

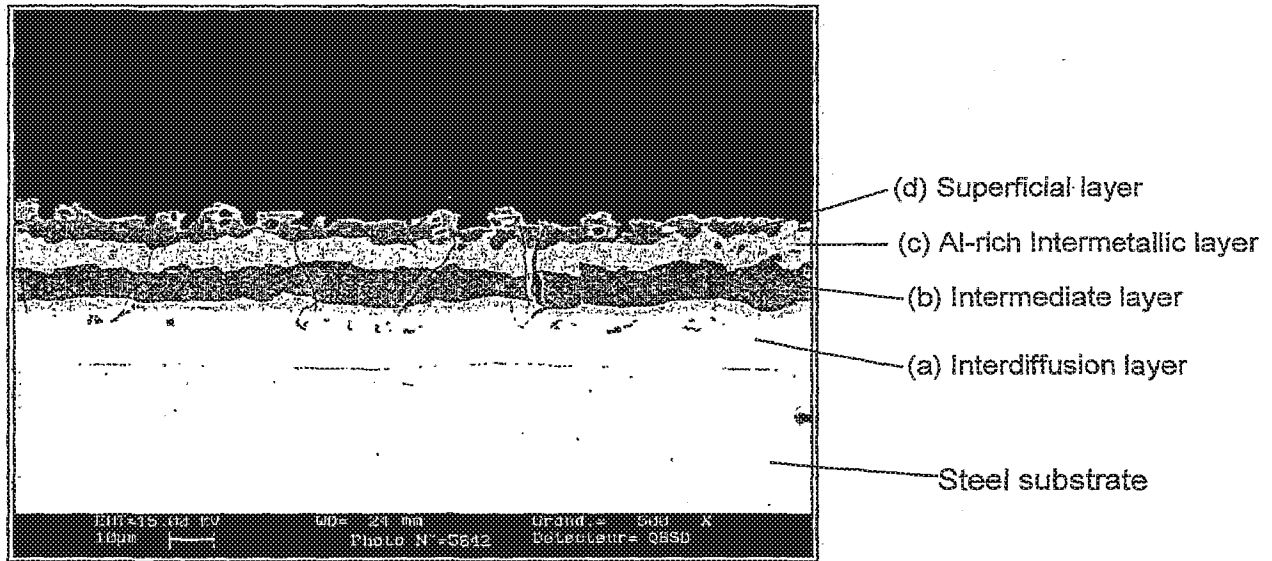


Fig. 1

10µm

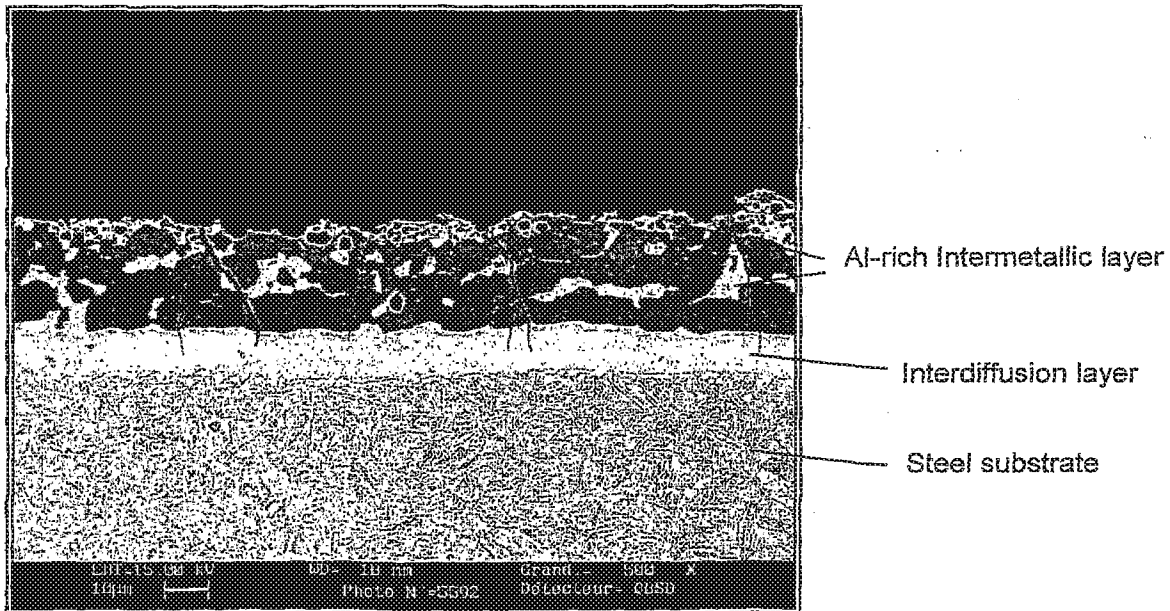


Fig.2

10µm

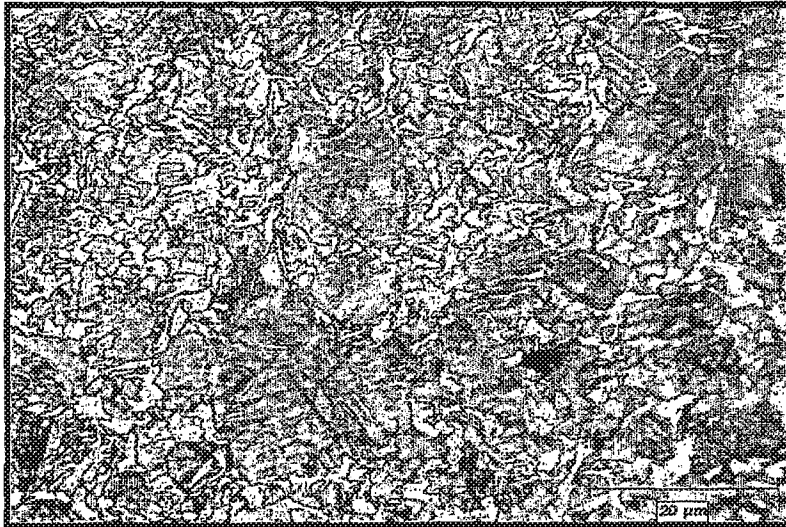


Fig. 3

10µm

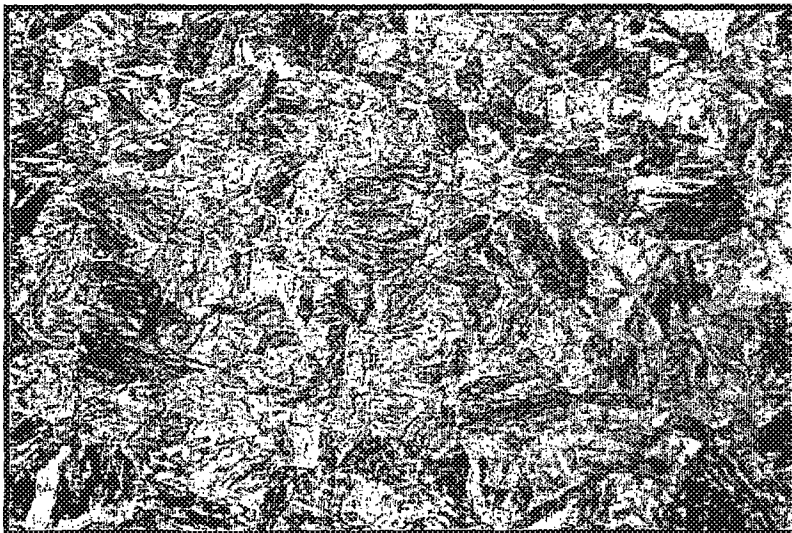


Fig. 4

10 µm

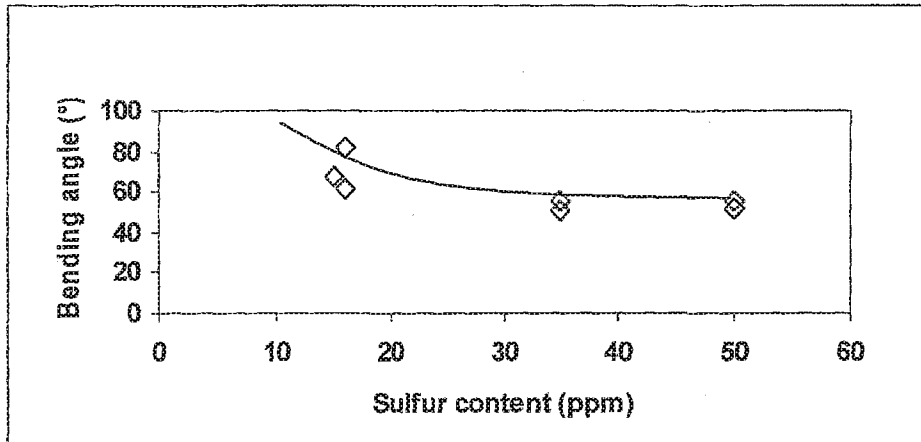


Fig. 5

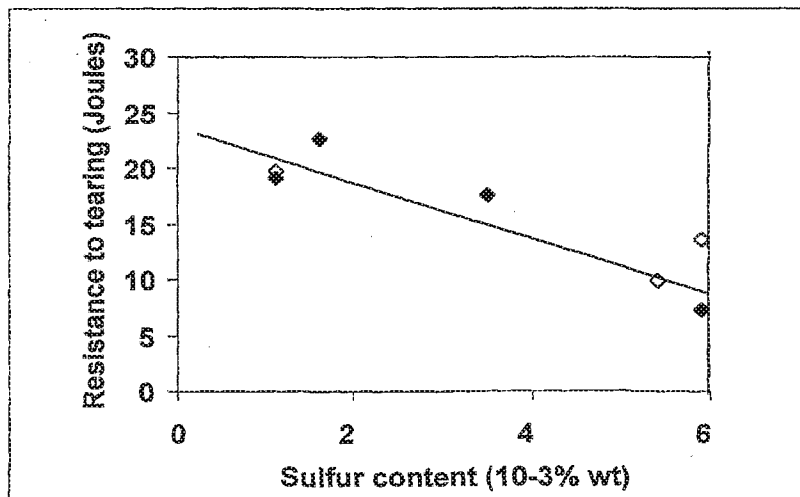


Fig. 6

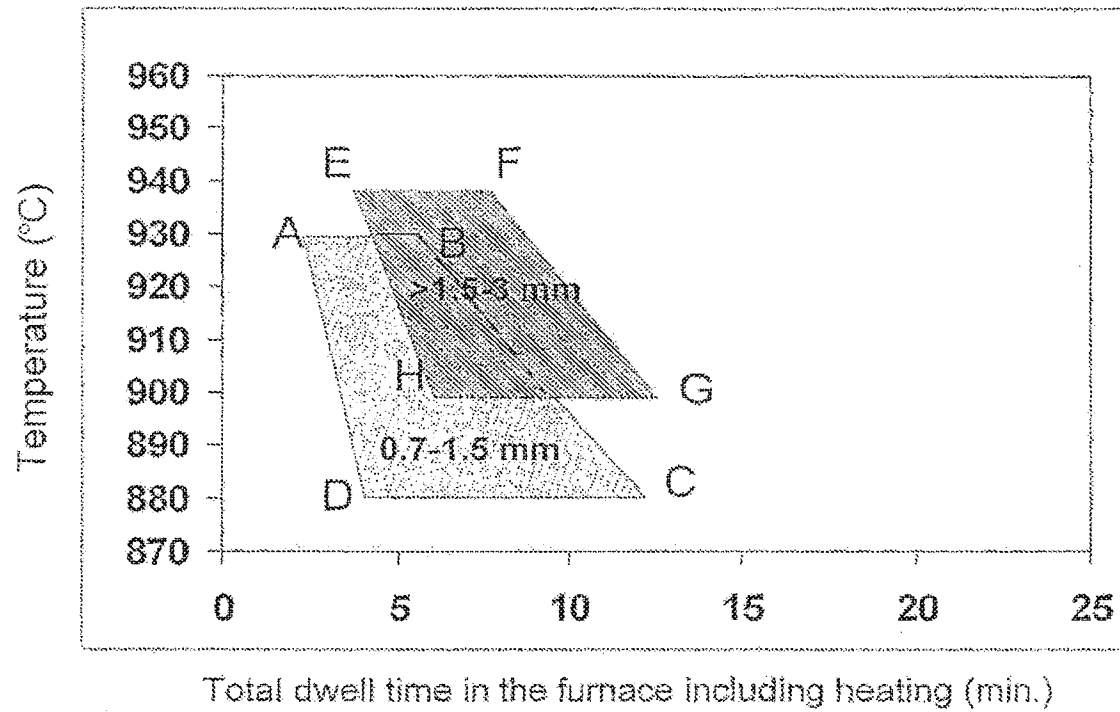


Fig. 7

## ABSTRACT

A pre-coated steel strip is provided. The steel strip includes a strip of base steel having a length, a width, a first side, and a second side. The length of the strip is at least 100m and the width is at least 600mm. An aluminum or an aluminum alloy pre-coating is on at least part of at least one of the first or second sides of the strip of base steel. A thickness  $t_p$  of the pre-coating is from 20 to 33 micrometers at every location on at least one of the first or second sides. Processes, coated stamped products and land motor vehicles are also provided.

What is claimed is:

1. A pre-coated steel strip, comprising:

- (a) a strip of base steel having a length, a width, a first side, and a second side;
  - (b) said length of said strip of base steel being at least 100m and said width being at least 600mm, and
  - (c) an aluminum or an aluminum alloy pre-coating on at least part of one of said first and second sides of said strip of base steel,
- wherein a thickness  $t_p$  of said pre-coating is from 20 to 33 micrometers at every location on at least one of said first and second sides.

2. A method for making a pre-coated steel strip according to claim 1, wherein the pre-coated steel strip has a thickness of not greater than 3 mm.

3. A process for making a hot stamped coated steel sheet product, comprising:

(A) heating an aluminum- or aluminum alloy pre-coated steel sheet in a furnace preheated to a temperature and during a time defined by trapezoid ABCD having heating time, temperature coordinates of about A (3 minutes, 930°C), B (6 minutes, 930 °C), C (13 minutes, 880 °C) and D (4.5 minutes, 880°C) if thickness of said sheet is greater than or equal to 0.7mm and less than or equal to 1.5mm, and by trapezoid EFGH having heating time, temperature coordinates of about E (4 minutes, 940°C), F (8 minutes, 940°C), G (13 minutes, 900°C), and H (6.5 minutes, 900°C) if thickness of said sheet is greater than 1.5mm and less than or equal to 3mm, at a heating rate  $V_c$  between 20 and 700°C comprised between 4 and 12°C/s, to obtain a heated blank;

(B) transferring said heated blank to a die; and

(C) stamping said heated blank in said die, to thereby obtain a hot stamped steel sheet product,

wherein the said heated product is cooled at a rate of at least 50°C/s during an interval between when said product exits said furnace and when said product reaches 400°C.

4. A process according to claim 3, wherein said heated blank is deformed by a quantity higher than 10% during said stamping.
5. A process according to claim 3, wherein the elapsed time between said heated blank exits said furnace and said stamping commences is not more than 10 seconds.
6. A process for making a hot stamped coated steel sheet product, comprising : hot dip pre-coating a steel strip having a first side and a second side with aluminum or aluminum alloy to obtain a pre-coated steel strip, the pre-coating thickness on at least one of said first side and said second side of said strip having a thickness of from 20 to 33  $\mu\text{m}$  at every location on at least one of said first and second sides of said strip;  
cutting said pre-coated steel strip to obtain a sheet;  
heating the sheet in a pre-heated furnace to obtain a heated blank in which the steel is austenitic;  
transferring said heated blank to a die while maintaining said steel in the austenitic phase;  
and  
stamping said heated blank in a die to obtain a hot stamped steel sheet product in which the steel microstructure is martensitic.
7. A hot stamped coated steel sheet product, which is prepared by a process comprising:  
(A) providing a pre-coated strip according to claim 1, said strip being pre-coated with aluminum or aluminum alloy ;  
(B) cutting said pre-coated steel strip to a predetermined size and shape to obtain a sheet ;  
(C) heating said steel sheet in a furnace preheated to a temperature and during a time defined by trapezoid ABCD having heating time, temperature coordinates of about A (3 minutes, 930°C), B (6 minutes, 930 °C), C (13 minutes, 880 °C) and D (4.5 minutes, 880°C) if thickness of said product is greater than or equal to 0.7mm and less than or equal to 1.5mm, and by trapezoid EFGH having heating time, temperature coordinates of about E (4 minutes, 940°C), F (8 minutes, 940°C), G (13 minutes, 900°C), and H (6.5 minutes, 900°C) if thickness of said product is greater than 1.5mm and less than or equal to 3mm, to obtain a heated blank;  
(D) transferring said heated blank to a die; and

(E) stamping said heated blank in said die, to thereby obtain a hot stamped steel sheet product,

wherein the elapsed time between said heated blank exits said furnace and said stamping commences is not more than 10 seconds, and wherein said heated product cools at a rate higher than 30°C/s during said stamping.

8. A process for making a hot stamped coated steel sheet product, comprising:

(A) providing a heated blank according to claim 7 ;

(B) transferring said heated blank to a die; and

(C) stamping said heated blank in said die, to thereby obtain a hot stamped steel sheet product,

wherein the elapsed time between said heated blank exits said furnace and said stamping commences is not more than 10 seconds, and wherein said heated product cools at such a rate during said stamping that the steel microstructure is martensitic.

9. The pre-coated steel strip according to claim 1, wherein the base steel comprises the following components by weight based on total weight:

0.15%<carbon<0.5%;

0.5%<manganese<3%;

0.1 %<silicon<0.5%;

0.01 %<chromium<1%;

titanium<0.2%;

aluminum<0.1%;

phosphorus<0.1 %;

sulfur<0.05%;

0.0005%<boron<0.08%;

iron; and

impurities inherent in processing.

10. The pre-coated steel strip according to claim 9, wherein the base steel comprises the following components by weight based on total weight:

0.20%<carbon<0.5%;  
0.8%<manganese<1.5%;  
0.1%<silicon<0.35%;  
0.01%<chromium<1%;  
titanium<0.1%;  
aluminum<0.1%;  
phosphorus<0.05%;  
sulfur<0.03%;  
0.0005%<boron<0.01%,  
iron;  
and impurities inherent in processing.

11. The pre-coated steel strip according to claim 9, wherein the base steel comprises 20 ppm or less of sulfur.

12. The pre-coated steel strip according to claim 9, wherein the ratio of titanium to nitrogen in the steel sheet in weight % is in excess of 3.42.

13. The pre-coated steel strip according to claim 1, wherein the aluminum or aluminum alloy pre-coating comprises from 8% to 11% silicon by weight, from 2% to 4% iron by weight, the remainder being aluminum and impurities inherent in processing.

14. The pre-coated steel strip according to claim 9, wherein the aluminum or aluminum alloy pre-coating comprises from 8% to 11% silicon by weight, from 2% to 4% iron by weight, the remainder being aluminum and impurities inherent in processing.

15. A coated steel stamped product, which comprises:

- (a) a strip of base steel having a first side and a second side; and
- (b) a coating on at least one of said first side of said strip of base steel and said second side of said strip of base steel, wherein:

(i) said coating results from the interdiffusion between said base steel and aluminium or aluminium alloy pre-coating, and

(ii) said coating comprises, proceeding from base steel outwards,

(1) Interdiffusion layer

(2) Intermediate layer

(3) Intermetallic layer

(4) Superficial layer.

wherein said coating has a thickness greater than 30 micrometers

16. A coated steel stamped product according to claim 15, wherein said layer (1) has a thickness less than 15 micrometers

17. A coated steel stamped product according to claim 15, wherein the said layers (3) and (4) are occupying at least 90% of the level corresponding to the considered said layers and wherein less than 10% of layer (3) is present at the extreme surface of the product

18. A coated steel stamped product according to claim 15, wherein the steel in the strip has a composition according to claim 9.

19. A coated steel stamped product according to claim 15, wherein the steel in the strip has a composition according to claim 10.

20. A coated steel stamped product according to claim 15, wherein the steel has a composition according to claim 11.

21. A coated steel stamped product according to claim 15, wherein the steel has a composition according to claim 12.

22. A coated steel stamped product according to claim 15, the aluminum or aluminum alloy pre-coating comprises from 8% to 11% silicon by weight, from 2% to 4% iron by weight, the remainder being aluminum and impurities inherent in processing.

23. A land motor vehicle comprising the heat treated coated steel product according to claim 7 or 15.

24. A land motor vehicle comprising the heat treated coated steel product produced according to claim 3, 6 or 8.

## Coated Steel Strips, Coated Stamped Products and Methods

### CROSS REFERENCE TO RELATED APPLICATIONS

[0000.1] This is a Continuation of U.S. Patent Application No. 13/621,015, filed September 15, 2012 which is a Divisional of U.S. Patent Application No. 12/447,777, filed April 29, 2009, which is a National Phase Application of International Patent Application PCT/IB06/004019, filed October 30, 2006. All of the above patent applications are hereby incorporated by reference herein.

[0001] The present invention relates to, among other things, coated steels, methods of making such coated steels, including hot dipping, methods of using such coated steels, stamping blanks prepared from coated steels, stamped products prepared from coated steels, and to various uses of the invention products such as in spot welding, etc.

[0002] Additional advantages and other features of the present invention will be set forth in part in the description that follows and in part will become apparent to those having ordinary skill in the art upon examination of the following or may be learned from the practice of the present invention. The advantages of the present invention may be realized and obtained as particularly pointed out in the appended claims. As will be realized, the present invention is capable of other and different embodiments, and its several details are capable of modifications in various obvious respects, all without departing from the present invention. The description is to be regarded as illustrative in nature, and not as restrictive.

### BACKGROUND OF THE INVENTION

[0003] In recent years the use of pre-coated steels in hot-stamping processes for the shaping of parts has become important, especially in the automotive industry. Fabrication of such parts may include the following main steps:

[0004] Pre-coating of a steel sheets, by hot dipping;

[0005] Trimming or cutting for obtaining blanks;

[0006] Heating the blanks in order to obtain alloying of the steel substrate with the pre-coating, as well as the austenitizing of the steel; and

[0007] Hot forming followed by rapid cooling of the part in order to obtain predominantly martensitic structures. See for Example U.S. Pat. No. 6,296,805, incorporated herein by reference.

[0008] Thanks to an alloying of the pre-coating with the steel substrate, which has the effect of creating intermetallic alloys with high melting temperature, the blanks having such coating may be heated in a temperature range where austenitizing of the metallic substrate takes place, allowing further hardening by quenching.

[0009] Heat treatments of the blanks in view of the intermetallic alloying of the coating and austenitizing of the substrate are most frequently performed in furnaces, where blanks are traveling on rollers. The thermal cycles experienced by the blanks include first a heating phase whose rate is a function of parameters such as blank thickness, furnace temperature, traveling speed, and coating reflectivity. After this heating phase, thermal cycles generally include a holding phase, whose temperature is the regulation temperature of the furnace. Problems however are experienced with the furnace operation: the rollers may become fouled by metallic deposits which come from the pre-coating of the blanks. If these deposits are excessive, maintenance of the rollers has to be performed and productivity is decreased.

[0010] Parts obtained after heating and rapid cooling display very high mechanical resistance and may be used for structural applications, for example for automotive industry applications. These parts must be frequently welded with others and high weldability is required. This means that:

[0011] The welding operation should be performable in a sufficiently wide operating range in order to guarantee that an eventual drift of the nominal welding parameters has no incidence on weld quality. For resistance welding, which is very common in the automotive industry, an operating welding range is defined by the combination of parameters: welding current intensity  $I$  and force  $F$  applied of the parts during welding being among the most important. A proper combination of these parameters helps to ensure that insufficient nugget diameter is not obtained (caused by too low intensity or too low force) and that no weld expulsion occurs.

[0012] The welding operation should also be performed in such a way that high mechanical resistance is obtained on the weld. This mechanical resistance may be evaluated by tests such as by shear-tensile tests or cross-tensile tests.

## SUMMARY OF THE INVENTION

[0013] There remains a need for coated steels which may be conveniently used to prepare shaped parts by a stamping process. There also remains a need for coated steels which may be used to prepare shaped parts by a stamping process which are suitable for welding. There also remains a need for processes for preparing such coated steels and stamped parts.

[0014] The inventors have discovered that certain coated steels in which a base steel strip is at least partially coated (sometimes termed "pre-coated," this prefix indicating that a transformation of the nature of the pre-coating will take place during heat treatment before stamping) on at least one side with a coating of either aluminum or an aluminum alloy and in which the coating has a defined thickness and is preferably substantially uniform, are conveniently formed into shaped parts after heating by stamping and are conveniently welded. In addition, the inventors have discovered that the roller fouling problem described above generally arises from an insufficient degree of intermetallic alloying between the substrate and the metallic pre-coating. Furthermore, it was discovered that the location of the fouling of the rollers corresponds to zones of the blanks in contact with the rollers where the metallic pre-coating thickness locally exceeds the average thickness. While not bound by a particular theory, it is believed that if a pre-coating is locally too thick, intermetallic alloying is insufficient and the pre-coating melts, fouling the rollers. Thus, the inventors have discovered that the control of the homogeneity of the pre-coating thickness over the entire sheet within given tolerances is an important factor for obtaining the desired degree of intermetallic alloying, allowing one to improve the resistance to the subsequent melting of the coating during travel on rollers.

[0015] The inventors have also discovered that particularly good weldability of aluminized and hot stamped parts is associated with a special succession of coating layers on the parts, proceeding from steel substrate outwards.

[0016] The inventors have also discovered that a specific combination of transfer time between the heating furnace and the stamping die, the amount of deformation during stamping, the temperature of stamping, the cooling rate of the product during stamping, leads to the fabrication of a part with a fully homogeneous martensitic structure and that an increase in ductility or energy absorption of the parts after stamping is obtained by a reduction of sulfur below a critical value, these two benefits being obtained with or without the invention aluminum/aluminum alloy coating, and with other coatings.

[0017] Accordingly, and in view of the above, it is one object of the present invention to provide novel pre-coated steels strips which may be conveniently processed into stamping blanks.

[0018] It is another object of the present invention to provide novel pre-coated steels strips or sheets which may be conveniently formed into parts by stamping.

[0019] It is another object of the present invention to provide novel coated steels which may be conveniently formed into parts by hot stamping.

[0020] It is another object of the present invention to provide novel methods for making such a coated steel.

[0021] It is another object of the present invention to provide novel stamping blanks which are prepared from such a coated steel.

[0022] It is another object of the present invention to provide novel methods of making such stamping blanks.

[0023] It is another object of the present invention to provide novel stamped parts which are prepared from such a coated steel.

[0024] It is another object of the present invention to provide novel methods of making such stamped parts.

[0025] It is another object of the present invention to provide novel articles of manufacture, such as a motor vehicle, which contain such stamped parts.

[0026] It is another object of the present invention to provide novel stamped parts.

[0027] It is another object of the present invention to provide novel methods of making welded stamped parts.

[0028] It is another object of the present invention to provide novel articles of manufacture, such as a motor vehicle, which contain such welded stamped parts.

[0029] It is another object of the present invention to provide novel welded coated steels and welded stamping blanks.

[0030] It is another object of the present invention to provide novel methods of making such welded coated steels and welded stamping blanks.

[0031] These and other objects, which will become apparent during the following detailed description.

#### BRIEF DESCRIPTION OF THE DRAWINGS

[0032] FIG. 1 illustrates a preferred embodiment of an invention coated part, after heat treatment and stamping. The succession of layers of the coating on the steel substrate is: (a) interdiffusion layer; (b) intermediate layer; (c) Intermetallic layer; and (d) superficial layer. This arrangement is particularly favorable for the further welding of the part.

[0033] FIG. 2 illustrates a coating of a steel substrate after heat treatment and stamping which does not correspond to the invention. This succession of layers (interdiffusion layer and intermetallic layer) yields inferior results in resistance spot welding.

[0034] FIG. 3 illustrates a microstructure of a steel part, hot stamped and cooled under

conditions not according to the invention.

[0035] FIG. 4 illustrates the microstructure of a steel part, hot stamped and cooled according to a preferred set of conditions according to the invention.

[0036] FIG. 5 illustrates the influence of sulfur on the bending angle of parts after hot stamping.

[0037] FIG. 6 illustrates the influence of sulfur on the initiation energy of fracture of parts after hot stamping.

[0038] FIG. 7 shows conditions of furnace temperature as a function of the total dwell time in the furnace for sheets of total thicknesses of from 0.7-1.5 and 1.5-3 mm that provide particularly favorable coatings for welding.

#### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0039] As noted above, the inventors' discovery that certain coated steels, in which a base steel is at least partially pre-coated on at least one side with a coating of either aluminum or an aluminum alloy and in which the pre-coating has a defined thickness and is substantially uniform, are conveniently formed into shaped parts by stamping, forms one basis for the invention.

[0040] In the context of the present invention, the terms first side (or side 1) and second side (or side 2) of the strip or sheet, etc., of base steel refer to the two large, opposite-facing, surfaces which have a surface area defined by the length and width of the strip of base steel. In contrast, the side edges of the strip of base steel are the two small, opposite-facing, surfaces which have a surface area defined by the length and thickness of the strip. The top and bottom edges of the strip of base steel are the two small, opposite-facing, surfaces which have a surface area defined by the width and thickness of the strip. In the following,  $t_p$  designates the thickness of the pre-coating, at any considered location on the sides 1 and 2 of a sheet or a blank. In particular, in the case of sheets coated on two sides 1 and 2,  $t_{p1}$  stands for the thickness on side 1, and  $t_{p2}$  for the thickness on side 2.

According to a highly preferred embodiment  $t_p$  is controlled in a precise range, expressed by ( $t_{pmin}$ ,  $t_{pmax}$ ), in order to improve the resistance to the fouling of the rollers. The thickness is preferably controlled, both in the longitudinal (or rolling) direction of the strip or sheet, as well in the transverse direction.

[0041] In relation to the problem of the fouling of rollers, the control of the pre-coating thickness on the side of the sheet or blank which is directly in contact with the rollers is especially important. Because different operations can follow the step of coating the steel sheet (for example hot-dip-coating which provides coated sides 1 and 2), it is preferable to carefully control the pre-coating on both sides of the sheet. For example, after any of coiling, handling, cutting, punching, etc., sides 1 and 2 may be not readily identifiable. But when control of  $t_p$  is effected on the two sides of the sheet that has been coated (first side and second side) it is not necessary to track sides 1 and 2 as neither side will foul the roller. In addition, it is not necessary to trim the sheet in order to obtain a preferred smaller sheet having a more uniform homogeneity of pre-coating thickness, thus providing a sheet that is pre-coated by, e.g., hot-dipping. In other words, important benefits are obtained when one controls the pre-coating minimum and maximum thickness of the first side ( $t_{pmin1}$ ,  $t_{pmax1}$ ) and the pre-coating minimum and maximum thickness of the second side ( $t_{pmin2}$ ,  $t_{pmax2}$ ) of the subject steel sheet or blank.

[0039.1] Hot-dip coated steels are the preferred steels herein. However, regardless of the coating method, the pre-coating thickness on one or both sides of the sheet can be measured and monitored continuously on a coating line directly after the coating operation. This may be realized by devices known per se, such as thickness gages relying on X-Ray absorption. At every moment, the measurement of thickness at a given location may be performed for example on an area of a few hundreds of mm.<sup>sup.2</sup>, this representing the dimension of the zone irradiated by X-Ray.

[0042] In a preferred embodiment, a plurality of such devices are positioned at different distances in the transverse direction of the strip in order to obtain a profile of the thickness of the pre-coating along the strip width.

[0043] The inventors have discovered that the resistance to the pollution or fouling of the rollers in the furnaces is improved when the minimum and maximum thickness respectively of at least one of the first side ( $t_{pmin1}$ ,  $t_{pmax1}$ ) and the second side ( $t_{pmin2}$ ,  $t_{pmax2}$ ) are respectively equal to 20 and 33 micrometers (a micrometer is the same as a micron and is a metric unit of length equal to one millionth of a meter). In other words, in a preferred embodiment, at every location on at least one face of the sheet or the blank, the thickness  $t_p$  of the pre-coating is preferably from 20-33 micrometers in thickness, including 21, 22, 23, 24, 25, 26, 27, 28, 29, 30, 31 and 32 micrometers and all ranges and subranges therebetween, and including all numbers between each listed value as if written out in its entirety (e.g., 22.34 micrometers). For hot-dip coating, precise control of this range of pre-coating thickness can be realized on an operating line for example using a system of nozzles blowing gas after coating, for example after exit of the strip or sheet from a bath, and by the flatness of the strip. Number, geometry and location of the nozzles, and flow rates are among the main parameters for a precise control of the thickness  $t_p$ . Given the present disclosure, one of ordinary skill in this art can control the pre-coating thickness as described herein without undue labor.

[0044] The invention refers to strips produced in industrial conditions, i.e. where control of pre-coating thickness is effective over a wide surface strip, i.e. with a length greater than 100 m, and with a width greater than 600 mm. In such a way, blanks cut or trimmed out these strips display very high homogeneity of pre-coating thickness, and the settings of the heating treatments in the furnace do not have to be changed for being adapted to eventual variations of this thickness.

[0045] While not bound by a particular theory of operation, the inventors believe that several of the benefits of the invention are related to this pre-coating thickness range, such as the following:

[0046] For a pre-coating thickness less than 20 micrometers, the alloyed layer which is formed during the heating of the blank has an insufficient roughness. Thus, the adhesion of subsequent painting is low on this surface, and the corrosion resistance is decreased.

[0047] If the pre-coating thickness is more than 33 micrometers at a given location on a sheet,

the risk is that the difference of thickness between this location and some other locations where the pre-coating is thinner, becomes too important: The settings of the heating treatment in the furnace may be adapted to the thinner value of the pre-coating, but not to the thicker one. Thus, the alloying reaction forming the intermetallic alloy may take place to an insufficient degree since the mean diffusion distance of the elements in the pre-coating becomes significantly less than the local value of the pre-coating thickness. As a matter of results, alloying will become much more difficult in the external (or superficial) part, particularly in the case of high heating rate.

[0048] Thus, in a first embodiment, the present invention provides certain coated steel strips, which comprise a strip of base steel and a pre-coating of aluminum or an aluminum alloy on at least a part of one side of the strip of the base steel. For many applications, the strip of base steel may comprise any type of steel which may be coated with either aluminum or an aluminum alloy. However, for certain applications, such as a structural part of an automobile, it is preferred that the strip of base steel comprise an ultra high strength steel (UHSS). In such cases, it is particularly preferred that the strip of base steel comprises a boron steel.

[0049] The inventors have also discovered that good welding results are achieved if the coating obtained on parts made out of blanks having undergone intermetallic alloying, austenitizing and hot stamping, displays distinctive features. It must be pointed out that this coating is different from the initial pre-coating, since the thermal treatment causes an alloying reaction with the steel substrate which modifies both the physico-chemical nature and the geometry of the pre-coating: in this regard, the inventors have discovered that particularly good weldability of aluminized and hot stamped parts is associated with the following succession of coating layers on the parts, proceeding from steel substrate outwards:

[0050] (a) Interdiffusion layer,

[0051] (b) Intermediate layer,

[0052] (c) Intermetallic layer,

[0053] (d) Superficial layer See, e.g., FIG. 1. In a preferred embodiment these layers are as follows:

[0054] (a) Interdiffusion layer, preferably with medium hardness (e.g., HV50g between 290 and 410, HV50g designating the hardness measured under a load of 50 grams) In a preferred

embodiment this layer has the following composition, by weight: 86-95% Fe, 4-10% Al, 0-5% Si

[0055] (b) Intermediate layer (HV50g around 900-1000 e.g., +/-10%)) In a preferred

embodiment this layer has the following composition, by weight: 39-47% Fe, 53-61% Al, 0-2% Si

[0056] (c) Intermetallic layer, with hardness HV50g around 580-650, e.g., +/-10%) In a preferred

embodiment this layer has the following composition, by weight: 62-67% Fe, 30-34% Al, 2-6% Si

[0057] (d) Superficial layer (HV50g around 900-1000 e.g., +/-10%)) In a preferred embodiment

this layer has the following composition, by weight: 39-47% Fe, 53-61% Al, 0-2% In a preferred

embodiment the total thickness of layers (a) to (d) is greater than 30 micrometers. In another

preferred embodiment, the thickness of layer (a) is less than 15 micrometers, for example 14, 12,

10, 8, 6, 4, 2, or 1 micrometers, and all whole numbers, ranges and subranges therebetween, and

including all numbers between each listed value as if written out in its entirety (e.g., 13.84

micrometers).

[0058] The inventors have discovered that high weldability is especially obtained when layers (c)

and (d) are essentially continuous (i.e.: occupying at least 90% of the level corresponding to the

considered layer) and when less than 10% of layer (c) is present at the extreme surface of the

part. Without being bound by a theory, it is thought that this particular layer disposal, in

particular layer (a) and, layers (c) and (d) influence the resistivity of the coating both by their

intrinsic characteristics and by the effect of roughness. Thus, current flow, heat generation at the

surfaces, and nugget formation in the initial stage of spot welding are affected by this particular

arrangement. This favorable layer disposition is obtained for example when aluminum- or

aluminum alloy pre-coated steel sheets, whose thickness range from, e.g., 0.7 to 3 mm, are

heated for 3 to 13 minutes (this dwell time includes the heating phase and the holding time) in a

furnace heated to a temperature of 880 to 940° C. Other conditions leading to such favorable

layer dispositions are found in FIG. 7:

[0059] For sheets of total thicknesses greater or equal to 0.7 mm, and less than or equal to 1.5

mm, the preferred treatment conditions: (furnace temperature, total dwell time in the furnace) are illustrated in FIG. 7 by conditions lying within the limits of diagram "ABCD"

For sheets of total thicknesses greater than 1.5 mm, and less than or equal to 3 mm, the preferred treatment conditions: (furnace temperature, total dwell time in the furnace) are illustrated in FIG. 7 by diagram "EFGH". The heating rate  $V_c$  is comprised between 4 and 12° C./s for producing a favorable alloyed layer disposition. In this regard, this "heating rate" reflects the temperature rise which is experienced by the pre-coated steel upon being located in the preheated furnace.  $V_c$  is defined as the mean heating rate between 20 and 700° C. The inventors have discovered that the control of  $V_c$  in this precise range is a key factor, because it controls directly the nature and the morphology of the alloyed layers which are formed. It is here underlined that the heating rate  $V_c$  is different from the mean heating rate, which is the heating rate between room temperature and furnace temperature. The rates of 6, 7, 8, 9, 10, and 11° C./s are included as are all numbers, ranges and subranges therebetween, and including all numbers between each listed value as if written out in its entirety (e.g., 7.7° C./s). In this regard, all of the conditions specified within FIG. 7 are incorporated herein by reference thereto. Particularly preferred conditions are:

[0060] (for thicknesses of 0.7-1.5 mm)

[0061] 930° C., from 3 minutes up to 6 minutes;

[0062] 880° C., from 4 minutes 30 seconds up to 13 minutes (for thicknesses of 1.5 to 3 mm)

[0063] 940° C., from 4 minutes up to 8 minutes;

[0064] 900° C., from 6 minutes 30 seconds up to 13 minutes.

[0064.1] A special advantage arises from pre-coatings whose thickness is comprised between 20 and 33 micrometers, since this thickness range yields favorable layer disposal, and since the homogeneity of the pre-coating thickness is associated to an homogeneity of the coating formed after alliation treatment.

[0065] Heated blanks are thereafter transferred to a die, hot stamped to obtain a part or product, and cooled at a rate of more than 30.degree. C./s. The cooling rate is defined here as the mean rate between the exit of the heated blank from the furnace, down to 400° C.

[0066] The strip of base steel is coated with either aluminum or an aluminum alloy.

Commercially pure aluminum is known in the art as Type 2 aluminum, while alloys of aluminum

with 5 to 11% by weight of silicon are known in the art as Type 1 aluminum. Silicon is present in order to prevent the formation of a thick iron-metallic intermetallic layer which reduces adherence and formability. Other alloying elements useful with aluminum herein include iron, between 2.5 and 3% by weight, and calcium, between 15 and 30 ppm by weight, including combinations of two or more thereof with aluminum.

[0067] A typical metal bath for an Al--Si coating generally contains in its basic composition by weight, from 8% to 11% silicon, from 2% to 4% iron, the remainder being aluminum or aluminum alloy, and impurities inherent in processing. Typical composition of Al--Si coating is: Al-9.3% Si-2.8% Fe. Invention coatings are not limited to these compositions, however.

[0068] The strip of base steel used herein may be any which can be coated by a conventional coating technique. For example, the strip of base steel may be any hot rolled strip, such as those prepared by hot rolling a steel slab (either with or without subsequent cold rolling). Typically, the strip of base steel will be stored and transported in the form of a coil both before and after the formation of the coating.

[0069] An example of a preferred steel for the strip of base steel is one having the following composition by weight:

[0070] 0.10%<carbon<0.5%

[0071] 0.5%<manganese<3%

[0072] 0.1%<silicon<1%

[0073] 0.01%<chromium<1%

[0074] titanium<0.2%

[0075] aluminum<0.1%

[0076] phosphorus<0.1%

[0077] sulfur<0.05%

[0078] 0.0005%<boron<0.010%, the remainder comprising, consisting essentially of, or consisting of iron and impurities inherent in processing. Use of such a steel provides a very high mechanical resistance after thermal treatment and the aluminum-based coating provides a high resistance to corrosion.

[0079] Particularly preferably, the composition by weight of the steel in the strip of base steel is the following:

[0080] 0.15%<carbon<0.25%

[0081] 0.8%<manganese<1.8%

[0082] 0.1%<silicon<0.35%

[0083] 0.01%<chromium<0.5%

[0084] titanium<0.1%

[0085] aluminum<0.1%

[0086] phosphorus<0.1%

[0087] sulfur<0.05%

[0088] 0.002%<boron<0.005%, the remainder comprising, consisting essentially of, or consisting of iron and impurities inherent in processing.

[0089] A preferred strip herein is 100 m long and 600 mm wide. Preferred thicknesses are 0.7 to 3 mm.

[0090] Even more preferably, in the composition by weight of the sheet, the weight ratio of titanium content with respect to the nitrogen content is in excess of 3.42, believed to be a level at which the boron is no longer able to combine with the nitrogen.

[0091] An example of preferred commercially available steel for use in the strip of base steel is 22MnB5.

[0092] Chromium, manganese, boron and carbon may be added, in the composition of the steel according to the invention, for their effect on hardenability. In addition, carbon makes it possible to achieve high mechanical characteristics thanks to its effect on the hardness of the martensite.

[0093] Aluminum is introduced into the composition, to perform deoxidation in the liquid state and to protect the effectiveness of the boron.

[0094] Titanium, the ratio of the content of which with respect to the nitrogen content should be in excess of 3.42, is introduced for example in order to prevent combining of the boron with the nitrogen, the nitrogen being combined with titanium.

[0095] The alloying elements, Mn, Cr, B, make possible a hardenability allowing hardening in the stamping tools or the use of mild hardening fluids limiting deformation of the parts at the time of thermal treatment. In addition, the composition according to the invention is optimized from the point of view of weldability.

[0096] The steel in the sheet may undergo a treatment for globularization of sulfides performed with calcium, which has the effect of improving the fatigue resistance of the sheet.

[0097] As mentioned above, ultra-high-strength can be provided with the steel sheet coated and hot stamped according to the invention. This high level of strength is sometimes associated with a limited ductility. In applications requiring a higher ductility, in particular when an ability for bending is required from a part or a product, the inventors have discovered that increased ductility can be obtained if sulfur is particularly controlled: when the sulfur level of base steel is lower than or equal to 0.002% (20 ppm), the bending angle can be greater than 60° and enhanced ductility and tearing resistance are obtained on the parts having experienced heat treatment and stamping. Preferred levels include 20, 18, 15, 13, 10, 8, 5, 2, etc ppm sulfur. In fact, this benefit applies to steels in general, and is not limited to coated steels or to steels coated with Al or Al alloy coatings. While not bound by a particular theory, when analyzing causes of premature failure of some parts in bending operations, the inventors observed that failure initiated on sulfide inclusions. It is thus believed that decohesions between inclusions and the martensitic or bainite-martensitic matrix act as stress-concentration factors and trigger further crack propagation in the ductile mode.

[0098] The invention also concerns a process for producing a part starting from an invention coated sheet, then cut into a blank which, after shaping, the coating of the blank is subjected to an increase in temperature at a speed in excess of 4° C./second, but lower than 12° C./second. The heating rate  $V_c$  is defined as the mean rate between 20 and 700° C.

[0099] The invention also concerns the use of a hot-rolled steel sheet which then can be cold-rolled and coated, for structural and/or anti-intrusion or substructure parts for a land motor vehicle, such as, for example, a bumper bar, a door reinforcement, a wheel spoke, etc.

[0100] The sheet according to the invention described above can derive, by reason of its processing, from a hot-rolling mill, and possibly may be cold-rerolled again depending on the final thickness desired. It then is coated with an aluminum-based coating, for example by dipping in a bath containing, in addition to the aluminum source/alloy, e.g., from 8% to 11% silicon and from 2% to 4% iron, the sheet having a high mechanical resistance after thermal treatment and a high resistance to corrosion, as well as a good capacity for painting and gluing.

[0101] The coating is preferably controlled as above, and has in particular the function of protecting the basic sheet against corrosion in various conditions. The thermal treatment applied at the time of a hot-forming process or after forming makes it possible to obtain high mechanical characteristics which can exceed 1500 MPa for mechanical resistance and 1200 MPa for yield stress. The final mechanical characteristics are adjustable and depend in particular on the martensite fraction of the structure, on the carbon content of the steel and on the thermal treatment. At the time of thermal treatment performed on a finished part or at the time of a hot-shaping process, the coating forms a layer having a substantial resistance to abrasion, wear, fatigue, shock, as well as a good resistance to corrosion and a good capacity for painting and gluing. The coating makes it possible to avoid different surface-preparation operations such as for steel sheets for thermal treatment not having any coating.

[0102] The steel sheet can be pre-coated by dipping, after pickling, in an aluminum bath containing for example only aluminum or either aluminum and from 8% to 11% silicon and 2% to 4% iron, or only from 2% to 4% iron, or even in an aluminum bath preferably containing from 9% to 10% silicon and 2% to 3.5% iron. The aluminum may be aluminum per se or an aluminum alloy.

[0103] In an example of implementation of a coating of the sheet by dipping in a metal bath containing an aluminum alloy comprising a proportion of approximately 90% aluminum, the

coating layer comprises a first alloy layer in contact with the surface of the steel. This layer, directly in contact with the surface of the sheet, is highly alloyed with iron.

[0104] A second coating layer, on top of the first, contains approximately 90% aluminum and may contain silicon and a small amount of iron, depending on the composition of the bath.

[0105] The first alloy layer may crack when the sheet is submitted to high strains during cold forming operations of the manufacture of parts.

[0106] According to the invention, after the forming of the part, the coating is subjected to an increase in temperature at a speed in excess of 4° C./second. This rise in temperature makes possible a rapid remelting of the aluminum which fills in the cracks generated by the operation of shaping of the part.

[0107] At the time of thermal treatment, the base coating, of aluminum for example, is transformed into a layer alloyed with iron and comprising different phases depending on the thermal treatment and having a considerable hardness which may exceed 600 HV50 g.

[0108] Another advantage of the invention lies in the fact that the diffusion of the iron in the coating is initiated at high temperature. One thus will have a better cohesion between coating and steel in the sheet. In another form of the invention, the thermal treatment may be performed locally, in heavily deformed zones.

According to the invention, the sheet, in the delivery state in a coil or in a sheet, the thickness of which may range between 0.25 mm and 15 mm, has good forming properties and a good resistance to corrosion as well as a good capacity for painting or gluing. Preferably, the steel sheet or blank has a thickness less than 3 mm, since the cooling rates that may be achieved after quenching are high and help to obtain martensitic structures.

[0109] The steel sheet, a coated product, has a substantial resistance to corrosion in the delivery state, during forming and thermal treatments as well as during usage of the finished part. The presence of the coating at the time of thermal treatment of the parts makes it possible to prevent

any decarburization of the base metal as well as any oxidation. That is an undeniable advantage, in particular in the case of hot forming. Furthermore, heating of the treated part does not require a furnace having a controlled atmosphere to prevent a decarburization.

[0110] Thermal treatment of the metal in the sheet comprising heating at a temperature ranging between  $A_{c1}$ , starting temperature of austenitic transformation when heating, for example  $750^{\circ}\text{C}$ . and  $1200^{\circ}\text{C}$ ., in a furnace, for a period which depends on the temperature to be reached and the thickness of the blank. The composition is optimized so as to limit the grain growth at the time of thermal treatment. If the structure sought is completely martensitic, the holding temperature should be in excess of  $A_{c3}$ , for example  $840^{\circ}\text{C}$ ., temperature of complete austenitic transformation. The temperature holding should be followed by a cooling adjusted to the final structure sought.

[0111] Blanks are thereafter transferred from the furnace to a stamping press. When the elapsed time between the exit of the blanks from the furnace and the introduction in the stamping press is more than 10 seconds, a partial transformation from austenite is susceptible to appear: if obtaining a full martensitic structure is desired, the transfer time between the exit of the furnace and stamping should be less than 10 s.

[0112] The inventors have also discovered that the obtaining of a fully martensitic structure is linked to the amount of deformation in the hot forming operation: the amount of local deformation caused by hot forming is closely linked to the shape of the part or product and may exceed locally 40 or 50% in some particular regions. The inventors found that, when the local strain exceeds a critical value of 10%, the cooling rate must be sufficiently high in order to get a total martensitic transformation. Otherwise, bainitic transformation can take place at a significant amount instead of martensitic transformation. Thus, the risk is that heterogeneous structure appears on parts with complex shape where some locations are much more deformed than others. In this respect, the inventors put into evidence that, on the locations of the parts where the forming strain is higher than 10%, the cooling rate must be increased beyond  $50^{\circ}\text{C./s}$  in order to guarantee full martensitic transformation. The cooling rate is defined as the mean rate between the exit of the heated blank from the furnace, down to  $400^{\circ}\text{C}$ .

[0113] But one may also seek to obtain ferrito-bainitic or ferrito-martensitic structures, by a heating at a temperature ranging between  $A_{c1}$ , for example  $750^{\circ}\text{C}$ . and  $A_{c3}$ , for example  $840^{\circ}\text{C}$ ., followed by an appropriate cooling. According to the level of resistance to be achieved and the thermal treatment applied, one or several of these constituents is/are present in variable proportions.

[0114] The modulation of thermal treatment parameters makes it possible to achieve, with a given composition, different levels of hot and cold sheet resistance according to the thickness sought. For the highest resistance levels, the structure is composed predominantly of martensite.

[0115] The steel is particularly suited to the production of structural and anti-intrusion parts.

[0116] The invention thus enables one to produce a hot- or cold-rolled steel sheet of a desired thickness, coated, and affording extensive forming possibilities and which, after thermal treatment performed on the finished part, makes it possible to obtain a mechanical resistance in excess of 1000 MPa, a substantial resistance to shocks, fatigue, abrasion and wear, while retaining a good resistance to corrosion as well as a good capacity for welding, painting and gluing.

[0117] The present invention is described by way of certain exemplary embodiments which are not intended to be limiting.

## EXAMPLES

### Example 1

[0118] In a first example of implementation, a cold rolled steel sheet, 1.9 mm thick, containing by weight 0.23% carbon, 1.25% manganese, 0.017% phosphorus, 0.002% sulfur, 0.27% silicon, 0.062% aluminum, 0.021% copper, 0.019% nickel, 0.208% chromium, 0.005% nitrogen, 0.038% titanium, 0.004% boron, 0.003% calcium-has been pre-coated with an aluminum-based alloy with composition 9.3% silicon, 2.8% iron, the remainder being aluminum and unavoidable

impurities. According to the conditions of fabrication, namely the settings of the blowing devices on the operating line, sheets of 120 m long and 650 mm wide with various thickness ranges were produced.

[0119] Sheet A (according to the invention): The thickness  $t_{p1}$  and  $t_{p2}$  on each side of the sheet was controlled to be within the range (20-33) micrometers, at every location of the two faces of the sheet, both in the longitudinal (or rolling) direction and in the transversal direction.

Measurement was performed continuously with thickness gages devices relying on X-Ray emission. At every moment, the spot of measurement of each gage was a circular zone of about 20 mm radius. The sheets were afterwards cut into blanks of 1.2.times.0.5 m.sup.2 of overall dimensions.

[0120] Sheet B (reference): On these sheets, the pre-coating thickness had a wider variability since the thickness  $t_{p1}$  and  $t_{p2}$  on the two sides of the sheet was comprised in the range (30-45) micrometers. Blanks cut out of these sheets exhibit the same pre-coating thickness.

[0121] The blanks were then submitted to heating in a furnace at  $T=920^{\circ}$  C. Heating time was 3 min, with 4 mn holding time. The microstructure is then fully austenitic. Blanks were thereafter transferred from the furnace to a stamping press. When the elapsed time between the exit of the blanks from the furnace and the transfer in the stamping press is more 10 seconds, a partial transformation from austenite was susceptible to appear, thus reducing the mechanical resistance of the stamped part.

The blanks were directly cooled afterwards without hot stamping in order to appreciate the eventual remelting of the coating. On series A, no melting of the pre-coating was found. Intermetallic alloying between the pre-coating and the steel substrate occurred completely. On series B, the pre-coating underwent mainly alloying, but some traces of remelting were found, particularly on the former thicker locations of the pre-coating. This partial remelting of the aluminium pre-coating contributes to the progressive fouling of the rollers in the furnace. The sheets according to the invention do not contribute to this progressive buildup on the rollers.

## Example 2

[0122] i) Conditions according to the invention: In a second example of implementation, a cold rolled steel sheet, 1.2 mm thick, 120 m long and 650 mm wide, with same composition and same pre-coating as in example 1, has been fabricated. The sheets were afterwards cut into blanks which were heated at 920° C. for 6 mn, this time including the heating phase and the holding time. Heating rate  $V_c$  between 20 and 700° C. was 10° C./s. The blanks were finally hot stamped and quenched in order to obtain full martensitic structures. The parts obtained after hot-stamping are covered by a coating, 40 micrometers thick, illustrated at FIG. 1, which has a four layer structure. Starting from the steel substrate, the layers are the following:

[0123] (a) Interdiffusion layer or intermetallic layer, 17 micrometers thick. This layer is itself composed of two sub-layers. Hardness HV50g ranges from 295 to 407, and the mean composition is: 90% Fe, 7% Al, 3% Si.

[0124] (b) Intermediate layer, appearing darker, 8 micrometers thick. This layer has a hardness of 940HV50g and a mean composition, by weight: 43% Fe, 57% Al, 1% Si.

[0125] (c) Intermetallic layer appearing as a pale phase, 8 micrometers thick, displaying a hardness of 610HV50g, a mean composition of 65% Fe, 31% Al, 4% Si

[0126] (d) Darker superficial layer, 7 micrometers thick, 950 HV50g, with a mean composition of 45% Fe, 54% Al, 1% Si. Layers (c) and (d) are quasi-continuous, i.e. occupying at least 90% of the level corresponding to the considered layer. In particular, layer (c) does not reach the extreme surface except very exceptionally. Anyway, this layer (c) occupies less than 10% of the extreme surface.

ii) Conditions of reference: On the other hand, blanks with the same base material and pre-coating parts were furnace-heated in different conditions: The blanks were heated to 950° C. for 7 minutes, this time including the heating phase. Heating rate  $V_c$  was 11° C./s. These conditions correspond to a degree of alloying which is more important than in conditions (i)

[0127] In this coating, the pale intermetallic layer (c), is not continuous and appears as to be scattered within the coating. About 50% of this layer is present at the extreme surface of the part. Moreover, the interdiffusion layer, 10 micrometers thick in contact with the steel substrate is thinner than in the previous case of FIG. 1.

[0128] Resistance spot welding was performed in the two situations i) and ii):

[0129] (i): Coating with quasi-continuous layers (c) and (d), layer (c) occupying less than 10% of the extreme surface

[0130] (ii): Coating with mixed and discontinuous layers, layer (c) occupying more than 10% of the extreme surface Resistance spot welding was performed by superposing two parts and joining them in the following conditions:

[0131] Squeeze force and welding force: 4000 N

[0132] Squeeze time: 50 periods

[0133] Welding and holding time: 18 periods respectively In each condition, the suitable intensity range was determined for obtaining:

[0134] No sputter during welding

[0135] Acceptable nugget size. For the condition i), the weldability range, expressed in terms of current intensity, is 1.4 kA. For the condition ii) the weldability range is extremely small.

[0136] Thus, it may be seen that the coating according to the invention, yields much more satisfactory results.

### Example 3

[0137] In a third example of implementation, a cold rolled steel sheet of the example 1 was cut into blanks of 500.times.500 mm.sup.2 which were heated at 920° C., during 6 mn, then hot stamped and cooled in tools, in such conditions that two different cooling rates were obtained:

[0138] (A): Cooling rate:  $V_A=30^{\circ} \text{ C./s}$

[0139] (B): Cooling rate:  $V_B=60^{\circ} \text{ C./s}$  Due to the shape of the parts, different deformation levels .epsilon. were created during hot stamping. In particular, some zones largely strained display deformation levels higher than 30%.

[0140] As illustrated on FIG. 3, metallographic observations reveal that when .epsilon.>10%, partial bainitic or ferritic transformation occurs on parts cooled with  $V_A=30^{\circ} \text{ C./s}$ , mainly on former austenitic grain boundaries. On the other hand, the parts cooled with  $V_B=60^{\circ} \text{ C./s}$  display fully martensitic microstructure as illustrated on FIG. 4. The latter structures display superior mechanic resistance and a great homogeneity in the case of mechanical solicitation. Thus, even in products or parts where straining is larger than 10%, the application of cooling according to

the invention guarantees a microstructural and mechanical homogeneity.

#### Example 4

[0141] In a fourth example of implementation, steel castings containing different values of sulfur, were elaborated. These steels were further hot rolled, then cold rolled steel into sheets, 2.2 mm thick. Sulfur content varies from 11 ppm (0.0011%) to 59 ppm (0.006%) in weight. Apart sulfur, the compositions of these different steel castings comprise in weight: 0.24% carbon, 1.17% manganese, 0.01% phosphorus, 0.25% silicon, 0.045% aluminum, 0.01% copper, 0.02% nickel, 0.2% chromium, 0.04% titanium, 0.003% boron, 0.002% calcium, the remainder being iron and unavoidable impurities. These sheets were pre-coated with an aluminum-based alloy of composition comprising 9.3% silicon, 2.8% iron, the remainder being aluminum and unavoidable impurities. The sheets were afterwards cut into blanks which were heated at 950° C. for 5 mn, then hot stamped and cooled in tools in order to obtain a full martensitic structure. Mechanical resistance exceeded 1450 MPa. Specimens were extracted according to the transverse sense of rolling direction and submitted to a bending test with alternate bending modes. The inventors put into evidence that the critical bending angle (angle at fracture) is closely related to the sulfur content of the steel: when sulfur content is lower than 0.002%, the bending angle exceeds 60°, which indicates higher ductility and energy absorption. Compact Tensile-Test type specimens were also extracted according to the transverse rolling direction in order to measure the resistance to tearing, i.e. the energy which is necessary for the initiation or propagation of an existing crack. The results, illustrated on FIG. 6, indicate that a initiation energy higher than 18 Joules is achieved when sulfur content is lower than 0.002% in weight. As these qualities of high resistance, high energy absorption and weldability are required in automobile industry, the parts or products fabricated according to the invention will be used with profit for such applications.

[0142] While the above description is clear with regard to the understanding of the invention, the following terms as used in the following list of preferred embodiments and claims have the following noted meanings in order to avoid any confusion:

[0143] pre-coating--the material (Al or Al alloy) coated on or located on at least a portion of the strip or sheet, etc., of base steel to form a pre-coating/base composite, the composite not having been subjected to an alliation reaction between the coated Al or Al alloy material and base steel

[0144] alliation--a reaction between the pre-coating and base steel, to produce at least one intermediate layer different in composition from both the base steel and the pre-coating. The alliation reaction happens during the heat treatment immediately preceding hot stamping. The alliation reaction affects the total thickness of the pre-coating. In a highly preferred embodiment the alliation reaction forms the following layers: (a) interdiffusion, (b) intermediate, (c) intermetallic, and (d) superficial as described above;

[0145] pre-coated steel--the pre-coating/base composite, not having been subjected to an alliation reaction between the coated material and base steel;

[0146] coating--the pre-coating after having been subjected to an alliation reaction between the pre-coating and base steel. In a highly preferred embodiment the coating comprises layers (a) interdiffusion, (b) intermediate, (c) intermetallic, and (d) superficial described above;

[0147] coated steel or product--the pre-coated steel or product that has been subjected to an alliation reaction between the pre-coating and base steel. In a highly preferred embodiment the coated steel is a strip or sheet, etc., of base steel having thereon an invention coating comprising layers (a) interdiffusion, (b) intermediate, (c) intermetallic, and (d) superficial described above;

[0148] blank--a shape cut from a strip.

[0149] product--a stamped blank

## Electronic Patent Application Fee Transmittal

|                                                                                                         |                                                          |                 |               |                             |
|---------------------------------------------------------------------------------------------------------|----------------------------------------------------------|-----------------|---------------|-----------------------------|
| <b>Application Number:</b>                                                                              |                                                          |                 |               |                             |
| <b>Filing Date:</b>                                                                                     |                                                          |                 |               |                             |
| <b>Title of Invention:</b>                                                                              | Coated Steel Strips, Coated Stamped Products and Methods |                 |               |                             |
| <b>First Named Inventor/Applicant Name:</b>                                                             | Pascal DRILLET                                           |                 |               |                             |
| <b>Filer:</b>                                                                                           | Cary S. Kappel                                           |                 |               |                             |
| <b>Attorney Docket Number:</b>                                                                          | 11020631.1028CON5                                        |                 |               |                             |
| Filed as Large Entity                                                                                   |                                                          |                 |               |                             |
| <b>Filing Fees for Track I Prioritized Examination - Nonprovisional Application under 35 USC 111(a)</b> |                                                          |                 |               |                             |
| <b>Description</b>                                                                                      | <b>Fee Code</b>                                          | <b>Quantity</b> | <b>Amount</b> | <b>Sub-Total in USD(\$)</b> |
| <b>Basic Filing:</b>                                                                                    |                                                          |                 |               |                             |
| UTILITY APPLICATION FILING                                                                              | 1011                                                     | 1               | 300           | 300                         |
| UTILITY SEARCH FEE                                                                                      | 1111                                                     | 1               | 660           | 660                         |
| UTILITY EXAMINATION FEE                                                                                 | 1311                                                     | 1               | 760           | 760                         |
| REQUEST FOR PRIORITIZED EXAMINATION                                                                     | 1817                                                     | 1               | 4000          | 4000                        |
| <b>Pages:</b>                                                                                           |                                                          |                 |               |                             |
| <b>Claims:</b>                                                                                          |                                                          |                 |               |                             |
| CLAIMS IN EXCESS OF 20                                                                                  | 1202                                                     | 3               | 100           | 300                         |
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| PUBL. FEE- EARLY, VOLUNTARY, OR NORMAL   | 1504     | 1        | 0      | 0                    |
| PROCESSING FEE, EXCEPT PROV. APPLS.      | 1830     | 1        | 140    | 140                  |
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|-----------------|-----------------------------------------------------|-------------------------------------|-------------------------------------------|------------------|------------------|
| 1               | TrackOne Request                                    | TrackOneRequest-EFS.PDF             | 118261                                    | no               | 2                |
|                 |                                                     |                                     | 6afca026592c8637a21469fe04e530f31414b62d  |                  |                  |
| Warnings:       |                                                     |                                     |                                           |                  |                  |
| Information:    |                                                     |                                     |                                           |                  |                  |
| 2               | Application Data Sheet                              | ADS-EFS.PDF                         | 1256750                                   | no               | 9                |
|                 |                                                     |                                     | 836709caa09f6b64af0c682076034fa2375172d9  |                  |                  |
| Warnings:       |                                                     |                                     |                                           |                  |                  |
| Information:    |                                                     |                                     |                                           |                  |                  |
| 3               | Oath or Declaration filed                           | delclarations.PDF                   | 645621                                    | no               | 5                |
|                 |                                                     |                                     | f97b559f237b6d1d317206eaea3adf8174f39883  |                  |                  |
| Warnings:       |                                                     |                                     |                                           |                  |                  |
| Information:    |                                                     |                                     |                                           |                  |                  |
| 4               | Power of Attorney                                   | poa.PDF                             | 68736                                     | no               | 1                |
|                 |                                                     |                                     | fd0ae6785cbb09a6bf74c259681c107e19becc5e  |                  |                  |
| Warnings:       |                                                     |                                     |                                           |                  |                  |
| Information:    |                                                     |                                     |                                           |                  |                  |
| 5               | Preliminary Amendment                               | 1028CON5-pre-amend-efs.pdf          | 137437                                    | no               | 7                |
|                 |                                                     |                                     | 1817e802d8236ca4e93b34fd7be2f432b133656   |                  |                  |
| Warnings:       |                                                     |                                     |                                           |                  |                  |
| Information:    |                                                     |                                     |                                           |                  |                  |
| 6               |                                                     | 11020631-1028CON5-APPL-DWGS-EFS.pdf | 3712140                                   | yes              | 34               |
|                 |                                                     |                                     | 9f21c931bfd10674c8156e911a04e17256aff4faa |                  |                  |
|                 | Multipart Description/PDF files in .zip description |                                     |                                           |                  |                  |
|                 | Document Description                                |                                     | Start                                     | End              |                  |

|  |                                             |    |    |
|--|---------------------------------------------|----|----|
|  | Drawings-only black and white line drawings | 31 | 34 |
|  | Abstract                                    | 30 | 30 |
|  | Claims                                      | 24 | 29 |
|  | Specification                               | 1  | 23 |

**Warnings:**

**Information:**

|   |                      |              |                                          |    |   |
|---|----------------------|--------------|------------------------------------------|----|---|
| 7 | Fee Worksheet (SB06) | fee-info.pdf | 41654                                    | no | 2 |
|   |                      |              | ed0736a381b52f35b5e65ffb8c757e8346727626 |    |   |

**Warnings:**

**Information:**

|                                     |         |
|-------------------------------------|---------|
| <b>Total Files Size (in bytes):</b> | 5980599 |
|-------------------------------------|---------|

**This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.**

**New Applications Under 35 U.S.C. 111**

**If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.**

**National Stage of an International Application under 35 U.S.C. 371**

**If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.**

**New International Application Filed with the USPTO as a Receiving Office**

**If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.**

**IN THE UNITED STATES PATENT & TRADEMARK OFFICE**

Application of: ARCELORMITTAL  
Inventor(s): Drillet et al.  
Application No.: TBA  
Filed: HEREWITH  
Attorney Docket No.: 20631.1028CON5  
Confirmation No.:  
Title: Coated Steel Strips, Coated Stamped Products and Methods

Submitted electronically via EFS-Web

May 16, 2019

Mail Stop:  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

**PRELIMINARY AMENDMENT**

Dear Madam or Sir:

Applicant requests that the following amendments be made in the above-identified matter prior to examination thereof:

**Amendments to the Specification and Abstract**

Please amend the specification as follows:

[0000.1] This is a Continuation of U.S. Patent Application No. 15/606,770 filed May 26, 2017 which is a Continuation of U.S. Patent Application No. 13/621,015, filed September 15, 2012 which is a Divisional of U.S. Patent Application No. 12/447,777, filed April 29, 2009, which is a National Phase Application of International Patent Application PCT/IB06/004019, filed October 30, 2006. All of the above patent applications are hereby incorporated by reference herein.

No new matter has been added.

**Amendments to the Claims:**

The following listing of claims will replace all prior versions and listings of claims in the application:

**Listing of Claims:**

Claims 1-24 (canceled).

Claim 25 ( New) A precoated steel product comprising:

a strip of base steel having a first side and a second side; and  
a precoat on at least one of the first side and the second side, the precoat being made of an aluminum alloy and having a controlled thickness to create a precoated steel strip heat treatable to form a coating comprising, proceeding from the base steel outwards:

- (a) an Interdiffusion layer,
- (b) an Intermediate layer,
- (c) an Intermetallic layer, and
- (d) a Superficial layer; wherein the coating has a thickness greater than 30 micrometers and wherein said layer (a) has a thickness less than 15 micrometers.

Claim 26 ( New) The precoated steel product according to claim 25, wherein the said layers (c) and (d) are quasi continuous by occupying at least 90% of a level corresponding to each said layer and wherein less than 10% of layer (c) is present at an extreme surface of the product.

Claim 27 ( New) The precoated steel product according to claim 25, wherein the base steel comprises the following components by weight based on total weight:

0.15%<carbon<0.5%;  
0.5%<manganese<3%;  
0.1%<silicon<0.5%;  
0.01%<chromium<1%;  
titanium<0.2%;  
aluminum<0.1%;  
phosphorus<0.1%;

sulfur<0.05%;

0.0005%<boron<0.08%;

and further comprises iron and impurities inherent in processing.

Claim 28 ( New) The precoated steel product according to claim 25, wherein the base steel comprises the following components by weight based on total weight:

0.20%<carbon<0.5%;

0.8%<manganese<1.5%;

0.1%<silicon<0.35%;

0.01%<chromium<1%;

titanium<0.1%;

aluminum<0.1%;

phosphorus<0.05%;

sulfur<0.03%;

0.0005%<boron<0.01%;

and further comprises iron and impurities inherent in processing.

Claim 29 ( New) The precoated steel product according to claim 25, wherein the base steel comprises 20 ppm or less of sulfur.

Claim 30 ( New) The precoated steel product according to claim 27, wherein the base steel comprises 20 ppm or less of sulfur.

Claim 31 ( New) The precoated steel product according to claim 25, wherein a ratio of titanium to nitrogen in the base steel in weight % is in excess of 3.42.

Claim 32 ( New) The precoated steel product according to claim 27, wherein a ratio of titanium to nitrogen in the base steel in weight % is in excess of 3.42.

Claim 33 ( New) The precoated steel product according to claim 30, wherein a ratio of titanium to nitrogen in the base steel in weight % is in excess of 3.42.

Claim 34 ( New). The precoated steel product according to claim 25, wherein the aluminum alloy precoating comprises from 8% to 11% silicon by weight, from 2% to 4% iron by weight, the remainder being aluminum and impurities inherent in processing.

Claim 35 ( New). The precoated steel product according to claim 25, wherein said layer (a) has a thickness less than 10 micrometers.

Claim 36 ( New). The precoated steel product according to claim 25, wherein a thickness  $t_p$  of said precoating is from 20 to 33 micrometers at every location on at least one of said first and second sides.

Claim 37 ( New). The precoated steel product according to claim 25, wherein a thickness  $t_p$  of said precoating is from 20 to 33 micrometers at every location on both said first and second sides.

Claim 38 ( New) The precoated steel product according to claim 34, wherein the aluminum alloy precoating comprises from 9% to 10% silicon by weight.

Claim 39 ( New) The precoated steel product according to claim 38, wherein the aluminum alloy precoating comprises from 9.3% silicon by weight.

Claim 40 ( New) The precoated steel product according to claim 25, wherein the Interdiffusion layer comprises the following components by weight based on total weight: 86 to 95% Fe, 4 to 10% Al, and 0 to 5% Si.

Claim 41 ( New) The precoated steel product according to claim 25, wherein the Intermediate layer comprises the following components by weight based on total weight: 39 to 47% Fe, 53 to 61% Al, and 0 to 2% Si.

Claim 42 ( New) The precoated steel product according to claim 25, wherein the Intermetallic layer comprises the following components by weight based on total weight: 62 to 67% Fe, 30 to 34% Al, and 2 to 6% Si.

Claim 43 ( New) The precoated steel product according to claim 25, wherein the Superficial layer comprises the following components by weight based on total weight: 39 to 47% Fe, 53to 61% Al, and 0 to 2% Si.

Claim 44 ( New) The precoated steel product according to claim 40, wherein the Intermediate layer comprises the following components by weight based on total weight: 39 to 47% Fe, 53 to 61% Al, and 0 to 2% Si.

Claim 45 ( New) The precoated steel product according to claim 44, wherein the Intermetallic layer comprises the following components by weight based on total weight: 62 to 67% Fe, 30 to 34% Al, and 2 to 6% Si.

Claim 46 ( New) The precoated steel product according to claim 45, wherein the Superficial layer comprises the following components by weight based on total weight: 39 to 47% Fe, 53to 61% Al, and 0 to 2% Si.

Claim 47 ( New) The precoated steel product according to claim 25, wherein the Interdiffusion layer has a thickness of from 1 micron to 15 microns.

**REMARKS**

Claims 1-24 are canceled. Claim 25-47 are added. The specification has been amended.  
No new matter has been added.

Reconsideration and allowance of the present application is respectfully requested.

Respectfully submitted,

DAVIDSON, DAVIDSON & KAPPEL, LLC

By \_\_\_\_\_ /Cary Kappel /  
Cary Kappel  
(Reg. No. 36,561)

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Customer No.: 23280  
Deposit Acct. No.: 50-0552

**CERTIFICATION AND REQUEST FOR PRIORITIZED EXAMINATION  
UNDER 37 CFR 1.102(e) (Page 1 of 1)**

|                       |                                                         |                                               |  |
|-----------------------|---------------------------------------------------------|-----------------------------------------------|--|
| First Named Inventor: | Pascal DRILLET                                          | Nonprovisional Application Number (if known): |  |
| Title of Invention:   | Coated Steel Strips Coated Stamped Products and Methods |                                               |  |

**APPLICANT HEREBY CERTIFIES THE FOLLOWING AND REQUESTS PRIORITIZED EXAMINATION FOR THE ABOVE-IDENTIFIED APPLICATION.**

1. The processing fee set forth in 37 CFR 1.17(i)(1) and the prioritized examination fee set forth in 37 CFR 1.17(c) have been filed with the request. The publication fee requirement is met because that fee, set forth in 37 CFR 1.18(d), is currently \$0. The basic filing fee, search fee, and examination fee are filed with the request or have been already been paid. I understand that any required excess claims fees or application size fee must be paid for the application.
2. I understand that the application may not contain, or be amended to contain, more than four independent claims, more than thirty total claims, or any multiple dependent claims, and that any request for an extension of time will cause an outstanding Track I request to be dismissed.
3. The applicable box is checked below:

**I. ☒ Original Application (Track One) - Prioritized Examination under § 1.102(e)(1)**

- i. (a) The application is an original nonprovisional utility application filed under 35 U.S.C. 111(a). This certification and request is being filed with the utility application via EFS-Web.  
---OR---  
(b) The application is an original nonprovisional plant application filed under 35 U.S.C. 111(a). This certification and request is being filed with the plant application in paper.
- ii. An executed inventor's oath or declaration under 37 CFR 1.63 or 37 CFR 1.64 for each inventor, or the application data sheet meeting the conditions specified in 37 CFR 1.53(f)(3)(i) is filed with the application.

**II. ☐ Request for Continued Examination - Prioritized Examination under § 1.102(e)(2)**

- i. A request for continued examination has been filed with, or prior to, this form.
- ii. If the application is a utility application, this certification and request is being filed via EFS-Web.
- iii. The application is an original nonprovisional utility application filed under 35 U.S.C. 111(a), or is a national stage entry under 35 U.S.C. 371.
- iv. This certification and request is being filed prior to the mailing of a first Office action responsive to the request for continued examination.
- v. No prior request for continued examination has been granted prioritized examination status under 37 CFR 1.102(e)(2).

|                                       |                                               |
|---------------------------------------|-----------------------------------------------|
| Signature <u>/Cary Kappel/</u>        | Date <u>May 16, 2019</u>                      |
| Name (Print/Typed) <u>Cary Kappel</u> | Practitioner Registration Number <u>36561</u> |

**Note:** This form must be signed in accordance with 37 CFR 1.33. See 37 CFR 1.4(d) for signature requirements and certifications. Submit multiple forms if more than one signature is required.\*

☒ \*Total of 1 forms are submitted.

## Privacy Act Statement

The **Privacy Act of 1974 (P.L. 93-579)** requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether disclosure of these records is required by the Freedom of Information Act.
2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (*i.e.*, GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspection or an issued patent.
9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

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|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |                                                          |                        |                   |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------|------------------------|-------------------|
| <b>Application Data Sheet 37 CFR 1.76</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |                                                          | Attorney Docket Number | 11020631.1028con5 |
|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |                                                          | Application Number     |                   |
| Title of Invention                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | Coated Steel Strips, Coated Stamped Products and Methods |                        |                   |
| <p>The application data sheet is part of the provisional or nonprovisional application for which it is being submitted. The following form contains the bibliographic data arranged in a format specified by the United States Patent and Trademark Office as outlined in 37 CFR 1.76.</p> <p>This document may be completed electronically and submitted to the Office in electronic format using the Electronic Filing System (EFS) or the document may be printed and included in a paper filed application.</p> |                                                          |                        |                   |

**Secrecy Order 37 CFR 5.2:**

|                          |                                                                                                                                                                                                                                       |
|--------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <input type="checkbox"/> | Portions or all of the application associated with this Application Data Sheet may fall under a Secrecy Order pursuant to 37 CFR 5.2 (Paper filers only. Applications that fall under Secrecy Order may not be filed electronically.) |
|--------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|

**Inventor Information:**

|                                                                                                                                    |              |                         |                                   |        |        |
|------------------------------------------------------------------------------------------------------------------------------------|--------------|-------------------------|-----------------------------------|--------|--------|
| Inventor                                                                                                                           | 1            |                         |                                   |        | Remove |
| Legal Name                                                                                                                         |              |                         |                                   |        |        |
| Prefix                                                                                                                             | Given Name   | Middle Name             | Family Name                       | Suffix |        |
|                                                                                                                                    | Pascal       |                         | DRILLET                           |        |        |
| Residence Information (Select One)    US Residency <input checked="" type="radio"/> Non US Residency    Active US Military Service |              |                         |                                   |        |        |
| City                                                                                                                               | Rozerieulles |                         | Country of Residence <sup>i</sup> | FR     |        |
| Mailing Address of Inventor:                                                                                                       |              |                         |                                   |        |        |
| Address 1                                                                                                                          |              | 9 Chemin de Banauvignes |                                   |        |        |
| Address 2                                                                                                                          |              |                         |                                   |        |        |
| City                                                                                                                               | Rozerieulles |                         | State/Province                    |        |        |
| Postal Code                                                                                                                        | 57160        |                         | Country <sup>i</sup>              | FR     |        |
| Inventor                                                                                                                           | 2            |                         |                                   |        | Remove |
| Legal Name                                                                                                                         |              |                         |                                   |        |        |
| Prefix                                                                                                                             | Given Name   | Middle Name             | Family Name                       | Suffix |        |
|                                                                                                                                    | Dominique    |                         | Spehner                           |        |        |
| Residence Information (Select One)    US Residency <input checked="" type="radio"/> Non US Residency    Active US Military Service |              |                         |                                   |        |        |
| City                                                                                                                               | Balan        |                         | Country of Residence <sup>i</sup> | FR     |        |
| Mailing Address of Inventor:                                                                                                       |              |                         |                                   |        |        |
| Address 1                                                                                                                          |              | 8 rue de la Porte       |                                   |        |        |
| Address 2                                                                                                                          |              |                         |                                   |        |        |
| City                                                                                                                               | Balan        |                         | State/Province                    | OT     |        |
| Postal Code                                                                                                                        | 08200        |                         | Country <sup>i</sup>              | FR     |        |
| Inventor                                                                                                                           | 3            |                         |                                   |        | Remove |
| Legal Name                                                                                                                         |              |                         |                                   |        |        |

|                                           |                                                          |                        |                   |
|-------------------------------------------|----------------------------------------------------------|------------------------|-------------------|
| <b>Application Data Sheet 37 CFR 1.76</b> |                                                          | Attorney Docket Number | 11020631.1028con5 |
|                                           |                                                          | Application Number     |                   |
| Title of Invention                        | Coated Steel Strips, Coated Stamped Products and Methods |                        |                   |

|                                                                                                                                    |                          |             |                                   |        |
|------------------------------------------------------------------------------------------------------------------------------------|--------------------------|-------------|-----------------------------------|--------|
| Prefix                                                                                                                             | Given Name               | Middle Name | Family Name                       | Suffix |
|                                                                                                                                    | Ronald                   |             | Kefferstein                       |        |
| Residence Information (Select One)    US Residency <input checked="" type="radio"/> Non US Residency    Active US Military Service |                          |             |                                   |        |
| City                                                                                                                               | Saint Mitre Les Ramparts |             | Country of Residence <sup>i</sup> | FR     |

|                                                                                                                                                                                    |                          |                      |    |  |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------|----------------------|----|--|
| <b>Mailing Address of Inventor:</b>                                                                                                                                                |                          |                      |    |  |
| Address 1                                                                                                                                                                          | 2 rue des Rocaledes      |                      |    |  |
| Address 2                                                                                                                                                                          |                          |                      |    |  |
| City                                                                                                                                                                               | Saint Mitre Les Ramparts | State/Province       | OT |  |
| Postal Code                                                                                                                                                                        | 13920                    | Country <sup>i</sup> | FR |  |
| All Inventors Must Be Listed - Additional Inventor Information blocks may be generated within this form by selecting the <b>Add</b> button. <span style="float: right;">Add</span> |                          |                      |    |  |

### Correspondence Information:

|                                                                                                                                       |                   |           |              |
|---------------------------------------------------------------------------------------------------------------------------------------|-------------------|-----------|--------------|
| Enter either Customer Number or complete the Correspondence Information section below.<br>For further information see 37 CFR 1.33(a). |                   |           |              |
| <input type="checkbox"/> An Address is being provided for the correspondence information of this application.                         |                   |           |              |
| Customer Number                                                                                                                       | 23280             |           |              |
| Email Address                                                                                                                         | ddk@ddkpatent.com | Add Email | Remove Email |

### Application Information:

|                                         |                                                          |                                           |                          |
|-----------------------------------------|----------------------------------------------------------|-------------------------------------------|--------------------------|
| Title of the Invention                  | Coated Steel Strips, Coated Stamped Products and Methods |                                           |                          |
| Attorney Docket Number                  | 11020631.1028con5                                        | Small Entity Status Claimed               | <input type="checkbox"/> |
| Application Type                        | Nonprovisional                                           |                                           |                          |
| Subject Matter                          | Utility                                                  |                                           |                          |
| Total Number of Drawing Sheets (if any) | 4                                                        | Suggested Figure for Publication (if any) |                          |

### Filing By Reference:

Only complete this section when filing an application by reference under 35 U.S.C. 111(c) and 37 CFR 1.57(a). Do not complete this section if application papers including a specification and any drawings are being filed. Any domestic benefit or foreign priority information must be provided in the appropriate section(s) below (i.e., "Domestic Benefit/National Stage Information" and "Foreign Priority Information").

For the purposes of a filing date under 37 CFR 1.53(b), the description and any drawings of the present application are replaced by this reference to the previously filed application, subject to conditions and requirements of 37 CFR 1.57(a).

|                                                        |                          |                                                         |
|--------------------------------------------------------|--------------------------|---------------------------------------------------------|
| Application number of the previously filed application | Filing date (YYYY-MM-DD) | Intellectual Property Authority or Country <sup>i</sup> |
|                                                        |                          |                                                         |

|                                           |                                                          |                        |                   |
|-------------------------------------------|----------------------------------------------------------|------------------------|-------------------|
| <b>Application Data Sheet 37 CFR 1.76</b> |                                                          | Attorney Docket Number | 11020631.1028con5 |
|                                           |                                                          | Application Number     |                   |
| Title of Invention                        | Coated Steel Strips, Coated Stamped Products and Methods |                        |                   |

**Publication Information:**

|                          |                                                                                                                                                                                                                                                                                                                                                                                               |
|--------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <input type="checkbox"/> | Request Early Publication (Fee required at time of Request 37 CFR 1.219)                                                                                                                                                                                                                                                                                                                      |
| <input type="checkbox"/> | <b>Request Not to Publish.</b> I hereby request that the attached application not be published under 35 U.S.C. 122(b) and certify that the invention disclosed in the attached application <b>has not and will not</b> be the subject of an application filed in another country, or under a multilateral international agreement, that requires publication at eighteen months after filing. |

**Representative Information:**

|                                                                                                                                                                                                                                                                                                                                                                                                                                                                 |                                                  |                                              |                                                         |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------|----------------------------------------------|---------------------------------------------------------|
| Representative information should be provided for all practitioners having a power of attorney in the application. Providing this information in the Application Data Sheet does not constitute a power of attorney in the application (see 37 CFR 1.32). Either enter Customer Number or complete the Representative Name section below. If both sections are completed the customer Number will be used for the Representative Information during processing. |                                                  |                                              |                                                         |
| Please Select One:                                                                                                                                                                                                                                                                                                                                                                                                                                              | <input checked="" type="radio"/> Customer Number | <input type="radio"/> US Patent Practitioner | <input type="radio"/> Limited Recognition (37 CFR 11.9) |
| Customer Number                                                                                                                                                                                                                                                                                                                                                                                                                                                 | 23280                                            |                                              |                                                         |

**Domestic Benefit/National Stage Information:**

This section allows for the applicant to either claim benefit under 35 U.S.C. 119(e), 120, 121, 365(c), or 386(c) or indicate National Stage entry from a PCT application. Providing benefit claim information in the Application Data Sheet constitutes the specific reference required by 35 U.S.C. 119(e) or 120, and 37 CFR 1.78.

When referring to the current application, please leave the "Application Number" field blank.

|                          |                        |                          |                                    |               |                         |
|--------------------------|------------------------|--------------------------|------------------------------------|---------------|-------------------------|
| Prior Application Status | Pending                | Remove                   |                                    |               |                         |
| Application Number       | Continuity Type        | Prior Application Number | Filing or 371(c) Date (YYYY-MM-DD) |               |                         |
|                          | Continuation of        | 15/606770                | 2017-05-26                         |               |                         |
| Prior Application Status | Patented               | Remove                   |                                    |               |                         |
| Application Number       | Continuity Type        | Prior Application Number | Filing Date (YYYY-MM-DD)           | Patent Number | Issue Date (YYYY-MM-DD) |
| 15/606770                | Continuation of        | 13621015                 | 2012-09-15                         | 9708683       | 2017-07-18              |
| Prior Application Status | Patented               | Remove                   |                                    |               |                         |
| Application Number       | Continuity Type        | Prior Application Number | Filing Date (YYYY-MM-DD)           | Patent Number | Issue Date (YYYY-MM-DD) |
| 13621015                 | Division of            | 12447777                 | 2011-03-18                         | 8307680       | 2012-11-13              |
| Prior Application Status | Abandoned              | Remove                   |                                    |               |                         |
| Application Number       | Continuity Type        | Prior Application Number | Filing or 371(c) Date (YYYY-MM-DD) |               |                         |
| 12447777                 | a 371 of international | PCT/IB06/04019           | 2006-10-30                         |               |                         |

|                                           |                                                          |                        |                   |
|-------------------------------------------|----------------------------------------------------------|------------------------|-------------------|
| <b>Application Data Sheet 37 CFR 1.76</b> |                                                          | Attorney Docket Number | 11020631.1028con5 |
|                                           |                                                          | Application Number     |                   |
| Title of Invention                        | Coated Steel Strips, Coated Stamped Products and Methods |                        |                   |

Additional Domestic Benefit/National Stage Data may be generated within this form by selecting the **Add** button.

Add

## Foreign Priority Information:

This section allows for the applicant to claim priority to a foreign application. Providing this information in the application data sheet constitutes the claim for priority as required by 35 U.S.C. 119(b) and 37 CFR 1.55. When priority is claimed to a foreign application that is eligible for retrieval under the priority document exchange program (PDX)<sup>i</sup> the information will be used by the Office to automatically attempt retrieval pursuant to 37 CFR 1.55(i)(1) and (2). Under the PDX program, applicant bears the ultimate responsibility for ensuring that a copy of the foreign application is received by the Office from the participating foreign intellectual property office, or a certified copy of the foreign priority application is filed, within the time period specified in 37 CFR 1.55(g)(1).

|                    |                      |                          | <b>Remove</b>                            |
|--------------------|----------------------|--------------------------|------------------------------------------|
| Application Number | Country <sup>i</sup> | Filing Date (YYYY-MM-DD) | Access Code <sup>i</sup> (if applicable) |
|                    |                      |                          |                                          |

Additional Foreign Priority Data may be generated within this form by selecting the **Add** button.

Add

## Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications

☐ This application (1) claims priority to or the benefit of an application filed before March 16, 2013 and (2) also contains, or contained at any time, a claim to a claimed invention that has an effective filing date on or after March 16, 2013.

NOTE: By providing this statement under 37 CFR 1.55 or 1.78, this application, with a filing date on or after March 16, 2013, will be examined under the first inventor to file provisions of the AIA.

|                                           |                                                          |                        |                   |
|-------------------------------------------|----------------------------------------------------------|------------------------|-------------------|
| <b>Application Data Sheet 37 CFR 1.76</b> |                                                          | Attorney Docket Number | 11020631.1028con5 |
|                                           |                                                          | Application Number     |                   |
| Title of Invention                        | Coated Steel Strips, Coated Stamped Products and Methods |                        |                   |

## Authorization or Opt-Out of Authorization to Permit Access:

When this Application Data Sheet is properly signed and filed with the application, applicant has provided written authority to permit a participating foreign intellectual property (IP) office access to the instant application-as-filed (see paragraph A in subsection 1 below) and the European Patent Office (EPO) access to any search results from the instant application (see paragraph B in subsection 1 below).

Should applicant choose not to provide an authorization identified in subsection 1 below, applicant **must opt-out** of the authorization by checking the corresponding box A or B or both in subsection 2 below.

**NOTE:** This section of the Application Data Sheet is **ONLY** reviewed and processed with the **INITIAL** filing of an application. After the initial filing of an application, an Application Data Sheet cannot be used to provide or rescind authorization for access by a foreign IP office(s). Instead, Form PTO/SB/39 or PTO/SB/69 must be used as appropriate.

### 1. Authorization to Permit Access by a Foreign Intellectual Property Office(s)

**A. Priority Document Exchange (PDX)** - Unless box A in subsection 2 (opt-out of authorization) is checked, the undersigned hereby **grants the USPTO authority** to provide the European Patent Office (EPO), the Japan Patent Office (JPO), the Korean Intellectual Property Office (KIPO), the State Intellectual Property Office of the People's Republic of China (SIPO), the World Intellectual Property Organization (WIPO), and any other foreign intellectual property office participating with the USPTO in a bilateral or multilateral priority document exchange agreement in which a foreign application claiming priority to the instant patent application is filed, access to: (1) the instant patent application-as-filed and its related bibliographic data, (2) any foreign or domestic application to which priority or benefit is claimed by the instant application and its related bibliographic data, and (3) the date of filing of this Authorization. See 37 CFR 1.14(h)(1).

**B. Search Results from U.S. Application to EPO** - Unless box B in subsection 2 (opt-out of authorization) is checked, the undersigned hereby **grants the USPTO authority** to provide the EPO access to the bibliographic data and search results from the instant patent application when a European patent application claiming priority to the instant patent application is filed. See 37 CFR 1.14(h)(2).

The applicant is reminded that the EPO's Rule 141(1) EPC (European Patent Convention) requires applicants to submit a copy of search results from the instant application without delay in a European patent application that claims priority to the instant application.

### 2. Opt-Out of Authorizations to Permit Access by a Foreign Intellectual Property Office(s)

☐ A. Applicant **DOES NOT** authorize the USPTO to permit a participating foreign IP office access to the instant application-as-filed. If this box is checked, the USPTO will not be providing a participating foreign IP office with any documents and information identified in subsection 1A above.

☐ B. Applicant **DOES NOT** authorize the USPTO to transmit to the EPO any search results from the instant patent application. If this box is checked, the USPTO will not be providing the EPO with search results from the instant application.

**NOTE:** Once the application has published or is otherwise publicly available, the USPTO may provide access to the application in accordance with 37 CFR 1.14.

|                                           |                                                          |                        |                   |
|-------------------------------------------|----------------------------------------------------------|------------------------|-------------------|
| <b>Application Data Sheet 37 CFR 1.76</b> |                                                          | Attorney Docket Number | 11020631.1028con5 |
|                                           |                                                          | Application Number     |                   |
| Title of Invention                        | Coated Steel Strips, Coated Stamped Products and Methods |                        |                   |

## Applicant Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.

|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |                                          |                                                  |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------|--------------------------------------------------|
| <b>Applicant</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              | 1                                        | <a href="#">Remove</a>                           |
| <p>If the applicant is the inventor (or the remaining joint inventor or inventors under 37 CFR 1.45), this section should not be completed. The information to be provided in this section is the name and address of the legal representative who is the applicant under 37 CFR 1.43; or the name and address of the assignee, person to whom the inventor is under an obligation to assign the invention, or person who otherwise shows sufficient proprietary interest in the matter who is the applicant under 37 CFR 1.46. If the applicant is an applicant under 37 CFR 1.46 (assignee, person to whom the inventor is obligated to assign, or person who otherwise shows sufficient proprietary interest) together with one or more joint inventors, then the joint inventor or inventors who are also the applicant should be identified in this section.</p> <p style="text-align: right;"><a href="#">Clear</a></p> |                                          |                                                  |
| <input checked="" type="radio"/> Assignee                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | Legal Representative under 35 U.S.C. 117 | Joint Inventor                                   |
| Person to whom the inventor is obligated to assign.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |                                          | Person who shows sufficient proprietary interest |
| If applicant is the legal representative, indicate the authority to file the patent application, the inventor is:                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |                                          |                                                  |
| <div style="border: 1px solid black; height: 20px; width: 100%;"></div>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       |                                          |                                                  |
| Name of the Deceased or Legally Incapacitated Inventor: <div style="border: 1px solid black; height: 20px; width: 100%;"></div>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |                                          |                                                  |
| If the Applicant is an Organization check here. <input checked="" type="checkbox"/>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |                                          |                                                  |
| Organization Name                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             | ArcelorMittal                            |                                                  |
| <b>Mailing Address Information For Applicant:</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |                                          |                                                  |
| Address 1                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | 24-26 Boulevard-d'Avranches              |                                                  |
| Address 2                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |                                          |                                                  |
| City                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          | Luxembourg                               | State/Province OT                                |
| Country                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       | LU                                       | Postal Code L-1160                               |
| Phone Number                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |                                          | Fax Number                                       |
| Email Address                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 |                                          |                                                  |
| Additional Applicant Data may be generated within this form by selecting the Add button. <a href="#">Add</a>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |                                          |                                                  |

## Assignee Information including Non-Applicant Assignee Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.

|                                           |                                                          |                        |                   |
|-------------------------------------------|----------------------------------------------------------|------------------------|-------------------|
| <b>Application Data Sheet 37 CFR 1.76</b> |                                                          | Attorney Docket Number | 11020631.1028con5 |
|                                           |                                                          | Application Number     |                   |
| Title of Invention                        | Coated Steel Strips, Coated Stamped Products and Methods |                        |                   |

|                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                      |                      |                      |                                       |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------|----------------------|----------------------|---------------------------------------|
| <b>Assignee</b>                                                                                                                                                                                                                                                                                                                                                                                                                                        | 1                    |                      |                      |                                       |
| Complete this section if assignee information, including non-applicant assignee information, is desired to be included on the patent application publication. An assignee-applicant identified in the "Applicant Information" section will appear on the patent application publication as an applicant. For an assignee-applicant, complete this section only if identification as an assignee is also desired on the patent application publication. |                      |                      |                      |                                       |
|                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                      |                      |                      | <input type="button" value="Remove"/> |
| If the Assignee or Non-Applicant Assignee is an Organization check here.                                                                                                                                                                                                                                                                                                                                                                               |                      |                      |                      | <input type="checkbox"/>              |
| Prefix                                                                                                                                                                                                                                                                                                                                                                                                                                                 | Given Name           | Middle Name          | Family Name          | Suffix                                |
| <input type="text"/>                                                                                                                                                                                                                                                                                                                                                                                                                                   | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/>                  |
| <b>Mailing Address Information For Assignee including Non-Applicant Assignee:</b>                                                                                                                                                                                                                                                                                                                                                                      |                      |                      |                      |                                       |
| Address 1                                                                                                                                                                                                                                                                                                                                                                                                                                              |                      | <input type="text"/> |                      |                                       |
| Address 2                                                                                                                                                                                                                                                                                                                                                                                                                                              |                      | <input type="text"/> |                      |                                       |
| City                                                                                                                                                                                                                                                                                                                                                                                                                                                   | <input type="text"/> | State/Province       | <input type="text"/> |                                       |
| Country <sup>i</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                   | <input type="text"/> | Postal Code          | <input type="text"/> |                                       |
| Phone Number                                                                                                                                                                                                                                                                                                                                                                                                                                           | <input type="text"/> | Fax Number           | <input type="text"/> |                                       |
| Email Address                                                                                                                                                                                                                                                                                                                                                                                                                                          | <input type="text"/> |                      |                      |                                       |
| Additional Assignee or Non-Applicant Assignee Data may be generated within this form by selecting the Add button.                                                                                                                                                                                                                                                                                                                                      |                      |                      |                      | <input type="button" value="Add"/>    |

**Signature:**

**NOTE:** This Application Data Sheet must be signed in accordance with 37 CFR 1.33(b). However, if this Application Data Sheet is submitted with the **INITIAL** filing of the application and either box A or B is not checked in subsection 2 of the "Authorization or Opt-Out of Authorization to Permit Access" section, then this form must also be signed in accordance with 37 CFR 1.14(c).

This Application Data Sheet **must** be signed by a patent practitioner if one or more of the applicants is a **juristic entity** (e.g., corporation or association). If the applicant is two or more joint inventors, this form must be signed by a patent practitioner, **all** joint inventors who are the applicant, or one or more joint inventor-applicants who have been given power of attorney (e.g., see USPTO Form PTO/AIA/81) on behalf of **all** joint inventor-applicants.

See 37 CFR 1.4(d) for the manner of making signatures and certifications.

|                                                                                     |               |           |                   |                                    |
|-------------------------------------------------------------------------------------|---------------|-----------|-------------------|------------------------------------|
| <b>Signature</b>                                                                    | /Cary Kappel/ |           | Date (YYYY-MM-DD) | 2019-05-16                         |
| First Name                                                                          | Cary          | Last Name | Kappel            | Registration Number                |
|                                                                                     |               |           |                   | 36561                              |
| Additional Signature may be generated within this form by selecting the Add button. |               |           |                   | <input type="button" value="Add"/> |

|                                           |                                                          |                        |                   |
|-------------------------------------------|----------------------------------------------------------|------------------------|-------------------|
| <b>Application Data Sheet 37 CFR 1.76</b> |                                                          | Attorney Docket Number | 11020631.1028con5 |
|                                           |                                                          | Application Number     |                   |
| Title of Invention                        | Coated Steel Strips, Coated Stamped Products and Methods |                        |                   |

This collection of information is required by 37 CFR 1.76. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 23 minutes to complete, including gathering, preparing, and submitting the completed application data sheet form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

# Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these records.
2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspections or an issued patent.
9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

Doc code: Oath

Document Description: Oath or declaration filed

PTO/AIA/02 (07-13)

Approved for use through 01/31/2014, OMB 0651-0032

U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

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**SUBSTITUTE STATEMENT IN LIEU OF AN OATH OR DECLARATION FOR UTILITY  
OR DESIGN PATENT APPLICATION (35 U.S.C. 115(d) AND 37 CFR 1.64)**

|                                                                                                                                                                                   |                                                          |         |         |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------|---------|---------|
| <b>Title of<br/>Invention</b>                                                                                                                                                     | Coated Steel Strips, Coated Stamped Products and Methods |         |         |
| This statement is directed to:                                                                                                                                                    |                                                          |         |         |
| <input type="checkbox"/> The attached application,                                                                                                                                |                                                          |         |         |
| OR                                                                                                                                                                                |                                                          |         |         |
| <input checked="" type="checkbox"/> United States application or PCT international application number <u>15/606,770</u> filed on <u>May 26, 2017</u>                              |                                                          |         |         |
| <b>LEGAL NAME of inventor to whom this substitute statement applies:</b>                                                                                                          |                                                          |         |         |
| (E.g., Given Name (first and middle (if any)) and Family Name or Surname)                                                                                                         |                                                          |         |         |
| Dominique Spohner                                                                                                                                                                 |                                                          |         |         |
| Residence (except for a deceased or legally incapacitated inventor):                                                                                                              |                                                          |         |         |
| City                                                                                                                                                                              | State                                                    | Country |         |
| Balan                                                                                                                                                                             |                                                          | FR      |         |
| Mailing Address (except for a deceased or legally incapacitated inventor):                                                                                                        |                                                          |         |         |
| 8 rue de la Porte                                                                                                                                                                 |                                                          |         |         |
| City                                                                                                                                                                              | State                                                    | Zip     | Country |
| Balan                                                                                                                                                                             |                                                          | 08200   | FR      |
| I believe the above-named inventor or joint inventor to be the original inventor or an original joint inventor of a claimed invention in the application.                         |                                                          |         |         |
| The above-identified application was made or authorized to be made by me.                                                                                                         |                                                          |         |         |
| I hereby acknowledge that any willful false statement made in this statement is punishable under 18 U.S.C. 1001 by fine or imprisonment of not more than five (5) years, or both. |                                                          |         |         |
| <b>Relationship to the inventor to whom this substitute statement applies:</b>                                                                                                    |                                                          |         |         |
| <input type="checkbox"/> Legal Representative (for deceased or legally incapacitated inventor only),                                                                              |                                                          |         |         |
| <input checked="" type="checkbox"/> Assignee,                                                                                                                                     |                                                          |         |         |
| <input type="checkbox"/> Person to whom the inventor is under an obligation to assign,                                                                                            |                                                          |         |         |
| <input type="checkbox"/> Person who otherwise shows a sufficient proprietary interest in the matter (petition under 37 CFR 1.46 is required), or                                  |                                                          |         |         |
| <input type="checkbox"/> Joint Inventor.                                                                                                                                          |                                                          |         |         |

[Page 1 of 2]

This collection of information is required by 35 U.S.C. 115 and 37 CFR 1.63. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 1 minute to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

**SUBSTITUTE STATEMENT**

Circumstances permitting execution of this substitute statement:

- ☐ Inventor is deceased,
- ☐ Inventor is under legal incapacity,
- ☒ Inventor cannot be found or reached after diligent effort, or
- ☐ Inventor has refused to execute the oath or declaration under 37 CFR 1.63.

If there are joint inventors, please check the appropriate box below:

- ☒ An application data sheet under 37 CFR 1.76 (PTO/AIA/14 or equivalent) naming the entire inventive entity has been or is currently submitted.
- OR
- ☐ An application data sheet under 37 CFR 1.76 (PTO/AIA/14 or equivalent) has not been submitted. Thus, a Substitute Statement Supplemental Sheet (PTO/AIA/11 or equivalent) naming the entire inventive entity and providing inventor information is attached. See 37 CFR 1.64(b).

**WARNING:**

Petitioner/applicant is cautioned to avoid submitting personal information in documents filed in a patent application that may contribute to identity theft. Personal information such as social security numbers, bank account numbers, or credit card numbers (other than a check or credit card authorization form PTO-2038 submitted for payment purposes) is never required by the USPTO to support a petition or an application. If this type of personal information is included in documents submitted to the USPTO, petitioners/applicants should consider redacting such personal information from the documents before submitting them to the USPTO. Petitioner/applicant is advised that the record of a patent application is available to the public after publication of the application (unless a non-publication request in compliance with 37 CFR 1.213(a) is made in the application) or issuance of a patent. Furthermore, the record from an abandoned application may also be available to the public if the application is referenced in a published application or an issued patent (see 37 CFR 1.14). Checks and credit card authorization forms PTO-2038 submitted for payment purposes are not retained in the application file and therefore are not publicly available.

**PERSON EXECUTING THIS SUBSTITUTE STATEMENT:**

Name: **Sophie Plaisant** Date (Optional): **24 July 2018**

Signature: **APPLICANT NAME AND TITLE OF PERSON EXECUTING THIS SUBSTITUTE STATEMENT:**

If the applicant is a juristic entity, list the applicant name and the title of the signer:

**ARCELORMITTAL**

Applicant Name:

Title of Person Executing This Substitute Statement: **Head of Central Patent Team, Corporate Officer**

The signer, whose title is supplied above, is authorized to act on behalf of the applicant.

**Residence of the signer (unless provided in an application data sheet, PTO/AIA/14 or equivalent):**

City **Montreuil** State \_\_\_\_\_ Country **France**

**Mailing Address of the signer (unless provided in an application data sheet, PTO/AIA/14 or equivalent)**

City \_\_\_\_\_ State \_\_\_\_\_ Zip \_\_\_\_\_ Country \_\_\_\_\_

Note: Use an additional PTO/AIA/02 form for each inventor who is deceased, legally incapacitated, cannot be found or reached after diligent effort, or has refused to execute the oath or declaration under 37 CFR 1.63.

## Privacy Act Statement

The **Privacy Act of 1974 (P.L. 93-579)** requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether disclosure of these records is required by the Freedom of Information Act.
2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspection or an issued patent.
9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

**ASSIGNMENT AND DECLARATION UNDER 37 C.F.R. § 1.63**  
(For Use with Signed Application Data Sheet)

**Title of Invention:** COATED STEEL STRIPS, COATED STAMPED PRODUCTS AND METHODS

**Attorney Docket No.:** 20631.1028CON

This assignment and declaration are directed to (check one):

- ☐ the attached application;  
☐ the application identified by the attorney docket no. and title of invention given above;  
☒ United States Application or PCT International Application No. 15/606,770, filed May 26, 2017.

**ASSIGNMENT**

For good and valuable consideration, the receipt and sufficiency of which are hereby acknowledged, as a below-named inventor I have sold and assigned, and hereby sell and assign, to:

**Name of Assignee:** ARCELORMITTAL

**Address of Assignee:** 24-26 Blvd d'Avranches L-1160 Luxembourg, Luxembourg

(hereinafter ASSIGNEE) all right, title, and interest in and to the above-identified invention and United States patent application, including : (i) any and all divisions or continuations thereof, and in and to any and all Letters Patent of the United States that may issue on any such application or for said invention, including any and all reissues or extensions thereof, to be held and enjoyed by said ASSIGNEE, its successors, or assigns to the full end of the term or terms for which any and all such Letters Patent may be granted; (ii) all applications for patents, utility models, and designs which may hereafter be filed for said invention in any country or countries foreign to the United States, together with the right to file such applications and the right to claim for the same the priority rights derived from said United States application under the Patent Laws of the United States, the International Convention for the Protection of Industrial Property, or any other international agreement or the domestic laws of the country in which any such application is filed, as may be applicable, to be held and enjoyed by said ASSIGNEE, its successors, or assigns to the full end of the term or terms for which any and all such patents, utility models, designs or other rights may be granted; and (iii) all forms of industrial property protection, including, without limitation, patents, utility models, inventors' certificates and designs which may be granted for said invention in any country or countries foreign to the United States and all extensions, renewals and reissues thereof, to be held and enjoyed by said ASSIGNEE, its successors, or assigns to the full end of the term or terms for which any and all such patents, utility models, designs or other rights may be granted.

I hereby authorize and request the Director of the U.S. Patent and Trademark Office to issue any and all such Letters Patent to said ASSIGNEE, its successors, or assigns in accordance herewith.

I warrant and covenant that I have the right to sell and assign the interests herein sold and assigned and that I have not executed and will not execute any document or instrument in conflict herewith.

I further covenant and agree that I will communicate to said ASSIGNEE, its successors, legal representatives, or assigns all information known to me relating to said invention or patent application and that I will execute and deliver any papers, make all rightful oaths, testify in any legal proceedings, and perform all other lawful acts necessary or desirable by said ASSIGNEE, its successors, legal representatives, or assigns to perfect title to said invention, to said application including divisions and continuations thereof and to any and all Letters Patent that may be granted therefor or thereon, including reissues or extensions, in said ASSIGNEE, its successors, or assigns, or to assist said ASSIGNEE, its successors, legal representatives, or assigns in obtaining, reissuing, or enforcing Letters Patent of the United States and any and all patents, utility models, designs or other rights that may be granted in the United States or of any country or countries foreign to the United States, for said invention.

**DECLARATION**

As a below-named inventor, I hereby declare that:

The above-identified application was made or authorized to be made by me.

I believe that I am the original inventor or an original joint inventor of a claimed invention in the application.

I hereby acknowledge that any willful false statement made in this declaration is punishable under 18 U.S.C. § 1001 by fine or imprisonment of not more than five (5) years, or both.

Signature: \_\_\_\_\_

Inventor: Pascal DRILLET

Date: 29/06/2017

**ASSIGNMENT AND DECLARATION UNDER 37 C.F.R. § 1.63**  
(For Use with Signed Application Data Sheet)

**Title of Invention:** COATED STEEL STRIPS, COATED STAMPED PRODUCTS AND METHODS

**Attorney Docket No.:** 20631.1028CON

This assignment and declaration are directed to (check one):

- ☐ the attached application;  
☐ the application identified by the attorney docket no. and title of invention given above;  
☒ United States Application or PCT International Application No. 15/606,770, filed May 26, 2017.

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**Address of Assignee:** 24-26 Blvd d'Avranches L-1160 Luxembourg, Luxembourg

(hereinafter ASSIGNEE) all right, title, and interest in and to the above-identified invention and United States patent application, including : (i) any and all divisions or continuations thereof, and in and to any and all Letters Patent of the United States that may issue on any such application or for said invention, including any and all reissues or extensions thereof, to be held and enjoyed by said ASSIGNEE, its successors, or assigns to the full end of the term or terms for which any and all such Letters Patent may be granted; (ii) all applications for patents, utility models, and designs which may hereafter be filed for said invention in any country or countries foreign to the United States, together with the right to file such applications and the right to claim for the same the priority rights derived from said United States application under the Patent Laws of the United States, the International Convention for the Protection of Industrial Property, or any other international agreement or the domestic laws of the country in which any such application is filed, as may be applicable, to be held and enjoyed by said ASSIGNEE, its successors, or assigns to the full end of the term or terms for which any and all such patents, utility models, designs or other rights may be granted; and (iii) all forms of industrial property protection, including, without limitation, patents, utility models, inventors' certificates and designs which may be granted for said invention in any country or countries foreign to the United States and all extensions, renewals and reissues thereof, to be held and enjoyed by said ASSIGNEE, its successors, or assigns to the full end of the term or terms for which any and all such patents, utility models, designs or other rights may be granted.

I hereby authorize and request the Director of the U.S. Patent and Trademark Office to issue any and all such Letters Patent to said ASSIGNEE, its successors, or assigns in accordance herewith.

I warrant and covenant that I have the right to sell and assign the interests herein sold and assigned and that I have not executed and will not execute any document or instrument in conflict herewith.

I further covenant and agree that I will communicate to said ASSIGNEE, its successors, legal representatives, or assigns all information known to me relating to said invention or patent application and that I will execute and deliver any papers, make all rightful oaths, testify in any legal proceedings, and perform all other lawful acts necessary or desirable by said ASSIGNEE, its successors, legal representatives, or assigns to perfect title to said invention, to said application including divisions and continuations thereof and to any and all Letters Patent that may be granted therefor or thereon, including reissues or extensions, in said ASSIGNEE, its successors, or assigns, or to assist said ASSIGNEE, its successors, legal representatives, or assigns in obtaining, reissuing, or enforcing Letters Patent of the United States and any and all patents, utility models, designs or other rights that may be granted in the United States or of any country or countries foreign to the United States, for said invention.

**DECLARATION**

As a below-named inventor, I hereby declare that:

The above-identified application was made or authorized to be made by me.

I believe that I am the original inventor or an original joint inventor of a claimed invention in the application.

I hereby acknowledge that any willful false statement made in this declaration is punishable under 18 U.S.C. § 1001 by fine or imprisonment of not more than five (5) years, or both.

Signature: \_\_\_\_\_

Inventor: \_\_\_\_\_

**Ronald KEFFERSTEIN**

Date: \_\_\_\_\_

1/7/2017

**POWER OF ATTORNEY TO PROSECUTE APPLICATIONS BEFORE  
THE UNITED STATES PATENT AND TRADEMARK OFFICE**

This Power of Attorney is submitted by:

Submitter Name: **ArcelorMittal**

Address: **24-26 Boulevard d'Avranches  
1160 Luxembourg  
LUXEMBOURG**

The Submitter hereby appoints the practitioners at **Davidson, Davidson & Kappel, LLC** associated with the Customer Number:

**23280**

as its attorney(s) or agent(s) to represent the Submitter before the United States Patent and Trademark Office (USPTO) and to transact all business before the USPTO in connection with any and all applications of which the Submitter is the/an applicant and the/an assignee or entity to whom the inventor(s) is/are obligated to assign.

Please direct all correspondence for any and all applications in which this Power of Attorney is submitted to the address associated with the Customer Number:

**23280**

The individual(s) whose name(s) and title(s) is/are supplied below is/are authorized to act on behalf of the Submitter.

Date: March 3, 2016

By 

Name: **Sophie Plaisant**  
Title: **Head of Central Patent Team  
Corporate Officer**

Date: \_\_\_\_\_

By \_\_\_\_\_

Name:

Title:



# UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE  
United States Patent and Trademark Office  
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P.O. Box 1450  
Alexandria, Virginia 22313-1450  
www.uspto.gov

| APPLICATION<br>NUMBER | FILING or<br>371(c) DATE | GRP ART<br>UNIT | FIL FEE REC'D | ATTY. DOCKET NO   | TOT CLAIMS | IND CLAIMS |
|-----------------------|--------------------------|-----------------|---------------|-------------------|------------|------------|
| 16/413,799            | 05/16/2019               | 1784            | 2020          | 11020631.1028con5 | 23         | 1          |

**CONFIRMATION NO. 7637**

## FILING RECEIPT



0000000108448728

23280  
Davidson, Davidson & Kappel, LLC  
589 8th Avenue  
16th Floor  
New York, NY 10018

Date Mailed: 05/28/2019

Receipt is acknowledged of this non-provisional utility patent application. The application will be taken up for examination in due course. Applicant will be notified as to the results of the examination. Any correspondence concerning the application must include the following identification information: the U.S. APPLICATION NUMBER, FILING DATE, NAME OF FIRST INVENTOR, and TITLE OF INVENTION. Fees transmitted by check or draft are subject to collection.

**Please verify the accuracy of the data presented on this receipt.** If an error is noted on this Filing Receipt, please submit a written request for a corrected Filing Receipt, including a properly marked-up ADS showing the changes with strike-through for deletions and underlining for additions. If you received a "Notice to File Missing Parts" or other Notice requiring a response for this application, please submit any request for correction to this Filing Receipt with your reply to the Notice. When the USPTO processes the reply to the Notice, the USPTO will generate another Filing Receipt incorporating the requested corrections provided that the request is grantable.

### Inventor(s)

Pascal DRILLET, Rozerieulles, FRANCE;  
Dominique Spehner, Balan, FRANCE;  
Ronald Kefferstein, Saint Mitre Les Ramparts, FRANCE;

### Applicant(s)

ArcelorMittal, Luxembourg, LUXEMBOURG;

**Power of Attorney:** The patent practitioners associated with Customer Number 23280

### Domestic Priority data as claimed by applicant

This application is a CON of 15/606,770 05/26/2017  
which is a CON of 13/621,015 09/15/2012 PAT 9708683  
which is a DIV of 12/447,777 03/18/2011 PAT 8307680  
which is a 371 of PCT/IB06/04019 10/30/2006

**Foreign Applications** for which priority is claimed (You may be eligible to benefit from the **Patent Prosecution Highway** program at the USPTO. Please see <http://www.uspto.gov> for more information.) - None.

*Foreign application information must be provided in an Application Data Sheet in order to constitute a claim to foreign priority. See 37 CFR 1.55 and 1.76.*

**Permission to Access Application via Priority Document Exchange:** Yes

**Permission to Access Search Results:** Yes

Applicant may provide or rescind an authorization for access using Form PTO/SB/39 or Form PTO/SB/69 as appropriate.

**If Required, Foreign Filing License Granted:** 05/23/2019

The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 16/413,799**

**Projected Publication Date:** 09/05/2019

**Non-Publication Request:** No

**Early Publication Request:** No  
**Title**

Coated Steel Strips, Coated Stamped Products and Methods

**Preliminary Class**

428

**Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications:** No

## **PROTECTING YOUR INVENTION OUTSIDE THE UNITED STATES**

Since the rights granted by a U.S. patent extend only throughout the territory of the United States and have no effect in a foreign country, an inventor who wishes patent protection in another country must apply for a patent in a specific country or in regional patent offices. Applicants may wish to consider the filing of an international application under the Patent Cooperation Treaty (PCT). An international (PCT) application generally has the same effect as a regular national patent application in each PCT-member country. The PCT process **simplifies** the filing of patent applications on the same invention in member countries, but **does not result** in a grant of "an international patent" and does not eliminate the need of applicants to file additional documents and fees in countries where patent protection is desired.

Almost every country has its own patent law, and a person desiring a patent in a particular country must make an application for patent in that country in accordance with its particular laws. Since the laws of many countries differ in various respects from the patent law of the United States, applicants are advised to seek guidance from specific foreign countries to ensure that patent rights are not lost prematurely.

Applicants also are advised that in the case of inventions made in the United States, the Director of the USPTO must issue a license before applicants can apply for a patent in a foreign country. The filing of a U.S. patent application serves as a request for a foreign filing license. The application's filing receipt contains further information and guidance as to the status of applicant's license for foreign filing.

Applicants may wish to consult the USPTO booklet, "General Information Concerning Patents" (specifically, the section entitled "Treaties and Foreign Patents") for more information on timeframes and deadlines for filing foreign patent applications. The guide is available either by contacting the USPTO Contact Center at 800-786-9199, or it can be viewed on the USPTO website at <http://www.uspto.gov/web/offices/pac/doc/general/index.html>.

For information on preventing theft of your intellectual property (patents, trademarks and copyrights), you may wish to consult the U.S. Government website, <http://www.stopfakes.gov>. Part of a Department of Commerce initiative, this website includes self-help "toolkits" giving innovators guidance on how to protect intellectual property in specific

countries such as China, Korea and Mexico. For questions regarding patent enforcement issues, applicants may call the U.S. Government hotline at 1-866-999-HALT (1-866-999-4258).

**LICENSE FOR FOREIGN FILING UNDER**  
**Title 35, United States Code, Section 184**  
**Title 37, Code of Federal Regulations, 5.11 & 5.15**

**GRANTED**

The applicant has been granted a license under 35 U.S.C. 184, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" followed by a date appears on this form. Such licenses are issued in all applications where the conditions for issuance of a license have been met, regardless of whether or not a license may be required as set forth in 37 CFR 5.15. The scope and limitations of this license are set forth in 37 CFR 5.15(a) unless an earlier license has been issued under 37 CFR 5.15(b). The license is subject to revocation upon written notification. The date indicated is the effective date of the license, unless an earlier license of similar scope has been granted under 37 CFR 5.13 or 5.14.

This license is to be retained by the licensee and may be used at any time on or after the effective date thereof unless it is revoked. This license is automatically transferred to any related applications(s) filed under 37 CFR 1.53(d). This license is not retroactive.

The grant of a license does not in any way lessen the responsibility of a licensee for the security of the subject matter as imposed by any Government contract or the provisions of existing laws relating to espionage and the national security or the export of technical data. Licensees should apprise themselves of current regulations especially with respect to certain countries, of other agencies, particularly the Office of Defense Trade Controls, Department of State (with respect to Arms, Munitions and Implements of War (22 CFR 121-128)); the Bureau of Industry and Security, Department of Commerce (15 CFR parts 730-774); the Office of Foreign Assets Control, Department of Treasury (31 CFR Parts 500+) and the Department of Energy.

**NOT GRANTED**

No license under 35 U.S.C. 184 has been granted at this time, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" DOES NOT appear on this form. Applicant may still petition for a license under 37 CFR 5.12, if a license is desired before the expiration of 6 months from the filing date of the application. If 6 months has lapsed from the filing date of this application and the licensee has not received any indication of a secrecy order under 35 U.S.C. 181, the licensee may foreign file the application pursuant to 37 CFR 5.15(b).

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The United States represents the largest, most dynamic marketplace in the world and is an unparalleled location for business investment, innovation, and commercialization of new technologies. The U.S. offers tremendous resources and advantages for those who invest and manufacture goods here. Through SelectUSA, our nation works to promote and facilitate business investment. SelectUSA provides information assistance to the international investor community; serves as an ombudsman for existing and potential investors; advocates on behalf of U.S. cities, states, and regions competing for global investment; and counsels U.S. economic development organizations on investment attraction best practices. To learn more about why the United States is the best country in the world to develop

technology, manufacture products, deliver services, and grow your business, visit <http://www.SelectUSA.gov> or call +1-202-482-6800.

| <b>PATENT APPLICATION FEE DETERMINATION RECORD</b><br>Substitute for Form PTO-875                                                                                                                                                                                                                                                                                                                          |                                                                 |                                  |          |                                    |               | Application or Docket Number<br>16/413,799 |                    |                         |                    |  |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------|----------------------------------|----------|------------------------------------|---------------|--------------------------------------------|--------------------|-------------------------|--------------------|--|
| <b>APPLICATION AS FILED - PART I</b>                                                                                                                                                                                                                                                                                                                                                                       |                                                                 |                                  |          |                                    |               |                                            |                    |                         |                    |  |
| (Column 1)                                                                                                                                                                                                                                                                                                                                                                                                 |                                                                 | (Column 2)                       |          | SMALL ENTITY                       |               | OTHER THAN SMALL ENTITY                    |                    |                         |                    |  |
| FOR                                                                                                                                                                                                                                                                                                                                                                                                        | NUMBER FILED                                                    | NUMBER EXTRA                     | RATE(\$) | FEE(\$)                            |               | RATE(\$)                                   | FEE(\$)            |                         |                    |  |
| BASIC FEE<br>(37 CFR 1.16(a), (b), or (c))                                                                                                                                                                                                                                                                                                                                                                 | N/A                                                             | N/A                              | N/A      |                                    |               | N/A                                        | 300                |                         |                    |  |
| SEARCH FEE<br>(37 CFR 1.16(k), (l), or (m))                                                                                                                                                                                                                                                                                                                                                                | N/A                                                             | N/A                              | N/A      |                                    |               | N/A                                        | 660                |                         |                    |  |
| EXAMINATION FEE<br>(37 CFR 1.16(o), (p), or (q))                                                                                                                                                                                                                                                                                                                                                           | N/A                                                             | N/A                              | N/A      |                                    |               | N/A                                        | 760                |                         |                    |  |
| TOTAL CLAIMS<br>(37 CFR 1.16(j))                                                                                                                                                                                                                                                                                                                                                                           | 23      minus 20 =                                              | *                                |          |                                    | OR            | x    100    =                              | 300                |                         |                    |  |
| INDEPENDENT CLAIMS<br>(37 CFR 1.16(h))                                                                                                                                                                                                                                                                                                                                                                     | 1      minus 3 =                                                | *                                |          |                                    |               | x    460    =                              | 0.00               |                         |                    |  |
| APPLICATION SIZE FEE<br>(37 CFR 1.16(s))<br>If the specification and drawings exceed 100 sheets of paper, the application size fee due is \$310 (\$155 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).                                                                                                                                  |                                                                 |                                  |          |                                    |               |                                            | 0.00               |                         |                    |  |
|                                                                                                                                                                                                                                                                                                                                                                                                            |                                                                 |                                  |          |                                    |               |                                            | 0.00               |                         |                    |  |
| MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j))                                                                                                                                                                                                                                                                                                                                                          |                                                                 |                                  |          |                                    |               |                                            | 0.00               |                         |                    |  |
|                                                                                                                                                                                                                                                                                                                                                                                                            |                                                                 |                                  | TOTAL    |                                    |               | TOTAL                                      | 2020               |                         |                    |  |
| * If the difference in column 1 is less than zero, enter "0" in column 2.                                                                                                                                                                                                                                                                                                                                  |                                                                 |                                  |          |                                    |               |                                            |                    |                         |                    |  |
| <b>APPLICATION AS AMENDED - PART II</b>                                                                                                                                                                                                                                                                                                                                                                    |                                                                 |                                  |          |                                    |               |                                            |                    |                         |                    |  |
| (Column 1)                                                                                                                                                                                                                                                                                                                                                                                                 |                                                                 | (Column 2)                       |          | (Column 3)                         |               | SMALL ENTITY                               |                    | OTHER THAN SMALL ENTITY |                    |  |
| AMENDMENT A                                                                                                                                                                                                                                                                                                                                                                                                |                                                                 | CLAIMS REMAINING AFTER AMENDMENT |          | HIGHEST NUMBER PREVIOUSLY PAID FOR | PRESENT EXTRA | RATE(\$)                                   | ADDITIONAL FEE(\$) | RATE(\$)                | ADDITIONAL FEE(\$) |  |
|                                                                                                                                                                                                                                                                                                                                                                                                            | Total<br>(37 CFR 1.16(i))                                       | *                                | Minus    | **                                 | =             | x      =                                   |                    | OR                      | x      =           |  |
|                                                                                                                                                                                                                                                                                                                                                                                                            | Independent<br>(37 CFR 1.16(h))                                 | *                                | Minus    | ***                                | =             | x      =                                   |                    | OR                      | x      =           |  |
|                                                                                                                                                                                                                                                                                                                                                                                                            | Application Size Fee (37 CFR 1.16(s))                           |                                  |          |                                    |               |                                            |                    | OR                      |                    |  |
|                                                                                                                                                                                                                                                                                                                                                                                                            | FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j)) |                                  |          |                                    |               |                                            |                    | OR                      |                    |  |
|                                                                                                                                                                                                                                                                                                                                                                                                            |                                                                 |                                  |          |                                    |               | TOTAL<br>ADD'L FEE                         |                    | OR                      | TOTAL<br>ADD'L FEE |  |
| AMENDMENT B                                                                                                                                                                                                                                                                                                                                                                                                |                                                                 | CLAIMS REMAINING AFTER AMENDMENT |          | HIGHEST NUMBER PREVIOUSLY PAID FOR | PRESENT EXTRA | RATE(\$)                                   | ADDITIONAL FEE(\$) | RATE(\$)                | ADDITIONAL FEE(\$) |  |
|                                                                                                                                                                                                                                                                                                                                                                                                            | Total<br>(37 CFR 1.16(i))                                       | *                                | Minus    | **                                 | =             | x      =                                   |                    | OR                      | x      =           |  |
|                                                                                                                                                                                                                                                                                                                                                                                                            | Independent<br>(37 CFR 1.16(h))                                 | *                                | Minus    | ***                                | =             | x      =                                   |                    | OR                      | x      =           |  |
|                                                                                                                                                                                                                                                                                                                                                                                                            | Application Size Fee (37 CFR 1.16(s))                           |                                  |          |                                    |               |                                            |                    | OR                      |                    |  |
|                                                                                                                                                                                                                                                                                                                                                                                                            | FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j)) |                                  |          |                                    |               |                                            |                    | OR                      |                    |  |
|                                                                                                                                                                                                                                                                                                                                                                                                            |                                                                 |                                  |          |                                    |               | TOTAL<br>ADD'L FEE                         |                    | OR                      | TOTAL<br>ADD'L FEE |  |
| * If the entry in column 1 is less than the entry in column 2, write "0" in column 3.<br>** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20".<br>*** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3".<br>The "Highest Number Previously Paid For" (Total or Independent) is the highest found in the appropriate box in column 1. |                                                                 |                                  |          |                                    |               |                                            |                    |                         |                    |  |



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www.uspto.gov

| APPLICATION NO.                  | FILING DATE | FIRST NAMED INVENTOR | ATTORNEY DOCKET NO. | CONFIRMATION NO. |
|----------------------------------|-------------|----------------------|---------------------|------------------|
| 16/413,799                       | 05/16/2019  | Pascal DRILLET       | 11020631.1028con5   | 7637             |
| 23280                            | 7590        | 06/04/2019           | EXAMINER            |                  |
| Davidson, Davidson & Kappel, LLC |             |                      | ART UNIT            | PAPER NUMBER     |
| 589 8th Avenue                   |             |                      | 1784                |                  |
| 16th Floor                       |             |                      | NOTIFICATION DATE   | DELIVERY MODE    |
| New York, NY 10018               |             |                      | 06/04/2019          | ELECTRONIC       |

**Please find below and/or attached an Office communication concerning this application or proceeding.**

The time period for reply, if any, is set in the attached communication.

Notice of the Office communication was sent electronically on above-indicated "Notification Date" to the following e-mail address(es):

ddk@ddkpatent.com

|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       |                                              |                                       |                                |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------|---------------------------------------|--------------------------------|
| <b><i>Decision Granting Request for Prioritized Examination (Track I)</i></b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         | <b>Application No.</b><br>16/413,799         | <b>Applicant(s)</b><br>DRILLET et al. |                                |
|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       | <b>Examiner</b><br>CHERYL P GIBSON<br>BAYLOR | <b>Art Unit</b><br>OPET               | <b>AIA (FITF) Status</b><br>No |
| <p>1. THE REQUEST FILED <u>16 May 2019</u> IS <b>GRANTED</b> .</p> <p>The above-identified application has met the requirements for prioritized examination</p> <p>A. <input checked="" type="checkbox"/> for an original nonprovisional application (Track I).</p> <p>B. <input type="checkbox"/> for an application undergoing continued examination (RCE).</p> <p>2. <b>The above-identified application will undergo prioritized examination.</b> The application will be accorded special status throughout its entire course of prosecution until one of the following occurs:</p> <p>A. filing a <b><u>petition for extension of time</u></b> to extend the time period for filing a reply;</p> <p>B. filing an <b><u>amendment to amend the application to contain more than four independent claims, more than thirty total claims</u></b>, or a multiple dependent claim;</p> <p>C. filing a <b><u>request for continued examination</u></b> ;</p> <p>D. filing a notice of appeal;</p> <p>E. filing a request for suspension of action;</p> <p>F. mailing of a notice of allowance;</p> <p>G. mailing of a final Office action;</p> <p>H. completion of examination as defined in 37 CFR 41.102; or</p> <p>I. abandonment of the application.</p> <p>Telephone inquiries with regard to this decision should be directed to CHERYL GIBSON BAYLOR at (571 )272-3213. In his/her absence, calls may be directed to Petition Help Desk at (571) 272-3282.</p> |                                              |                                       |                                |
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(54) **COATED STEEL STRIPS, METHODS OF MAKING THE SAME, METHODS OF USING THE SAME, STAMPING BLANKS PREPARED FROM THE SAME, STAMPED PRODUCTS PREPARED FROM THE SAME, AND ARTICLES OF MANUFACTURE WHICH CONTAIN SUCH A STAMPED PRODUCT**

(57) Coated steel having a well defined and uniform thickness is useful and advantageous for preparing products by hot stamping. Products prepared by hot stamping

such a steel are particularly advantageous when subjected to a subsequent spot welding step.

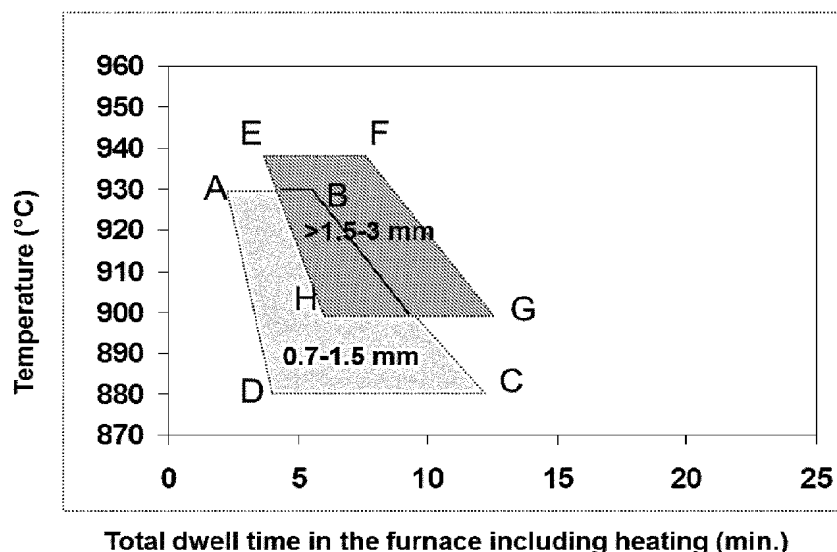


**FIG.7**

## Description

### FIELD OF THE INVENTION

**[0001]** The present invention relates to, among other things, coated steels, methods of making such coated steels, including hot dipping, methods of using such coated steels, stamping blanks prepared from coated steels, stamped products prepared from coated steels, and to various uses of the invention products such as in spot welding, etc.

**[0002]** Additional advantages and other features of the present invention will be set forth in part in the description that follows and in part will become apparent to those having ordinary skill in the art upon examination of the following or may be learned from the practice of the present invention. The advantages of the present invention may be realized and obtained as particularly pointed out in the appended claims. As will be realized, the present invention is capable of other and different embodiments, and its several details are capable of modifications in various obvious respects, all without departing from the present invention. The description is to be regarded as illustrative in nature, and not as restrictive.

### BACKGROUND OF THE INVENTION

**[0003]** In recent years the use of pre-coated steels in hot-stamping processes for the shaping of parts has become important, especially in the automotive industry. Fabrication of such parts may include the following main steps:

- Pre-coating of a steel sheets, by hot dipping
- Trimming or cutting for obtaining blanks
- Heating the blanks in order to obtain alloying of the steel substrate with the pre-coating, as well as the austenitizing of the steel
- Hot forming followed by rapid cooling of the part in order to obtain predominantly martensitic structures

See for Example U.S. 6,296,805, incorporated herein by reference.

**[0004]** Thanks to an alloying of the pre-coating with the steel substrate, which has the effect of creating intermetallic alloys with high melting temperature, the blanks having such coating may be heated in a temperature range where austenitizing of the metallic substrate takes place, allowing further hardening by quenching.

**[0005]** Heat treatments of the blanks in view of the intermetallic alloying of the coating and austenitizing of the substrate are most frequently performed in furnaces, where blanks are traveling on rollers. The thermal cycles experienced by the blanks include first a heating phase whose rate is a function of parameters such as blank thickness, furnace temperature, traveling speed, and coating reflectivity. After this heating phase, thermal cycles generally include a holding phase, whose tempera-

ture is the regulation temperature of the furnace. Problems however are experienced with the furnace operation: the rollers may become fouled by metallic deposits which come from the pre-coating of the blanks. If these deposits are excessive, maintenance of the rollers has to be performed and productivity is decreased.

**[0006]** Parts obtained after heating and rapid cooling display very high mechanical resistance and may be used for structural applications, for example for automotive industry applications. These parts must be frequently welded with others and high weldability is required. This means that:

- The welding operation should be performable in a sufficiently wide operating range in order to guarantee that an eventual drift of the nominal welding parameters has no incidence on weld quality. For resistance welding, which is very common in the automotive industry, an operating welding range is defined by the combination of parameters: welding current intensity I and force F applied of the parts during welding being among the most important. A proper combination of these parameters helps to ensure that insufficient nugget diameter is not obtained (caused by too low intensity or too low force) and that no weld expulsion occurs.
- The welding operation should also be performed in such a way that high mechanical resistance is obtained on the weld. This mechanical resistance may be evaluated by tests such as by shear-tensile tests or cross-tensile tests.

**[0007]** There remains a need for coated steels which may be conveniently used to prepare shaped parts by a stamping process. There also remains a need for coated steels which may be used to prepare shaped parts by a stamping process which are suitable for welding. There also remains a need for processes for preparing such coated steels and stamped parts.

### SUMMARY OF THE INVENTION

**[0008]** The inventors have discovered that certain coated steels in which a base steel strip is at least partially coated (sometimes termed "pre-coated," this prefix indicating that a transformation of the nature of the pre-coating will take place during heat treatment before stamping) on at least one side with a coating of either aluminum or an aluminum alloy and in which the coating has a defined thickness and is preferably substantially uniform, are conveniently formed into shaped parts after heating by stamping and are conveniently welded. In addition, the inventors have discovered that the roller fouling problem described above generally arises from an insufficient degree of intermetallic alloying between the substrate and the metallic pre-coating. Furthermore, it was discovered that the location of the fouling of the rollers corresponds to zones of the blanks in contact with the rollers where

the metallic pre-coating thickness locally exceeds the average thickness. While not bound by a particular theory, it is believed that if a pre-coating is locally too thick, intermetallic alloying is insufficient and the pre-coating melts, fouling the rollers. Thus, the inventors have discovered that the control of the homogeneity of the pre-coating thickness over the entire sheet within given tolerances is an important factor for obtaining the desired degree of intermetallic alloying, allowing one to improve the resistance to the subsequent melting of the coating during travel on rollers.

**[0009]** The inventors have also discovered that particularly good weldability of aluminized and hot stamped parts is associated with a special succession of coating layers on the parts, proceeding from steel substrate outwards.

**[0010]** The inventors have also discovered that a specific combination of transfer time between the heating furnace and the stamping die, the amount of deformation during stamping, the temperature of stamping, the cooling rate of the product during stamping, leads to the fabrication of a part with a fully homogeneous martensitic structure and that an increase in ductility or energy absorption of the parts after stamping is obtained by a reduction of sulfur below a critical value, these two benefits being obtained with or without the invention aluminum/aluminum alloy coating, and with other coatings.

#### OBJECTS OF THE INVENTION

**[0011]** Accordingly, and in view of the above, it is one object of the present invention to provide novel pre-coated steels strips which may be conveniently processed into stamping blanks.

**[0012]** It is another object of the present invention to provide novel pre-coated steels strips or sheets which may be conveniently formed into parts by stamping.

**[0013]** It is another object of the present invention to provide novel coated steels which may be conveniently formed into parts by hot stamping.

**[0014]** It is another object of the present invention to provide novel methods for making such a coated steel.

**[0015]** It is another object of the present invention to provide novel stamping blanks which are prepared from such a coated steel.

**[0016]** It is another object of the present invention to provide novel methods of making such stamping blanks.

**[0017]** It is another object of the present invention to provide novel stamped parts which are prepared from such a coated steel.

**[0018]** It is another object of the present invention to provide novel methods of making such stamped parts.

**[0019]** It is another object of the present invention to provide novel articles of manufacture, such as a motor vehicle, which contain such stamped parts.

**[0020]** It is another object of the present invention to provide novel stamped parts.

**[0021]** It is another object of the present invention to

provide novel methods of making welded stamped parts.

**[0022]** It is another object of the present invention to provide novel articles of manufacture, such as a motor vehicle, which contain such welded stamped parts.

**[0023]** It is another object of the present invention to provide novel welded coated steels and welded stamping blanks.

**[0024]** It is another object of the present invention to provide novel methods of making such welded coated steels and welded stamping blanks.

**[0025]** These and other objects, which will become apparent during the following detailed description.

#### BRIEF DESCRIPTION OF THE DRAWINGS

##### **[0026]**

Figure 1 illustrates a preferred embodiment of an invention coated part, after heat treatment and stamping. The succession of layers of the coating on the steel substrate is: (a) interdiffusion layer; (b) intermediate layer; (c) Intermetallic layer; and (d) superficial layer. This arrangement is particularly favorable for the further welding of the part.

Figure 2 illustrates a coating of a steel substrate after heat treatment and stamping which does not correspond to the invention. This succession of layers (interdiffusion layer and intermetallic layer) yields inferior results in resistance spot welding.

Figure 3 illustrates a microstructure of a steel part, hot stamped and cooled under conditions not according to the invention.

Figure 4 illustrates the microstructure of a steel part, hot stamped and cooled according to a preferred set of conditions according to the invention.

Figure 5 illustrates the influence of sulfur on the bending angle of parts after hot stamping.

Figure 6 illustrates the influence of sulfur on the initiation energy of fracture of parts after hot stamping.

Figure 7 shows conditions of furnace temperature as a function of the total dwell time in the furnace for sheets of total thicknesses of from 0.7-1.5 and 1.5-3 mm that provide particularly favorable coatings for welding.

#### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

**[0027]** As noted above, the inventors' discovery that certain coated steels, in which a base steel is at least partially pre-coated on at least one side with a coating of either aluminum or an aluminum alloy and in which the pre-coating has a defined thickness and is substantially uniform, are conveniently formed into shaped parts by stamping, forms one basis for the invention.

**[0028]** In the context of the present invention, the terms first side (or side 1) and second side (or side 2) of the strip or sheet, etc., of base steel refer to the two large,

opposite-facing, surfaces which have a surface area defined by the length and width of the strip of base steel. In contrast, the side edges of the strip of base steel are the two small, opposite-facing, surfaces which have a surface area defined by the length and thickness of the strip. The top and bottom edges of the strip of base steel are the two small, opposite-facing, surfaces which have a surface area defined by the width and thickness of the strip. In the following,  $t_p$  designates the thickness of the pre-coating, at any considered location on the sides 1 and 2 of a sheet or a blank. In particular, in the case of sheets coated on two sides 1 and 2,  $t_{p1}$  stands for the thickness on side 1, and  $t_{p2}$  for the thickness on side 2. According to a highly preferred embodiment  $t_p$  is controlled in a precise range, expressed by  $(t_{pmin}, t_{pmax})$ , in order to improve the resistance to the fouling of the rollers. The thickness is preferably controlled, both in the longitudinal (or rolling) direction of the strip or sheet, as well in the transverse direction.

**[0029]** In relation to the problem of the fouling of rollers, the control of the pre-coating thickness on the side of the sheet or blank which is directly in contact with the rollers is especially important. Because different operations can follow the step of coating the steel sheet (for example hot-dip-coating which provides coated sides 1 and 2), it is preferable to carefully control the pre-coating on both sides of the sheet. For example, after any of coiling, handling, cutting, punching, etc., sides 1 and 2 may be not readily identifiable. But when control of  $t_p$  is effected on the two sides of the sheet that has been coated (first side and second side) it is not necessary to track sides 1 and 2 as neither side will foul the roller. In addition, it is not necessary to trim the sheet in order to obtain a preferred smaller sheet having a more uniform homogeneity of pre-coating thickness, thus providing a sheet that is pre-coated by, e.g., hot-dipping. In other words, important benefits are obtained when one controls the pre-coating minimum and maximum thickness of the first side ( $t_{pmin1}$ ,  $t_{pmax1}$ ) and the pre-coating minimum and maximum thickness of the second side ( $t_{pmin2}$ ,  $t_{pmax2}$ ) of the subject steel sheet or blank.

Hot-dip coated steels are the preferred steels herein. However, regardless of the coating method, the pre-coating thickness on one or both sides of the sheet can be measured and monitored continuously on a coating line directly after the coating operation. This may be realized by devices known per se, such as thickness gages relying on X-Ray absorption. At every moment, the measurement of thickness at a given location may be performed for example on an area of a few hundreds of  $mm^2$ , this representing the dimension of the zone irradiated by X-Ray.

**[0030]** In a preferred embodiment, a plurality of such devices are positioned at different distances in the transverse direction of the strip in order to obtain a profile of the thickness of the pre-coating along the strip width.

**[0031]** The inventors have discovered that the resistance to the pollution or fouling of the rollers in the furnaces

is improved when the minimum and maximum thickness respectively of at least one of the first side ( $t_{pmin1}$ ,  $t_{pmax1}$ ) and the second side ( $t_{pmin2}$ ,  $t_{pmax2}$ ) are respectively equal to 20 and 33 micrometers (a micrometer is the same as a micron and is a metric unit of length equal to one millionth of a meter). In other words, in a preferred embodiment, at every location on at least one face of the sheet or the blank, the thickness  $t_p$  of the pre-coating is preferably from 20 - 33 micrometers in thickness, including 21, 22, 23, 24, 25, 26, 27, 28, 29, 30, 31 and 32 micrometers and all ranges and subranges therebetween, and including all numbers between each listed value as if written out in its entirety (e.g., 22.34 micrometers). For hot-dip coating, precise control of this range of pre-coating thickness can be realized on an operating line for example using a system of nozzles blowing gas after coating, for example after exit of the strip or sheet from a bath, and by the flatness of the strip. Number, geometry and location of the nozzles, and flow rates are among the main parameters for a precise control of the thickness  $t_p$ . Given the present disclosure, one of ordinary skill in this art can control the pre-coating thickness as described herein without undue labor.

The invention refers to strips produced in industrial conditions, i.e. where control of pre-coating thickness is effective over a wide surface strip, i.e. with a length greater than 100m, and with a width greater than 600mm. In such a way, blanks cut or trimmed out these strips display very high homogeneity of pre-coating thickness, and the settings of the heating treatments in the furnace do not have to be changed for being adapted to eventual variations of this thickness.

**[0032]** While not bound by a particular theory of operation, the inventors believe that several of the benefits of the invention are related to this pre-coating thickness range, such as the following :

- For a pre-coating thickness less than 20 micrometers, the alloyed layer which is formed during the heating of the blank has an insufficient roughness. Thus, the adhesion of subsequent painting is low on this surface, and the corrosion resistance is decreased.
- If the pre-coating thickness is more than 33 micrometers at a given location on a sheet, the risk is that the difference of thickness between this location and some other locations where the pre-coating is thinner, becomes too important : The settings of the heating treatment in the furnace may be adapted to the thinner value of the pre-coating, but not to the thicker one. Thus, the alloying reaction forming the intermetallic alloy may take place to an insufficient degree since the mean diffusion distance of the elements in the pre-coating becomes significantly less than the local value of the pre-coating thickness. As a matter of results, alloying will become much more difficult in the external (or superficial) part, particularly in the case of high heating rate.

**[0033]** Thus, in a first embodiment, the present invention provides certain coated steel strips, which comprise a strip of base steel and a pre-coating of aluminum or an aluminum alloy on at least a part of one side of the strip of the base steel. For many applications, the strip of base steel may comprise any type of steel which may be coated with either aluminum or an aluminum alloy. However, for certain applications, such as a structural part of an automobile, it is preferred that the strip of base steel comprise an ultra high strength steel (UHSS). In such cases, it is particularly preferred that the strip of base steel comprises a boron steel.

**[0034]** The inventors have also discovered that good welding results are achieved if the coating obtained on parts made out of blanks having undergone intermetallic alloying, austenitizing and hot stamping, displays distinctive features. It must be pointed out that this coating is different from the initial pre-coating, since the thermal treatment causes an alloying reaction with the steel substrate which modifies both the physico-chemical nature and the geometry of the pre-coating : in this regard, the inventors have discovered that particularly good weldability of aluminized and hot stamped parts is associated with the following succession of coating layers on the parts, proceeding from steel substrate outwards :

- (a) Interdiffusion layer,
- (b) Intermediate layer,
- (c) Intermetallic layer,
- (d) Superficial layer

See, e.g., Figure 1. In a preferred embodiment these layers are as follows:

- (a) Interdiffusion layer, preferably with medium hardness (e.g., HV50g between 290 and 410, HV50g designating the hardness measured under a load of 50 grams) In a preferred embodiment this layer has the following composition, by weight: 86-95%Fe, 4-10%Al, 0-5%Si
- (b) Intermediate layer (HV50g around 900 - 1000 e.g., +/- 10%) In a preferred embodiment this layer has the following composition, by weight: 39-47% Fe, 53-61%Al, 0-2%Si
- (c) Intermetallic layer, with hardness HV50g around 580-650, e.g., +/- 10%) In a preferred embodiment this layer has the following composition, by weight: 62-67%Fe, 30-34 %Al, 2-6%Si
- (d) Superficial layer (HV50g around 900 - 1000 e.g., +/- 10%)

**[0035]** In a preferred embodiment this layer has the following composition, by weight: 39-47% Fe, 53-61 %Al, 0-2%

**[0036]** In a preferred embodiment the total thickness of layers (a) to (d) is greater than 30 micrometers.

**[0037]** In another preferred embodiment, the thickness of layer (a) is less than 15 micrometers, for example 14,

12, 10, 8, 6, 4, 2, or 1 micrometers, and all whole numbers, ranges and subranges therebetween, and including all numbers between each listed value as if written out in its entirety (e.g., 13.84 micrometers).

**[0038]** The inventors have discovered that high weldability is especially obtained when layers (c) and (d) are essentially continuous (i.e.: occupying at least 90% of the level corresponding to the considered layer) and when less than 10% of layer (c) is present at the extreme surface of the part. Without being bound by a theory, it is thought that this particular layer disposal, in particular layer (a) and layers (c) and (d) influence the resistivity of the coating both by their intrinsic characteristics and by the effect of roughness. Thus, current flow, heat generation at the surfaces, and nugget formation in the initial stage of spot welding are affected by this particular arrangement.

**[0039]** This favorable layer disposition is obtained for example when aluminum- or aluminum alloy pre-coated steel sheets, whose thickness range from, e.g., 0.7 to 3mm, are heated for 3 to 13 minutes (this dwell time includes the heating phase and the holding time) in a furnace heated to a temperature of 880 to 940°C. Other conditions leading to such favorable layer dispositions are found in Figure 7:

For sheets of total thicknesses greater or equal to 0.7mm, and less than or equal to 1.5mm, the preferred treatment conditions : (furnace temperature, total dwell time in the furnace) are illustrated in figure 7 by conditions lying within the limits of diagram "AB-CD"

For sheets of total thicknesses greater than 1.5mm, and less than or equal to 3mm, the preferred treatment conditions : (furnace temperature, total dwell time in the furnace) are illustrated in figure 7 by diagram "EFGH" .

The heating rate  $V_c$  is comprised between 4 and 12°C/s for producing a favorable alloyed layer disposition. In this regard, this "heating rate" reflects the temperature rise which is experienced by the pre-coated steel upon being located in the preheated furnace.  $V_c$  is defined as the mean heating rate between 20 and 700°C. The inventors have discovered that the control of  $V_c$  in this precise range is a key factor, because it controls directly the nature and the morphology of the alloyed layers which are formed. It is here underlined that the heating rate  $V_c$  is different from the mean heating rate, which is the heating rate between room temperature and furnace temperature. The rates of 6, 7, 8, 9, 10, and 11 °C/s are included as are all numbers, ranges and subranges therebetween, and including all numbers between each listed value as if written out in its entirety (e.g., 7.7 °C/s). In this regard, all of the conditions specified within Figure 7 are incorporated herein by reference thereto. Particularly preferred conditions are:

(for thicknesses of 0.7-1.5mm)

**[0040]**

- 930°C, from 3 minutes up to 6 minutes;
- 880°C, from 4 minutes 30 seconds up to 13 minutes

(for thicknesses of 1.5 to 3 mm)

**[0041]**

- 940°C, from 4 minutes up to 8 minutes;
- 900°C, from 6 minutes 30 seconds up to 13 minutes

A special advantage arises from pre-coatings whose thickness is comprised between 20 and 33 micrometers, since this thickness range yields favorable layer disposal, and since the homogeneity of the pre-coating thickness is associated to an homogeneity of the coating formed after alliation treatment.

**[0042]** Heated blanks are thereafter transferred to a die, hot stamped to obtain a part or product, and cooled at a rate of more than 30°C/s. The cooling rate is defined here as the mean rate between the exit of the heated blank from the furnace, down to 400°C.

**[0043]** The strip of base steel is coated with either aluminum or an aluminum alloy. Commercially pure aluminum is known in the art as Type 2 aluminum, while alloys of aluminum with 5 to 11 % by weight of silicon are known in the art as Type 1 aluminum. Silicon is present in order to prevent the formation of a thick iron-metallic intermetallic layer which reduces adherence and formability. Other alloying elements useful with aluminum herein include iron, between 2.5 and 3% by weight, and calcium, between 15 and 30ppm by weight, including combinations of two or more thereof with aluminum.

**[0044]** A typical metal bath for an Al-Si coating generally contains in its basic composition by weight, from 8% to 11% silicon, from 2% to 4% iron, the remainder being aluminum or aluminum alloy, and impurities inherent in processing. Typical composition of Al-Si coating is: Al-9,3%Si-2,8%Fe. Invention coatings are not limited to these compositions, however.

**[0045]** The strip of base steel used herein may be any which can be coated by a conventional coating technique. For example, the strip of base steel may be any hot rolled strip, such as those prepared by hot rolling a steel slab (either with or without subsequent cold rolling). Typically, the strip of base steel will be stored and transported in the form of a coil both before and after the formation of the coating.

**[0046]** An example of a preferred steel for the strip of base steel is one having the following composition by weight:

0.10% < carbon < 0.5%  
0.5% < manganese < 3%  
0.1 % < silicon < 1%

0.01 % < chromium < 1 %  
titanium < 0.2%  
aluminum < 0.1 %  
phosphorus < 0.1 %  
sulfur < 0.05%  
0.0005% < boron < 0.010%,

the remainder comprising, consisting essentially of, or consisting of iron and impurities inherent in processing.

Use of such a steel provides a very high mechanical resistance after thermal treatment and the aluminum-based coating provides a high resistance to corrosion.

**[0047]** Particularly preferably, the composition by weight of the steel in the strip of base steel is the following:

0.15% < carbon < 0.25%  
0.8% < manganese < 1.8%  
0.1 % < silicon < 0.35%  
0.01% < chromium < 0.5%  
titanium < 0.1%  
aluminum < 0.1 %  
phosphorus < 0.1 %  
sulfur < 0.05%  
0.002% < boron < 0.005%,

the remainder comprising, consisting essentially of, or consisting of iron and impurities inherent in processing.

**[0048]** A preferred strip herein is 100 m long and 600mm wide. Preferred thicknesses are 0.7 to 3 mm.

**[0049]** Even more preferably, in the composition by weight of the sheet, the weight ratio of titanium content with respect to the nitrogen content is in excess of 3.42, believed to be a level at which the boron is no longer able to combine with the nitrogen.

**[0050]** An example of preferred commercially available steel for use in the strip of base steel is 22MnB5.

**[0051]** Chromium, manganese, boron and carbon may be added, in the composition of the steel according to the invention, for their effect on hardenability. In addition, carbon makes it possible to achieve high mechanical characteristics thanks to its effect on the hardness of the martensite.

**[0052]** Aluminum is introduced into the composition, to perform deoxidation in the liquid state and to protect the effectiveness of the boron.

**[0053]** Titanium, the ratio of the content of which with respect to the nitrogen content should be in excess of 3.42, is introduced for example in order to prevent combining of the boron with the nitrogen, the nitrogen being combined with titanium.

**[0054]** The alloying elements, Mn, Cr, B, make possible a hardenability allowing hardening in the stamping tools or the use of mild hardening fluids limiting deformation of the parts at the time of thermal treatment. In addition, the composition according to the invention is optimized from the point of view of weldability.

**[0055]** The steel in the sheet may undergo a treatment for globularization of sulfides performed with calcium,

which has the effect of improving the fatigue resistance of the sheet.

**[0056]** As mentioned above, ultra-high-strength can be provided with the steel sheet coated and hot stamped according to the invention. This high level of strength is sometimes associated with a limited ductility. In applications requiring a higher ductility, in particular when an ability for bending is required from a part or a product, the inventors have discovered that increased ductility can be obtained if sulfur is particularly controlled: when the sulfur level of base steel is lower than or equal to 0.002% (20 ppm), the bending angle can be greater than 60° and enhanced ductility and tearing resistance are obtained on the parts having experienced heat treatment and stamping. Preferred levels include 20, 18, 15, 13, 10, 8, 5, 2, etc ppm sulfur. In fact, this benefit applies to steels in general, and is not limited to coated steels or to steels coated with Al or Al alloy coatings. While not bound by a particular theory, when analyzing causes of premature failure of some parts in bending operations, the inventors observed that failure initiated on sulfide inclusions. It is thus believed that decohesions between inclusions and the martensitic or bainite-martensitic matrix act as stress-concentration factors and trigger further crack propagation in the ductile mode.

**[0057]** The invention also concerns a process for producing a part starting from an invention coated sheet, then cut into a blank which, after shaping, the coating of the blank is subjected to an increase in temperature at a speed in excess of 4°C/second, but lower than 12°C/second. The heating rate  $V_c$  is defined as the mean rate between 20 and 700°C.

**[0058]** The invention also concerns the use of a hot-rolled steel sheet which then can be cold-rolled and coated, for structural and/or anti-intrusion or substructure parts for a land motor vehicle, such as, for example, a bumper bar, a door reinforcement, a wheel spoke, etc.

**[0059]** The sheet according to the invention described above can derive, by reason of its processing, from a hot-rolling mill, and possibly may be cold-rolled again depending on the final thickness desired. It then is coated with an aluminum-based coating, for example by dipping in a bath containing, in addition to the aluminum source/alloy, e.g., from 8% to 11% silicon and from 2% to 4% iron, the sheet having a high mechanical resistance after thermal treatment and a high resistance to corrosion, as well as a good capacity for painting and gluing.

**[0060]** The coating is preferably controlled as above, and has in particular the function of protecting the basic sheet against corrosion in various conditions. The thermal treatment applied at the time of a hot-forming process or after forming makes it possible to obtain high mechanical characteristics which can exceed 1500 MPa for mechanical resistance and 1200 MPa for yield stress. The final mechanical characteristics are adjustable and depend in particular on the martensite fraction of the structure, on the carbon content of the steel and on the thermal treatment. At the time of thermal treatment performed on

a finished part or at the time of a hot-shaping process, the coating forms a layer having a substantial resistance to abrasion, wear, fatigue, shock, as well as a good resistance to corrosion and a good capacity for painting and gluing. The coating makes it possible to avoid different surface-preparation operations such as for steel sheets for thermal treatment not having any coating.

**[0061]** The steel sheet can be pre-coated by dipping, after pickling, in an aluminum bath containing for example only aluminum or either aluminum and from 8% to 11% silicon and 2% to 4% iron, or only from 2% to 4% iron, or even in an aluminum bath preferably containing from 9% to 10% silicon and 2% to 3.5% iron. The aluminum may be aluminum per se or an aluminum alloy.

**[0062]** In an example of implementation of a coating of the sheet by dipping in a metal bath containing an aluminum alloy comprising a proportion of approximately 90% aluminum, the coating layer comprises a first alloy layer in contact with the surface of the steel. This layer, directly in contact with the surface of the sheet, is highly alloyed with iron.

**[0063]** A second coating layer, on top of the first, contains approximately 90% aluminum and may contain silicon and a small amount of iron, depending on the composition of the bath.

**[0064]** The first alloy layer may crack when the sheet is submitted to high strains during cold forming operations of the manufacture of parts.

**[0065]** According to the invention, after the forming of the part, the coating is subjected to an increase in temperature at a speed in excess of 4°C/second. This rise in temperature makes possible a rapid remelting of the aluminum which fills in the cracks generated by the operation of shaping of the part.

**[0066]** At the time of thermal treatment, the base coating, of aluminum for example, is transformed into a layer alloyed with iron and comprising different phases depending on the thermal treatment and having a considerable hardness which may exceed 600 HV50 g.

**[0067]** Another advantage of the invention lies in the fact that the diffusion of the iron in the coating is initiated at high temperature. One thus will have a better cohesion between coating and steel in the sheet. In another form of the invention, the thermal treatment may be performed locally, in heavily deformed zones.

According to the invention, the sheet, in the delivery state in a coil or in a sheet, the thickness of which may range between 0.25 mm and 15 mm, has good forming properties and a good resistance to corrosion as well as a good capacity for painting or gluing. Preferably, the steel sheet or blank has a thickness less than 3 mm, since the cooling rates that may be achieved after quenching are high and help to obtain martensitic structures.

**[0068]** The steel sheet, a coated product, has a substantial resistance to corrosion in the delivery state, during forming and thermal treatments as well as during usage of the finished part.

The presence of the coating at the time of thermal treat-

ment of the parts makes it possible to prevent any decarburization of the base metal as well as any oxidation. That is an undeniable advantage, in particular in the case of hot forming. Furthermore, heating of the treated part does not require a furnace having a controlled atmosphere to prevent a decarburization.

**[0069]** Thermal treatment of the metal in the sheet comprising heating at a temperature ranging between  $A_{c1}$ , starting temperature of austenitic transformation when heating, for example  $750^{\circ}\text{C}$  and  $1200^{\circ}\text{C}$ , in a furnace, for a period which depends on the temperature to be reached and the thickness of the blank. The composition is optimized so as to limit the grain growth at the time of thermal treatment. If the structure sought is completely martensitic, the holding temperature should be in excess of  $A_{c3}$ , for example  $840^{\circ}\text{C}$ , temperature of complete austenitic transformation. The temperature holding should be followed by a cooling adjusted to the final structure sought.

**[0070]** Blanks are thereafter transferred from the furnace to a stamping press. When the elapsed time between the exit of the blanks from the furnace and the introduction in the stamping press is more 10 seconds, a partial transformation from austenite is susceptible to appear: if obtaining a full martensitic structure is desired, the transfer time between the exit of the furnace and stamping should be less than 10s.

**[0071]** The inventors have also discovered that the obtaining of a fully martensitic structure is linked to the amount of deformation in the hot forming operation: the amount of local deformation caused by hot forming is closely linked to the shape of the part or product and may exceed locally 40 or 50% in some particular regions. The inventors found that, when the local strain exceeds a critical value of 10%, the cooling rate must be sufficiently high in order to get a total martensitic transformation. Otherwise, bainitic transformation can take place at a significant amount instead of martensitic transformation. Thus, the risk is that heterogeneous structure appears on parts with complex shape where some locations are much more deformed than others. In this respect, the inventors put into evidence that, on the locations of the parts where the forming strain is higher than 10%, the cooling rate must be increased beyond  $50^{\circ}\text{C/s}$  in order to guarantee full martensitic transformation. The cooling rate is defined as the mean rate between the exit of the heated blank from the furnace, down to  $400^{\circ}\text{C}$ .

**[0072]** But one may also seek to obtain ferrito-bainitic or ferrito-martensitic structures, by a heating at a temperature ranging between  $A_{c1}$ , for example  $750^{\circ}\text{C}$  and  $A_{c3}$ , for example  $840^{\circ}\text{C}$ , followed by an appropriate cooling. According to the level of resistance to be achieved and the thermal treatment applied, one or several of these constituents is/are present in variable proportions.

**[0073]** The modulation of thermal treatment parameters makes it possible to achieve, with a given composition, different levels of hot and cold sheet resistance according to the thickness sought. For the highest resist-

ance levels, the structure is composed predominantly of martensite.

**[0074]** The steel is particularly suited to the production of structural and anti-intrusion parts.

**[0075]** The invention thus enables one to produce a hot- or cold-rolled steel sheet of a desired thickness, coated, and affording extensive forming possibilities and which, after thermal treatment performed on the finished part, makes it possible to obtain a mechanical resistance in excess of 1000 MPa, a substantial resistance to shocks, fatigue, abrasion and wear, while retaining a good resistance to corrosion as well as a good capacity for welding, painting and gluing.

**[0076]** The present invention will now be further described by way of a certain exemplary embodiments which are not intended to be limiting.

## EXAMPLES

### Example 1 :

**[0077]** In a first example of implementation, a cold rolled steel sheet, 1.9 mm thick, containing by weight: 0.23 % carbon, 1.25% manganese, 0.017% phosphorus, 0.002% sulfur, 0.27% silicon, 0.062% aluminum, 0.021 % copper, 0.019% nickel, 0.208% chromium, 0.005% nitrogen, 0.038% titanium, 0.004% boron, 0.003% calcium-has been pre-coated with an aluminum-based alloy with composition 9.3% silicon, 2.8% iron, the remainder being aluminum and unavoidable impurities. According to the conditions of fabrication, namely the settings of the blowing devices on the operating line, sheets of 120m long and 650mm wide with various thickness ranges were produced.

- Sheet A (according to the invention): The thickness  $t_{p1}$  and  $t_{p2}$  on each side of the sheet was controlled to be within the range (20-33) micrometers, at every location of the two faces of the sheet, both in the longitudinal (or rolling) direction and in the transversal direction. Measurement was performed continuously with thickness gages devices relying on X-Ray emission. At every moment, the spot of measurement of each gage was a circular zone of about 20mm radius. The sheets were afterwards cut into blanks of  $1.2 \times 0.5\text{m}^2$  of overall dimensions.
- Sheet B (reference): On these sheets, the pre-coating thickness had a wider variability since the thickness  $t_{p1}$  and  $t_{p2}$  on the two sides of the sheet was comprised in the range (30-45) micrometers. Blanks cut out of these sheets exhibit the same pre-coating thickness.

**[0078]** The blanks were then submitted to heating in a furnace at  $T=920^{\circ}\text{C}$ . Heating time was 3 min, with 4 mn holding time. The microstructure is then fully austenitic. Blanks were thereafter transferred from the furnace to a stamping press. When the elapsed time between the exit

of the blanks from the furnace and the transfer in the stamping press is more 10 seconds, a partial transformation from austenite was susceptible to appear, thus reducing the mechanical resistance of the stamped part. The blanks were directly cooled afterwards without hot stamping in order to appreciate the eventual remelting of the coating

On series A, no melting of the pre-coating was found. Intermetallic alloying between the pre-coating and the steel substrate occurred completely.

On series B, the pre-coating underwent mainly alloying, but some traces of remelting were found, particularly on the former thicker locations of the pre-coating. This partial remelting of the aluminium pre-coating contributes to the progressive fouling of the rollers in the furnace. The sheets according to the invention do not contribute to this progressive buildup on the rollers.

#### Example 2 :

##### [0079]

i) Conditions according to the invention: In a second example of implementation, a cold rolled steel sheet, 1,2mm thick, 120m long and 650mm wide, with same composition and same pre-coating as in example 1, has been fabricated. The sheets were afterwards cut into blanks which were heated at 920°C for 6mn, this time including the heating phase and the holding time. Heating rate  $V_c$  between 20 and 700°C was 10°C/s. The blanks were finally hot stamped and quenched in order to obtain full martensitic structures.

The parts obtained after hot-stamping are covered by a coating, 40 micrometers thick, illustrated at figure 1, which has a four layer structure. Starting from the steel substrate, the layers are the following:

- (a) Interdiffusion layer or intermetallic layer, 17 micrometers thick. This layer is itself composed of two sub-layers. Hardness HV50g ranges from 295 to 407, and the mean composition is: 90%Fe, 7%Al, 3%Si.
- (b) Intermediate layer, appearing darker, 8 micrometers thick. This layer has a hardness of 940HV50g and a mean composition, by weight : 43%Fe, 57%Al, 1 %Si.
- (c) Intermetallic layer appearing as a pale phase, 8 micrometers thick, displaying a hardness of 610HV50g, a mean composition of 65%Fe, 31%Al, 4%Si
- (d) Darker superficial layer, 7 micrometers thick, 950 HV50g, with a mean composition of 45%Fe, 54%Al, 1 %Si

Layers (c) and (d) are quasi-continuous, i.e. occupying at least 90% of the level corresponding to the considered layer. In particular, layer (c) does not

reach the extreme surface except very exceptionally. Anyway, this layer (c) occupies less than 10% of the extreme surface.

ii) Conditions of reference: On the other hand, blanks with the same base material and pre-coating parts were furnace-heated in different conditions: The blanks were heated to 950°C for 7 minutes, this time including the heating phase. Heating rate  $V_c$  was 11°C/s. These conditions correspond to a degree of alloying which is more important than in conditions (i)

- In this coating, the pale intermetallic layer (c), is not continuous and appears as to be scattered within the coating. About 50% of this layer is present at the extreme surface of the part. Moreover, the interdiffusion layer, 10 micrometers thick in contact with the steel substrate is thinner than in the previous case of figure 1.

Resistance spot welding was performed in the two situations i) and ii):

- (i): Coating with quasi-continuous layers (c) and (d), layer (c) occupying less than 10% of the extreme surface
- (ii) : Coating with mixed and discontinuous layers, layer (c) occupying more than 10% of the extreme surface

[0080] Resistance spot welding was performed by superposing two parts and joining them in the following conditions:

- Squeeze force and welding force : 4000 N
- Squeeze time: 50 periods
- Welding and holding time: 18 periods respectively

[0081] In each condition, the suitable intensity range was determined for obtaining:

- No sputter during welding
- Acceptable nugget size.

[0082] For the condition i), the weldability range, expressed in terms of current intensity, is 1,4kA. For the condition ii) the weldability range is extremely small.

[0083] Thus, it may be seen that the coating according to the invention, yields much more satisfactory results.

#### Example 3 :

[0084] In a third example of implementation, a cold rolled steel sheet of the example 1 was cut into blanks of 500x500mm<sup>2</sup> which were heated at 920°C, during 6mn, then hot stamped and cooled in tools, in such conditions that two different cooling rates were obtained :

- (A): Cooling rate:  $V_A = 30^\circ\text{C/s}$

- (B) : Cooling rate:  $V_B = 60^\circ\text{C/s}$

**[0085]** Due to the shape of the parts, different deformation levels  $\varepsilon$  were created during hot stamping. In particular, some zones largely strained display deformation levels higher than 30%.

- As illustrated on figure 3, metallographic observations reveal that when  $\varepsilon > 10\%$ , partial bainitic or ferritic transformation occurs on parts cooled with  $V_A = 30^\circ\text{C/s}$ , mainly on former austenitic grain boundaries. On the other hand, the parts cooled with  $V_B = 60^\circ\text{C/s}$  display fully martensitic microstructure as illustrated on figure 4. The latter structures display superior mechanic resistance and a great homogeneity in the case of mechanical sollicitation. Thus, even in products or parts where straining is larger than 10%, the application of cooling according to the invention guarantees a microstructural and mechanical homogeneity.

#### Example 4:

**[0086]** In a fourth example of implementation, steel castings containing different values of sulfur, were elaborated. These steels were further hot rolled, then cold rolled steel into sheets, 2,2mm thick. Sulfur content varies from 11ppm (0,0011%) to 59ppm (0,006%) in weight. Apart sulfur, the compositions of these different steel castings comprise in weight:

0,24% carbon, 1,17% manganese, 0,01% phosphorus, 0,25% silicon, 0,045% aluminum, 0,01% copper, 0,02% nickel, 0,2% chromium, 0,04% titanium, 0,003% boron, 0,002% calcium, the remainder being iron and unavoidable impurities.

**[0087]** These sheets were pre-coated with an aluminum-based alloy of composition comprising 9,3% silicon, 2.8% iron, the remainder being aluminum and unavoidable impurities.

**[0088]** The sheets were afterwards cut into blanks which were heated at  $950^\circ\text{C}$  for 5mn, then hot stamped and cooled in tools in order to obtain a full martensitic structure. Mechanical resistance exceeded 1450 MPa. Specimens were extracted according to the transverse sense of rolling direction and submitted to a bending test with alternate bending modes. The inventors put into evidence that the critical bending angle (angle at fracture) is closely related to the sulfur content of the steel: when sulfur content is lower than 0,002%, the bending angle exceeds  $60^\circ$ , which indicates higher ductility and energy absorption.

**[0089]** Compact Tensile-Test type specimens were also extracted according to the transverse rolling direction in order to measure the resistance to tearing, i.e. the energy which is necessary for the initiation or propagation of an existing crack. The results, illustrated on figure 6,

indicate that a initiation energy higher than 18 Joules is achieved when sulfur content is lower than 0,002% in weight.

**[0090]** As these qualities of high resistance, high energy absorption and weldability are required in automobile industry, the parts or products fabricated according to the invention will be used with profit for such applications.

**[0091]** While the above description is clear with regard to the understanding of the invention, the following terms as used in the following list of preferred embodiments and claims have the following noted meanings in order to avoid any confusion:

**pre-coating** - the material (Al or Al alloy) coated on or located on at least a portion of the strip or sheet, etc., of base steel to form a pre-coating/base composite, the composite not having been subjected to an alliation reaction between the coated Al or Al alloy material and base steel

**alliation** - a reaction between the pre-coating and base steel, to produce at least one intermediate layer different in composition from both the base steel and the pre-coating. The alliation reaction happens during the heat treatment immediately preceding hot stamping. The alliation reaction affects the total thickness of the pre-coating. In a highly preferred embodiment the alliation reaction forms the following layers: (a) interdiffusion, (b) intermediate, (c) intermetallic, and (d) superficial as described above;

**pre-coated steel** - the pre-coating/base composite, not having been subjected to an alliation reaction between the coated material and base steel;

**coating** - the pre-coating after having been subjected to an alliation reaction between the pre-coating and base steel. In a highly preferred embodiment the coating comprises layers (a) interdiffusion, (b) intermediate, (c) intermetallic, and (d) superficial described above;

**coated steel or product** - the pre-coated steel or product that has been subjected to an alliation reaction between the pre-coating and base steel. In a highly preferred embodiment the coated steel is a strip or sheet, etc., of base steel having thereon an invention coating comprising layers (a) interdiffusion, (b) intermediate, (c) intermetallic, and (d) superficial described above;

**blank** - a shape cut from a strip.

**product** - a stamped blank

**[0092]** Thus, the present invention provides, among other things, the following preferred embodiments:

1. A pre-coated steel strip, comprising:

- (a) a strip of base steel having a length, a width, a first side, and a second side;
- (b) said length of said strip of base steel being

at least 100m and said width being at least 600mm, and

(c) an aluminum or an aluminum alloy pre-coating on at least part of one of said first and second sides of said strip of base steel, wherein

(i) the thickness  $t_p$  of the said pre-coating is from 20 - 33 micrometers at every location on at least one of said first and second sides.

2. A method for making a pre-coated steel strip according to embodiment 1 wherein the pre-coated steel strip has a thickness of not greater than 3 mm.

3. A process for making a hot stamped coated steel sheet product, comprising:

(A) heating an aluminum- or aluminum alloy pre-coated steel sheet in a furnace preheated to a temperature and during a time defined by diagram ABCD of figure 7 if thickness of said sheet is greater than or equal to 0.7mm and less than or equal to 1.5mm, and by diagram EFGH of figure 7 if thickness of said sheet is greater than 1.5mm and less than or equal to 3mm, at a heating rate  $V_c$  between 20 and 700°C comprised between 4 and 12°C/s, to obtain a heated blank; (B) transferring said heated blank to a die; and (C) stamping said heated blank in said die, to thereby obtain a hot stamped steel sheet product, and

wherein the said heated product is cooled at a rate of at least 30°C/s.

4. A process for making a hot stamped coated steel sheet product, comprising:

(A) providing a heated blank according to embodiment 3; and (B) transferring said heated blank to a die; and (C) stamping said heated blank in said die, to thereby obtain a hot stamped steel sheet product,

wherein the elapsed time between said heated blank exits said furnace and said stamping commences is not more than 10 seconds, and wherein said heated blank is deformed by a quantity higher than 10% during said stamping and the said heated product is cooled at a rate of at least 50°C/s.

5. A process for making a hot stamped coated steel sheet product, comprising :

hot dip pre-coating a steel strip having a first side and a second side with aluminum or aluminum alloy to obtain a pre-coated steel strip, the

pre-coating thickness on at least one of said first side and said second side of said strip having a thickness of from 20 to 33  $\mu\text{m}$  at every location on at least one of said first and second sides of said strip;

cutting said pre-coated steel strip to obtain a sheet;

heating the sheet in a pre-heated furnace to obtain a heated blank in which the steel is austenitic;

transferring said heated blank to a die while maintaining said steel in the austenitic phase; and

stamping said heated blank in a die to obtain a hot stamped steel sheet product in which the steel microstructure is martensitic.

6. A hot stamped coated steel sheet product, which is prepared by a process comprising :

(A) providing a pre-coated strip according to embodiment 1, said strip being pre-coated with aluminum or aluminum alloy

(B) Cutting said pre-coated steel strip to a predetermined size and shape to obtain a sheet

(C) heating said steel sheet in a furnace preheated to a temperature and during a time defined by diagram ABCD of figure 7 if thickness of said product is greater than or equal to 0.7mm and less than or equal to 1.5mm, and by diagram EFGH of figure 7 if thickness of said product is greater than 1.5mm and less than or equal to 3mm, to obtain a heated blank;

(D) transferring said heated blank to a die; and

(E) stamping said heated blank in said die, to thereby obtain a hot stamped steel sheet product,

wherein the elapsed time between said heated blank exits said furnace and said stamping commences is not more than 10 seconds, and wherein said heated product cools at a rate higher than 30°C/s during said stamping.

7. A process for making a hot stamped coated steel sheet product, comprising:

(A) providing a heated blank according to embodiment 6

(B) transferring said heated blank to a die; and

(C) stamping said heated blank in said die, to thereby obtain a hot stamped steel sheet product,

wherein the elapsed time between said heated blank exits said furnace and said stamping commences is not more than 10 seconds, and wherein said heated product cools at such a rate during said stamping

that the steel microstructure is martensitic.

8. The pre-coated steel strip according to embodiment 1, wherein the base steel comprises the following components by weight based on total weight:

0.15%<carbon<0.5%  
0.5%<manganese<3%  
0.1%<silicon<0.5%  
0.01 %<chromium<1 %  
titanium<0.2%  
aluminum<0.1%  
phosphorus<0.1 %  
sulfur<0.05%  
0.0005%<boron<0.08%,

and further comprises iron and impurities inherent in processing.

9. The pre-coated steel strip according to embodiment 8, wherein the base steel comprises the following components by weight based on total weight:

0.20%<carbon<0.5%  
0.8%<manganese<1.5%  
0.1%<silicon<0.35%  
0.01 %<chromium<1 %  
titanium<0.1 %  
aluminum<0.1%  
phosphorus<0.05%  
sulfur<0.03%  
0.0005%<boron<0.01%,

and further comprises iron and impurities inherent in processing.

10. The pre-coated steel strip according to embodiment 8 or 9, wherein the base steel comprises 20 ppm or less of sulfur.

11. The pre-coated steel strip according to any of embodiments 8 to 10, wherein the ratio of titanium to nitrogen in the steel sheet in weight % is in excess of 3.42.

12. The pre-coated steel strip according to any of the embodiments 1 or 8 to 11, wherein the aluminum or aluminum alloy pre-coating comprises from 8% to 11% silicon by weight, from 2% to 4% iron by weight, the remainder being aluminum and impurities inherent in processing.

13. A coated steel stamped product, which comprises:

- (a) a strip of base steel having a first side and a second side; and
- (b) a coating on at least one of said first side of

said strip of base steel and said second side of said strip of base steel,

wherein;

- (i) said coating results from the interdiffusion between said base steel, and aluminium or aluminium alloy pre-coating,
- (ii) said coating comprises, proceeding from base steel outwards,

- (a) Interdiffusion layer
- (b) Intermediate layer
- (c) Intermetallic layer
- (d) Superficial layer

14. A coated steel stamped product according to embodiment 13, wherein said coating has a thickness greater than 30 micrometers

15. A coated steel stamped product according to embodiments 13 or 14, wherein said layer (a) has a thickness less than 15 micrometers

16. A coated steel stamped product according to any of the embodiments 13 to 15, wherein the said layers (c) and (d) are occupying at least 90% of the level corresponding to the considered said layers and wherein less than 10% of layer (c) is present at the extreme surface of the product

17. A coated steel stamped product according to any of the embodiments 13 to 16, wherein the steel in the strip has a composition according to embodiment 8

18. A coated steel stamped product according to any of the embodiments 13 to 16, wherein the steel in the strip has a composition according to embodiment 9

19. A coated steel stamped product according to any of the embodiments 13 to 18, wherein the steel has a composition according to embodiment 10

20. A coated steel stamped product according to any of the embodiments 13 to 19, wherein the steel has a composition according to embodiment 11

21. A coated steel stamped product according to any of the embodiments 13 to 20, the aluminum or aluminum alloy pre-coating comprises from 8% to 11% silicon by weight, from 2% to 4% iron by weight, the remainder being aluminum and impurities inherent in processing.

22. A land motor vehicle comprising the heat treated coated steel product according to any of the embod-

iments 6, 13 to 21

23. A land motor vehicle comprising the heat treated coated steel product produced according to any of the embodiments 3, 4, 5, 7

[0093] The above written description of the invention provides a manner and process of making and using it such that any person skilled in this art is enabled to make and use the same, this enablement being provided in particular for the subject matter of the appended claims, which make up a part of the original description. As used above, the phrases "selected from the group consisting of," "chosen from," and the like include mixtures of the specified materials. Terms such as "contain(s)" and the like as used herein are open terms meaning 'including at least' unless otherwise specifically noted. All references, patents, applications, tests, standards, documents, publications, brochures, texts, articles, etc. mentioned herein are incorporated herein by reference. Where a numerical limit or range is stated, the endpoints are included. Also, all values and subranges within a numerical limit or range are specifically included as if explicitly written out. The above description is presented to enable a person skilled in the art to make and use the invention, and is provided in the context of a particular application and its requirements. Various modifications to the preferred embodiments will be readily apparent to those skilled in the art, and the generic principles defined herein may be applied to other embodiments and applications without departing from the spirit and scope of the invention. Thus, this invention is not intended to be limited to the embodiments shown, but is to be accorded the widest scope consistent with the principles and features disclosed herein.

## Claims

1. A process for making a hot stamped coated steel sheet product, comprising:

(C) heating an aluminum- or aluminum alloy pre-coated steel sheet in a furnace preheated to a temperature and during a time defined by diagram ABCD of figure 7 if thickness of said sheet is greater than or equal to 0.7mm and less than or equal to 1.5mm, and by diagram EFGH of figure 7 if thickness of said sheet is greater than 1.5mm and less than or equal to 3mm, at a heating rate  $V_c$  between 20 and 700°C comprised between 4 and 12°C/s, to obtain a heated blank, the heating rate  $V_c$  being different from the mean heating rate between room temperature and the furnace temperature;

(D) transferring said heated blank to a die; and

(E) hot stamping said heated blank in said die, to thereby obtain a hot stamped steel sheet

product, and

wherein said heated blank is cooled at a mean rate between the exit of the heated blank from the furnace, down to 400°C, of at least 30°C/s.

2. The process according to claim 1, wherein the elapsed time between said heated blank exits said furnace and said stamping commences is not more than 10 seconds, and wherein said heated blank is deformed by a quantity higher than 10% during said stamping and the heated blank is cooled at a mean rate between the exit of the heated blank from the furnace, down to 400°C of at least 50°C/s.
3. The process according to any one of claims 1 or 2, further comprising:

(A) providing a pre-coated strip obtained by hot dip pre-coating a steel strip having a first side and a second side with aluminium or aluminium alloy, the pre-coating thickness on at least one of said first side and said second side of said strip having a thickness of from 20 to 33  $\mu\text{m}$  at every location on at least one of said first and second sides of said strip;

(B) cutting said pre-coated steel strip to obtain the aluminum- or aluminum alloy pre-coated steel sheet.

4. The process according to any one of claims 1 to 3, wherein the aluminum or aluminum alloy pre-coating comprises from 8% to 11% silicon by weight, from 2% to 4% iron by weight, the remainder being aluminum and impurities inherent in processing.

5. A coated steel stamped product, which comprises:

(a) a strip of base steel having a first side and a second side; and

(b) a coating on at least one of said first side of said strip of base steel and said second side of said strip of base steel,

wherein:

(i) said coating results from the interdiffusion between said base steel, and aluminium or aluminium alloy pre-coating,

(ii) said coating comprises, proceeding from base steel outwards,

- (a) Interdiffusion layer
- (b) Intermediate layer
- (c) Intermetallic layer
- (d) Superficial layer

And wherein said layers (c) and (d) are occupying at least 90% of the level corresponding to the considered said layers and wherein less than 10% of layer (c) is present at the extreme surface of the product.

6. A coated steel stamped product according to claim 5, wherein said coating has a thickness greater than 30 micrometers

7. A coated steel stamped product according to any one of claims 5 or 6, wherein said layer (a) has a thickness less than 15 micrometers

8. A coated steel stamped product according to any of the claims 5 to 7, wherein the steel in the strip has a composition comprising, by weight:

0.15%<carbon<0.5%  
0.5%<manganese<3%  
0.1%<silicon<0.5%  
0.01 %<chromium<1 %  
titanium<0.2%  
aluminum<0.1%  
phosphorus<0.1 %  
sulfur<0.05%  
0.0005%<boron<0.08%,

and further comprises iron and impurities inherent in processing.

9. A coated steel stamped product according to any of the claims 5 to 7, wherein the steel in the strip has a composition comprising, by weight:

0.20%<carbon<0.5%  
0.8%<manganese<1.5%  
0.1%<silicon<0.35%  
0.01 %<chromium<1 %  
titanium<0.1 %  
aluminum<0.1%  
phosphorus<0.05%  
sulfur<0.03%  
0.0005%<boron<0.01%,

and further comprises iron and impurities inherent in processing.

10. A coated steel stamped product according to any of the claims 5 to 9, wherein the steel comprises 20 ppm or less of sulfur.

11. A coated steel stamped product according to any of the claims 5 to 10, wherein the ratio of titanium to nitrogen in the steel in weight% is in excess of 3.42.

12. A coated steel stamped product according to any of the claims 5 to 11, wherein the aluminum or alumi-

num alloy pre-coating comprises from 8% to 11% silicon by weight, from 2% to 4% iron by weight, the remainder being aluminum and impurities inherent in processing.

13. A coated steel stamped product according to any of the claims 5 to 12, wherein the interdiffusion layer has the following composition, by weight: 86-95% Fe, 4-10% Al, 0-5% Si.

14. A coated steel stamped product according to any of the claims 5 to 13, wherein the intermediate layer has the following composition, by weight: 39-47% Fe, 53-61% Al, 0-2% Si.

15. A coated steel stamped product according to any of the claims 5 to 14, wherein the intermetallic layer has the following composition, by weight: 62-67% Fe, 30-34% Al, 2-6% Si.

16. A coated steel stamped product according to any of the claims 5 to 15, wherein the superficial layer has the following composition, by weight: 39-47% Fe, 53-61% Al, 0-2% Si.

17. A land motor vehicle comprising the coated steel stamped product according to any of the claims 5 to 16.

18. A land motor vehicle comprising the hot stamped coated steel sheet product produced according to any of the claims 1 to 4.

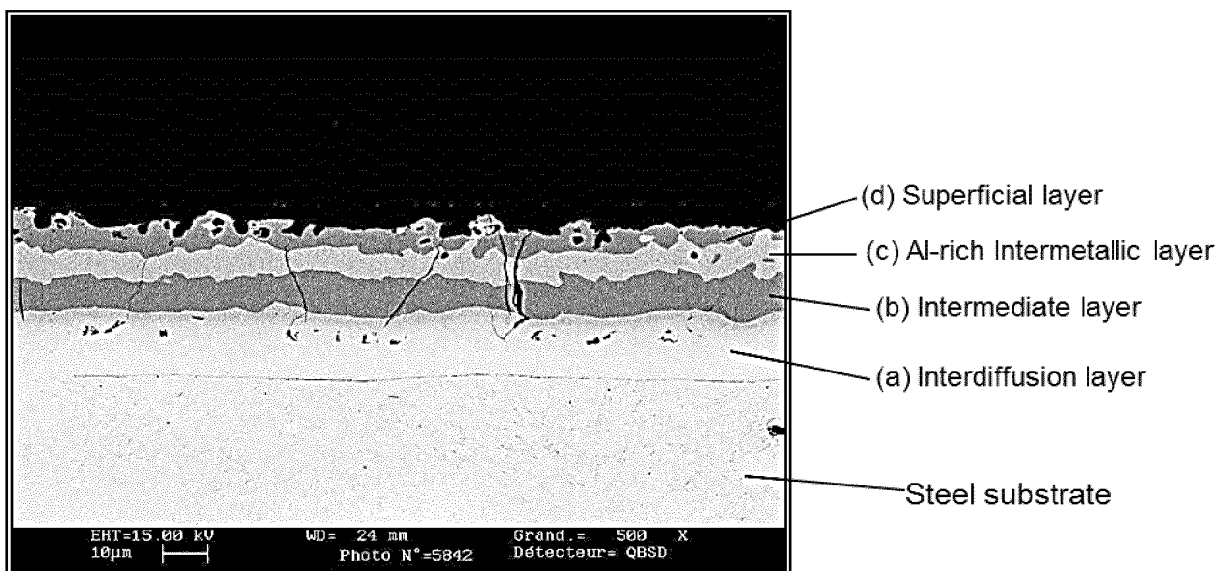


**FIG.1**

10µm

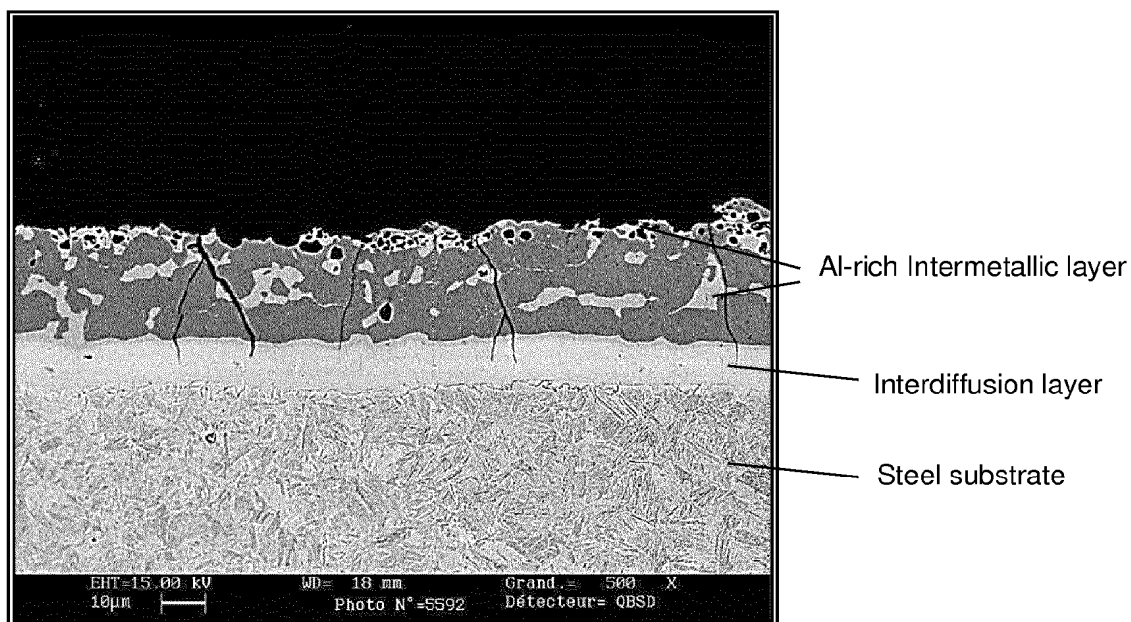


**FIG.2**

10µm

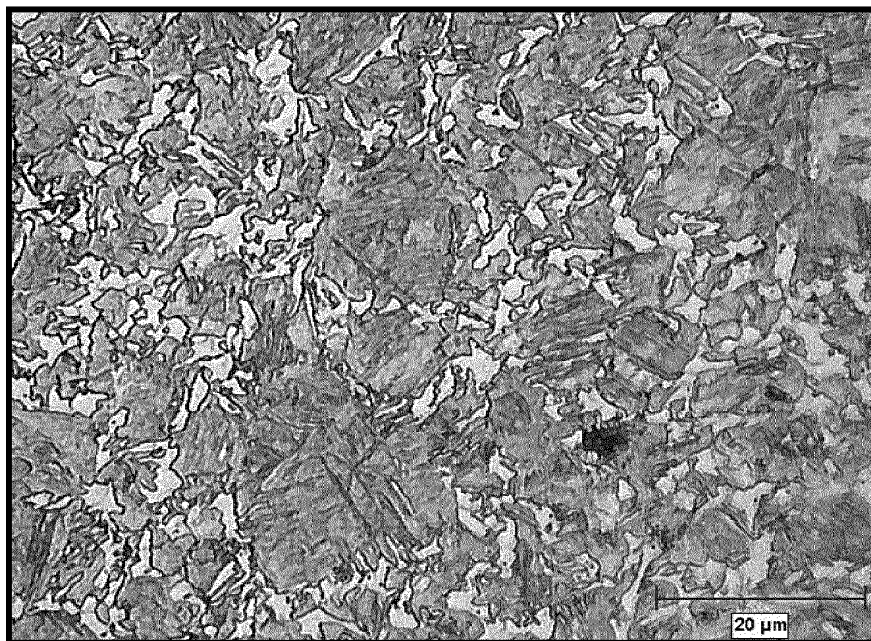


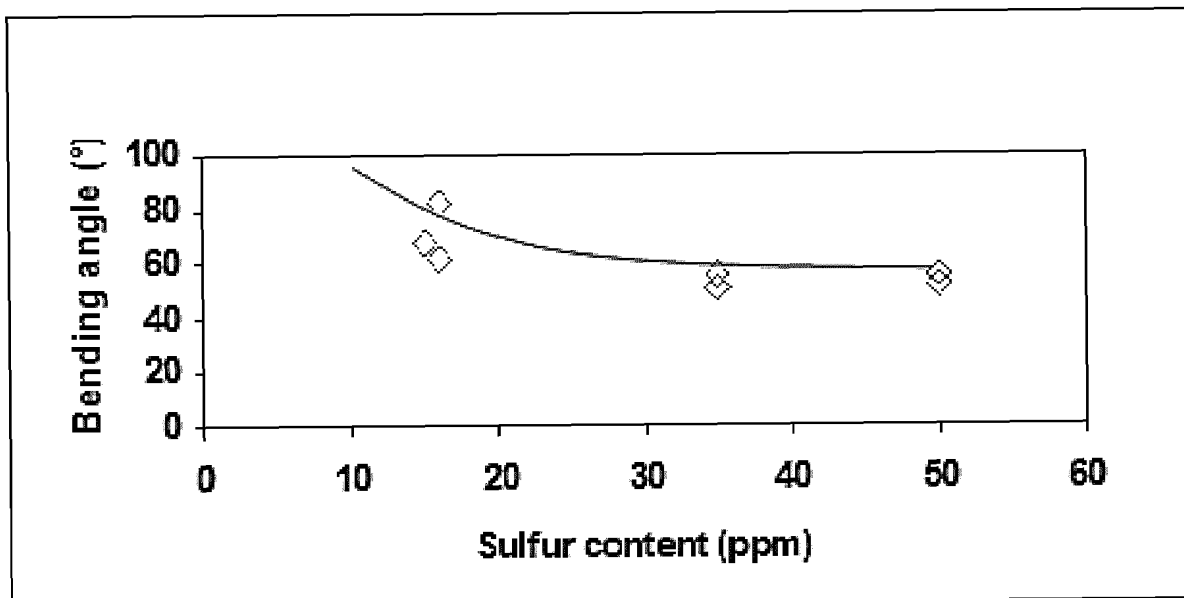
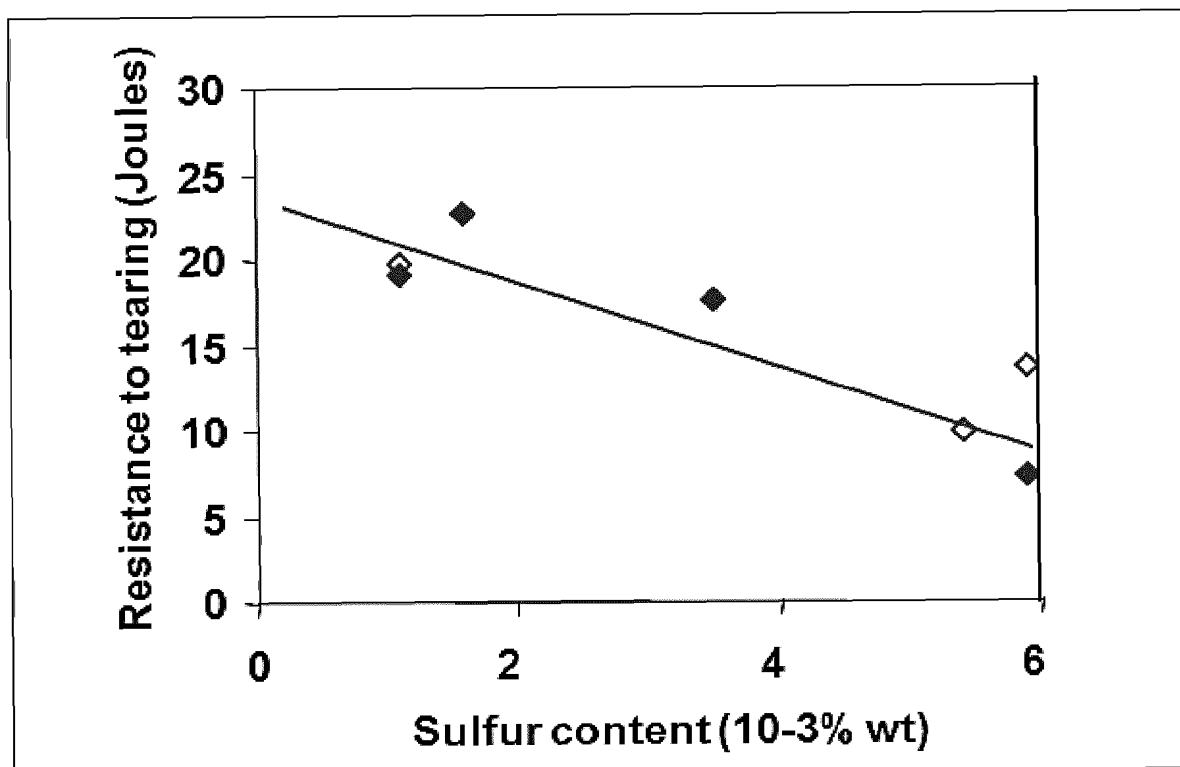
**FIG.3**

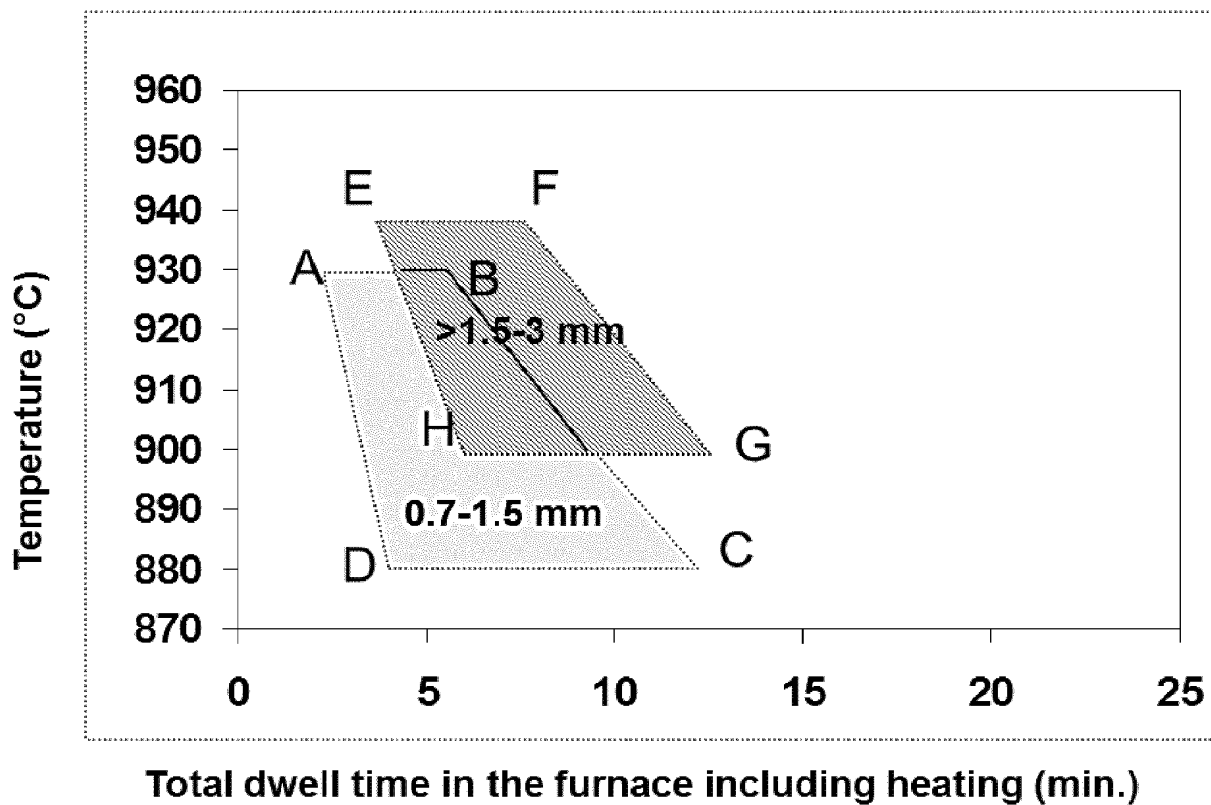
10μm



**FIG.4**

10 μm

**FIG.5****FIG.6**

**FIG. 7**



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Espacenet

## Bibliographic data: FR2758571 (A1) — 1998-07-24

Steel sheet coated with double layer of aluminium@ alloys

**Inventor(s):** GUESDON PHILIPPE; HENNECHART JEAN PAUL; SPEHNER DOMINIQUE ± (GUESDON PHILIPPE, ; HENNECHART JEAN PAUL, ; SPEHNER DOMINIQUE)

**Applicant(s):** LORRAINE LAMINAGE [FR] ± (SOLLAC)

**Classification:** - international: **C23C2/12**; (IPC1-7): C21D9/46; C23C2/12  
- cooperative: **C23C2/12**

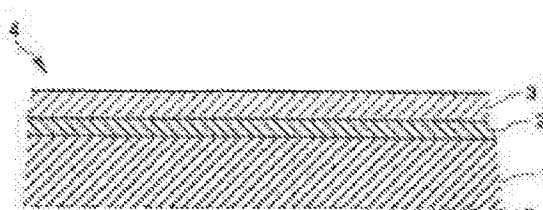
**Application number:** FR19970000565 19970121

**Priority number(s):** FR19970000565 19970121

**Also published as:** FR2758571 (B1)

### Abstract of FR2758571 (A1)

A steel sheet (1) is coated on both sides with a layer (3) containing by weight 95-98% of aluminium and less than 4% iron, separated from sheet (1) by an intermediate layer (2) comprising 40-65% Al, 30-50% Fe and 3-12% of one or more elements whose atomic diameter is within 15% of that of Fe. Also claimed is dipping a steel sheet in a melt of 88-95% Al and 7-12% of the one or more elements in order to form layer (2) thereon, draining excess alloy from the sheet to leave a layer (2) 3-7  $\mu\text{m}$  thick and then dipping the sheet in molten Al.



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## DESCRIPTION FR2758571

### STEEL SHEET HAVING A ALUMINUM COATING.

The present invention relates to the field of coated sheets.

It relates specifically to sheets provided with an aluminum-based coating, called aluminized sheets.

These aluminized sheets are particularly used in the automotive field for producing exhaust lines, heat shields intended to isolate the body from exhaust lines, fuel tanks, or suspension arms, for example.

They are also used in the building industry in the production of, for example, fire doors, flues, chimney inserts, ducts or covering elements.

Aluminum sheets are also used in the field of household appliances for the production of toasters, minifours, deep fryers, boiler combustion chambers and burners.

It is known to use aluminized sheets made of a steel substrate coated with an alloy based on aluminum and silicon.

Such an aluminized sheet is produced by dipping a steel strip in a molten bath containing about 90% aluminum and 10% silicon, in order to obtain a coating layer, for example of 20 microns, thickness.

After diffusion of the iron atoms of the substrate into the coating layer, it has a first highly alloyed layer in contact with the substrate containing by weight approximately 45% iron, 53% aluminum, 2% silicon and then Intermediate layer containing about 32% iron, 58% aluminum, 10% silicon, and finally an outer layer containing about 90% aluminum, 9% silicon and very little iron (less than 3%).

At the traditional strength of the substrate steel, this type of aluminum-silicon coating provides good resistance to hot oxidation, good resistance to high temperature and good resistance to corrosion, which confers this type of Aluminized sheet metal, a particular ability to produce automobile exhaust lines, heat shields and, more generally, parts subjected to high temperatures.

The high alloy layer containing about 45% of iron being very small, on the order of a micrometer, and the intermediate layer still containing an average of 32% of iron being also weak, on the order of 4 micrometers, An alloy layer of small thickness, of the order of 5 micrometers, which gives this type of coating a good adhesion and therefore permits deep drawing.

It is also known to use aluminized sheets made of a steel substrate coated with an aluminum alloy.

Such an aluminized sheet is produced by dipping a strip of steel in an aluminum melt in order to obtain a coating layer on the order of 35 to 60 microns thick.

After diffusion of the iron atoms of the substrate into the coating layer, the latter has a first highly alloyed layer in contact with the substrate containing by weight approximately 42% iron, 58% aluminum and then an outer layer containing about 98 % Of aluminum and almost no iron (less than 3% present as precipitates  $\text{FeAl}_3$ ,  $\text{Fe}_2\text{Al}_5$  and  $\text{Fe}_2\text{Al}_{17}$ ).

This type of aluminized sheet has excellent resistance to industrial, urban and marine corrosion, excellent resistance to aggressive corrosion of hydrocarbon combustion products and corrosion in the presence of heat.

Moreover, this type of aluminized sheet offers a very good radiation reflectance (greater than 85%), close to that of a pure aluminum sheet.

It also has good resistance to heat and fire. Up to a temperature of 450 ° C., the coating is not altered, and between 450 and 600 ° C., a visual transformation of the coating layer is observed but without any appreciable alteration of its protective power.

This type of coating is perfectly suited for the construction of building roofs and for heat insulation parts.

However, the adhesion of the coating is modest because of the importance of the highly alloyed layer, generally greater than 12 micrometres thick for this type of aluminized sheet.

It is for this reason that the coating layer is of large thickness, usually between 35 and 60 micrometers, in order to guarantee the presence of a sufficient non-alloy layer.

Due to this adhesion problem, it is impossible to carry out deep drawings on this type of aluminized sheet without altering the coating, and in the case of folding of such a sheet, certain precautions must be observed, particularly in terms of radii Of curvature in order to avoid degradation of the coating. Indeed, in the case of stamping or bending under severe conditions, cracks are created in the highly alloyed layer, resulting in cracking, overlap and decohesion of the coating.

Thus, the known technique proposes either an aluminized sheet having a very good resistance to corrosion and very good reflective properties close to those of pure but not very formable aluminum sheets or an aluminized sheet allowing deep drawing but having a Less corrosion resistance and less good reflective properties.

The object of the present invention is to propose an aluminized sheet having both very good resistance to corrosion, a very good resistance to hot oxidation, very good reflectivity properties close to those of pure aluminum sheets, allowing Deep drawings.

The subject of the invention is a coated steel sheet made of a steel substrate provided on both sides with an aluminum-based coating, characterized in that the coating is a coating consisting of two superimposed layers: An internal layer in contact with the substrate comprising by weight between 40 and 65% of aluminum, between 30 and 50% of iron and between 3 and 12% of one or more elements selected from those whose relative difference in atomic diameter with An iron atom is less than or equal to 15%; An outer layer comprising by weight between 95 and 98% of aluminum and less than 4% of iron.

According to other characteristics of the invention, the internal layer comprises by weight between 40 and 65% of aluminum, between 30 and 50% of iron and between 3 and 12% of one or more elements chosen from silicon, Titanium, beryllium, manganese, the thickness of the inner layer is between 2 and 7 micrometers, the outer layer comprises by weight between 95 and 98% of aluminum, less than 4% of iron and less than 2 % Of one or more elements selected from those whose relative difference in atomic diameter with an iron atom is less than or equal to 15%; The thickness of the outer layer is between 5 and 50 micrometers.

The invention also relates to a method for producing such a coated steel sheet, characterized in that it comprises at least three steps: a first step in which, in order to form an alloyed inner layer containing by weight Between 40 and 65% of aluminum, between 30 and 50% of iron and between 3 and 12% of one or more elements selected from those whose relative difference in atomic diameter with an iron atom is less than or equal to 15% , A steel sheet is quenched in a melt containing between 88 and 95% aluminum and between 7 and 12% of one or more elements selected from those whose relative difference in atomic diameter with an iron atom is Less than or equal to 15%; A second step in which the sheet is wiped to eliminate the surface portion of unalloyed coating and to retain only an alloyed inner layer with a thickness of between 2 and 7 micrometers; A third stage in which the sheet is quenched in a bath of molten aluminum.

The invention also relates to another method of manufacturing such a coated steel sheet, characterized in that: an annealing of a steel strip under a non-oxidizing atmosphere containing atoms in gaseous form, d One or more elements whose relative difference in atomic diameter with an iron atom is less than or equal to 15%, so as to form an inner layer; And then the strip is

soaked in a bath of molten aluminum.

According to other characteristics: the non-oxidizing atmosphere in which the strip is annealed contains one or more elements chosen from silicon, titanium, beryllium, manganese, in gaseous form; The non-oxidizing atmosphere in which the strip is annealed contains silane.

**BRIEF DESCRIPTION OF THE DRAWINGS** The characteristics and advantages will appear more clearly from the following description given solely by way of example, with reference to the accompanying drawings, in which: FIG. 1 is a schematic view of an aluminized sheet according to the invention FIG. 2 is a graph showing the diameter factors of the main elements, FIG. 3 is a schematic view of an installation making it possible to implement a first process for manufacturing an aluminized sheet according to the invention; FIG. 4 is a schematic view of an installation for implementing a second process for manufacturing an aluminized sheet according to the invention, FIG. 5 is a diagrammatic view of another installation enabling the method to be implemented. FIG. Second method of manufacturing an aluminized sheet according to the invention.

As can be seen in FIG. 1, the aluminized steel sheet according to the invention consists of a substrate 1 made of steel coated with two layers of a distinct coating.

The first internal layer 2 in contact with the substrate 1 comprises, by weight, between 40 and 65% of aluminum, between 30 and 50% of iron and between 3 and 12% of one or more elements selected from those whose difference Relative atomic diameter with an iron atom is less than or equal to 15%.

These elements are identified in FIG. 2 between the two dashed lines which indicate the limits between which the relative difference in atomic diameter with an iron atom is less than 15% according to the Hume-Rothery classification.

These elements are beryllium, silicon, phosphorus, vanadium, chromium, manganese, cobalt, nickel, copper, zinc, germanium, arsenic, molybdenum, ruthenium, rhodium, Palladium, tin, tungsten, rhenium, iridium, platinum, titanium, niobium, silver, antimony, and gold.

The second outer layer 3 comprises by weight between 95 and 98% of aluminum and less than 4% of iron.

This second outer layer 3 may also comprise less than 2% of one or more elements selected from those whose relative difference in atomic diameter with an iron atom is less than or equal to 15%, for example beryllium, silicon, Phosphorus, vanadium, chromium, manganese, cobalt, nickel, copper, zinc, germanium, arsenic, molybdenum, ruthenium, rhodium, palladium, tin, tungsten, Rhenium, iridium, platinum, titanium, niobium, silver, antimony, or gold.

The first inner layer 2 has a low thickness between 2 and 7 micrometers in order to preserve the adhesive qualities of the coating and the second layer 3 may be between 5 and 50 micrometers according to the total thickness of coating desired. Preferably, the thickness of the layer 2 is less than or equal to 5 micrometers.

The element or elements whose atomic diameter is such that the relative difference in atomic diameter of each of these elements with an iron atom is less than 15%, contained in the layer 2 in contact with the substrate, make it possible to limit the phenomenon of The alloy between this coating layer and the substrate.

Indeed, elements whose atomic diameter is greater than that of iron have difficulty dislodging and thus block the migration of iron atoms.

On the other hand, elements whose atomic diameter is less than that of iron can more easily dislodge, but the space left free is insufficient for an iron atom or an aluminum atom.

In practice, the non-toxic elements and the lower cost elements will preferably be chosen. Thus, the use of arsenic, silver, gold or platinum, although limiting the phenomenon of alloying, will be very rarely used.

Moreover, depending on the type of steel of the substrate, elements which tend to combine with components of the steel, such as, for example, niobium, which tends to trap the carbon contained in the steel, will be avoided.

Preferably, one or more elements chosen from silicon, titanium, beryllium and manganese will be used.

A first process for producing an aluminized sheet according to the invention consists in annealing a steel strip in a non-oxidizing atmosphere containing atoms in gaseous form of one or more elements whose relative difference in atomic diameter with an iron atom is less than or equal to 15%, and then soaking the strip in a bath of molten aluminum.

As can be seen in FIG. 3, a strip 4 of steel continuously passes through an annealing chamber 5 in which care has been taken to maintain a non-oxidizing atmosphere.

During annealing, one or more elements whose atmospheric difference in atomic diameter with an iron atom is less than or equal to 15% are introduced into the atmosphere of the annealing enclosure.

It is very important that the gaseous form under which these elements are introduced into the annealing enclosure is a non-oxidizing form. For example, in the case of silicon, the silicon atoms are introduced in the form of silane  $\text{SiH}_4$ .

At the outlet of the annealing chamber, the strip is held in a non-oxidizing atmosphere in a chute 6 until it is soaked in a bath 7 of molten aluminum.

In the annealing chamber 5 and in the chute 6, the non-oxidizing atmosphere is composed, for example, of a mixture of nitrogen and hydrogen, and the temperature of the strip is brought to a value close to the temperature of Bath 7, preferably between  $660^\circ\text{C}$ . and  $730^\circ\text{C}$ .

The nitrogen used to constitute the non-oxidizing atmosphere preferably contains less than 5 ppm of oxygen and has a dew point of less than  $-60^\circ\text{C}$ .

When the strip 4 is heated in the annealing chamber, its main faces are charged with atoms of the element or elements introduced into said chamber.

At the outlet of the bath of molten aluminum, the two faces coated with the strip 4 are drained by a gas blown through the wiper nozzles 8 and the strip is discharged and discharged into sheets.

This process makes it possible to produce the bi-layer coating according to the invention by dipping the strip in a single molten bath, constituted solely of aluminum. The first inner layer 2 is produced by the introduction of elements introduced into the annealing chamber.

A second method for manufacturing an aluminized sheet according to the invention consists in depositing the two inner layers 2 and outer layer 3 separately.

The strip is first soaked in a molten bath containing an aluminum-based alloy containing between 88 and 95% aluminum and between 7 and 12% of one or more elements selected from those whose relative diameter difference Atomic ratio with an iron atom is less than or equal to 15%, preferably silicon, titanium, beryllium, manganese, in order to form an alloyed inner layer containing by weight between 40 and 65% aluminum between 30 and 50% of iron and between 3 and 12% of one or more elements selected from those whose relative difference in atomic diameter with an iron atom is less than or equal to 15%.

Thereafter, the strip is drained to remove the surface portion of the unalloyed coating and to retain only a layer containing between 40 and 65% aluminum, between 30 and 50% iron, and between 3 and 12% of the element Of addition, with a thickness of between 3 and 7 micrometers.

Finally, the strip is dipped in a bath of molten aluminum.

Before soaking the strip in the first bath, the strip advantageously undergoes an annealing under a non-oxidizing atmosphere in order to bring its temperature to a value close to the temperature of the bath.

This method has the advantage of perfectly controlling the thickness of the first coating layer 2.

The installation illustrated in FIG. 4 makes it possible to implement this method.

The steel strip 4 penetrates into an annealing chamber 5 in which the atmosphere is non-oxidizing and is deflected by a roller 9 to pass through a chute 6 and to dip into a coating metal bath 10, for

example a bath containing Between 88 and 95% aluminum and between 7 and 12% silicon.

Then, after being deflected by a roller 11 disposed in the bath 10, the strip 4 emerges from the bath 10 and is dewatered by blown gas through the nozzles 8.

This dewatering operation makes it possible to eliminate the part of the coating layer deposited on the non-alloy strip. The strip then comprises a first coating layer 2, with a thickness of between 3 and 7 micrometers, preferably equal to 5 micrometers.

The strip 4 then enters immediately into a bath of molten aluminum.

It is very important to cause the strip to penetrate into the bath 12 immediately after its dewatering in order to avoid any oxidation.

In the illustrated embodiment, the strip 4 passes vertically through the bath 10 and is then evacuated after having undergone a further dewatering operation, for example by blown gas through the nozzles 8.

The installation illustrated in FIG. 5 also makes it possible to implement this method.

In this installation, the strip 4 emerges from the bath 10, is wringed by blown gas through the nozzles 8.

Then, after being deflected by means of a roller 13, it is immersed in a bath 14 of molten aluminum, in which it is again deflected by means of a roller 15 disposed in the bottom of the bath 14 and then Strip spring of the bath 14 and undergoes a new operation of scavenging by gas blown through the nozzles 8.

Optionally, if the path of the strip between the wiper nozzles 8 and the second bath 14 is long, the strip is protected by arranging a chute 16 between said wiping nozzles and the second bath, in which a non- oxidizing.

The aluminized steel sheet according to the invention offers a very good reflectance to radiation (greater than 85%), close to that of a pure aluminum sheet, good resistance to heat and fire.

It allows deep drawing because the alloy layer 2 is of very small thickness and makes a barrier to the diffusion of the aluminum atoms of the external layer 3 towards the substrate 1 and the diffusion of the iron atoms of the substrate 1 towards the layer External 3.

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## CLAIMS FR2758571

### CLAIMS

1

- Coated steel sheet consisting of a steel substrate provided on both sides with an aluminum-based coating, characterized in that the coating consists of two superimposed layers: an inner layer (2) in contact With the substrate comprising by weight between 40 and 65% aluminum, between 30 and 50% iron and between 3 and 12% of one or more elements selected from those whose relative difference in atomic diameter with an iron atom is Less than or equal to 15%; An outer layer (3) comprising by weight between 95 and 98% of aluminum and less than 4% of iron.

2

Coated steel sheet according to claim 1, characterized in that the inner layer (2) comprises by weight between 40 and 65% of aluminum, between 30 and 50% of iron and between 3 and 12% of Or several elements selected from silicon, titanium, beryllium, manganese.

3

Coated steel sheet according to claim 1 or 2, characterized in that the thickness of the inner layer (2) is between 2 and 7 micrometers.

4

Coated steel sheet according to claim 1, characterized in that the outer layer (3) comprises, by weight, between 95 and 98% of aluminum, less than 4% of iron and less than 2% of one or more elements Chosen from those whose relative difference in atomic diameter with an iron atom is less than or equal to 15%;

5

Coated steel sheet according to claim 1 or 4, characterized in that the thickness of the outer layer (3) is between 5 and 50 micrometers.

6

A method of manufacturing a steel sheet coated on its two main faces with an aluminum-based coating according to claim 1, characterized in that it comprises at least three steps: a first step in which, In order to form an alloyed inner layer containing by weight between 40 and 65% of aluminum, between 30 and 50% of iron and between 3 and 12% of one or more elements selected from those whose relative difference in atomic diameter With an iron atom being less than or equal to 15%, a steel sheet is quenched in a molten bath containing between 88 and 95% aluminum and between 7 and 12% of one or more elements selected from those of which The relative difference in atomic diameter with an iron atom is less than or equal to 15%; A second stage in which the sheet is drained to remove the surface portion of unalloyed coating and to retain only an alloyed inner layer with a thickness of between 3 and 7 micrometers; A third stage in which the sheet is quenched in a bath of molten aluminum.

7

Process according to claim 6, characterized in that prior to the first step, the steel strip is annealed in a non-oxidizing atmosphere.

8

Process for the production of a steel sheet coated on its two main faces with an aluminum-based coating according to claim 1, characterized in that: an annealing of a strip of steel under an atmosphere Non-oxidizing composition containing atoms, in gaseous form, of one or more elements whose relative difference in atomic diameter with an iron atom is less than or equal to 15%, so as to form an inner layer; And then the strip is soaked in a bath of molten aluminum.

9

Process according to claim 8, characterized in that the non-oxidizing atmosphere in which the strip is annealed contains one or more elements chosen from silicon, titanium, beryllium and manganese in gaseous form.

10

Process according to claim 9, characterized in that the non-oxidizing atmosphere in which the strip is annealed contains silane.

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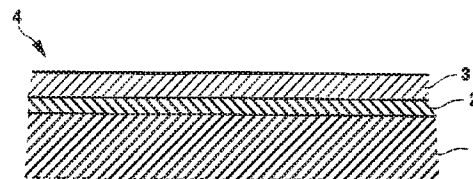
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54 TOLE D'ACIER MUNIE D'UN REVETEMENT A BASE D'ALUMINIUM.

57 L'invention a pour objet une tôle d'acier revêtue. Le re-  
vêtement est constitué de deux couches juxtaposées:

- une couche interne en contact avec le substrat compre-  
nant en poids entre 40 et 65% d'aluminium, entre 30 et  
50% de fer et entre 3 et 12% d'un ou plusieurs éléments  
choisis parmi ceux dont la différence relative de diamètre  
atomique avec un atome de fer est inférieure ou égale à  
15%, par exemple le silicium, le titane, le béryllium, le man-  
ganèse;

- une couche externe comprenant en poids entre 95 et  
98% d'aluminium et moins de 4% de fer.§.



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## TOLE D'ACIER MUNIE D'UN REVETEMENT A BASE D'ALUMINIUM.

La présente invention concerne le domaine des tôles revêtues.

Elle a trait spécifiquement aux tôles munies d'un revêtement à  
5 base d'aluminium, dites tôles aluminées.

Ces tôles aluminées sont particulièrement utilisées dans le domaine automobile pour réaliser des lignes d'échappement, des écrans thermiques destinées à isoler la caisse des lignes d'échappement, des réservoirs à carburant, ou des bras de suspension par exemple.

10 Elles trouvent également leur utilisation dans le domaine du bâtiment dans la réalisation par exemple de portes coupe-feu, de conduits de fumées, d'inserts de cheminée, de gaines ou d'éléments de couverture.

On utilise aussi les tôles aluminées dans le domaine de l'électroménager pour la réalisation par exemple de grille-pains, de mini-  
15 fours, de friteuses, de chambre de combustion de chauffe-eau, de brûleurs.

Il est connu d'utiliser des tôles aluminées constituées d'un substrat d'acier revêtu d'un alliage à base d'aluminium et de silicium.

Une telle tôle aluminée est réalisée par trempage d'une bande d'acier dans un bain en fusion contenant environ 90 % d'aluminium et 10 %  
20 de silicium, dans le but d'obtenir une couche de revêtement par exemple de 20 microns d'épaisseur.

Après diffusion des atomes de fer du substrat dans la couche de revêtement, celui-ci présente une première couche fortement alliée en contact avec le substrat contenant en poids environ 45 % de fer, 53 %  
25 d'aluminium, 2 % de silicium, puis une couche intermédiaire contenant environ 32 % de fer, 58 % d'aluminium, 10 % de silicium, et enfin une couche externe contenant environ 90 % d'aluminium, 9 % de silicium et très peu de fer (moins de 3 %).

A la résistance traditionnelle de l'acier du substrat, ce type de  
30 revêtement aluminium-silicium apporte une bonne tenue à l'oxydation à chaud, une bonne résistance à haute température et une bonne tenue à la corrosion, ce qui confère à ce type de tôle aluminée une aptitude particulière à la réalisation de lignes d'échappement de véhicules automobile, d'écrans thermiques, et plus généralement de pièces soumises à des températures  
35 élevées.

La couche fortement alliée contenant environ 45 % de fer étant très faible, de l'ordre du micromètre, et la couche intermédiaire contenant encore en moyenne 32 % de fer étant également faible, de l'ordre de 4 micromètres, cela permet d'obtenir une couche alliée de faible épaisseur, de l'ordre de 5 micromètres, ce qui confère à ce type de revêtement une bonne adhérence et autorise donc des emboutissages profonds.

Il est également connu d'utiliser des tôles aluminées constituées d'un substrat d'acier revêtu d'un alliage d'aluminium.

Une telle tôle aluminée est réalisée par trempage d'une bande d'acier dans un bain en fusion d'aluminium, dans le but d'obtenir une couche de revêtement de l'ordre de 35 à 60 microns d'épaisseur.

Après diffusion des atomes de fer du substrat dans la couche de revêtement, celui-ci présente une première couche fortement alliée en contact avec le substrat contenant en poids environ 42 % de fer, 58 % d'aluminium, puis une couche externe contenant environ 98 % d'aluminium et quasiment pas de fer (moins de 3 % présent sous forme de précipités  $\text{FeAl}_3$ ,  $\text{Fe}_2\text{Al}_5$  et  $\text{Fe}_2\text{Al}_7$ ).

Ce type de tôle aluminée présente une excellente résistance à la corrosion industrielle, urbaine et marine, une excellente résistance à la corrosion agressive des produits de combustion des hydrocarbures et à la corrosion en présence de chaleur.

De plus, ce type de tôle aluminée offre une très bonne réflectance au rayonnement (supérieure à 85 %), proche de celle d'une tôle en aluminium pur.

Elle présente également une bonne résistance à la chaleur et au feu. Jusqu'à une température égale à 450 °C, le revêtement n'est pas altéré, et entre 450 et 600 °C, on assiste à une transformation visuelle de la couche de revêtement mais sans altération notable de son pouvoir de protection.

Ce type de revêtement est parfaitement adapté pour la réalisation de toitures de bâtiments et pour les pièces de calorifugeage.

Mais l'adhérence du revêtement est modeste du fait de l'importance de la couche fortement alliée, d'épaisseur généralement supérieure à 12 micromètres pour ce type de tôle aluminée.

C'est pour cette raison que la couche de revêtement est d'épaisseur importante, entre 35 et 60 micromètres habituellement afin de garantir la présence d'une couche non alliée suffisante.

Du fait de ce problème d'adhérence, il est impossible de réaliser  
5 des emboutissages profonds sur ce type de tôle aluminisée sans altérer le revêtement, et en cas de pliage d'une telle tôle, il convient de respecter certaines précautions notamment en terme de rayons de courbure afin d'éviter de dégrader le revêtement. En effet, en cas d'emboutissage ou de pliage en conditions sévères, on crée des fissures dans la couche fortement  
10 alliée, entraînant fissuration, chevauchement et décohésion du revêtement.

Ainsi la technique connue propose soit une tôle aluminisée présentant une très bonne tenue à la corrosion, et de très bonnes propriétés réfléchives, proches de celles des tôles en aluminium pur, mais peu formables, soit une tôle aluminisée autorisant des emboutissages profonds,  
15 mais présentant une moins bonne tenue à la corrosion et de moins bonnes propriétés réfléchives.

La présente invention a pour but de proposer une tôle aluminisée présentant à la fois une très bonne tenue à la corrosion, une très bonne tenue à l'oxydation à chaud, de très bonnes propriétés réfléchives proches de  
20 celles des tôles en aluminium pur, autorisant des emboutissages profonds.

L'invention a pour objet une tôle d'acier revêtue constituée d'un substrat en acier muni sur ses deux faces d'un revêtement à base d'aluminium, caractérisé en ce que le revêtement est un revêtement constitué de deux couches superposées :

25 - une couche interne en contact avec le substrat comprenant en poids entre 40 et 65 % d'aluminium, entre 30 et 50 % de fer et entre 3 et 12 % d'un ou plusieurs éléments choisis parmi ceux dont la différence relative de diamètre atomique avec un atome de fer est inférieure ou égale à 15 % ;

30 - une couche externe comprenant en poids entre 95 et 98 % d'aluminium et moins de 4 % de fer.

Selon d'autres caractéristiques de l'invention :

- la couche interne comprend en poids entre 40 et 65 % d'aluminium, entre 30 et 50 % de fer, et entre 3 et 12 % d'un ou plusieurs éléments choisis parmi le silicium, le titane, le béryllium, le manganèse ;

35 - l'épaisseur de la couche interne est comprise entre 2 et 7 micromètres ;

- la couche externe comprend en poids entre 95 et 98 % d'aluminium, moins de 4 % de fer et moins de 2% d'un ou plusieurs éléments choisis parmi ceux dont la différence relative de diamètre atomique avec un atome de fer est inférieure ou égale à 15 % ;

5                   - l'épaisseur de la couche externe est comprise entre 5 et 50 micromètres.

L'invention concerne également un procédé de fabrication d'une telle tôle d'acier revêtue, caractérisé en ce qu'il comprend au moins trois étapes :

10                   - une première étape dans laquelle, dans le but de former une couche interne alliée contenant en poids entre 40 et 65 % d'aluminium, entre 30 et 50 % de fer et entre 3 et 12 % d'un ou plusieurs éléments choisis parmi ceux dont la différence relative de diamètre atomique avec un atome de fer est inférieure ou égale à 15 %, on trempe une tôle d'acier dans un bain en  
15 fusion contenant entre 88 et 95 % d'aluminium et entre 7 et 12 % d'un ou plusieurs éléments choisis parmi ceux dont la différence relative de diamètre atomique avec un atome de fer est inférieure ou égale à 15 % ;

                    - une seconde étape dans laquelle on effectue un essorage de la tôle pour éliminer la partie superficielle de revêtement non allié et ne  
20 conserver qu'une couche interne alliée d'épaisseur comprise entre 2 et 7 micromètres ;

                    - une troisième étape dans laquelle on trempe la tôle dans un bain d'aluminium en fusion.

L'invention concerne aussi un autre procédé de fabrication  
25 d'une telle tôle d'acier revêtue, caractérisé en ce que :

                    - on effectue un recuit d'une bande d'acier sous atmosphère non oxydante contenant des atomes, sous forme gazeuse, d'un ou plusieurs éléments dont la différence relative de diamètre atomique avec un atome de fer est inférieure ou égale à 15 %, de manière à former une couche interne ;

30                   - puis on trempe la bande dans un bain d'aluminium en fusion.

Selon d'autres caractéristiques :

                    - l'atmosphère non oxydante dans laquelle la bande est recuite contient un ou plusieurs éléments choisis parmi le silicium, le titane, le béryllium, le manganèse, sous forme gazeuse ;

35                   - l'atmosphère non oxydante dans laquelle la bande est recuite contient du silane.

Les caractéristiques et avantages apparaîtront mieux à la suite de la description qui suit, donnée uniquement à titre d'exemple, faite en référence aux dessins annexés, dans lesquels :

- la figure 1 est une vue schématique d'une tôle aluminisée selon l'invention,
- la figure 2 est un graphique représentant les facteurs de diamètre des principaux éléments,
- la figure 3 est une vue schématique d'une installation permettant de mettre en oeuvre un premier procédé de fabrication d'une tôle aluminisée selon l'invention,
- la figure 4 est une vue schématique d'une installation permettant de mettre en oeuvre un second procédé de fabrication d'une tôle aluminisée selon l'invention,
- la figure 5 est une vue schématique d'une autre installation permettant de mettre en oeuvre le second procédé de fabrication d'une tôle aluminisée selon l'invention.

Comme on peut le voir sur la figure 1, la tôle d'acier aluminisée selon l'invention est constituée d'un substrat 1 en acier revêtu de deux couches d'un revêtement distinct.

La première couche interne 2 en contact avec le substrat 1 comprend, en poids, entre 40 et 65 % d'aluminium, entre 30 et 50 % de fer et entre 3 et 12 % d'un ou plusieurs éléments choisis parmi ceux dont la différence relative de diamètre atomique avec un atome de fer est inférieure ou égale à 15 %.

Ces éléments sont identifiés sur la figure 2 entre les deux droites en tirets qui indiquent les limites entre lesquelles la différence relative de diamètre atomique avec un atome de fer est inférieure à 15 %, selon la classification de Hume-Rothery.

Ces éléments sont le béryllium, le silicium, le phosphore, le vanadium, le chrome, le manganèse, le cobalt, le nickel, le cuivre, le zinc, le germanium, l'arsenic, le molybdène, le ruthénium, le rhodium, le palladium, l'étain, le tungstène, le rhénium, l'iridium, le platine, le titane, le niobium, l'argent, l'antimoine, et l'or.

La seconde couche externe 3 comprend en poids entre 95 et 98 % d'aluminium et moins de 4 % de fer.

Cette seconde couche externe 3 peut également comprendre moins de 2% d'un ou plusieurs éléments choisis parmi ceux dont la différence relative de diamètre atomique avec un atome de fer est inférieure ou égale à 15 %, par exemple le béryllium, le silicium, le phosphore, le vanadium, le chrome, le manganèse, le cobalt, le nickel, le cuivre, le zinc, le germanium, l'arsenic, le molybdène, le ruthénium, le rhodium, le palladium, l'étain, le tungstène, le rhénium, l'iridium, le platine, le titane, le niobium, l'argent, l'antimoine, ou l'or.

La première couche interne 2 présente une épaisseur faible, entre 2 et 7 micromètres, afin de préserver les qualités d'adhérence du revêtement et la seconde couche 3 peut être comprise entre 5 et 50 micromètres selon l'épaisseur totale de revêtement désiré. De préférence, l'épaisseur de la couche 2 est inférieure ou égale à 5 micromètres.

Le ou les éléments dont le diamètre atomique est telle que la différence relative de diamètre atomique de chacun de ces éléments avec un atome de fer est inférieure à 15 %, contenus dans la couche 2 en contact avec le substrat, permettent de limiter le phénomène d'alliation entre cette couche de revêtement et le substrat.

En effet, les éléments dont le diamètre atomique est supérieur à celui du fer ont des difficultés à se déloger et ainsi bloquent la migration des atomes de fer.

En revanche, les éléments dont le diamètre atomique est inférieur à celui du fer peuvent plus facilement se déloger, mais la place libre laissée est insuffisante pour un atome de fer ou un atome d'aluminium.

Dans la pratique, on choisira de manière préférentielle les éléments exempts de toxicité et les éléments de plus faible coût. Ainsi, l'utilisation de l'arsenic, de l'argent, l'or ou le platine, bien que permettant de limiter le phénomène d'alliation, ne seront que très rarement utilisés.

De plus, selon le type d'acier du substrat, on évitera les éléments qui auront tendance à se combiner avec des constituants de l'acier, comme par exemple le niobium qui a tendance à piéger le carbone contenu dans l'acier.

De préférence, on utilisera un ou plusieurs éléments choisis parmi le silicium, le titane, le béryllium et le manganèse.

Un premier procédé pour fabriquer une tôle aluminée selon l'invention consiste à effectuer un recuit d'une bande d'acier sous atmosphère

non oxydante contenant des atomes, sous forme gazeuse, d'un ou plusieurs éléments dont la différence relative de diamètre atomique avec un atome de fer est inférieure ou égale à 15 %, puis à tremper la bande dans un bain d'aluminium en fusion.

5 Comme on peut le voir sur la figure 3, une bande 4 en acier défile en continu dans une enceinte de recuit 5 dans laquelle on a pris soin de faire régner une atmosphère non oxydante.

Au cours du recuit, on introduit dans l'atmosphère de l'enceinte de recuit un ou plusieurs éléments dont la différence relative de diamètre  
10 atomique avec un atome de fer est inférieure ou égale à 15 %.

Il est très important que la forme gazeuse sous laquelle ces éléments sont introduits dans l'enceinte de recuit soit une forme non oxydante. Par exemple dans le cas du silicium, on introduit les atomes de silicium sous forme de silane  $\text{SiH}_4$ .

15 A la sortie de l'enceinte de recuit, la bande est maintenue dans une atmosphère non oxydante dans une goulotte 6 jusqu'à ce qu'elle soit trempée dans un bain 7 d'aluminium en fusion.

Dans l'enceinte de recuit 5 et dans la goulotte 6, l'atmosphère non oxydante est composée par exemple d'un mélange d'azote et  
20 d'hydrogène, et la température de la bande est amenée à une valeur voisine de la température du bain 7, de préférence comprise entre 660°C et 730°C.

L'azote utilisé pour constituer l'atmosphère non oxydante contient de préférence moins de 5 ppm d'oxygène et a un point de rosée inférieur à -60°C.

25 Lorsque la bande 4 est réchauffée dans l'enceinte de recuit, ses faces principales se chargent des atomes de l'élément ou des éléments introduits dans ladite enceinte.

A la sortie du bain d'aluminium en fusion, les deux faces revêtues de la bande 4 sont essorées par un gaz soufflé à travers les buses  
30 d'essorage 8, et la bande est évacuée et débitée en tôles.

Ce procédé permet de réaliser le revêtement bi-couches selon l'invention en trempant la bande dans un seul bain en fusion, constitué uniquement d'aluminium. La première couche interne 2 est réalisée grâce à l'apport de ou des éléments introduits dans l'enceinte de recuit.

Un second procédé pour fabriquer une tôle aluminée selon l'invention consiste à déposer les deux couches interne 2 et externe 3 séparément.

La bande est d'abord trempée dans un bain en fusion contenant  
5 un alliage à base d'aluminium contenant entre 88 et 95 % d'aluminium et entre 7 et 12 % d'un ou plusieurs éléments choisis parmi ceux dont la différence relative de diamètre atomique avec un atome de fer est inférieure ou égale à 15 %, de préférence le silicium, le titane, le béryllium, le manganèse, dans le but de former une couche interne alliée contenant en  
10 poids entre 40 et 65 % d'aluminium, entre 30 et 50 % de fer et entre 3 et 12 % d'un ou plusieurs éléments choisis parmi ceux dont la différence relative de diamètre atomique avec un atome de fer est inférieure ou égale à 15 %.

Ensuite, la bande est essorée pour éliminer la partie superficielle de revêtement non alliée et ne conserver qu'une couche  
15 contenant entre 40 et 65 % d'aluminium, entre 30 et 50 % de fer, et entre 3 et 12 % du ou des éléments d'addition, d'épaisseur comprise entre 3 et 7 micromètres.

Enfin, la bande est trempée dans un bain d'aluminium en fusion.

Avant de tremper la bande dans le premier bain, la bande subit  
20 avantageusement un recuit sous atmosphère non oxydante pour amener sa température à une valeur voisine de la température du bain.

Ce procédé présente l'avantage de parfaitement contrôler l'épaisseur de la première couche de revêtement 2.

L'installation illustrée sur la figure 4 permet de mettre en oeuvre  
25 ce procédé.

La bande 4 en acier pénètre dans une enceinte de recuit 5 dans laquelle l'atmosphère est non oxydante, et est déviée par un rouleau 9 pour traverser une goulotte 6 et pour plonger dans un bain 10 de métal de revêtement, par exemple un bain contenant entre 88 et 95 % d'aluminium et  
30 entre 7 et 12 % de silicium.

Ensuite, après être déviée par un rouleau 11 disposé dans le bain 10, la bande 4 ressort du bain 10 et est essorée par du gaz soufflé à travers les buses 8.

Cette opération d'essorage permet d'éliminer la partie de la  
35 couche de revêtement déposé sur la bande non alliée. La bande comporte

alors une première couche de revêtement 2, d'épaisseur comprise entre 3 et 7 micromètres, de préférence égale à 5 micromètres.

La bande 4 pénètre alors immédiatement dans un bain 12 d'aluminium en fusion.

5 Il est très important de faire pénétrer la bande dans le bain 12 immédiatement après son essorage pour éviter toute oxydation.

Dans l'exemple de réalisation représenté, la bande 4 traverse verticalement le bain 10 puis est évacuée après avoir subie éventuellement une nouvelle opération d'essorage par exemple par du gaz soufflé à travers  
10 les buses 8.

L'installation illustrée sur la figure 5 permet également de mettre en oeuvre ce procédé.

Dans cette installation, la bande 4 ressort du bain 10, est essorée par du gaz soufflé à travers les buses 8.

15 Ensuite, après être déviée au moyen d'un rouleau 13, elle plonge dans un bain 14 d'aluminium en fusion, dans lequel elle est à nouveau déviée au moyen d'un rouleau 15, disposé dans le fond du bain 14, puis la bande ressort du bain 14 et subit une nouvelle opération d'essorage par du gaz soufflé à travers les buses 8.

20 Eventuellement, si le trajet de la bande entre les buses d'essorage 8 et le second bain 14 est long, on protège la bande en disposant une goulotte 16 entre lesdites buses d'essorage et le second bain, dans laquelle on maintient une atmosphère non oxydante.

La tôle d'acier aluminisée selon l'invention offre une très bonne  
25 réflectance au rayonnement (supérieur à 85 %), proche de celle d'une tôle en aluminium pur, une bonne résistance à la chaleur et au feu.

Elle autorise des emboutissages profonds car la couche alliée 2 est d'épaisseur très faible et fait barrière à la diffusion des atomes d'aluminium de la couche externe 3 vers le substrat 1 et à la diffusion des  
30 atomes de fer du substrat 1 vers la couche externe 3.

## REVENDEICATIONS

1 - Tôle d'acier revêtue constituée d'un substrat en acier muni sur ses deux faces d'un revêtement à base d'aluminium, caractérisé en ce que le revêtement est constitué de deux couches superposées :

- une couche interne (2) en contact avec le substrat comprenant en poids entre 40 et 65 % d'aluminium, entre 30 et 50 % de fer et entre 3 et 12 % d'un ou plusieurs éléments choisis parmi ceux dont la différence relative de diamètre atomique avec un atome de fer est inférieure ou égale à 15 % ;
- une couche externe (3) comprenant en poids entre 95 et 98 % d'aluminium et moins de 4 % de fer.

2 - Tôle d'acier revêtue selon la revendication 1, caractérisée en ce que la couche interne (2) comprend en poids entre 40 et 65 % d'aluminium, entre 30 et 50 % de fer, et entre 3 et 12 % d'un ou plusieurs éléments choisis parmi le silicium, le titane, le béryllium, le manganèse.

3 - Tôle d'acier revêtue selon les revendications 1 ou 2, caractérisée en ce que l'épaisseur de la couche interne (2) est comprise entre 2 et 7 micromètres.

4 - Tôle d'acier revêtue selon la revendication 1, caractérisée en ce que la couche externe (3) comprend en poids entre 95 et 98 % d'aluminium, moins de 4 % de fer et moins de 2% d'un ou plusieurs éléments choisis parmi ceux dont la différence relative de diamètre atomique avec un atome de fer est inférieure ou égale à 15 % ;

5 - Tôle d'acier revêtue selon les revendications 1 ou 4, caractérisée en ce que l'épaisseur de la couche externe (3) est comprise entre 5 et 50 micromètres.

6 - Procédé de fabrication d'une tôle d'acier revêtue sur ses deux faces principales d'un revêtement à base d'aluminium selon la revendication 1, caractérisé en ce qu'il comprend au moins trois étapes :

- une première étape dans laquelle, dans le but de former une couche interne alliée contenant en poids entre 40 et 65 % d'aluminium, entre

30 et 50 % de fer et entre 3 et 12 % d'un ou plusieurs éléments choisis parmi ceux dont la différence relative de diamètre atomique avec un atome de fer est inférieure ou égale à 15 %, on trempe une tôle d'acier dans un bain en fusion contenant entre 88 et 95 % d'aluminium et entre 7 et 12 % d'un ou  
5 plusieurs éléments choisis parmi ceux dont la différence relative de diamètre atomique avec un atome de fer est inférieure ou égale à 15 % ;

- une seconde étape dans laquelle on effectue un essorage de la tôle pour éliminer la partie superficielle de revêtement non allié et ne conserver qu'une couche interne alliée d'épaisseur comprise entre 3 et 7  
10 micromètres ;

- une troisième étape dans laquelle on trempe la tôle dans un bain d'aluminium en fusion.

7 - Procédé selon la revendication 6, caractérisé en ce que  
15 préalablement à la première étape, on effectue un recuit de la bande d'acier dans une atmosphère non oxydante.

8 - Procédé de fabrication d'une tôle d'acier revêtue sur ses deux faces principales d'un revêtement à base d'aluminium selon la  
20 revendication 1, caractérisé en ce que :

- on effectue un recuit d'une bande d'acier sous atmosphère non oxydante contenant des atomes, sous forme gazeuse, d'un ou plusieurs éléments dont la différence relative de diamètre atomique avec un atome de fer est inférieure ou égale à 15 %, de manière à former une couche interne ;

25 - puis on trempe la bande dans un bain d'aluminium en fusion.

9 - Procédé selon la revendication 8, caractérisé en ce que l'atmosphère non oxydante dans laquelle la bande est recuite contient un ou plusieurs éléments choisis parmi le silicium, le titane, le béryllium, le  
30 manganèse, sous forme gazeuse.

10 - Procédé selon la revendication 9, caractérisé en ce que l'atmosphère non oxydante dans laquelle la bande est recuite contient du silane.

1/4

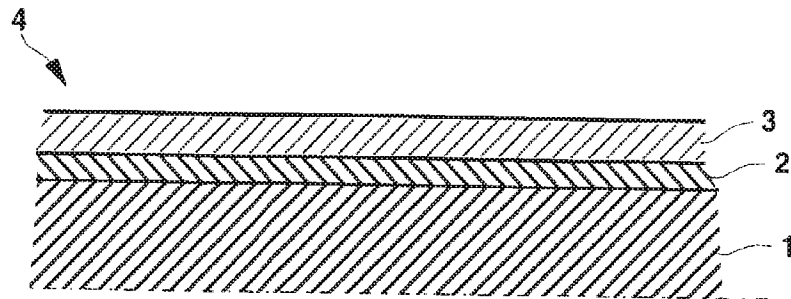


Fig. 1

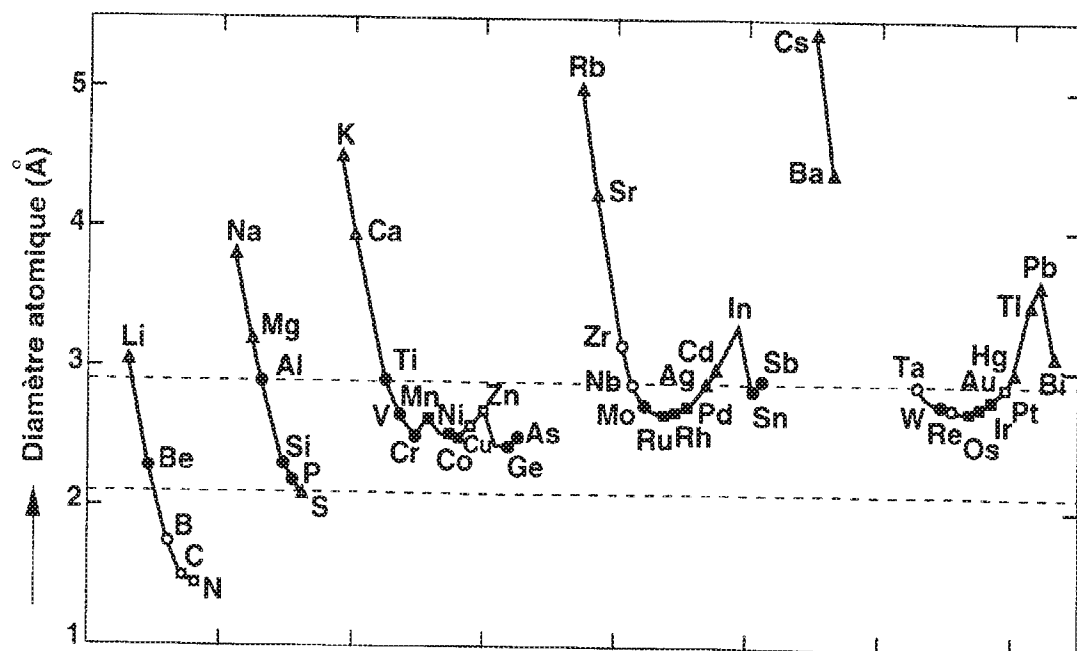


Fig. 2

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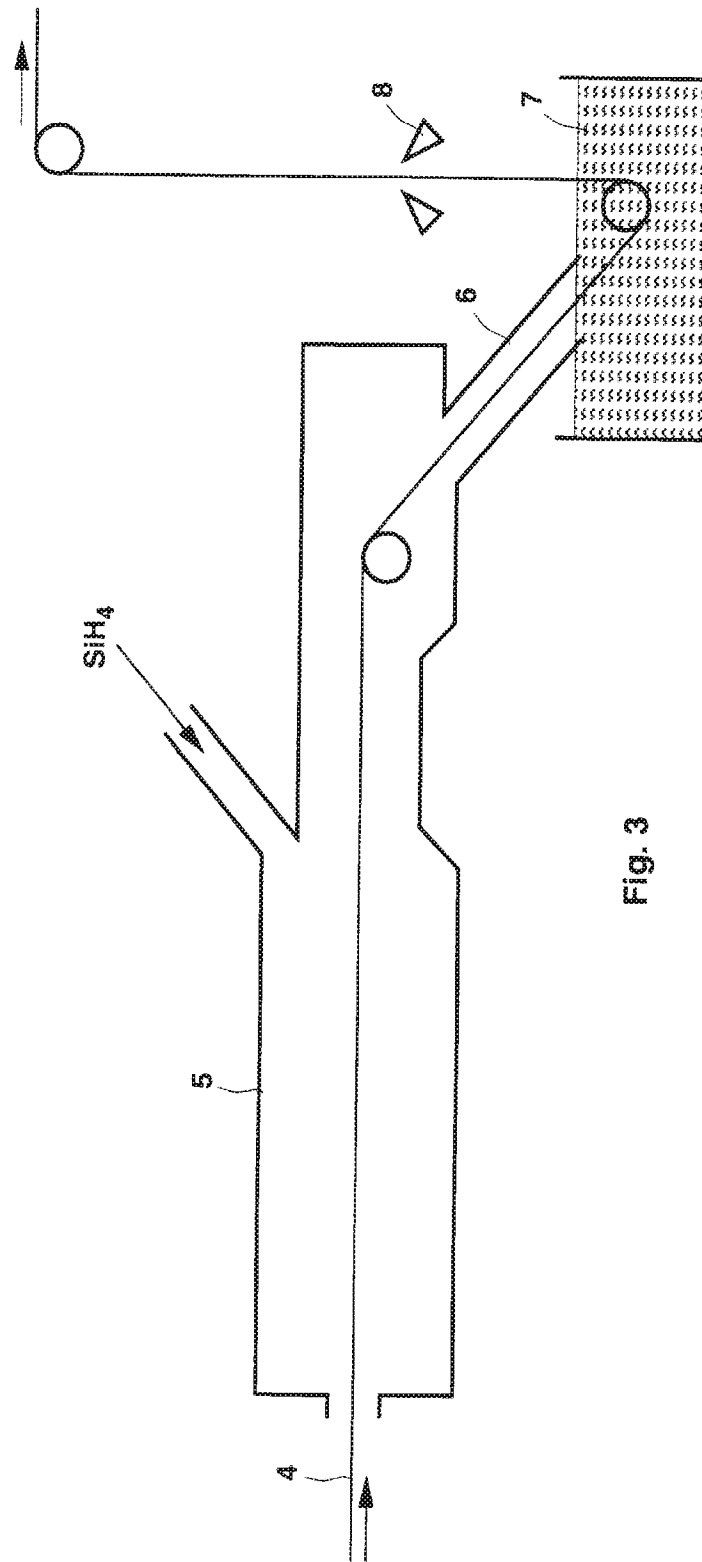


Fig. 3

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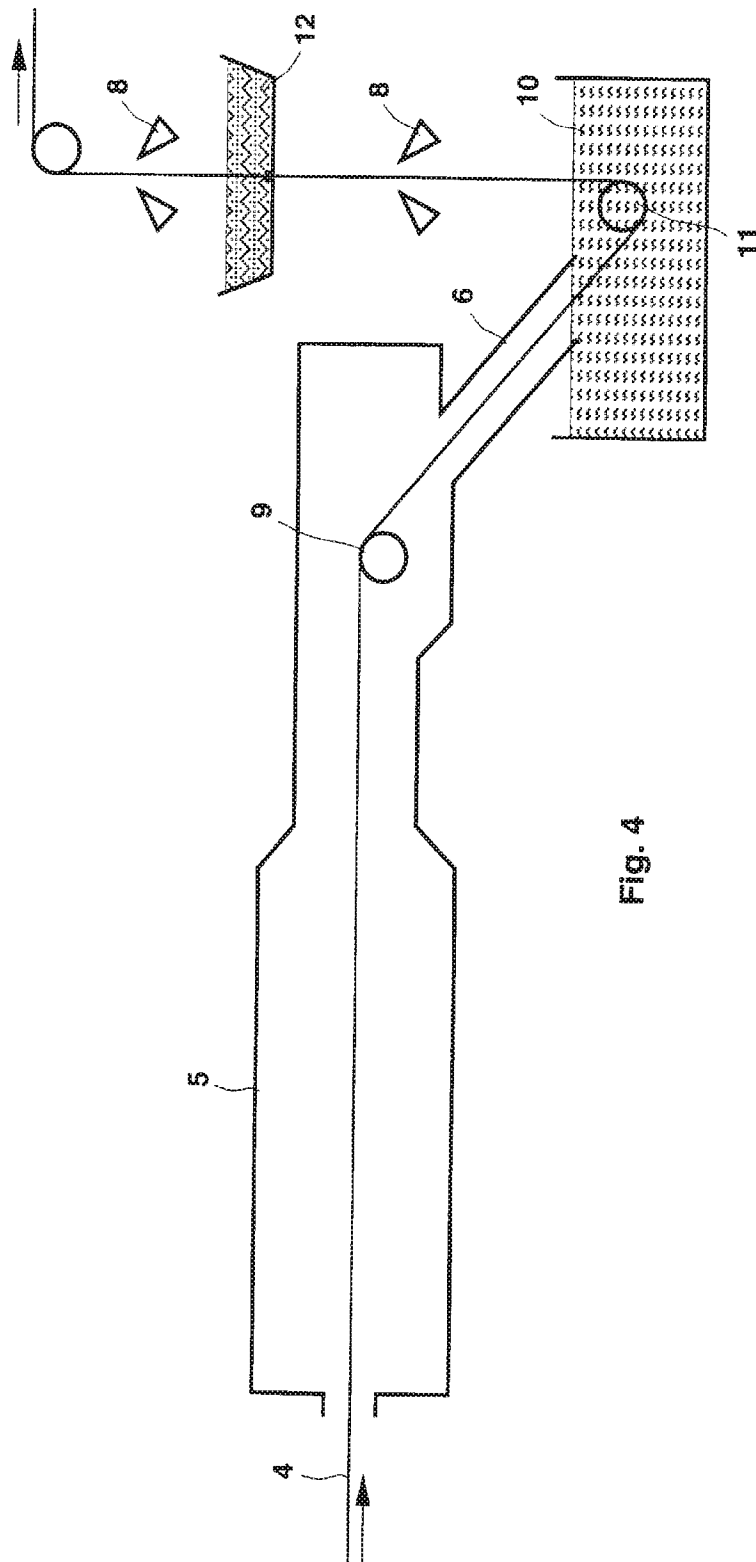


Fig. 4

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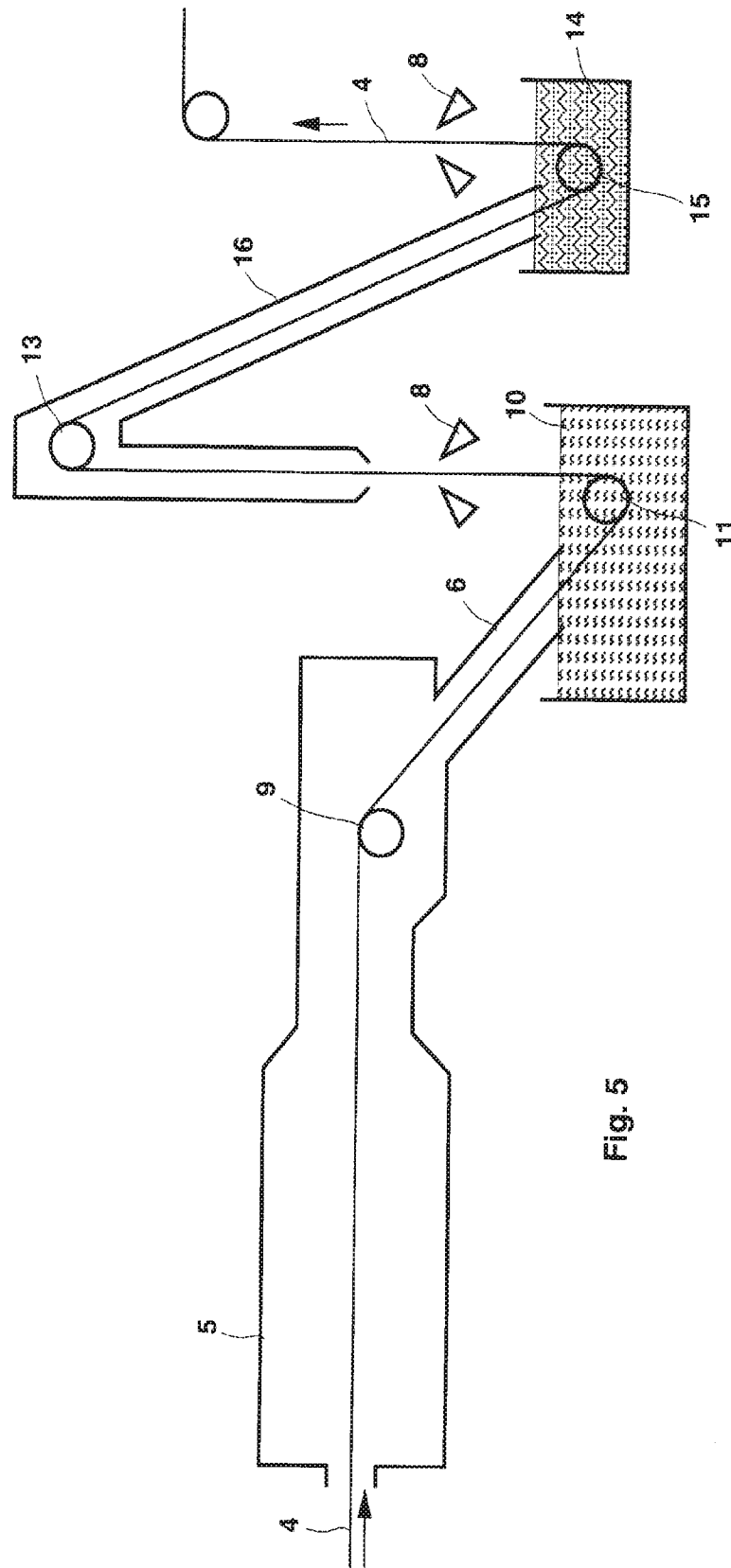


Fig. 5

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établi sur la base des dernières revendications  
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N° d'enregistrement  
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| DOCUMENTS CONSIDERES COMME PERTINENTS                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 |                                                                                                                                                        | Revendications<br>concernées<br>de la demande<br>examinée |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------|
| Catégorie                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             | Citation du document avec indication, en cas de besoin,<br>des parties pertinentes                                                                     |                                                           |
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| 6 octobre 1997                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                                                                                                                                                        | Elsen, D                                                  |
| <p>CATEGORIE DES DOCUMENTS CITES</p> <p>X : particulièrement pertinent à lui seul<br/>Y : particulièrement pertinent en combinaison avec un<br/>autre document de la même catégorie<br/>A : pertinent à l'encontre d'au moins une revendication<br/>ou arrière-plan technologique général<br/>O : divulgation non-écrite<br/>P : document intercalaire</p> <p>T : théorie ou principe à la base de l'invention<br/>E : document de brevet bénéficiant d'une date antérieure<br/>à la date de dépôt et qui n'a été publié qu'à cette date<br/>de dépôt ou qu'à une date postérieure.<br/>D : cité dans la demande<br/>L : cité pour d'autres raisons<br/>&amp; : membre de la même famille, document correspondant</p> |                                                                                                                                                        |                                                           |

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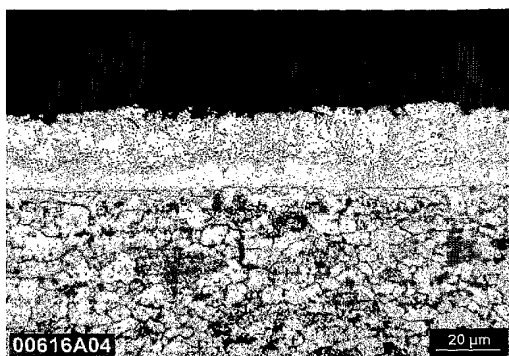
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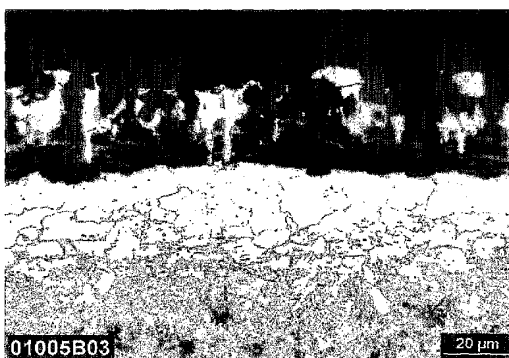
[Suite sur la page suivante]

(54) Title: METHOD FOR MAKING A COATED STEEL PART HAVING VERY HIGH RESISTANCE AFTER HEAT TREATMENT

(54) Titre : PROCEDE DE FABRICATION D'UNE PIECE D'ACIER REVETU PRESENTANT UNE TRES HAUTE RESISTANCE APRES TRAITEMENT THERMIQUE



1



4

(57) Abstract: The invention concerns a method for making a part with very high mechanical characteristics from a hot- or cold-rolled steel strip, comprising precoating said strip with aluminum or an aluminum alloy, cold process deformation of the coated strip, optionally cutting off excess plate metal in view of the final shape of the part, heating the part so as to produce an intermetallic compound from the steel-coating interface and to austenize the steel, transferring the part into an equipment, cooling the part inside the equipment at a speed such that the steel structure after cooling is martensitic, or bainitic or martensite-bainitic. The precoating is performed by electroplating, chemical or physical vapour deposition, or combined rolling.

(57) Abrégé : L'invention concerne un procédé de fabrication d'une pièce à très hautes caractéristiques mécaniques à partir d'une bande d'acier laminée à chaud ou à froid, comportant un pré-revêtement de ladite bande par de l'aluminium ou un alliage d'aluminium, une déformation à froid de la bande revêtue, une découpe éventuelle des excédents de tôle en vue de la géométrie finale de la pièce, un chauffage de la pièce de façon à réaliser un composé intermétallique à partir de l'interface acier-revêtement et à austénitiser l'acier, un transfert de la pièce au sein d'un outillage, un refroidissement de la pièce au sein de l'outillage avec une vitesse telle que la structure de l'acier après refroidissement soit martensitique, ou bainitique ou martensite-bainitique. Le pré-revêtement est effectué par électrodéposition, dépôt chimique ou physique en phase vapeur, ou colaminage.



KM, KN, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, LY, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

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En ce qui concerne les codes à deux lettres et autres abréviations, se référer aux "Notes explicatives relatives aux codes et abréviations" figurant au début de chaque numéro ordinaire de la Gazette du PCT.

**PROCEDE DE FABRICATION D'UNE PIECE D'ACIER REVÊTU  
PRESENTANT UNE TRES HAUTE RESISTANCE APRES TRAITEMENT  
THERMIQUE**

5 L'invention concerne la fabrication de pièces d'acier revêtu laminé à chaud ou à froid, présentant une haute résistance mécanique ainsi qu'une bonne résistance à la corrosion.

Pour certaines applications, on cherche à réaliser des pièces en acier combinant une résistance mécanique élevée, une grande résistance aux  
10 chocs et une bonne tenue à la corrosion. Ce type de combinaison est particulièrement désirable dans l'industrie automobile où l'on recherche un allègement significatif des véhicules. Ceci peut être notamment obtenu grâce à l'utilisation d'aciers à très hautes caractéristiques mécaniques : Des pièces anti-intrusion, de structure ou de sécurité des véhicules automobiles  
15 (traverses de pare-choc, renforts de portière ou de pied milieu, bras de roue) nécessitent par exemple les qualités évoquées plus haut.

Le brevet FR 2807447 divulgue un procédé de fabrication où on approvisionne une tôle d'acier de base avec un pré-revêtement métallique, l'acier possédant une résistance à la rupture de l'ordre de 500MPa, on  
20 procède à une opération de mise en forme à froid, par exemple d'emboutissage ou de profilage à froid, puis l'on effectue un traitement thermique en vue d'une trempe ultérieure au sein d'un outillage de forme adapté à la géométrie de la pièce. Durant la phase de chauffage de ce traitement thermique, on forme un revêtement intermétallique à la surface de  
25 la pièce par alliation du pré-revêtement initial et de l'acier de base. De la sorte, on obtient par exemple des pièces avec une résistance mécanique supérieure à 1500MPa offrant une résistance à la corrosion.

La tôle d'acier de base peut être prérevêtue d'aluminium ou d'un alliage d'aluminium par un procédé au trempé. On peut cependant rencontrer des  
30 limitations à la mise en œuvre de ce procédé dans certains cas. Lors d'opérations de mise en forme à froid de la pièce avant traitement thermique, certaines zones peuvent être soumises à une déformation plus sévère, et l'on peut éventuellement assister à un endommagement de l'interface entre le

substrat et le pré-revêtement, sous forme de décohésion locale. Dans ce cas, le traitement thermique ultérieur peut conduire à la formation de calamine au voisinage de la couche interfaciale d'alliage. La présence de cette calamine est néfaste à la réalisation d'une alliation satisfaisante entre l'acier de base et le pré-revêtement aluminé.

Par ailleurs, après mise en forme à froid des pièces aluminées, on peut effectuer une découpe, un poinçonnage ou un détourage en vue d'enlever l'excédent de matière avant le traitement thermique ultérieur d'alliation. La présence du pré-revêtement aluminé au trempé peut contribuer à l'usure de l'outillage de découpe.

D'autre part, le pré-revêtement des tôles aluminées au trempé peut présenter une variation d'épaisseur par rapport à l'épaisseur nominale. Le chauffage lors du traitement thermique d'alliation est effectué assez rapidement, typiquement en quelques minutes. En cas de surépaisseur excessive, on assiste à une alliation incomplète du revêtement. La température de fusion des pré-revêtements usuels étant de 660°C pour l'aluminium ou de 580°C pour un alliage aluminium-10% silicium, on pourra assister à une fusion prématurée du côté le plus épais du revêtement avant d'atteindre la température d'austénitisation de la pièce. Les traitements thermiques étant effectués généralement dans des fours où les pièces sont déplacées sur des rouleaux, la surface de ces derniers est polluée d'une couche provenant de la fusion partielle du pré-revêtement, ce qui nuit à la bonne marche des fours. En outre, une alliation incomplète du pré-revêtement est néfaste lors d'opérations ultérieures de cataphorèse

La présente invention a pour but de résoudre les problèmes évoqués ci-dessus. Elle vise en particulier à mettre à disposition un procédé de fabrication de pièces d'acier laminées à chaud ou à froid pré-revêtues d'aluminium ou d'alliage d'aluminium, permettant une déformation importante préalable à froid avant traitement d'alliation sans risque de conséquence ultérieure sur le traitement d'alliation. Elle vise à réduire l'usure de l'outillage lors de l'usinage mécanique avant le traitement thermique d'alliation. Elle vise également à obtenir, après traitement thermique, une alliation complète du pré-revêtement d'aluminium ou d'alliage d'aluminium.

Dans ce but, l'invention a pour objet un procédé de fabrication d'une pièce à très hautes caractéristiques mécaniques à partir d'une bande d'acier, laminée à chaud ou à froid, comportant les étapes successives suivantes :

- 5       - On effectue un pré-revêtement de la bande par de l'aluminium ou d'un alliage d'aluminium. Ce pré-revêtement peut être réalisé par une ou plusieurs étapes selon les modes ci-dessous, seuls ou en combinaison :
  - 10       - pré-revêtement par une ou plusieurs étapes d'électrodéposition d'aluminium ou d'alliage d'aluminium
  - 10       - pré-revêtement par une ou plusieurs étapes de dépôt chimique en phase vapeur d'aluminium ou d'alliage d'aluminium
  - 10       - pré-revêtement par une ou plusieurs étapes de dépôt physique en phase vapeur d'aluminium ou d'alliage d'aluminium.
  - 15       - pré-revêtement par une ou plusieurs étapes de colaminage entre la bande d'acier et un feuillard d'aluminium ou d'alliage d'aluminium.
  - 15       - Grâce à la mise en œuvre de ces modes de pré-revêtement, l'interface entre la bande d'acier et le pré-revêtement ne comporte pas de phase intermétallique.
- 20       - On déforme à froid la bande revêtue
- 20       - On retire éventuellement les excédents de tôle en vue de la géométrie finale de ladite pièce
- 25       - On chauffe la pièce, par exemple au sein d'un four, de façon à réaliser un composé intermétallique à la surface de la pièce, à partir de l'interface acier-revêtement et à austénitiser l'acier. Durant la phase de chauffage de ce traitement thermique, on forme, par alliation de la couche de pré-revêtement initial et de l'acier de base, un revêtement intermétallique à la surface de la pièce, cette alliation étant réalisée dans la totalité de l'épaisseur de la couche de revêtement.
- 30       - Après chauffage, on transfère la pièce au sein d'un outillage. Le temps de transfert entre la phase de chauffage et le contact avec l'outillage est suffisamment court pour qu'une transformation de l'austénite n'intervienne pas durant ce laps de temps. La géométrie et la conception de l'outillage sont adaptées à la pièce à traiter et à la

drasticité de la trempe. En particulier, ces outils peuvent être refroidis, notamment par circulation de fluide pour augmenter la productivité des opérations et/ou accroître la sévérité de trempe. Un effort de serrage peut assurer un contact intime entre les pièces et l'outillage, permettant ainsi un refroidissement efficace par conduction et une déformation minimale. On refroidit la pièce au sein de l'outillage avec une vitesse telle que la structure de l'acier après refroidissement soit martensitique, ou bainitique ou martensito-bainitique.

Selon un mode particulier, le taux de la déformation généralisée de la déformation à froid est supérieure à 20% en au moins un point de la pièce.

L'invention a également pour objet l'utilisation d'une pièce à très hautes caractéristiques mécaniques à partir d'une bande d'acier, fabriquée selon l'un des modes ci-dessus, pour la fabrication de pièces de structures ou de sécurité pour véhicule automobile terrestre à moteur.

D'autres caractéristiques et avantages de l'invention apparaîtront au cours de la description ci-dessous, donnée à titre d'exemple.

La figure 1 présente un exemple d'interface acier-alliage d'aluminium déposé au trempé, avant déformation à froid

La figure 2 présente l'évolution de cette interface après déformation généralisée à froid supérieure à 20%.

La figure 3 présente un exemple d'interface acier-alliage d'aluminium déposé au trempé, sans déformation à froid, après traitement d'alliation.

La figure 4 illustre la couche superficielle après déformation à froid supérieure à 20%, suivie par un traitement d'alliation.

On a examiné l'évolution de l'interface acier-revêtement au cours d'un procédé de fabrication conventionnel : dans ce but, on a considéré des pièces d'acier de 1,2 ou 2mm d'épaisseur, de composition pondérale suivante :

Carbone : 0,15 à 0,25%

Manganèse : 0,8 à 1,5%

Silicium : 0,1 à 0,35%

Chrome : 0,01 à 0,2%

Titane < 0,1%

Phosphore < 0,05%

Soufre < 0,03%

B : 0,0005% à 0,01%,

5 Celles-ci ont été pré-revêtues grâce à un procédé conventionnel au trempé dans un bain à base d'aluminium comportant :

Silicium : 9-10%

Fer : 2 à 3,5%

Le solde étant constitué d'aluminium et des inévitables impuretés

10 Il est connu que le contact d'un acier dans un bain d'aluminium pur à plus de 660°C conduit à une formation très rapide d'une épaisse couche d'alliage intermétallique, comportant notamment FeAl<sub>3</sub>-Fe<sub>2</sub>Al<sub>5</sub>. Cette couche présentant une faible aptitude à la déformation, une addition de 10% de silicium dans le bain permet de réduire l'épaisseur de cette couche intermédiaire. La figure 1 indique que la couche intermétallique, d'une dureté

15 de 600 à 800HV, a une épaisseur d'environ 7 micromètres, celle-ci étant surmontée par une couche métallique à base d'aluminium de 15 micromètres environ.

Les pièces pré-revêtues ont été soumises à une déformation à froid sur éprouvettes de type Nakazima, sollicitant celles-ci selon différents modes :

20 traction uniaxiale, expansion équi-biaxiale. Au moyen de grilles à motifs circulaires préalablement photodéposées, on a mesuré en divers points les déformations principales  $\varepsilon_1$ ,  $\varepsilon_2$ , c'est à dire les déformations dans un repère principal. On en déduit la déformation généralisée :

$$\bar{\varepsilon} = \frac{2}{\sqrt{3}} \sqrt{(\varepsilon_1^2 + \varepsilon_1 \varepsilon_2 + \varepsilon_2^2)} \text{ relative à ces différents points.}$$

25 Simultanément, on a observé en ces différents emplacements le comportement du pré-revêtement : Les observations révèlent que, jusqu'à une déformation généralisée de l'ordre de 10%, la couche intermédiaire est fissurée de manière fine et régulière, sans conséquence sur la couche supérieure d'aluminium métallique surmontant celle-ci. Un traitement

30 thermique ultérieur en four à 900°C pendant 5 ou 7 minutes, suivi d'une trempe au sein d'un outillage refroidi à l'eau conduit à une alliation complète du revêtement initial et à la disparition de ce réseau limité de fissures (figure

3). Au-delà de 20% de déformation généralisée, on assiste à une fragmentation de la couche intermétallique (figure 2), et par endroits, à une dégradation du revêtement métallique à base d'aluminium. Le traitement thermique ultérieur d'alliation peut alors provoquer la croissance d'une  
5 couche de calamine ou une décarburation à la surface de l'acier (figure 4) néfaste à la mise en œuvre ultérieure de la pièce, par exemple à une mise en peinture

Ainsi, la mise en œuvre d'un procédé de déformation à froid avec une déformation sévère peut conduire à des difficultés à partir d'un pré-  
10 revêtement conventionnel à base d'aluminium. Dans le cadre de l'invention, on a mis en évidence que ce problème était résolu lorsque l'interface entre l'acier et l'aluminium ne comportait pas de phase intermétallique. En effet, compte tenu de la ductilité intrinsèque de l'aluminium ou de l'alliage d'aluminium, liée à sa structure cubique-face-centrée, une déformation à froid  
15 importante d'un acier pré-revêtu ne conduit à aucune dégradation de l'interface ou du pré-revêtement, si bien que le traitement d'alliation ultérieur intervient dans des conditions optimales.

Le pré-revêtement d'aluminium ou d'un alliage d'aluminium est réalisé par électrodéposition, par dépôt physique ou chimique en phase vapeur, ou  
20 encore par colaminage entre une bande d'acier et un feuillard d'aluminium ou d'alliage d'aluminium. Par ces différentes étapes, on aboutit ainsi à une pièce sans couche intermétallique entre l'acier de base et le pré-revêtement avant le traitement d'alliation. Le procédé selon l'invention peut être mis en œuvre en appliquant une même étape de pré-revêtement en une seule reprise, ou  
25 en appliquant celui-ci à plusieurs reprises. De même, le procédé peut être mis en œuvre selon l'invention en combinant successivement différentes étapes de pré-revêtements de façon à exploiter les avantages intrinsèques aux différents procédés et aux différentes caractéristiques des dépôts.

L'application du procédé selon l'invention facilite la mise en œuvre dans le  
30 cas d'un découpage, d'un poinçonnage ou d'un détournage des pièces après l'opération de mise en forme à froid. En effet, un usinage intermédiaire peut s'avérer utile dans le but de diminuer le volume de métal à réchauffer dans le traitement d'alliation. Selon l'invention, cet usinage intermédiaire est facilité

par l'absence de la couche intermétallique dure (de 600 à 800 HV) que l'on rencontre dans le procédé conventionnel. De la sorte, l'usure des outils de découpe est réduite.

Par ailleurs, les étapes de pré-revêtement selon l'invention sont associées à des dépôts d'une grande régularité d'épaisseur : par exemple, les conditions de pré-revêtement en phase vapeur peuvent être associées à un dépôt d'épaisseur compris entre 15 et 20 micromètres avec une variation d'épaisseur d'ordre micrométrique.

Selon les procédés d'aluminage au trempé, la variation d'épaisseur du pré-revêtement mesurée sur coupe micrographique peut être de l'ordre de  $\pm 10$  micromètres pour une épaisseur moyenne de 25 micromètres. Dans le but de maximiser la productivité, on souhaite que le chauffage lors du traitement thermique d'alliation s'effectue le plus rapidement possible. La constatation de la présence d'une surépaisseur peut conduire à allonger la phase de chauffage, pour que l'alliation soit complète. Pour un traitement thermique donné, la méconnaissance d'une surépaisseur excessive peut avoir pour conséquence une alliation incomplète, se traduisant par une fusion partielle du pré-revêtement.

L'étape de pré-revêtement selon l'invention conduit à une faible variabilité de l'épaisseur, ce qui diminue le risque de fusion et augmente la stabilité de la marche des fours.

Par ailleurs, après austénitisation, le traitement de trempe par refroidissement au sein d'un outillage confère une structure martensitique ou bainitique ou martensito-bainitique à l'acier. Selon la composition de l'acier, en particulier sa teneur en carbone, ainsi qu'en manganèse, chrome et bore, la résistance maximale obtenue sur les pièces selon l'invention, varie de 1200 à 1700 MPa. Selon l'invention, la coupe s'effectuant de manière plus nette en raison de l'absence de couche intermétallique, l'effet d'entaille en bord de découpe est moindre après traitement de trempe, puisque l'on sait que des structures totalement ou partiellement martensitiques sont par essence plus sensibles aux effets de concentrations locales de contraintes.

Ainsi, l'invention permet la fabrication de pièces à hautes caractéristiques revêtues, avec des formes plus complexes, puisque la déformation à froid

peut atteindre des taux importants. L'invention est mise en œuvre de façon particulièrement avantageuse lorsque le taux de la déformation généralisée à froid préalable au traitement d'alliation est supérieur à 20%. Elle permet une réduction de l'usure de l'outillage lors des opérations intermédiaires de  
5 découpe et conduit à une plus grande efficacité du traitement final d'alliation.

## REVENDICATIONS

1. Procédé de fabrication d'une pièce à très hautes caractéristiques  
5 mécaniques à partir d'une bande d'acier, laminée à chaud ou à froid,  
comportant les étapes successives suivantes :
- On effectue un pré-revêtement de ladite bande par de l'aluminium ou  
un alliage d'aluminium
  - On déforme à froid ladite bande revêtue
  - 10 - On retire éventuellement les excédents de tôle en vue de la géométrie  
finale de ladite pièce
  - On chauffe ladite pièce de façon à réaliser un composé intermétallique  
à la surface de ladite pièce à partir de l'interface acier-revêtement et à  
austénitiser l'acier
  - 15 - On transfère ladite pièce au sein d'un outillage
  - On refroidit ladite pièce au sein d'un outillage avec une vitesse telle  
que la structure de l'acier après refroidissement soit martensitique ou  
bainitique ou martensito-bainitique,
  - caractérisé en ce que l'interface entre ladite bande d'acier et le pré-  
20 revêtement avant ledit chauffage ne comporte pas de phase  
intermétallique et que l'on réalise ledit pré-revêtement par au moins  
une étape d'électrodéposition d'aluminium ou d'alliage d'aluminium,  
par au moins une étape de dépôt chimique en phase vapeur  
d'aluminium ou d'alliage d'aluminium, par au moins une étape de  
25 dépôt physique en phase vapeur d'aluminium ou d'alliage d'aluminium,  
par au moins une étape de colaminage entre ladite bande d'acier et un  
feuillard d'aluminium ou d'alliage d'aluminium, lesdites au moins une  
étape de pré-revêtement pouvant être réalisées seules ou en  
combinaison

30

2 Procédé de fabrication selon la revendication 1 caractérisé en ce que le taux de la déformation généralisée de ladite déformation à froid est supérieur à 20% en au moins un point de ladite pièce

5

3 Utilisation d'une pièce à très hautes caractéristiques mécaniques à partir d'une bande d'acier fabriquée selon la revendication 1 ou 2 pour la fabrication de pièces de structures ou de sécurité pour véhicule automobile

10 terrestre à moteur.

1/2

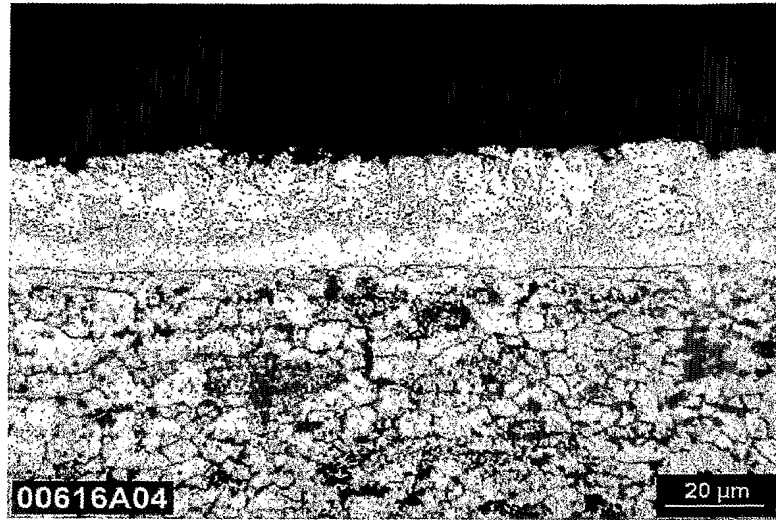


Figure 1

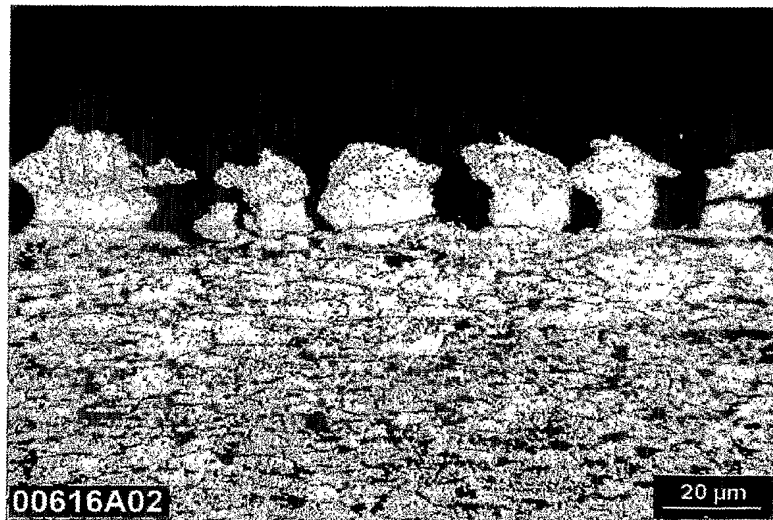


Figure 2

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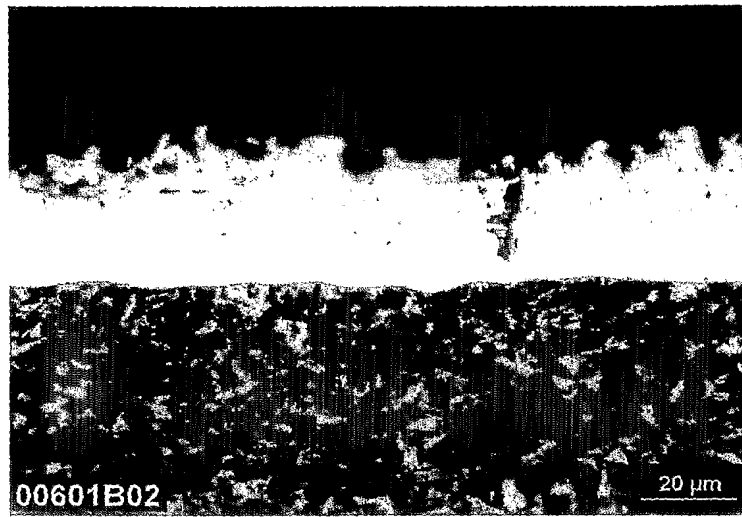


Figure 3

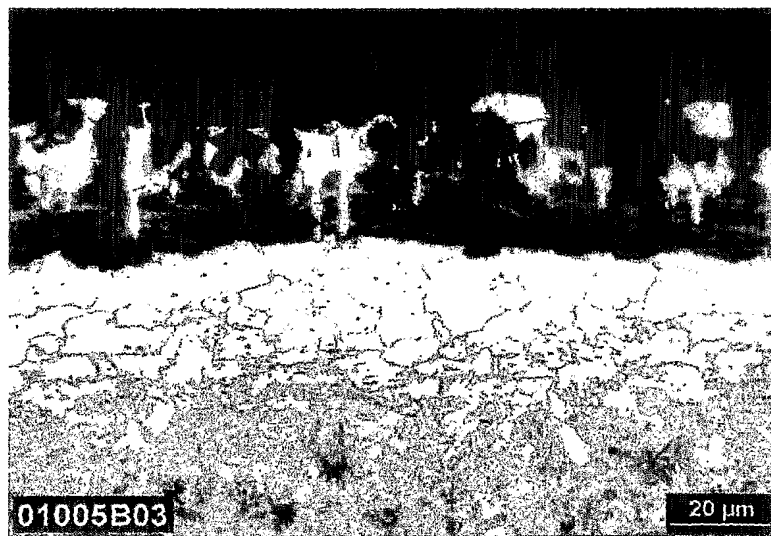


Figure 4

# INTERNATIONAL SEARCH REPORT

International application No  
PCT/FR2006/000466

**A. CLASSIFICATION OF SUBJECT MATTER**  
INV. C23C22/78 C23F17/00

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)  
C21D C25D C23F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ, WPI Data

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

| Category* | Citation of document, with indication, where appropriate, of the relevant passages                                                                                                                 | Relevant to claim No. |
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☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

\* Special categories of cited documents :

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

- \*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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- \*Z\* document member of the same patent family

Date of the actual completion of the international search

23 June 2006

Date of mailing of the international search report

30/06/2006

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## INTERNATIONAL SEARCH REPORT

International application No  
PCT/FR2006/000466

| C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT |                                                                                                                                                                                                                                                                              |                       |
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Information on patent family members

International application No

PCT/FR2006/000466

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# RAPPORT DE RECHERCHE INTERNATIONALE

Demande internationale n°

PCT/FR2006/000466

**A. CLASSEMENT DE L'OBJET DE LA DEMANDE**  
INV. C23C22/78 C23F17/00

Selon la classification internationale des brevets (CIB) ou à la fois selon la classification nationale et la CIB

**B. DOMAINES SUR LESQUELS LA RECHERCHE A PORTE**

Documentation minimale consultée (système de classification suivi des symboles de classement)  
C21D C25D C23F

Documentation consultée autre que la documentation minimale dans la mesure où ces documents relèvent des domaines sur lesquels a porté la recherche

Base de données électronique consultée au cours de la recherche internationale (nom de la base de données, et si cela est réalisable, termes de recherche utilisés)

EPO-Internal, PAJ, WPI Data

**C. DOCUMENTS CONSIDERES COMME PERTINENTS**

| Catégorie* | Identification des documents cités, avec, le cas échéant, l'indication des passages pertinents                                                                                                               | no. des revendications visées |
|------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------|
| Y          | US '6 197 132 B1 (ANDERSSON-DRUGGE<br>ING-MARIE) 6 mars 2001 (2001-03-06)<br>colonne 1, ligne 5-11<br>colonne 2, ligne 1-12,35-67; figures 2,4;<br>exemple 2<br>colonne 4, ligne 10-13<br>revendications 1-4 | 1-3                           |
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☒ Voir la suite du cadre C pour la fin de la liste des documents

☒ Les documents de familles de brevets sont indiqués en annexe

\* Catégories spéciales de documents cités:

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\*X\* document particulièrement pertinent; l'invention revendiquée ne peut être considérée comme nouvelle ou comme impliquant une activité inventive par rapport au document considéré isolément

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\*S\* document qui fait partie de la même famille de brevets

Date à laquelle la recherche internationale a été effectivement achevée

23 juin 2006

Date d'expédition du présent rapport de recherche internationale

30/06/2006

Nom et adresse postale de l'administration chargée de la recherche internationale  
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Fonctionnaire autorisé

Handrea-Haller, M

| C(suite). DOCUMENTS CONSIDERES COMME PERTINENTS |                                                                                                                                                                                                                                                                                            |                               |
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| Y                                               | GB 1 489 618 A (SUMITOMO METAL IND LTD)<br>26 octobre 1977 (1977-10-26)<br>page 1, ligne 7-9,25-46<br>page 2, ligne 1-12; exemples 2,4<br>revendications 1-4,6-8<br>-----                                                                                                                  | 1-3                           |
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| A                                               | DE 12 90 024 B (INTERLICENCE S. A)<br>27 février 1969 (1969-02-27)<br>le document en entier<br>-----                                                                                                                                                                                       | 1-3                           |
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# RAPPORT DE RECHERCHE INTERNATIONALE

Renseignements relatifs aux membres de familles de brevets

Demande internationale n°

PCT/FR2006/000466

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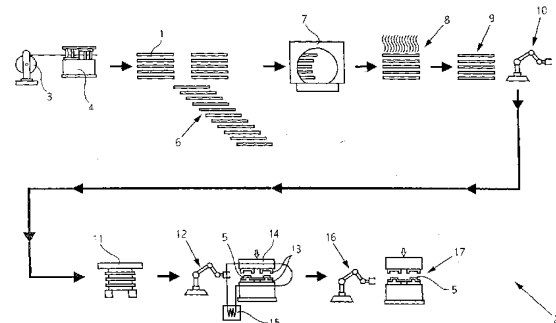
**Die folgenden Angaben sind den vom Anmelder eingereichten Unterlagen entnommen**

Prüfungsantrag gemäß § 44 PatG ist gestellt.

(54) Bezeichnung: **Verfahren zur Herstellung eines Bauteils durch Umformen einer Platine und Vorrichtung zur Durchführung des Verfahrens**

(57) Zusammenfassung: Die Erfindung bezieht sich auf ein Verfahren zur Herstellung eines Bauteils (5) durch Umformen einer beschichteten Platine (1) aus einem Vergütungsstahl, wobei sie vor dem Umformen über eine erste Wärmebehandlung austenitisiert wird, wobei sich darüber hinaus ein Schichtdickenwachstum vollzieht.

Eine Optimierung im Prozessablauf und ein Vermeiden von Ausschussplatinen bei Prozessstörungen ist dadurch erreichbar, dass nach einem raschen Abkühlen die wärmebehandelte Platine (1) zwischengelagert wird, dass unmittelbar vor dem Umformen zum Bauteil (5) die Platine einer erneuten, kurzzeitigen Erwärmung auf Austenitisierungstemperatur unterzogen wird und dass nach erfolgter Gefügeumwandlung das Umformen und Härten der Platine (1) vonstatten geht. Vorzugsweise geschieht die zweite Erwärmung der Platine (1) durch Induktion.



## Beschreibung

**[0001]** Die Erfindung bezieht sich auf ein Verfahren zur Herstellung eines Bauteils durch Umformen einer beschichteten, vorzugsweise aluminiumbeschichteten Platine aus einem Vergütungsstahl nach dem Oberbegriff des Patentanspruches 1. Die Erfindung betrifft des weiteren eine Vorrichtung zur Durchführung des Verfahrens.

**[0002]** Im Stand der Technik sind verschiedene Umformverfahren für Platinen aus einem Vergütungsstahl in Verbindung mit Härtungsprozessen allgemein bekannt. Bei einem sogenannten „direkten“ Umformverfahren wird eine ebene Platine aus einem Vergütungsstahl in einem Ofen, vorzugsweise einem Durchlaufofen, unter Schutzgasatmosphäre austenitisiert. Beispielsweise kann ein Vergütungsstahl in der Qualität 22MnB5 verwendet werden, der für die Austenitisierung mehrere Minuten bei ca. 950°C geglüht wird. Anschließend wird mit einer vorzugsweise automatisierten Transfereinrichtung die heiße austenitisierte ebene Platine in ein für Serienprozesse gekühltes Umform-/Vergütungswerkzeug eingelegt. Dieses Werkzeug ist Bestandteil einer Presse und beim Schließen derselben wird die heiße Platine zu einem Bauteil auf Endform umgeformt und im geschlossenen Werkzeug bei aufgebrachter Schließkraft relativ schnell abgekühlt und dadurch gehärtet. Das gehärtete Bauteil wird aus dem Werkzeug entnommen und, falls es sich um ein unbeschichtetes Blech handelt, in einem Reinigungsschritt beispielsweise durch Sand- oder Kugelstrahlen entzundert (dies ist bei beschichteten Bauteilen entbehrlich, da z. B. aluminisierte Bleche ausreichend Korrosionsschutz bieten und eine Verzunderung verhindert wird). Abschließend folgt ein Fertigungskontur- und Lochbeschnitt des fertig geformten und gehärteten Bauteiles, vorzugsweise mittels Laserschneiden. Auch ein mechanisches Schneiden im sogenannten Pressenverbund ist denkbar.

**[0003]** Während der Wärmebehandlung im Ofen erfährt z. B. eine im Ausgangszustand etwa 25 µm starke Aluminiumbeschichtung ein Schichtdickenwachstum auf etwa 45 µm, wobei sich unmittelbar an das Grundmaterial der Platine angrenzend eine Al-Si-Schicht mit eindiffundiertem Eisen heraus bildet, die eine darüber hinaus sich bildende, relativ harte und spröde AlSi-Schicht trägt, die die eigentliche Korrosionsschutzfunktion erfüllt.

**[0004]** Ein typischer Verfahrensablauf (Warmumformkurve 20) hinsichtlich der Wärmebehandlung der Platine im Zuge ihres Umformens ist beispielsweise in Fig. 1 in einem Zeit-Temperatur-Diagramm dargestellt. Je nach verwendeter Vergütungsstahlqualität, Blechdicke, Ausgangsschichtdicke u.s.w. sind die in Fig. 1 angegebenen Werte natürlich auch gewissen Schwankungen unterworfen (Wärmebehandlungun-

ter-/obergrenze 18, 19). So ist ohne weiteres denkbar, dass die Platine sich über eine Verweildauer von bis zu 30 Minuten im Ofen befindet.

**[0005]** Der verwendete Ofen ist häufig ein sogenannter Durchlaufofen mit Formnestern oder Platinaufnahmen oder ein Rostdurchschubofen mit Rosten, welche die Platine tragen und diese innerhalb von etwa 2 Minuten mittels Gasbrenner auf Austenitisierungstemperatur erwärmen und anschließend mehrere Minuten mittels elektrischer Heizung auf dieser Temperatur halten. Vorzug des Gasbrenners ist eine höhere Leistung, wohingegen elektrische Heizungen besser regelbar sind.

**[0006]** Gemäß Fig. 1 wird also die Platine im Ofen auf eine Solltemperatur von etwa 950° C erhitzt und auf dieser Temperatur gehalten. Die Austenitisierung vollzieht sich bei Temperaturen oberhalb etwa 720° C. Üblicherweise beträgt die Verweildauer im Ofen ca. 9 Minuten, wobei sich die Platine innerhalb der ersten beiden Minuten auf Solltemperatur erwärmt, während in den darauffolgenden etwa 7 Minuten das Umkörnen des Grundmaterials vom kubisch-raumzentrierten Ferrit/Perlit-Gefüge ins kubisch-flächenzentrierte Austenit, das für ein Härten notwendig ist, vonstatten geht. Daneben ist die genannte Zeitspanne vor allem auch wichtig, um ein ausreichendes Anwachsen der AlSi-Schutzschicht zu erreichen.

**[0007]** Insbesondere hinsichtlich minimaler Glüh-temperatur und maximaler Verweildauer der Platine im Ofen gibt es gewisse, mehr oder weniger enge Grenzen, innerhalb derer der Prozess noch Gutteile liefert, d. h., dass die aus dem Ofen entnommenen Platinen überhaupt noch für den Umformprozess und die weitere Verwendung verwertbar sind.

**[0008]** Stellt sich nun im weiteren Ablauf eine Störung ein, sei es bei der Entnahme einer Platine aus dem Ofen und ihrem Weitertransport zum Umform-/Vergütungswerkzeug bzw. innerhalb der Station des Fertigungskontur- und Lochbeschnittes, so kann für die Dauer der Störung keine Platine mehr aus dem Ofen entnommen werden, die maximal zulässige Verweildauer wird in der Regel überschritten und sämtliche im Ofen befindlichen Platinen sind Ausschuss und müssen von daher entsorgt werden.

## Aufgabenstellung

**[0009]** Der Erfindung liegt die Aufgabe zugrunde, das gattungsgemäße Verfahren zur Herstellung eines Bauteiles durch Umformen einer beschichteten Platine aus einem Vergütungsstahl in der Weise weiterzubilden, dass eine Optimierung im Prozessablauf erreichbar ist und dass insbesondere bei eventuellen Prozessstörungen dem kostenträchtigen Anfallen von Ausschussplatinen vorgebeugt werden kann.

**[0010]** Diese Aufgabe wird hinsichtlich des Verfahrens mit den weiteren Merkmalen nach dem Kennzeichen des Patentanspruches 1 und hinsichtlich der Vorrichtung zur Durchführung des erfindungsgemäßen Verfahrens mit den Merkmalen des Patentanspruches 10 gelöst.

**[0011]** Die Vorteile der erfindungsgemäßen Vorgehensweise sind mannigfaltig. So besteht nun keine Abhängigkeit mehr bezüglich der Verweildauer der Platinen innerhalb des Ofens und Ablaufstörungen im Prozess außerhalb des Ofens. Durch die Entkopplung der Abläufe ergibt sich für den eigentlichen Prozess der Platinenumformung ein geringerer Flächenbedarf und ein Weniger an Infrastruktur. Eine Pufferung/Zwischenlagerung der wärmebehandelten Platinen ist möglich, so dass eine Wärmebehandlung, unter anderem zur Beeinflussung der AlSi-Schicht, ohne weiteres auch bereits beim Stahlhersteller bzw. Blechlieferanten erfolgen kann.

#### Stand der Technik

**[0012]** Eine solche ausgelagerte, vorgeschaltete Wärmebehandlung ist an sich bereits bekannt, wie aus der EP 0 946 311 B1 und der DE 102 12 400 C1 hervorgeht. Auch eine Glühbehandlung mittels Induktionserwärmung ist für sich gesehen bereits bekannter Stand der Technik, wie dies im letztgenannten Dokument beispielsweise auch erwähnt ist.

**[0013]** Vorteilhafte Ausgestaltungen und Weiterbildungen der Erfindung sind in den jeweiligen Unteransprüchen beansprucht.

#### Ausführungsbeispiel

**[0014]** Die Erfindung ist nachstehend anhand eines Ausführungsbeispiels näher erläutert. In der Zeichnung zeigen:

**[0015]** Fig. 1 den erfindungsgemäßen Verfahrensablauf zur Herstellung eines Bauteiles durch Umformen einer Platine,

**[0016]** Fig. 2 ein Temperatur- Zeit- Diagramm der ersten Platinen-Wärmebehandlung und

**[0017]** Fig. 3 ein Temperatur- Zeit- Diagramm der zweiten Platinen- Wärmebehandlung.

**[0018]** In Fig. 1 ist schematisch der erfindungsgemäße Verfahrensablauf zur Herstellung eines Bauteiles 5 durch Umformen einer beschichteten Platine 1 aus einem Vergütungsstahl mittels einer hierfür geeigneten Vorrichtung 2 gezeigt. Von dem im aufgerolltem Zustand angelieferten Stahl, einem sogenannten Coil 3, wird mittels eines Werkzeuges 4 der Stahl abgerollt, geglättet und die für das fertige Bauteil 5 notwendige Größe der Platine 1 ausgestanzt

bzw. abgeschnitten. Von dort aus werden die Platinen 1 einer Pufferzone 6 zugeführt. Dieses Zwischenlagern ist allerdings nicht unbedingt erforderlich, vielmehr können die Platinen 1 auch unmittelbar nach Verlassen des Werkzeuges 4 einem ersten Ofen 7 zugeführt werden, in dem sie eine Wärmebehandlung gemäß dem Temperatur- Zeit- Diagramm nach Fig. 2 erfahren. Unmittelbar dem ersten Ofen 7 nachgelagert ist eine Abkühlzone 8, in der die Platinen 1 abgeschreckt werden und die abschließenden Phasen der Wärmebehandlung durchlaufen. Die Abkühlzone 8 verlassend werden die wärmebehandelten Platinen 1 einem Zwischenlager 9 zugeführt.

**[0019]** Der erste Ofen 7 kann vom konstruktiven Aufbau her ein bereits erwähnter Durchlaufofen, ein Karussell-Ofen oder dergleichen sein.

**[0020]** Die einzelnen Phasen der Wärmebehandlung wurden bereits eingangs mit Hinweis auf Fig. 2 erläutert. Das relativ langsam vonstatten gehende Aufheizen auf Solltemperatur sowie die übrige Verweildauer im ersten Ofen 7 zum Herbeiführen der Austenitisierung und zum Verändern der Topographie (Beschichtungsaufbau, Schichtdicke) summiert sich auf eine Gesamt-Verweildauer von etwa 9 Minuten, wobei erfahrungsgemäß eine maximale Verweildauer von 30 Minuten nicht überschritten werden darf, damit die Platine nicht unbrauchbar wird. Der Transport in die Abkühlzone 8 und die dortige Abschreckung der Platine 1 vollzieht sich innerhalb relativ kurzer Zeiträume, während ein restliches Abkühlen auf Raumtemperatur RT im Zwischenlager 9 erfolgen kann. Am Ende der Wärmebehandlung weist die Platine 1 ein Martensitgefüge auf.

**[0021]** Mittels einer geeigneten Transportvorrichtung 10, beispielsweise einem Gelenkarmroboter, werden die Platinen 1 einem Induktionsofen 11 zugeführt, von wo aus sie über eine weitere Transportvorrichtung 12, z. B. wiederum ein Gelenkarmroboter, in ein für Serienprozesse geeignetes, gekühltes Umform-Vergütungswerkzeug 13 eingelegt werden. Diesem ist eine Presseneinrichtung 14 sowie eine Kühlvorrichtung 15 zugeordnet, und beim Schließen der Presseneinrichtung 14 wird die heiße Platine 1 zu einem Bauteil 5 auf Entform umgeformt und im geschlossenen Umform-Vergütungswerkzeug 13 bei aufgebrachtter Schließkraft schnell abgekühlt und dadurch gehärtet. In einem letzten Verfahrensschritt wird jedes Bauteil 5 über eine Transportvorrichtung 16 einer Beschnittvorrichtung 17 zugeführt, wo ein Fertigkontur- und Lochbeschnitt des fertig geformten und gehärteten Bauteiles 5 vorzugsweise mittels Laserschneiden durchgeführt wird. Selbstverständlich kann dies auch über geeignete Beschnittmesser auf mechanischem Wege erfolgen.

**[0022]** Die im Induktionsofen 11 und im nachgeschalteten Umform-Vergütungswerkzeug 13 vonstat-

ten gehende Wärmebehandlung der Platine **1** ist im Temperatur- Zeit- Diagramm gemäß **Fig. 3** anhand der Warmumformkurve **20** und der Wärmebehandlungsunter-/obergenze **18, 19** dargestellt. Sie ist gekennzeichnet durch eine ausgesprochen kurze Verweildauer der Platine im Induktionsofen **11**. Während das Aufheizen auf Solltemperatur (Austenitisierungstemperatur) innerhalb weniger Sekunden (ca. zehn sec.) erfolgt dient eine nachgeschaltete kurze Verweilzeit von etwa zehn Sekunden bis maximal zwei Minuten dazu, eine Gefügeumwandlung vorstatten gehen zu lassen. Eine Änderung von Dicke und Aufbau der Beschichtung ist nicht mehr erforderlich, da dies bereits im ersten Ofen **7** erfolgt ist. Nach demzufolge äußerst kurzer Verweildauer im Induktionsofen **11** kann die Platine bereits dem Umform-Vergütungs-werkzeug **13** zugeführt werden, in dem neben der Umformung das Abschrecken in gleicher Weise (gleicher Verlauf der Warmumformkurve **20**) vorstatten geht wie in der Abkühlzone **8**. Beim Verlassen des Umform-Vergütungs-werkzeuges **13** weist das Bauteil **5** bereits Martensitgefüge auf, die Abkühlung auf Raumtemperatur RT kann beim Weitertransport zur bzw. innerhalb der Beschnittvorrichtung **17** erfolgen.

**[0023]** Auf diese Weise wurde eine eine ursprüngliche Zugfestigkeit von etwa 500 bis 600 N/mm<sup>2</sup> aufweisende Platine **1** in ein eine Zugfestigkeit von etwa 1300 bis 1500 N/mm<sup>2</sup> aufweisendes Bauteil **5** umgeformt.

**[0024]** In vorteilhafter Weiterbildung der Erfindung wäre denkbar, im Induktionsofen **11** die Platine **1** partiell unterschiedlich stark zu erwärmen, mit der Folge, dass, falls dies gewünscht ist, das umgeformte und abgeschreckte Bauteil **5** partiell unterschiedliche Festigkeiten aufweist.

**[0025]** Weiter wäre vorteilhaft möglich, die Platine **1** vor der zweiten Wärmebehandlung (Induktionsofen **11**) zum Beispiel durch Aufschweißen von Verstärkungsblechen (Patches) lokal zu verstärken. Ein solchermaßen gepatchtes Verbundblech könnte dann dem zweiten Ofen und danach dem Umform-Vergütungs-werkzeug **13** zugeführt werden. Dies hätte insgesamt positive Auswirkungen auf die Werkstoffeigenschaften und die Formgenauigkeit.

**[0026]** Das erfindungsgemäße Verfahren ist auch bei Verwendung von Tailored blanks Platinen vorteilhaft anwendbar.

**[0027]** Ein wesentlicher Vorteil der Erfindung ist die Möglichkeit der Entkopplung der einzelnen Verfahrensschritte. So kann die erste Wärmebehandlung im Ofen **7** bereits beim Stahl- bzw. Blechhersteller erfolgen und die so vorbehandelten Platinen **1** können dann dem weiterverarbeitenden Unternehmen (z. B. Fahrzeughersteller) bereitgestellt werden (Zwischenschicht **9**).

**[0028]** In einer weiteren vorteilhaften Weiterbildung der Erfindung ist denkbar, der Transportvorrichtung **10** einen Induktor zuzuordnen bzw. diesen in die Transportvorrichtung **10** baulich zu integrieren, so dass die Wärmebehandlung der Platine **1** während ihres Transportes zum Umform-Vergütungs-werkzeug **13** vorstatten gehen kann. Ein separater Induktionsofen **11** sowie eine diesem nachgeschaltete weitere Transportvorrichtung **12** könnten dadurch entfallen.

### Patentansprüche

1. Verfahren zur Herstellung eines Bauteils durch Umformen einer beschichteten, vorzugsweise aluminiumbeschichteten Platine aus einem Vergütungsstahl, wobei vor dem Umformen in einem ersten Verfahrensschritt die Platine einem ersten Ofen zugeführt und dort austenitisiert wird und wobei die Verweildauer der Platine im ersten Ofen so gewählt wird, dass neben der Gefügeumwandlung ein Schichtdickenwachstum vorstatten geht, gekennzeichnet durch folgende weitere Verfahrensschritte:

- schnelles Abkühlen und anschließendes Zwischenlagern der wärmebehandelten Platine (**1**)
- erneutes, kurzzeitiges Erwärmen der Platine (**1**) in einem zweiten Ofen (**11**) auf Austenitisierungstemperatur unmittelbar vor dem Umformen zum Bauteil (**5**) und
- nach erfolgter Gefügeumwandlung Umformen und Härten der Platine (**1**).

2. Verfahren nach Patentanspruch 1, gekennzeichnet durch eine Verweildauer im ersten Ofen (**7**) zwischen neun Minuten und dreißig Minuten.

3. Verfahren nach Patentanspruch 1, dadurch gekennzeichnet, dass bei der wiederholten Erwärmung der Platine (**1**) auf Austenitisierungstemperatur im zweiten Ofen (**11**) die Verweildauer so gewählt wird, dass lediglich eine Gefügeumwandlung, aber kein Schichtdickenwachstum mehr statt findet.

4. Verfahren nach Patentanspruch 3, gekennzeichnet durch eine Verweildauer der Platine (**1**) im zweiten Ofen (**11**) von zehn Sekunden bis zweieinhalb Minuten.

5. Verfahren nach Patentanspruch 1, dadurch gekennzeichnet, dass die Erwärmung der Platine (**1**) im ersten Ofen (**7**) auf Elektro- oder Gas-Basis erfolgt, während die Erwärmung im zweiten Ofen (**11**) durch Induktion vorstatten geht.

6. Verfahren nach Patentanspruch 1, dadurch gekennzeichnet, dass die erste Erwärmung beim Stahl- bzw. Blechhersteller erfolgt, während die zweite Wärmebehandlung beim weiterverarbeitenden Unternehmen, z. B. Fahrzeughersteller, vorstatten geht.

7. Verfahren nach Patentanspruch 1, dadurch ge-

kennzeichnet, dass während der zweiten Wärmebehandlung die Platine (1) über ihre Oberfläche unterschiedlich stark erwärmt wird.

8. Verfahren nach Patentanspruch 1, dadurch gekennzeichnet, dass die Platine (1) vor dem erneuten Erwärmen im zweiten Ofen (11) durch Aufbringen von mindestens einem Verstärkungsblech lokal verstärkt wird.

9. Verfahren nach Patentanspruch 1, gekennzeichnet durch die Verwendung eines Tailored blanks als Platine (1).

10. Vorrichtung zur Durchführung des Verfahrens nach einem der Patentansprüche 1–9, gekennzeichnet durch

- ein Werkzeug (4) zum Herstellen von Platinen (1) aus einem Coil (3),
- einen ersten Ofen (7) zur ersten Wärmebehandlung einschließlich Herbeiführen eines Schichtdickenwachstums der Platinen (1),
- eine Abkühlzone (8) für die Platinen (1),
- ein Zwischenlager (9) für die Platinen (1),
- einen zweiten Ofen (11) zur erneuten Wärmebehandlung der Platinen (1),
- ein Umform-Vergütungswerkzeug (13) mit einer Presseneinrichtung (14) und einer Kühlvorrichtung (15) und
- eine Beschnittvorrichtung (17) zur Herstellung eines Fertigkontur- und Lochbeschnittes.

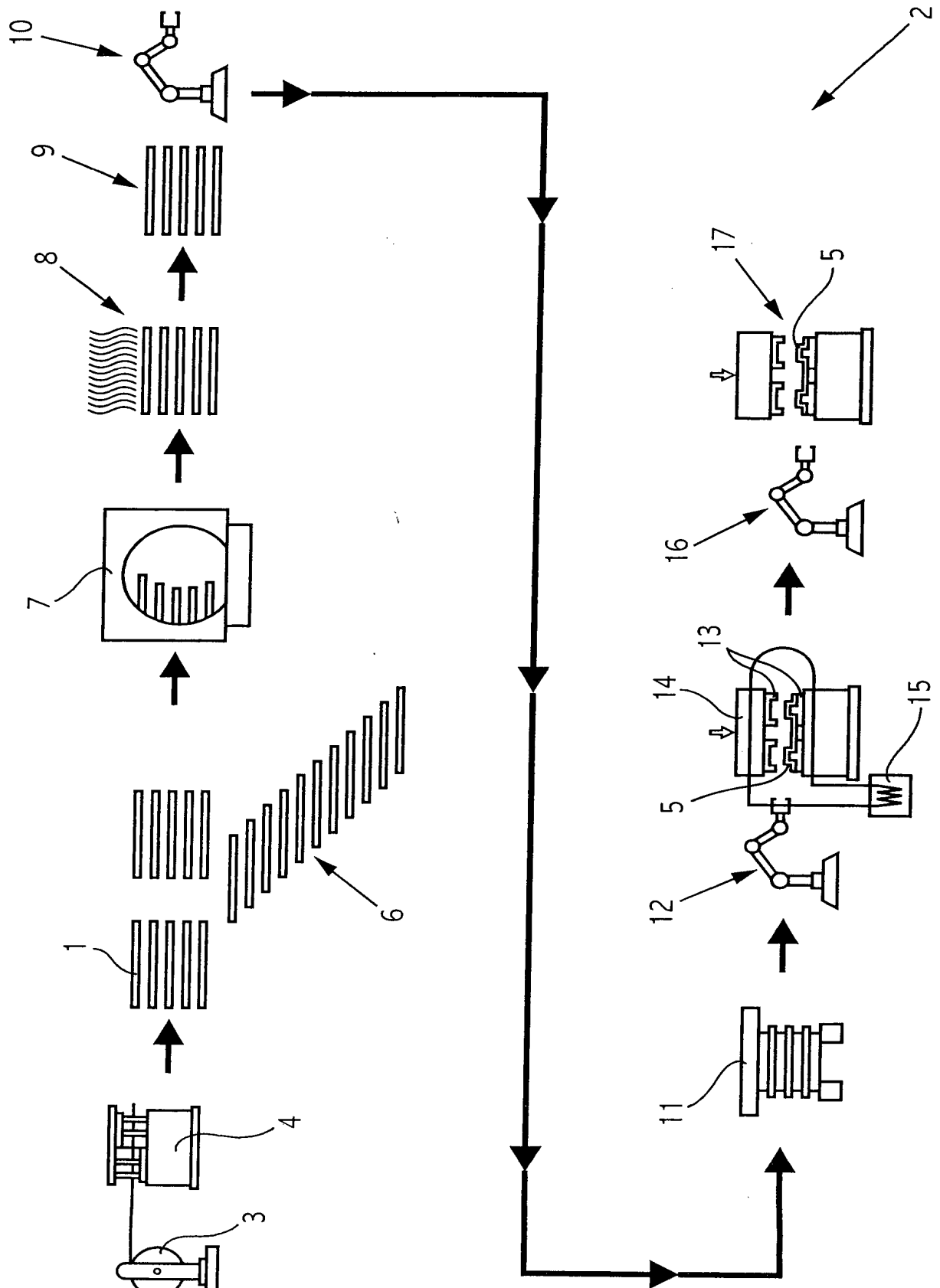
11. Vorrichtung nach Patentanspruch 10, gekennzeichnet durch einen ersten Ofen (7) auf Elektro- und/oder Gas-Basis und einen Induktionsofen (11) zur zweiten Wärmebehandlung.

12. Vorrichtung nach Patentanspruch 11, dadurch gekennzeichnet, dass ein Induktor in eine Transportvorrichtung (10) integriert ist, die zwischen Zwischenlager (9) und Umform-Vergütungswerkzeug (13) angeordnet ist.

13. Vorrichtung nach Patentanspruch 10, dadurch gekennzeichnet, dass zwischen der Abkühlzone (8) und dem zweiten Ofen (11) eine Station zum Aufbringen, insbesondere Aufschweißen, von mindestens einem Verstärkungsblech auf die Platine (1) angeordnet ist.

Es folgen 3 Blatt Zeichnungen

FIG.1



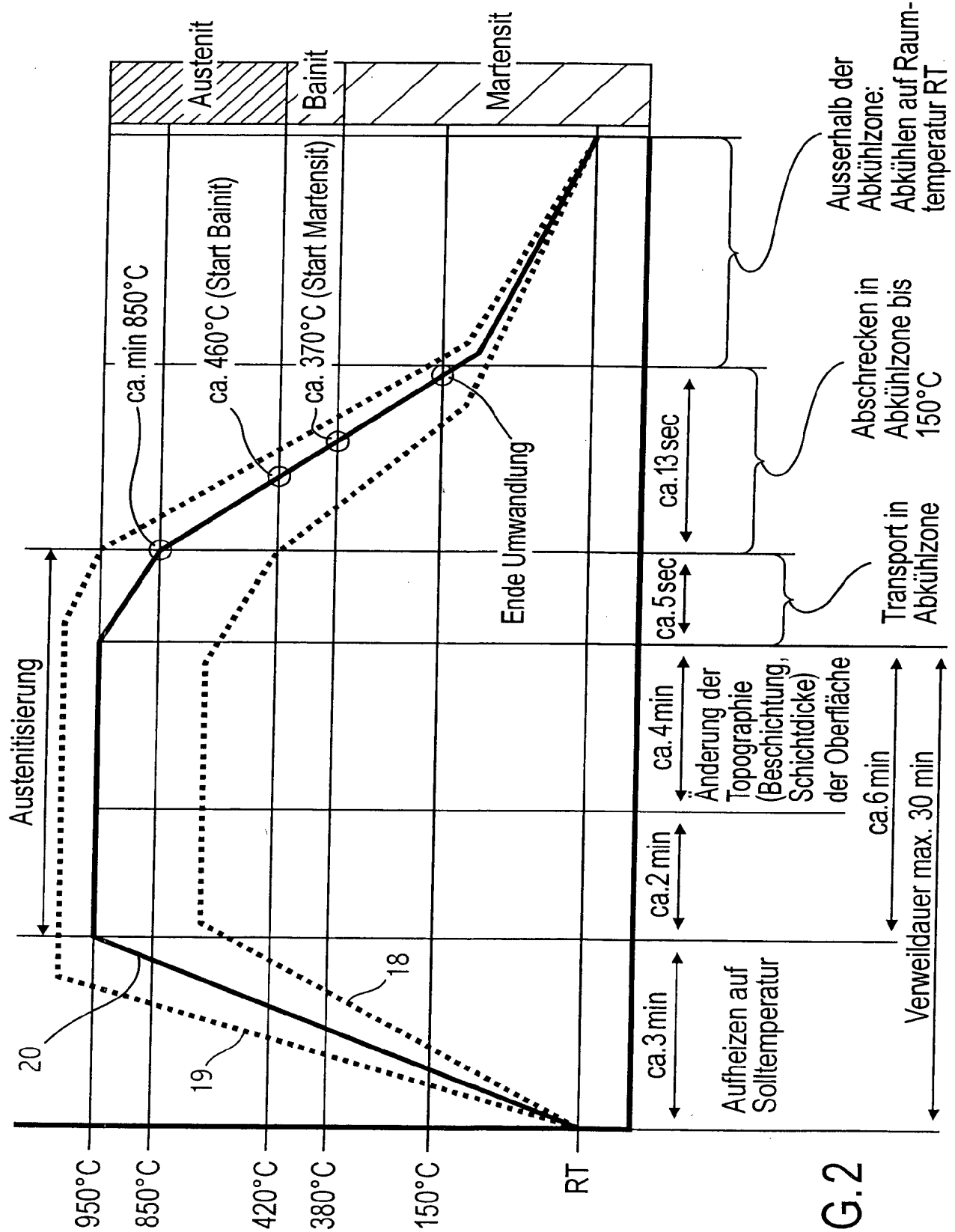


FIG. 2

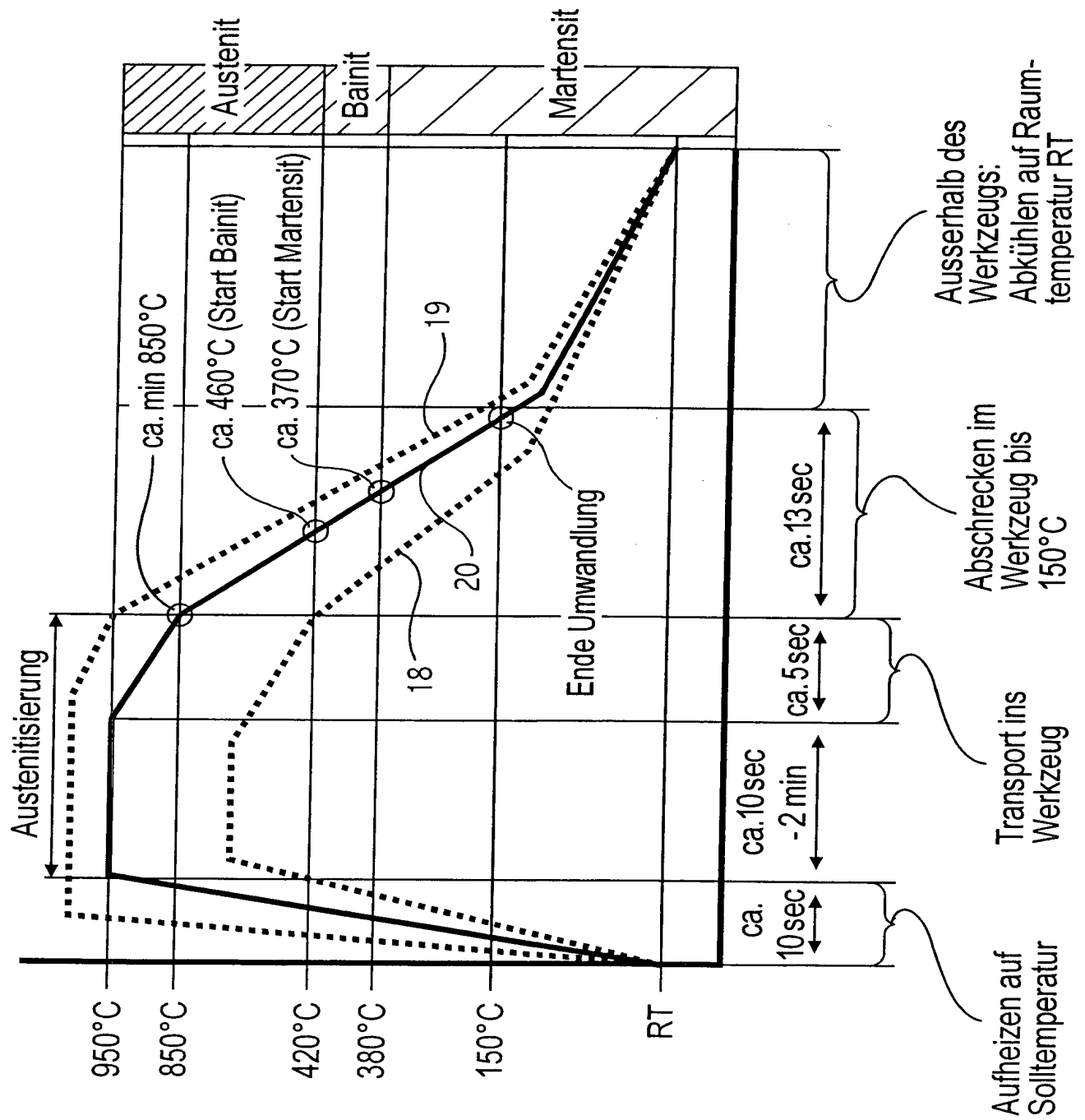


FIG. 3

(19)



(11)

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(12)

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**B21D 22/02** <sup>(2006.01)</sup> **C21D 8/02** <sup>(2006.01)</sup>  
**C22C 38/32** <sup>(2006.01)</sup> **C22C 38/38** <sup>(2006.01)</sup>  
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**WO 2009/090555 (23.07.2009 Gazette 2009/30)**

(54) **PROCESS FOR MANUFACTURING STAMPED PRODUCTS, AND STAMPED PRODUCTS  
PREPARED FROM THE SAME**

PROZESS FÜR DIE HERSTELLUNG DER STANZLINGEN UND STANZLINGE VORBEREITET AUS  
DEN SELBEN

PROCÉDÉ DE FABRICATION DE PRODUITS ESTAMPÉS ET PRODUITS ESTAMPÉS PRÉPARÉS  
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## Description

### FIELD OF THE INVENTION

**[0001]** The present invention relates to methods of manufacturing hot stamped products prepared from coated steels and to various uses of the invention products such as in spot welding.

### BACKGROUND OF THE INVENTION

**[0002]** In recent years the use of coated steels in hot-stamping processes for the shaping of parts has become important, especially in the automotive industry. Fabrication of such parts or products may include the successive following main steps:

- Coating of steel strips or sheets,
- Trimming or cutting for obtaining blanks
- Heating the blanks in order to obtain alloying of the steel substrate with the pre-coating, as well as the austenitizing of the steel
- Hot forming followed by rapid cooling of the part in order to obtain predominantly martensitic structures

This is illustrated for example by U.S. 6,296,805, incorporated herein by reference.

Thanks to an alloying of the pre-coating with the steel substrate, which has the effect of creating intermetallic alloys with high melting temperature, the blanks having such coating may be heated in a temperature range where austenitizing of the metallic substrate takes place, allowing further hardening by quenching.

**[0003]** Heat treatments of the blanks in view of the intermetallic alloying of the coating and austenitizing of the substrate are most frequently performed in furnaces. The thermal cycles experienced by the blanks include first a heating phase whose rate is a function of parameters such as furnace temperature settings, travelling speed, blank thickness, heating process, and coating reflectivity. After this heating phase, thermal cycles generally include a holding phase, whose temperature is the regulation temperature of the furnace.

**[0004]** Parts or products obtained after heating, hot stamping and rapid cooling display very high mechanical resistance and may be used for structural applications, for example for automotive industry applications. These parts must be frequently welded with others and high weldability is required. This means that:

- The welding operation should be performable in a sufficiently wide operating range in order to guarantee that an eventual drift of the nominal welding parameters has no incidence on weld quality. For resistance welding, which is very common in the automotive industry, an operating welding range is defined by the combination of parameters: welding current intensity  $I$  and force  $F$  applied of the parts during

welding being among the most important. A proper combination of these parameters helps to ensure that insufficient nugget diameter is not obtained (caused by too low intensity or too low force) and that no weld expulsion occurs.

- The welding operation should also be performed in such a way that high mechanical resistance is obtained in the weld. This mechanical resistance may be evaluated by tests such as by shear-tensile tests or cross-tensile tests.

EP1380666 discloses also a process including hot stamping of Al-coated steel sheets for the fabrication of welded structural members. But the weldability needs to be further improved.

There remains a need for a process making possible to prepare stamped parts or products which are very suitable to spot welding, which are easy to paint and which display good corrosion resistance.

### SUMMARY OF THE INVENTION

**[0005]** The inventors have discovered that certain coated steels in which a base steel strip or sheet is at least partially coated (sometimes termed "pre-coated," this prefix indicating that a transformation of the nature of the pre-coating will take place during heat treatment before hot stamping or forming) on at least one side with a coating of either aluminum or an aluminum alloy and in which the coating has a defined thickness, are conveniently formed into shaped parts after heating in particular conditions, and thereby display particular improved weldability.

**[0006]** The inventors have also discovered that particular good weldability of aluminized and hot stamped parts is associated with a special succession of coating layers on the parts, proceeding from steel substrate outwards, and a controlled fraction of porosities in these layers.

**[0007]** The inventors have also discovered that this special disposal of layers is associated to specific heating conditions.

### OBJECTS OF THE INVENTION

**[0008]** It is an object of the present invention to provide novel hot stamped parts which are prepared from a pre-coated steel.

**[0009]** It is another object of the present invention to provide novel articles of manufacture, such as a motor vehicle, which contain such stamped parts.

**[0010]** It is another object of the present invention to provide novel methods of making stamped parts displaying high weldability.

**[0011]** These and other objects, which will become apparent during the following detailed description.

# BRIEF DESCRIPTION OF THE DRAWINGS

[0012]

Figure 1 shows conditions of furnace temperature as a function of the total dwell time in the furnace for sheets of total thicknesses of from 0.7-1.5 mm and 1.5-3 mm that provide particularly favorable coatings for welding.

## DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0013] The invention is implemented with certain pre-coated steel strips, which comprise a strip of base steel and a pre-coating of aluminum or an aluminum alloy on at least a part of one side of the strip of the base steel. For many applications, the strip or sheet of base steel may comprise any type of steel which may be coated with either aluminum or an aluminum alloy. However, for certain applications, such as a structural part of an automobile, it is preferred that the strip of base steel comprises a steel for providing ultra high strength on the part, higher than 1000MPa. In such cases, it is particularly preferred that the strip of base steel comprises a boron steel.

[0014] The strip can derive, by reason of its processing, from a hot-rolling mill, and possibly may be cold-rolled again depending on the final thickness desired. Preferred thicknesses are 0.7 to 3 mm. Typically, the strip of base steel will be stored and transported in the form of a coil both before and after the formation of the coating.

[0015] An example of a preferred steel for the strip of base steel is one having the following composition by weight:

0.10% < carbon < 0.5%

0.5% < manganese < 3%

0.1% < silicon < 1%

0.01% < chromium < 1%

nickel < 0.1%

copper < 0.1%

titanium < 0.2%

aluminum < 0.1%

phosphorus < 0.1%

sulfur < 0.05%

0.0005% < boron < 0.010%,

the remainder comprising, consisting essentially of, or consisting of iron and impurities inherent in processing. Use of such a steel provides a very high mechanical resistance after thermal treatment and the aluminum-based coating provides a high resistance to corrosion.

[0016] Particularly preferably, the composition by weight of the steel in the strip of base steel is the following:

0.15% < carbon < 0.25%

0.8% < manganese < 1.8%

0.1% < silicon < 0.35%

0.01% < chromium < 0.5%

nickel < 0.1%

copper < 0.1%

titanium < 0.1%

aluminum < 0.1%

phosphorus < 0.1%

sulfur < 0.05%

0.002% < boron < 0.005%,

the remainder comprising, consisting essentially of, or consisting of iron and impurities inherent in processing. An example of preferred commercially available steel for use in the strip of base steel is 22MnB5.

**[0017]** Chromium, manganese, boron and carbon may be added, in the composition of the steel according to the invention, for their effect on hardenability. In addition, carbon makes it possible to achieve high mechanical characteristics thanks to its effect on the hardness of the martensite.

**[0018]** Aluminum is introduced into the composition, to perform deoxidation in the liquid state and to protect the effectiveness of the boron.

**[0019]** Titanium, the ratio of the content of which with respect to the nitrogen content should be in excess of 3.42, is introduced for example in order to prevent combining of the boron with the nitrogen, the nitrogen being combined with titanium.

**[0020]** The alloying elements, Mn, Cr, B, make possible a hardenability allowing hardening in the stamping tools or the use of mild hardening fluids limiting deformation of the parts at the time of thermal treatment. In addition, the composition according to the invention is optimized from the point of view of weldability. Additions of Ni and Cu, up to 0.1%, may also be performed.

**[0021]** The steel may undergo a treatment for globularization of sulfides performed with calcium, which has the effect of improving the fatigue resistance of the sheet.

**[0022]** The strip of base steel is coated (or pre-coated, this prefix indicating that a transformation of the nature of the pre-coating will take place during heat treatment before stamping) with either aluminum or an aluminum alloy, preferably with hot-dip. A typical metal bath for an Al-Si coating generally contains in its basic composition by weight, from 8% to 11 % silicon, from 2% to 4% iron, the remainder being aluminum or aluminum alloy, and impurities inherent in processing. Silicon is present in order to prevent the formation of a thick iron-metallic intermetallic layer which reduces adherence and formability. Other alloying elements useful with aluminum herein include iron, and calcium, between 15 and 30ppm by weight, including combinations of two or more thereof with aluminium. Typical composition of Al-Si coating is: Al-9,3%Si-2,8%Fe. Invention coatings are not limited to these compositions, however.

While not bound by a particular theory of operation, the inventors believe that several of the benefits of the invention are first related to a specific range of pre-coating thickness tp of 20 to 33 micrometers :

- For a pre-coating thickness less than 20 micrometers, the alloyed layer which is formed during the heating of the blank has an insufficient roughness. Thus, the adhesion of subsequent painting is low on

this surface, and the corrosion resistance is decreased.

- If the pre-coating thickness is more than 33 micrometers at a given location on a sheet, the risk is that the difference of thickness between this location and some other locations where the pre-coating is thinner, becomes too important, and that alloying during the heating of the blank becomes uneven. The inventors have also shown that the control of the pre-coating thickness in the narrow range presented above, contributes to form coatings after alliation whose thickness is also controlled in a precise range. This is also a factor for ensuring that the range of resistance welding parameters applied on parts after alliation is not subject to variability.

The pre-coated steel sheets or strips are then cut into blanks, and submitted to heat treatments in furnace prior to hot stamping, in order to obtain products or parts. The inventors have discovered that very good welding properties are achieved if the coating obtained on parts or products made out of blanks having undergone intermetallic alloying, austenitizing and hot stamping, displays distinctive features. It must be pointed out that this coating is different from the initial pre-coating, since the thermal treatment causes an alloying reaction with the steel substrate which modifies both the physicochemical nature and the geometry of the pre-coating : in this regard, the inventors have discovered that particularly good weldability of aluminized and hot stamped parts is associated with the following succession of coating layers on the parts, proceeding from steel substrate outwards :

- (a) Interdiffusion layer,
- (b) Intermediate layer,
- (c) Intermetallic layer,
- (d) Superficial layer

The inventors have also discovered that particular good weldability is obtained with a limited quantity of porosities in the coating layers, as will be detailed below.

In a preferred embodiment, the layers are as follows:

- (a) Interdiffusion layer, preferably with medium hardness (e.g., HV50g between 290 and 410, HV50g designating the hardness measured under a load of 50 grams) In a preferred embodiment this layer has the following composition, by weight: 86-95%Fe, 4-10%Al, 0-5%Si
- (b) Intermediate layer (HV50g around 900 - 1000 e.g., +/- 10%) In a preferred embodiment this layer has the following composition, by weight: 39-47% Fe, 53-61%Al, 0-2%Si
- (c) Intermetallic layer, with hardness HV50g around 580-650, e.g., +/- 10%) In a preferred embodiment this layer has the following composition, by weight: 62-67%Fe, 30-34 %Al, 2-6%Si
- (d) Superficial layer (HV50g around 900 - 1000 e.g.,

+/- 10%)) In a preferred embodiment this layer has the following composition, by weight : 39-47% Fe, 53-61%Al, 0-2%Si

In a preferred embodiment the total thickness of layers (a) to (d) is greater than 30 micrometers.

In another preferred embodiment, the thickness of layer (a) is less than 15 micrometers.

**[0023]** The inventors have discovered that high weldability is especially obtained when layers (c) and (d) are essentially continuous; the character of essential continuity of these layers is defined in the following manner: the layers may be fully continuous. But they may be fragmented in some areas due to layer parts coming from lower or upper levels. According to the invention, this fragmentation must be limited, i.e. layers (c) and (d) must occupy at least 90% of their respective level. High weldability is obtained when less than 10% of layer (c) is present at the extreme surface of the part. Without being bound by a theory, it is thought that this particular layer disposal, in particular layer (a) and layers (c) and (d) influence the resistivity of the coating both by their intrinsic characteristics and by the effect of roughness. Thus, current flow, heat generation at the surfaces, and nugget formation in the initial stage of spot welding are affected by this particular arrangement.

This favorable layer disposition is obtained for example when aluminum- or aluminum alloy pre-coated steel sheets, whose thickness range from, e.g., 0.7 to 3mm, are heated for 3 to 13 minutes (this dwell time includes the heating phase and the holding time) in a furnace without special atmosphere heated to a temperature of 880 to 940°C. The invention does not require a furnace with a controlled atmosphere. Other conditions leading to such favorable layer dispositions are found in Figure 1 and below.

Particularly preferred conditions are:

- for thicknesses of 0.7-1.5mm
  - 930°C, from 3 minutes up to 6 minutes;
  - 880°C, from 4 minutes 30 seconds up to 13 minutes
- for thicknesses of 1.5 to 3 mm
  - 940°C, from 4 minutes up to 8 minutes;
  - 900°C, from 6 minutes 30 seconds up to 13 minutes

For sheets of total thicknesses greater or equal to 0.7mm, and less than or equal to 1.5mm, the preferred treatment conditions : (furnace temperature, total dwell time in the furnace) are illustrated in figure 1 by conditions lying within the limits of diagram "ABCD" For sheets of total thicknesses greater than 1.5mm, and less than or equal to 3mm, the preferred treatment conditions: (furnace temperature, total dwell time in the furnace) are illustrated in

figure 1 by diagram "EFGH".

The heating rate  $V_c$  is comprised between 4 and 12°C/s for producing a favorable alloyed layer disposition.  $V_c$ , depending in particular of furnace settings, is defined as the mean heating rate between 20 and 700°C experienced by the pre-coated steel blank in the preheated furnace. The inventors have discovered that the control of  $V_c$  in this particular range allows to influence the nature and the morphology of the alloyed layers which are formed. It is here underlined that the heating rate  $V_c$  is different from the mean heating rate, which is the heating rate between room temperature and furnace holding temperature.

The inventors have discovered in a surprising manner that special heating conditions are particularly favourable for the formation of alloyed layers, leading to less porosities formation. Without being bound by a theory of the invention, it is believed that the formation of the preferred alloyed layers takes place in a particular temperature range due to the particular kinetics of alliation in this range: in this respect, it has been discovered that the control of the heating rate in the particular temperature range between 500 and 700°C (designated here as  $V_c'$ ) is especially important and that the value of  $V_c'$  has to be comprised between 1.5 and 6°C/s.

When  $V_c'$  is lower than 1.5°C/s, there is a risk that the kinetics of oxidation, resulting from the interaction of oxygen of the furnace atmosphere with the pre-coating surface, competes with the kinetics of alliation between the steel substrate and the pre-coating. Thus, the desired alloyed layer disposal is not obtained. Furthermore slow heating rate  $V_c'$  causes a too high quantity of porosities in the coating.

When  $V_c'$  is higher than 6°C/s, the intermetallic layer (c) has a tendency to be present in more than 10% at the extreme surface of the part, thus reducing weldability. When  $V_c$  is comprised between 1.5 and 6°C/s, the character of essential continuity of layers (c) and (d) is fully ensured.

Without being bound by a theory, it is thought that the porosity formation and its influence on weldability, may be explained as follows:

- Porosities appear mainly during the interdiffusion of pre-coating with the steel substrate, due to the difference of diffusion fluxes, This implies a flux of vacancies with a creation of Kirkendal defects. This manifestation of vacancies under the form of porosities appears to be optimized when heating rate  $V_c'$  is comprised between 1.5 and 6°C/s.

During spot welding of welding products, current flows initially around the porosities, which collapse progressively due to pressure and temperature elevation. Thus, the current flows through a coating whose some properties may change discontinuously, which in turn may lead to increased sparking and splashings during the welding

operation.

Increased spot weldability is observed when the coating resulting from interdiffusion contains, in surfacic fraction, less than 10% of porosities. For a given area representative of the coating, this fraction is the total surface occupied by porosities, as referred to the area of the coating.

Special good weldability is experienced when the superficial layer has a controlled compacity, which means that the superficial layer (d) contains less than 20% porosities: this fraction is the surface of porosities in the superficial layer (d), as referred to the area of this superficial layer. A special advantage arises from pre-coatings whose thickness is comprised between 20 and 33 micrometers, since this thickness range yields favorable layer disposal, and since the homogeneity of the pre-coating thickness is associated to an homogeneity of the coating formed after alliation treatment.

**[0024]** Heated blanks are thereafter transferred from the furnace to a die, hot stamped in a press to obtain a part or product, and cooled at a rate  $V_r$  of more than 30°C/s. The cooling rate  $V_r$  is defined here as the mean rate between the exit of the heated blank from the furnace, down to 400°C. In these conditions, austenite formed at high temperature mainly transform into martensitic or martensitic-bainitic structures with high strength.

In a preferred embodiment, the elapsed time between the exit of the heated blank and the introduction of the blank in the hot stamping press is not more than 10 seconds. Otherwise, a partial transformation from austenite is susceptible to appear: if obtaining a full martensitic structure is desired, the transfer time between the exit of the furnace and stamping should be less than 10s.

The coating obtained has in particular the function of protecting the basic sheet against corrosion in various conditions. At the time of thermal treatment performed on a finished part or at the time of a hot-shaping process, the coating forms a layer having a substantial resistance to abrasion, wear, fatigue, shock, as well as a good resistance to corrosion and a good capacity for painting and gluing. The coating makes it possible to avoid different surface-preparation operations such as for steel sheets for thermal treatment not having any coating.

The thermal treatment applied at the time of a hot-forming process or after forming makes it possible to obtain high mechanical characteristics which can exceed 1500 MPa for mechanical resistance and 1200 MPa for yield stress. The final mechanical characteristics are adjustable and depend in particular on the martensite fraction of the structure, on the carbon content of the steel and on the thermal treatment.

The invention also concerns the use of a hot-rolled steel sheet which then can be cold-rolled and coated, for structural and/or anti-intrusion or substructure parts for a land motor vehicle, such as, for example, a bumper bar, a door reinforcement, a wheel spoke, etc.

The present invention will now be further described by

way a certain exemplary embodiments which are not intended to be limiting.

## EXAMPLES

### [0025]

i) - Conditions according to the invention: in an example of implementation, a cold rolled steel sheet, 1.2 mm thick, has been fabricated: it contains by weight: 0.23 % carbon, 1.25% manganese, 0.017% phosphorus, 0.002% sulfur, 0.27% silicon, 0.062% aluminum, 0.021% copper, 0.019% nickel, 0.208% chromium, 0.005% nitrogen, 0.038% titanium, 0.004% boron, 0.003% calcium. The sheet has been pre-coated with an aluminum-based alloy with composition 9.3% silicon, 2.8% iron, the remainder being aluminum and unavoidable impurities. The thickness on each side of the sheet was controlled to be within the range (20-33) micrometers.

The sheets were afterwards cut into blanks which were heated at 920°C for 6mn, this time including the heating phase and the holding time. Heating rate  $V_c$  between 20 and 700°C was 10°C/s. The heating rate  $V_c$  between 500 and 700°C was 5°C/s. No special control of furnace atmosphere was performed. The blanks were transferred from the furnace to a press in less than 10s, hot stamped and quenched in order to obtain full martensitic structures.

The parts obtained after hot-stamping are covered by a coating, 40 micrometers thick, which has a four layer structure. Starting from the steel substrate, the layers are the following:

- (a) Interdiffusion layer or intermetallic layer, 17 micrometers thick. This layer is itself composed of two sub-layers. Hardness HV50g ranges from 295 to 407, and the mean composition is, by weight: 90%Fe, 7%Al, 3%Si.
- (b) Intermediate layer, 8 micrometers thick. This layer has a hardness of 940HV50g and a mean composition, by weight: 43%Fe, 57%Al, 1%Si,
- (c) Intermetallic layer, 8 micrometers thick, displaying a hardness of 610HV50g, a mean composition of, by weight: 65%Fe, 31%Al, 4%Si
- (d) Superficial layer, 7 micrometers thick, 950 HV50g, with a mean composition of, by weight: 45%Fe, 54%Al, 1%Si

Layers (c) and (d) are quasi-continuous, i.e. occupying at least 90% of the level corresponding to the considered layer. In particular, layer (c) does not reach the extreme surface except very exceptionally. Anyway, this layer (c) occupies less than 10% of the extreme surface.

A small number of porosities were observed in the coating, their surfacic fraction in this coating being lower than 10%. The surfacic fraction of porosities

in the superficial layer (d) is lower than 20%.

ii) Conditions of reference: blanks with the same base material and pre-coating were furnace-heated in different conditions: The blanks were heated to 950°C for 7 minutes, this time including the heating phase. Heating rate  $V_c$  was 11°C/s. Heating rate  $V_c'$  between 500 and 700°C was 7°C/s. These conditions correspond to a degree of alloying which is more important than in conditions (i)

- In this coating, the intermetallic layer (c), is not continuous and appears as to be scattered within the coating. About 50% of this layer is present at the extreme surface of the part. The interdiffusion layer, 10 micrometers thick in contact with the steel substrate is thinner than in the previous case. Moreover the porosities are much more numerous than in condition (i) since their surfacic fraction in the coating exceeds 10%. These porosities are especially more numerous in the superficial layer (d) wherein the surfacic fraction exceeds 20%.

Resistance spot welding was performed in the two situations i) and ii):

- (i): Coating with quasi-continuous layers (c) and (d), layer (c) occupying less than 10% of the extreme surface, and low surfacic fraction of porosities
- (ii) : Coating with mixed and discontinuous layers, layer (c) occupying more than 10% of the extreme surface, and higher surfacic fraction of porosities

Resistance spot welding was performed by superposing two parts and joining them in the following conditions:

- Squeeze force and welding force : 4000 N
- Squeeze time: 50 periods
- Welding and holding time: 18 periods respectively

In each condition, the suitable intensity range was determined for obtaining:

- No sputter during welding
- Acceptable nugget size.

Tensile tests were also performed to assess the weldability range.

- For the condition i), the weldability range, expressed in terms of current intensity, is 1,4kA. For the condition ii) the weldability range is extremely small. The higher fraction of porosities and the layer disposal are associated to sparks and coating splashing.

Thus, it may be seen that the coating according to the invention, yields much more satisfactory results.

[0026] While the above description is clear with regard to the understanding of the invention, the following terms as used in the following list of preferred embodiments and claims have the following noted meanings in order to avoid any confusion:

pre-coating : - the material (Al or Al alloy) coated on or located on at least a portion of the strip or sheet, etc., of base steel to form a pre-coating/base composite, the composite not having been subjected to an alliation reaction between the coated Al or Al alloy material and base steel

alliation or alloying : - a reaction between the pre-coating and base steel, to produce at least one intermediate layer different in composition from both the base steel and the pre-coating. The alliation reaction happens during the heat treatment immediately preceding hot stamping. The alliation reaction affects the total thickness of the pre-coating. In a highly preferred embodiment the alliation reaction forms the following layers: (a) interdiffusion, (b) intermediate, (c) intermetallic, and (d) superficial as described above;

pre-coated steel : - the pre-coating/base composite, not having been subjected to an alliation reaction between the coated material and base steel;

coating : - the pre-coating after having been subjected to an alliation reaction between the pre-coating and base steel. In a highly preferred embodiment the coating comprises layers (a) interdiffusion, (b) intermediate, (c) intermetallic, and (d) superficial described above;

coated steel or product : - the pre-coated steel or product that has been subjected to an alliation reaction between the pre-coating and base steel. In a highly preferred embodiment the coated steel is a strip or sheet, etc., of base steel having thereon an invention coating comprising layers (a) interdiffusion, (b) intermediate, (c) intermetallic, and (d) superficial described above;

blank : - a shape cut from a strip.

product : - a hot stamped blank

The above written description of the invention provides a manner and process of making and using it such that any person skilled in this art is enabled to make and use the same.

## Claims

1. A process for making a hot stamped coated steel blank, comprising:

- pre-coating a steel strip or sheet with aluminium- or aluminium alloy, by hot dip of said steel strip or sheet having a first side and a second side, in an aluminium or aluminium alloy bath, the thickness  $t_p$  of the said pre-coating being from 20 to 33 micrometers at every location on said first and second sides of said strip or sheet, then

- cutting said pre-coated steel strip or sheet to obtain a pre-coated steel blank, then

- heating said aluminum- or aluminum alloy pre-coated steel blank in a furnace preheated to a temperature and during a time defined by diagram ABCD of figure 1 if thickness of said sheet is greater than or equal to 0.7mm and less than or equal to 1.5mm, and by diagram EFGH of figure 1 if thickness of said sheet is greater than 1.5mm and less than or equal to 3mm, at a heating rate  $V_c$  between 20 and 700°C comprised between 4 and 12°C/s, and at a heating rate  $V_c'$  between 500 and 700°C comprised between 1.5 and 6°C/s, to obtain a heated blank; then

- transferring said heated blank to a die; then

- hot stamping said heated blank in said die, to thereby obtain a hot stamped steel blank, then

- cooling said hot stamped steel blank at a mean rate  $V_r$  between the exit of said heated blank from the furnace, down to 400°C, of at least 30°C/s.

2. A process according to claim 1, wherein the elapsed time between said heated blank exits said furnace and said stamping commences is not more than 10 seconds

3. A hot stamped coated steel blank, which comprises:

(a) a strip of base steel having a first side and a second side; and

(b) a coating on at least one of said first side of said strip of base steel and said second side of said strip of base steel, wherein:

(i) said coating results from the interdiffusion between said base steel, and aluminium or aluminium alloy pre-coating,

(ii) said coating comprises, proceeding from base steel outwards,

- (a) Interdiffusion layer
- (b) Intermediate layer
- (c) Intermetallic layer
- (d) Superficial layer

(iii) said coating contains, in surfacic fraction, less than 10% of porosities

and wherein the said layers (c) and (d) are quasi-continuous by occupying at least 90% of their respective level and wherein less than 10% of layer (c) is present at the extreme surface of said hot stamped coated steel blank

4. A hot stamped coated steel blank according to claim 3, wherein said superficial layer (d) contains, in surfacic fraction, less than 20% of porosities

5. A hot stamped coated steel blank according to claims 3 or 4, wherein said coating has a thickness greater than 30 micrometers

6. A hot stamped coated steel blank according to any of claims 3 to 5, wherein said layer (a) has a thickness less than 15 micrometers

7. A hot stamped coated steel blank according to any of the claims 3 to 6, wherein the steel composition in the strip comprises the following components by weight based on total weight:

$0.15\% < \text{carbon} < 0.5\%$

$0.5\% < \text{manganese} < 3\%$

$0.1\% < \text{silicon} < 0.5\%$

$0.01\% < \text{chromium} < 1\%$

$\text{nickel} < 0.1\%$

$\text{copper} < 0.1\%$

$\text{titanium} < 0.2\%$

$\text{aluminum} < 0.1\%$

$\text{phosphorus} < 0.1\%$

$\text{sulfur} < 0.05\%$

0.0005%<boron<0.08%,

the balance being iron and unavoidable impurities. 5

8. A hot stamped coated steel blank according to any of the claims 3 to 6, wherein the steel composition in the strip comprises the following components by weight based on total weight:

0.20%<carbon<0.5%

0.8%<manganese<1.5% 15

0.1%<silicon<0.35% 20

0.01%<chromium<1%

nickel<0.1% 25

copper<0.1% 30

titanium<0.1%

aluminum<0.1% 35

phosphorus<0.05%

sulfur<0.03% 40

0.0005%<boron<0.01%, 45

the balance being iron and unavoidable impurities.

9. A hot stamped coated steel blank according to any of the claims 3 to 8, wherein the aluminum or aluminum alloy pre-coating comprises from 8% to 11% silicon by weight, from 2% to 4% iron by weight, the remainder being aluminum and impurities inherent in processing. 50

10. Use of a hot stamped coated steel blank according to any of the claims 3 to 9 for the manufacturing of a land motor vehicle 55

11. Use of a hot stamped coated steel blank manufactured according to a process according to any of the claims 1 or 2, for the manufacturing of a land motor vehicle.

## Patentansprüche

1. Verfahren zum Herstellen eines heißgestanzten beschichteten Stahlabschnitts, umfassend:

- Vorbeschichten eines Stahlstreifens oder-blechs mit Aluminium oder einer Aluminiumlegierung durch Schmelztauchen des eine erste und eine zweite Seite aufweisenden Stahlstreifens oder-blechs in ein Aluminium- oder Aluminiumlegierungsbad, wobei die Dicke tp der Vorbeschichtung von 20 bis 33 µm an jeder Stelle der ersten und zweiten Seite des Streifens oder Blechs ist, dann
- Schneiden des vorbeschichteten Stahlstreifens oder-blechs, um einen vorbeschichteten Stahlabschnitt zu erhalten, Heizen des vorbeschichteten Aluminium- oder Aluminiumlegierungs-Stahlabschnitts in einem Ofen, der auf eine Temperatur und während eines Zeitraums angeheizt wird, die durch das Diagramm ABCD der Figur 1, wenn die Dicke des Blechs größer als oder gleich 0,7 mm und kleiner als oder gleich 1,5 mm ist, und durch das Diagramm EFGH der Figur 1 definiert sind, wenn die Dicke des Blechs größer als 1,5 mm und kleiner als oder gleich 3 mm ist, zwischen 20 und 700°C bei einer Heizrate  $V_c$  zwischen 4 und 12°C/s und zwischen 500 und 700°C bei einer Heizrate  $V_c'$  zwischen 1,5 und 6°C/s, um einen aufgeheizten Abschnitt zu erhalten; dann
- Übertragen des aufgeheizten Abschnitts in ein Presswerkzeug; dann
- Heißstanzen des aufgeheizten Abschnitts in dem Presswerkzeug, um dabei einen heißgestanzten Stahlabschnitt zu erhalten, dann
- Abkühlen des heißgestanzten Stahlabschnitts zwischen dem Austritt des aufgeheizten Abschnitts aus dem Ofen herunter auf 400°C bei einer mittleren Rate  $V_r$  von mindestens 30°C/s.

2. Verfahren nach Anspruch 1, bei der die Zeit, die zwischen dem Austritt des aufgeheizten Abschnitts aus dem Ofen und dem Beginn des Stanzens abläuft, nicht mehr als 10 Sekunden ist.

3. Heißgestanzter beschichteter Stahlabschnitt, der umfasst:

- (a) einen Streifen eines Basisstahls mit einer ersten Seite und einer zweiten Seite; und
- (b) eine Beschichtung auf mindestens einer der

ersten Seite des Stahlstreifens aus Basisstahl und der zweiten Seite des Streifens aus Basisstahl, wobei:

- (i) die Beschichtung aus der Interdiffusion zwischen dem Basisstahl und einer Aluminium- oder Aluminiumlegierung-Vorbeschichtung resultiert, 5  
(ii) die Beschichtung umfasst, beginnend vom Basisstahl nach außen, eine 10

- (a) Interdiffusionsschicht
- (b) Zwischenschicht
- (c) Intermetallische Schicht
- (d) Oberflächenschicht 15

- (iii) die Beschichtung enthält in Oberflächenbruchteilen weniger als 10% Porosität und wobei die Schichten (c) und (d) quasi kontinuierlich durch Einnehmen von mindestens 90% ihres jeweiligen Niveaus und wobei weniger als 10% der Schicht (c) an der extremen Fläche des heißgestanzten beschichteten Stahlabschnitts vorhanden ist. 20

4. Heißgestanzter beschichteter Stahlabschnitt nach Anspruch 3, wobei die Oberflächenschicht (d) in Oberflächenbruchteilen weniger als 20% Porosität enthält. 25

5. Heißgestanzter beschichteter Stahlabschnitt nach Anspruch 3 oder 4, wobei die Beschichtung eine Dicke größer als 30 µm aufweist. 30

6. Heißgestanzter beschichteter Stahlabschnitt nach einem der Ansprüche 3 bis 5, bei dem die Schicht (a) eine Dicke kleiner als 15 µm aufweist. 35

7. Heißgestanzter beschichteter Stahlabschnitt nach einem der Ansprüche 3 bis 6, bei dem die Stahlsammensetzung in dem Streifen die folgenden Komponenten in Gewichtsprozenten basierend auf dem Gesamtgewicht umfasst: 40

0,15%<Kohlenstoff<0,5% 45

0,5%<Mangan<3% 50

0,1%<Silizium<0,5% 55

0,01%<Chrom<1%

Nickel<0,1%

Kupfer<0,1%

Titan<0,2%

Aluminium<0,1%

Phosphor<0,1%

Schwefel<0,05%

0,0005%<Bor<0,08%,

wobei der Ausgleich Eisen und nicht vermeidbare Verunreinigungen sind.

8. Heißgestanzter beschichteter Stahlabschnitt nach einem der Ansprüche 3 bis 6, bei dem die Stahlsammensetzung in dem Streifen die folgenden Komponenten in Gewichtsprozenten basierend auf dem Gesamtgewicht umfasst: 25

0,20%<Kohlenstoff<0,5% 30

0,8%<Mangan<1,5% 35

0,1%<Silizium<0,35% 40

0,01%<Chrom<1% 45

Nickel<0,1%

Kupfer<0,1%

Titan<0,1%

Aluminium<0,1%

Phosphor<0,05%

Schwefel<0,03%

0,0005%<Bor<0,01%,

wobei der Ausgleich Eisen und nicht vermeidbare Verunreinigungen sind.

9. Heißgestanzter beschichteter Stahlabschnitt nach einem der Ansprüche 3 bis 8, wobei die Aluminium- oder Aluminiumlegierung-Vorbeschichtung 8 bis 11 Gew.-% Silizium, 2 bis 4 Gew.-% Eisen, als Rest Aluminium und dem Verfahren inhärente Verunreinigungen umfasst.
10. Verwendung eines heißgestanzten beschichteten Stahlabschnitts nach einem der Ansprüche 3 bis 9 für die Herstellung von Landmotorfahrzeugen.
11. Verwendung eines heißgestanzten beschichteten Stahlabschnitts, hergestellt nach einem Verfahren nach einem der Ansprüche 1 oder 2 für die Herstellung von Landmotorfahrzeugen.

#### Revendications

1. Procédé pour produire une ébauche en acier revêtue estampée à chaud, comprenant les étapes consistant à :
  - pré-revêtir une bande ou tôle d'acier avec de l'aluminium ou un alliage d'aluminium, par trempage à chaud de ladite bande ou tôle d'acier ayant un premier côté et un deuxième côté, dans un bain d'aluminium ou d'alliage d'aluminium, l'épaisseur  $t_p$  dudit pré-revêtement étant de 20 à 33 micromètres en tout emplacement sur lesdits premier et deuxième côtés de ladite bande ou tôle, puis
  - découper ladite bande ou tôle d'acier pré-revêtue pour obtenir une ébauche en acier pré-revêtue, puis
  - chauffer ladite ébauche en acier pré-revêtue d'aluminium ou d'alliage d'aluminium dans un four préchauffé à une température pendant un temps défini par le diagramme ABCD de la Figure 1 si l'épaisseur de ladite tôle est supérieure ou égale à 0,7 mm et inférieure ou égale à 1,5 mm, et par le diagramme EFGH de la Figure 1 si l'épaisseur de ladite tôle est supérieure à 1,5 mm et inférieure ou égale à 3 mm, à une vitesse

de chauffage  $V_c$  de 20 à 700°C comprise entre 4 et 12°C/s, et à une vitesse de chauffage  $V_c'$  de 500 à 700°C comprise entre 1,5 et 6°C/s, pour obtenir une ébauche chauffée ; puis

- transférer ladite ébauche chauffée dans une matrice ; puis
- estamper à chaud ladite ébauche chauffée dans ladite matrice, de façon à obtenir ainsi une ébauche en acier estampée à chaud, puis
- refroidir ladite ébauche en acier estampée à chaud à une vitesse moyenne  $V_r$  depuis la sortie de ladite ébauche chauffée hors du four, jusqu'à 400°C, d'au moins 30°C/s.

2. Procédé selon la revendication 1, dans lequel le temps écoulé entre la sortie hors dudit four de ladite ébauche chauffée et le début dudit estampage ne dépasse pas 10 secondes.

3. Ebauche en acier revêtue estampée à chaud, qui comprend :

(a) une bande d'acier de base ayant un premier côté et un deuxième côté ; et  
 (b) un revêtement sur au moins l'un parmi ledit premier côté de ladite bande d'acier de base et ledit deuxième côté de ladite bande d'acier de base, dans laquelle :

(i) ledit revêtement résulte de l'interdiffusion entre ledit acier de base, et un pré-revêtement d'aluminium ou d'alliage d'aluminium,  
 (ii) ledit revêtement comprend, en allant de l'acier de base vers l'extérieur,

- (a) une couche d'interdiffusion,
- (b) une couche intermédiaire
- (c) une couche intermétallique,
- (d) une couche superficielle,

(iii) ledit revêtement contient, en fraction surfacique, moins de 10 % de porosités,

et dans laquelle lesdites couches (c) et (d) sont quasi-continues en occupant au moins 90 % de leur niveau respectif et dans laquelle moins de 10 % de la couche (c) est présent à la surface extrême de ladite ébauche en acier revêtue estampée à chaud.

4. Ebauche en acier revêtue estampée à chaud selon la revendication 3, dans laquelle ladite couche superficielle (d) contient, en fraction surfacique, moins de 20 % de porosités.
5. Ebauche en acier revêtue estampée à chaud selon la revendication 3 ou 4, dans laquelle ledit revête-

ment a une épaisseur supérieure à 30 micromètres.

au poids total :

6. Ebauche en acier revêtue estampée à chaud selon l'une quelconque des revendications 3 à 5, dans laquelle ladite couche (a) a une épaisseur inférieure à 15 micromètres.

0,20 % < carbone < 0,5 %

0,8 % < manganèse < 1,5 %

7. Ebauche en acier revêtue estampée à chaud selon l'une quelconque des revendications 3 à 6, dans laquelle la composition de l'acier dans la bande comprend les composants suivants, en poids par rapport au poids total :

0,1 % < silicium < 0,35 %

0,15 % < carbone < 0,5 %

0,01 % < chrome < 1 %

0,5 % < manganèse < 3 %

nickel < 0,1 %

0,1 % < silicium < 0,5 %

cuivre < 0,1 %

0,01 % < chrome < 1 %

titane < 0,1 %

nickel < 0,1 %

aluminium < 0,1 %

cuivre < 0,1 %

phosphore < 0,05 %

titane < 0,2 %

soufre < 0,03 %

aluminium < 0,1 %

0,0005 % < bore < 0,01 %

phosphore < 0,1 %

le reste étant du fer et des impuretés inévitables.

soufre < 0,05 %

0,0005 % < bore < 0,08 %

9. Ebauche en acier revêtue estampée à chaud selon l'une quelconque des revendications 3 à 8, dans laquelle le pré-revêtement d'aluminium ou d'alliage d'aluminium comprend de 8 % à 11 % en poids de silicium, de 2 % à 4 % en poids de fer, le reste étant de l'aluminium et des impuretés inhérentes au traitement.

10. Utilisation d'une ébauche en acier revêtue estampée à chaud selon l'une quelconque des revendications 3 à 9 pour la fabrication d'un véhicule à moteur terrestre.

11. Utilisation d'une ébauche en acier revêtue estampée à chaud fabriquée conformément à un procédé selon

le reste étant du fer et des impuretés inévitables.

8. Ebauche en acier revêtue estampée à chaud selon l'une quelconque des revendications 3 à 6, dans laquelle la composition de l'acier dans la bande comprend les composants suivants, en poids par rapport

l'une quelconque des revendications 1 et 2, pour la fabrication d'un véhicule à moteur terrestre.

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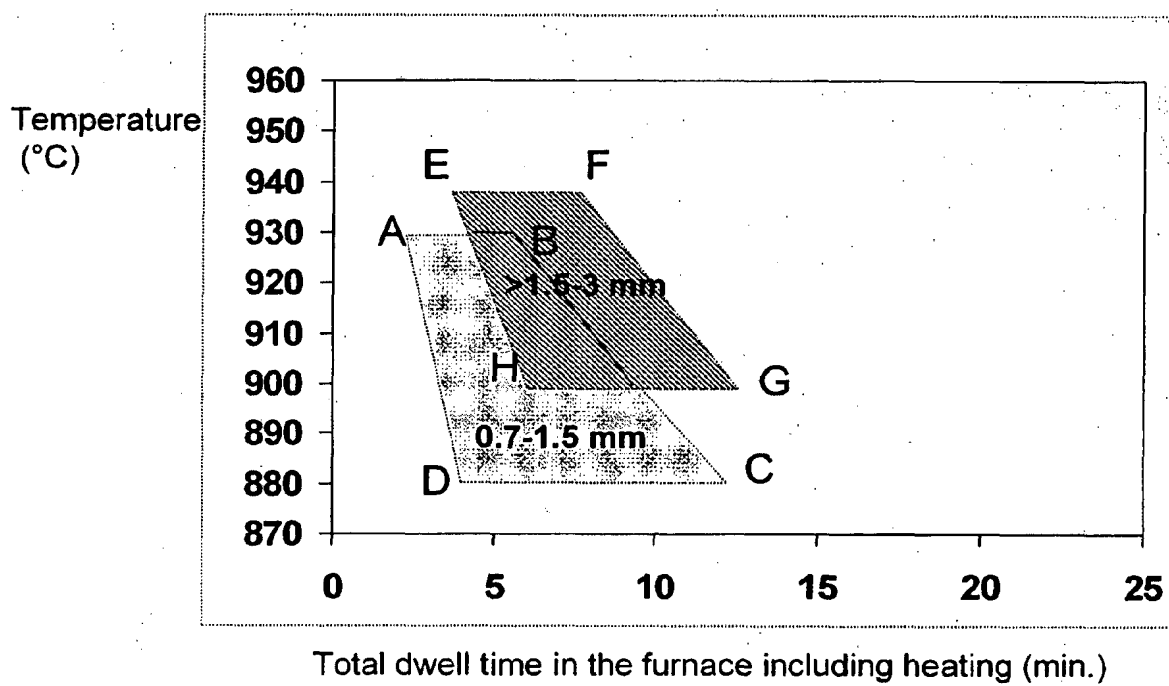


Fig. 1

**REFERENCES CITED IN THE DESCRIPTION**

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**Patent documents cited in the description**

- US 6296805 B [0002]
- EP 1380666 A [0004]

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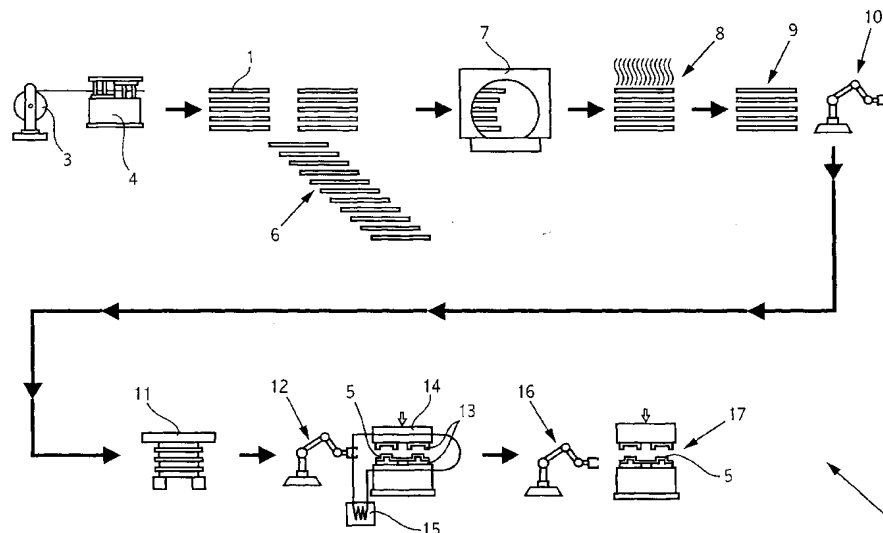
(81) Bestimmungsstaaten (soweit nicht anders angegeben, für  
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AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH,  
CN, CO, CR, CU, CZ, DK, DM, DZ, EC, EE, EG, ES, FI,  
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EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL,

[Fortsetzung auf der nächsten Seite]

(54) Title: METHOD FOR PRODUCING A COMPONENT BY RESHAPING A PLATE, AND DEVICE FOR CARRYING OUT  
SAID METHOD

(54) Bezeichnung: VERFAHREN ZUR HERSTELLUNG EINES BAUTEILS DURCH UMFORMEN EINER PLATINE UND  
VORRICHTUNG ZUR DURCHFÜHRUNG DES VERFAHRENS



(57) Abstract: The invention relates to a method for producing a component (5) by reshaping a coated plate (1) consisting of tempering steel, said plate being austenitised before the reshaping by means of a first heat treatment, followed by the growth of the layer thickness. The aim of the invention is to optimise the process and to prevent the production of scrap plates caused by interruptions to the process. To this end, after being rapidly cooled, the heat-treated plate (1) is temporarily stored, and is briefly heated again to the austenitisation temperature, directly before being reshaped to form the component (5). Once the structure has been modified, the plate (1) is reshaped and hardened. Preferably, the plate (1) is heated a second time by induction.

[Fortsetzung auf der nächsten Seite]



PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

**Erklärungen gemäß Regel 4.17:**

- hinsichtlich der Berechtigung des Anmelders, ein Patent zu beantragen und zu erhalten (Regel 4.17 Ziffer ii) für die folgenden Bestimmungsstaaten AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW, ARIPO Patent (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW),

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europäisches Patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE,  
ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL,  
PT, RO, SE, SI, SK, TR), OAPI Patent (BF, BJ, CF, CG, CI,  
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**Veröffentlicht:**

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Zur Erklärung der Zweibuchstaben-Codes und der anderen Abkürzungen wird auf die Erklärungen ("Guidance Notes on Codes and Abbreviations") am Anfang jeder regulären Ausgabe der PCT-Gazette verwiesen.

**(57) Zusammenfassung:** Die Erfindung bezieht sich auf ein Verfahren zur Herstellung eines Bauteils (5) durch Umformen einer beschichteten Platine (1) aus einem Vergütungsstahl, wobei sie vor dem Umformen über eine erste Wärmebehandlung austenitisiert wird, wobei sich darüber hinaus ein Schichtdickenwachstum vollzieht. Eine Optimierung im Prozessablauf und ein Vermeiden von Ausschußplatinen bei Prozessstörungen ist dadurch erreichbar, dass nach einem raschen Abkühlen die wärmebehandelte Platine (1) zwischengelagert wird, dass unmittelbar vor dem Umformen zum Bauteil (5) die Platine einer erneuten, kurzzeitigen Erwärmung auf Austenitisierungstemperatur unterzogen wird und dass nach erfolgter Gefügeumwandlung das Umformen und Härten der Platine (1) vonstatten geht. Vorzugsweise geschieht die zweite Erwärmung der Platine (1) durch Induktion.

## **Verfahren zur Herstellung eines Bauteils durch Umformen einer Platine und Vorrichtung zur Durchführung des Verfahrens**

### **B E S C H R E I B U N G**

Die Erfindung bezieht sich auf ein Verfahren zur Herstellung eines Bauteils durch Umformen einer beschichteten, vorzugsweise aluminiumbeschichteten Platine aus einem Vergütungsstahl nach dem Oberbegriff des Patentanspruches 1. Die Erfindung betrifft des weiteren eine Vorrichtung zur Durchführung des Verfahrens.

Im Stand der Technik sind verschiedene Umformverfahren für Platinen aus einem Vergütungsstahl in Verbindung mit Härtingsprozessen allgemein bekannt. Bei einem sogenannten „direkten“ Umformverfahren wird eine ebene Platine aus einem Vergütungsstahl in einem Ofen, vorzugsweise einem Durchlaufofen, unter Schutzgasatmosphäre austenitisiert. Beispielsweise kann ein Vergütungsstahl in der Qualität 22MnB5 verwendet werden, der für die Austenitisierung mehrere Minuten bei ca. 950°C geglüht wird. Anschließend wird mit einer vorzugsweise automatisierten Transfereinrichtung die heiße austenitisierte ebene Platine in ein für Serienprozesse gekühltes Umform-/Vergütungswerkzeug eingelegt. Dieses Werkzeug ist Bestandteil einer Presse und beim Schließen derselben wird die heiße Platine zu einem Bauteil auf Endform umgeformt und im geschlossenen Werkzeug bei aufgebrachter Schließkraft relativ schnell abgekühlt und dadurch gehärtet. Das gehärtete Bauteil wird aus dem Werkzeug entnommen und, falls es sich um ein unbeschichtetes Blech handelt, in einem Reinigungsschritt beispielsweise durch Sand- oder Kugelstrahlen entzundert (dies ist bei beschichteten Bauteilen entbehrlich, da z. B. aluminisierte Bleche ausreichend Korrosionsschutz bieten und eine Verzunderung verhindert wird). Abschließend folgt ein Fertigkontur- und Lochbeschnitt des fertig geformten und gehärteten Bauteiles, vorzugsweise mittels Laserschneiden. Auch ein mechanisches Schneiden im sogenannten Pressenverbund ist denkbar.

Während der Wärmebehandlung im Ofen erfährt z. B. eine im Ausgangszustand etwa 25 µm starke Aluminiumbeschichtung ein Schichtdickenwachstum auf etwa 45 µm, wobei sich unmittelbar an das Grundmaterial der Platine angrenzend eine AlSi-Schicht mit eindiffundiertem Eisen heraus bildet, die eine darüber hinaus sich bildende, relativ

harte und spröde AlSi-Schicht trägt, die die eigentliche Korrosionsschutzfunktion erfüllt.

Ein typischer Verfahrensablauf (Warmumformkurve 20) hinsichtlich der Wärmebehandlung der Platine im Zuge ihres Umformens ist beispielsweise in Fig. 1 in einem Zeit- Temperatur- Diagramm dargestellt. Je nach verwendeter Vergütungsstahlqualität, Blechdicke, Ausgangsschichtdicke u.s.w. sind die in Fig.1 angegebenen Werte natürlich auch gewissen Schwankungen unterworfen (Wärmebehandlungsunter- / obergrenze 18, 19). So ist ohne weiteres denkbar, dass die Platine sich über eine Verweildauer von bis zu 30 Minuten im Ofen befindet.

Der verwendete Ofen ist häufig ein sogenannter Durchlaufofen mit Formnestern oder Platinenaufnahmen oder ein Rostdurchschubofen mit Rosten, welche die Platine tragen und diese innerhalb von etwa 2 Minuten mittels Gasbrenner auf Austenitisierungstemperatur erwärmen und anschließend mehrere Minuten mittels elektrischer Heizung auf dieser Temperatur halten. Vorzug des Gasbrenners ist eine höhere Leistung, wohingegen elektrische Heizungen besser regelbar sind.

Gemäß Fig. 1 wird also die Platine im Ofen auf eine Solltemperatur von etwa 950° C erhitzt und auf dieser Temperatur gehalten. Die Austenitisierung vollzieht sich bei Temperaturen oberhalb etwa 720° C. Üblicherweise beträgt die Verweildauer im Ofen ca. 9 Minuten, wobei sich die Platine innerhalb der ersten beiden Minuten auf Solltemperatur erwärmt, während in den darauffolgenden etwa 7 Minuten das Umkörnen des Grundmaterials vom kubisch- raumzentrierten Ferrit/Perlit- Gefüge ins kubisch- flächenzentrierte Austenit, das für ein Härten notwendig ist, vonstatten geht. Daneben ist die genannte Zeitspanne vor allem auch wichtig, um ein ausreichendes Anwachsen der AlSi-Schutzschicht zu erreichen.

Insbesondere hinsichtlich minimaler Glühtemperatur und maximaler Verweildauer der Platine im Ofen gibt es gewisse, mehr oder weniger enge Grenzen, innerhalb derer der Prozess noch Gutteile liefert, d. h., dass die aus dem Ofen entnommenen Platinen überhaupt noch für den Umformprozess und die weitere Verwendung verwertbar sind.

Stellt sich nun im weiteren Ablauf eine Störung ein, sei es bei der Entnahme einer Platine aus dem Ofen und ihrem Weitertransport zum Umform-/ Vergütungswerkzeug bzw. innerhalb der Station des Fertigkontur- und Lochbeschnittes, so kann für die Dauer der Störung keine Platine mehr aus dem Ofen entnommen werden, die maximal zulässige Verweildauer wird in der Regel überschritten und sämtliche im Ofen befindlichen Platinen sind Ausschuss und müssen von daher entsorgt werden.

Der Erfindung liegt die Aufgabe zugrunde, das gattungsgemäße Verfahren zur Herstellung eines Bauteiles durch Umformen einer beschichteten Platine aus einem Vergütungsstahl in der Weise weiterzubilden, dass eine Optimierung im Prozessablauf erreichbar ist und dass insbesondere bei eventuellen Prozessstörungen dem kostenträchtigen Anfallen von Ausschussplatinen vorgebeugt werden kann.

Diese Aufgabe wird hinsichtlich des Verfahrens mit den weiteren Merkmalen nach dem Kennzeichen des Patentanspruches 1 und hinsichtlich der Vorrichtung zur Durchführung des erfindungsgemäßen Verfahrens mit den Merkmalen des Patentanspruches 10 gelöst.

Die Vorteile der erfindungsgemäßen Vorgehensweise sind mannigfaltig. So besteht nun keine Abhängigkeit mehr bezüglich der Verweildauer der Platinen innerhalb des Ofens und Ablaufstörungen im Prozess außerhalb des Ofens. Durch die Entkopplung der Abläufe ergibt sich für den eigentlichen Prozess der Platinenumformung ein geringerer Flächenbedarf und ein Weniger an Infrastruktur. Eine Pufferung / Zwischenlagerung der wärmebehandelten Platinen ist möglich, so dass eine Wärmebehandlung, unter anderem zur Beeinflussung der AlSi-Schicht, ohne weiteres auch bereits beim Stahlhersteller bzw. Blechlieferanten erfolgen kann.

Eine solche ausgelagerte, vorgeschaltete Wärmebehandlung ist an sich bereits bekannt, wie aus der EP 0 946 311 B1 und der DE 102 12 400 C1 hervorgeht. Auch eine Glühbehandlung mittels Induktionserwärmung ist für sich gesehen bereits bekannter Stand der Technik, wie dies im letztgenannten Dokument beispielsweise auch erwähnt ist.

Vorteilhafte Ausgestaltungen und Weiterbildungen der Erfindung sind in den jeweiligen Unteransprüchen beansprucht.

Die Erfindung ist nachstehend anhand eines Ausführungsbeispiels näher erläutert. In der Zeichnung zeigen:

Fig. 1 den erfindungsgemäßen Verfahrensablauf zur Herstellung eines Bauteiles durch Umformen einer Platine,

Fig. 2 ein Temperatur- Zeit- Diagramm der ersten Platinen- Wärmebehandlung und

Fig. 3 ein Temperatur- Zeit- Diagramm der zweiten Platinen- Wärmebehandlung.

In Fig. 1 ist schematisch der erfindungsgemäße Verfahrensablauf zur Herstellung eines Bauteiles 5 durch Umformen einer beschichteten Platine 1 aus einem Vergütungsstahl mittels einer hierfür geeigneten Vorrichtung 2 gezeigt. Von dem im aufgerolltem Zustand angelieferten Stahl, einem sogenannten Coil 3, wird mittels eines Werkzeuges 4

der Stahl abgerollt, geglättet und die für das fertige Bauteil 5 notwendige Größe der Platine 1 ausgestanzt bzw. abgeschnitten. Von dort aus werden die Platinen 1 einer Pufferzone 6 zugeführt. Dieses Zwischenlagern ist allerdings nicht unbedingt erforderlich, vielmehr können die Platinen 1 auch unmittelbar nach Verlassen des Werkzeuges 4 einem ersten Ofen 7 zugeführt werden, in dem sie eine Wärmebehandlung gemäß dem Temperatur- Zeit- Diagramm nach Fig. 2 erfahren. Unmittelbar dem ersten Ofen 7 nachgelagert ist eine Abkühlzone 8, in der die Platinen 1 abgeschreckt werden und die abschließenden Phasen der Wärmebehandlung durchlaufen. Die Abkühlzone 8 verlassend werden die wärmebehandelten Platinen 1 einem Zwischenlager 9 zugeführt.

Der erste Ofen 7 kann vom konstruktiven Aufbau her ein bereits erwähnter Durchlauf-ofen, ein Karussell- Ofen oder dergleichen sein.

Die einzelnen Phasen der Wärmebehandlung wurden bereits eingangs mit Hinweis auf Fig. 2 erläutert. Das relativ langsam vonstatten gehende Aufheizen auf Solltemperatur sowie die übrige Verweildauer im ersten Ofen 7 zum Herbeiführen der Austenitisierung und zum Verändern der Topographie (Beschichtungsaufbau, Schichtdicke) summiert sich auf eine Gesamt- Verweildauer von etwa 9 Minuten, wobei erfahrungsgemäß eine maximale Verweildauer von 30 Minuten nicht überschritten werden darf, damit die Platine nicht unbrauchbar wird. Der Transport in die Abkühlzone 8 und die dortige Abschreckung der Platine 1 vollzieht sich innerhalb relativ kurzer Zeiträume, während ein restliches Abkühlen auf Raumtemperatur RT im Zwischenlager 9 erfolgen kann. Am Ende der Wärmebehandlung weist die Platine 1 ein Martensitgefüge auf.

Mittels einer geeigneten Transportvorrichtung 10, beispielsweise einem Gelenkarmroboter, werden die Platinen 1 einem Induktionsofen 11 zugeführt, von wo aus sie über eine weitere Transportvorrichtung 12, z. B. wiederum ein Gelenkarmroboter, in ein für Serienprozesse geeignetes, gekühltes Umform-/Vergütungswerkzeug 13 eingelegt werden. Diesem ist eine Presseneinrichtung 14 sowie eine Kühlvorrichtung 15 zugeordnet, und beim Schließen der Presseneinrichtung 14 wird die heiße Platine 1 zu einem Bauteil 5 auf Entform umgeformt und im geschlossenen Umform-/Vergütungswerkzeug 13 bei aufgebrachter Schließkraft schnell abgekühlt und dadurch gehärtet. In einem letzten Verfahrensschritt wird jedes Bauteil 5 über eine Transportvorrichtung 16 einer Beschnittvorrichtung 17 zugeführt, wo ein Fertigkontur- und Lochbeschnitt des fertig geformten und gehärteten Bauteiles 5 vorzugsweise mittels Laserschneiden durchgeführt wird. Selbstverständlich kann dies auch über geeignete Beschnittmesser auf mechanischem Wege erfolgen.

Die im Induktionsofen 11 und im nachgeschalteten Umform-/Vergütungswerkzeug 13 vonstatten gehende Wärmebehandlung der Platine 1 ist im Temperatur- Zeit- Diagramm gemäß Fig. 3 anhand der Warmumformkurve 20 und der Wärmebehandlungsunter- / obergrenze 18, 19 dargestellt. Sie ist gekennzeichnet durch eine ausgesprochen kurze Verweildauer der Platine im Induktionsofen 11. Während das Aufheizen auf Solltemperatur (Austenitisierungstemperatur) innerhalb weniger Sekunden (ca. zehn sec. ) erfolgt dient eine nachgeschaltete kurze Verweilzeit von etwa zehn Sekunden bis maximal zwei Minuten dazu, eine Gefügeumwandlung vonstatten gehen zu lassen. Eine Änderung von Dicke und Aufbau der Beschichtung ist nicht mehr erforderlich, da dies bereits im ersten Ofen 7 erfolgt ist. Nach demzufolge äußerst kurzer Verweildauer im Induktionsofen 11 kann die Platine bereits dem Umform-/Vergütungswerkzeug 13 zugeführt werden, in dem neben der Umformung das Abschrecken in gleicher Weise (gleicher Verlauf der Warmumformkurve 20) vonstatten geht wie in der Abkühlzone 8. Beim Verlassen des Umform-/Vergütungswerkzeuges 13 weist das Bauteil 5 bereits Martensitgefüge auf, die Abkühlung auf Raumtemperatur RT kann beim Weitertransport zur bzw. innerhalb der Beschnittvorrichtung 17 erfolgen.

Auf diese Weise wurde eine eine ursprüngliche Zugfestigkeit von etwa 500 bis 600 N/mm<sup>2</sup> aufweisende Platine 1 in ein eine Zugfestigkeit von etwa 1300 bis 1500 N/mm<sup>2</sup> aufweisendes Bauteil 5 umgeformt.

In vorteilhafter Weiterbildung der Erfindung wäre denkbar, im Induktionsofen 11 die Platine 1 partiell unterschiedlich stark zu erwärmen, mit der Folge, dass, falls dies gewünscht ist, das umgeformte und abgeschreckte Bauteil 5 partiell unterschiedliche Festigkeiten aufweist.

Weiter wäre vorteilhaft möglich, die Platine 1 vor der zweiten Wärmebehandlung (Induktionsofen 11) zum Beispiel durch Aufschweißen von Verstärkungsblechen (Patches) lokal zu verstärken. Ein solchermaßen gepatchtes Verbundblech könnte dann dem zweiten Ofen und danach dem Umform-/Vergütungswerkzeug 13 zugeführt werden. Dies hätte insgesamt positive Auswirkungen auf die Werkstoffeigenschaften und die Formgenauigkeit.

Das erfindungsgemäße Verfahren ist auch bei Verwendung von Tailored blanks Platinen vorteilhaft anwendbar.

Ein wesentlicher Vorteil der Erfindung ist die Möglichkeit der Entkopplung der einzelnen Verfahrensschritte. So kann die erste Wärmebehandlung im Ofen 7 bereits beim Stahl- bzw. Blechhersteller erfolgen und die so vorbehandelten Platinen 1 können dann

dem weiterverarbeitenden Unternehmen (z. B. Fahrzeughersteller) bereitgestellt werden (Zwischenlager 9).

In einer weiteren vorteilhaften Weiterbildung der Erfindung ist denkbar, der Transportvorrichtung 10 einen Induktor zuzuordnen bzw. diesen in die Transportvorrichtung 10 baulich zu integrieren, so dass die Wärmebehandlung der Platine 1 während ihres Transportes zum Umform-/Vergütungswerkzeug 13 vonstatten gehen kann. Ein separater Induktionsofen 11 sowie eine diesem nachgeschaltete weitere Transportvorrichtung 12 könnten dadurch entfallen.

## ANSPRÜCHE

1. Verfahren zur Herstellung eines Bauteils durch Umformen einer beschichteten, vorzugsweise aluminiumbeschichteten Platine aus einem Vergütungsstahl, wobei vor dem Umformen in einem ersten Verfahrensschritt die Platine einem ersten Ofen zugeführt und dort austenitisiert wird und wobei die Verweildauer der Platine im ersten Ofen so gewählt wird, dass neben der Gefügeumwandlung ein Schichtdickenwachstum vonstatten geht, gekennzeichnet durch folgende weitere Verfahrensschritte:
  - schnelles Abkühlen und anschließendes Zwischenlagern der wärmebehandelten Platine (1)
  - erneutes, kurzzeitiges Erwärmen der Platine (1) in einem zweiten Ofen (11) auf Austenitisierungstemperatur unmittelbar vor dem Umformen zum Bauteil (5) und
  - nach erfolgter Gefügeumwandlung Umformen und Härten der Platine (1).
2. Verfahren nach Patentanspruch 1, gekennzeichnet durch eine Verweildauer im ersten Ofen (7) zwischen neun Minuten und dreißig Minuten.
3. Verfahren nach Patentanspruch 1, dadurch gekennzeichnet, dass bei der wiederholten Erwärmung der Platine (1) auf Austenitisierungstemperatur im zweiten Ofen (11) die Verweildauer so gewählt wird, dass lediglich eine Gefügeumwandlung, aber kein Schichtdickenwachstum mehr statt findet.
4. Verfahren nach Patentanspruch 3, gekennzeichnet durch eine Verweildauer der Platine (1) im zweiten Ofen (11) von zehn Sekunden bis zweieinhalb Minuten.
5. Verfahren nach Patentanspruch 1, dadurch gekennzeichnet, dass die Erwärmung der Platine (1) im ersten Ofen (7) auf Elektro- oder Gas-Basis erfolgt, während die Erwärmung im zweiten Ofen (11) durch Induktion vonstatten geht.
6. Verfahren nach Patentanspruch 1, dadurch gekennzeichnet, dass die erste Erwärmung beim Stahl- bzw. Blechhersteller erfolgt, während die zweite Wärmebehand-

lung beim weiterverarbeitenden Unternehmen, z. B. Fahrzeughersteller, vonstatten geht.

7. Verfahren nach Patentanspruch 1, dadurch gekennzeichnet, dass während der zweiten Wärmebehandlung die Platine (1) über ihre Oberfläche unterschiedlich stark erwärmt wird.
8. Verfahren nach Patentanspruch 1, dadurch gekennzeichnet, dass die Platine (1) vor dem erneuten Erwärmen im zweiten Ofen (11) durch Aufbringen von mindestens einem Verstärkungsblech lokal verstärkt wird.
9. Verfahren nach Patentanspruch 1, gekennzeichnet durch die Verwendung eines Tailored blanks als Platine (1).
10. Vorrichtung zur Durchführung des Verfahrens nach einem der Patentansprüche 1-9, gekennzeichnet durch
  - ein Werkzeug (4) zum Herstellen von Platinen (1) aus einem Coil (3),
  - einen ersten Ofen (7) zur ersten Wärmebehandlung einschließlich Herbeiführen eines Schichtdickenwachstums der Platinen (1),
  - eine Abkühlzone (8) für die Platinen (1),
  - ein Zwischenlager (9) für die Platinen (1),
  - einen zweiten Ofen (11) zur erneuten Wärmebehandlung der Platinen (1),
  - ein Umform-/Vergütungswerkzeug (13) mit einer Presseneinrichtung (14) und einer Kühlvorrichtung (15) und
  - eine Beschnittvorrichtung (17) zur Herstellung eines Fertigkontur- und Lochbeschnittes.
11. Vorrichtung nach Patentanspruch 10, gekennzeichnet durch einen ersten Ofen (7) auf Elektro-und/oder Gas-Basis und einen Induktionsofen (11) zur zweiten Wärmebehandlung.
12. Vorrichtung nach Patentanspruch 11, dadurch gekennzeichnet, dass ein Induktor in eine Transportvorrichtung (10) integriert ist, die zwischen Zwischenlager (9) und Umform-Vergütungswerkzeug (13) angeordnet ist.
13. Vorrichtung nach Patentanspruch 10, dadurch gekennzeichnet, dass zwischen der Abkühlzone (8) und dem zweiten Ofen (11) eine Station zum Aufbringen, insbesondere Aufschweißen, von mindestens einem Verstärkungsblech auf die Platine (1) angeordnet ist.

I/III

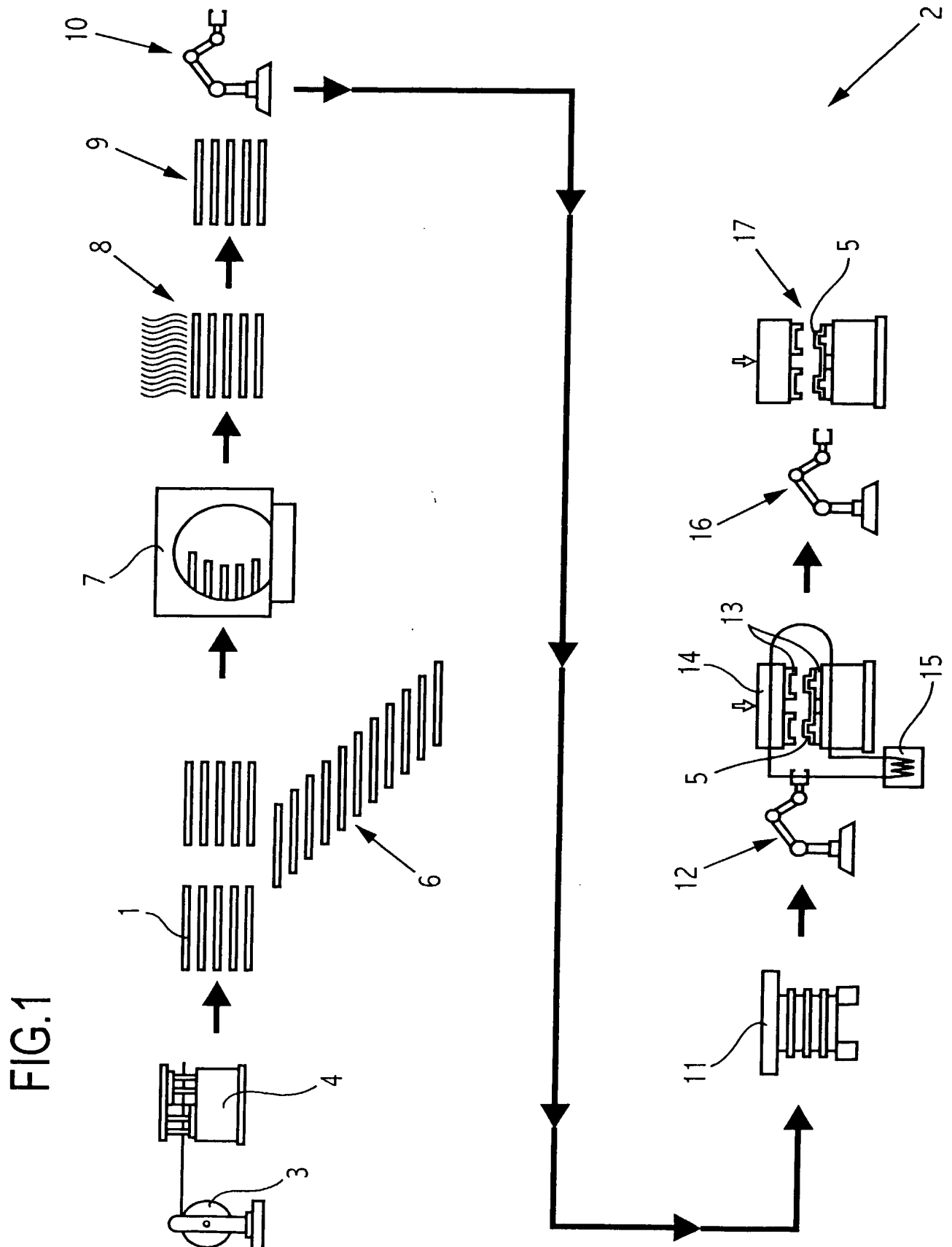


**FIG. 1**

II/III

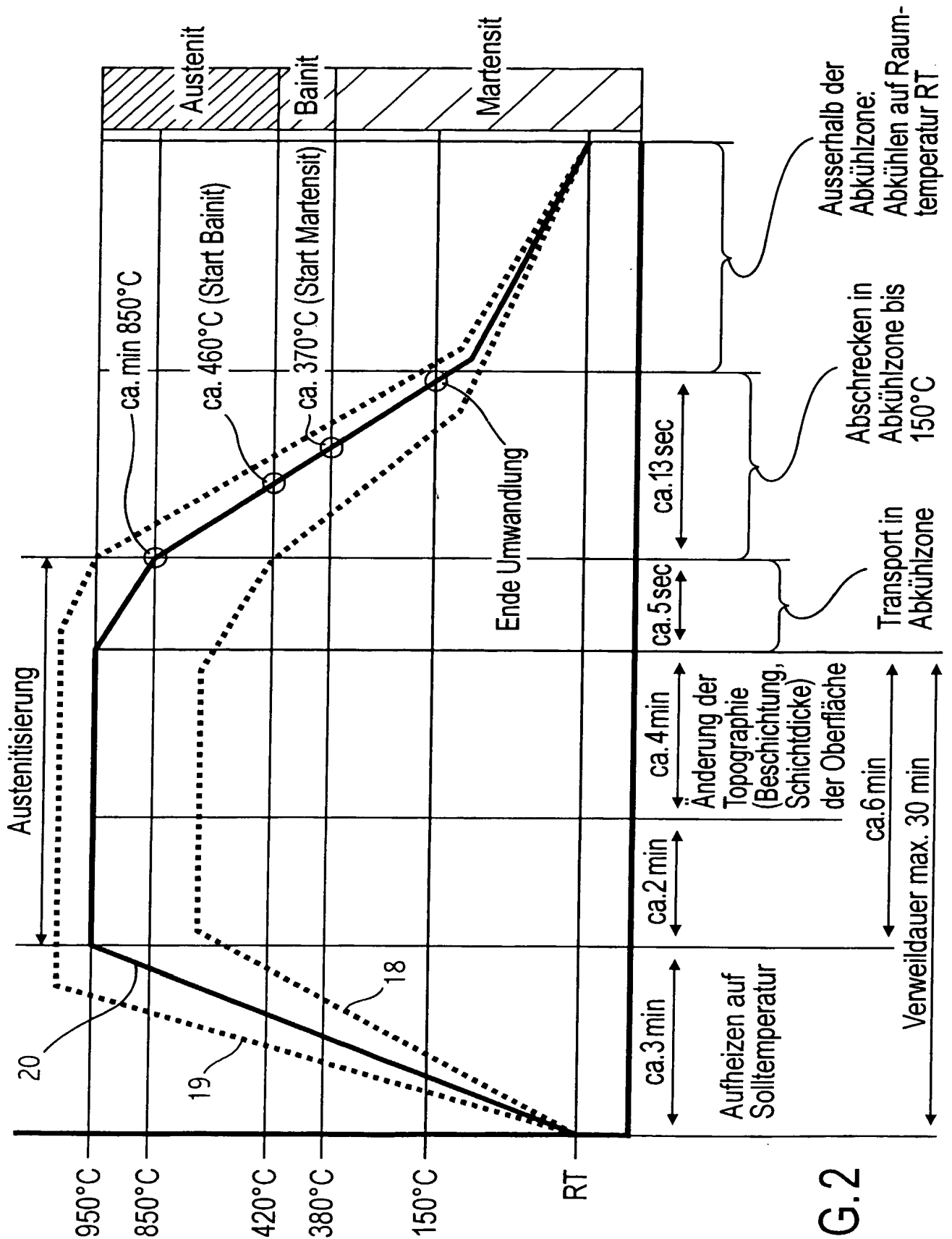
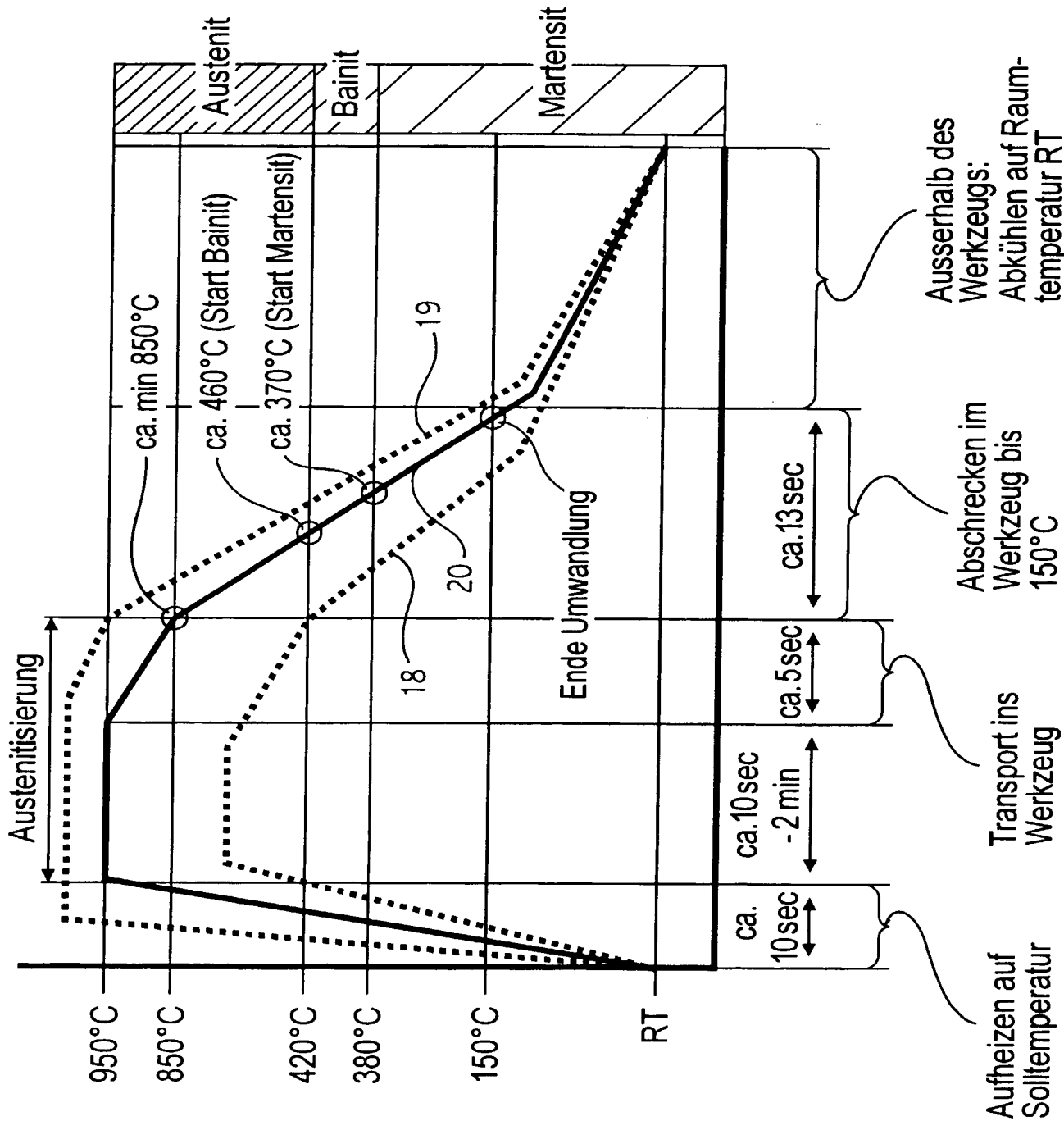


**FIG. 2**

日/日



## INTERNATIONAL SEARCH REPORT

International Application No  
PCT/EP2005/000853

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 7 C21D9/46

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
IPC 7 C21D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

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## C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category * | Citation of document, with indication, where appropriate, of the relevant passages                                                                                            | Relevant to claim No. |
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|            | -----<br>-/--                                                                                                                                                                 |                       |

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Date of the actual completion of the international search

25 April 2005

Date of mailing of the international search report

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## INTERNATIONAL SEARCH REPORT

International Application No  
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## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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Information on patent family members

International Application No

PCT/EP2005/000853

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**A. KLASSIFIZIERUNG DES ANMELDUNGSGEGENSTANDES**  
IPK 7 C21D9/46

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**B. RECHERCHIERTE GEBIETE**Recherchierter Mindestprüfstoff (Klassifikationssystem und Klassifikationssymbole )  
IPK 7 C21D

Recherchierte aber nicht zum Mindestprüfstoff gehörende Veröffentlichungen, soweit diese unter die recherchierten Gebiete fallen

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**C. ALS WESENTLICH ANGESEHENE UNTERLAGEN**

| Kategorie* | Bezeichnung der Veröffentlichung, soweit erforderlich unter Angabe der in Betracht kommenden Teile                                                                             | Betr. Anspruch Nr. |
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Fax: (+31-70) 340-3016

Bevollmächtigter Bediensteter

Swiatek, R

## C.(Fortsetzung) ALS WESENTLICH ANGESEHENE UNTERLAGEN

| Kategorie° | Bezeichnung der Veröffentlichung, soweit erforderlich unter Angabe der in Betracht kommenden Teile                                            | Betr. Anspruch Nr. |
|------------|-----------------------------------------------------------------------------------------------------------------------------------------------|--------------------|
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Angaben zu Veröffentlichungen, die zur selben Patentfamilie gehören

Internationales Aktenzeichen

PCT/EP2005/000853

| Im Recherchenbericht<br>angeführtes Patentdokument |    | Datum der<br>Veröffentlichung | Mitglied(er) der<br>Patentfamilie | Datum der<br>Veröffentlichung |
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| DE 10212400                                        | C1 | 03-04-2003                    | FR 2837216 A1                     | 19-09-2003                    |



**Espacenet**

## Bibliographic data: FR2833504 (A1) — 2003-06-20

**Hot forming of motor vehicle wheel components involves hot stamping of pre-coated hot- or cold-rolled steel sheet**

**Inventor(s):** LAURENT JEAN PIERRE; DERUY SYLVAIN; DEVROC JACQUES ±  
(LAURENT JEAN PIERRE, ; DERUY SYLVAIN, ; DEVROC JACQUES)

**Applicant(s):** USINOR [FR] ± (USINOR)

**Classification:** - **international:** B21D53/26; B21D53/30; (IPC1-7): B21D53/26;  
C21D8/02; C22C38/32; C22C38/38  
- **cooperative:** B21D53/26; B21D53/30; C21D1/673

**Application number:** FR20010016185 20011214

**Priority number(s):** FR20010016185 20011214

### Abstract of FR2833504 (A1)

A method for hot forming, notably to obtain all or part of a motor vehicle wheel, such as, for example, a hub cap, utilizes, in a hot stamping operation, a pre-coated, heat treated, hot- or cold-rolled steel sheet. The composition of the steel sheet is (by weight): 0.15 - 0.5% carbon; 0.5 - 3% manganese; 0.1 - 0.5% silicon; 0 - 1% chromium; less than 0.2% titanium; less than 0.1% aluminum; less than 0.1% phosphorus; less than 0.05% sulfur; and 0.0005 - 0.08% boron. The coating is aluminum-based with some silicon, iron, and possibly zinc.

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## DESCRIPTION FR2833504

<Desc / Clms Page number 1>

Hot forming method and in particular obtaining all or part of a motor vehicle wheel.

The present invention relates to obtaining a workpiece by a hot forming process and in particular obtaining all or part of a motor vehicle wheel.

Currently, the webs of wheels are obtained either by cold stamping of a sheet blank or flow spinning.

In the case of obtaining a web by stamping a sheet blank, it is necessary to perform several shaping operations, which requires the realization of several stamping tools. The number of stamping tools is of the order of 7, 8 or 9.

The main disadvantages of the present technique cited are the following: - Any change in shape requires the machining or manufacture of 7 to 9 pairs of different tools.

- Any change of manufacture causes the disassembly then the assembly and the setting of 7 to 9 tools as well as the setting of the associated presses.

- These many tools must be stored.

- The cost of manufacturing tools does not allow the production of small series in an economical way.

- It is not possible to make thick parts.

In the case of obtaining, for example, a spinning wheel veil, the productivity is reduced compared to the stamping, which limits the production in large series.

In both techniques mentioned: - The use of drawing oil or lubricant is imperative and requires degreasing before painting.

- It is necessary to store and treat these oils as well as the effluents.

From the point of view of the desired characteristics: The increase of the mechanical characteristics leads to a decrease of the formability, which limits the shape of the sails, and generates an increased elastic return, penalizing the dimensional guarantees.

- The increase in mechanical characteristics requires the use of increasingly powerful presses.

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- The temporary protection against corrosion, before painting for example a stamped film, requires oiling the part then degrease it. It is also necessary to remove the stamping oil before painting, which involves a cost and an ecological risk.

The object of the invention is the production of a part by hot stamping, in a single pass, respecting the dimensional guarantees, while having the possibility of obtaining complex shapes.

The object of the invention is to obtain a part by a hot forming process and in particular to obtain all or part of a motor vehicle wheel, for example a wheel cover, characterized in that In a hot stamping operation, a hot-rolled steel sheet for heat treatment, pre-coated, is used.

The other characteristics of the invention are: the steel sheet is a pre-coated hot or cold rolled steel sheet of the following general weight composition: 0, 15% <carbon <0.5% 0.5% <manganese <3% 0, 1% <silicon <0, 5% 0% <chromium <1% titanium <0.2% aluminum <0, 1% phosphorus <0, 1% sulfur <0.05% 0, 0005 % <boron <0.08% the other characteristics of the invention are: - the coating is based on aluminum, - preferably, the steel sheet is a pre-coated hot rolled or cold rolled steel sheet of the following composition by weight: 0.15% <carbon <0.5% 0.8% <manganese <1.5% 0.1% <silicon <0.35% 0% <chromium <1% titanium <0, 1% aluminum <0.1%

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phosphorus <0.05% sulfur <0.03% 0.0005% <boron <0.01% in the weight composition of the sheet, the titanium content with respect to the nitrogen content is greater than 3.42, - the metal bath of the coating comprises, in its basic weight composition, from 9% to 10% of silicon, from 2% to 3.5% of iron, the rest of the bath being composed of aluminum and impurities, - the metal bath the coating comprises in its basic composition by weight of 2% to 4% of iron, the metal bath of the coating may contain in its basic composition by weight, from 0 to 50% of zinc.

The following description will make the invention clear.

The problem is to achieve a part by a hot forming process and in particular obtaining all or part of a motor vehicle wheel such as, for example, a VL wheel or PL wheel (light car or weight heavy) or a wheel cover and a part of the wheel rim, in one pass, respecting the dimensional guarantees, while achieving complex shapes.

It is known from the patent application FR No. 98 08793 a hot and cold steel sheet coated and having a very high resistance after heat treatment. It is also known from the patent application FR No. 98 16477 a method of producing a part from a strip of rolled steel sheet and in particular hot rolled.

According to the invention, a particularly important use of a sheet and the method presented has

been demonstrated, in obtaining a part by a hot forming process and, in particular, obtaining all or part of a wheel of a motor vehicle, in a single hot stamping pass, while the techniques used to date, requires numerous tools, complex, and many operations formatting.

Against known industrial habits, the inventors have obtained, in a single pass, by hot stamping a pre-coated hot rolled steel sheet, a wheel cover, which previously was made in 7 or 8 stamping operations, the pre-cut sheet metal plane having the following general weight composition:

<Desc / Clms Page number 4>

0, 15% <carbon <0, 5% 0, 5% <manganese <3% <img class = "EMIRef" id = "011716631-00040001" />

0, 1% <silicon <0, 5% 0, 01% <chromium <1% titanium <0.2% aluminum <0, 1% phosphorus <0, 1% sulfur <0.05% 0.0005% <boron <0.08 The use of a pre-coated heat treatment steel makes it possible to obtain a notable gain in fatigue resistance without posing a welding problem and exhibiting an increased resistance to corrosion after painting.

In addition, the process and the steel used allow a noticeable lightening and an increase in fatigue life. Finally, the veil obtained can be welded and painted without modification of existing lines.

<img class = "EMIRef" id = "011716631-00040002" />

Preferably, the steel of the sheet has the following weight composition: 0.15% <carbon <0.5% 0.8% <manganese <1.5% 0.1% <silicon <0.35% 0.01 % <chromium <1% titanium <0, 1% aluminum <0.1% phosphorus <0.05% sulfur <0.03% 0.0005% <boron <0.01% the sheet ensuring a very high mechanical strength after heat treatment and aluminum-based coating ensuring high resistance to corrosion and fatigue.

To obtain the part, in a single pass, by hot stamping of the hot rolled coated sheet, it is preferable that the titanium content with respect to the nitrogen content is greater than 3.42.

The coating of the aluminum-based, hot-rolled coated sheet comprises in its basic weight composition from 9% to 10% silicon, from 2% to 3.5% iron.

The coating of the coated hot-rolled sheet may also comprise in its basic weight composition from 2% to 4% iron. The metal bath may contain in its basic composition by weight of 0 to 50% zinc.

In an exemplary embodiment, the steel according to the invention containing 0.231% of carbon; 1.145% manganese; 0.020% phosphorus; 0.0038% sulfur; 0.239% silicon; 0.043% aluminum; 0.009% copper; 0.020% nickel; 0.179% chromium; 0.0042% nitrogen; 0.032% titanium; 0.0028% boron; 0.0051% of calcium in the form of a sheet is coated with an aluminum-based layer having about 25  $\mu\text{m}$  in thickness prior to heat treatment.

The sheet blank is heated in an oven at a temperature between 750°C and 1000 °C and more generally at a temperature between 850 °C and 950°C for a time varying according to the thickness. For a thickness of 2 mm, the sheet blank stays for 6 minutes in the oven. The rate of rise in temperature is greater than 3 °C / s. Due to the coating, it is not necessary to use an inert atmosphere furnace.

During the reheating of the sheet blank, the coating is alloyed with the substrate to form an intermetallic alloy. The alloying causes, by inter-diffusion, an increase in the thickness of the layer which, for the treatment above specified, generally corresponds to an increase of 50%.

The blank of hot sheet is then transferred into the tool cooled or not, depending on the rate, the thickness of the sheet and the target cooling rate, to be shaped. There should be no blank greenhouse pressure to prevent tearing of the sheet due to its low resistance to high temperature. The rate of deformation must be sufficient to avoid deforming the steel at a temperature below 400 °C.

The wheel veil thus obtained is held in the tool until a temperature of less than 200°C and preferably less than 100 °C is reached in order to respect the dimensional guarantees. The average cooling rate must be greater than 27 °C / s, between the outlet temperature of the oven and 400 °C, if the target structure for the substrate is a martensitic structure. If the average cooling rate is less than this value, bainite, ferrite or even perlite may appear. It is thus possible to obtain different structures and therefore different properties depending on the target cooling rate.

Nevertheless, the best

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Fatigue results are obtained with a steel structure, totally martensitic.

Because of the hot forming, there is no damage to the metallurgical structure. This has a beneficial effect on the fatigue resistance due to the elimination of internal defects that can be used to initiate fatigue cracks. It is not necessary to add stamping lubricant because the pre-coating acts as a lubricant.

The piece thus obtained can undergo the painting cycle directly. If this is not the case, it is not necessary to apply temporary protection against corrosion because the alloy layer slows down the appearance of corrosion. During the thermal cycle, the coating, whether or not alloyed, protects the steel from oxidation and decarburization.

The alloy layer produced on the part has a hardness gradient. Starting with  id = "011716631-00060001" />

from the extreme layer and moving towards the sheet, the hardness varies from 750HVO, 1 to 300 HVO, 1 to the interface layer-coating sheet, then increases up to about 500HVO, 1 in the sheet, when the structure Steel is totally martensitic. It is the 300HVO layer, 1 which, by stopping cracks, gives the piece an increased fatigue resistance.

If using the steel of the proposed solution but not pre-coated, there is a significant degradation of average endurance limit, as shown in Table 1 below mainly related to the decarburization of steel. The proposed solution, because of the presence of pre-protection, avoids this phenomenon. The fatigue tests in alternating plane flexion show a noticeable gain.

Table 1.  id = "011716631-00060002" />

Steel RpO. 2 Rm Number of cycles of Limit of endurance (MPa) (MPa) validation of the Average (MPa) Travers Travers limitation of the limit of endurance <SEP> (millions of cycles) USIDUR 355 example of 432 514 1. 106 279 comparison USIFORM 800 example 766 872 2.106 388 from comparison <tb> Obtaining according to 980 1490 2.106 249 the invention, no pre coated Obtaining according to < SEP> 980 1480 2. 106 439 the invention

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Compared to the USIDUR 355 sheet, commonly used industrially for the production of wheel sails, the gain is 40% in life or lightening.

Compared to the mentioned grade used for the wheel sails, the endurance limit has a gain of more than 12%.

The advantages of obtaining a part by a hot forming process and in particular obtaining all or part of a wheel of a motor vehicle are the following: a realization of a wheel sail in a single pass of forming, - because of the important formability to hot, only one tool is necessary. it is therefore possible to make parts of complex shapes in series economically such as the wheels of style, - a very good dimensional guarantee because there is no springback as in the case of cold stamping or flow spinning, - the high mechanical characteristics after quenching in the stamping tool allow significant lightening, - the pre-coating ensures an increased guarantee against corrosion and wear, - an increased formability during the heating of the blank of sheet metal makes it possible to heat-form thicknesses that can usually be shaped only by spinning, - deformation can take place in the austenitic phase when hot, without damage, which guarantees a better resistance to fatigue by eliminating internal defects generally created during a cold deformation, - because of the pre-coating, it is useless to lubricate the sheet during hot forming, thus eliminating i the degreasing step before painting, - thanks to the coating, it is useless to apply a temporary protection against corrosion on the parts waiting to paint, - the pre-coating not heat treated, unlike coatings based on zinc, does not degrade the fatigue strength and even increases it, - the impact behavior is reinforced by the use of the selected type of steel, - the heat treated pre-coating does not require special welding equipment and precaution.

Other parts can benefit from this method of obtaining. Mention may in particular be made of the ground connection parts dimensioned in fatigue such as: the suspension arms, wheel arms, etc.



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## CLAIMS FR2833504

### CLAIMS.

1.

Hot forming method and in particular, obtaining all or part of a motor vehicle wheel such as a wheel cover, characterized in that, in a hot stamping operation, a sheet metal is used. hot rolled or cold rolled steel for pre-coated heat treatment.

2.

Process according to Claim 1, characterized in that the steel sheet is a hot-rolled steel sheet pre-coated with the following general weight composition: 0.15% <carbon <0.5% 0.5% <manganese <3% 0, 1% <silicon <0, 5% 0% <chromium <1% titanium <0.2% aluminum <0.1% phosphorus <0.1% sulfur <0.05% 0.0005% < boron <0.08% 3.

Process according to Claim 1, characterized in that the coating is based on aluminum.

4.

Process according to Claim 1, characterized in that the steel sheet is preferably a hot rolled or cold rolled steel sheet pre-coated with the following composition by weight: 0.15% <carbon <0.5% 0, 8% <manganese <1.5% 0, 1% <silicon <0, 35% 0.01% <chromium <1% titanium <0.1% aluminum <0.1% phosphorus <0.05% sulfur < 0.03% 0.0005% <boron <0.01%

5.

Process according to Claim 1, characterized in that, in the weight composition of the sheet, the titanium content with respect to the nitrogen content is greater than 3.42.

6.

Process according to Claim 1, characterized in that the metal bath of the coating comprises, in its basic weight composition, from 9% to 10% of silicon, from 2% to 3.5% of iron, the rest of the bath being composed of aluminum and impurities.

7.

Process according to Claim 1, characterized in that the metal bath of the coating comprises in its basic weight composition from 2% to 4% of iron.

8.

Process according to Claim 1, characterized in that the metal bath of the coating may contain, in its basic weight composition, from 0 to 50% of zinc.

⑫

DEMANDE DE BREVET D'INVENTION

A1

②② Date de dépôt : 14.12.01.

③⑦ Priorité :

④③ Date de mise à la disposition du public de la  
demande : 20.06.03 Bulletin 03/25.

⑤⑥ Liste des documents cités dans le rapport de  
recherche préliminaire : *Se reporter à la fin du  
présent fascicule*

⑥⑦ Références à d'autres documents nationaux  
apparentés :

⑦① Demandeur(s) : *USINOR Société anonyme* — FR.

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⑦③ Titulaire(s) :

⑦④ Mandataire(s) :

⑤④ PROCÉDE DE MISE EN FORME A CHAUD ET NOTAMMENT L'OBTENTION DE TOUT OU PARTIE D'UNE  
ROUE DE VEHICULE AUTOMOBILE.

⑤⑦ Procédé de mise en forme à chaud et notamment, ob-  
tention de tout ou partie d'une roue de véhicule automobile  
comme par exemple un voile de roue, caractérisé en ce  
qu'on utilise, dans une opération d'emboutissage à chaud,  
une tôle d'acier laminée à chaud pour traitement thermique  
pré revêtu.

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**Procédé de mise en forme à chaud et notamment l'obtention de tout ou partie d'une roue de véhicule automobile.**

La présente invention concerne l'obtention d'une pièce par un procédé de mise en forme à chaud et notamment l'obtention de tout ou partie d'une roue de véhicule automobile.

Actuellement, les voiles de roues sont obtenus soit par emboutissage à froid d'un flan de tôle soit par fluotournage.

Dans le cas de l'obtention d'un voile par emboutissage d'un flan de tôle, il est nécessaire de réaliser plusieurs opérations de mise en forme, ce qui impose la réalisation de plusieurs outils d'emboutissage. Le nombre d'outils d'emboutissage est de l'ordre de 7, 8 ou 9.

Les principaux inconvénients de la présente technique citée sont les suivants :

- Tout changement de forme nécessite l'usinage ou la fabrication de 7 à 9 paires d'outils différents.
- Tout changement de fabrication occasionne le démontage puis le montage et le réglage de 7 à 9 outils ainsi que le réglage des presses associées.
- Ces nombreux outils doivent être stockés.
- Le coût de fabrication des outils ne permet pas la réalisation de petites séries de manière économique.
- Il n'est pas possible de réaliser des pièces de forte épaisseur.

Dans le cas de l'obtention, par exemple, d'un voile de roue par fluotournage, la productivité est réduite comparativement à l'emboutissage, ce qui limite la fabrication en grandes séries.

Dans les deux techniques citées :

- L'utilisation d'huile d'emboutissage ou de lubrifiant est impératif et nécessite un dégraissage avant mise en peinture.
- Il faut stocker et traiter ces huiles ainsi que les effluents.

Du point de vue des caractéristiques recherchées :

- L'augmentation des caractéristiques mécaniques entraîne une diminution de la formabilité, ce qui limite la forme des voiles, et génère un retour élastique accru, pénalisant les garanties dimensionnelles.
- L'augmentation des caractéristiques mécaniques impose d'utiliser des presses de plus en plus puissantes.

- La protection temporaire contre la corrosion, avant peinture par exemple d'un voile embouti, nécessite d'huiler la pièce puis de la dégraisser. Il est également nécessaire de retirer l'huile d'emboutissage avant peinture, ce qui comporte un coût et un risque écologique.

Le but de l'invention est la réalisation d'une pièce par emboutissage à chaud, en une seule passe, en respectant les garanties dimensionnelles, tout en ayant la possibilité d'obtenir des formes complexes.

L'invention a pour objet l'obtention d'une pièce par un procédé de mise en forme à chaud et notamment l'obtention de tout ou partie d'une roue de véhicule automobile comme par exemple un voile de roue, caractérisée en ce qu'on utilise, dans une opération d'emboutissage à chaud, une tôle d'acier laminée à chaud pour traitement thermique, pré revêtue.

Les autres caractéristiques de l'invention sont :

- la tôle d'acier est une tôle d'acier laminée à chaud ou à froid pré revêtue de composition générale pondérale suivante :

0,15% < carbone < 0,5%

0,5% < manganèse < 3%

0,1% < silicium < 0,5%

0% < chrome < 1%

titane < 0,2%

aluminium < 0,1%

phosphore < 0,1%

soufre < 0,05%

0,0005% < bore < 0,08%

les autres caractéristiques de l'invention sont :

- le revêtement est à base d'aluminium,
- de préférence, la tôle d'acier est une tôle d'acier laminée à chaud ou à froid pré revêtue de la composition pondérale suivante :

0,15% < carbone < 0,5%

0,8% < manganèse < 1,5%

0,1% < silicium < 0,35%

0% < chrome < 1%

titane < 0,1%

aluminium < 0,1%

phosphore < 0,05%

soufre < 0,03%

0,0005% < bore < 0,01%

- dans la composition pondérale de la tôle, la teneur en titane par rapport à la teneur en azote est supérieure à 3,42,
- le bain métallique du revêtement comprend dans sa composition pondérale de base, de 9% à 10% de silicium, de 2% à 3,5% de fer, le reste du bain étant composé d'aluminium et des impuretés,
- le bain métallique du revêtement comprend dans sa composition pondérale de base de 2% à 4% de fer,
- le bain métallique du revêtement peut contenir dans sa composition pondérale de base, de 0 à 50% de zinc.

La description qui suit fera bien comprendre l'invention.

Le problème posé est de réaliser une pièce par un procédé de mise en forme à chaud et notamment l'obtention de tout ou partie d'une roue de véhicule automobile comme par exemple, un voile de roue de VL ou PL (voiture légère ou poids lourd) ou un voile de roue et une partie de la jante de roue, en une seule passe, en respectant les garanties dimensionnelles, tout en réalisant des formes complexes.

Il est connu de la demande de brevet FR N° 98 08793 une tôle d'acier à chaud et à froid revêtue et comportant une très haute résistance après traitement thermique. Il est également connu de la demande de brevet FR N° 98 16477 un procédé de réalisation d'une pièce à partir d'une bande de tôle d'acier laminée et notamment laminée à chaud.

Selon l'invention, une utilisation particulièrement importante d'une tôle et du procédé présentés a été démontrée, dans l'obtention d'une pièce par un procédé de mise en forme à chaud et, notamment, l'obtention de tout ou partie d'une roue de véhicule automobile, en une seule passe d'emboutissage à chaud, alors que les techniques utilisées à ce jour, demande des outillages nombreux, complexes, ainsi que des opérations nombreuses de mise en forme.

A l'encontre des habitudes industrielles connues, les inventeurs ont obtenu, en une seule passe, par emboutissage à chaud d'une tôle d'acier laminée à chaud pré revêtu, un voile de roue, qui antérieurement, était réalisé en 7 ou 8 opérations d'emboutissage, le plan de tôle prédécoupé ayant la composition pondérale générale suivante :

0,15% < carbone < 0,5%  
 0,5% < manganèses < 3%  
 0,1% < silicium < 0,5%  
 0,01% < chrome < 1%  
 titane < 0,2%  
 aluminium < 0,1%  
 phosphore < 0,1%  
 soufre < 0,05%  
 0,0005% < bore < 0,08%

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10 L'utilisation d'un acier pour traitement thermique pré revêtu permet d'obtenir un gain notable en résistance à la fatigue sans poser de problème de soudage et en présentant une résistance accrue à la corrosion après peinture.

De plus, le procédé et l'acier utilisé permettent un allègement notable et une augmentation de la durée de vie en fatigue. Enfin, le voile obtenu peut être soudé puis  
 15 peint sans modification des lignes existantes.

De préférence l'acier de la tôle a la composition pondérale suivante :

0,15% < carbone < 0,5%  
 0,8% < manganèses < 1,5%  
 0,1% < silicium < 0,35%  
 0,01% < chrome < 1%  
 titane < 0,1%  
 aluminium < 0,1%  
 phosphore < 0,05%  
 soufre < 0,03%  
 0,0005% < bore < 0,01%

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la tôle assurant une très haute résistance mécanique après traitement thermique et le revêtement à base d'aluminium assurant une haute tenue à la corrosion et à la fatigue.

Pour l'obtention de la pièce, en une seule passe, par emboutissage à chaud de la tôle laminée à chaud revêtue, il est préférable que la teneur en titane par rapport à la  
 30 teneur en azote soit supérieure à 3,42.

Le revêtement de la tôle laminée à chaud revêtue, à base d'aluminium, comprend dans sa composition pondérale de base, de 9% à 10% de silicium, de 2% à 3,5% de fer.

Le revêtement de la tôle laminée à chaud revêtue peut également comprendre dans sa composition pondérale de base, de 2% à 4% de fer. Le bain métallique peut contenir dans sa composition pondérale de base de 0 à 50% de zinc.

Dans un exemple de réalisation, l'acier selon l'invention contenant 0,231% de carbone ; 1,145% de manganèse ; 0,020% de phosphore ; 0,0038% de soufre ; 0,239% de silicium ; 0,043% d'aluminium ; 0,009% de cuivre ; 0,020% de nickel ; 0,179% de chrome ; 0,0042% d'azote ; 0,032% de titane ; 0,0028% de bore ; 0,0051% de calcium, sous la forme d'une tôle est revêtue d'une couche à base d'aluminium ayant environ 25  $\mu\text{m}$  d'épaisseur avant traitement thermique.

Le flan de tôle est chauffé dans un four, à une température comprise entre 750°C et 1000°C et plus généralement à une température comprise entre 850°C et 950°C pendant un temps variant en fonction de l'épaisseur. Pour une épaisseur de 2 mm, le flan de tôle séjourne pendant 6 minutes dans le four. La vitesse de montée en température est supérieure à 3°C/s. Du fait du revêtement, il n'est pas nécessaire d'utiliser un four à atmosphère inerte.

Lors du réchauffage du flan de tôle, le revêtement s'allie avec le substrat pour former un alliage intermétallique. L'alliation entraîne, par inter diffusion, une augmentation de l'épaisseur de la couche qui, pour le traitement ci-dessus spécifié, correspond généralement à une augmentation de 50%.

Le flan de tôle chaud est ensuite transféré dans l'outil refroidi ou non, en fonction de la cadence, de l'épaisseur de la tôle et de la vitesse de refroidissement visée, pour être mis en forme. Il ne doit pas y avoir de pression de serre flan afin d'éviter le déchirement de la tôle dû à sa faible résistance à haute température. La vitesse de déformation doit être suffisante pour éviter de déformer l'acier à une température inférieure à 400°C.

Le voile de roue ainsi obtenu est maintenu dans l'outil jusqu'à atteindre une température inférieure à 200°C et de préférence inférieure à 100°C afin de respecter les garanties de dimension. La vitesse de refroidissement moyenne doit être supérieure à 27°C/s, entre la température de sortie du four et 400°C, si la structure visée pour le substrat est une structure martensitique. Si la vitesse moyenne de refroidissement est inférieure à cette valeur, de la bainite, de la ferrite, voire de la perlite peuvent apparaître. Il est ainsi possible d'obtenir différentes structures et donc différentes propriétés en fonction de la vitesse de refroidissement visée. Néanmoins, les meilleurs

résultats en fatigue sont obtenus avec une structure de l'acier, totalement martensitique.

Du fait du formage à chaud, il n'y a pas d'endommagement de la structure métallurgique. Ceci a un effet bénéfique sur la tenue en fatigue du fait de la suppression des défauts internes pouvant servir d'initiation des fissures en fatigue. Il n'est pas nécessaire d'ajouter de lubrifiant d'emboutissage car le pré revêtement fait office de lubrifiant.

La pièce ainsi obtenue peut subir directement le cycle de peinture. Si ce n'est pas le cas, il n'est pas nécessaire d'appliquer une protection temporaire contre la corrosion car la couche alliée ralentit l'apparition de la corrosion. Lors du cycle thermique, le revêtement, alliée ou non, protège l'acier de l'oxydation et de la décarburation.

La couche alliée réalisée sur la pièce présente un gradient de dureté. En partant de la couche extrême et en se déplaçant vers la tôle, la dureté varie de 750HV0,1 à 300 HV0,1 à l'interface couche de revêtement - tôle, puis augmente jusqu'à environ 500HV0,1 dans la tôle, lorsque la structure de l'acier est totalement martensitique. C'est la couche à 300HV0,1 qui, en arrêtant les fissures, confère à la pièce une tenue en fatigue accrue.

Si on utilise l'acier de la solution proposée mais non pré revêtu, on note une dégradation importante de limite d'endurance moyenne, comme matérialisé sur le tableau 1 ci-dessous liée principalement à la décarburation de l'acier. La solution proposée, de part la présence de la pré protection, évite ce phénomène. Les essais de fatigue en flexion plane alternée montre un gain notable.

Tableau 1.

| Acier                                              | Rp0.2<br>(MPa)<br>Travers | Rm<br>(MPa)<br>Travers | Nombre de cycles de<br>validation de la<br>limitation de la limite<br>d'endurance (millions<br>de cycles) | Limite d'endurance<br>Moyenne (MPa) |
|----------------------------------------------------|---------------------------|------------------------|-----------------------------------------------------------------------------------------------------------|-------------------------------------|
| USIDUR 355 exemple de<br>comparaison               | 432                       | 514                    | 1. 106                                                                                                    | 279                                 |
| USIFORM 800 exemple<br>de comparaison              | 766                       | 872                    | 2. 106                                                                                                    | 388                                 |
| Obtention selon<br>l'invention, non pré<br>revêtue | 980                       | 1490                   | 2. 106                                                                                                    | 249                                 |
| Obtention selon<br>l'invention                     | 980                       | 1480                   | 2. 106                                                                                                    | 439                                 |

Par rapport à la tôle USIDUR 355, couramment utilisé industriellement pour la réalisation des voiles de roues, le gain est de 40% en durée de vie ou en allègement. Par rapport à la nuance citée, utilisée pour les voiles de roues, la limite d'endurance présente un gain supérieur à 12%.

5 Les avantages de l'obtention d'une pièce par un procédé de mise en forme à chaud et notamment l'obtention de tout ou partie d'une roue de véhicule automobile sont les suivants :

- une réalisation de voile de roue en une seule passe de formage,
- du fait de la formabilité importante à chaud, un seul outil est nécessaire. il est donc possible de faire des pièces de formes complexes en série de manière économique  
10 telles que les roues de style,
- une très bonne garantie dimensionnelle car il n'y a pas de retour élastique comme dans le cas de l'emboutissage à froid ou du fluotournage,
- les caractéristiques mécaniques élevées après trempe dans l'outil d'emboutissage  
15 permettent un allègement significatif,
- le pré revêtement assure une garantie accrue à la corrosion et à l'usure,
- une formabilité accrue lors de la chauffe du flan de tôle permet de former à chaud des épaisseurs ne pouvant habituellement être mise en forme que par fluotournage,
- une déformation peut avoir lieu dans la phase austénitique à chaud, sans endommagement, ce qui garantie une meilleure tenue à la fatigue par élimination  
20 des défauts internes généralement créés lors d'une déformation à froid,
- du fait du pré revêtement, il est inutile de lubrifier la tôle lors du formage à chaud, supprimant ainsi l'étape de dégraissage avant peinture,
- grâce au revêtement, il est inutile d'appliquer une protection temporaire contre la corrosion sur les pièces en attente de peinture,  
25
- le pré revêtement non traité thermiquement, contrairement aux revêtements à base de zinc, ne dégrade pas la tenue à la fatigue et même l'augmente,
- le comportement aux chocs est renforcé par l'utilisation du type d'acier choisi,
- le pré revêtement traité thermiquement ne nécessite pas de matériel de soudage et  
30 de précaution particuliers.

D'autres pièces peuvent bénéficier de cette méthode d'obtention. On peut citer en particulier les pièces de liaison au sol dimensionnées en fatigue telles que : les bras de suspension, bras de roue...

## REVENDECATIONS.

1. Procédé de mise en forme à chaud et notamment, obtention de tout ou partie d'une  
roue de véhicule automobile comme par exemple un voile de roue, caractérisé en ce  
5 qu'on utilise, dans une opération d'emboutissage à chaud, une tôle d'acier laminée à  
chaud ou à froid pour traitement thermique pré revêtu.
2. Procédé selon la revendication 1, caractérisé en ce que la tôle d'acier est une tôle  
d'acier laminée à chaud pré revêtu de la composition générale pondérale suivante :
- 10 0,15% < carbone < 0,5%  
0,5% < manganèse < 3%  
0,1% < silicium < 0,5%  
0% < chrome < 1%  
titane < 0,2%  
15 aluminium < 0,1%  
phosphore < 0,1%  
soufre < 0,05%  
0,0005% < bore < 0,08%
- 20 3. Procédé selon la revendication 1, caractérisé en ce que le revêtement est à base  
d'aluminium.
4. Procédé selon la revendication 1, caractérisé en ce que de préférence, la tôle  
d'acier est une tôle d'acier laminée à chaud ou à froid pré revêtue de la composition  
25 pondérale suivante :
- 0,15% < carbone < 0,5%  
0,8% < manganèse < 1,5%  
0,1% < silicium < 0,35%  
0,01% < chrome < 1%  
30 titane < 0,1%  
aluminium < 0,1%  
phosphore < 0,05%  
soufre < 0,03%  
0,0005% < bore < 0,01%

5. Procédé selon la revendication 1, caractérisé en ce que dans la composition pondérale de la tôle, la teneur en titane par rapport à la teneur en azote est supérieure à 3,42.

5

6. Procédé selon la revendication 1, caractérisé en ce que le bain métallique du revêtement comprend dans sa composition pondérale de base, de 9% à 10% de silicium, de 2% à 3,5% de fer, le reste du bain étant composé d'aluminium et des impuretés.

10

7. Procédé selon la revendication 1, caractérisé en ce que le bain métallique du revêtement comprend dans sa composition pondérale de base, de 2% à 4% de fer.

8. Procédé selon la revendication 1, caractérisé en ce que le bain métallique du revêtement peut contenir dans sa composition pondérale de base, de 0 à 50% de zinc.

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N° d'enregistrement  
national

# RAPPORT DE RECHERCHE PRÉLIMINAIRE

établi sur la base des dernières revendications  
déposées avant le commencement de la recherche

FA 611824  
FR 0116185

| DOCUMENTS CONSIDÉRÉS COMME PERTINENTS                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |                                                                                                 | Revendication(s)<br>concernée(s) | Classement attribué<br>à l'invention par l'INPI      |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------|----------------------------------|------------------------------------------------------|
| Catégorie                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          | Citation du document avec indication, en cas de besoin,<br>des parties pertinentes              |                                  |                                                      |
| D,X                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                | FR 2 780 984 A (LORRAINE LAMINAGE)<br>14 janvier 2000 (2000-01-14)<br>* le document en entier * | 1-8                              | B21D53/26<br>C21D8/02<br>C22C38/32<br>C22C38/38      |
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|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |                                                                                                 |                                  | <b>DOMAINES TECHNIQUES<br/>RECHERCHÉS (Int.CL.7)</b> |
|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |                                                                                                 |                                  | B21D                                                 |
| Date d'achèvement de la recherche                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |                                                                                                 | Examineur                        |                                                      |
| 14 août 2002                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       |                                                                                                 | Peeters, L                       |                                                      |
| <b>CATÉGORIE DES DOCUMENTS CITÉS</b><br>X : particulièrement pertinent à lui seul<br>Y : particulièrement pertinent en combinaison avec un autre document de la même catégorie<br>A : arrière-plan technologique<br>O : divulgation non-écrite<br>P : document intercalaire<br>T : théorie ou principe à la base de l'invention<br>E : document de brevet bénéficiant d'une date antérieure à la date de dépôt et qui n'a été publié qu'à cette date de dépôt ou qu'à une date postérieure.<br>D : cité dans la demande<br>L : cité pour d'autres raisons<br>& : membre de la même famille, document correspondant |                                                                                                 |                                  |                                                      |

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EPO FORM 1503 12.99 (P04C14)

**ANNEXE AU RAPPORT DE RECHERCHE PRÉLIMINAIRE**  
**RELATIF A LA DEMANDE DE BREVET FRANÇAIS NO. FR 0116185 FA 611824**

La présente annexe indique les membres de la famille de brevets relatifs aux documents brevets cités dans le rapport de recherche préliminaire visé ci-dessus.  
 Les dits membres sont contenus au fichier informatique de l'Office européen des brevets à la date du **14-08-2002**  
 Les renseignements fournis sont donnés à titre indicatif et n'engagent pas la responsabilité de l'Office européen des brevets, ni de l'Administration française

| Document brevet cité<br>au rapport de recherche | Date de<br>publication | Membre(s) de la<br>famille de brevet(s) | Date de<br>publication |
|-------------------------------------------------|------------------------|-----------------------------------------|------------------------|
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EPO FORM P0485

Pour tout renseignement concernant cette annexe : voir Journal Officiel de l'Office européen des brevets, No.12/82



**Espacenet**

## Bibliographic data: EP1013785 (A1) — 2000-06-28

Process for manufacturing of a part from a hot-rolled sheet

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- cooperative: C21D1/673; C21D9/48; C23C2/12; C23C2/26;  
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**Also published as:** EP1013785 (B1) EP1013785 (B2) AT343658 (T)  
DE69933751 (T2) DE69933751 (T3) more

### Abstract of EP1013785 (A1)

A component is produced from a strip of rolled (especially hot-rolled) sheet steel, by coating with aluminum or its alloy and pressing operations. The process involves: (a) coating the rolled sheet with a metal or an alloy assuring protection of the surface and the steel; (b) raising the temperature of the coated sheet for forming; (c) producing, at the surface, an intermetallic alloy composite assuring protection against corrosion and decarburization of the steel, with the intermetallic composite able to assure a lubrication function; (d) forming the coated sheet, notably by pressing; (e) cooling the formed component in order to confer increased hardness of the steel and increased surface hardness in the coating. The metal used for coating the steel sheet is aluminum or an aluminum alloy, so that the intermetallic alloy composite is made up of aluminum, iron and silicon.

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## DESCRIPTION EP1013785

[0001]

The invention relates to a method of producing a part from a strip of rolled steel sheet and in particular hot rolled.

[0002]

Steel sheets to be subjected to high temperature forming and / or heat treatment are not delivered coated for coating considerations during heat treatment. The coating is therefore performed on finished part which requires a careful cleaning of surfaces and hollow parts. This cleaning requires the use of acids and / or bases whose recycling and storage are a significant financial burden and present risks for operators and the environment. In addition, the heat treatment must be carried out under a controlled atmosphere in order to avoid decarburization and oxidation of the steel. Then, in the case of hot forming, calamine, by virtue of its abrasive power, damages the forming tools, which reduces the quality of the parts obtained from the dimensional and aesthetic point of view, or forces frequent and expensive tool repairs. Finally, to increase their resistance to corrosion, the parts thus obtained must receive an expensive post-processing whose application is difficult or impossible, especially in the case of parts with depressions.

[0003]

The object of the invention is to propose sheet steel rolled from 0.2 mm to about 20 mm thick, coated in particular after hot rolling, and to be shaped, either hot or cold followed by a heat treatment, the temperature rise to be ensured without decarburization of the steel of the sheet,

without oxidation of the surface of said sheet, before, during and after hot forming and or heat treatment .

[0004]

The subject of the invention is a process for producing a part from a strip of rolled steel sheet, in particular hot rolled, which is characterized in that: the sheet metal coated with a metal or a metal alloy providing a protection of the surface and steel, the coated sheet is subjected to a temperature rise for its shaping, thereby producing an intermetallic alloy compound, on the surface, providing protection against the corrosion, against the decarburization of the steel, the intermetallic compound being able to provide a lubricating function, the shaping is carried out in particular by stamping, the formed part is cooled to impart high mechanical hardness characteristics of the steel and a high surface hardness of the coating.

[0005]

The other characteristics of the invention are: the hot rolled sheet is coated after hot rolling and pickling, the metal or the metal coating alloy is aluminum or an aluminum alloy. the intermetallic alloy is a compound based mainly on aluminum, iron and silicon, the coating of metal or metal alloy and carried out by hot dipping. the coated sheet is subjected to a temperature rise above 700 ° C before shaping and or heat treatment, the coated sheet is hot stamped. the piece obtained in particular by stamping is cooled to undergo quenching at a speed greater than the critical quenching speed.

[0006]

The invention also relates to a hot-rolled sheet including a surface-deposited metal or metal alloy coating, for hot-forming an intermetallic compound, the intermetallic compound providing, a protection against corrosion, a barrier against the decarburization of the steel of the sheet, a lubrication during the hot shaping of the sheet, and on the finished part, after shaping and cooling, the mechanical properties of hardness of the high steel as well as a high surface hardness of the surface of said finished part.

[0007]

Another feature of the invention is: the sheet has a surface deposit of aluminum or an aluminum alloy.

[0008]

The invention also relates to the use of the sheet in the hot forming, in particular by stamping parts with high mechanical properties in hardness and high surface hardness characteristics ensuring very good resistance to abrasion.

The following description will make the invention clear.

[0009]

The process according to the invention consists in coating a steel, especially a hot-rolled steel, for heat treatment and hot forming, with a metal or a metal alloy such as aluminum or an aluminum alloy composed, for example, of aluminum and silicon.

The metal or metal alloy coating is selected to have corrosion protection and to withstand a high temperature.

During a heat treatment, or a temperature rise for forming, the coating forms an alloy layer having a high resistance to corrosion, abrasion, wear and fatigue.

The coating does not alter the formability properties of the steel and thus allows a wide variety of cold and hot forming.

[0010]

After hot rolling the strip can be pickled and cold rolled before being coated.

In the case where the sheet is cold rolled, it can be annealed before being coated.

[0011]

The laminated sheet can be coated with, for example, the following two types of metal alloy: Alloy No. 1: Si: 9% to 10%; Fe: 2% to 3.5%; Balance: Al and impurities. Alloy No. 2: Fe: 2% to 4%; Balance: Al and impurities. For alloy No. 1 the coating thickness is between 5 microns and 100 microns, and preferably between 10 microns and 25 microns. For alloy No. 2 the coating thickness is between 15 microns and 100 microns, and preferably between 30 microns and 50 microns.

[0012]

For shaping or heat treatment, the sheet is subjected to a temperature rise preferably between 750 ° C and 1200 ° C in an oven with an atmosphere no longer requiring control because of the barrier to oxidation formed by the coating. When raising the temperature, the aluminum-based coating is transformed into an alloyed surface layer having different phases depending on the temperature treatment and having a high hardness of more than 600 HV 100g.

[0013]

According to the process of the invention, it is possible to produce sheets having a thickness of between 0.2 mm and 20 mm, having good shaping properties as well as good resistance to corrosion.

[0014]

The sheets delivered coated, have a significant resistance to corrosion during temperature rises, shaping, heat treatments, and during the use of finished formed parts.

[0015]

The presence of the coating during heat treatments or hot forming avoids in addition to corrosion, the decarburization of the base steel.

This has an undeniable advantage in the case of hot forming, for example in a stamping press.

Indeed, the formed intermetallic alloy avoids the formation of calamine, the wear of the tools by calamine, and thereby allows an extension of the average life of said tools. It has been noticed that the hot formed intermetallic alloy has a high temperature lubricant function. In addition, the

effect of protection against decarburization of the intermetallic alloy allows the use of high temperature furnace exceeding 900 ° C having an uncontrolled atmosphere, and this, even for heating times of several hours.

[0016]

Out of the oven, it is no longer necessary to strip the piece obtained, resulting in a saving due to the removal of the pickling bath of finished parts.

[0017]

Due to the characteristics of the coating after temperature rise, the resulting pieces have increased resistance to fatigue, wear, abrasion and corrosion.

In addition, the coating is weldable before and after temperature rise.

[0018]

The steel of the sheet ensures, by cooling quenching effect, high mechanical characteristics of the part obtained after forming, the coating converted into a hot intermetallic alloy, for its part, because of its lubricant qualities and resistance to friction, improved shaping, especially in the field of hot stamping.

[0019]

In one exemplary embodiment, a strip of hot-rolled steel sheet with the following weight composition is used: carbon: 0.15% to 0.25%, manganese: 0.8% to 1.5%, silicon: 0, 1% to 0.35%, chromium: 0.01% to 0.2%, titanium: less than 0.1%, aluminum: less than 0.1%, phosphorus: less than 0.05%, sulfur: less of 0.03%, boron: 0.0005% to 0.01%, the sheet metal strip is coated with a coating of alloy type No. 1 or 2, the sheet is austenitized at 950 ° C. before forming and quenched in the tool, the coating providing a role of lubricant during shaping, in addition to its protection functions against cold corrosion, hot and against decarburization.

During quenching, the alloy coating does not interfere with the extraction of heat by the tool and can promote it. After forming and quenching, it is no longer necessary to strip the part or to protect

it, the base coating providing protection throughout the process.

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## CLAIMS EP1013785

1.

Process for producing a workpiece from a strip of rolled steel sheet, in particular hot-rolled steel sheet, characterized in that: - the rolled metal sheet is coated with a metal or a metal alloy providing protection against the surface and steel, - the coated sheet is subjected to an elevation in temperature for its shaping, - is thus realized an intermetallic alloy compound, surface, providing protection against corrosion, against decarburization of steel, the intermetallic compound being able to provide a lubricating function, the forming is carried out in particular by stamping, the part formed is cooled to give high mechanical hardness characteristics of the steel and a high surface hardness of coating.

2.

Process according to Claim 1, characterized in that the hot-rolled sheet is coated immediately after hot rolling,

3.

Process according to claim 1 characterized in that the metal or metal coating alloy is aluminum or an aluminum alloy.

4.

Process according to Claim 1, characterized in that the intermetallic alloy is a compound based on aluminum, iron and silicon.

5.

Process according to Claim 1, characterized in that the coating of metal or metal alloy is carried out under hot dipping.

6.

A method according to claim 1 characterized in that the coated sheet is subjected to a temperature rise above 700 ° C before shaping and / or heat treatment.

7.

Process according to Claim 1, characterized in that the coated sheet is hot-drawn.

8.

Process according to Claim 1, characterized in that the part obtained, in particular by stamping, is cooled to undergo quenching at a speed greater than the critical quenching speed.

9.

Rolled sheet, especially when hot, comprising a surface-deposited metal or metal alloy coating for hot-forming an intermetallic compound, the intermetallic compound providing, a protection against corrosion, a barrier against the decarburization of steel of sheet metal, lubrication during the hot shaping of the sheet, and on the finished part, after shaping and cooling, the high mechanical hardness characteristics of the steel and a high surface hardness of the surface of said finished part.

10.

Hot-rolled sheet according to claim 9, characterized in that it comprises a surface deposit of aluminum or an aluminum alloy.

11.

Use of the sheet according to claims 9 or 10 in the hot forming, in particular by stamping parts with high mechanical properties in hardness and high surface hardness characteristics ensuring a very good resistance to abrasion.

(19)



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**EP 1 013 785 A1**

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(54) **Procédé de réalisation d'une pièce à partir d'une bande de tôle d'acier laminée et notamment laminée à chaud**

(57) Procédé de réalisation d'une pièce à partir d'une bande de tôle d'acier laminée et notamment laminée à chaud, caractérisée en ce que:

- on revêt la tôle laminée d'un métal ou d'un alliage métallique assurant une protection de la surface et de l'acier,
- on soumet la tôle revêtue à une élévation en température pour sa mise en forme,

- on réalise, de ce fait un composé allié intermétallique, en surface, assurant une protection contre la corrosion, contre la décarburation de l'acier, le composé intermétallique pouvant assurer une fonction de lubrification,
- on effectue la mise en forme notamment par emboutissage,
- on refroidit la pièce formée pour conférer des caractéristiques mécaniques de dureté élevées de l'acier et une dureté superficielle élevée du revêtement.

**EP 1 013 785 A1**

**Description**

[0001] L'invention concerne un procédé de réalisation d'une pièce à partir d'une bande de tôle d'acier laminée et notamment laminée à chaud.

[0002] Les tôles en acier devant subir un formage à haute température et ou un traitement thermique ne sont pas livrées revêtues pour des considérations de tenue du revêtement lors du traitement thermique. Le revêtement est donc effectué sur pièce finie ce qui nécessite un nettoyage soigné des surfaces et des parties creuses. Ce nettoyage requiert l'utilisation d'acides et ou de bases dont le recyclage et le stockage sont une charge financière importante et présentent des risques pour les opérateurs et pour l'environnement. De plus, le traitement thermique doit être effectué sous atmosphère contrôlée afin d'éviter toute décarburation et oxydation de l'acier. Ensuite, dans le cas du formage à chaud, la calamine, de par son pouvoir abrasif, endommage les outils de mise en forme, ce qui diminue la qualité des pièces obtenues du point de vue dimensionnel et esthétique, ou oblige à de fréquentes et coûteuses réparations d'outils. Enfin, pour augmenter leur résistance à la corrosion, les pièces ainsi obtenues doivent recevoir un post traitement coûteux dont l'application est difficile, voire impossible, en particulier dans le cas de pièces comportant des creux.

[0003] Le but de l'invention est de proposer des tôles d'acier laminées de 0,2 mm à environ 20 mm d'épaisseur, revêtues notamment après laminage à chaud, et devant subir une mise en forme, soit à chaud, soit à froid suivie d'un traitement thermique, l'élévation de température devant être assurée sans décarburation de l'acier de la tôle, sans oxydation de la surface de ladite tôle, avant, pendant et après la mise en forme à chaud et ou le traitement thermique.

[0004] L'invention a pour objet un procédé de réalisation d'une pièce à partir d'une bande de tôle d'acier laminée et notamment laminée à chaud et qui se caractérise en ce que:

- on revêt la tôle laminée d'un métal ou d'un alliage métallique assurant une protection de la surface et de l'acier,
- on soumet la tôle revêtue à une élévation en température pour sa mise en forme,
- on réalise, de ce fait un composé allié intermétallique, en surface, assurant une protection contre la corrosion, contre la décarburation de l'acier, le composé intermétallique pouvant assurer une fonction de lubrification,
- on effectue la mise en forme notamment par emboutissage,
- on refroidit la pièce formée pour conférer des caractéristiques mécaniques de dureté élevées de l'acier et une dureté superficielle élevée du revêtement.

[0005] Les autres caractéristiques de l'invention sont:

- on revêt la tôle laminée à chaud après laminage à chaud et décapage,
- le métal ou l'alliage métallique de revêtement est de l'aluminium ou un alliage d'aluminium.
- l'alliage intermétallique est un composé à base principalement d'aluminium, de fer et de silicium,
- le revêtement de métal ou d'alliage métallique et effectué au trempé à chaud.
- la tôle revêtue est soumise à une élévation de température supérieure à 700°C avant la mise en forme et ou le traitement thermique,
- la tôle revêtue est emboutie à chaud.
- la pièce obtenue notamment par emboutissage est refroidie pour subir une trempe, à une vitesse supérieure à la vitesse critique de trempe.

[0006] L'invention concerne également une tôle laminée notamment à chaud comportant un revêtement de métal ou d'alliage métallique déposé en surface, pour la réalisation à chaud d'un composé intermétallique, le composé intermétallique assurant, une protection contre la corrosion, une barrière contre la décarburation de l'acier de la tôle, une lubrification lors de la mise en forme à chaud de la tôle, et sur la pièce finie, après mise en forme et refroidissement, des caractéristiques mécaniques de dureté de l'acier élevées ainsi qu'une dureté superficielle élevée de la surface de ladite pièce finie.

[0007] Une autre caractéristique de l'invention est:

- la tôle comporte un dépôt en surface d'aluminium ou d'un alliage d'aluminium.

[0008] L'invention concerne encore l'utilisation de la tôle dans la mise en forme à chaud notamment par emboutissage de pièces de hautes caractéristiques mécaniques en dureté et de hautes caractéristiques de dureté en surface assurant une très bonne tenue à l'abrasion. La description qui suit fera bien comprendre l'invention.

[0009] Le procédé selon l'invention consiste à revêtir un acier notamment laminé à chaud, pour traitement thermique et ou formage à chaud, avec un métal ou un alliage métallique tels que de l'aluminium ou un alliage d'aluminium composé par exemple d'aluminium et de silicium. Le revêtement de métal ou d'alliage métallique est choisi de manière à avoir une protection contre la corrosion et de manière à résister à une haute température. Lors d'un traitement thermique, ou d'une élévation de température pour la mise en forme, le revêtement forme une couche allié présentant une grande résistance à la corrosion, à l'abrasion, à l'usure et à la fatigue. Le revêtement ne modifie pas les propriétés de formabilité de l'acier et autorise ainsi une grande variété de mise en forme à froid et à chaud.

[0010] Après laminage à chaud la bande peut être décapée et laminée à froid avant d'être revêtue. Dans le

cas ou la tôle est laminée à froid, celle-ci peut être recuite avant d'être revêtue.

[0011] On peut revêtir la tôle laminée avec, par exemple, les deux types d'alliage métallique suivants:

Alliage N°1: Si: 9% à 10%; Fe: 2% à 3,5%; Solde: Al et des impuretés.

Alliage N°2: Fe: 2% à 4%; Solde: Al et des impuretés.

Pour l'alliage N°1 l'épaisseur du revêtement est comprise entre 5 µm et 100 µm, et de préférence entre 10 µm et 25 µm. Pour l'alliage N°2 l'épaisseur du revêtement est comprise entre 15 µm et 100 µm, et de préférence entre 30 µm et 50 µm.

[0012] Pour la mise en forme ou le traitement thermique, la tôle est soumise à une élévation de température comprise de préférence entre 750°C et 1200°C dans un four comportant une atmosphère ne nécessitant plus de contrôle du fait de la barrière à l'oxydation formée par le revêtement. Lors de l'élévation de la température, le revêtement à base d'aluminium se transforme en une couche alliée en surface comportant différentes phases dépendant du traitement en température et présentant une grande dureté pouvant dépasser 600 HV 100g.

[0013] Selon le procédé de l'invention, on peut produire des tôles dont l'épaisseur est comprise entre 0,2 mm et 20 mm, ayant de bonnes propriétés de mise en forme ainsi qu'une bonne résistance à la corrosion.

[0014] Les tôles livrées revêtues, présentent une résistance importante à la corrosion pendant les élévations de température, la mise en forme, les traitements thermiques, et lors de l'utilisation des pièces formées finies.

[0015] La présence du revêtement lors de traitements thermiques ou de formages à chaud permet d'éviter en plus de la corrosion, la décarburation de l'acier de base. Cela a un avantage indéniable dans le cas d'une mise en forme à chaud par exemple dans une presse d'emboutissage. En effet, l'alliage intermétallique formé évite, la formation de la calamine, l'usure des outils par la calamine, et permet de ce fait, un allongement de la vie moyenne desdits outils. Il a été remarqué que l'alliage intermétallique formé à chaud, a une fonction de lubrifiant à haute température. De plus, l'effet de protection contre la décarburation de l'alliage intermétallique permet l'usage de four haute température dépassant 900°C ayant une atmosphère non contrôlée, et cela, même pour des temps de chauffe de plusieurs heures.

[0016] En sortie de four, il n'est plus nécessaire de décaper la pièce obtenue, d'où une économie du fait de la suppression du bain de décapage des pièces finies.

[0017] De par les caractéristiques du revêtement après élévation de température, les pièces obtenues ont une résistance accrue à la fatigue, à l'usure, à l'abrasion et à la corrosion. De plus, le revêtement est soudable avant et après élévation de température.

[0018] L'acier de la tôle assure, par effet de trempe

au refroidissement, des caractéristiques mécaniques élevées de la pièce obtenue après mise en forme, le revêtement transformé en un alliage intermétallique à chaud assurant pour sa part, du fait de ses qualités de lubrifiant et de résistance aux frottement, une amélioration de la mise en forme, notamment, dans le domaine de l'emboutissage à chaud.

[0019] Dans un exemple de réalisation, on utilise une bande de tôle laminée à chaud en acier de composition pondérale suivante:

carbone: 0,15% à 0,25%,  
manganèse: 0,8% à 1,5%,  
silicium: 0,1% à 0,35%,  
chrome: 0,01 % à 0,2%,  
titane: moins de 0,1%,  
aluminium: moins de 0,1 %,  
phosphore: moins de 0,05%,  
soufre: moins de 0,03%,  
bore : 0,0005% à 0,01%,

on revêt la bande de tôle d'un revêtement de type alliage N°1 ou 2, on austénitise la tôle à 950°C avant formage et trempe dans l'outil, le revêtement assurant un rôle de lubrifiant lors de la mise en forme, en plus de ses fonctions de protection contre la corrosion à froid, à chaud et contre la décarburation. Lors de la trempe, le revêtement allié ne gêne pas l'extraction de la chaleur par l'outil et peut la favoriser. Après formage et trempe, il n'est plus nécessaire de décaper la pièce ou de la protéger, le revêtement de base assurant la protection tout au long du procédé.

## Revendications

1. Procédé de réalisation d'une pièce à partir d'une bande de tôle d'acier laminée et notamment laminée à chaud, caractérisée en ce que:

- on revêt la tôle laminée d'un métal ou d'un alliage métallique assurant une protection de la surface et de l'acier,
- on soumet la tôle revêtue à une élévation en température pour sa mise en forme,
- on réalise, de ce fait un composé allié intermétallique, en surface, assurant une protection contre la corrosion, contre la décarburation de l'acier, le composé intermétallique pouvant assurer une fonction de lubrification,
- on effectue la mise en forme notamment par emboutissage,
- on refroidit la pièce formée pour conférer des caractéristiques mécaniques de dureté élevées de l'acier et une dureté superficielle élevée du revêtement.

2. Procédé selon la revendication 1 caractérisé en ce

que on revêt le tôle laminée à chaud immédiatement après laminage à chaud,

3. Procédé selon la revendication 1 caractérisé en ce que le métal ou l'alliage métallique de revêtement est de l'aluminium ou un alliage d'aluminium. 5
4. Procédé selon la revendication 1 caractérisé en ce que l'alliage intermétallique est un composé à base d'aluminium, de fer et de silicium. 10
5. Procédé selon la revendication 1 caractérisé en ce que le revêtement de métal ou d'alliage métallique est effectué au trempé à chaud. 15
6. Procédé selon la revendication 1 caractérisé en ce que la tôle revêtue est soumise à une élévation de température supérieure à 700°C avant la mise en forme et ou le traitement thermique. 20
7. Procédé selon la revendication 1 caractérisé en ce que la tôle revêtue est emboutie à chaud.
8. Procédé selon la revendication 1 caractérisé en ce que la pièce obtenue notamment par emboutissage est refroidie pour subir une trempe, à une vitesse supérieure à la vitesse critique de trempe. 25
9. Tôle laminée notamment à chaud comportant un revêtement de métal ou d'alliage métallique déposé en surface, pour la réalisation à chaud d'un composé intermétallique, le composé intermétallique assurant, une protection contre la corrosion, une barrière contre la décarburation de l'acier de la tôle, une lubrification lors de la mise en forme à chaud de la tôle, et sur la pièce finie, après mise en forme et refroidissement, des caractéristiques mécaniques de dureté de l'acier élevées ainsi qu'une dureté superficielle élevée de la surface de ladite pièce finie. 30 35 40
10. Tôle laminée à chaud selon la revendication 9 caractérisée en ce qu'elle comporte un dépôt en surface d'aluminium ou d'un alliage d'aluminium. 45
11. Utilisation de la tôle selon les revendications 9 ou 10 dans la mise en forme à chaud notamment par emboutissage de pièces de hautes caractéristiques mécaniques en dureté et de hautes caractéristiques de dureté en surface assurant une très bonne tenue à l'abrasion. 50

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## RAPPORT DE RECHERCHE EUROPEENNE

Numéro de la demande  
EP 99 40 3227

| DOCUMENTS CONSIDERES COMME PERTINENTS                                                                                                                                                                                               |                                                                                                                                                                                                                                                                                                                           |                                                                                                                                                                                                                                                                 |                                                                  |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------|
| Catégorie                                                                                                                                                                                                                           | Citation du document avec indication, en cas de besoin, des parties pertinentes                                                                                                                                                                                                                                           | Revendication concernée                                                                                                                                                                                                                                         | CLASSEMENT DE LA DEMANDE (Int.Cl.7)                              |
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| Le présent rapport a été établi pour toutes les revendications                                                                                                                                                                      |                                                                                                                                                                                                                                                                                                                           |                                                                                                                                                                                                                                                                 |                                                                  |
| Lieu de la recherche<br>LA HAYE                                                                                                                                                                                                     |                                                                                                                                                                                                                                                                                                                           | Date d'achèvement de la recherche<br>8 mars 2000                                                                                                                                                                                                                | Examineur<br>Ceulemans, J                                        |
| CATEGORIE DES DOCUMENTS CITES                                                                                                                                                                                                       |                                                                                                                                                                                                                                                                                                                           | T : théorie ou principe à la base de l'invention<br>E : document de brevet antérieur, mais publié à la date de dépôt ou après cette date<br>D : cité dans la demande<br>L : cité pour d'autres raisons<br>& : membre de la même famille, document correspondant |                                                                  |
| X : particulièrement pertinent à lui seul<br>Y : particulièrement pertinent en combinaison avec un autre document de la même catégorie<br>A : arrière-plan technologique<br>O : divulgation non-écrite<br>P : document intercalaire |                                                                                                                                                                                                                                                                                                                           |                                                                                                                                                                                                                                                                 |                                                                  |

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# RAPPORT DE RECHERCHE EUROPEENNE

Numéro de la demande

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| DOCUMENTS CONSIDERES COMME PERTINENTS                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |                                                                                                                                    |                                                         |                                           |
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| Catégorie                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | Citation du document avec indication, en cas de besoin, des parties pertinentes                                                    | Revendication concernée                                 | CLASSEMENT DE LA DEMANDE (Int.Cl.7)       |
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| Le présent rapport a été établi pour toutes les revendications                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            |                                                                                                                                    |                                                         |                                           |
| Lieu de la recherche<br><b>LA HAYE</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |                                                                                                                                    | Date d'achèvement de la recherche<br><b>8 mars 2000</b> | Examineur<br><b>Ceulemans, J</b>          |
| <p><b>CATEGORIE DES DOCUMENTS CITES</b></p> <p>X : particulièrement pertinent à lui seul<br/>Y : particulièrement pertinent en combinaison avec un autre document de la même catégorie<br/>A : arrière-plan technologique<br/>O : divulgation non-écrite<br/>P : document intercalaire</p> <p>T : théorie ou principe à la base de l'invention<br/>E : document de brevet antérieur, mais publié à la date de dépôt ou après cette date<br/>D : cité dans la demande<br/>L : cité pour d'autres raisons<br/>&amp; : membre de la même famille, document correspondant</p> |                                                                                                                                    |                                                         |                                           |

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**ANNEXE AU RAPPORT DE RECHERCHE EUROPEENNE  
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EP 99 40 3227

La présente annexe indique les membres de la famille de brevets relatifs aux documents brevets cités dans le rapport de recherche européenne visé ci-dessus.  
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| Document brevet cité<br>au rapport de recherche | Date de<br>publication | Membre(s) de la<br>famille de brevet(s) | Date de<br>publication |
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(54) Title: METHOD OF HOT FORMING A STEEL BLANK AND THE HOT FORMED PART

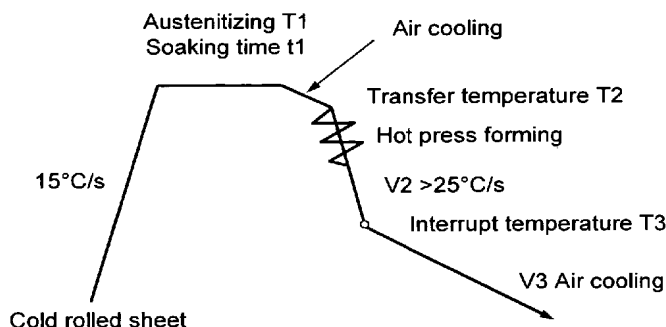


Fig. 1

(57) Abstract: According to a first aspect the present invention provides a method of hot forming a steel blank into an article the method comprising the following steps: d) cooling a heated steel blank to form an article during hot forming, starting at a starting temperature T2 above Ar3; to an interrupt temperature T3 in the range of 400 - 550 °C at a cooling rate V2 of at least 25 °C/s; e) immediately further cooling the article from the interrupt temperature T3 to ambient temperature at a cooling rate V3 of 0.2 - 10 °C/s, wherein the interrupt temperature T3 and cooling rate V3 are selected such that the article thus obtained has multiphase microstructure comprising by volume fraction: 55 - 90% of bainitic ferrite 5 - 15% of retained austenite 5 - 30% martensite.

## METHOD OF HOT FORMING A STEEL BLANK AND THE HOT FORMED PART

The invention relates to a method of hot forming a steel blank into a hot formed article having very high mechanical properties, such as an automotive part, to an article thus hot formed and to a steel strip, sheet or blank for use in a thermomechanical treatment process, in particular for use in a hot forming method according to the invention.

Recent progress in developing advanced high strength steels (AHSS) enables automotive manufacturers to increase vehicle's safety and crashworthiness of automotive parts and to reduce the weight of car bodies. Despite AHSS's high strength and light weight, inferior formability, poor ductility and toughness, compared to conventional steels, have become major hurdles for the application of AHSS to complex automotive parts such as A- or B-pillars reinforcements.

In attempts to solve the formability problem, hot (press) forming (hot stamping or press hardening) has been developed. By hot (press) forming, the strength of the steel article is improved through fast cooling after heating it to a temperature range where an austenitic phase exists and through the phase transformations of the austenite to harder phases such as bainite and martensite.

The basics of the hot forming technique and steel compositions adapted to be used were for the first time described in GB1490535. Afterwards a lot of Carbon - Manganese - Boron based steels have been developed for use in hot press forming.

A typical steel used for the hot press forming is based on a composition system of 22MnB5, i.e. 0.22% of C, 1.2% of Mn, maximum 50 ppm of B, specified in EN10083. Hot press forming of 22MnB5 steel can produce complex parts such as bumpers and pillars with ultrahigh strength, minimum springback, and

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reduced sheet thickness. The tensile strength of boron steels is up to 1600 MPa, which is far above that of the highest-strength conventional cold stamping steels. However the ductility of total elongation A5 is less than 6%.

- 5 To improve the crash-resistance of the steel, the ductility and toughness should be further increased. Advanced high strength steels with TRIP (transformation induced plasticity) added multiphase microstructure are a solution. These steels are characterized by a ferritic-bainitic microstructure with retained austenite, which undergoes martensitic transformation during forming of products,  
10 additionally contributing to the strengthening of a ready part.

- US2008/0286603 A1 has disclosed a steel sheet that exhibits an ultra-high strength after hot press forming followed by rapid cooling, and an enhanced yield strength after painting. The steel sheet has a composition (expressed in % by  
15 weight) comprising 0.1 to 0.5 C, 0.01 to 1.0 Si, 0.5 to 4.0 Mn, 0.1 or less P, 0.03 or less S, 0.1 soluble Al, 0.01 to 0.1 N, 0.3 or less W, and the balance being Fe and other inevitable impurities. Further a hot-pressed part made of the steel sheet and a method for manufacturing the hot-pressed part are disclosed. The hot-pressed part achieves a high increment in yield strength after heat treatment  
20 for painting while ensuring an ultra-high tensile strength. The steel provided according to this US patent application has an ultimate tensile strength (UTS) of 1500 MPa with the total elongation being less than 8%.

- From US2008/0251160 A1 a high-strength cold-rolled steel sheet having an  
25 uniform elongation is known, including (in wt.%): 0.10 - 0.28 C; 1.0 - 2.0 Si; and 1.0 - 3.0 Mn. The structure has the space factors: 30 - 65% of bainitic ferrite; 30 - 50% of polygonal ferrite; and 5 - 20% of residual austenite. The desired microstructures were obtained according to the following process. After hot rolling and cold rolling, the steel sheet was heated for soaking up to a  
30 temperature which is equal to or higher than the A3 transformation point (A3), then cooled down temporarily to a temperature  $T_q$  expressed by the formula  $A3-250 (^{\circ}C) \leq T_q \leq A3-20 (^{\circ}C)$  at an average cooling rate of 1 - 10  $^{\circ}C/s$ , and then quenched from this temperature down into a bainitic transformation

temperature range at an average cooling rate of 11 °C/s or faster. The tensile properties of the steel after such a heat treatment are typically, UTS = 1000 MPa and total elongation less than 15%.

- 5 US2008/0308194 A1 has disclosed a process for manufacturing a part made of steel having a multiphase microstructure. The composition of steel comprises, in % by weight: C 0.01 - 0.50, Mn 0.5 - 3.0, Si 0.001 - 3.0, Al 0.005 - 3.0, Mo  $\leq$  1.0, Cr  $\leq$  1.5, P  $\leq$  0.10, Ti  $\leq$  0.20, V  $\leq$  1.0 and optionally, one or more elements such as: Ni  $\leq$  2.0, Cu  $\leq$  2.0, S  $\leq$  0.05 and Nb  $\leq$  0.15, the balance of the composition  
10 being iron and inevitable impurities. In the process the steel sheet is heated to reach a soaking temperature  $T_s$  above  $A_{c1}$  but below  $A_{c3}$  and is held at this soaking temperature  $T_s$  for a soaking time  $t_s$  so that the steel has an austenite content equal to or greater than 25 % by area; the steel blank is transferred into a forming tool for hot forming; and the part obtained is cooled within the tool at a  
15 cooling rate  $V$  to obtain a multiphase microstructure comprising ferrite, martensite or bainite and retained austenite, and ferrite is homogeneous in each of the regions.

- Ryu H.-B et al., in a paper "Effect of thermomechanical processing on the retained austenite content in a Si-Mn transformation-induced-plasticity steel" (published in METALLURGICAL & MATERIALS TRANSACTIONS A, Vol. 33A, No. 9, 2002, p. 2811-2816), has reported a process of hot forming a steel specimen into a deformed article. This known process comprises - after austenizing for 5 minutes at 1000 °C - compression deformation at 920 °C. After  
20 this hot forming step is completed at this temperature, the deformed specimens are cooled at rates ranging from 10 - 35 °C/s to a simulated coiling temperature in the range of 420 - 480 °C. At this temperature the specimens are slowly cooled to 420 °C over a 2-minute period to complete bainitic transformation. Finally the specimens are air cooled to ambient temperature.

30

From EP1686195 A1 a heat treatment procedure - without deformation - for an already drawn wire is known. This heat treatment procedure comprises an intermediate isothermal transformation period of 60 - 3600 seconds at a

temperature between Ms - 50 °C and Bs.

JP2003193193 teaches steel sheet having a two phase microstructure comprising bainite/bainitic ferrite as a main phase and austenite as a second  
5 phase. The manufacturing process comprises an isothermal heat treatment at 200 - 450 °C for 1 - 3000 seconds.

In order to meet the requirements of the automotive industry there is an ongoing demand for steel articles having improved mechanical properties.

10

It is an object of the present invention to provide a manufacturing method of such steel articles, as well as steel compositions, in particular suitable for use in this method, more particularly advanced high strength TBF (TRIP-aided bainitic ferrite) steel products suitable for hot press forming as a Boron steel alternative  
15 for automotive applications.

Another object of the present invention is to provide a multiphase microstructured steel resulting in simultaneously improved strength and ductility.

20 Yet a further object is to provide a steel having an ultimate tensile strength higher than 1400 MPa, such as about 1500 Mpa. Still another object is to provide a steel having a total elongation higher than 8%, such as about 12%.

According to a first aspect the invention provides a method of hot forming a  
25 steel blank into an article, such as an automotive part, as defined in claim 1, wherein the method comprises the following steps:

- d) cooling a heated steel blank to form an article during hot forming, starting at a starting temperature T2 above Ar3 to an interrupt temperature T3 in the range of 400 - 550 °C at a cooling rate V2 of at  
30 least 25 °C/s;
- e) immediately further cooling the article from the interrupt temperature T3 to ambient temperature at a cooling rate V3 of 0.2 - 10 °C/s

- 5 -

wherein the interrupt temperature T3 and cooling rate V3 are selected such that the article thus obtained has a multiphase microstructure comprising by volume fraction:

55 - 90% of bainitic ferrite

5                   5 - 15% of retained austenite

5 - 30% martensite.

The steel blank as a starting material for executing the method according to the invention can be obtained by standard casting processes, or indirectly from steel strip or sheet material. In hot rolling the cast ingot, the preheating temperature may be about 1100 - 1250 °C. Traditional hot rolling passes and rolling conditions may be implemented to roll the steel ingot to a sheet product of about 3 to 5 mm in thickness. The finish rolling temperature is about 850 - 880 °C. After hot rolling, the steel sheet is cooled at an average cooling rate of 5 - 50 °C/s to a coiling temperature between 550 and 700 °C. An excess of martensite or bainite is formed at a coiling temperature lower than 550 °C, resulting in an excessive increase in the strength of the hot-rolled steel sheet. The excessively increased strength acts as a load during subsequent cold rolling for the production of a cold-rolled steel sheet causing problems, such as a poor appearance. During cold rolling the coiled hot-rolled sheet is pickled and cold-rolled. Cold rolling can also be carried out under standard conditions at a reduction of about 30 - 75%. However, in view of prevention of uneven recrystallization, the cold rolling reduction is preferably controlled to range from 40 to 70%. After cold rolling, the sheet has a thickness of about 1 to 2 mm according to product requirements. After cold rolling the sheet may be decoiled and blanked to proper size suitable for hot forming according to the invention. A cold rolled sheet is a preferred starting product in the method according to the invention.

In the present method of cooling during hot forming, the two-step cooling pattern and the interrupt cooling temperature are properly controlled.

Advantageously the heated steel blank used in step d) has an austenite structure. In a preferred embodiment for obtaining such austenite structure the method according to the invention also comprises the steps of

- 5 a) heating the steel blank to an austenitizing temperature  $T_1$  above  $Ac_3$ , preferably in the range of  $Ac_3 + 20\text{ }^{\circ}\text{C}$  to  $Ac_3 + 60\text{ }^{\circ}\text{C}$ , more preferably at a heating rate of  $10 - 25\text{ }^{\circ}\text{C/s}$ ;
- b) soaking the steel blank in said range, preferably during a soaking time  $t_1$  of  $1 - 5$  minutes;
- 10 c) optionally transferring the heated and soaked blank to a hot forming facility.

These austenitizing steps of the method for obtaining an austenite structure are performed by heating steel sheets, strips of blanks e.g. in a furnace or in a hot forming facility itself, to a temperature  $T_1$  above  $Ac_3$ , more preferably in the

15 range of  $Ac_3 + 20\text{ }^{\circ}\text{C}$  to  $Ac_3 + 60\text{ }^{\circ}\text{C}$ . Preferably the heating rate is in the range of  $10 - 25\text{ }^{\circ}\text{C/s}$ . This heating step is followed by soaking at a temperature above  $Ac_3$ , preferably in the range of  $Ac_3 + 20\text{ }^{\circ}\text{C}$  to  $Ac_3 + 60\text{ }^{\circ}\text{C}$ , for a short period of time, preferably  $1 - 5$  min ( $Ac_3$  being the temperature at which transformation of ferrite into austenite is completed in hypoeutectoid steel, upon heating). The

20 austenitizing temperature ( $T_1$ ) and soaking time are selected to ensure complete dissolution of carbides. If applicable, then the steel blank thus soaked is transferred to the hot forming device such as a hot forming press. If applicable, the transferring time is controlled, usually to a short transfer time such as less than 10 seconds to prevent cooling of the blank below the starting temperature

25  $T_2$  of the next hot forming step. The starting temperature  $T_2$  for hot forming and simultaneous cooling should be above the  $Ar_3$  point (typically in the range of  $780\sim 830\text{ }^{\circ}\text{C}$ ) to prevent any ferrite phase transformation, e.g. during transferring. ( $Ar_3$  being the temperature at which austenite begins to convert to ferrite upon cooling a steel.) During hot forming the blank is deformed and at the same time

30 cooled down to an interrupt quenching temperature  $T_3$  in the range of  $400$  to  $550\text{ }^{\circ}\text{C}$  at a cooling rate  $V_2$  higher than  $25\text{ }^{\circ}\text{C/s}$  to avoid formation of proeutectoid ferrite and pearlite. If the cooling rate  $V_2$  is less than  $25\text{ }^{\circ}\text{C/s}$ , pearlite may be generated during quenching. Immediately, i.e. without holding

the blank for a predetermined time at a temperature of about T3, such as isothermal austempering at T3, the steel blank is allowed to cool down further to room temperature at a further cooling rate V3 of 0.2 - 10 °C/s. The higher cooling rates above 5 °C/s within said range are advantageous for achieving a high total elongation (At). The range of 0.5 - 5 °C/s is preferred in view of high Ultimate Tensile Strength (UTS). The combined adjustment of the interrupt temperature T3 and the further cooling rate V3 in the final step effectively controls the volume fraction of the various phases in the final microstructure of the article thus manufactured. The interrupt quenching temperature T3 is in the bainitic transformation range. If T3 is too high, generally above 550 °C, some pearlite structure may form during the following slow cooling step at cooling rate V3. If T3 is too low, below Ms point, bainitic structure may be obtained in an insufficient amount in the final microstructure. If V3 is too fast, too much martensite is obtained in the final microstructure, and it is impossible to ensure sufficient elongation. If V3 is too small, too less martensite is obtained in order to guarantee the high strength. Moreover, the combination of T3 and V3 is selected such that the article obtained comprises a multiphase microstructure comprising by volume fraction 55 - 90% of bainitic ferrite, 5 - 15% retained austenite and 5 - 30% martensite. A low martensite content within this range, typically below 10 %, offers a relatively high total elongation. A relatively high UTS is achieved if the multiphase microstructure comprises 55 - 85% bainitic ferrite, 5 - 15% retained austenite and 10 - 30% martensite. Generally the higher the interrupt temperature T3, the lower V3 has to be in order to get this multiphase microstructure.

Preferably the steel, in particular TBF steel, in the article as manufactured, is characterized by a multiphase structure comprising, more preferably consisting of, carbide free bainitic ferrite, carbon enriched retained austenite and a relatively small amount of martensite. In the multiphase structure, the mother-phase structure (matrix) comprises fine plates of essentially carbide free bainitic ferrite. The formation of such a microstructure is due to the fact that the precipitation of cementite during bainitic transformation is suppressed by alloying the steel with a sufficient amount of Si and/or Al, which have very low

solubility in cementite and greatly retards growth of cementite from austenite. Carbon that is rejected from the bainitic ferrite enriches the residual austenite, thereby transforming to martensite during cooling after bainitic transformation or stabilizing it down to room temperature.

5

The advantages of this type of microstructure are manifold. The bainitic ferrite in TBF steel is present in the form of plates with an ultra fine grain size, usually the length ranging up to about 15 micrometer at most and the thickness ranging up to 0.3 micrometer at most (typically ~10  $\mu\text{m}$  long and ~0.2  $\mu\text{m}$  thick).  
10 Furthermore the strength and ductility are improved simultaneously. The high flow stresses are due to the small thickness of the bainitic laths and to the absence of proeutectoid polygonal ferrite contrary to existing TRIP (transformation induced plasticity) steels. It is assumed that the retained austenite offers an additional TRIP effect, and that it is useful for improving total  
15 elongation. The presence of martensite islands leads to a high strength and to high instantaneous hardening rates. If the synergy effect is secured between transformation induced plasticity attained by residual austenite and use of fine plate bainitic ferrite in TBF steel, a dramatic improvement of elongation of TBF steel is attained. Toughness and crash performance are improved as well.

20

Moreover, to reach a high strength and a high ductility of the steel, the constituents of the various phases in the final microstructure preferably comprise, by volume fraction, bainitic ferrite: 55 - 90%, retained austenite: 5 - 15% and martensite: 5 - 30%.

25

The multiphase microstructure according to the invention can be obtained by designing steel with a specific composition and by applying careful control of the thermomechanical treatment process as discussed above.

30 Thus, in other words in a first aspect a preferred embodiment of the present invention relates to a method of hot forming a steel blank into an article, wherein the method comprises heating the steel blank to an austenitizing temperature  $T_1$ , advantageously in the range of  $A_{c3} + 20\text{ }^{\circ}\text{C}$  and  $A_{c3} + 60\text{ }^{\circ}\text{C}$ ,

and soaking the steel blank in the austenitizing temperature range, such that carbides are completely dissolved; introducing the heated and soaked blank in a hot forming device, such as a hot press, while preventing ferrite phase transformation; hot forming the steel blank to form a shaped article starting at a starting temperature  $T_2$  above  $A_{r3}$  and cooling the article thus being hot formed to an interrupt temperature  $T_3$  in the bainitic transformation range at a cooling rate such that formation of proeutectoid ferrite and pearlite is avoided; and further cooling the article from the interrupt temperature  $T_3$  to ambient temperature at a cooling rate such that the volume fraction of bainitic ferrite is 55 - 90%, of retained austenite 5 - 15%, and of martensite 5 - 30% in the article thus manufactured.

Advantageously, the steel comprises the following elements (in wt. %): C 0.15 - 0.45, Si 0.6 - 2.5, Mn 1.0 - 3.0, Mo 0 - 0.5, Cr 0 - 1.0, P 0.001 - 0.05, S < 0.03, Ca < 0.003, Ti 0.1 or less and V 0.1 or less and the balance Fe and other inevitable impurities. Optionally, the steel may also contain Al less than 1.5%, partially replacing the same amount of Si, provided that the sum of Si and Al is in the range of 1.2 - 2.5%. Preferably, the amounts of Mn and Cr satisfy  $Mn + Cr \leq 3\%$ , while also preferably, the amounts of C and Mo satisfy  $C + 1/3Mo \leq 0.45\%$  for better control of the multiphase microstructure.

Hereinbelow the functions of each element in the steel composition is described. Content is represented as % by weight of the total composition.

C is an element for securing high strength, and for securing retained austenite. C is added in an amount of 0.15% or more to form the desired multiphase microstructure to achieve ultra-high strength and ductility. Meanwhile, when the C content exceeds 0.45%, it is difficult to obtain the multiphase microstructure through the method according to the invention comprising a contiguous cooling subprocess. Moreover, there is a great possibility that the toughness and weldability of the steel sheet will be deteriorated. C is preferably present in an amount of 0.2 - 0.4%, more preferably 0.2 - 0.35%.

Mn is one of the main elements in the steel composition according to the invention. The functions of Mn include stabilizing the austenite and obtaining the desired multiphase microstructure. If Mn is less than 1.0%, the effects are not sufficiently marked. Whereas if the content exceeds 3%, a fully martensite structure is easily created. As a result, the steel is hardened and embrittled during press forming. Besides, Mn is an element that is useful in lowering the Ac3 temperature. A higher Mn content is advantageous in lowering the temperature necessary for hot press forming. The Mn content is limited to the range of 1.0 - 3.0%, preferably 1.5 - 2.5% and more preferably 1.6 - 2.5%, even more preferably 1.7 - 2.4%.

Si is an element effective for reinforcing a solid solution, and is useful for suppressing production of carbide due to decomposition of retained austenite. To obtain high ductility, toughness and formability, the formation of carbides (either transition carbide or cementite) should be avoided as much as possible. Si suppresses the precipitation of brittle cementite during bainite formation, and hence results in an improvement in formability and toughness. A minimum of 1.0% Si is needed to form carbide free bainite. However, Si is also known to deteriorate galvanizability due to the formation of oxides adherent to the steel substrate. Therefore, the upper limit of Si is controlled below 2.5%. The Si content is advantageously limited to the range of 1.2 - 2.5% and more preferably to the range of 1.4 - 2.0%.

Al is also an element useful for suppressing production of carbide due to decomposition of, particularly, retained austenite. Partial replacement of Si by a same amount of Al has been shown to effectively retard cementite formation without a detrimental effect on hot-dip coatability in TBF steels. However, a high concentration of Al leads to higher possibility of polygonal ferrite to be generated, which is less effective than fine plate ferrite in view of strength. A full substitution of Si by an equivalent amount of Al leads to a marked deterioration of the strength-ductility balance. If added, the amount of Al is limited to 1.5% or smaller.

P is an element useful for maintaining desired retained austenite, and its effect is exerted by an amount of P of 0.001% or larger, more preferably 0.005% or larger, but P may deteriorate the workability of the steel when it is added in an excess amount. Accordingly, the P content is preferably limited to 0.05% or less.

S is a harmful element which forms sulfide based inclusions such as MnS, which initiates crack formation, and deteriorates processability. Therefore, it is desirable to reduce the amount of S as much as possible. Accordingly, S is controlled to 0.03% or smaller.

Mo and Cr serve to improve the hardenability of the steel and facilitate the formation of bainite ferrite. At the same time, they are elements having similar effectiveness useful for stabilizing retained austenite. Therefore, Mo and Cr are very effective for process control. It is advantageous that each of them is contained at 0.05% or larger. However, when each of them is added excessively, the effect is saturated and a higher addition is not economical. Therefore, the amount of Mo is 0.5% or smaller, and the amount of Cr is 1% or smaller.

Ti and V have the effect of forming strengthening precipitates and refining microstructure. The amount of each of them is 0.1% or smaller, preferably 0.05% or smaller.

Ca is an element effective for controlling a form of sulfide in the steel, and improving processability. It is recommended that Ca is contained at 0.0003% or more. However, when it is added excessively, the effect is saturated. Therefore, the preferred amount is 0.0003 - 0.003%.

Regarding a practical manufacturing process, the heat treatment described above may be carried out by heating and cooling in a continuous annealing facility (CAL), hot press forming facility, salt bath or the like.

Advantageously the first combined deformation/cooling step d) is performed in a hot forming facility. In a preferred embodiment the second cooling step e) is performed in air or in stock outside the hot forming facility.

5

Preferably the steel of the formed article has an ultimate tensile strength (UTS) of at least 1400 MPa, advantageously at least 1500 MPa, more preferably at least 1600 MPa, and most preferably at least 1700 MPa.

- 10 Advantageously the steel of the formed article has a total elongation of at least 8%, preferably at least 10%, more preferably at least 12%, and most preferably at least 14%.

The heat treatment processes can be simply performed by applying the hot (press) forming in a standard hot forming facility with only modification of the simultaneous cooling process, including cooling interrupt temperature and velocities. After austenitizing, the heat blank is inserted into the die sets of a hot press, in which the blank is shaped and cooled. The transfer time (e.g. 5 to 10 s) between furnace and the press forming tools is controlled to secure that the starting deformation temperature is higher than Ar<sub>3</sub>. The cooling rate V<sub>2</sub> of the steel part in the forming tool depends on the deformation and on the quality of the contact between the tool and the steel blank. The forming tools may be cooled for example by using circulation of a liquid to ensure that the cooling rate is high enough (>25 °C/s) during quenching in the press mould. The interrupt cooling temperature can be controlled by the time for separation of the pressing tools. The formed article is then removed from the mould and cooled to room temperature in air. The formed articles, e.g. sheets, may also be stacked and then cooled to ambient temperature in air.

- 30 After cooling down to room temperature, the parts will be built in the car body structure. Then the paintbake process is performed. The paintbake cycle does not affect the properties.

In a second aspect the present invention provides a steel article, preferably formed according to the method of the present invention, wherein the steel has a microstructure comprising by volume fraction:

- 55 - 90% of bainitic ferrite
- 5        5 - 15% of retained austenite
- 5 - 30% martensite.

The details of the composition and microstructure as discussed above in view of the method according to the invention, in particular the advantageous and/or preferred embodiments are similarly applicable to the second aspect of the invention. Preferably the steel has an Ultimate Tensile Strength of at least 1400 MPa, advantageously at least 1500 MPa, preferably at least 1600 MPa, more preferably at least 1700 MPa and/or a total elongation of at least 8%, preferably at least 10%, more preferably at least 12%, most preferably at least 14%.

According to a third aspect the present invention provides a steel strip, sheet or strip for use in a thermomechanical treatment, in particular a hot forming method according to the invention described above, having a composition, in weight %:

|    |     |              |
|----|-----|--------------|
| 20 | C:  | 0.15 - 0.45  |
|    | Si: | 0.6 - 2.5    |
|    | Mn: | 1.0 - 3.0    |
|    | Al: | 0 - 1.5      |
|    | Mo: | 0 - 0.5      |
| 25 | Cr: | 0 - 1.0      |
|    | P:  | 0.001 - 0.05 |
|    | S:  | < 0.03       |
|    | Ca: | < 0.003      |
|    | Ti: | < 0.1        |
| 30 | V:  | < 0.1        |

the balance being Fe and inevitable impurities,  
wherein  $Si + Al = 1.2 - 2.5\%$ .

Preferably the relationships  $Mn + Cr \leq 3\%$  and  $C + 1/3Mo \leq 0.45\%$  are applied.

In preferred compositions of the steel strip, sheet or blank according to the third aspect of the invention comprises, the alloying elements are present in, expressed in weight %,:  
5

|    |     |                                         |
|----|-----|-----------------------------------------|
|    | C:  | 0.2 - 0.4, preferably 0.2 - 0.35 and/or |
|    | Si: | 0.8 - 2.0, preferably 1.2 - 1.8 and/or  |
|    | Mn: | 1.5 - 2.5, preferably 1.7 - 2.4 and/or  |
|    | Mo: | 0.05 - 0.5 and/or                       |
|    | Cr: | 0.05 - 1.0 and/or                       |
| 10 | P:  | 0.005 - 0.05 and/or                     |
|    | Ca: | 0.0003 - 0.003.                         |

The articles obtained from the strip, sheet or blank exhibit a high tensile strength by rapid cooling after heat treatment and achieve a high increment in yield  
15 strength after heat treatment, especially for painting. Based on these advantages, excellent impact properties of the steel article according to the present invention are attained. In addition, the steel article according to the present invention advantageously exhibits good adhesion to a coating layer. Furthermore, other advantages of the steel article according to the present  
20 invention are good surface appearance and superior corrosion resistance after painting.

A schematic representation of a practical embodiment of the method according to the invention is shown in Fig. 1, showing a temperature vs. time plot. A steel  
25 blank is heated at a heating rate of 15 °C to the austenitizing temperature T1 in the range of Ac3 + 20 °C to Ac3 + 60 °C and soaked at that temperature during a soaking time t1. The thus heated and soaked blank is transferred from the furnace to the hot forming facility, during which cooling by air occurs to some extent. Care is taken that the temperature T2 is not decreased below Ar3  
30 before hot press forming of the blank. After hot press forming the blank thus formed is cooled down to the interrupt temperature T3 at a rate of more than 25 °C. Then air cooling is carried out.

The invention is further illustrated by the following examples.

Steels having compositions A-F as specified in Table 1 were cast to ingots of about 25 kg (100x110x330 mm). The ingots were reheated and then roughly hot rolled to slabs having a thickness of 40 mm. Then finish hot rolling was applied with the following process parameters: preheating at 1200°C for 30 min; multi-pass rolling to thickness 4 mm (40-27-18-12-8-6-4 mm); and finishing rolling temperature  $860 \pm 20$  °C. Controlled cooling at a rate 30 °C/s to 600 °C in a run-out-table was carried out in order to simulate the commercially used coiling process. After the coiling process the steel plates contain a microstructure consisting of ferrite and pearlite and have a tensile strength less than 700 MPa. The plate was then cold rolled from 4 to 1 mm sheet.

Table 1. Chemical compositions (in wt. %) of TBF steel

| Alloy | C    | Si   | Mn   | Mo   | Cr  | Ti   | P    | S    | Ac3<br>(°C) | Ms<br>(°C) |
|-------|------|------|------|------|-----|------|------|------|-------------|------------|
| A     | 0.31 | 1.47 | 1.66 | -    | -   | 0.01 | 0.03 | 0.01 | 863         | 334        |
| B     | 0.40 | 1.48 | 1.67 | -    | -   | 0.01 | 0.03 | 0.01 | 848         | 306        |
| C     | 0.29 | 1.76 | 2.39 | -    | -   | 0.01 | 0.03 | 0.01 | 879         | 313        |
| D     | 0.22 | 1.57 | 2.35 | 0.25 | -   | 0.01 | 0.03 | 0.01 | 890         | 340        |
| E     | 0.30 | 1.50 | 2.40 | 0.25 | -   | 0.01 | 0.03 | 0.01 | 876         | 318        |
| F     | 0.30 | 1.50 | 2.00 | 0.25 | 0.5 | 0.01 | 0.03 | 0.01 | 874         | 315        |

V and Al were below detection level.

The critical temperatures such as Ac3 and Ms were determined by applying standard dilatometric analysis to facilitate the determination of the temperatures in the process according to the invention.

20

The resulting cold rolled sheets were subjected to a heat treatment by using a CASIM simulator. Specifically, the steel sheets were heated to 870 to 920°C at a rate of 15 °C/s, holding the sheets at this temperature for 2 min, then cooling at a rate of 50 °C/s to an interrupt temperature between 400 and 550 °C, thereafter, cooling at a rate of 0.2 to 10 °C/s to simulate different air cooling conditions.

25

Tensile tests were conducted by applying JIS 5 tensile test specimen to measure tensile strength and elongation of the specimens with the required microstructures. The volume fraction of bainite and/or martensite in the microstructures was estimated by using metallographic characterisation in combination with dilatametric analysis. The volume fraction of the retained austenite was determined by using TEM. Other measurements are standard. The tensile test results and the invented alloys with the required the microstructure constituents are given in Table 2.

The experimental results indicate that the microstructures and the properties are essentially independent from the austenitizing temperature  $T_1$  as long as the  $T_1$  is above  $A_{c3}$ , advantageously between  $A_{c3} + 20\text{ }^{\circ}\text{C}$  and  $A_{c3} + 60\text{ }^{\circ}\text{C}$ . It is also proven that the transferring temperature  $T_2$  and the cooling rate  $V_2$  do not significantly affect the microstructures and properties as well provided that  $T_2$  is higher than  $A_{r3}$  and  $V_2$  is equal to or more than  $25\text{ }^{\circ}\text{C/s}$  and preferably less than  $100\text{ }^{\circ}\text{C/s}$ . However, the microstructures and the properties are strongly dependent on the interrupt temperature  $T_3$  and the cooling rate  $V_3$ . The C content and alloying element contents have a large effect on the selection of  $T_3$  and  $V_3$ . From these results, the conditions for the required multiphase microstructures of the alloys according to the invention can be obtained by careful adjustment of  $T_3$  and  $V_3$ . For alloys with higher C or higher Mo contents, the cooling rate  $V_3$  can be controlled in a lower range  $0.25$  to  $2\text{ }^{\circ}\text{C/s}$ ; for alloys containing lower C or lower Mo, the cooling rate  $V_3$  can be controlled in a higher range  $2$  to  $10^{\circ}\text{ C/s}$ . For a given alloy composition at a fixed  $T_3$  temperature, higher cooling rate will result in less bainitic ferrite, but relatively more martensite. Therefore higher strength will be obtained. Lower cooling rate will result in more bainitic ferrite, but relatively less martensite, then higher elongation will be achieved. The higher  $T_3$  temperature is, the relatively more bainitic ferrite will be in the final microstructure for a given cooling rate, resulting in high strength but lower elongation.

Table 2. Hardness and tensile properties of the alloys and their microstructures

- 17 -

| Alloy    | T3   | V3     | Final microstructure |      |       | HV5 | YS    | UTS   | At   |
|----------|------|--------|----------------------|------|-------|-----|-------|-------|------|
|          | (°C) | (°C/s) | BF(%)                | M(%) | Ar(%) |     | (MPa) | (MPa) | (%)  |
| <b>A</b> | 450  | 4      | 65                   | 23   | 12    | 435 | 1024  | 1503  | 10.5 |
| <b>A</b> | 500  | 8      | 76                   | 16   | 10    | 410 | 943   | 1435  | 14   |
| <b>B</b> | 400  | 1      | 74                   | 20   | 6     | 476 | 1035  | 1660  | 12   |
| <b>B</b> | 450  | 1      | 87                   | 8    | 5     | 435 | 965   | 1420  | 14.2 |
| <b>C</b> | 450  | 1      | 77                   | 14   | 9     | 460 | 1023  | 1572  | 12.5 |
| <b>C</b> | 500  | 1      | 85                   | 8    | 7     | 455 | 987   | 1475  | 13.6 |
| <b>D</b> | 450  | 2      | 85                   | 5    | 10    | 485 | 914   | 1451  | 12.5 |
| <b>E</b> | 450  | 0.5    | 65                   | 25   | 10    | 491 | 1025  | 1796  | 9    |
| <b>E</b> | 550  | 0.5    | 68                   | 23   | 9     | 480 | 1016  | 1693  | 8.9  |
| <b>F</b> | 500  | 0.5    | 80                   | 12   | 8     | 475 | 997   | 1599  | 11   |
| <b>F</b> | 550  | 0.5    | 72                   | 20   | 8     | 519 | 1011  | 1695  | 9.5  |

In the above table the following abbreviations are used:

BF = Bainitic Ferrite

M = Martensite

5 Ar = Retained Austenite

HV5 = Vickers hardness measured at load of 5 kgf

YS = Yield Strength

UTS = Ultimate Tensile Strength

At = Total Elongation

10

**CLAIMS**

1. Method of hot forming a steel blank into an article, the method comprising  
5 the following steps:

d) cooling a heated steel blank to form an article during hot forming,  
starting at a starting temperature T2 above Ar3 to an interrupt  
temperature T3 in the range of 400 - 550 °C at a cooling rate V2 of  
10 at least 25 °C/s;

e) immediately further cooling the article from the interrupt temperature  
T3 to ambient temperature at a cooling rate V3 of 0.2 - 10 °C/s,  
wherein the interrupt temperature T3 and cooling rate V3 are selected  
such that the article thus obtained has a multiphase microstructure  
15 comprising by volume fraction:

55 - 90% of bainitic ferrite  
5 - 15% of retained austenite  
5 - 30% martensite.

20 2. Method according to claim 1, wherein the blank, optionally produced from  
a steel strip, sheet, has the following composition in weight %:

|        |              |
|--------|--------------|
| C:     | 0.15 - 0.45  |
| Si:    | 0.6 - 2.5    |
| Mn:    | 1.0 - 3.0    |
| 25 Al: | 0 - 1.5      |
| Mo:    | 0 - 0.5      |
| Cr:    | 0 - 1.0      |
| P:     | 0.001 - 0.05 |
| S:     | < 0.03       |
| 30 Ca: | < 0.003      |
| Ti:    | < 0.1        |
| V:     | < 0.1        |

the balance being Fe and inevitable impurities,

wherein  $\text{Si} + \text{Al} = 1.2 - 2.5\%$ .

3. Method according to claim 2, wherein  $\text{Mn} + \text{Cr} \leq 3\%$  and  $\text{C} + 1/3 \text{ Mo} \leq 0.45\%$ .

5

4. Method according to claim 2 or 3, wherein the composition comprises, in weight%:

|     |                     |
|-----|---------------------|
| C:  | 0.2 - 0.4 and/or    |
| Si: | 0.8 - 2.0 and/or    |
| Mn: | 1.5 - 2.5 and/or    |
| Mo: | 0.05 - 0.5 and/or   |
| Cr: | 0.05 - 1.0 and/or   |
| P:  | 0.005 - 0.05 and/or |
| Ca: | 0.0003 - 0.003.     |

15

5. Method according to any one of claims 2-4, wherein the composition comprises, in weight %:

|     |                   |
|-----|-------------------|
| C:  | 0.2 - 0.35 and/or |
| Si: | 1.2 - 1.8 and/or  |
| Mn: | 1.7 - 2.4.        |

20

6. Method according to any one of the preceding claims, wherein the bainitic ferrite is essentially carbide free and the retained austenite is carbon enriched.

25

7. Method according to any one of the preceding claims, wherein the grains of bainitic ferrite have a length of at most 15  $\mu\text{m}$  and a thickness of at most 0.3  $\mu\text{m}$ .

- 30 8. Method according to any one of the preceding claims, further comprising - prior to the hot forming step d) - the steps of

- 5
- a) heating the steel blank to an austenitizing temperature T1 above Ac3, preferably in the range of  $Ac3 + 20\text{ }^{\circ}\text{C}$  -  $Ac3 + 60\text{ }^{\circ}\text{C}$ , preferably at a heating rate of  $10 - 25\text{ }^{\circ}\text{C/s}$ ;
- b) soaking the steel blank in said range, preferably during a soaking time of 1-5 minutes;
- c) optionally transferring the heated and soaked blank to a hot forming facility;
- 10
9. Method according to claim 5, wherein step a) is performed in a continuous annealing facility, or a hot forming facility, or a salt bath or equivalent.
10. Method according to any one of the preceding claims, wherein step d) is performed in a hot forming facility.
- 15
11. Method according to any one of the preceding claims, wherein step e) is performed outside a hot forming facility in air.
12. Steel article preferably formed according to the method of any one of the preceding claims, wherein the steel has a microstructure comprising by volume fraction:
- 20
- 55 - 90% of bainitic ferrite  
5 - 15% of retained austenite  
5 - 30% martensite.
- 25
13. Steel article according to claim 13, wherein the steel has an Ultimate Tensile Strength of at least 1400 MPa, advantageously at least 1500 MPa, preferably at least 1600 MPa, more preferably at least 1700 MPa and/or a total elongation of at least 8%, preferably at least 10%, more preferably at least 12%, most preferably at least 14%.
- 30
14. Steel strip, sheet or blank for use in a thermomechanical treatment process, preferably the method according to any one of the preceding claims 1-12, having a composition, in weight %:

- 21 -

|    |     |              |
|----|-----|--------------|
|    | C:  | 0.15 - 0.45  |
|    | Si: | 0.6 - 2.5    |
|    | Mn: | 1.0 - 3.0    |
|    | Al: | 0 - 0.6      |
| 5  | Mo: | 0 - 0.5      |
|    | Cr: | 0 - 1.0      |
|    | P:  | 0.001 - 0.05 |
|    | S:  | < 0.03       |
|    | Ca: | < 0.003      |
| 10 | Ti: | < 0.1        |
|    | V:  | < 0.1        |

the balance being Fe and inevitable impurities,  
wherein Si + Al = 1.2 - 2.5%.

- 15 15. Steel strip, sheet or blank according to claim 14, wherein  $Mn + Cr \leq 3\%$   
and  $C + 1/3 Mo \leq 0.45\%$ .

16. Steel strip, sheet or blank according to claim 14 or 15, wherein,  
expressed in weight %,::

|    |     |                                         |
|----|-----|-----------------------------------------|
| 20 | C:  | 0.2 - 0.4, preferably 0.2 - 0.35 and/or |
|    | Si: | 0.8 - 2.0, preferably 1.2 - 1.8 and/or  |
|    | Mn: | 1.5 - 2.5, preferably 1.7 - 2.4 and/or  |
|    | Mo: | 0.05 - 0.5 and/or                       |
|    | Cr: | 0.05 - 1.0 and/or                       |
| 25 | P:  | 0.005 - 0.05 and/or                     |
|    | Ca: | 0.0003 - 0.003.                         |

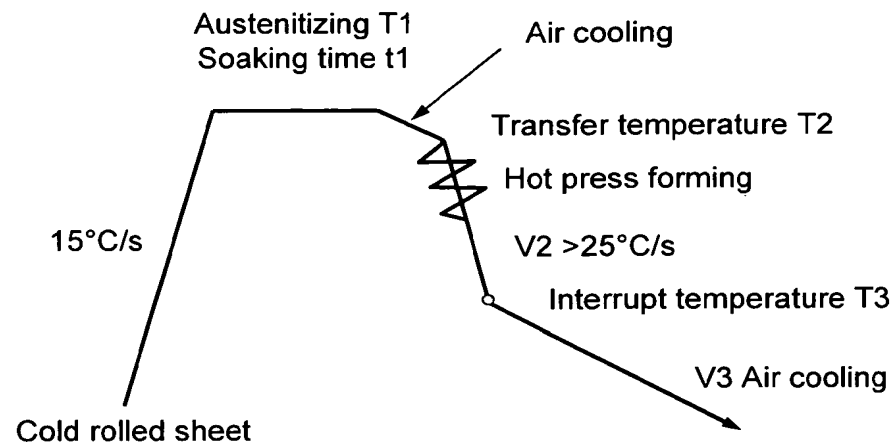


Fig. 1

## INTERNATIONAL SEARCH REPORT

International application No  
PCT/EP2011/005053

## A. CLASSIFICATION OF SUBJECT MATTER

INV. C21D9/46 C22C38/02 C22C38/04 C22C38/12 C22C38/14  
C21D1/673 C21D9/48

ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C21D C22C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, CHEM ABS Data, WPI Data

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages                                                                                                                                                                                                                                                                                                                                                                                                                  | Relevant to claim No. |
|-----------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------|
| X         | RYU H-B ET AL: "Effect of thermomechanical processing on the retained austenite content in a Si-Mn transformation-induced-plasticity steel", METALLURGICAL AND MATERIALS TRANSACTIONS A: PHYSICAL METALLURGY & MATERIALS SCIENCE, ASM INTERNATIONAL, MATERIALS PARK, OH, US, vol. 33A, no. 9, 1 September 2002 (2002-09-01), pages 2811-2816, XP002584719, ISSN: 1073-5623, DOI: DOI:10.1007/S11661-002-0266-3 [retrieved on 2007-04-07] page 1 - page 2; figures 3, 6, 7; table 1<br>-----<br>-/-- | 1-16                  |

☒ Further documents are listed in the continuation of Box C.☒ See patent family annex.

## \* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
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Date of the actual completion of the international search

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## INTERNATIONAL SEARCH REPORT

International application No

PCT/EP2011/005053

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages                                                                                                                | Relevant to claim No. |
|-----------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------|
| X         | EP 1 686 195 A1 (KOBE STEEL LTD [JP];<br>SHINSHU TLO CO LTD [JP])<br>2 August 2006 (2006-08-02)<br>paragraph [0018] - paragraph [0028]<br>page 7, line 5 - line 45<br>example E; table 1<br>----- | 1-16                  |
| X         | JP 2003 193193 A (NIPPON STEEL CORP)<br>9 July 2003 (2003-07-09)<br>paragraph [0022] - paragraph [0026];<br>example c1; tables 1-3<br>-----                                                       | 1-16                  |

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2011/005053

| Patent document<br>cited in search report |    | Publication<br>date | Patent family<br>member(s) | Publication<br>date |
|-------------------------------------------|----|---------------------|----------------------------|---------------------|
| EP 1686195                                | A1 | 02-08-2006          | AT 535624 T                | 15-12-2011          |
|                                           |    |                     | EP 1686195 A1              | 02-08-2006          |
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|                                           |    |                     | US 2006169367 A1           | 03-08-2006          |
| -----                                     |    |                     |                            |                     |
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[Continued on next page]

(54) Title: METHOD FOR HOT FORMING A COATED METAL PART AND FORMED PART

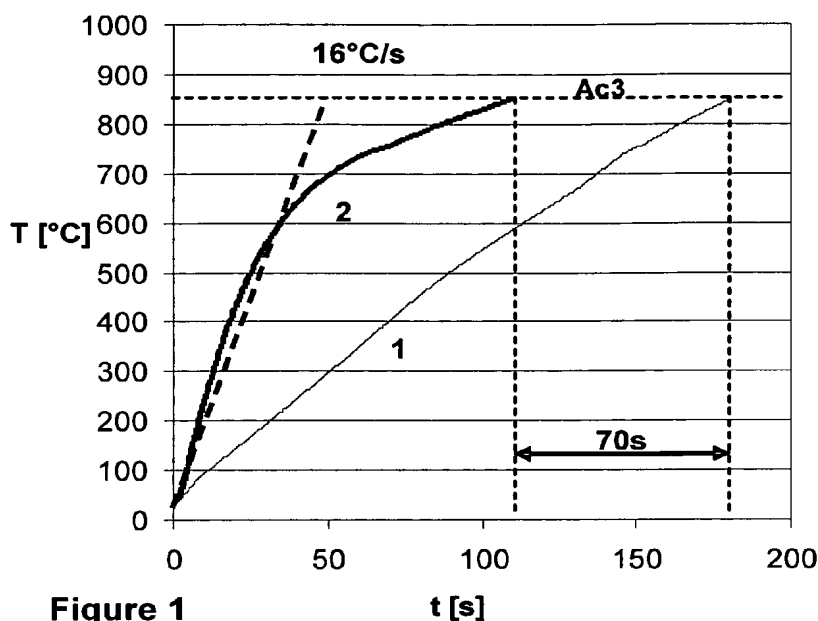


Figure 1

(57) Abstract: The invention relates to a method for manufacturing a coated part having very high mechanical properties using hot forming techniques. According to the invention, the method comprises the following steps: - providing an annealed steel strip or blank or pre-shaped part that has been coated with a metal or metal alloy before the annealing - if a strip is provided, cutting a blank from the strip before or after the annealing - optionally forming a pre-shaped part from the blank - heating the blank or pre-shaped part to a temperature of 500° C at an average heating rate of 16° C/s or more - further heating the blank or pre-shaped part to a temperature between 700 and 1000° C - hot forming the blank or pre-shaped part into a hot formed part - hardening the hot formed part. The invention also relates to a method for manufacturing a coated steel strip, blank or pre-shaped part, and to a coated steel strip, blank or part and a hot formed part.

SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, **Published:**  
GW, ML, MR, NE, SN, TD, TG).

— *with international search report (Art. 21(3))*

## METHOD FOR HOT FORMING A COATED METAL PART AND FORMED PART

The invention relates to a method for manufacturing a coated part using hot forming techniques. The invention also relates to a method for manufacturing a coated steel strip, blank or pre-formed part for the hot forming method, and to a coated steel strip, blank, part or pre-formed part and a hot formed part.

The use of hot forming techniques for the forming of a part is well known, especially for automotive purposes. Starting from a sheet that can be easily formed, the hot forming techniques provide a formed part having very high mechanical properties, such as a tensile strength above 1200 MPa.

Usually the hot forming is performed by providing a blank, heating the blank to a temperature between 700° and 1000° C and keeping the blank at that temperature for a few minutes, placing the heated blank in a hot forming apparatus, forming the blank into a part in the hot forming apparatus, and hardening the hot formed part.

When using uncoated steel, the heating of the blank before the hot forming in a hot forming apparatus can be performed under a protective atmosphere to prevent oxidation and decarburization of the steel. However, the hot forming itself and the hardening take place in air, and thus oxidation occurs; therefore, after the hot forming the hot formed parts must be descaled. To overcome this drawback, in the last ten years it has been proposed to use coated steel sheets, which sheets are heated to a temperature above the Ac1 temperature. During the heating a diffusion layer is formed between the coating and the steel sheet, providing protection against oxidation and a good adherence of the coating to the steel sheet, also at the elevated temperatures which are used for hot forming.

Though a protective atmosphere is not necessary anymore when using coated steel sheets, the known method has some drawbacks. One of the main problems is that the heating velocity of the coated steel sheets has been found to be critical. This makes the whole process more difficult to control. It also results in the heating of a steel sheet taking a considerable time, for instance 5 minutes, whereas the hot forming in the hot forming apparatus and the subsequent hardening can be performed in less than 1 minute. Manufacturing at a high production rate, as made possible by the hot forming

apparatus, can be performed by heating a number of coated steel sheets in an oven. However, when there is a delay at the hot forming apparatus the coated steel sheets remain too long in the oven, which means that they have to be scrapped. This has a considerable influence on the cost of the hot forming process. Moreover, the oven has  
5 to be very long.

It is an object of the invention to provide a method for manufacturing a coated part using hot forming techniques, which makes it possible to control the process in a more flexible and robust manner.

It is also an object of the invention to provide a method for manufacturing a  
10 coated part using hot forming techniques, which makes it possible to easily and effectively produce hot formed parts.

It is a further object of the invention to provide a method for manufacturing a coated part using hot forming techniques, which is more cost-effective than the known method.

15 Furthermore, it is an object of the invention to provide a coated steel strip, a coated steel blank and a coated pre-shaped part, and a method to produce these, which can be used in the method according to the invention.

According to the invention one or more of these objects is reached by providing a method for manufacturing a coated part having very high mechanical properties using  
20 hot forming techniques, comprising the following steps:

1. providing an annealed steel strip or blank or pre-shaped part that has been coated with a metal or metal alloy before the annealing
2. if a strip is provided, cutting a blank from the strip before or after the annealing
3. optionally forming a pre-shaped part from the blank
- 25 4. heating the blank or pre-shaped part to a temperature of 500° C at an average heating rate of 16° C/s or more
5. further heating the blank or pre-shaped part to a temperature between 700 and 1000° C
6. hot forming the blank or pre-shaped part into a hot formed part
- 30 7. hardening the hot formed part.

The inventors have found that due to the use of annealed coated steel, the heating of the annealed steel strip or blank or pre-shaped part can be performed at an average heating rate of 16° C/s or more up to a temperature of 500° C. In this way, the

heating of the blank or pre-shaped part before the hot forming thereof can be performed much faster than usual. The heating of the blank or pre-shaped part can now be performed within 3 minutes before the blank or pre-shaped part is taken out of the oven and put in the hot forming apparatus. This fast heating according to the invention is possible using a blank, which is used in the direct hot forming process, or using a pre-shaped part, which is used in the indirect hot forming process.

According to a preferred embodiment, the blank or pre-shaped part in step 4 is heated to a temperature of 625° C at an average heating rate of 16° C/s or more, and further heated in step 5 to a temperature between 700 and 1000° C before the blank or pre-shaped part is hot formed. By heating the blank or pre-shaped part to a temperature of 700° C at the high heating rate of 16° C/s or more, the heating of the blank or pre-shaped part can be performed in an even shorter time period.

According to a further preferred embodiment, the blank or pre-shaped part in step 4 is heated to a temperature of 700° C at an average heating rate of 16° C/s or more, and optionally further heated in step 5 to a temperature between 700 and 1000° C before the blank or pre-shaped part is hot formed. By heating the blank or pre-shaped part to a temperature of 700° C at the high heating rate of 16° C/s or more, the heating of the blank or pre-shaped part can be performed in an even shorter time period of less than 2 minutes.

According to a further preferred embodiment, the blank or pre-shaped part in step 4 is heated to a temperature between 700 and 900° C at an average heating rate of 16° C/s or more, and optionally further heated in step 5 to a temperature of at most 1000° C before the blank or pre-shaped part is hot formed. By heating the blank or pre-shaped part to a temperature between 700 and 900° C at the high heating rate of 16° C/s or more, the heating of the blank or pre-shaped part can be performed in a very short time period of less than 90 seconds when the average heating rate of 16° C/s or more is used to temperatures above the Ac1 temperature.

Preferably the metal or metal alloy is zinc or a zinc alloy. The inventors have found that the use of an annealed zinc or zinc alloy coating makes it possible to use very high heating rates.

According to a preferred embodiment the coating on the blank or pre-shaped part remains solid during step 4 and step 5. This has of course the advantage that the coating remains on the blank or pre-shaped part, and especially for pre-shaped parts

remains evenly spread on non-horizontal portions of the pre-shaped part and does not flow. Because the coating remains solid it does not stick to handling equipment. Moreover, no energy is needed to transform the coating from the solid into the liquid state.

5            Preferably, the heating rate is 20° C/s or more, and more preferably 25° C/s or more. These high heating rates make it possible to heat the coated blank or pre-shaped part before the hot forming of the part in a time period shorter than one minute.

            Preferably, in step 4 the blank or pre-shaped part is heated at an average heating rate of at most 50° C/s. Higher heating rates make it difficult to control the highest  
10          temperature to which the blank or pre-shaped part are heated.

            According to a preferred embodiment, the steel has the following composition in weight percent:

$0,1 < C < 0,5$   
             $0,5 < Mn < 3,0$   
15           $0,1 < Si < 0,5$   
             $Cr < 1,0$   
             $Ti < 0,2$   
             $Al < 0,1$   
             $P < 0,1$   
20           $S < 0,05$   
             $0,0005 < B < 0,08$   
            optionally:  
             $Nb < 0,1$   
             $V < 0,1$   
25          unavoidable impurities  
            the remainder being iron.

Though other metal composition are also possible, it has been found that the steel composition as given above will give very good results in most cases.

            According to a preferred embodiment the metal alloy is a zinc alloy having the  
30          following composition in weight percent:

$1.0 < Al < 5.0$ , preferably  $1.5 < Al < 2.0$   
             $1.0 < Mg < 5.0$ , preferably  $1.5 < Mg < 2.0$   
            at most 0.2 in total of one or more standard alloying elements

remainder zinc and inevitable impurities.

The inventors have found that using a zinc alloy having this composition, the blank or pre-shaped part can be heated at very high heating rates above 16° C/s. The standard alloying elements are Pb, Sb, Ti, Ca, Mn, La, Ce, Cr, Ni, Zr, and Bi.

5 According to a second aspect of the invention there is provided a method for manufacturing the annealed steel strip or blank or pre-shaped part that has been coated with a metal or metal alloy before the annealing as provided in the first aspect of the invention, comprising the following steps:

- 1 - providing a steel strip or blank or pre-shaped part
- 10 2 - coating the steel with a layer of metal or metal alloy, preferably zinc or zinc alloy
- 3 - heating the coated steel to a temperature between 700° C and 1000 ° C and keeping the strip or blank or pre-shaped part at that temperature during a time period of at most 30 minutes
- 15 4 - cooling the coated steel.

This method for manufacturing a coated and annealed steel strip or blank or pre-shaped part is thus performed independently from the hot forming process as such. The choice for a high diffusion temperature between 700° C and 1000° C means that a relatively short production time for the forming of the diffusion layer can be obtained.

20 Preferably in the method according to the second aspect of the invention, if a strip is provided the steel strip is cut to form a blank from the strip, and a pre-shaped part is formed from the blank after step 1, 2, 3 or 4. Since blanks or pre-shaped parts are used in the hot forming process, it is preferred to store and transport blanks or pre-shaped parts which can be directly used in the hot forming process.

25 According to a third aspect of the invention the coated steel strip, blank, part or pre-shaped part as produced according to the method of the second aspect of the invention is provided with a coating of zinc or zinc alloy, wherein the outer layer of the coating on average contains more than 5 weight % Fe over a depth of 3 µm. A coated steel having more than 5 weight % Fe over a depth of 3 µm can be used in the hot  
30 forming of the blank or pre-shaped part.

Preferably the outer layer of the coating on average contains more than 10 weight % Fe over a depth of 3 µm, more preferably more than 20 weight % Fe, still more preferably more than 30 weight % Fe, and most preferably more than 40 weight

% Fe. A higher amount of Fe in the outer layer of the coating means that the coating and the Fe from the steel have better diffused.

According to a fourth aspect of the invention a hot formed coated part is provided that is manufactured using the method according to the first aspect of the invention.

5 The invention will be elucidated referring to the experiments hereinafter.

Figure 1 shows the heating curves for a substrate covered with two different coatings.

The substrate is a 1.5 mm thick 22MnB5 boron steel.

10 Example 1 is a blank which is covered by a zinc alloy containing 1.6 weight% Al and 1.6 weight% Mg, the remainder being zinc. The coating has a coating weight of 140 g/m<sup>2</sup> (70 g/m<sup>2</sup> per side).

Example 2 is a blank which is covered by the same coating as in example 1, but the blank has been annealed at 750° C. Due to the annealing, the average Fe content of the coating is more than 20%.

15 The two blanks have been put in an oven together. The oven temperature had been set on 920° C. The blanks have been heated to a temperature of approximately 850° C; the temperature of the blanks has been measured using a thermocouple.

Figure 1 shows the heating curves of the blanks. The horizontal axis shows the dwell time t of the blanks in the oven in seconds; the vertical axis shows the 20 temperature T of the blanks in ° C.

Inspection of the blanks showed that the coating of example 1 became liquid.

The coating of example 2 remained solid through the full heating up to 850° C.

Figure 1 shows that the blank of example 2 is heated with a heating rate of more than 16° C/s up to a temperature of 500° C, and that the average heating rate is 16° C up 25 to a temperature of approximately 650° C. Contrary to this the heating rate of the blank of example 1 is far below 16° C/s.

The time to reach the Ac3 temperature, at which the steel will fully transform into austenite and which is approximately 850° C/s for the present substrate, is approximately 110 seconds for example 2 and approximately 180 seconds for example 30 1.

After the blanks have reached the Ac3 temperature they can be given some additional time to homogenise into austenite before they can be put in a hot forming apparatus.

**CLAIMS**

1. Method for manufacturing a coated part having very high mechanical properties using hot forming techniques, comprising the following steps:
  - 5           1. providing an annealed steel strip or blank or pre-shaped part that has been coated with a metal or metal alloy before the annealing
  2. if a strip is provided, cutting a blank from the strip before or after the annealing
  3. optionally forming a pre-shaped part from the blank
  - 10          4. heating the blank or pre-shaped part to a temperature of 500° C at an average heating rate of 16° C/s or more
  5. further heating the blank or pre-shaped part to a temperature between 700 and 1000° C
  6. hot forming the blank or pre-shaped part into a hot formed part
  - 15          7. hardening the hot formed part.
2. Method for manufacturing a coated part according to claim 1, wherein the blank or pre-shaped part in step 4 is heated to a temperature of 625° C at an average heating rate of 16° C/s or more, and further heated in step 5 to a temperature  
20           between 700 and 1000° C before the blank or pre-shaped part is hot formed.
3. Method for manufacturing a coated part according to claim 1, wherein the blank or pre-shaped part in step 4 is heated to a temperature of 700° C at an average heating rate of 16° C/s or more, and optionally further heated in step 5 to a  
25           temperature between 700 and 1000° C before the blank or pre-shaped part is hot formed.
4. Method for manufacturing a coated part according to claim 1, wherein the blank or pre-shaped part in step 4 is heated to a temperature between 700 and 900° C at  
30           an average heating rate of 16° C/s or more, and optionally further heated in step 5 to a temperature of at most 1000° C before the blank or pre-shaped part is hot formed.

5. Method for manufacturing a coated part according to any one of the preceding claims, wherein the metal or metal alloy is zinc or a zinc alloy.
6. Method according to any one of the preceding claims, wherein the coating on the blank or pre-shaped part remains solid during step 4 and step 5.
7. Method according to any one of the preceding claims, wherein in step 4 the blank or pre-shaped part is heated at an average heating rate of at most 50 ° C/s.
8. Method according to any one of the preceding claims, wherein the steel has the following composition in weight percent:
- $0,1 < C < 0,5$   
 $0,5 < Mn < 3,0$   
 $0,1 < Si < 0,5$   
 $Cr < 1,0$   
 $Ti < 0,2$   
 $Al < 0,1$   
 $P < 0,1$   
 $S < 0,05$   
 $0,0005 < B < 0,08$   
optionally:  
 $Nb < 0,1$   
 $V < 0,1$   
unavoidable impurities  
the remainder being iron.
9. Method according to any one of the preceding claims, wherein the metal alloy is a zinc alloy having the following composition in weight percent:
- $1.0 < Al < 5.0$ , preferably  $1.5 < Al < 2.0$   
 $1.0 < Mg < 5.0$ , preferably  $1.5 < Mg < 2.0$   
at most 0.2 in total of one or more standard alloying elements  
remainder zinc and inevitable impurities.

10. Method for manufacturing the annealed steel strip or blank or pre-shaped part that has been coated with a metal or metal alloy before the annealing, as provided in any one of the preceding claims, comprising the following steps:
- 1 - providing a steel strip or blank or pre-shaped part
  - 5 2 - coating the steel with a layer of metal or metal alloy, preferably zinc or zinc alloy
  - 3 - heating the coated steel to a temperature between 700° C and 1000 ° C and keeping the strip or blank or pre-shaped part at that temperature during a time period of at most 30 minutes
  - 10 4 - cooling the coated steel.
11. Method according to claim 10, wherein if a strip is provided the steel strip is cut to form a blank from the strip and a pre-shaped part is formed from the blank after step 1, 2, 3 or 4.
- 15 12. Coated steel strip, blank, part or pre-shaped part provided with a coating of zinc or zinc alloy as produced according to the method of claim 10 or 11, wherein the outer layer of the coating on average contains more than 5 weight % Fe over a depth of 3 µm.
- 20 13. Coated steel strip, blank, part or pre-shaped part according to claim 12, wherein the outer layer of the coating on average contains more than 10 weight % Fe over a depth of 3 µm, preferably more than 20 weight % Fe, more preferably more than 30 weight % Fe, still more preferably more than 40 weight % Fe.
- 25 14. Part provided by performing the method according to any one of the claims 1 – 9.

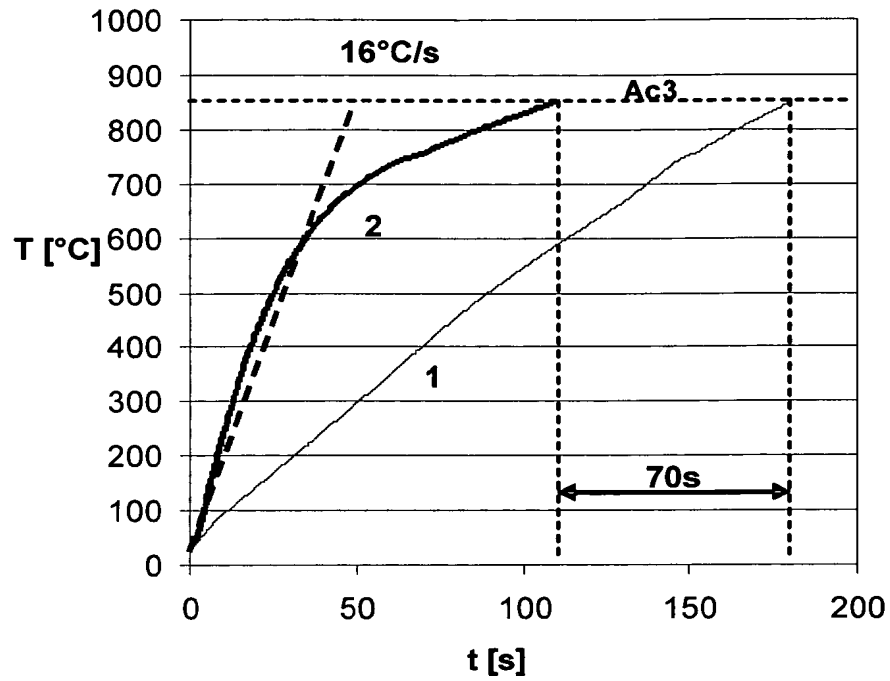


Figure 1

## INTERNATIONAL SEARCH REPORT

International application No  
PCT/EP2011/003601

## A. CLASSIFICATION OF SUBJECT MATTER

INV. C21D8/04 C21D9/46 C22C38/02 C22C38/04 C21D1/673  
C21D9/48

ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C21D C22C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, CHEM ABS Data, WPI Data

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Further documents are listed in the continuation of Box C.



See patent family annex.

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

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## INTERNATIONAL SEARCH REPORT

International application No

PCT/EP2011/003601

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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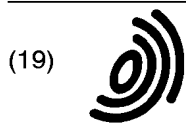
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Information on patent family members

International application No

PCT/EP2011/003601

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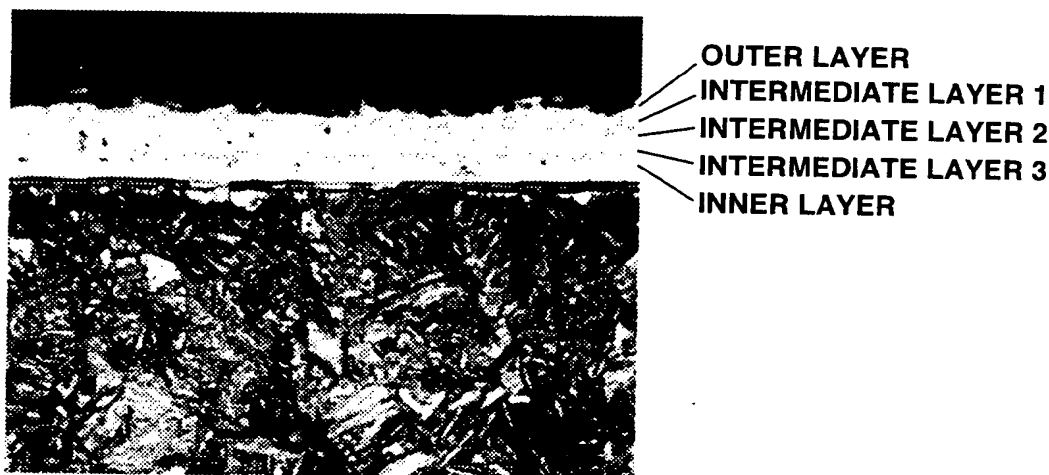
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(54) **Aluminium-coated structural member and production method**

(57) An aluminum-coated structural member includes an Al-Si-Fe alloy layer formed on a steel substrate layer. The alloy layer includes a softer region having a hardness smaller than or equal to a hardness of

the steel substrate layer, extends from the surface of the steel substrate layer toward a surface of the alloy layer, over a depth range greater than or equal to 50% of a thickness of the alloy layer. The Al-Si-Fe alloy layer has an oxide weight smaller than or equal to 500 mg/dm<sup>2</sup>.

**FIG.2A**



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**Description****BACKGROUND OF THE INVENTION**

5 [0001] The present invention relates to an aluminum-coated structural member and a production method thereof. More specifically, the present invention relates to an aluminum-coated structure member, such as a structural member used for motor vehicles, produced by applying heat treatment and forming operation to aluminum-coated steel sheet, and coated with an alloy layer of an Al-Si-Fe alloy for improvement in formability, weldability and corrosion resistance.

10 [0002] A Published Japanese Patent Application Publication No. 2000-204463 shows a hot-dip aluminum-coated steel sheet which can be used for motor vehicles as fuel tank, muffler, heat shield plate, etc., from the viewpoint of excellent corrosion resistance due to oxide film, and less burden on the environment due to non-inclusion of heavy metal such as lead.

**SUMMARY OF THE INVENTION**

15 [0003] A hard Al-Fe alloy layer contained in a hot-dip aluminum-coated steel sheet at an interface with a steel substrate is more or less problematical in the formability. Moreover, the alloy layer tends to suffer cracks in a forming operation, and hence sometimes entails another treatment to ensure sufficient corrosion resistance. Weldability is another problem because an aluminum coating layer is low in melting point, and an aluminum single surface layer is insufficient in thermal conductivity. Accordingly, the hot-dip aluminum-coated steel sheet is limited in application.

20 [0004] It is an object of the present invention to provide aluminum-coated structural member and production method to the advantage of corrosion resistance, formability and weldability and wider application in motor vehicles.

25 [0005] According to one aspect of the present invention, an aluminum-coated structural member comprises: a steel substrate layer; and an Al-Si-Fe alloy layer formed on a surface of the steel substrate layer, the alloy layer including a softer region having a hardness smaller than or equal to a hardness of the steel substrate layer, extending from the surface of the steel substrate layer toward a surface of the alloy layer, and having a thickness greater than or equal to 50% of a thickness of the alloy layer, the Al-Si-Fe alloy layer having an oxide weight smaller than or equal to 500 mg/dm<sup>2</sup>.

30 [0006] According to another aspect of the invention, a production method of an aluminum-coated structural member comprises: a first method element of heating a hot-dip aluminum-coated steel sheet at a heating rate in a range of 1~10°C/sec; a second method element of holding the hot-dip aluminum steel sheet at a raised temperature in a temperature range of 900~950°C for a duration in a range of 2~8 minutes; a third method element of cooling the hot-dip aluminum-coated steel sheet to a temperature in a temperature range of 700~800°C at a cooling rate in a range of 5~15°C/sec; a fourth method element of forming the hot-dip aluminum-coated steel sheet into a predetermined shape in the temperature range of 700~800°C; and a fifth method element of cooling the hot-dip aluminum-coated steel sheet in the predetermined shape rapidly from the temperature range of 700~800°C to a lower temperature lower than or equal to 300°C at a cooling rate in a range of 20~100°C/sec.

**BRIEF DESCRIPTION OF THE DRAWINGS**

40 [0007] FIG. 1 is a perspective view showing the shape and dimensions of specimens used in a test 1 according to an embodiment of the present invention, for evaluation of the formability.

[0008] FIG. 2A is a view of a photograph showing a microstructure of a coating layer in a structural member in a practical example No. 1 in the test 1.

45 [0009] FIG. 2B is a view of a photograph showing a microstructure of a coating layer in a structural member in a practical example No. 2 in the test 1.

[0010] FIG. 3 is a graph showing performance of consecutive spot welds in spot welding, of a structural member in the embodiment of the present invention in comparison with an ordinary hot-dip aluminum-coated steel sheet.

[0011] FIG. 4 is a view of a table 1 showing the result of test 1 in the embodiment.

50 [0012] FIG. 5 is a view of a table 2 showing the result of test 3 in the embodiment.

**DETAILED DESCRIPTION OF THE INVENTION**

55 [0013] In general, a hot-dip aluminum-coated steel sheet is formed by immersing a strip continuously in a bath of molten aluminum. According to the coating metal component, hot-dip aluminum-coated steel sheets are classified broadly into two categories. One employs, as coating metal, pure aluminum (for weather resistance), and the other employs Al-Si alloy (for heat resistance) containing Si in addition to Al. An embodiment according to the present invention employs heat resisting coated steel sheet containing an additive of Si (approximately 3~11 % Si).

[0014] The addition of Si to the coating layer is for restraining the growth of a hard Al-Fe alloy layer in a boundary

between the steel and coating during the process of aluminizing, and thereby for improving the adhesion and corrosion resistance of the Al coating layer. In such a hot-dip aluminum-coated steel sheet, there is formed an Al-Si-Fe alloy layer in the boundary between the Al coating and the steel substrate layer.

**[0015]** When a hot-dip aluminum-coated steel sheet of such a type is subjected to heat treatment under certain conditions, the Al-Si-Fe layer can grow and spread entirely until the surface is reached. FIG. 2A shows a sectional structure in one example of an aluminum-coated steel sheet including such an Al-Si-Fe layer grown to the surface by the heat treatment.

**[0016]** During the growth of the Al-Si-Fe alloy layer by the heat treatment, mutual diffusion between Al coating and steel substrate layer produces various Al-Si-Fe ternary intermetallic compounds such as  $\alpha$ -FeAlSi phase (alpha FeAlSi),  $\beta$ -FeAlSi phase (beta FeAlSi) and  $\gamma$ -FeAlSi phase (gamma FeAlSi). The Al-Si-Fe alloy layer formed by the heat treatment has a layered structure made up of these intermetallic compounds, and offers advantages of formability, corrosion resistance and weldability.

**[0017]** When the hardness of Al-Si-Fe alloy layer exceeds the hardness of steel substrate, the Al-Si-Fe alloy layer exerts adverse influence on the formability in the forming process of the steel sheet, and on the corrosion resistance by deteriorating the compliance of the coating and increasing the possibility of cracks in the coating. Therefore, in this embodiment, there is formed a less hard or soft layer having a hardness smaller than the hardness of the steel substrate at or near the boundary between the steel and coating. This soft layer prevents crack, even if produced by any chance, from extending deeper from the surface toward the steel substrate. In this embodiment, the thickness of this soft layer is made equal to or greater than 50% of the total thickness of the alloy coating layer. The thick soft layer extending from a level at or near the boundary with the steel substrate, to or beyond the middle in the depth of the coating layer is effective for preventing progress of crack and securing sufficient corrosion resistance.

**[0018]** Oxygen contained in the form of oxides in the coating alloy layer causes pinholes in the process of electro deposition and thereby deteriorates the adhesion between the coating and deposited film if the amount of oxygen is increased. Therefore, the oxide quantity or oxide weight is made equal to or less than 500 mg/dm<sup>2</sup>. In this specification, "oxide weight" is defined, as mentioned later, as a difference in mass between the mass of a specimen after a heat treatment at 950°C and the mass of the specimen before the heat treatment.

**[0019]** The mechanism of formation of the layered structure is not fully evident. Currently, the presumption by the inventors of the present invention is as follows: (1) The retention of a specimen at an elevated temperature causes the diffusion of Fe, and produce a concentration gradient in Fe and Si in the coating layer. (2) In this case, the coating alloy phase is urged to transform into a stable compound phase determined by the composition at each depth, so that there are formed different compound layers in different regions of depth. (3) Therefore, by controlling the raised temperature of the heating and the holding time at the raised temperature, the diffusion quantity of Fe is varied and the degree of transformation into a stable compound is varied.

**[0020]** The Al-Si-Fe alloy layer contiguous with the steel substrate layer is softened by the diffusion of Fe from the steel substrate layer, and made more adhesive with steel, and hence more formable. Specifically, a preferable range of the Fe content is 85~95%. When the Fe content is in this range, the hardness of the soft layer contacting with the steel substrate is equal to or lower than 80% (300~400 Hv) of an average hardness of the coating layer. When the Fe content is less than 85%, the above-mentioned advantageous effect by the diffusion of Fe tends to be insufficient. When the Fe content is more than 95%, the diffusion of Fe tends to become excessive and to make the Al content too low to obtain the corrosion resistance in the outermost layer in the multi-layer structure. In this specification, the percent % is used to mean "mass %" (percent by mass) unless otherwise specified.

**[0021]** The Al-Si-Fe alloy layer is made softer by the heat treatment and diffusion of Fe from the steel substrate. However, excessive diffusion of Fe could decrease the Al content below a level needed for the corrosion resistance. In order to maintain the minimum Al content from the viewpoint of corrosion resistance, and to form a layer softer than the steel base material, it is preferable to set the Al content within the range of 25~40% at least in one of three or more layers of the multi-layer structure. The corrosion resistance tends to be insufficient when the Al content is less than 25%. The formation of a softer layer becomes difficult, and the adhesion with the steel base could become poor when the Al content is more than 40%.

**[0022]** In the case of a multi-layer structure including three or more subordinate layers differing in composition or compound, there is one or more intermediate layers between an innermost layer contiguous with the steel substrate layer and an outermost layer forming the surface of the coating layer. Preferably, the intermediate layer or at least one of the intermediate layers contains 25~40% Al.

**[0023]** The weldability is improved by alloying of the aluminum coating layer with Fe diffused from the steel base material. The alloyed Al-Si-Fe coating layer is higher in melting point than an Al coating layer formed by hot dipping, so that welding is feasible from low current condition.

Moreover, adhesion of coating metal to a welding electrode is prevented in spot welding, to the advantage of improvement in consecutive spot welding.

**[0024]** Thermal stability is improved by alloying an Al-Si-Fe coating layer entirely into a multi-layer structure such as

a five-layer structure having five layers of different alloy compositions. A multi-layer structure including a hard outermost layer is advantageous for maintaining good contact with a welding gun in spot welding even under great pressure for high tension material.

**[0025]** As the Fe content increases with the progress of alloying between the aluminum coating layer and Fe diffused from the steel base, the thermal conductivity of the coating layer decreases and the welding with lower current becomes feasible. It is possible to combine weldability and corrosion resistance by making the Fe concentration in the surface or outermost layer of the coating in the range of 35~50%. The coating layer having a surface Fe concentration in this range can be formed by forming an Al-Si-Fe layer having a multi-layer structure, or by forming an Al-Si-Fe layer having a concentration gradient of Fe content. The improvement in weldability is insufficient when the Fe concentration in the outermost layer is less than 35%. The Al content is decreased relatively and the corrosion resistance degrades when the Fe concentration in the outermost layer is more than 50%.

**[0026]** An aluminum-coated structural member having an Al-Si-Fe alloy layer on the surface as mentioned above can be obtained by the following process. First, a heat resisting hot-dip aluminum-coated steel sheet containing Si in a coating layer is heated at a rate of 1~10°C/sec to a temperature in the temperature range of 900~950°C, and held at that raised temperature for a holding time (or duration) of 2~8 minutes. In this case, the aluminum coating could melt locally when the hot-dip aluminum-coated steel sheet is heated at a heating rate faster than 10°C/sec to a temperature higher than 900°C. The coating layer could oxidize when the heating rate is slower than 1°C/sec. The progress of austenitizing in the steel base material is too slow to obtain a sufficient strength as a structural member when the raised temperature at which the specimen is held is lower than 900°C. When the raised temperature is higher than 950°C, the undesired oxidation of the coating layer proceeds, and the diffusion of Fe proceeds too much up to the surface of the coating layer. When the time for holding at the raised temperature is shorter than 2 minutes, then the formation of Al-Si-Fe alloy layer becomes insufficient. When the time for holding at the raised temperature is longer than 8 minutes, the oxidation of coating layer proceeds, and undesired influence is exerted on the adhesiveness of the coating film by electro deposition.

**[0027]** After the heat treatment for holding in the above-mentioned temperature range, the steel sheet is carried from a heating furnace to a forming press. During the transportation, cooling starts. In this case, the rate of cooling after the heat treatment is required to be 5~15°C/sec in order to perform the forming operation at a predetermined temperature and the rapid cooling from a predetermined temperature. When the cooling rate is slower than 5°C/sec, the oxidation of the coating layer becomes conspicuous. For cooling at a rate higher than 15°C/sec, the need arises for forced cooling.

**[0028]** The forming operation into a desired shape is started in the temperature range of 700~800°C. After the forming operation, the shaped sheet is cooled rapidly from the temperature range of 700~800°C, to a temperature lower than or equal to 300°C at a rate in the range of 20~100°C/sec. By this rapid cooling, the steel base material is cooled while preserving the Al-Si-Fe alloy layer formed by the heat treatment, and an aluminum-coated structural member of a desired shape is obtained. The above-mentioned conditions of the heat treatment are adequate also for quench hardening of the steel base material, and hence effective for enhance the strength of the material. To start the forming operation at a temperature higher than 800°C is not desirable because reheating is required and oxidation of coating is promoted. When the forming operation is started at a temperature lower than 700°C, the quenching temperature is too low for martensitic transformation. When the cooling rate after the forming operation is slower than 20°C/sec, the transformation to martensite is insufficient, and the diffusion of Fe proceeds too much. When the cooling rate after the forming operation is faster than 100°C/sec, the cost for facility is increased significantly for the need for improving a cooling system in production equipment. The rapid cooling to a temperature lower than or equal to 300°C is effective for preventing ferrite phase from being left over, and promoting martensitic transformation sufficiently.

**[0029]** The following is explanation on practical examples of the embodiment.

**[0030]** (Test 1) Aluminum-coated or aluminized structural members were produced under the following conditions and investigation was conducted on the formability, corrosion resistance and weldability.

**[0031]** [Hot-dip aluminum-coated steel sheet to be tested]

Composition of original sheet for coating: 0.22%C, 0.22%Si, 1.0%Mn, 0.015%P, 0.007%S.

Sheet thickness: 1.4mm.

Si concentration in Aluminum coating: 5%.

Thickness of aluminum coating layer: 10~20 µm.

**[0032]** [Test Procedure] From the above-mentioned hot-dip aluminum-coated steel sheet, a flat plate having a width of 160 mm, and a length of 160mm was cut out, heat-treated under the following conditions and formed into a shape shown in FIG. 1. Then, investigation was conducted on the composition of the alloy coating layer, hardness, formability, corrosion resistance and weldability.

|                                |         |
|--------------------------------|---------|
| <Conditions of Heat treatment> |         |
| Heating rate                   | 8°C/sec |

(continued)

| <Conditions of Heat treatment>          |           |
|-----------------------------------------|-----------|
| Heating temperature                     | 900~950°C |
| Holding time                            | 2~10min   |
| Rate of Primary cooling                 | 7°C/sec   |
| Temperature at a start of forming       | 720°C     |
| Temperature at a start of rapid cooling | 720°C     |
| Rate of Secondary cooling (quenching)   | 30°C/sec  |
| Temperature at an end of cooling        | 100°      |

**[0033]** [Results of Testing] Table 1 shows the results of the test. In all the cases in Table 1, a softest layer in a coating is a layer contiguous with a steel substrate layer of steel base material. Table 1 includes a column of production conditions, divided into a column of raised temperature and a column of hold time; a column of coating layer structure and composition (%), divided into a column of component and hardness, a column of inner layer (INNER), a column of first intermediate layer (INTER1), a column of a second intermediate layer (INTER2), a column of a third intermediate layer (INTER3), and a column of outer layer (OUTER); a column of hardness of steel base material (Hv); a column of hardness of coating layer (Hv), divided into a column of softest layer, a column of average, and a column of a range (%) softer than the base material; a column of oxide weight (mg/dm<sup>2</sup>); a column of formability; a column of corrosion resistance; and a column of weldability.

**[0034]** <Formability> Cracking was checked in the Al-Si-Fe alloy layer after the forming operation. Test samples having no cracks reaching the steel substrate layer were evaluated as proper and indicated by a sign of a circle. Samples having one or more cracks reaching the steel substrate layer were evaluated as improper and indicated by a sign of a cross. The samples showed good formability except the structural member of No. 2 coated with a coating layer so hardened that an average hardness of the coating layer is greater than the hardness of the steel base material, and the hardness of the coating layer is harder in a region spreading over a depth range greater than 50% of the thickness of the alloy layer. As a typical example, FIG. 2A shows a micro structure of the coating layer in the structural member of No. 1 having no cracks, and FIG. 2B show a micro structure of the coating layer in the structural member of No. 2 suffering a crack.

**[0035]** <Corrosion Resistance> Produced structural members were subjected to a salt spray test (SST) as defined by JIS Z2371, to evaluate the corrosion resistance of each structural member. As a result, a poorer corrosion resistance was shown by the structural member of No. 3 in which the diffusion of Fe was excessive because of too long a holding time at the raised temperature, and hence the aluminum content was too low in the outermost layer.

**[0036]** <Weldability> Produced structural members were subjected to spot welding in succession under the conditions mentioned below. As the number of consecutive spots increases, in general, a nugget diameter tends to decrease and the weld strength tends to decrease. Accordingly, spot welding was carried out under the following welding conditions until the number of spots reaches a number smaller than a class A judgment reference in JIS Z3140. The weldability was determined in accordance with the number of consecutive spots. The No. 3 structural member was judged as improper and indicated by X since the number of consecutive spots was about 500 below the level of the judgment reference. As to the structural members other than the No. 3 structural member, numbers of spot welds up to 3000, to a level satisfying the judgment reference were obtained and the spot welding was ended at that point. Thus, the structural members other than the No. 3 specimen are indicated as proper by circle. Thus, the results of the test show that the weldability is degraded when the holding time for the heat treatment is too long and the diffusion of Fe softens the coating layer, as in the No. 3 sample. The No. 3 sample is poorer in corrosion resistance and weldability. However, the No. 3 sample is good in formability. Therefore, the No. 3 sample is still adequate for some applications.

| [Welding Conditions] |                                                                                                                               |
|----------------------|-------------------------------------------------------------------------------------------------------------------------------|
| Base material        | Aluminum-coated steel sheet (thickness of 1.2mm) and GA material (2.0 mm thick) of a type having a tensile strength of 440Mpa |
| Pressing Force       | 5.98 kN                                                                                                                       |
| Squeeze              | 50 Hz                                                                                                                         |
| Up slope             | 3 Hz                                                                                                                          |
| Energization         | 9kA×18Hz                                                                                                                      |
| Hold                 | 21 Hz                                                                                                                         |

[0037] (Test 2) By using a hot-dip aluminum-coated steel sheet heat-treated in the same manner as the No. 1 sample of Test 1, and a hot-dip aluminum-coated steel sheet not subjected to heat treatment, as a comparative sample, comparison was made about the characteristic of consecutive spot welds under the following conditions. Fig. 3 shows the results of the test 2 for samples of the embodiment and comparative samples. The sample using the hot-dip aluminum-coated steel sheet without heat treatment shows about 500 points whereas the sample of this example according to the present invention shows 3000 points or more.

| (Test Conditions) |                                                                                                         |
|-------------------|---------------------------------------------------------------------------------------------------------|
| Base material     | Aluminum-coated steel sheet (1.2t) and GA material (2.0t) of a type having a tensile strength of 440Mpa |
| Pressing Force    | 610 kgf                                                                                                 |
| Squeeze           | 50 cyc                                                                                                  |
| Up slope          | 3 cyc                                                                                                   |
| Energization      | 9kA×18 cyc                                                                                              |
| Hold              | 21 cyc                                                                                                  |

[0038] (Test 3) Hot-dip aluminum-coated steel sheets were held at 950°C for various lengths of time, to form an Al-Si-Fe alloy layer, and subjected to electro deposition coating onto the alloy layer. Then, the samples were checked to examine a relation between the oxide weight in the alloy layer and the paintability. The results are shown in Table 2. The oxide weight herein is defined as a difference in mass between the result of mass measurement by a chemical balance before the heating and the result of mass measurement after the heating.

[0039] As evident from Table 2, when the oxide weight becomes greater than 500mg/dm<sup>2</sup>, the occurrence of pinhole becomes noticeable in the deposited coating film. In the samples impaired with pinholes, the adhesion of the coating film is insufficient, and the corrosion resistance is not improved sufficiently by the electro deposition coating.

[0040] In the aluminum-coated structural member according to the embodiment of the present invention, the Al-Si-Fe alloy layer on the steel substrate or base layer prevents a hard layer from being formed between the steel substrate and the coating, and instead forms a softer region ranging wide in the depth direction. Moreover, the alloy layer is low in the oxide weight. Therefore, the embodiment can improve the formability and the paintability, and attain both corrosion resistance and weldability by balancing the Al content and Fe content.

[0041] In the production method according to the embodiment of the present invention, a steel sheet coated with a hot-dip aluminum coating layer containing Si is heat-treated, formed into a desired shape, and cooled rapidly. Therefore, this production method can improve the corrosion resistance, paintability, weldability and formability.

[0042] This application is based on a prior Japanese Patent Application No. 2002-202770 filed on July 11, 2002. The entire contents of this Japanese Patent Applications No. 2002-202770 are hereby incorporated by reference.

[0043] Although the invention has been described above by reference to certain embodiments of the invention, the invention is not limited to the embodiments described above. Modifications and variations of the embodiments described above will occur to those skilled in the art in light of the above teachings. The scope of the invention is defined with reference to the following claims.

## Claims

1. An aluminum-coated structural member comprising:

a steel substrate layer; and  
an Al-Si-Fe alloy layer formed on a surface of the steel substrate layer, the alloy layer including a softer region having a hardness smaller than or equal to a hardness of the steel substrate layer, extending from the surface of the steel substrate layer toward a surface of the alloy layer, and having a thickness greater than or equal to 50% of a thickness of the alloy layer, the Al-Si-Fe alloy layer having an oxide weight smaller than or equal to 500 mg/dm<sup>2</sup>.

2. The aluminum-coated structural member as claimed in Claim 1, wherein the Al-Si-Fe alloy layer has a multi-layer structure including an inner layer contiguous with the steel substrate layer, and an outer layer forming the surface of the alloy layer.

3. The aluminum-coated structural member as claimed in Claim 2, wherein the inner layer is a softest layer in the

multi-layer structure.

4. The aluminum-coated structural member as claimed in Claim 2 or 3, wherein the inner layer contains 85~95% Fe, and the inner layer is softer in Hv hardness than an average hardness of the alloy layer by a percentage more than or equal to 20%.

5. The aluminum-coated structural member as claimed in Claim 2, 3 or 4, wherein the multi-layer structure of the Al-Si-Fe alloy layer further comprises an intermediate layer containing 25~40% Al, and the intermediate layer is lower in hardness than the steel substrate layer.

6. The aluminum-coated structural member as claimed in Claim 2, 3 or 4, wherein the multi-layer structure of the Al-Si-Fe alloy layer further comprises a plurality of intermediate layers, at least one of the intermediate layers containing 25~40% Al, and being lower in hardness than the steel substrate layer.

7. The aluminum-coated structural member as claimed in Claim 2, 3, 4, 5 or 6, wherein the outer layer of the Al-Si-Fe alloy layer is harder than the steel substrate layer.

8. The aluminum-coated structural member as claimed in Claim 2, 3, 4, 5, 6 or 7, wherein the outer layer contains 35~50% Fe.

9. An aluminum-coated structural member comprising:

a steel substrate layer; and

an Al-Si-Fe alloy layer formed on a surface of the steel substrate layer, the Al-Si-Fe alloy layer having a multi-layer structure including an inner layer contiguous with the steel substrate layer, an outer layer forming a surface of the alloy layer and an intermediate layer formed between the inner and outer layers, the inner layer containing 85~95% Fe, the intermediate layer containing 25~40% Al, and the outer layer being harder than the steel substrate layer.

10. A production method of an aluminum-coated structural member comprising:

heating a hot-dip aluminum-coated steel sheet at a heating rate in a range of 1~10°C/sec;

holding the hot-dip aluminum steel sheet at a raised temperature in a temperature range of 900~950°C for a duration in a range of 2~8 minutes;

cooling the hot-dip aluminum-coated steel sheet to a temperature in a temperature range of 700~800°C at a cooling rate in a range of 5~15°C/sec;

forming the hot-dip aluminum-coated steel sheet into a predetermined shape in the temperature range of 700~800°C; and

cooling the hot-dip aluminum-coated steel sheet in the predetermined shape rapidly from the temperature range of 700~800°C to a lower temperature lower than or equal to 300°C at a cooling rate in a range of 20~100°C/sec.

**FIG.1**

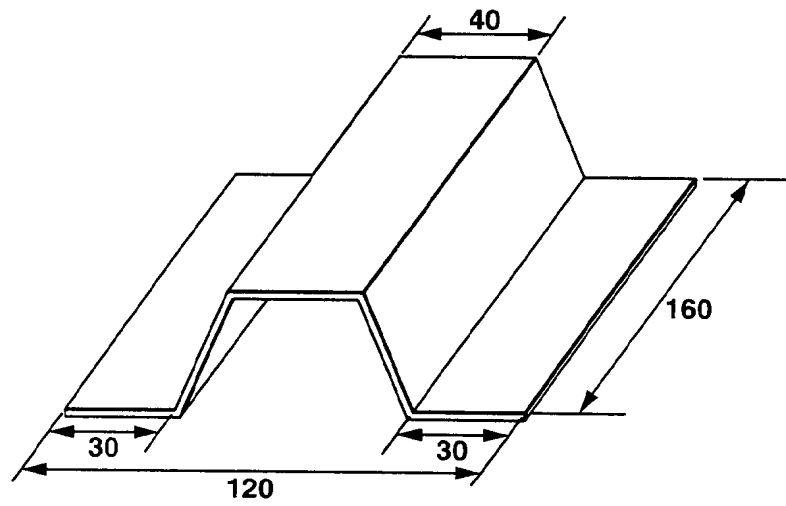


**FIG.2A**

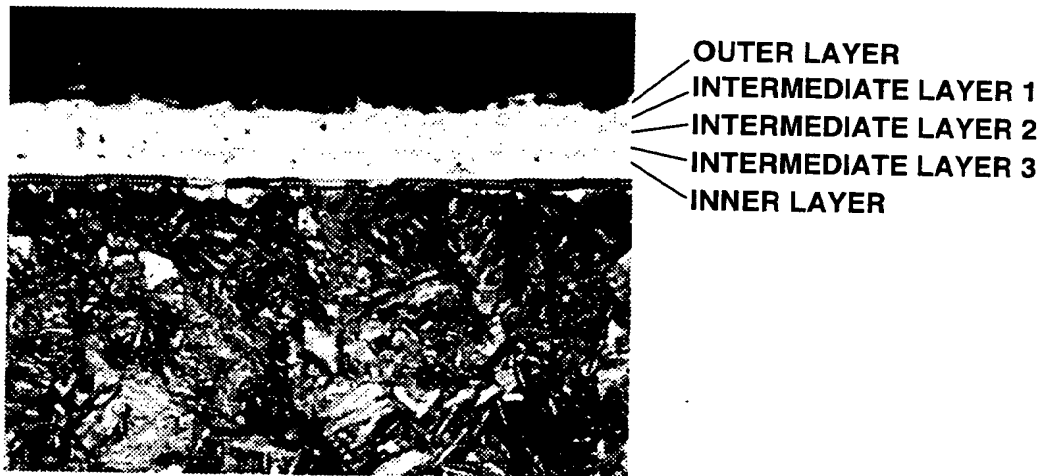


**FIG.2B**

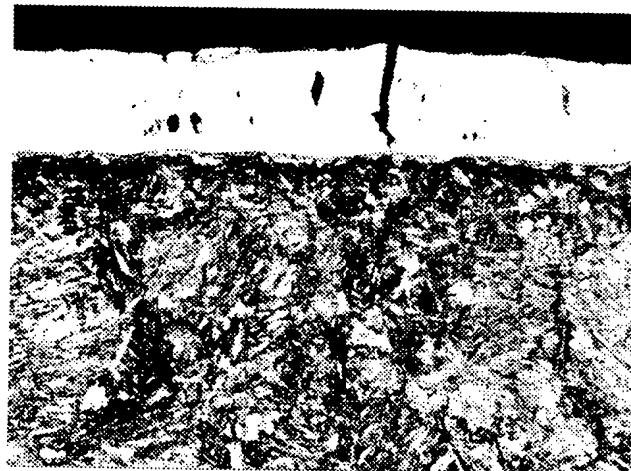


**FIG.3**

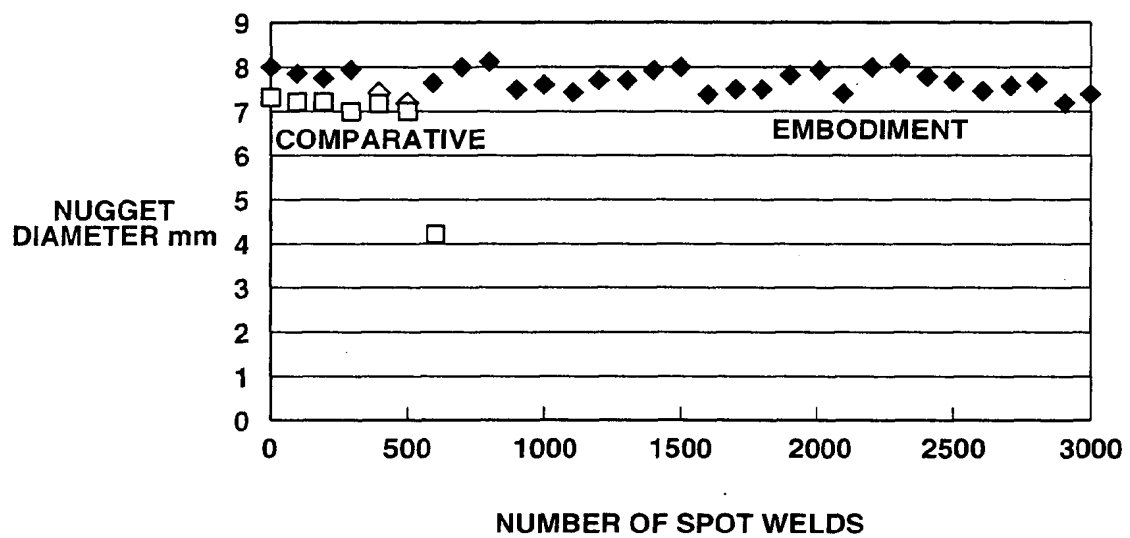


FIG.4

TABLE 1

| No. | PRODUCTION              |                  | COATING LAYER STRUCTURE & COMPOSITION (%) |       |         |         |         |       |                  |                  | STEEL BASE HARDNESS (Hv) | COATING LAYER HARDNESS (Hv) |     |   | OXIDE WEIGHT (mg/dm <sup>2</sup> ) | FORMABILITY | CORROSION RESISTANCE | WELDABILITY |
|-----|-------------------------|------------------|-------------------------------------------|-------|---------|---------|---------|-------|------------------|------------------|--------------------------|-----------------------------|-----|---|------------------------------------|-------------|----------------------|-------------|
|     | RAISED TEMPERATURE (°C) | HOLD TIME (min.) | COMPONENT & HARDNESS                      | INNER | INTER 1 | INTER 2 | INTER 3 | OUTER | SOFTTEST AVERAGE | SOFTER RANGE (%) |                          |                             |     |   |                                    |             |                      |             |
| 1   | 920                     | 3                | Al                                        | 7.7   | 31.0    | 48.4    | 31.6    | 54.8  | 450              | 310              | 415                      | 51                          | 300 | ○ | ○                                  | ○           |                      |             |
|     |                         |                  | Si                                        | 2.0   | 3.9     | 1.2     | 5.7     | 0.8   |                  |                  |                          |                             |     |   |                                    |             |                      |             |
|     |                         |                  | Fe                                        | 89.6  | 65.9    | 50.1    | 62.0    | 44.5  |                  |                  |                          |                             |     |   |                                    |             |                      |             |
|     |                         |                  | Hv                                        | 310   | 360     | 750     | 700     | 800   |                  |                  |                          |                             |     |   |                                    |             |                      |             |
| 2   | 900                     | 2                | Al                                        | 27.2  | 48.4    | 31.6    | —       | 54.9  | 415              | 380              | 455                      | 22                          | 275 | × | ○                                  | ○           |                      |             |
|     |                         |                  | Si                                        | 4.1   | 1.2     | 5.7     | —       | 0.8   |                  |                  |                          |                             |     |   |                                    |             |                      |             |
|     |                         |                  | Fe                                        | 67.2  | 50.1    | 61.9    | —       | 44.1  |                  |                  |                          |                             |     |   |                                    |             |                      |             |
|     |                         |                  | Hv                                        | 380   | 760     | 720     | —       | 810   |                  |                  |                          |                             |     |   |                                    |             |                      |             |
| 3   | 930                     | 10               | Al                                        | 9.4   | —       | —       | —       | 11.6  | 400              | 305              | 385                      | 78                          | 550 | ○ | ×                                  | ×           |                      |             |
|     |                         |                  | Si                                        | 1.3   | —       | —       | —       | 1.8   |                  |                  |                          |                             |     |   |                                    |             |                      |             |
|     |                         |                  | Fe                                        | 88.4  | —       | —       | —       | 85.7  |                  |                  |                          |                             |     |   |                                    |             |                      |             |
|     |                         |                  | Hv                                        | 305   | —       | —       | —       | 410   |                  |                  |                          |                             |     |   |                                    |             |                      |             |
| 4   | 950                     | 5                | Al                                        | 6.0   | 27.8    | 51.9    | 30.4    | 47.4  | 440              | 280              | 400                      | 59                          | 385 | ○ | ○                                  | ○           |                      |             |
|     |                         |                  | Si                                        | 1.5   | 4.3     | 0.6     | 5.3     | 2.2   |                  |                  |                          |                             |     |   |                                    |             |                      |             |
|     |                         |                  | Fe                                        | 90.7  | 66.7    | 46.8    | 63.1    | 49.8  |                  |                  |                          |                             |     |   |                                    |             |                      |             |
|     |                         |                  | Hv                                        | 280   | 340     | 720     | 690     | 800   |                  |                  |                          |                             |     |   |                                    |             |                      |             |
| 5   | 900                     | 4                | Al                                        | 3.2   | 54.5    | 33.4    | —       | 54.5  | 440              | 300              | 410                      | 56                          | 335 | ○ | ○                                  | ○           |                      |             |
|     |                         |                  | Si                                        | 0.4   | 2.5     | 15.3    | —       | 6.8   |                  |                  |                          |                             |     |   |                                    |             |                      |             |
|     |                         |                  | Fe                                        | 94.3  | 42.7    | 51.4    | —       | 38.4  |                  |                  |                          |                             |     |   |                                    |             |                      |             |
|     |                         |                  | Hv                                        | 300   | 740     | 700     | —       | 810   |                  |                  |                          |                             |     |   |                                    |             |                      |             |

# FIG.5

**TABLE 2**

| HEATING<br>TEMPERATURE<br>(°C) | HOLDING<br>TIME (min.) | OXIDE<br>WEIGHT<br>(mg/dm <sup>2</sup> ) | ADHESION |                   |
|--------------------------------|------------------------|------------------------------------------|----------|-------------------|
|                                |                        |                                          | PIN HOLE | CROSS CUT<br>TEST |
| 950                            | 3                      | 290                                      | NONE     | ○                 |
|                                | 5                      | 385                                      | NONE     | ○                 |
|                                | 10                     | 540                                      | PRODUCED | ×                 |
|                                | 15                     | 690                                      | PRODUCED | ×                 |
|                                | 20                     | 760                                      | PRODUCED | ×                 |
|                                | 25                     | 790                                      | PRODUCED | ×                 |
|                                | 30                     | 805                                      | PRODUCED | ×                 |



European Patent  
Office

# EUROPEAN SEARCH REPORT

Application Number  
EP 03 01 3731

| DOCUMENTS CONSIDERED TO BE RELEVANT                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |                                                                                                                                                                                    |                                                             |                                              |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------|----------------------------------------------|
| Category                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             | Citation of document with indication, where appropriate, of relevant passages                                                                                                      | Relevant to claim                                           | CLASSIFICATION OF THE APPLICATION (Int.Cl.7) |
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|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |                                                                                                                                                                                    |                                                             | C23C<br>B32B                                 |
| The present search report has been drawn up for all claims                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |                                                                                                                                                                                    |                                                             |                                              |
| Place of search<br><b>MUNICH</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |                                                                                                                                                                                    | Date of completion of the search<br><b>11 November 2003</b> | Examiner<br><b>Brisson, O</b>                |
| <p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone<br/>Y : particularly relevant if combined with another document of the same category<br/>A : technological background<br/>O : non-written disclosure<br/>P : intermediate document</p> <p>T : theory or principle underlying the invention<br/>E : earlier patent document, but published on, or after the filing date<br/>D : document cited in the application<br/>L : document cited for other reasons</p> <p>&amp; : member of the same patent family, corresponding document</p> |                                                                                                                                                                                    |                                                             |                                              |

EPO FORM 1503 03/02 (P04001)

**ANNEX TO THE EUROPEAN SEARCH REPORT  
ON EUROPEAN PATENT APPLICATION NO.**

EP 03 01 3731

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.  
The members are as contained in the European Patent Office EDP file on  
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11-11-2003

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(54) **COATED STEEL STRIPS, METHODS OF MAKING THE SAME, METHODS OF USING THE SAME, STAMPING BLANKS PREPARED FROM THE SAME, STAMPED PRODUCTS PREPARED FROM THE SAME, AND ARTICLES OF MANUFACTURE WHICH CONTAIN SUCH A STAMPED PRODUCT**

(57) Coated steel having a well defined and uniform thickness is useful and advantageous for preparing products by hot stamping. Products prepared by hot stamping

such a steel are particularly advantageous when subjected to a subsequent spot welding step.

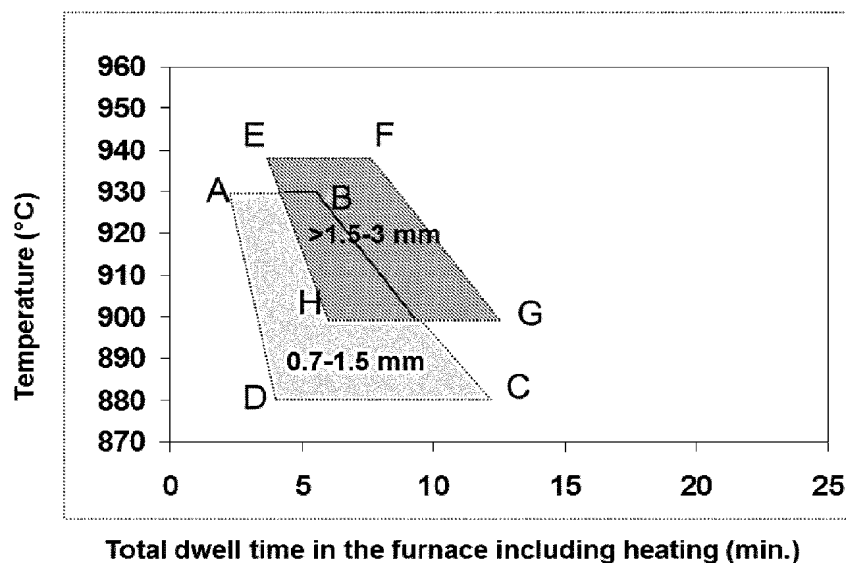


**FIG. 7**

## Description

### FIELD OF THE INVENTION

**[0001]** The present invention relates to, among other things, coated steels, methods of making such coated steels, including hot dipping, methods of using such coated steels, stamping blanks prepared from coated steels, stamped products prepared from coated steels, and to various uses of the invention products such as in spot welding, etc.

**[0002]** Additional advantages and other features of the present invention will be set forth in part in the description that follows and in part will become apparent to those having ordinary skill in the art upon examination of the following or may be learned from the practice of the present invention. The advantages of the present invention may be realized and obtained as particularly pointed out in the appended claims. As will be realized, the present invention is capable of other and different embodiments, and its several details are capable of modifications in various obvious respects, all without departing from the present invention. The description is to be regarded as illustrative in nature, and not as restrictive.

### BACKGROUND OF THE INVENTION

**[0003]** In recent years the use of pre-coated steels in hot-stamping processes for the shaping of parts has become important, especially in the automotive industry. Fabrication of such parts may include the following main steps:

- Pre-coating of a steel sheets, by hot dipping
- Trimming or cutting for obtaining blanks
- Heating the blanks in order to obtain alloying of the steel substrate with the pre-coating, as well as the austenitizing of the steel
- Hot forming followed by rapid cooling of the part in order to obtain predominantly martensitic structures

See for Example U.S. 6,296,805, incorporated herein by reference.

**[0004]** Thanks to an alloying of the pre-coating with the steel substrate, which has the effect of creating intermetallic alloys with high melting temperature, the blanks having such coating may be heated in a temperature range where austenitizing of the metallic substrate takes place, allowing further hardening by quenching.

**[0005]** Heat treatments of the blanks in view of the intermetallic alloying of the coating and austenitizing of the substrate are most frequently performed in furnaces, where blanks are traveling on rollers. The thermal cycles experienced by the blanks include first a heating phase whose rate is a function of parameters such as blank thickness, furnace temperature, traveling speed, and coating reflectivity. After this heating phase, thermal cycles generally include a holding phase, whose tempera-

ture is the regulation temperature of the furnace. Problems however are experienced with the furnace operation: the rollers may become fouled by metallic deposits which come from the pre-coating of the blanks. If these deposits are excessive, maintenance of the rollers has to be performed and productivity is decreased.

**[0006]** Parts obtained after heating and rapid cooling display very high mechanical resistance and may be used for structural applications, for example for automotive industry applications. These parts must be frequently welded with others and high weldability is required. This means that:

- The welding operation should be performable in a sufficiently wide operating range in order to guarantee that an eventual drift of the nominal welding parameters has no incidence on weld quality. For resistance welding, which is very common in the automotive industry, an operating welding range is defined by the combination of parameters: welding current intensity I and force F applied of the parts during welding being among the most important. A proper combination of these parameters helps to ensure that insufficient nugget diameter is not obtained (caused by too low intensity or too low force) and that no weld expulsion occurs.
- The welding operation should also be performed in such a way that high mechanical resistance is obtained on the weld. This mechanical resistance may be evaluated by tests such as by shear-tensile tests or cross-tensile tests.

**[0007]** There remains a need for coated steels which may be conveniently used to prepare shaped parts by a stamping process. There also remains a need for coated steels which may be used to prepare shaped parts by a stamping process which are suitable for welding. There also remains a need for processes for preparing such coated steels and stamped parts.

### SUMMARY OF THE INVENTION

**[0008]** The inventors have discovered that certain coated steels in which a base steel strip is at least partially coated (sometimes termed "pre-coated," this prefix indicating that a transformation of the nature of the pre-coating will take place during heat treatment before stamping) on at least one side with a coating of either aluminum or an aluminum alloy and in which the coating has a defined thickness and is preferably substantially uniform, are conveniently formed into shaped parts after heating by stamping and are conveniently welded. In addition, the inventors have discovered that the roller fouling problem described above generally arises from an insufficient degree of intermetallic alloying between the substrate and the metallic pre-coating. Furthermore, it was discovered that the location of the fouling of the rollers corresponds to zones of the blanks in contact with the rollers where

the metallic pre-coating thickness locally exceeds the average thickness. While not bound by a particular theory, it is believed that if a pre-coating is locally too thick, intermetallic alloying is insufficient and the pre-coating melts, fouling the rollers. Thus, the inventors have discovered that the control of the homogeneity of the pre-coating thickness over the entire sheet within given tolerances is an important factor for obtaining the desired degree of intermetallic alloying, allowing one to improve the resistance to the subsequent melting of the coating during travel on rollers.

**[0009]** The inventors have also discovered that particularly good weldability of aluminized and hot stamped parts is associated with a special succession of coating layers on the parts, proceeding from steel substrate outwards.

**[0010]** The inventors have also discovered that a specific combination of transfer time between the heating furnace and the stamping die, the amount of deformation during stamping, the temperature of stamping, the cooling rate of the product during stamping, leads to the fabrication of a part with a fully homogeneous martensitic structure and that an increase in ductility or energy absorption of the parts after stamping is obtained by a reduction of sulfur below a critical value, these two benefits being obtained with or without the invention aluminum/aluminum alloy coating, and with other coatings.

#### OBJECTS OF THE INVENTION

**[0011]** Accordingly, and in view of the above, it is one object of the present invention to provide novel pre-coated steels strips which may be conveniently processed into stamping blanks.

**[0012]** It is another object of the present invention to provide novel pre-coated steels strips or sheets which may be conveniently formed into parts by stamping.

**[0013]** It is another object of the present invention to provide novel coated steels which may be conveniently formed into parts by hot stamping.

**[0014]** It is another object of the present invention to provide novel methods for making such a coated steel.

**[0015]** It is another object of the present invention to provide novel stamping blanks which are prepared from such a coated steel.

**[0016]** It is another object of the present invention to provide novel methods of making such stamping blanks.

**[0017]** It is another object of the present invention to provide novel stamped parts which are prepared from such a coated steel.

**[0018]** It is another object of the present invention to provide novel methods of making such stamped parts.

**[0019]** It is another object of the present invention to provide novel articles of manufacture, such as a motor vehicle, which contain such stamped parts.

**[0020]** It is another object of the present invention to provide novel stamped parts.

**[0021]** It is another object of the present invention to

provide novel methods of making welded stamped parts.

**[0022]** It is another object of the present invention to provide novel articles of manufacture, such as a motor vehicle, which contain such welded stamped parts.

**[0023]** It is another object of the present invention to provide novel welded coated steels and welded stamping blanks.

**[0024]** It is another object of the present invention to provide novel methods of making such welded coated steels and welded stamping blanks.

**[0025]** These and other objects, which will become apparent during the following detailed description.

#### BRIEF DESCRIPTION OF THE DRAWINGS

##### **[0026]**

Figure 1 illustrates a preferred embodiment of an invention coated part, after heat treatment and stamping. The succession of layers of the coating on the steel substrate is: (a) interdiffusion layer; (b) intermediate layer; (c) Intermetallic layer; and (d) superficial layer. This arrangement is particularly favorable for the further welding of the part.

Figure 2 illustrates a coating of a steel substrate after heat treatment and stamping which does not correspond to the invention. This succession of layers (interdiffusion layer and intermetallic layer) yields inferior results in resistance spot welding.

Figure 3 illustrates a microstructure of a steel part, hot stamped and cooled under conditions not according to the invention.

Figure 4 illustrates the microstructure of a steel part, hot stamped and cooled according to a preferred set of conditions according to the invention.

Figure 5 illustrates the influence of sulfur on the bending angle of parts after hot stamping.

Figure 6 illustrates the influence of sulfur on the initiation energy of fracture of parts after hot stamping.

Figure 7 shows conditions of furnace temperature as a function of the total dwell time in the furnace for sheets of total thicknesses of from 0.7-1.5 and 1.5-3 mm that provide particularly favorable coatings for welding.

#### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

**[0027]** As noted above, the inventors' discovery that certain coated steels, in which a base steel is at least partially pre-coated on at least one side with a coating of either aluminum or an aluminum alloy and in which the pre-coating has a defined thickness and is substantially uniform, are conveniently formed into shaped parts by stamping, forms one basis for the invention.

**[0028]** In the context of the present invention, the terms first side (or side 1) and second side (or side 2) of the strip or sheet, etc., of base steel refer to the two large,

opposite-facing, surfaces which have a surface area defined by the length and width of the strip of base steel. In contrast, the side edges of the strip of base steel are the two small, opposite-facing, surfaces which have a surface area defined by the length and thickness of the strip. The top and bottom edges of the strip of base steel are the two small, opposite-facing, surfaces which have a surface area defined by the width and thickness of the strip. In the following,  $t_p$  designates the thickness of the pre-coating, at any considered location on the sides 1 and 2 of a sheet or a blank. In particular, in the case of sheets coated on two sides 1 and 2,  $t_{p1}$  stands for the thickness on side 1, and  $t_{p2}$  for the thickness on side 2. According to a highly preferred embodiment  $t_p$  is controlled in a precise range, expressed by  $(t_{pmin}, t_{pmax})$ , in order to improve the resistance to the fouling of the rollers. The thickness is preferably controlled, both in the longitudinal (or rolling) direction of the strip or sheet, as well in the transverse direction.

**[0029]** In relation to the problem of the fouling of rollers, the control of the pre-coating thickness on the side of the sheet or blank which is directly in contact with the rollers is especially important. Because different operations can follow the step of coating the steel sheet (for example hot-dip-coating which provides coated sides 1 and 2), it is preferable to carefully control the pre-coating on both sides of the sheet. For example, after any of coiling, handling, cutting, punching, etc., sides 1 and 2 may be not readily identifiable. But when control of  $t_p$  is effected on the two sides of the sheet that has been coated (first side and second side) it is not necessary to track sides 1 and 2 as neither side will foul the roller. In addition, it is not necessary to trim the sheet in order to obtain a preferred smaller sheet having a more uniform homogeneity of pre-coating thickness, thus providing a sheet that is pre-coated by, e.g., hot-dipping. In other words, important benefits are obtained when one controls the pre-coating minimum and maximum thickness of the first side ( $t_{pmin1}$ ,  $t_{pmax1}$ ) and the pre-coating minimum and maximum thickness of the second side ( $t_{pmin2}$ ,  $t_{pmax2}$ ) of the subject steel sheet or blank.

Hot-dip coated steels are the preferred steels herein. However, regardless of the coating method, the pre-coating thickness on one or both sides of the sheet can be measured and monitored continuously on a coating line directly after the coating operation. This may be realized by devices known per se, such as thickness gages relying on X-Ray absorption. At every moment, the measurement of thickness at a given location may be performed for example on an area of a few hundreds of  $mm^2$ , this representing the dimension of the zone irradiated by X-Ray.

**[0030]** In a preferred embodiment, a plurality of such devices are positioned at different distances in the transverse direction of the strip in order to obtain a profile of the thickness of the pre-coating along the strip width.

**[0031]** The inventors have discovered that the resistance to the pollution or fouling of the rollers in the furnaces

is improved when the minimum and maximum thickness respectively of at least one of the first side ( $t_{pmin1}$ ,  $t_{pmax1}$ ) and the second side ( $t_{pmin2}$ ,  $t_{pmax2}$ ) are respectively equal to 20 and 33 micrometers (a micrometer is the same as a micron and is a metric unit of length equal to one millionth of a meter). In other words, in a preferred embodiment, at every location on at least one face of the sheet or the blank, the thickness  $t_p$  of the pre-coating is preferably from 20 - 33 micrometers in thickness, including 21, 22, 23, 24, 25, 26, 27, 28, 29, 30, 31 and 32 micrometers and all ranges and subranges therebetween, and including all numbers between each listed value as if written out in its entirety (e.g., 22.34 micrometers). For hot-dip coating, precise control of this range of pre-coating thickness can be realized on an operating line for example using a system of nozzles blowing gas after coating, for example after exit of the strip or sheet from a bath, and by the flatness of the strip. Number, geometry and location of the nozzles, and flow rates are among the main parameters for a precise control of the thickness  $t_p$ . Given the present disclosure, one of ordinary skill in this art can control the pre-coating thickness as described herein without undue labor.

The invention refers to strips produced in industrial conditions, i.e. where control of pre-coating thickness is effective over a wide surface strip, i.e. with a length greater than 100m, and with a width greater than 600mm. In such a way, blanks cut or trimmed out these strips display very high homogeneity of pre-coating thickness, and the settings of the heating treatments in the furnace do not have to be changed for being adapted to eventual variations of this thickness.

**[0032]** While not bound by a particular theory of operation, the inventors believe that several of the benefits of the invention are related to this pre-coating thickness range, such as the following :

- For a pre-coating thickness less than 20 micrometers, the alloyed layer which is formed during the heating of the blank has an insufficient roughness. Thus, the adhesion of subsequent painting is low on this surface, and the corrosion resistance is decreased.
- If the pre-coating thickness is more than 33 micrometers at a given location on a sheet, the risk is that the difference of thickness between this location and some other locations where the pre-coating is thinner, becomes too important : The settings of the heating treatment in the furnace may be adapted to the thinner value of the pre-coating, but not to the thicker one. Thus, the alloying reaction forming the intermetallic alloy may take place to an insufficient degree since the mean diffusion distance of the elements in the pre-coating becomes significantly less than the local value of the pre-coating thickness. As a matter of results, alloying will become much more difficult in the external (or superficial) part, particularly in the case of high heating rate.

**[0033]** Thus, in a first embodiment, the present invention provides certain coated steel strips, which comprise a strip of base steel and a pre-coating of aluminum or an aluminum alloy on at least a part of one side of the strip of the base steel. For many applications, the strip of base steel may comprise any type of steel which may be coated with either aluminum or an aluminum alloy. However, for certain applications, such as a structural part of an automobile, it is preferred that the strip of base steel comprise an ultra high strength steel (UHSS). In such cases, it is particularly preferred that the strip of base steel comprises a boron steel.

**[0034]** The inventors have also discovered that good welding results are achieved if the coating obtained on parts made out of blanks having undergone intermetallic alloying, austenitizing and hot stamping, displays distinctive features. It must be pointed out that this coating is different from the initial pre-coating, since the thermal treatment causes an alloying reaction with the steel substrate which modifies both the physico-chemical nature and the geometry of the pre-coating : in this regard, the inventors have discovered that particularly good weldability of aluminized and hot stamped parts is associated with the following succession of coating layers on the parts, proceeding from steel substrate outwards :

- (a) Interdiffusion layer,
- (b) Intermediate layer,
- (c) Intermetallic layer,
- (d) Superficial layer

See, e.g., Figure 1. In a preferred embodiment these layers are as follows:

- (a) Interdiffusion layer, preferably with medium hardness (e.g., HV50g between 290 and 410, HV50g designating the hardness measured under a load of 50 grams) In a preferred embodiment this layer has the following composition, by weight: 86-95%Fe, 4-10%Al, 0-5%Si
- (b) Intermediate layer (HV50g around 900 - 1000 e.g., +/- 10%) In a preferred embodiment this layer has the following composition, by weight : 39-47% Fe, 53-61%Al, 0-2%Si
- (c) Intermetallic layer, with hardness HV50g around 580-650, e.g., +/- 10%) In a preferred embodiment this layer has the following composition, by weight : 62-67%Fe, 30-34 %Al, 2-6%Si
- (d) Superficial layer (HV50g around 900 - 1000 e.g., +/- 10%)

**[0035]** In a preferred embodiment this layer has the following composition, by weight : 39-47% Fe, 53-61%Al, 0-2%

In a preferred embodiment the total thickness of layers (a) to (d) is greater than 30 micrometers.

In another preferred embodiment, the thickness of layer (a) is less than 15 micrometers, for example 14, 12, 10,

8, 6, 4, 2, or 1 micrometers, and all whole numbers, ranges and subranges therebetween, and including all numbers between each listed value as if written out in its entirety (e.g., 13.84 micrometers).

**[0036]** The inventors have discovered that high weldability is especially obtained when layers (c) and (d) are essentially continuous (i.e.: occupying at least 90% of the level corresponding to the considered layer) and when less than 10% of layer (c) is present at the extreme surface of the part. Without being bound by a theory, it is thought that this particular layer disposal, in particular layer (a) and layers (c) and (d) influence the resistivity of the coating both by their intrinsic characteristics and by the effect of roughness. Thus, current flow, heat generation at the surfaces, and nugget formation in the initial stage of spot welding are affected by this particular arrangement.

This favorable layer disposition is obtained for example when aluminum- or aluminum alloy pre-coated steel sheets, whose thickness range from, e.g., 0.7 to 3mm, are heated for 3 to 13 minutes (this dwell time includes the heating phase and the holding time) in a furnace heated to a temperature of 880 to 940°C. Other conditions leading to such favorable layer dispositions are found in Figure 7:

For sheets of total thicknesses greater or equal to 0.7mm, and less than or equal to 1.5mm, the preferred treatment conditions : (furnace temperature, total dwell time in the furnace) are illustrated in figure 7 by conditions lying within the limits of diagram "ABCD"

For sheets of total thicknesses greater than 1.5mm, and less than or equal to 3mm, the preferred treatment conditions : (furnace temperature, total dwell time in the furnace) are illustrated in figure 7 by diagram "EFGH" .

The heating rate  $V_c$  is comprised between 4 and 12°C/s for producing a favorable alloyed layer disposition. In this regard, this "heating rate" reflects the temperature rise which is experienced by the pre-coated steel upon being located in the preheated furnace.  $V_c$  is defined as the mean heating rate between 20 and 700°C. The inventors have discovered that the control of  $V_c$  in this precise range is a key factor, because it controls directly the nature and the morphology of the alloyed layers which are formed. It is here underlined that the heating rate  $V_c$  is different from the mean heating rate, which is the heating rate between room temperature and furnace temperature. The rates of 6, 7, 8, 9, 10, and 11 °C/s are included as are all numbers, ranges and subranges therebetween, and including all numbers between each listed value as if written out in its entirety (e.g., 7.7 °C/s). In this regard, all of the conditions specified within Figure 7 are incorporated herein by reference thereto. Particularly preferred conditions are:

(for thicknesses of 0.7-1.5mm)

- 930°C, from 3 minutes up to 6 minutes;
- 880°C, from 4 minutes 30 seconds up to 13 minutes

(for thicknesses of 1.5 to 3 mm)

- 940°C, from 4 minutes up to 8 minutes;
- 900°C, from 6 minutes 30 seconds up to 13 minutes

A special advantage arises from pre-coatings whose thickness is comprised between 20 and 33 micrometers, since this thickness range yields favorable layer disposal, and since the homogeneity of the pre-coating thickness is associated to an homogeneity of the coating formed after alliation treatment.

**[0037]** Heated blanks are thereafter transferred to a die, hot stamped to obtain a part or product, and cooled at a rate of more than 30°C/s. The cooling rate is defined here as the mean rate between the exit of the heated blank from the furnace, down to 400°C.

**[0038]** The strip of base steel is coated with either aluminum or an aluminum alloy. Commercially pure aluminum is known in the art as Type 2 aluminum, while alloys of aluminum with 5 to 11 % by weight of silicon are known in the art as Type 1 aluminum. Silicon is present in order to prevent the formation of a thick iron-metallic intermetallic layer which reduces adherence and formability. Other alloying elements useful with aluminum herein include iron, between 2.5 and 3% by weight, and calcium, between 15 and 30ppm by weight, including combinations of two or more thereof with aluminum.

**[0039]** A typical metal bath for an Al-Si coating generally contains in its basic composition by weight, from 8% to 11% silicon, from 2% to 4% iron, the remainder being aluminum or aluminum alloy, and impurities inherent in processing. Typical composition of Al-Si coating is: Al-9,3%Si-2,8%Fe. Invention coatings are not limited to these compositions, however.

**[0040]** The strip of base steel used herein may be any which can be coated by a conventional coating technique. For example, the strip of base steel may be any hot rolled strip, such as those prepared by hot rolling a steel slab (either with or without subsequent cold rolling). Typically, the strip of base steel will be stored and transported in the form of a coil both before and after the formation of the coating.

**[0041]** An example of a preferred steel for the strip of base steel is one having the following composition by weight:

0.10% < carbon < 0.5%  
 0.5% < manganese < 3%  
 0.1% < silicon < 1%  
 0.01% < chromium < 1%  
 titanium < 0.2%  
 aluminum < 0.1%  
 phosphorus < 0.1%  
 sulfur < 0.05%  
 0.0005% < boron < 0.010%,

the remainder comprising, consisting essentially of, or consisting of iron and impurities inherent in processing.

Use of such a steel provides a very high mechanical resistance after thermal treatment and the aluminum-based coating provides a high resistance to corrosion.

**[0042]** Particularly preferably, the composition by weight of the steel in the strip of base steel is the following:

0.15% < carbon < 0.25%  
 0.8% < manganese < 1.8%  
 0.1% < silicon < 0.35%  
 0.01% < chromium < 0.5%  
 titanium < 0.1%  
 aluminum < 0.1%  
 phosphorus < 0.1%  
 sulfur < 0.05%  
 0.002% < boron < 0.005%,

the remainder comprising, consisting essentially of, or consisting of iron and impurities inherent in processing.

**[0043]** A preferred strip herein is 100 m long and 600mm wide. Preferred thicknesses are 0.7 to 3 mm.

**[0044]** Even more preferably, in the composition by weight of the sheet, the weight ratio of titanium content with respect to the nitrogen content is in excess of 3.42, believed to be a level at which the boron is no longer able to combine with the nitrogen.

**[0045]** An example of preferred commercially available steel for use in the strip of base steel is 22MnB5.

**[0046]** Chromium, manganese, boron and carbon may be added, in the composition of the steel according to the invention, for their effect on hardenability. In addition, carbon makes it possible to achieve high mechanical characteristics thanks to its effect on the hardness of the martensite.

**[0047]** Aluminum is introduced into the composition, to perform deoxidation in the liquid state and to protect the effectiveness of the boron.

**[0048]** Titanium, the ratio of the content of which with respect to the nitrogen content should be in excess of 3.42, is introduced for example in order to prevent combining of the boron with the nitrogen, the nitrogen being combined with titanium.

**[0049]** The alloying elements, Mn, Cr, B, make possible a hardenability allowing hardening in the stamping tools or the use of mild hardening fluids limiting deformation of the parts at the time of thermal treatment. In addition, the composition according to the invention is optimized from the point of view of weldability.

**[0050]** The steel in the sheet may undergo a treatment for globularization of sulfides performed with calcium, which has the effect of improving the fatigue resistance of the sheet.

**[0051]** As mentioned above, ultra-high-strength can be provided with the steel sheet coated and hot stamped according to the invention. This high level of strength is sometimes associated with a limited ductility. In applications requiring a higher ductility, in particular when an ability for bending is required from a part or a product, the inventors have discovered that increased ductility can

be obtained if sulfur is particularly controlled: when the sulfur level of base steel is lower than or equal to 0.002% (20 ppm), the bending angle can be greater than 60° and enhanced ductility and tearing resistance are obtained on the parts having experienced heat treatment and stamping. Preferred levels include 20, 18, 15, 13, 10, 8, 5, 2, etc ppm sulfur. In fact, this benefit applies to steels in general, and is not limited to coated steels or to steels coated with Al or Al alloy coatings. While not bound by a particular theory, when analyzing causes of premature failure of some parts in bending operations, the inventors observed that failure initiated on sulfide inclusions. It is thus believed that decohesions between inclusions and the martensitic or bainite-martensitic matrix act as stress-concentration factors and trigger further crack propagation in the ductile mode.

**[0052]** The invention also concerns a process for producing a part starting from an invention coated sheet, then cut into a blank which, after shaping, the coating of the blank is subjected to an increase in temperature at a speed in excess of 4°C/second, but lower than 12°C/second. The heating rate  $V_c$  is defined as the mean rate between 20 and 700°C.

**[0053]** The invention also concerns the use of a hot-rolled steel sheet which then can be cold-rolled and coated, for structural and/or anti-intrusion or substructure parts for a land motor vehicle, such as, for example, a bumper bar, a door reinforcement, a wheel spoke, etc.

**[0054]** The sheet according to the invention described above can derive, by reason of its processing, from a hot-rolling mill, and possibly may be cold-rerolled again depending on the final thickness desired. It then is coated with an aluminum-based coating, for example by dipping in a bath containing, in addition to the aluminum source/alloy, e.g., from 8% to 11% silicon and from 2% to 4% iron, the sheet having a high mechanical resistance after thermal treatment and a high resistance to corrosion, as well as a good capacity for painting and gluing.

**[0055]** The coating is preferably controlled as above, and has in particular the function of protecting the basic sheet against corrosion in various conditions. The thermal treatment applied at the time of a hot-forming process or after forming makes it possible to obtain high mechanical characteristics which can exceed 1500 MPa for mechanical resistance and 1200 MPa for yield stress. The final mechanical characteristics are adjustable and depend in particular on the martensite fraction of the structure, on the carbon content of the steel and on the thermal treatment. At the time of thermal treatment performed on a finished part or at the time of a hot-shaping process, the coating forms a layer having a substantial resistance to abrasion, wear, fatigue, shock, as well as a good resistance to corrosion and a good capacity for painting and gluing. The coating makes it possible to avoid different surface-preparation operations such as for steel sheets for thermal treatment not having any coating.

**[0056]** The steel sheet can be pre-coated by dipping, after pickling, in an aluminum bath containing for example

only aluminum or either aluminum and from 8% to 11% silicon and 2% to 4% iron, or only from 2% to 4% iron, or even in an aluminum bath preferably containing from 9% to 10% silicon and 2% to 3.5% iron. The aluminum may be aluminum per se or an aluminum alloy.

**[0057]** In an example of implementation of a coating of the sheet by dipping in a metal bath containing an aluminum alloy comprising a proportion of approximately 90% aluminum, the coating layer comprises a first alloy layer in contact with the surface of the steel. This layer, directly in contact with the surface of the sheet, is highly alloyed with iron.

**[0058]** A second coating layer, on top of the first, contains approximately 90% aluminum and may contain silicon and a small amount of iron, depending on the composition of the bath.

**[0059]** The first alloy layer may crack when the sheet is submitted to high strains during cold forming operations of the manufacture of parts.

**[0060]** According to the invention, after the forming of the part, the coating is subjected to an increase in temperature at a speed in excess of 4°C/second. This rise in temperature makes possible a rapid remelting of the aluminum which fills in the cracks generated by the operation of shaping of the part.

**[0061]** At the time of thermal treatment, the base coating, of aluminum for example, is transformed into a layer alloyed with iron and comprising different phases depending on the thermal treatment and having a considerable hardness which may exceed 600 HV50 g.

**[0062]** Another advantage of the invention lies in the fact that the diffusion of the iron in the coating is initiated at high temperature. One thus will have a better cohesion between coating and steel in the sheet. In another form of the invention, the thermal treatment may be performed locally, in heavily deformed zones.

According to the invention, the sheet, in the delivery state in a coil or in a sheet, the thickness of which may range between 0.25 mm and 15 mm, has good forming properties and a good resistance to corrosion as well as a good capacity for painting or gluing. Preferably, the steel sheet or blank has a thickness less than 3 mm, since the cooling rates that may be achieved after quenching are high and help to obtain martensitic structures.

**[0063]** The steel sheet, a coated product, has a substantial resistance to corrosion in the delivery state, during forming and thermal treatments as well as during usage of the finished part.

The presence of the coating at the time of thermal treatment of the parts makes it possible to prevent any decarburization of the base metal as well as any oxidation. That is an undeniable advantage, in particular in the case of hot forming. Furthermore, heating of the treated part does not require a furnace having a controlled atmosphere to prevent a decarburization.

**[0064]** Thermal treatment of the metal in the sheet comprising heating at a temperature ranging between Ac1, starting temperature of austenitic transformation

when heating, for example 750°C and 1200°C, in a furnace, for a period which depends on the temperature to be reached and the thickness of the blank. The composition is optimized so as to limit the grain growth at the time of thermal treatment. If the structure sought is completely martensitic, the holding temperature should be in excess of Ac3, for example 840°C, temperature of complete austenitic transformation. The temperature holding should be followed by a cooling adjusted to the final structure sought.

**[0065]** Blanks are thereafter transferred from the furnace to a stamping press. When the elapsed time between the exit of the blanks from the furnace and the introduction in the stamping press is more 10 seconds, a partial transformation from austenite is susceptible to appear: if obtaining a full martensitic structure is desired, the transfer time between the exit of the furnace and stamping should be less than 10s.

**[0066]** The inventors have also discovered that the obtaining of a fully martensitic structure is linked to the amount of deformation in the hot forming operation: the amount of local deformation caused by hot forming is closely linked to the shape of the part or product and may exceed locally 40 or 50% in some particular regions. The inventors found that, when the local strain exceeds a critical value of 10%, the cooling rate must be sufficiently high in order to get a total martensitic transformation. Otherwise, bainitic transformation can take place at a significant amount instead of martensitic transformation. Thus, the risk is that heterogeneous structure appears on parts with complex shape where some locations are much more deformed than others. In this respect, the inventors put into evidence that, on the locations of the parts where the forming strain is higher than 10%, the cooling rate must be increased beyond 50°C/s in order to guarantee full martensitic transformation. The cooling rate is defined as the mean rate between the exit of the heated blank from the furnace, down to 400°C.

**[0067]** But one may also seek to obtain ferrito-bainitic or ferrito-martensitic structures, by a heating at a temperature ranging between Ac1, for example 750°C and Ac3, for example 840°C, followed by an appropriate cooling. According to the level of resistance to be achieved and the thermal treatment applied, one or several of these constituents is/are present in variable proportions.

**[0068]** The modulation of thermal treatment parameters makes it possible to achieve, with a given composition, different levels of hot and cold sheet resistance according to the thickness sought. For the highest resistance levels, the structure is composed predominantly of martensite.

**[0069]** The steel is particularly suited to the production of structural and anti-intrusion parts.

**[0070]** The invention thus enables one to produce a hot- or cold-rolled steel sheet of a desired thickness, coated, and affording extensive forming possibilities and which, after thermal treatment performed on the finished part, makes it possible to obtain a mechanical resistance

in excess of 1000 MPa, a substantial resistance to shocks, fatigue, abrasion and wear, while retaining a good resistance to corrosion as well as a good capacity for welding, painting and gluing.

**[0071]** The present invention will now be further described by way a certain exemplary embodiments which are not intended to be limiting.

## EXAMPLES

### Example 1 :

**[0072]** In a first example of implementation, a cold rolled steel sheet, 1.9 mm thick, containing by weight: 0.23 % carbon, 1.25% manganese, 0.017% phosphorus, 0.002% sulfur, 0.27% silicon, 0.062% aluminum, 0.021% copper, 0.019% nickel, 0.208% chromium, 0.005% nitrogen, 0.038% titanium, 0.004% boron, 0.003% calcium- has been pre-coated with an aluminum-based alloy with composition 9.3% silicon, 2.8% iron, the remainder being aluminum and unavoidable impurities. According to the conditions of fabrication, namely the settings of the blowing devices on the operating line, sheets of 120m long and 650mm wide with various thickness ranges were produced.

- Sheet A (according to the invention): The thickness  $t_{p1}$  and  $t_{p2}$  on each side of the sheet was controlled to be within the range (20-33) micrometers, at every location of the two faces of the sheet, both in the longitudinal (or rolling) direction and in the transversal direction. Measurement was performed continuously with thickness gages devices relying on X-Ray emission. At every moment, the spot of measurement of each gage was a circular zone of about 20mm radius. The sheets were afterwards cut into blanks of 1.2 x 0.5m<sup>2</sup> of overall dimensions.
- Sheet B (reference): On these sheets, the pre-coating thickness had a wider variability since the thickness  $t_{p1}$  and  $t_{p2}$  on the two sides of the sheet was comprised in the range (30-45) micrometers. Blanks cut out of these sheets exhibit the same pre-coating thickness.

**[0073]** The blanks were then submitted to heating in a furnace at T=920°C. Heating time was 3 min, with 4 mn holding time. The microstructure is then fully austenitic. Blanks were thereafter transferred from the furnace to a stamping press. When the elapsed time between the exit of the blanks from the furnace and the transfer in the stamping press is more 10 seconds, a partial transformation from austenite was susceptible to appear, thus reducing the mechanical resistance of the stamped part. The blanks were directly cooled afterwards without hot stamping in order to appreciate the eventual remelting of the coating

On series A, no melting of the pre-coating was found. Intermetallic alloying between the pre-coating and the

steel substrate occurred completely.

On series B, the pre-coating underwent mainly alloying, but some traces of remelting were found, particularly on the former thicker locations of the pre-coating. This partial remelting of the aluminium pre-coating contributes to the progressive fouling of the rollers in the furnace. The sheets according to the invention do not contribute to this progressive buildup on the rollers.

#### Example 2 :

##### [0074]

i) Conditions according to the invention: In a second example of implementation, a cold rolled steel sheet, 1,2mm thick, 120m long and 650mm wide, with same composition and same pre-coating as in example 1, has been fabricated. The sheets were afterwards cut into blanks which were heated at 920°C for 6mn, this time including the heating phase and the holding time. Heating rate  $V_c$  between 20 and 700°C was 10°C/s. The blanks were finally hot stamped and quenched in order to obtain full martensitic structures.

The parts obtained after hot-stamping are covered by a coating, 40 micrometers thick, illustrated at figure 1, which has a four layer structure. Starting from the steel substrate, the layers are the following:

- (a) Interdiffusion layer or intermetallic layer, 17 micrometers thick. This layer is itself composed of two sub-layers. Hardness HV50g ranges from 295 to 407, and the mean composition is: 90%Fe, 7%Al, 3%Si.
- (b) Intermediate layer, appearing darker, 8 micrometers thick. This layer has a hardness of 940HV50g and a mean composition, by weight : 43%Fe, 57%Al, 1%Si.
- (c) Intermetallic layer appearing as a pale phase, 8 micrometers thick, displaying a hardness of 610HV50g, a mean composition of 65%Fe, 31%Al, 4%Si
- (d) Darker superficial layer, 7 micrometers thick, 950 HV50g, with a mean composition of 45%Fe, 54%Al, 1%Si

Layers (c) and (d) are quasi-continuous, i.e. occupying at least 90% of the level corresponding to the considered layer. In particular, layer (c) does not reach the extreme surface except very exceptionally. Anyway, this layer (c) occupies less than 10% of the extreme surface.

ii) Conditions of reference: On the other hand, blanks with the same base material and pre-coating parts were furnace-heated in different conditions: The blanks were heated to 950°C for 7 minutes, this time including the heating phase. Heating rate  $V_c$  was 11°C/s. These conditions correspond to a degree of

alloying which is more important than in conditions (i)

- In this coating, the pale intermetallic layer (c), is not continuous and appears as to be scattered within the coating. About 50% of this layer is present at the extreme surface of the part. Moreover, the interdiffusion layer, 10 micrometers thick in contact with the steel substrate is thinner than in the previous case of figure 1.

Resistance spot welding was performed in the two situations i) and ii):

- (i): Coating with quasi-continuous layers (c) and (d), layer (c) occupying less than 10% of the extreme surface
- (ii) : Coating with mixed and discontinuous layers, layer (c) occupying more than 10% of the extreme surface

**[0075]** Resistance spot welding was performed by superposing two parts and joining them in the following conditions:

- Squeeze force and welding force : 4000 N
- Squeeze time: 50 periods
- Welding and holding time: 18 periods respectively

**[0076]** In each condition, the suitable intensity range was determined for obtaining:

- No sputter during welding
- Acceptable nugget size.

**[0077]** For the condition i), the weldability range, expressed in terms of current intensity, is 1,4kA. For the condition ii) the weldability range is extremely small.

**[0078]** Thus, it may be seen that the coating according to the invention, yields much more satisfactory results.

#### Example 3 :

**[0079]** In a third example of implementation, a cold rolled steel sheet of the example 1 was cut into blanks of 500x500mm<sup>2</sup> which were heated at 920°C, during 6mn, then hot stamped and cooled in tools, in such conditions that two different cooling rates were obtained :

- (A): Cooling rate:  $V_A = 30^\circ\text{C/s}$
- (B) : Cooling rate:  $V_B = 60^\circ\text{C/s}$

**[0080]** Due to the shape of the parts, different deformation levels  $\epsilon$  were created during hot stamping. In particular, some zones largely strained display deformation levels higher than 30%.

- As illustrated on figure 3, metallographic observations reveal that when  $\epsilon > 10\%$ , partial bainitic or fer-

ritic transformation occurs on parts cooled with  $V_A = 30^\circ\text{C/s}$ , mainly on former austenitic grain boundaries. On the other hand, the parts cooled with  $V_B = 60^\circ\text{C/s}$  display fully martensitic microstructure as illustrated on figure 4. The latter structures display superior mechanic resistance and a great homogeneity in the case of mechanical solicitation.

**[0081]** Thus, even in products or parts where straining is larger than 10%, the application of cooling according to the invention guarantees a microstructural and mechanical homogeneity.

#### Example 4:

**[0082]** In a fourth example of implementation, steel castings containing different values of sulfur, were elaborated. These steels were further hot rolled, then cold rolled steel into sheets, 2,2mm thick. Sulfur content varies from 11ppm (0,0011%) to 59ppm (0,006%) in weight. Apart sulfur, the compositions of these different steel castings comprise in weight:

0,24% carbon, 1,17% manganese, 0,01% phosphorus, 0,25% silicon, 0,045% aluminum, 0,01% copper, 0,02% nickel, 0,2% chromium, 0,04% titanium, 0,003% boron, 0,002% calcium, the remainder being iron and unavoidable impurities.

**[0083]** These sheets were pre-coated with an aluminum-based alloy of composition comprising 9,3% silicon, 2.8% iron, the remainder being aluminum and unavoidable impurities.

**[0084]** The sheets were afterwards cut into blanks which were heated at  $950^\circ\text{C}$  for 5mn, then hot stamped and cooled in tools in order to obtain a full martensitic structure. Mechanical resistance exceeded 1450 MPa.

**[0085]** Specimens were extracted according to the transverse sense of rolling direction and submitted to a bending test with alternate bending modes.

**[0086]** The inventors put into evidence that the critical bending angle (angle at fracture) is closely related to the sulfur content of the steel: when sulfur content is lower than 0,002%, the bending angle exceeds  $60^\circ$ , which indicates higher ductility and energy absorption.

**[0087]** Compact Tensile-Test type specimens were also extracted according to the transverse rolling direction in order to measure the resistance to tearing, i.e. the energy which is necessary for the initiation or propagation of an existing crack. The results, illustrated on figure 6, indicate that a initiation energy higher than 18 Joules is achieved when sulfur content is lower than 0,002% in weight.

**[0088]** As these qualities of high resistance, high energy absorption and weldability are required in automobile industry, the parts or products fabricated according to the invention will be used with profit for such applications.

**[0089]** While the above description is clear with regard to the understanding of the invention, the following terms as used in the following list of preferred embodiments and claims have the following noted meanings in order to avoid any confusion:

**pre-coating** - the material (Al or Al alloy) coated on or located on at least a portion of the strip or sheet, etc., of base steel to form a pre-coating/base composite, the composite not having been subjected to an alliation reaction between the coated Al or Al alloy material and base steel

**alliation** - a reaction between the pre-coating and base steel, to produce at least one intermediate layer different in composition from both the base steel and the pre-coating. The alliation reaction happens during the heat treatment immediately preceding hot stamping. The alliation reaction affects the total thickness of the pre-coating. In a highly preferred embodiment the alliation reaction forms the following layers: (a) interdiffusion, (b) intermediate, (c) intermetallic, and (d) superficial as described above;

**pre-coated steel** - the pre-coating/base composite, not having been subjected to an alliation reaction between the coated material and base steel;

**coating** - the pre-coating after having been subjected to an alliation reaction between the pre-coating and base steel. In a highly preferred embodiment the coating comprises layers (a) interdiffusion, (b) intermediate, (c) intermetallic, and (d) superficial described above;

**coated steel or product** - the pre-coated steel or product that has been subjected to an alliation reaction between the pre-coating and base steel. In a highly preferred embodiment the coated steel is a strip or sheet, etc., of base steel having thereon an invention coating comprising layers (a) interdiffusion, (b) intermediate, (c) intermetallic, and (d) superficial described above;

**blank** - a shape cut from a strip.

**product** - a stamped blank

Thus, the present invention provides, among other things, the following preferred embodiments:

#### 1. A pre-coated steel strip, comprising:

- (a) a strip of base steel having a length, a width, a first side, and a second side;
- (b) said length of said strip of base steel being at least 100m and said width being at least 600mm, and
- (c) an aluminum or an aluminum alloy pre-coating on at least part of one of said first and second sides of said strip of base steel, wherein (i) the thickness  $t_p$  of the said pre-coating is from 20 - 33 micrometers at every location on at least one of said first and second sides.

2. A method for making a pre-coated steel strip according to embodiment 1 wherein the pre-coated steel strip has a thickness of not greater than 3 mm.

3. A process for making a hot stamped coated steel sheet product, comprising:

(A) heating an aluminum- or aluminum alloy pre-coated steel sheet in a furnace preheated to a temperature and during a time defined by diagram ABCD of figure 7 if thickness of said sheet is greater than or equal to 0.7mm and less than or equal to 1.5mm, and by diagram EFGH of figure 7 if thickness of said sheet is greater than 1.5mm and less than or equal to 3mm, at a heating rate  $V_c$  between 20 and 700°C comprised between 4 and 12°C/s, to obtain a heated blank; (B) transferring said heated blank to a die; and (C) stamping said heated blank in said die, to thereby obtain a hot stamped steel sheet product, and

wherein the said heated product is cooled at a rate of at least 30°C/s.

4. A process for making a hot stamped coated steel sheet product, comprising:

(A) providing a heated blank according to embodiment 3; and (B) transferring said heated blank to a die; and (C) stamping said heated blank in said die, to thereby obtain a hot stamped steel sheet product,

wherein the elapsed time between said heated blank exits said furnace and said stamping commences is not more than 10 seconds, and wherein said heated blank is deformed by a quantity higher than 10% during said stamping and the said heated product is cooled at a rate of at least 50°C/s.

5. A process for making a hot stamped coated steel sheet product, comprising :

hot dip pre-coating a steel strip having a first side and a second side with aluminum or aluminum alloy to obtain a pre-coated steel strip, the pre-coating thickness on at least one of said first side and said second side of said strip having a thickness of from 20 to 33  $\mu\text{m}$  at every location on at least one of said first and second sides of said strip; cutting said pre-coated steel strip to obtain a sheet; heating the sheet in a pre-heated furnace to obtain a heated blank in which the steel is austenitic;

transferring said heated blank to a die while maintaining said steel in the austenitic phase; and stamping said heated blank in a die to obtain a hot stamped steel sheet product in which the steel microstructure is martensitic.

6. A hot stamped coated steel sheet product, which is prepared by a process comprising :

(A) providing a pre-coated strip according to embodiment 1, said strip being pre-coated with aluminum or aluminum alloy (B) Cutting said pre-coated steel strip to a predetermined size and shape to obtain a sheet (C) heating said steel sheet in a furnace preheated to a temperature and during a time defined by diagram ABCD of figure 7 if thickness of said product is greater than or equal to 0.7mm and less than or equal to 1.5mm, and by diagram EFGH of figure 7 if thickness of said product is greater than 1.5mm and less than or equal to 3mm, to obtain a heated blank; (D) transferring said heated blank to a die; and (E) stamping said heated blank in said die, to thereby obtain a hot stamped steel sheet product,

wherein the elapsed time between said heated blank exits said furnace and said stamping commences is not more than 10 seconds, and wherein said heated product cools at a rate higher than 30°C/s during said stamping.

7. A process for making a hot stamped coated steel sheet product, comprising:

(A) providing a heated blank according to embodiment 6 (B) transferring said heated blank to a die; and (C) stamping said heated blank in said die, to thereby obtain a hot stamped steel sheet product,

wherein the elapsed time between said heated blank exits said furnace and said stamping commences is not more than 10 seconds, and wherein said heated product cools at such a rate during said stamping that the steel microstructure is martensitic.

8. The pre-coated steel strip according to embodiment 1, wherein the base steel comprises the following components by weight based on total weight:

0.15% < carbon < 0.5%  
0.5% < manganese < 3%

0.1%<silicon<0.5%

0.01%<chromium<1%

titanium<0.2%  
aluminum<0.1%  
phosphorus<0.1%  
sulfur<0.05%

0.0005%<boron<0.08%,

and further comprises iron and impurities inherent in processing.

9. The pre-coated steel strip according to embodiment 8, wherein the base steel comprises the following components by weight based on total weight:

0.20%<carbon<0.5%  
0.8%<manganese<1.5%  
0.1%<silicon<0.35%  
0.01%<chromium<1%

titanium<0.1%  
aluminum<0.1%

phosphorus<0.05%

sulfur<0.03%

0.0005%<boron<0.01 %,

and further comprises iron and impurities inherent in processing.

10. The pre-coated steel strip according to embodiment 8 or 9, wherein the base steel comprises 20 ppm or less of sulfur.

11. The pre-coated steel strip according to any of embodiments 8 to 10, wherein the ratio of titanium to nitrogen in the steel sheet in weight % is in excess of 3.42.

12. The pre-coated steel strip according to any of the embodiments 1 or 8 to 11, wherein the aluminum or aluminum alloy pre-coating comprises from 8% to 11% silicon by weight, from 2% to 4% iron by weight, the remainder being aluminum and impurities inherent in processing.

13. A coated steel stamped product, which comprises:

(a) a strip of base steel having a first side and a second side; and

(b) a coating on at least one of said first side of said strip of base steel and said second side of

said strip of base steel,

wherein:

(i) said coating results from the interdiffusion between said base steel, and aluminium or aluminium alloy pre-coating,

(ii) said coating comprises, proceeding from base steel outwards,

- (a) Interdiffusion layer
- (b) Intermediate layer
- (c) Intermetallic layer
- (d) Superficial layer

14. A coated steel stamped product according to embodiment 13, wherein said coating has a thickness greater than 30 micrometers

15. A coated steel stamped product according to embodiments 13 or 14, wherein said layer (a) has a thickness less than 15 micrometers

16. A coated steel stamped product according to any of the embodiments 13 to 15, wherein the said layers (c) and (d) are occupying at least 90% of the level corresponding to the considered said layers and wherein less than 10% of layer (c) is present at the extreme surface of the product

17. A coated steel stamped product according to any of the embodiments 13 to 16, wherein the steel in the strip has a composition according to embodiment 8

18. A coated steel stamped product according to any of the embodiments 13 to 16, wherein the steel in the strip has a composition according to embodiment 9

19. A coated steel stamped product according to any of the embodiments 13 to 18, wherein the steel has a composition according to embodiment 10

20. A coated steel stamped product according to any of the embodiments 13 to 19, wherein the steel has a composition according to embodiment 11

21. A coated steel stamped product according to any of the embodiments 13 to 20, the aluminum or aluminum alloy pre-coating comprises from 8% to 11% silicon by weight, from 2% to 4% iron by weight, the remainder being aluminum and impurities inherent in processing.

22. A land motor vehicle comprising the heat treated

coated steel product according to any of the embodiments 6, 13 to 21

23. A land motor vehicle comprising the heat treated coated steel product produced according to any of the embodiments 3, 4, 5, 7

[0090] The above written description of the invention provides a manner and process of making and using it such that any person skilled in this art is enabled to make and use the same, this enablement being provided in particular for the subject matter of the appended claims, which make up a part of the original description. As used above, the phrases "selected from the group consisting of," "chosen from," and the like include mixtures of the specified materials. Terms such as "contain(s)" and the like as used herein are open terms meaning 'including at least' unless otherwise specifically noted. All references, patents, applications, tests, standards, documents, publications, brochures, texts, articles, etc. mentioned herein are incorporated herein by reference. Where a numerical limit or range is stated, the endpoints are included. Also, all values and subranges within a numerical limit or range are specifically included as if explicitly written out. The above description is presented to enable a person skilled in the art to make and use the invention, and is provided in the context of a particular application and its requirements. Various modifications to the preferred embodiments will be readily apparent to those skilled in the art, and the generic principles defined herein may be applied to other embodiments and applications without departing from the spirit and scope of the invention. Thus, this invention is not intended to be limited to the embodiments shown, but is to be accorded the widest scope consistent with the principles and features disclosed herein.

## Claims

1. A process for making a hot stamped coated steel sheet product, comprising:

hot dip pre-coating a steel strip having a first side and a second side with aluminum or aluminum alloy to obtain a pre-coated steel strip, the pre-coating thickness on at least one of said first side and said second side of said strip having a thickness of from 20 to 33  $\mu\text{m}$  at every location on at least one of said first and second sides of said strip;  
cutting said pre-coated steel strip to obtain a sheet;  
heating the sheet in a pre-heated furnace to obtain a heated blank in which the steel is austenitic;  
transferring said heated blank to a die while maintaining said steel in the austenitic phase;

and

stamping said heated blank in a die to obtain a hot stamped steel sheet product in which the steel microstructure is martensitic.

2. The process according to claim 1, wherein the sheet is heated to a temperature and during a time defined by diagram ABCD of figure 7 if thickness of said sheet is greater than or equal to 0.7mm and less than or equal to 1.5mm, and by diagram EFGH of figure 7 if thickness of said sheet is greater than 1.5mm and less than or equal to 3mm, at a heating rate  $V_c$  between 20 and 700°C comprised between 4 and 12°C/s, the heating rate  $V_c$  being different from the mean heating rate between room temperature and the furnace temperature.
3. The process according to any one of claims 1 or 2, wherein the elapsed time between said heated blank exits said furnace and said stamping commences is not more than 10 seconds.
4. The process according to any one of claims 1 to 3, wherein said heated blank is deformed by a quantity higher than 10% during said stamping.
5. The process according to any one of claims 1 to 4, wherein the heated blank is cooled at a mean rate between the exit of the heated blank from the furnace, down to 400°C of at least 30°C/s.
6. The process according to any one of claims 1 to 5, wherein the heated blank is cooled at a mean rate between the exit of the heated blank from the furnace, down to 400°C of at least 50°C/s.
7. The process according to any one of claims 1 to 6, wherein the steel strip is made of a base steel comprising the following components by weight based on total weight:

0.15%<carbon<0.5%  
0.5%<manganese<3%  
0.1%<silicon<0.5%  
0.01%<chromium<1%  
titanium<0.2%  
aluminum<0.1%  
phosphorus<0.1%  
sulfur<0.05%  
0.0005%<boron<0.08%,

and further comprises iron and impurities inherent in processing.

8. The process according to claim 7, wherein the base steel comprises the following components by weight based on total weight:

0.20%<carbon<0.5%  
 0.8%<manganese<1.5%  
 0.1%<silicon<0.35%  
 0.01%<chromium<1%  
 titanium<0.1%  
 aluminum<0.1%  
 phosphorus<0.05%  
 sulfur<0.03%  
 0.0005%<boron<0.01 %,

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and further comprises iron and impurities inherent in processing.

9. The process according to claim 7 or 8, wherein the base steel comprises 20 ppm or less of sulfur. 15
10. The process according to any of claims 7 to 9, wherein the ratio of titanium to nitrogen in the base steel in weight % is in excess of 3.42. 20
11. The process according to any of the claims 1 to 10, wherein the aluminum or aluminum alloy pre-coating comprises from 8% to 11% silicon by weight, from 2% to 4% iron by weight, the remainder being aluminum and impurities inherent in processing. 25
12. A land motor vehicle comprising the heat treated coated steel product produced according to any of the claims 1 to 11. 30

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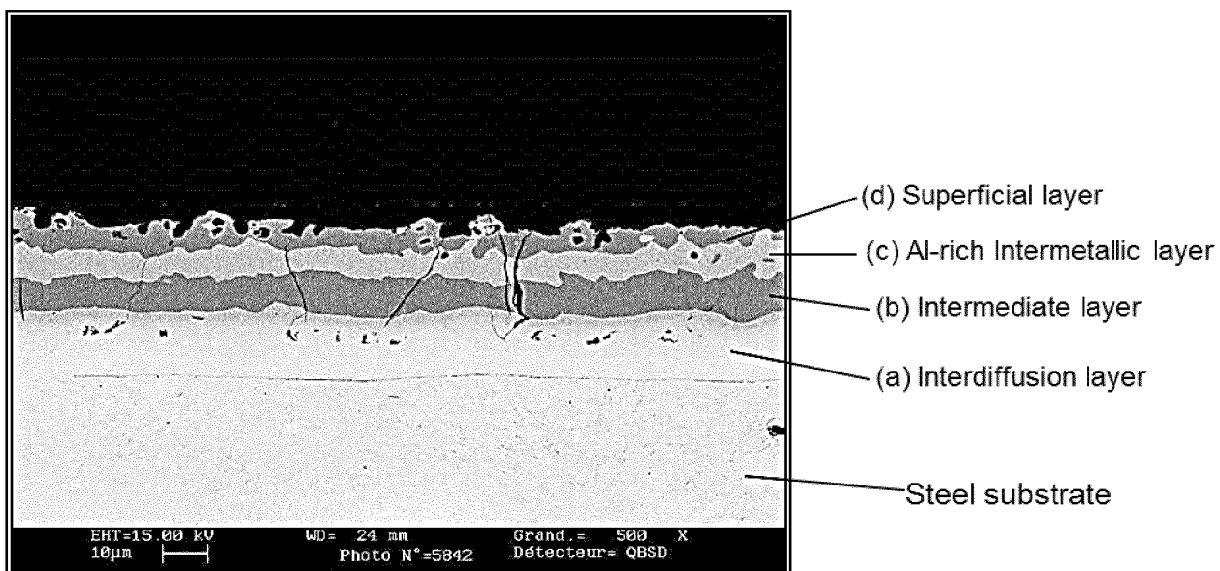


**FIG.1**

10µm

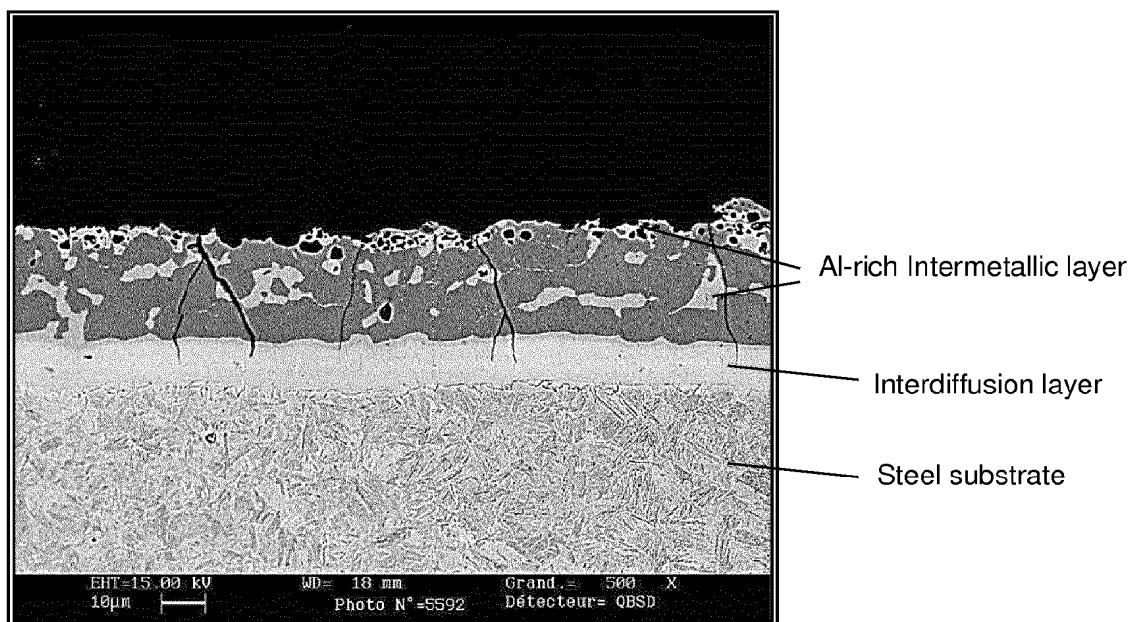


**FIG.2**

10µm

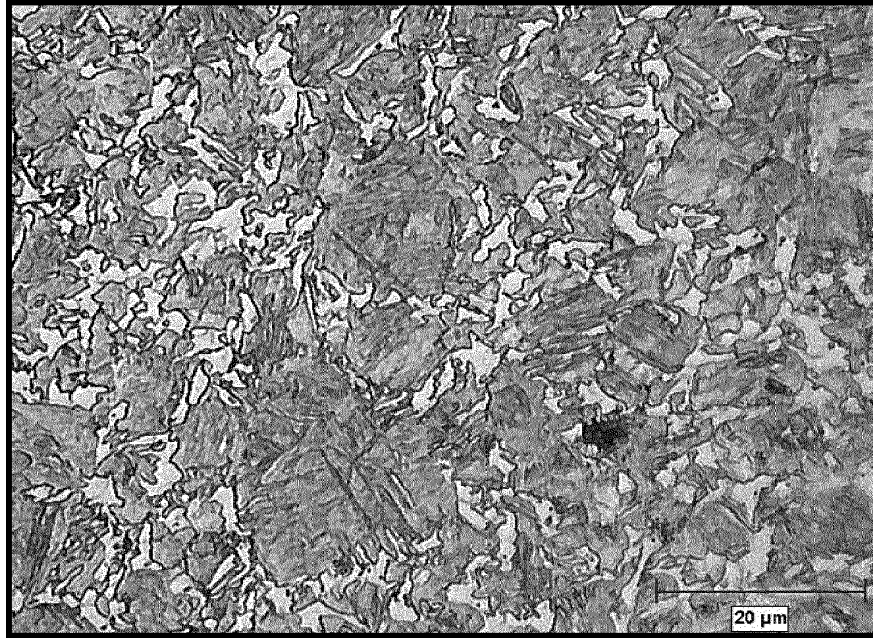


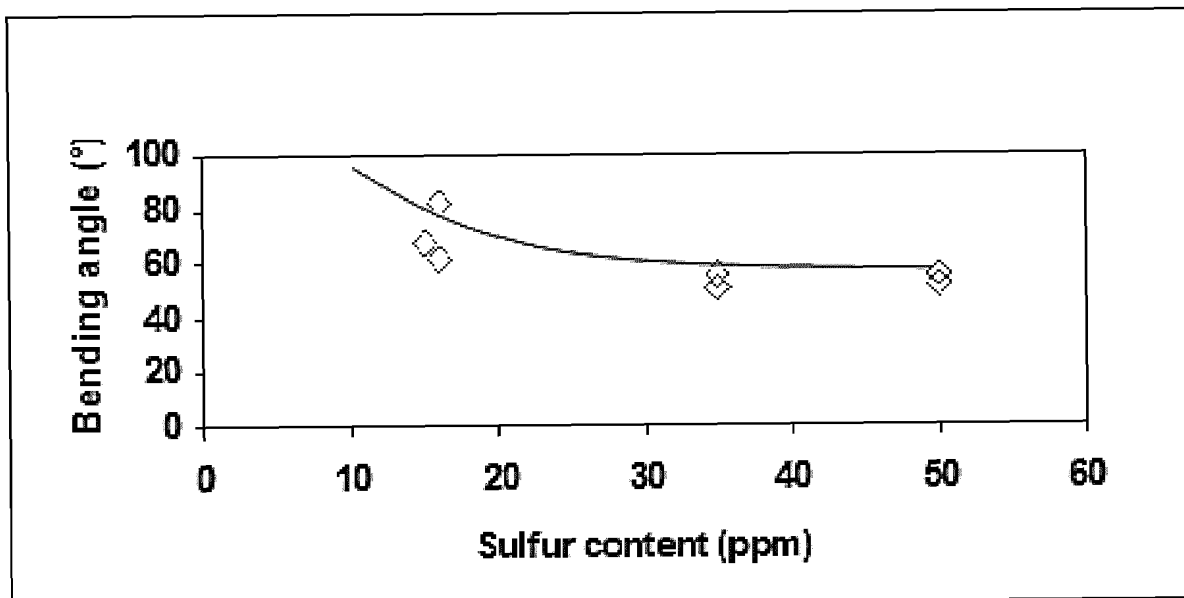
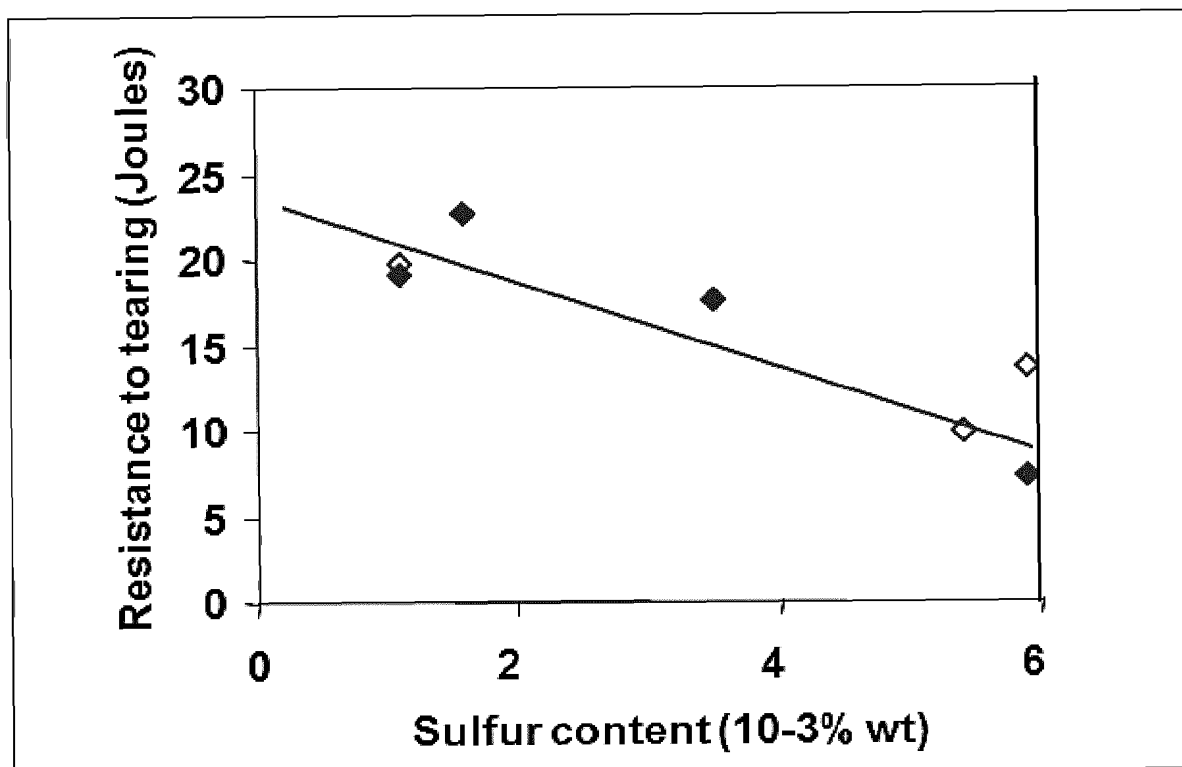
**FIG.3**

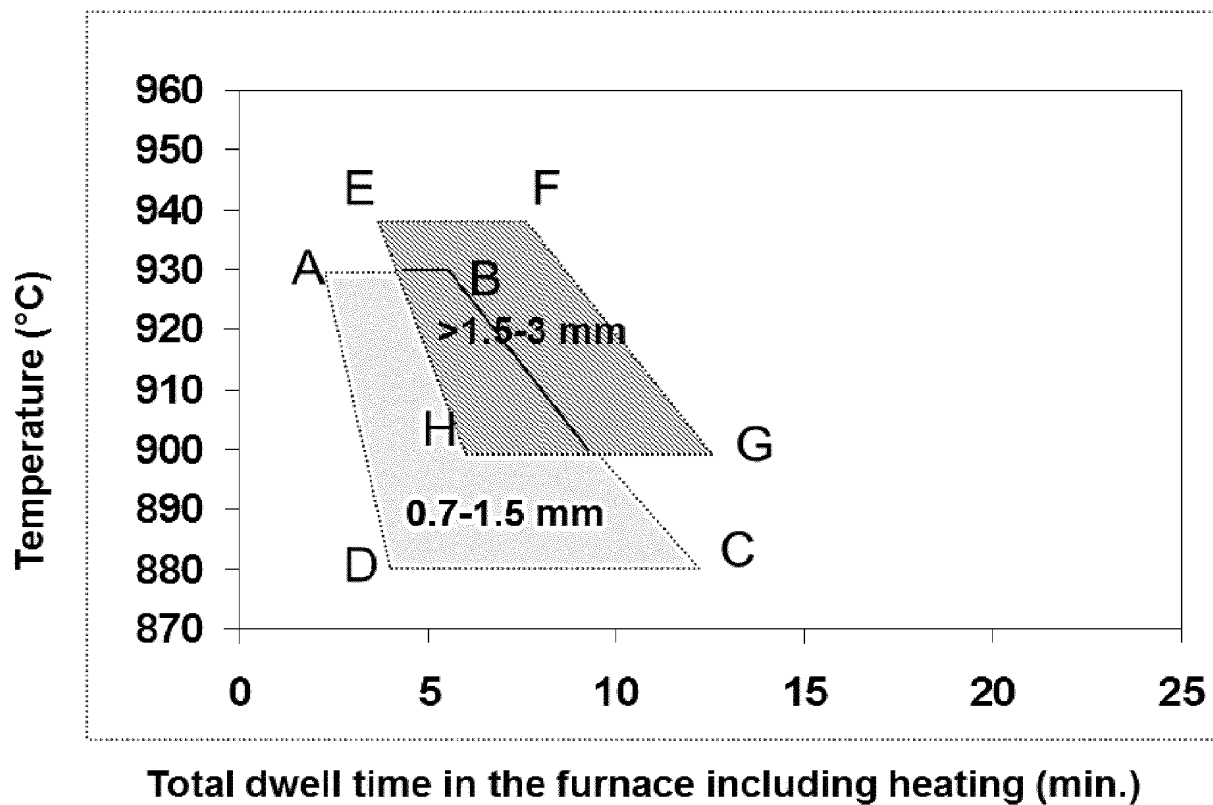
10μm



**FIG.4**

10 μm

**FIG.5****FIG.6**

**FIG. 7**