

DECLARATION OF JUNE ANN MUNFORD

1. My name is June Ann Munford. I am over the age of 18, have personal knowledge of the facts set forth herein, and am competent to testify to the same.
2. I earned a Master of Library and Information Science (MLIS) from the University of Wisconsin-Milwaukee in 2009. I have over ten years of experience in the library/information science field. Beginning in 2004, I have served in various positions in the public library sector including Assistant Librarian, Youth Services Librarian and Library Director. I have attached my Curriculum Vitae as Appendix CV.
3. During my career in the library profession, I have been responsible for materials acquisition for multiple libraries. In that position, I have cataloged, purchased and processed incoming library works. That includes purchasing materials directly from vendors, recording publishing data from the material in question, creating detailed material records for library catalogs and physically preparing that material for circulation. In addition to my experience in acquisitions, I was also responsible for analyzing large collections of library materials, tailoring library records for optimal catalog

search performance and creating lending agreements between libraries during my time as a Library Director.

4. I am familiar with the Internet Archive, a digital library formally certified by the State of California as a public library. Among other services that the Internet Archive makes available to the general public is the Wayback Machine, an online archive. The Internet Archive's Wayback Machine service archives webpages as of a certain capture date to track changes in the web over time. The Internet Archive has been in operation as a nonprofit library since 1996 and has hosted the Wayback Machine service since its inception in 2001. During my time as a librarian, I frequently used the Internet Archive's Wayback Machine for research and instruction purposes. This includes teaching instructional classes on using the Wayback Machine to library patrons and using the Wayback Machine to research reference inquiries that require hard-to-find online resources. I consider the Internet Archive's recordskeeping to be as rigorous and detailed as other formal library recordskeeping practices such as MARC records, OCLC records and Dublin Core.
5. I have reviewed Exhibit EX-1053, a document entitled "Properties of Aluminum-casted Steels for Hot-forming" by Masayoshi Suehiro, et al as

published in Nippon Steel Technical Report July 2003. I have also reviewed Exhibit EX-1054, a document entitled "ホットプレス用アルミニウムめつき鋼板の諸特性" by Masayoshi Suehiro, et al as published in 自動車用材料特集 July 2003. Based on the bibliographic information contained within each exhibit, EX-1054 appears to be a Japanese-language version of EX-1053.

6. Attached hereto as Appendix SUEHIRO01 is a screen capture of a webpage entitled No. 88 - Special Issue on Materials for Automotive use as hosted on the Nippon Steel Technical Report website. I secured these screen captures myself from <http://www.nipponsteel.com/en/tech/report/nsc/no88.html>.
7. Appendix SUEHIRO01 contains a true and correct copy of "Properties of Aluminum-casted Steels for Hot-forming" by Masayoshi Suehiro, et al as published in Nippon Steel Technical Report July 2003. This copy was downloaded directly from the publisher of the report. In comparing Appendix SUEHIRO01 to Exhibit EX-1053, it is my determination that Exhibit EX-1053 is a true and correct copy of "Properties of Aluminum-casted Steels for Hot-forming" by Masayoshi Suehiro, et al as published in Nippon Steel Technical Report July 2003.

8. Attached hereto as Appendix SUEHIRO02 is a screen capture of the Internet Archive Wayback Machine entry for <http://www0.nsc.co.jp/kenkyusho/contenthtml/S378/s37805.pdf>. I secured these screen captures myself from <https://web.archive.org/web/20041119035423/http://www0.nsc.co.jp/kenkyusho/contenthtml/S378/s37805.pdf>. This record was captured as of November 19, 2004.
9. Appendix SUEHIRO02 is an Internet Archive capture of a web page entitled Properties of Aluminum Coated Steels for Hot-forming and ホットプレス用アルミニウムめっき鋼板の諸特性 as hosted on the Nippon Steel Corporation website. Appendix SUEHIRO02 contains a true and correct copy of a Japanese-language edition of "Properties of Aluminum-casted Steels for Hot-forming".
10. Attached hereto as Appendix SUEHIRO03 is a screen capture of the Internet Archive Wayback Machine entry for <http://www0.nsc.co.jp/kenkyusho/contenthtml/S378/s378.html>. I secured these screen captures myself from <https://web.archive.org/web/20031002234705/http://www0.nsc.co.jp/kenkyu>

sho/contenthtml/S378/s378.html. This record was captured as of October 2, 2003.

11. Appendix SUEHIRO03 is an Internet Archive capture of a web page entitled 自動車用材料特集 (No. 378 2003年7月) as hosted on the Nippon Steel Corporation website. Appendix SUEHIRO03 is a table of contents for the Japanese-language edition of Nippon Steel Technical Report July 2003 issue containing "Properties of Aluminum-casted Steels for Hot-forming". This page contains a link entitled ホットプレス用アルミニウムめっき鋼板の諸特性 that provides a download of the PDF copy of the Japanese-language edition of "Properties of Aluminum-casted Steels for Hot-forming" presented in this declaration as Appendix SUEHIRO02. As this page was first captured as of October 2, 2003, it is my determination that "Properties of Aluminum-casted Steels for Hot-forming" by Masayoshi Suehiro, et al was first made available to the public as of October 2, 2003 via the Internet Archive and likely earlier via the original Nippon Steel website.

12. Appendices SUEHIRO02 and SUEHIRO03 indicate that Nippon Steel was distributing "Properties of Aluminum-casted Steels for Hot-forming" to any member of the public capable of visiting the Nippon Steel website from 2003-2004. The Table of Contents page found in Appendix SUEHIRO03

provides an accurate description of the July 2003 issue of Nippon Steel Technical Report as represented in Exhibit EX-1053, Exhibit EX-1054, Appendix SUEHIRO01 and Appendix SUEHIRO03. The links included in Appendix SUEHIRO03 indicate users visiting this Table of Contents webpage as of October 2, 2003 would have been provided a download link to each paper contained within this volume, including "Properties of Aluminum-casted Steels for Hot-forming". Following the URL link to "Properties of Aluminum-casted Steels for Hot-forming" found in Appendix SUEHIRO03 produces the document presented in this declaration as Appendix SUEHIRO02, a true and correct copy of the Japanese-language version of "Properties of Aluminum-casted Steels for Hot-forming". Appendix SUEHIRO02 was captured by the Internet Archive on November 19, 2004, as a distinct record separate from the Table of Contents page. As such, it is my determination that "Properties of Aluminum-casted Steels for Hot-forming" was made available to the public as of October 2, 2003 via the Table of Contents page and certainly no later than November 19, 2004, the earliest possible date to download the paper from the Internet Archive.

13. I have been retained on behalf of the Petitioner to provide assistance in the above-illustrated matter in establishing the authenticity and public

availability of the documents discussed in this declaration. I am being compensated for my services in this matter at the rate of \$200.00 per hour plus reasonable expenses. My statements are objective, and my compensation does not depend on the outcome of this matter.

14. I declare under penalty of perjury that the foregoing is true and correct. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made to the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code.

Dated: 12/22/2024

A handwritten signature in black ink, appearing to read 'June Ann Munford', with a stylized flourish at the end.

June Ann Munford

Appendix CV

June A. Munford
Curriculum Vitae

Education

University of Wisconsin-Milwaukee - MS, Library & Information Science, 2009
Milwaukee, WI

- Coursework included cataloging, metadata, data analysis, library systems, management strategies and collection development.
- Specialized in library advocacy, cataloging and public administration.

Grand Valley State University - BA, English Language & Literature, 2008
Allendale, MI

- Coursework included linguistics, documentation and literary analysis.
- Minor in political science with a focus in local-level economics and government.

Professional Experience

Researcher / Expert Witness, October 2017 – present
Freelance ● Pittsburgh, Pennsylvania & Grand Rapids, Michigan

- Material authentication and public accessibility determination. Declarations of authenticity and/or public accessibility provided upon research completion. Experienced with appeals and deposition process.
- Research provided on topics of public library operations, material publication history, digital database services and legacy web resources.
- Past clients include Alston & Bird, Arnold & Porter, Baker Botts, Fish & Richardson, Erise IP, Irell & Manella, O'Melveny & Myers, Perkins-Coie, Pillsbury Winthrop Shaw Pittman and Slayden Grubert Beard.

Library Director, February 2013 - March 2015
Dowagiac District Library ● Dowagiac, Michigan

- Executive administrator of the Dowagiac District Library. Located in

Southwest Michigan, this library has a service area of 13,000, an annual operating budget of over \$400,000 and total assets of approximately \$1,300,000.

- Developed careful budgeting guidelines to produce a 15% surplus during the 2013-2014 & 2014-2015 fiscal years while being audited.
- Using this budget surplus, oversaw significant library investments including the purchase of property for a future building site, demolition of existing buildings and building renovation projects on the current facility.
- Led the organization and digitization of the library's archival records.
- Served as the public representative for the library, developing business relationships with local school, museum and tribal government entities.
- Developed an objective-based analysis system for measuring library services - including a full collection analysis of the library's 50,000+ circulating items and their records.

November 2010 - January 2013

Librarian & Branch Manager, Anchorage Public Library ● Anchorage, Alaska

- Headed the 2013 Anchorage Reads community reading campaign including event planning, staging public performances and creating marketing materials for mass distribution.
- Co-led the social media department of the library's marketing team, drafting social media guidelines, creating original content and instituting long-term planning via content calendars.
- Developed business relationships with The Boys & Girls Club, Anchorage School District and the US Army to establish summer reading programs for children.

June 2004 - September 2005, September 2006 - October 2013

Library Assistant, Hart Area Public Library
Hart, MI

- Responsible for verifying imported MARC records and original MARC

cataloging for the local-level collection as well as the Michigan Electronic Library.

- Handled OCLC Worldcat interlibrary loan requests & fulfillment via ongoing communication with lending libraries.

Professional Involvement

Alaska Library Association - Anchorage Chapter

- Treasurer, 2012

Library Of Michigan

- Level VII Certification, 2008
- Level II Certification, 2013

Michigan Library Association Annual Conference 2014

- New Directors Conference Panel Member

Southwest Michigan Library Cooperative

- Represented the Dowagiac District Library, 2013-2015

Professional Development

Library Of Michigan Beginning Workshop, May 2008

Petoskey, MI

- Received training in cataloging, local history, collection management, children's literacy and reference service.

Public Library Association Intensive Library Management Training, October 2011

Nashville, TN

- Attended a five-day workshop focused on strategic planning, staff management, statistical analysis, collections and cataloging theory.

Alaska Library Association Annual Conference 2012 - Fairbanks, February 2012

Fairbanks, AK

- Attended seminars on EBSCO advanced search methods, budgeting, cataloging, database usage and marketing.

Depositions

2019 ● Fish & Richardson

Apple v. Qualcomm (IPR2018-001281, 39521-00421IP, IPR2018-01282 and 39521-00421IP2)

2019 ● Erise IP

Implicit, LLC v. Netscout Systems, Inc (Civil Action No. 2:18-cv-53-JRG)

2019 ● Perkins-Coie

Adobe Inc. v. RAH Color Technologies LLC (Cases IPR2019-00627, IPR2019-00628, IPR2019-00629 and IPR2019-00646)

2020 ● O'Melveny & Myers

Maxell, Ltd. v. Apple Inc. (Case No. 5:19-cv-00036-RWS)

2021 ● Pillsbury Winthrop Shaw Pittman LLP

Intel v. SRC (IPR2020-1449)

2022 ● Perkins-Coie

Realtek v. Future Link (IPR2021-01182)

2023 ● Fish & Richardson

Neuroderm Ltd. v. Abbvie, Inc (Case No. PGR2022-00040)

2023 ● Fish & Richardson

Nearmap US Inc. v. Pictometry International Corp. (IPR2022-00735)

2023 ● Fish & Richardson

Samsung Electronics v. MemoryWeb LLC (Case No. 39843-0136PS1)

2023 ● Pillsbury Winthrop Shaw Whitman LLP

Gravel Rating Systems v. Costco Wholesale Corp. (Civil Action No. 4:21-cv-149-ALM)

2024 ● Willkie-Farr

Netflix, Inc. v. VideoLabs. Inc. (IPR2023-00628)

Limited Case and Clientele History

Alston & Bird

- Ericsson

v. Collision Communications (IPR2022-01233)

- Nokia

v. Neptune Subsea, Xtera (Case No. 1:17-cv-01876)

- Universal Electronics Inc

v. Roku Inc (IPR2022-00818)

Arnold & Porter

- Ivantis

v. Glaukos (Case No. 8:18-cv-00620)

- Samsung

v. Jawbone (Case No. 2:21-cv-00186)

Benesch Friedlander Coplan & Aronoff

- Voyis
 - v. Cathx (Case No. 5:21-cv-00077-RWS)

Deschert LLP

- Smaxtec Animal Care
 - v. ST Reproductive Technologies, LLC (IPR2024-00885)

Erise I.P.

- Apple
 - v. Ericsson Inc. (IPR2022-00715)
 - v. Future Link Systems (IPRs 6317804, 6622108, 6807505, and 7917680)
 - v. INVT (Case No. 20-1881)
 - v. Navblazer LLC (IPR2020-01253)
 - v. Qualcomm (IPR2018-001281, 39521-00421IP, IPR2018-01282, 39521-00421IP2)
 - v. Quest Nettech Corp (Case No. 2:19-cv-00118-JRG)
 - v. Telefonaktiebolaget LM Ericsson (IPR2022-00275)
 - v. Theta IP, LLC (IPR2024-00818)

- Fanduel
 - v. CGT (Case No. 19-1393)

- Garmin
 - v. Phillips North America LLC (Case No. 2:19-cv-6301-AB-KS)

- Netscout

- v. Longhorn HD LLC (Case No. 2:20-cv-00349)

- v. Implicit, LLC (Civil Action No. 2:18-cv-53-JRG)

- Sony Interactive Entertainment LLC

- v. Bot M8 LLC (IPR2020-01288)

- v. Infernal Technology LLC (Case No. 2:19-CV-00248-JRG)

- Unified Patents

- v. GE Video Compression (Civil Action No. 2:19-cv-248)

Fish & Richardson

- Apple

- v. AliveCor (3:21-cv-03958)

- v. LBS Innovations (Case No. 2:19-cv-00119-JRG-RSP)

- v. Koss Corporation (IPR2021-00305)

- v. Masimo (IPR 50095-0012IP1, 50095-0012IP2, 50095-0013IP1, 50095-0013IP2, 50095-0006IP1, 50095-0135IP1)

- v. Neonode (Case No. 21-cv-08872-EMC)

- v. Qualcomm (IPR2018-001281, 39521-00421IP, IPR2018-01282, 39521-00421IP2)

- Dell

- v. Neo Wireless (IPR2022-00616)

- Dish Network

- v. Entropic Communications, LLC (Case No. 2:2023-CV-01043)

- v. Realtime Adaptive Streaming (Case No. 1:17-CV-02097-RBJ)
 - v. TQ Delta LLC (Case No. 18-1798)
- Evapco Dry Cooling
 - v. SPG Dry Cooling (IPR2021-00688)
- Genetec
 - v. Sensormatic Electronics (Case No. 1:20-CV-00760)
- Huawei
 - v. Bell Northern Research LLC (IPR2019-01174)
- Kianxis
 - v. Blue Yonder (Case No. 3:20-cv-03636)
- LG Electronics
 - v. Bell Northern Research LLC (Case No. 3:18-cv-2864-CAB-BLM)
- Metaswitch
 - v. Sonus Networks (IPR2018-01719)
- Microsoft
 - v. Throughputer Inc (IPR2022-00757)
- Mom Enterprises
 - v. Ddrops Company (Case No. 1:22-cv-00332)
- MLC Intellectual Property

- v. MicronTech (Case No. 3:14-cv-03657-SI)
- Nearmap Inc
 - v. EagleView Technologies (IPR2022-01009)
- Neuroderm Ltd.
 - v. Abbvie, Inc (Case No. PGR2022-00040)
- Realtek Semiconductor
 - v. Future Link (IPR2021-01182)
- Quectel
 - v. Koninklijke Philips (Case No. 1:20-cv-01710)
- Samsung
 - v. Aire Technology (IPR2022-00877)
 - v. Bell Northern Research (Case No. 2:19-cv-00286-JRG)
 - v. Communication Technologies Inc (IPR2022-01221)
 - v. Jawbone Innovations (IPR2022-00865)
 - v. MemoryWeb LLC (IPR2022-00885)
 - v. Dodots Licensing Solutions (IPR2023-00701)
 - v. Telefonaktiebolaget LM Ericsson (IPR2021-00615)
- Texas Instruments
 - v. Vantage Micro LLC (IPR2020-01261)
- Xilinx
 - v. Sentient Sensors LCC (Case No. 1:22-cv-00173)

Groombridge Wu

- Nearmap

v. Eagle View Technologies Inc. and Pictometry International Corp.
(Case No. 2:21-cv-00283-TS-DAO)

Irell & Manella

- Curium

O'Melveny & Myers

- Apple

v. Maxell (Case No. 5:19-cv-00036-RWS)

- Disney Media & Entertainment Distribution LLC

v. Digital Media Technology Holdings, LLC (Case No. 2:22-cv-01642)

- Micron Technology Inc.

v. Besang Inc (IPR2023-00900)

- Samsung

v. Daedalus Prime LLC (1335 Investigation)

Perkins-Coie

- Amazon

v. Nokia (IPR2024-00847 and IPR2024-00848)

- Heru Industries
 - v. The UAB Research Foundation (IPR2022-01148)
- Intel Corporation
 - v. BeSang Inc. (IPR2023-00991)
 - v. BitMICRO (Case No. 5:23-cv-00625)
 - v. Telefonaktiebolaget L M Ericsson (IPR2024-00610)
- Realtek Semiconductor
 - v. Future Link (IPR2021-01182)
- Twitter Inc
 - v. VOIP-Pal.com (Case No. 3:20-cv-02397-JD)
- TCL Industries
 - v. Koninklijke Philips NV (IPR2021-00495, IPR2021-00496 and IPR2021-00497)

Pillsbury Winthrop Shaw Pittman

- Intel
 - v. FG SRC LLC (Case No. 6:20-cv-00315)
- Gravel Rating Systems
 - v. Costco (Case No. 4:21-cv-149)
 - v. Lowe's Home Centers (Case No. 4:21-cv-150)
 - v. T-Mobile USA (Case No. 4:21-cv-152)
 - v. Kohl's Inc. (Case No. 4:21-cv-258)
 - v. Under Armor (Case No. 4:21-cv-356)

Quinn Emanuel

- ServiceNow, Inc.
 - v. InQuisient, Inc. (Case No. 22-900-CJB)
- Wolfspeed
 - v. The Trustees of Purdue University (Case No. 1:2021-cv-00840)

Sheppard, Mullin, Richter & Hampton LLP

- Cadence Design Systems Inc.
 - v. Semiconductor Design Technologies (Case No. 3:23-cv-01001)

Stern, Kessler, Goldstein & Fox

- Preliminary Research

Willkie, Farr & Gallagher LLP

- Neurent
 - v. The Foundry, LLC (IPR2024-00669)
- Netflix, Inc.
 - v. VideoLabs, Inc. (IPR2023-00628)

Appendix SUEHIRO01

No.88 Special Issue on Materials for Automotive Use

Jul. 2003

Contents

1. Remarks on Special Issue on Materials for Automotive Use (43KB)
2. Development of High Strength Steels for Automobiles (129KB)
3. High Strength Hot-rolled Steel Sheets for Automobiles (155KB)
4. Developments of Ultra-High-Strength Cold-Rolled Steel Sheets for Automotive Use (72KB)
5. Properties of Aluminum-coated Steels for Hot-forming (122KB)
6. Material Characterization at High Strain Rates for Optimizing Car Body Structure for Crash Events (55KB)
7. Shape Control Techniques for High Strength Steel in Sheet Metal Forming (128KB)
8. Factors to Determine Static Strengths of Spot-weld for High Strength Steel Sheets and Developments of High-strength Steel Sheets with Strong and Stable Welding Characteristics (42KB)
9. Welding Methods and Forming Characteristics of Tailored Blanks (TBs) (228KB)
10. Chromate-free Electro-galvanized Steel Sheets for Automobile Use, "ZINKOTE MZ" (42KB)
11. Lubricant Coating Film for Zinc-plated Steel Sheets (215KB)
12. Pb-free Coated Steel Sheets for Fuel Tanks (328KB)
13. Electrical Steel Sheet for Traction Motors of Hybrid/Electric Vehicles (403KB)
14. Present and Future Trends of Stainless Steel for Automotive Exhaust System (717KB)

- 15. Application of Titanium and Its Alloys for Automobile Parts (450KB)
- 16. Development of Lead Free Micro Alloyed Steel for Crank Shafts (172KB)
- 17. Development of Anti-coarsening Extra-fine Steel for Carburizing (280KB)
- 18. State of the Art for High Tensile Strength Steel Cord (211KB)

Properties of Aluminum-coated Steels for Hot-forming

Masayoshi SUEHIRO*¹
Kazuhisa KUSUMI*¹
Toshihiro MIYAKOSHI*²

Jun MAKI*¹
Masahiro OHGAMI*¹

Abstract

Production of parts with high strength by means of hot-forming process has been receiving considerable attention lately. Application of aluminized steel with good hardenability for this process has been investigated. Tensile strength over than 1,500MPa was obtained with steel containing around 0.2mass% C. Surface coated layer changed from Al-10% Si to Fe-Al(-Si) phases through the hot-forming process. The steel with Fe-Al(-Si) surface layer showed good paintability even without phosphatized treatment. After painting, they showed good corrosion resistance in JASO-CCT as galvanized steel did. The steel showed good spot weldability due to the stable surface layer at high temperature. (*license product within the frame of global strategic alliance with Arcelor (USIBOR1500))*

1. Introduction

High-strength steel sheets are used more and more for automobile components in recent years to reduce the weight of an automobile to improve environmental problems and collision safety. Many car components and structural members can be made significantly lighter by using high-strength steel sheets and reducing their thicknesses. With this as a background, the strength of the steel sheets used therefor has increased from 440- or 590-MPa class to 780-MPa class. Generally speaking, as the strength of a steel sheet increases, its formability deteriorates, and to overcome this problem, various types of high-strength steel sheet products with controlled microstructures have been developed¹⁾. In spite of these efforts, however, press forming complicated shapes is difficult when the strength of a steel sheet is as high as approximately 1,500 MPa.

In Europe, a forming method called die quenching, hot forming, hot stamping or hot pressing is employed for fabricating automobile structural members having a strength of 1,500 MPa or so²⁾. By this method, the strength of a steel sheet is increased through quenching after heating it to a temperature in the austenite temperature range, say, around 900°C, while forming dies are maintained at room temperature. Techniques similar to this are being studied in Japan; similar processes have been studied or put into actual practice using steel

sheets without surface treatment^{3,4)}. In Europe, Arcelor has developed and commercially launched an aluminized steel sheet product called USIBOR 1500 for that application. It has excellent hot-forming properties and corrosion resistance qualities^{2,5-6)}. Based on a global strategic alliance with Arcelor, Nippon Steel Corporation has entered into a license agreement regarding USIBOR 1500 and studied the production technologies and properties of the product. This paper reports the results of the company's studies of the properties and characteristics that are required of aluminized steel sheet products for hot forming application.

2. Hot Forming Process

The hot forming method is already a practical reality; the process is outlined in **Fig. 1**. In this method it is necessary to heat a steel sheet to a temperature in the austenite temperature range, equal to or higher than the A_{c3} temperature: for example, steel containing about 0.2 mass % C must be heated to 850°C or higher. Heated steel sheet is extracted from a heating furnace, transferred to a pressing machine, formed into a prescribed shape using dies maintained at room temperature, and thus quenched. At the forming work, the press machine is retained at the lower dead point until the entire steel sheet is quenched sufficiently.

*¹ Yawata R&D Lab.

*² Yawata Works

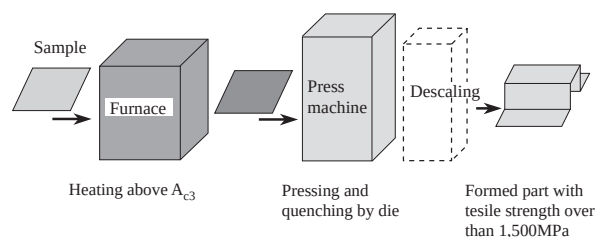


Fig. 1 Schematic illustration of hot forming process

In these processes, when a steel sheet without surface treatment is used, the heating must be done in a non-oxidizing atmosphere for suppressing scale formation and decarburization of its surface layers. Even in the above case, the processes from the exit from the furnace to the pressing machine are carried out inevitably in the normal atmosphere and for this reason, scale forms on the steel sheet surfaces however short the processing time may be. A formed product will be finally coated for use, but the scale causes a problem for the coating work. Therefore, when a steel sheet without surface treatment is used, an additional process step for removing the scale is required. The scale removal can be done through shot blasting, pickling, or the like, but this results in considerable increases in costs. Further, if shot blasting is employed, the shape of a work may be deteriorated. If a steel sheet with heat-resistant plating is used, the additional process step can be eliminated.

3. Material

Since a steel sheet is rapidly cooled in the hot forming process for hardening it as explained above, a material steel sheet must have good hardenability. To eliminate the furnace atmosphere control and the descaling process to simplify the work processes, the surfaces of the material steel sheet must be coated with plating layers excellent in heat resistance. An aluminized steel sheet having the chemical composition shown in **Table 1** satisfies all these requirements. The properties and characteristics of the specimen steel sheets used for authors' studies, the coating weights of which ranged from 120 to 160 g/m², are described below.

4. Properties of Base Material

4.1 properties before and after quenching

The properties of the steel sheets before and after the quenching are listed in **Table 2**. The tensile strength was measured using JIS No. 5 test pieces cut out from steel sheets of 1.4 mm thickness. Steel sheets of 490- and 590-MPa classes were quenched by the dies to a strength of 1,500 MPa or more. As seen in **Fig. 2**, the strength after

Table 1 Chemical composition of steel sheets used

C	Mn	Cr	B
0.22	1.2	0.15	0.002

Table 2 Tensile properties before and after quenching

	Yield strength (MPa)	Tensile strength (MPa)	Elongation (%)
As coated	394	615	26
As formed	1,280	1,530	8

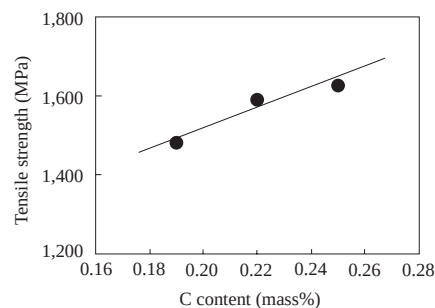


Fig. 2 Relationship between C content and tensile strength

quenching can be controlled by C content. As has been known, the mechanical properties of steel after quenching change depending on its C content and consequently, the strength after quenching, or the strength of a product, can be controlled by properly adjusting the C content. Note that substitute solid solution elements such as Mn and Cr are known to have only small influence on the strength after quenching. However, since these elements have influences over hardenability, they are essential for securing hardenability, which will be discussed in the following section. It should be noted that an excessive addition of these elements increases the strength of a base steel sheet, even though the effect on the strength is small comparing to that of C.

4.2 Hardenability

Fig. 3 shows the continuous cooling transformation curves (CCT curves) of the specimen steels from 950°C, and **Fig. 4** the relationship between the cooling rate and the hardness after cooling. Mn

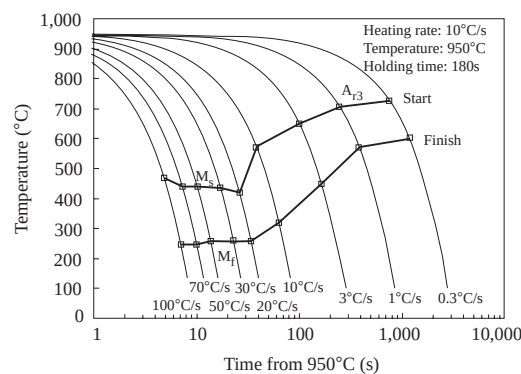


Fig. 3 CCT curves

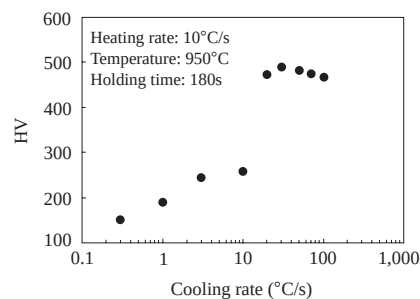


Fig. 4 Influence of cooling rate on hardness

and B had been added to the steels for increasing hardenability and as a result, when they were heated to 950°C and then cooled at a cooling rate of roughly 20°C/s or higher, a hardness of HV 450 to 500 was obtained. The cooling rate for quenching is determined by the conditions for avoiding the transformations of austenite into ferrite, pearlite and bainite during cooling. As the grain size of austenite becomes smaller, or the heating temperature becomes lower, the transformations into these phases take place quicker and as a consequence, the cooling rate for the quenching must be higher. The authors studied this aspect changing the heating temperature from 900 to 1,000°C, but no significant difference was observed in this heating temperature range. The critical cooling rate was 20 to 30°C/s or so.

From Figs. 3 and 4 and the results of the tests at other heating temperatures, it is understood that a sufficient hardness is obtained as far as the cooling rate during the hot forming is equal to or higher than 30°C/s. However, in actual practice, the cooling rate during the transfer from the heating furnace to the pressing machine and the time period before the commencement of the pressing work is close to that in the air.

When a steel sheet with thickness of about 1.4 mm is cooled in the normal atmosphere, the cooling rate is 10 to 15°C/s. It is clear from Figs. 3 and 4 that the strength of the steel sheet is decreased when it is cooled to 600°C or lower at this cooling rate. To cool a steel sheet from 950 to 600°C it takes 23 to 35 s; therefore, a material steel sheet can be quenched if its cooling with the pressing dies is commenced within 23 s after it is extracted from a heating furnace. It should be noted that this time allowance changes depending on heating conditions, the atmosphere during the cooling and surrounding environment and for this reason, it has to be determined on the basis of actual work environment.

4.3 Toughness

In the case where strength is enhanced through formation of martensite by quenching, there is a fear that toughness is lowered, especially at a low temperature. Figs. 5 and 6 show the results of Charpy impact tests carried out for studying this aspect. Fig. 5 shows the relationship between impact toughness and test temperature, and Fig. 6 the relationship between percent brittle fracture and test temperature. 2.5 mm sub-size test pieces with a 2-mm U-shaped notch were cut out from water-quenched steel sheets of 4 mm in thickness and used for the tests. Whereas the absorbed energy does not change significantly from room temperature to -60°C, the percent brittle fracture increases at -40°C and lower, reaching 80% at -80°C. The transition temperature ($T_{r_{50}}$) is somewhere around -60°C. Since the percent brittle fracture tends to decrease as the material thickness

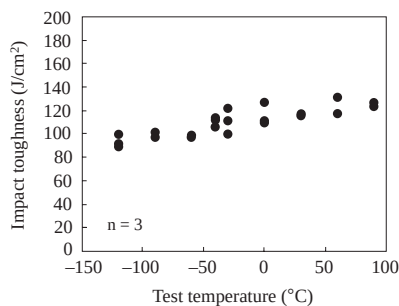


Fig. 5 Relationship between Charpy impact toughness and test temperature

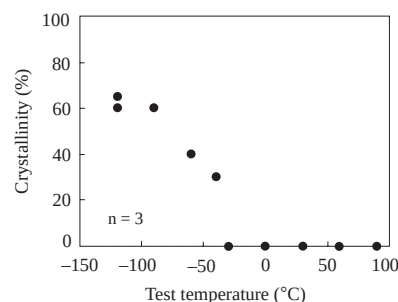


Fig. 6 Relationship between percent brittle fracture and test temperature

decreases, the transition temperature is presumably lower yet if the thickness is less than 2.5 mm. Based on these results, it is presumed that the steel sheet product, which has an as-quenched metallographic structure mainly consisting of a martensitic phase when it is finally used, does not display any abrupt change in absorbed energy or the mode of fracture even if it is exposed to a low temperature under normal conditions of use.

5. Spot Weldability

Test pieces for spot welding tests were prepared by heating aluminumized steel sheets of 1.2 mm thickness having a coating weight of 120 g/m² to 950°C, retaining at the temperature for 5 min, and then quenching by pinching them with metal dies from both the sides. The plating layers were of an Fe-Al alloy as explained in the following sections. The test pieces were spot welded to 440-MPa class cold-rolled steel sheets of 1.8 mm thickness using DR6φ type electrodes of alumina-dispersed copper at a welding time of 17 cycles (60 Hz) and an electrode force of 400 kgf. Fig. 7 shows the relationship between the nugget diameter and the current density; the welding current range is roughly 3 kA from the current value at which the nugget diameter becomes $4\sqrt{t}$ relative to the sheet thickness t to that at which expulsion occurs. Fig. 8 shows the relationship between tensile shear strength and welding current, and Fig. 9 shows that between cross tension strength and the welding current. Further, Fig. 10 shows hardness distribution near a nugget. Hardness is approximately HV 480 at the inside of a nugget and the base metal, and it falls to HV 300 or so at heat-affected zones (HAZ). Because of such a hardness distribution, failure occurs at the HAZ in most cases.

Fig. 11 shows the evaluation results of consecutive spot weldability. The test was continued up to 3,000 consecutive spot welds and no significant change in the nugget diameter was observed, and

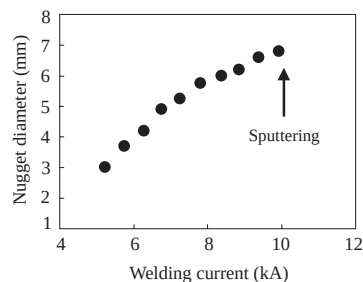


Fig. 7 Relationship between nugget diameter of spot-welded portion and welding current

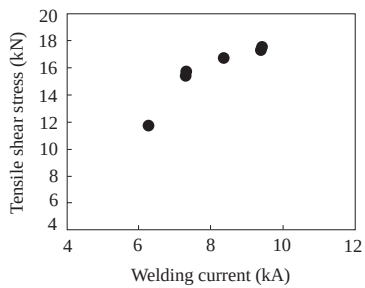


Fig. 8 Relationship between tensile shear strength of spot-welded portion and welding current

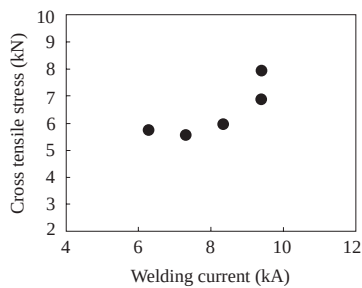


Fig. 9 Relationship between cross tension strength of spot-welded portion and welding current

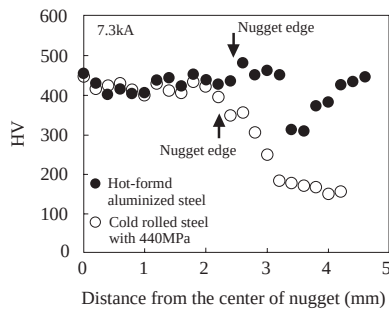


Fig. 10 Hardness distribution near spot-welded portion

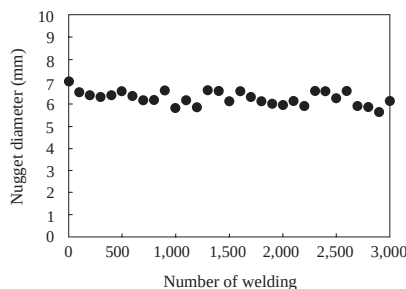


Fig. 11 Change of nugget diameter in consecutive spot welds

more consecutive spot welds were presumed to be attainable. The plating layer of the steel sheet consists of an Fe-Al alloy phase having a melting point higher than 1,000°C, and this is considered to be one of the reasons for the good electrode service life.

No data related to arc-weldability are presented herein. However, since the Fe-Al alloy phase has a boiling point higher than 2,000°C, the occurrence of blowholes, which cause a problem in the arc welding of galvanized steel sheets, does not take place.

6. Coating Layer Characteristics

6.1 Alloying of coating layer during heating

The coating layer of the steel sheet product is what is called the Type 1 coating layer, a plating layer of aluminum containing 10% Si. Its melting point is approximately 600°C, and the layer melts when heated quickly. However, during heating at a heating rate attainable with an electric furnace, an Fe-Al alloy having a high melting point forms from the interface with the base metal, and the alloying quickly reaches the surface and for this reason, no molten alloy sticks to furnace structures. The time that is required for reaching a holding temperature in an electric furnace of normal furnace atmosphere is about 2 min in the case of a 1.6-mm thick aluminized steel sheet.

Fig. 12 shows an alloy layer structure after retaining an aluminized steel sheet at 950°C for 0.5 min, and Fig. 13 the concentrations of Fe, Al and Si in the plating layer measured in the thickness direction by SEM-EDS. As the alloying proceeds, the coating layer develops a sub-layer structure as shown in the figure. In the case of

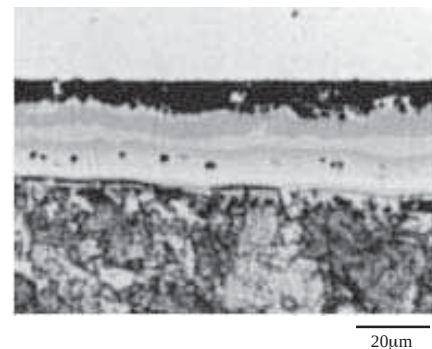


Fig. 12 Sectional optical micrograph showing plating layer structure after heated at 950°C for 30 s

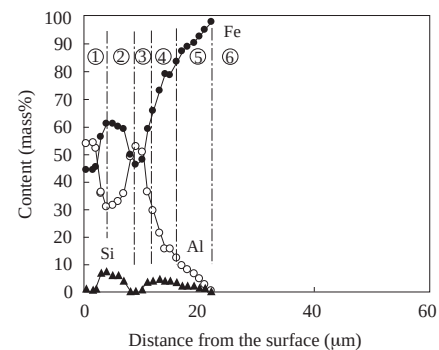


Fig. 13 Change of Fe, Al, and Si concentrations in thickness direction of sample corresponding to Fig. 12

the above heating condition, the plating layer consisted of five sub-layers, and the Al concentrations in the sub-layers were 50, 30, 50, 30 and 10%, respectively, from the surface to inside. The concentrations of the elements changed continuously from the fourth sub-layer to the base metal.

Fig. 14 is a ternary phase diagram of an Fe-Al-Si system at 950°C calculated using Thermo-Calc⁷⁾ based on the thermodynamic database of the Fe-Al-Si system presented by Z. Liu et al.⁸⁾ The authors superimposed the compositions of the plots of Fig. 13 upon the phase diagram and as a result, the sub-layers in Fig. 13 were identified, presumably, as Fe₂Al₅, an ordered BCC phase, FeAl₂, an ordered BCC phase, and a disordered BCC phase, respectively, from the surface to inside. As the holding time becomes longer, Al diffuses into inner layers and finally into the base steel and as a consequence, the sub-layers disappear gradually from the outermost to inside. **Fig. 15** shows a plating layer after retaining at the temperature for 5 min. Although only one layer could be observed in sectional observation, an SEM-EDS analysis revealed the existence of two sub-layers; they were presumed to be an ordered and a disordered BCC phase.

6.2 Paintability and corrosion resistance after painting

Steel sheets are used as automobile components after painting by electro-deposition. Usually, they are subjected to a chemical treatment prior to the painting for ensuring good paintability. The chemical treatment generally consists of dipping a steel sheet in a solution of zinc phosphate to form coating films of phosphate on the surfaces. However, the coating films do not form on the surface of the

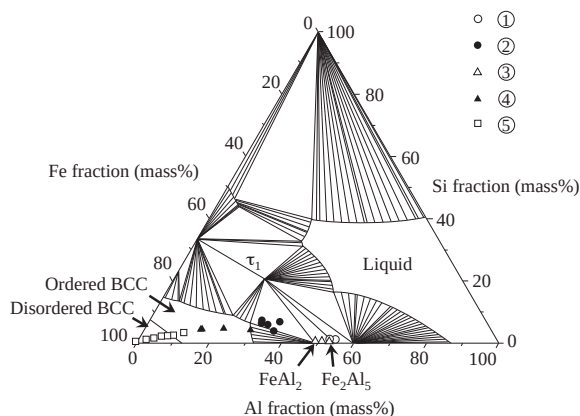


Fig. 14 Isothermal section of Fe-Al-Si ternary phase diagram (at 950°C)

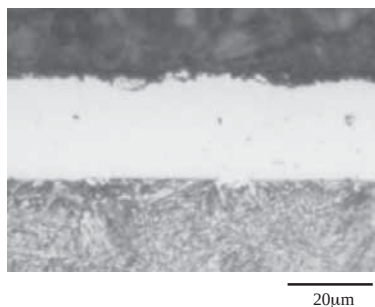


Fig. 15 Sectional optical micrograph showing plating layer structure after retention at 950°C for 5 min

Fe-Al alloy layer because the alloy is very stable against acid. The surface of the Fe-Al alloy phase after the heat treatment is rough as seen in **Fig. 16** and thanks to an anchoring effect of the surface, its paintability is good without the chemical treatment. **Table 3** shows a comparison of the adhesion of electro-deposited paint of about 20 μm thickness between samples with and without the chemical treatment; it is clear that the aluminized steel sheets hot-formed have sufficiently good paint adhesion regardless of the application or otherwise of the chemical treatment.

An aluminized steel sheet with a coating weight of 160 g/m² was heated to 900°C for 2 min to form an Fe-Al alloy layer at the surface, immersed in a chemical treatment solution, coated with electro-deposited paint of roughly 25 μm thickness, then, after cross-cutting the paint coating, subjected to 150 cycles (50 days) of the following cyclic corrosion test (CCT) according to JASO M 610:

Salt spray (5% NaCl, 35°C, 2 h) → drying (60°C, 4 h) → wetting (50°C, 2h)

Fig. 17 shows its surface after the CCT together with comparative samples. A cold-rolled steel sheet without surface treatment and a galvanized steel sheet (coating weight 45 g/m²) were subjected to the chemical treatment and then, after confirming the formation of the chemical coating films, to the CCT as the comparative samples. **Fig. 18** shows the change of coating blister of the above three kinds of samples during the course of the CCT, in terms of blister width on one side of a crosscut line. The corrosion depths of these samples were measured in the thickness direction after 150 cycles of the CCT, at 5 points per sample, using a laser beam. The mean values and standard deviations are shown in **Fig. 19**. It is clear from these results that a steel sheet having coating layers of Fe-Al alloy phases is superior to a cold-rolled steel sheet and equivalent to a galvanized steel sheet in terms of coating blister width and corrosion depth. When especially good corrosion resistance after paint coating is required, it is desirable to increase the aluminizing coating weight to 150 g/m² or so.

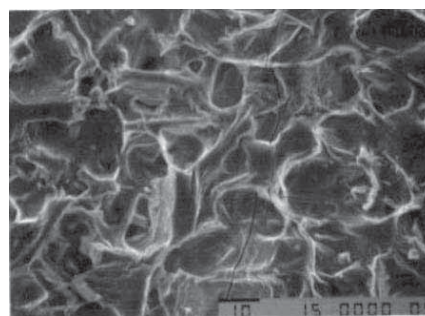


Fig. 16 SEM micrograph showing surface structure of plating layer after retention at 950°C for 5 min

Table 3 Evaluation of paint adhesion

	Surface preparation	Primary adhesion*	Secondary adhesion**
Hot-formed aluminized steel	Phosphated	100/100	100/100
Hot-formed aluminized steel	Degreased	100/100	100/100
Galvanized steel	Phosphated	100/100	100/100
Steel	Phosphated	100/100	100/100

*Primary adhesion test: 100 grid cross-cutting by 2mm and taping after painting

**Secondary test: dipping at 40°C water for 10 days and cross-cutting and taping

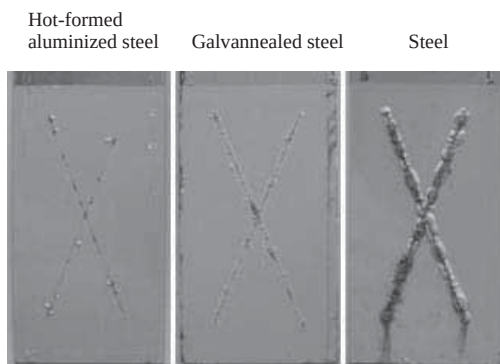


Fig. 17 Appearance of samples after 50 days of CCT according to JASO

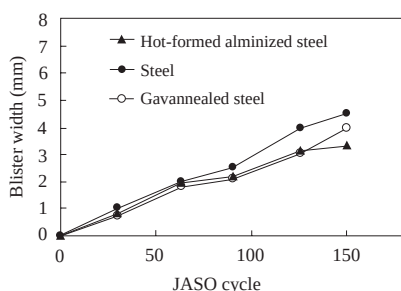


Fig. 18 Change of paint blister width during CCT according to JASO

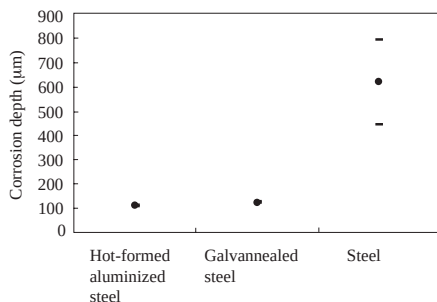


Fig. 19 Change of corrosion depth after 50 days of CCT according to JASO

6.3 Corrosion resistance of spot-welded portion

Fig. 20 shows a sectional structure of a portion just beneath a spot welding electrode and another outside a clamped portion. From these photomicrographs it is clear that the plating layer just beneath an electrode does not change in spite of the welding heat and retains its thickness throughout the welding process. For this reason, the corrosion resistance of these portions after paint coating is the same as that of the other portions. Fig. 21 shows samples of the aluminized steel sheet with paint coating after heat-treatment of hot-forming including spot welded portions after 2,000 h of a salt spray test (SST); no corrosion is observed in any of them. In the case of other coated steel sheet products, the plating layer beneath an electrode may melt during spot welding, be squeezed out from beneath the electrode, and the plating metal can sometimes form protrusions around the portion. At such portions, paint coating applied thereafter

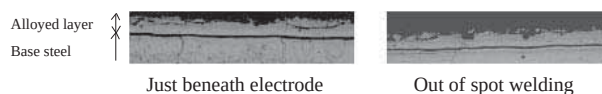


Fig. 20 Plating layers just beneath spot welding electrode and other portion (etched with bromic acid)

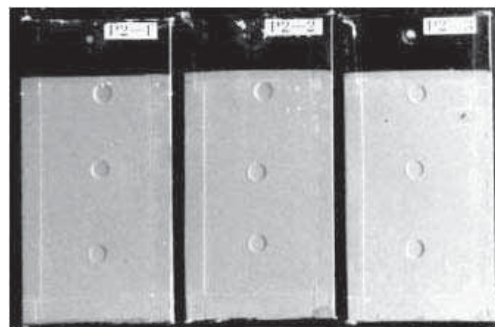


Fig. 21 Appearance of spot-welded and painted portions after SST for 2,000 h

may become thinner and there may be problems in corrosion resistance.

7. Summary

The properties of the base material and surface properties of an aluminized steel sheet for hot forming use have been explained herein. Even if the cooling rate at the die quenching is uneven during the press formation process, the base material has uniform strength after the hot forming as far as a cooling rate of 30°C/s or higher is attained. A yield strength of 1,280 MPa, a tensile strength of 1,530 MPa and an elongation of 8% can be obtained after the hot forming. The aluminum plating layer of the product transforms during the heating before the press forming into an Fe-Al alloy phase having a high melting point, and thanks to this, the spot weldability of the product is not adversely affected by the presence of coating layers. As a consequence, the consecutive spot weldability of the aluminized steel sheet product is excellent. The Fe-Al alloy phase displays a better corrosion resistance than a steel sheet without surface treatment and almost equivalent corrosion resistance to a galvannealed steel sheet.

The application of the steel sheet product having the above properties to the hot-forming process will make it possible to produce automobile components excellent in strength and corrosion resistance and having comparatively complicated shapes.

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Appendix SUEHIRO02



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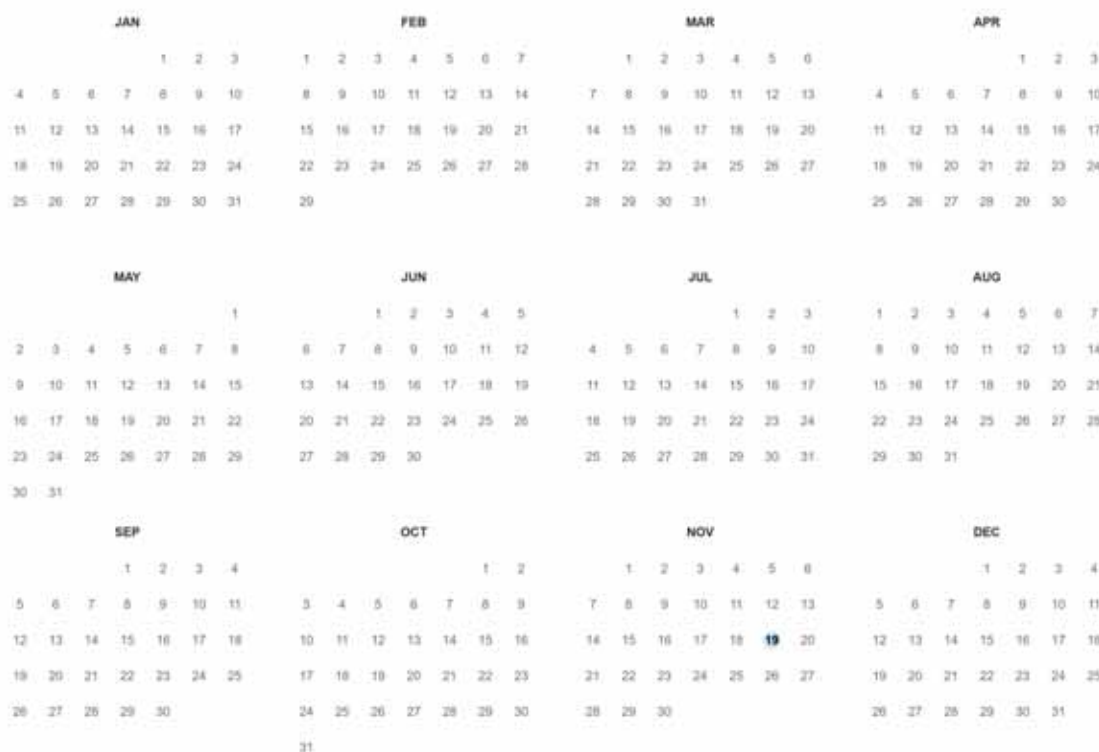
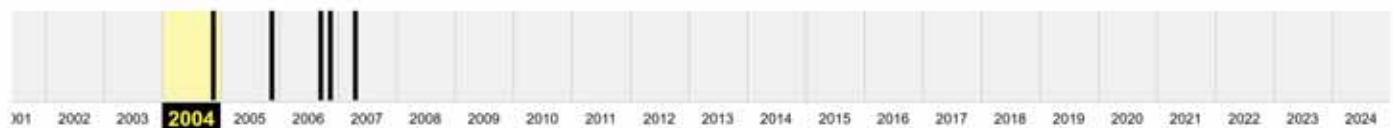
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ホットプレス用アルミニウムめっき鋼板の諸特性

Properties of Aluminum Coated Steels for Hot-forming

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抄 録

オーステナイト域の温度に加熱した鋼板をプレス成形すると同時に焼入れを行い高強度の部品を製作するホットプレスが注目されている。このホットプレスには加熱時の表面酸化とその後の耐食性確保にアルミニウムめっき鋼板が適していると考えられる^{*}。焼入れ性に優れた母材にアルミニウムめっきを施した鋼板をこのホットプレスに適用した際の材質特性、表面特性、スポット溶接性等に関し検討した。金型での焼入れ後に 500MPa 以上の強度となること、また、表面のめっき層は加熱により Fe-Al 合金層となり、JASO 法 CCT 試験による耐食性試験結果では合金化溶融亜鉛めっき鋼板と同等の耐食性を示すこと、スポット溶接性にも優れることが明らかとなった。^{*}(Anebr 社とのグローバル戦略提携契約に基づくライセンス契約商品 (US BGR 1500))

ホットプレス用アルミニウムめっき鋼板の諸特性

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Abstract

Production of parts with high strength by means of hot-forming process has been receiving considerable attention lately. Application of aluminized steel with good hardenability for this process has been investigated^{*}. Tensile strength over than 1500MPa was obtained with steel containing around 0.2mass% C. Surface coated layer changed from Al-10% Si to Fe-Al(-Si) phases through the hot-forming process. The steel with Fe-Al(-Si) surface layer showed good paintability even without phosphatized treatment. After painting, they showed good corrosion resistance in JASO-CCT as galvanized steel did. The steel showed good spot weldability due to the stable surface layer at high temperature. (^{*}license product within the frame of global strategic alliance with Arcelor (USIBOR1500))

1. 緒 言

近年、環境問題に端を発する自動車の軽量化や衝突時の安全性向上といった観点から高強度鋼板の使用が積極的に行われている。部材によってはより高強度の鋼板を用いることで鋼板厚みをより低減でき大きな軽量化を可能とできることから、用いられる鋼板強度も440MPaや590MPa級にとどまらず780MPa級以上へと上昇している。鋼板強度が増加するにつれ一般的には成形性が劣化する。これを克服するため各種組織制御を行った高強度鋼板が開発されてきた¹⁾。しかしながら、鋼板強度が1500MPa程度になると複雑な形状へのプレス成形は困難になると思われる。

1500MPa程度の強度を持つ部材を得るため欧州ではダイクエンチ、ホットフォーム、ホットスタンプあるいはホットプレスと呼ばれる成形法が実用化されている²⁾。この成形法では鋼板は900℃程度のオーステナイト域の温度に加熱され常温の金型で成形することにより焼入れ鋼板強度を高める。また、このような工法は国内においても検討されており、めっきを施していない鋼板を用い検討あるいは実用化されている^{3,4)}。また欧州においてはArcelor社がホットプレス特性と優れた耐食性を有するアルミニウムめっき(以下アルミ

めっき)製品USIBOR1500をすでに開発実用化している^{2,5,6)}。新日本製鐵は、Arcelor社とのグローバル戦略提携契約に基づき、USIBOR1500のライセンス契約を結び、商品の製造技術および製品特性の検討を進めてきた。本論文ではホットプレス用のアルミめっき鋼板の各種特性に関し検討した結果を報告する。

2. ホットプレス工程

ホットプレス工程は既に実用化されている。図1にその工程を簡単に示す。この製法では鋼板をオーステナイト域の温度(A_{c3} 以上)ま

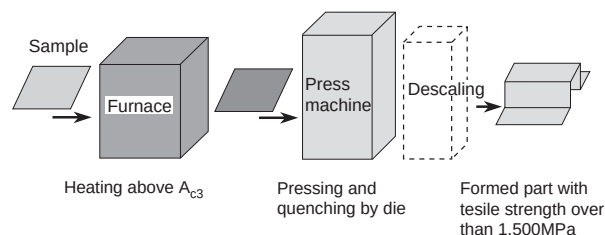


図1 ホットプレス工程の模式図

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^{*(3)} 八幡製鉄所 薄板部 マネジャー

で加熱することが必要である。例えば0.2mass%程度のC量を含有する鋼では850℃以上の温度に加熱することが必要となる。加熱後、炉から取り出された鋼板はプレス機まで搬送され常温の金型で成形し同時に焼入れが行われる。成形後鋼板全体が充分焼入れされるまで下死点で保持する。

このような成形工程をとるが、めっきを施していない鋼板を使用する場合には加熱時のスケール生成や表層脱炭を抑えるため非酸化雰囲気とする必要がある。また、非酸化雰囲気中で加熱した場合でも炉からプレス機までの間は大気雰囲気となるため短時間でも表層にスケールが生成する。プレス成形品は使用時には塗装が付与されるが表層にスケールが存在すると問題となる。したがって、めっきを施していない鋼板を使用した場合にはスケールを除去するプロセスが必要となる。スケール層の除去はショットブラストや酸洗が可能であるがこれらはコスト上昇を招く。また、ショットブラストを用いた場合には形状変化を生じる懸念がある。耐熱性に優れためっき鋼板を用いるとこの工程が不要となる。

3. 素 材

前述したようにホットプレス工程では鋼板を急速に冷却することで強度を高めるため焼入れ性に優れることが必要となる。表面に関しては耐熱性に優れためっきを施しておくことで前述した加熱炉の雰囲気制御やスケール除去のためのプロセスが不要となり、製造を容易にすることができる。以上のような条件を満足する鋼板として、表1に示すような成分系のアルミニウムめっき鋼板があげられる。なおめっき付着量は120～160g/m²のものをを用いた。この鋼板の各種特性を以下に概説する。

4. 母材特性

4.1 焼入れ前後の引張特性

本鋼板の焼入れ前後の特性を表2に示す。引張試験は1.4mmの厚みの素材からJIS5号引張試験片を作製し行った。出荷時の強度は490～590MPa級であり、金型での焼入れ後は1 500MPa以上となる。なお強度は図2に示すようにC量で調整することが可能である。図2は実験室的にC量を変化させた素材を真空熔解で溶製し焼入れ後の機械的特性を調査した結果である。これまでに知られているように焼入れ後の機械的特性はC量とともに増加することがわかる。焼入れ後の強度、つまり成形部品の強度はC量を調整することで制御可能である。なお、焼入れ時の強度に対するMn、Cr等の置換型固溶元素の影響は小さいことが知られている。これら置換型固溶元素は焼入れ性に影響するため、次章で述べる焼入れ性を確保するためには重要となる。ただし、これら元素を過剰に添加すると製品板の強度が高くなることを考慮しておく必要がある。

表1 検討に用いた鋼板の成分(mass%)

C	Mn	Cr	B
0.22	1.2	0.15	0.002

表2 焼入れ前後の引張特性

	Yield strength (MPa)	Tensile strength (MPa)	Elongation (%)
As coated	394	615	26
As formed	1,280	1,530	8

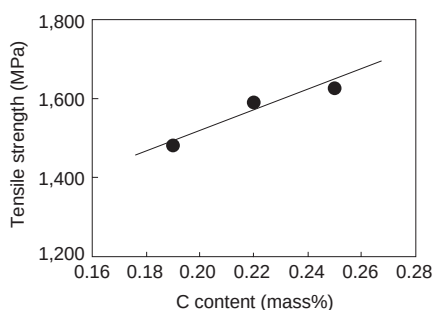


図2 C量と引張特性の関係

4.2 焼入れ性

図3にこの鋼のCCT曲線(950℃からの連続冷却曲線)を、図4に冷却速度と硬さの関係を示す。この鋼板は焼入れ性を高めるためMnやBが添加されており、加熱温度が950℃の場合約20℃/s以上の冷却速度で硬度をHV450～500の範囲とすることができる。この焼入れ速度は冷却中でのフェライト、パーライトおよびベイナイトへの変態を避ける条件で決まる。これらの変態はオーステナイト粒径が小さいと、つまり加熱温度が低いと速くなるため、焼入れに必要な冷却速度が速くなる。この点については加熱温度を900～1 000℃として検討を行ったが、この範囲ではあまり大きな差はなくどの加熱温度においても20～30℃/s程度に臨界冷却速度があった。

図3および図4および前述した他の加熱温度での実験結果からホットプレス時の鋼板の冷却速度が30℃/s以上であればどのような冷却速度をとっても十分な硬さが得られることがわかる。しかしながら、実際のプレス工程では加熱炉からプレス機への搬送とプレス開始までの間は空冷に近い冷却速度となる。

1.4mm程度の厚みの鋼板では大気雰囲気中で冷却すると10～15℃/s

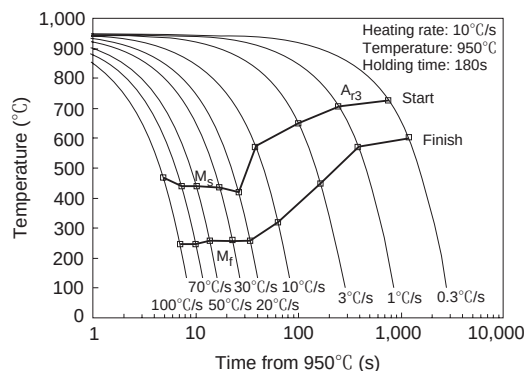


図3 CCT曲線

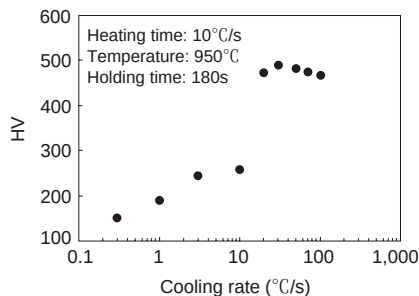


図4 硬さにおよぼす冷却速度の影響

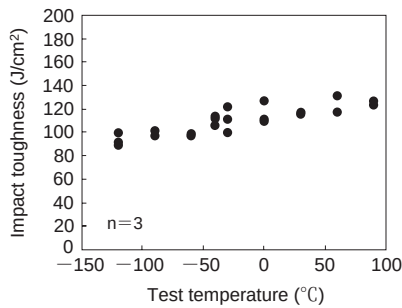


図5 シャルピー試験時の衝撃値と温度の関係

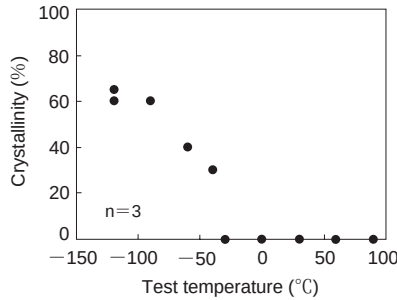


図6 シャルピー試験時の脆性破面率と温度の関係

の冷却速度となるが、この冷却速度で600°C以下まで冷却されると鋼板の強度が低下することが図3および図4からわかる。950°Cから600°Cまでの時間は23～35秒となり素材を炉から取り出してから23秒以内に金型での冷却を開始すれば素材を焼入れることが可能となる。なお、この関係は加熱条件、冷却中の雰囲気や周りの環境により変化するため、実際のホットプレス作業環境に応じ決定する必要がある。

4.3 靱性

焼入れによりマルテンサイト相で強度を確保した場合には靱性、特に低温での靱性が劣化する懸念がある。この点に関しシャルピー衝撃試験にて検討した結果を図5および図6に示す。図5は衝撃値と温度の関係を示す。この試験は4mmの熱間圧延板を水焼入れ後に2mmUノッチ2.5mmサブサイズ衝撃試験片を作製して行った。吸収エネルギーは室温から-60°Cまであまり大きな変化を示さない。一方、脆性破面率は-40°C以下で増加し、-80°Cでは約80%となり、遷移温度(Tr_{550})は-60°C程度である。脆性破面率は板厚が薄くなると低減する傾向にあり、2.5mmより薄い板厚の場合には遷移温度はより低下するものと考えられる。以上の結果から、この鋼板は焼入れのままのマルテンサイト相を主体とする組織で使用するが、通常的环境下で低温になって急激に吸収エネルギーが変化したり破壊形態が変化することはないものと推定されることがわかる。

5. スポット溶接性

めっき付着量を120g/m²とする板厚1.2mmのアルミニウムめっき鋼板を950°Cで5分加熱し、金型で両側から挟み込むことで焼入れを行いスポット溶接用サンプルとした。表層のめっき層の変化に関しては次章で説明するが、Fe-Al合金めっきとなっている。相手材としては板厚1.8mmの440MPa級の冷間圧延薄鋼板を用いた。スポット溶接は、DR6φ型のアルミナ分散銅を電極とし、通電を17サイクル

(60Hz)、加圧を400kgfとして行った。図7にナゲット径と電流密度の関係を示すがナゲット径が板厚を t として $4\sqrt{t}$ となる電流値とちりが発生する電流値の範囲は約3kA存在することがわかる。図8はせん断引張強度と電流値の関係を示す。図9は十字引張強度と電流値の関係を示す。図10にナゲット近傍の硬さ分布を示すが、ナゲット内および母材部は約480のビッカース硬さを持ち溶接熱影響部(HAZ)にて300程度に軟化する。このような硬さ分布を持つため、ナゲット径が充分な大きさを持つ場合破断部はこのHAZとなることが多い。

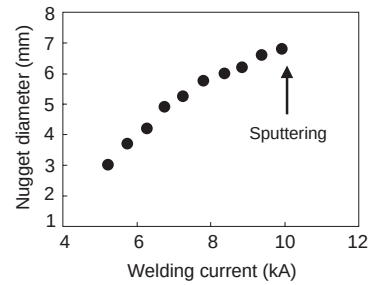


図7 スポット溶接部のナゲット径と電流値の関係

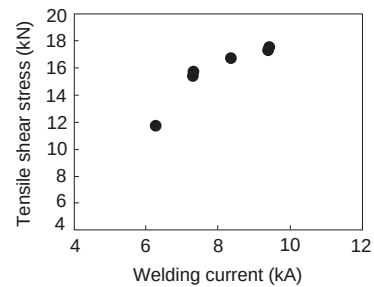


図8 スポット溶接部のせん断引張強度と電流値の関係

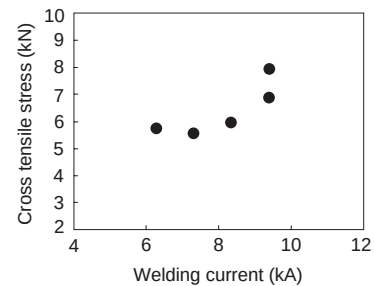


図9 スポット溶接部の十字引張強度と電流値の関係

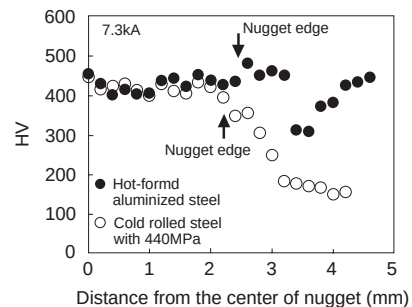


図10 ナゲット近傍の硬さ分布

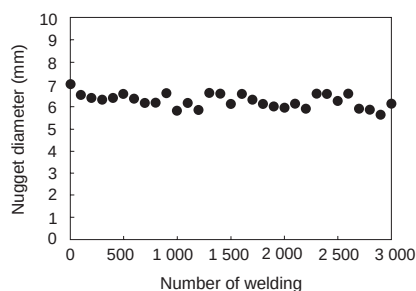


図11 連続打点によるナゲット径の変化

図11は連続打点性を評価した結果である。試験は3000点まで行ったが、この範囲ではナゲット径の変化はあまりなく3000点以上の連続打点も可能であると推定される。この銅板の表層のめっき層は融点が1000℃以上のFe-Al合金相となっているが、このことがこのような連続打点性を持つ理由と考えられる。

ここではアーク溶接性に関しては示していないが、Fe-Al合金相は沸点が2000℃以上であり、亜鉛系のめっき銅板で問題となるアーク溶接時のブローホールの発生は無い。

6. めっき特性

6.1 加熱時のめっき層の合金化

本銅板の表層めっきはSiを10%含有するアルミめっきであり、Type 1と呼ばれているものである。このめっき層の溶融温度は約600℃であるため急速に加熱するとめっき層は一旦溶融するが、電気炉で得られる加熱速度では加熱中に融点の高いFe-Al合金層が生成し、速やかに表層まで達するため溶融物が炉材等に付着することはない。大気雰囲気中の電気炉で板厚1.6mmの素材を加熱した場合、保温度に達するまでの時間は約2分であった。

図12に950℃で0.5分間保定した際の合金化の状況を、図13は表層のめっき層のFe、AlおよびSi濃度を板厚方向にSEM-EDSで測定した結果を示す。合金化が進行すると図に示すようなAlおよびFe濃度を持つ層状の合金相が形成される。この加熱条件下では合金相は5層からなり、Al濃度は最表層から順に約50%、30%、50%、30%、10%と変化している。4層目から母層にかけては元素濃度が連続的に変化している。

図14にThermo-calc⁷⁾により計算したFe-Al-Si系の950℃での状態図を示す。この図に図13で示した各点の組成を重ねた。なおこの計算のFe-Al-Si系熱力学データベースはZ. Liu⁸⁾によるものを使用した。図14から合金層は表層から Fe_2Al_3 、BCC規則相、 FeAl_2 、BCC規

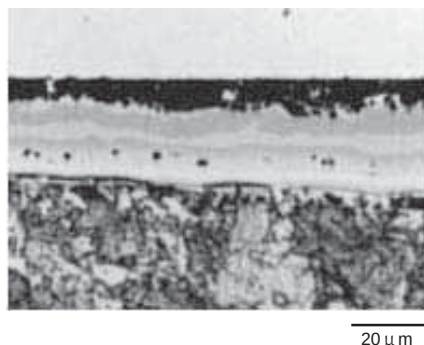


図12 950℃で30秒保定した試料のめっき層構造を示す断面光学顕微鏡写真

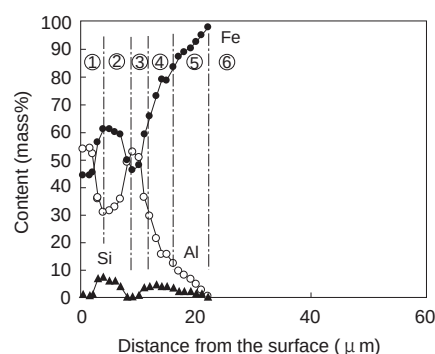


図13 図12に対応したサンプルの板厚方向のFe、Al、Si元素濃度変化

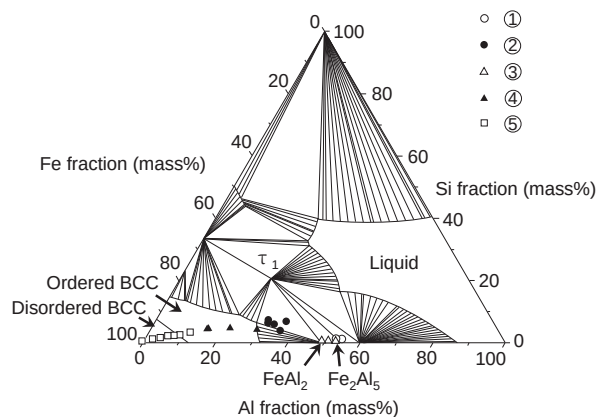


図14 Fe-Al-Si平衡状態図の950℃での等温断面図

則相、BCC不規則相と推定される。表層の合金層は保定時間が長くなるとAlが母層へと拡散していくため最表層から消滅していく。図15は保定時間を5分としたときのめっき層を示す。断面組織からは1相しか識別できなくなっているが、SEM-EDSで分析すると2相が存在していることがわかった。このときの相はBCC規則相と不規則相と考えられる。

6.2 塗装性および塗装後耐食性

銅板は自動車への適用に際して電着塗装されて使用されるが、通常、塗装性を確保するため塗装前に化成処理が施される。化成処理は一般にりん酸亜鉛系の溶液で行われ、表面にりん酸塩の皮膜を形成させる。この皮膜により塗装性、塗料密着性が確保されるが、Fe-Al合金相は化成処理液に浸漬してもこの化成皮膜が形成されない。これはFe-Al合金相が酸に対して非常に安定な相であるためである。しかしながら図16でわかるように加熱処理後の表面は凹凸が

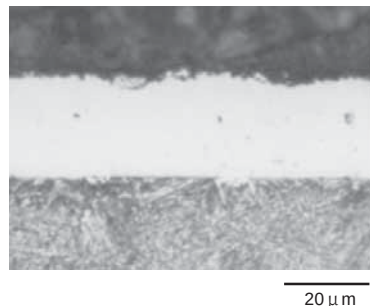


図15 950℃で5分保定後のめっき層構造を示す断面光学顕微鏡写真

ありこれによるアンカー効果で塗装性が確保される。表3は化成処理を施した試料及び施さない試料に約20 μ m厚みの電着塗装を行ない、塗装後の密着性を検討した結果であるが、十分な特性を確保していることがわかる。このように塗料密着性は化成処理の有無にかかわらず確保可能である。

めっき付着量を160g/m²とするアルミめっき鋼板を900℃で2分加熱することで表層をFe-Al合金めっきとした鋼板を化成処理液に浸漬し、約25 μ mの電着塗装後、表面の塗膜にクロス状の疵(クロスカット)を入れJASOM610に定めた複合腐食試験(CCT)を150サイクル(50日間)行った後の外観を図17に示す。JASO法CCTの試験条件を以下に示す。

塩水噴霧(5%NaCl, 35℃, 2h) → 乾燥(60℃, 4h) → 湿潤(50℃, 2h)

比較材としてめっきを施していない冷間圧延鋼板および合金化溶融亜鉛めっき鋼板(付着量片面45g/m²)を使用した。これらの材料は化成処理を施し、化成皮膜が生成していることを確認した後に試験に供した。腐食試験の途中でクロスカット部からの塗膜膨れ幅を片側最大膨れ幅として評価した結果を図18に、また150サイクル後の板厚方向への腐食深さを表面からレーザー光により1試料当たり各

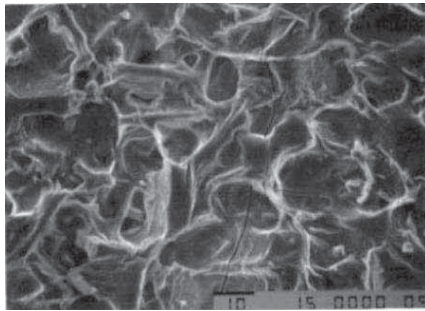


図16 950℃で5分保定後のめっき層表面形態を示すSEM写真

表3 塗料密着性評価結果

	Surface preparation	Primary adhesion*	Secondary adhesion**
Hot-formed aluminized steel	Phosphated	100/100	100/100
Hot-formed aluminized steel	Degreased	100/100	100/100
Galvannealed steel	Phosphated	100/100	100/100
Steel	Phosphated	100/100	100/100

*Primary adhesion test: 100 grid cross-cutting by 2mm and taping after painting

**Secondary test: dipping at 40℃ water for 10 days and cross-cutting and taping

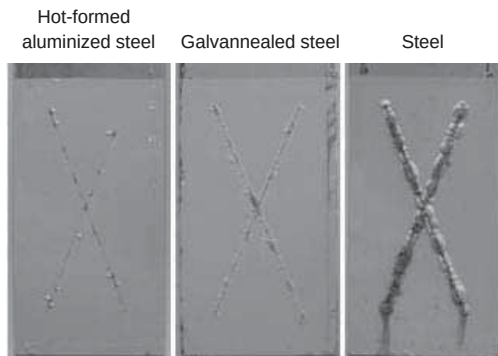


図17 JASO法CCT試験50日後の試料外観

5点づつ測定して平均値並びに標準偏差を算出した結果を図19に示す。これらの結果から、Fe-Al合金相をめっき層とする鋼板の耐食性は剥離幅、腐食深さとも冷間圧延鋼板より優れ、合金化溶融亜鉛めっき鋼板と同等の特性を持つ事がわかる。塗装後耐食性を重視する場合にはめっき付着量を150g/m²程度とすることが望ましい。

6.3 スポット溶接部の耐食性

図20はスポット溶接時に電極と接していた部分および電極と接触していなかった部分の断面組織を示す。この図から、電極と接していた部分のめっき層はスポット溶接時に温度が上昇してもほとんど変化せずスポット溶接前と同様の厚みを有していることがわかる。そのためこの部分の塗装後耐食性は平板部と全く変わらず、図21に示すように塩水噴霧試験(SST)にて2000時間経過後においても全く

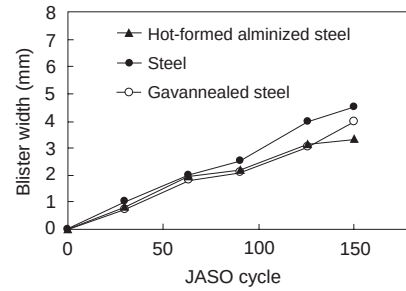


図18 JASO法CCT試験による塗膜膨れ幅の変化

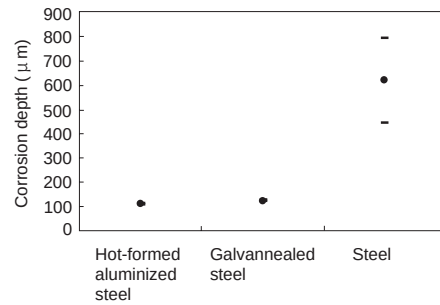


図19 JASO法CCT試験50日後の板厚方向腐食深さの変化

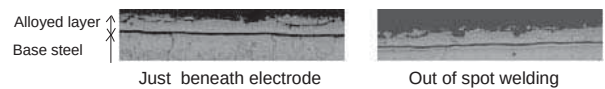


図20 スポット溶接後の電極直下および通常部のめっき層(腐食液:しゅう酸)

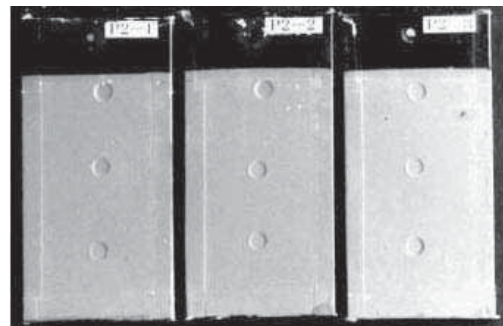


図21 スポット溶接部の塗装後耐食性のSSTによる評価, 2000時間後の外観

腐食していないことがわかる。他のめっき鋼板ではスポット溶接を行った際、電極と接していた部分のめっきは溶融し電極と母材の間から押出されめっき金属が盛り上がり突起状になることがあるが、この部分は十分な塗装厚みが確保できず耐食性に問題が生じることがある。

7. 結 言

本報告ではホットプレス用アルミめっき鋼板の母材特性および表面特性について述べた。母材特性に関しては、プレス成形時の金型焼入れで冷却速度がばらついても30℃/s以上の冷却速度が確保できれば均一な強度を有する部材が得られること、引張特性に関しては降伏強度：1280MPa、引張強度：1530MPa、伸び：8%が得られること、耐水素脆性にすぐれること等を述べた。スポット溶接性に関しては、表層のアルミめっきがプレス前の加熱で融点の高いFe-Al合金相となることに起因してめっき層の存在による劣化はないことや連続打点性に優れることを示した。また耐食性に関しては、Fe-Al合金相はめっきを施していない鋼板と合金化溶融亜鉛めっき鋼板

の間に位置することを示した。

以上のような特徴を持つ鋼板をホットプレスに適用することで、比較的複雑な形状を持つ高強度で耐食性に優れた部材の製造が可能となる。

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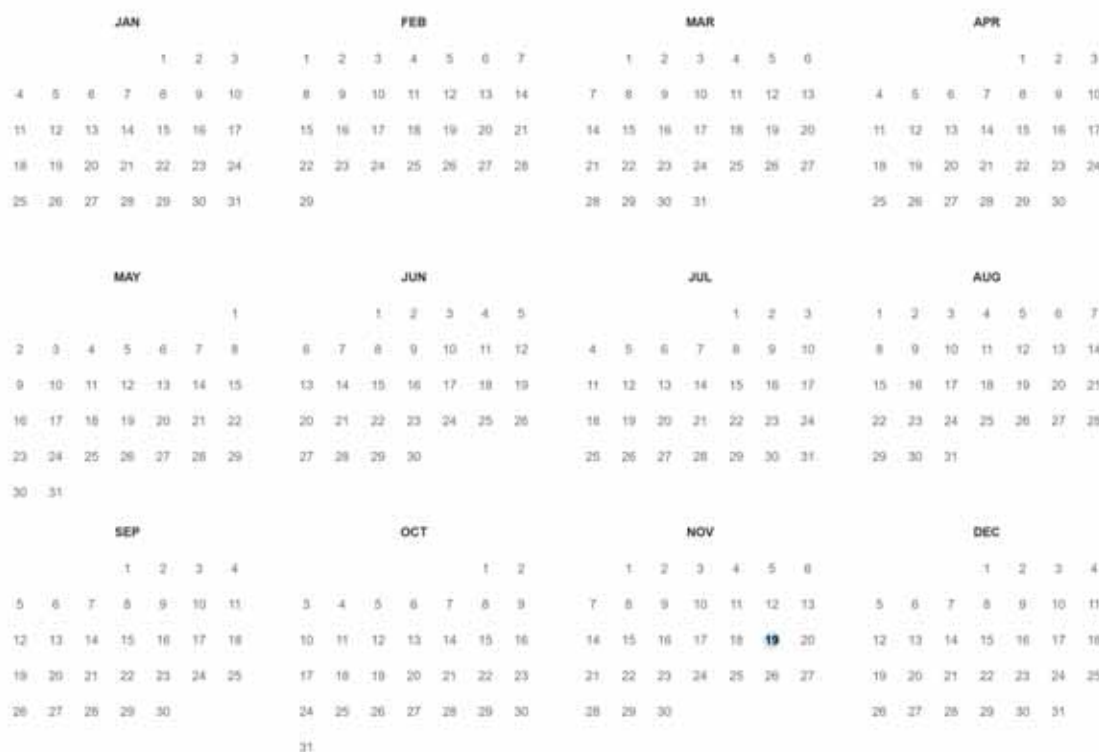
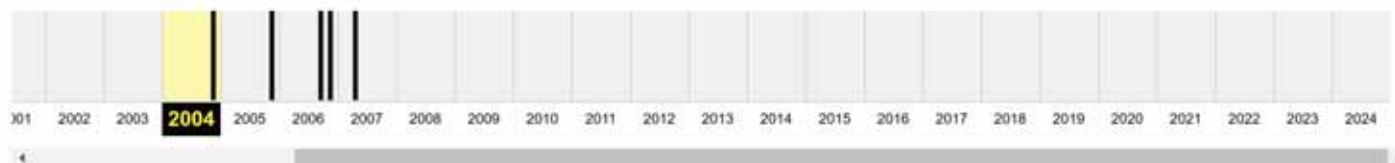
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