

Rationalisation of Vicat Softening Point Measurements

S. P. Namhata, L. Santolini & G. Locati

EniChem Tecnoresine, Centro Tecnologico, Via Iannozzi 1,
20097 San Donato Milanese, Milano, Italy

(Received 1 June 1989; accepted 9 July 1989)

ABSTRACT

The utility of the Vicat softening point is rationalised one step further. Light is thrown on Vicat measurements and they are interpreted from a qualitative viewpoint; it is stated that the Vicat softening point is nothing but a temperature at which the modulus has reached a particular level. So the Vicat softening point is not just an arbitrary point measurement but a well-defined point in the modulus–temperature diagram, being related to the fundamental properties of the material. This interpretation enables one to understand and control the thermal behavior of polymers, especially in developments of new blends where a target Vicat softening point is often pre-specified.

The validity of the above statement is established through experimental work. The quantitative values of modulus levels related to different Vicat measurements (Vicat A and B) are estimated. The Vicat values generally supplied in material literature can now be used for design purposes and for other critical structural calculations which are nowadays a common practice for the plastics designers.

1 INTRODUCTION

In the characterisation of polymeric materials the Vicat softening point determination holds a very important position as a measure of the upper limit of usable temperature for a short period. In all existing standards of polymer testing, e.g. ASTM, DIN and ISO, there is

75

Polymer Testing 0142-9418/90/\$03.50 © 1990 Elsevier Science Publishers Ltd, England.
Printed in Northern Ireland

provision for this type of measurement. Inclusion of Vicat values in all technical publications indicates their universal acceptability. But, as a long time has passed, we feel that these types of measurements should be reassessed from the viewpoint of modern requirements.

For all practical purposes, the Vicat softening point is believed to be a value close to the glass transition temperature of the amorphous polymer; hence sometimes its determination is referred to as a practical method to make an estimate of the glass transition temperature, or sometimes the Vicat values are predicted from the T_g values of the materials.

The above view is correct if we consider only simple polymeric systems. But the situation is not so simple if we consider materials such as reinforced (filled) polymer systems or polymer blends, where the Vicat softening point cannot be interpreted just as a value close to the value of the T_g of the material. Indeed on many occasions, as for example in the case of semi-compatible or incompatible blends, there exist two T_g values instead of a single one. In these cases it is difficult to predict the Vicat softening point just by knowing the value of T_g because the Vicat may be close to either one of the two T_g values or intermediate between them. Changing the concentration of the components may also change the Vicat values significantly.

Considering all these facts one can realise that a clearer interpretation of the Vicat value is necessary which would be able to explain and predict the Vicat values for polymers irrespective of their type. Particularly, such an interpretation for the Vicat is very important in order to have a better control over the work of developing new material by blending or filling where the goal is pre-specified.

Our present objective was to establish such a rationalisation of the Vicat test. We have shown that a Vicat value can be better explained if we consider it as a temperature at which the modulus of the material reaches a particular value.

2 EXPERIMENTAL

For our work we used different types of polymeric materials:

- | | |
|----------------------------|---|
| (1) Simple | Polycarbonate
Poly(ethylene terephthalate)
Polyamide 6 |
| (2) Reinforced
(filled) | Polyamide 6 (reinforced with glass fibre)
Polyamide 6 (reinforced with mineral filler) |

- (3) Blends Polycarbonate and ABS
 Polycarbonate and poly(ethylene
 terephthalate)
 A commercial material called Bayblend T45

All the experimental blends were prepared by melt blending (except Bayblend T45) in a twin-screw laboratory extruder.

Prepared blends and the other polymeric materials were all injection-moulded under optimum conditions. These moulded pieces were then subjected to evaluation without any other conditioning.

2.1 Evaluation

In our study we performed two types of evaluation.

- (1) Measurements of Vicat softening point using an instrument manufactured by an Italian Company, ATS.
- (2) Dynamic-mechanical measurements (real part of torsional shear modulus as a function of temperature) using a rheometer model RDS-II manufactured by Rheometrics Inc., USA.

For one set of materials we measured Vicat A values (1 kg load and heating rate 50 °C/h) and for another set Vicat B (1 kg load and heating rate 120 °C/h). These measurement conditions were in accordance with ASTM D1525. All the observed Vicat values are tabulated in Tables 1 and 2. Dynamic-mechanical measurements were performed on bars of dimension 50 mm × 12 mm × 3 mm. The conditions of testing were:

- (1) Heating rate 120 °C/h.
- (2) Frequency 1 Hz.
- (3) Oscillation angle 5×10^{-3} rad.

All the curves generated from this evaluation are shown in Figs 1A, 1B, 1C, 1D and 1E. In the figures, G' values are in Pa.

3 RESULTS AND DISCUSSION

To establish a correlation of the Vicat softening point with the fundamental properties of materials, one has to realise more clearly the significance of the Vicat softening test.

The test is actually a measure of penetration—that is a measurement of a particular deformation under a constant load. So the Vicat softening temperature is a temperature at which a particular prefixed

TABLE 1
 Vicat A Values of the Materials
 ASTM Standard D1525: 1 kg, 50 °C/h.

<i>Sample code</i>	<i>Source</i>	<i>Vicat A value (°C)</i>	<i>Composition</i>
Sample 203	Experimental ^a	135.5	PC and ABS
Sample 204	Experimental ^a	137.5	PC and ABS
Sample 205	Experimental ^a	138.5	PC and ABS
Sample 206	Experimental ^a	133.5	PC and ABS
Sample 207	Experimental ^a	134.0	PC and ABS
Sample 211	Experimental ^a	119.0	PC and ABS
Sample 212	Experimental ^a	106.0	PC and ABS
Sample 215	Experimental ^a	137.5	PC and ABS
Bayblend T45	Commercial	126.0	PC and ABS

^a Prepared in EniChem Tecnoresine Laboratory.

TABLE 2
 Vicat B Values of the Materials
 ASTM Standard D1525: 1 kg, 120 °C/h.

<i>Sample code</i>	<i>Source</i>	<i>Vicat B value (°C)</i>	<i>Composition</i>
PET (amorphous)	Enichem	80.0	Pure PET
Sinvet 251	Enichem	152.0	Pure PC
PP1	Experimental ^a	149.5	PC and PET
PP2	Experimental ^a	148.5	PC and PET
PP3	Experimental ^a	146.5	PC and PET
PP4	Experimental ^a	120.5	PC and PET
PP5	Experimental ^a	92.0	PC and PET
PP8	Experimental ^a	150.0	PC and PET
PP9	Experimental ^a	151.5	PC and PET
Nivionplast 273MR	Enichem	209.0	Pure PA6
Nivionplast R35GS	Enichem	211.0	Glass-filled PA6
Nivionplast R30GR	Enichem	216.0	Glass-filled PA6
Nivionplast C35M	Enichem	216.0	Mineral-filled PA6

^a Prepared in EniChem Tecnoresine Laboratory.

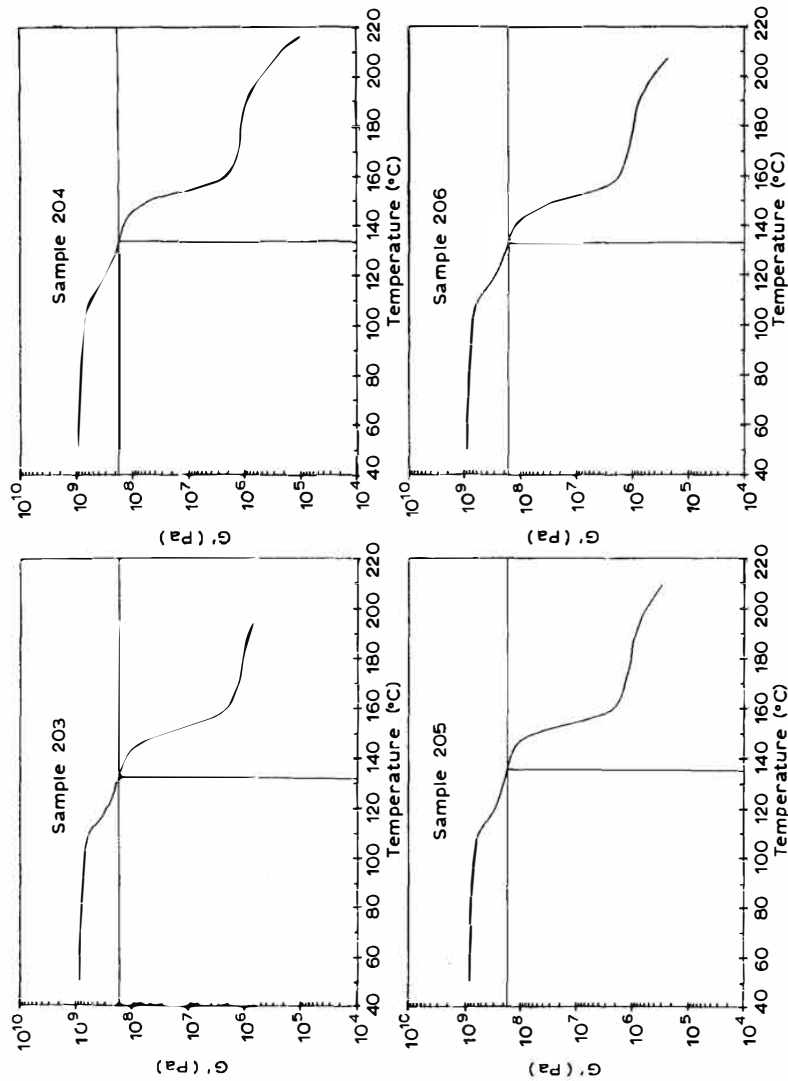


Fig. 1A. Typical G' versus temperature curves for some polycarbonate-ABS blends. (See Table 1.)

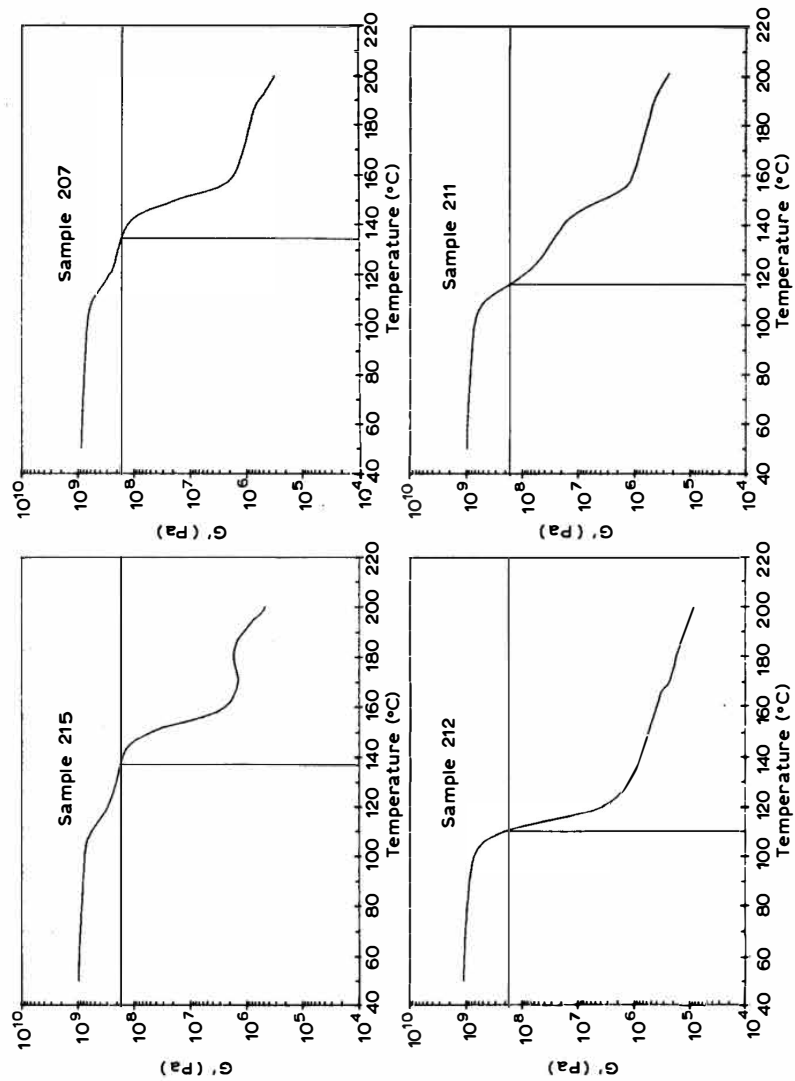


Fig. 1B. Typical G' versus temperature curves for some polycarbonate-ABS blends. (See Table 1.)

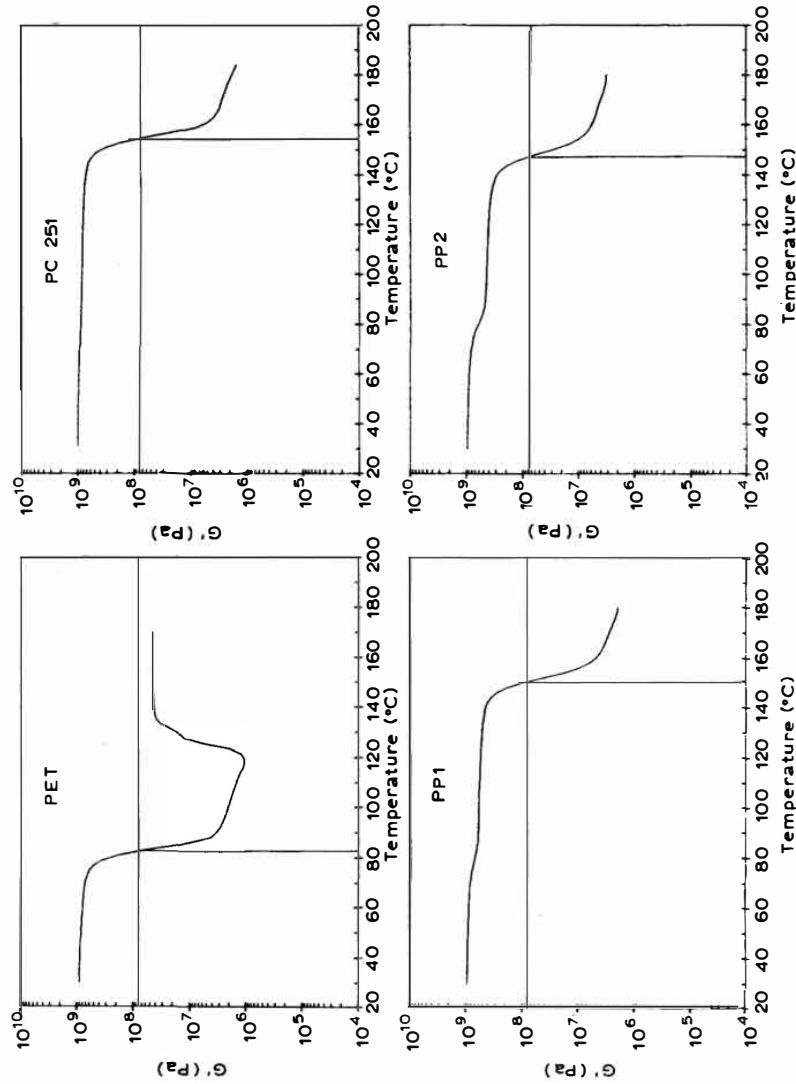


Fig. 1C. Typical G' versus temperature curves for polycarbonate, polyethyleneterephthalate, and for their blends. (See Table 2).

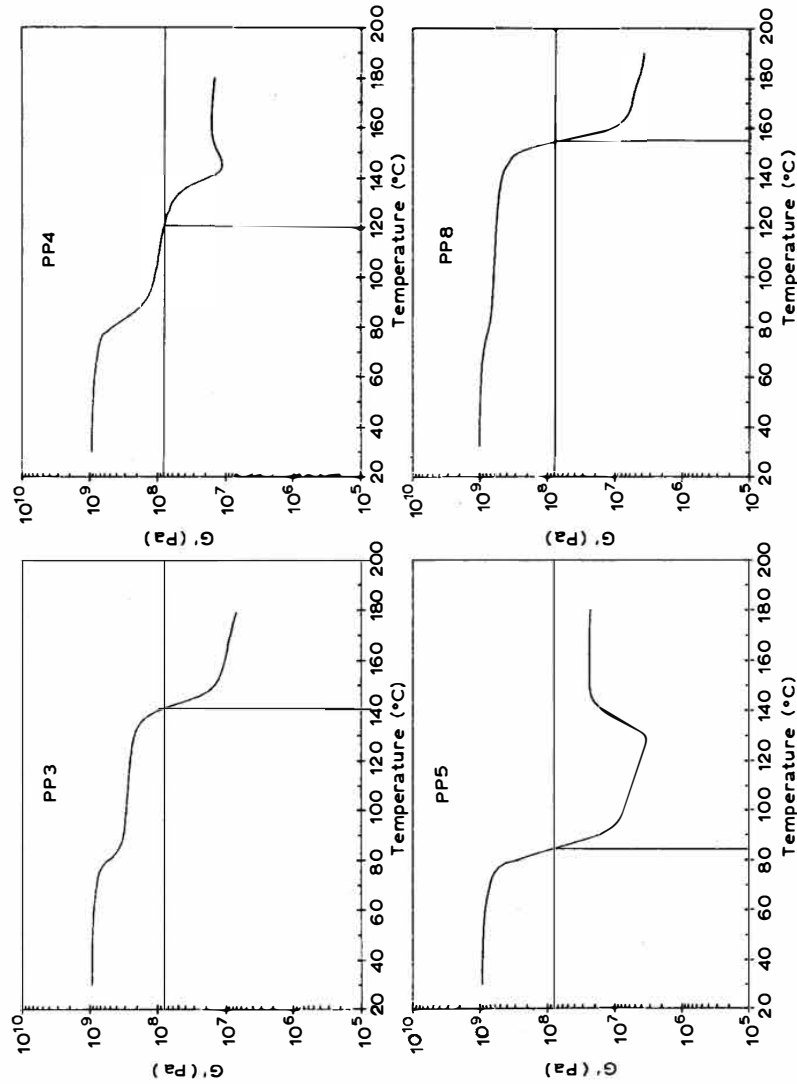


Fig. 1D. Typical G' versus temperature curves for some polycarbonate-polyethyleneterephthalate blends. (See Table 2.)

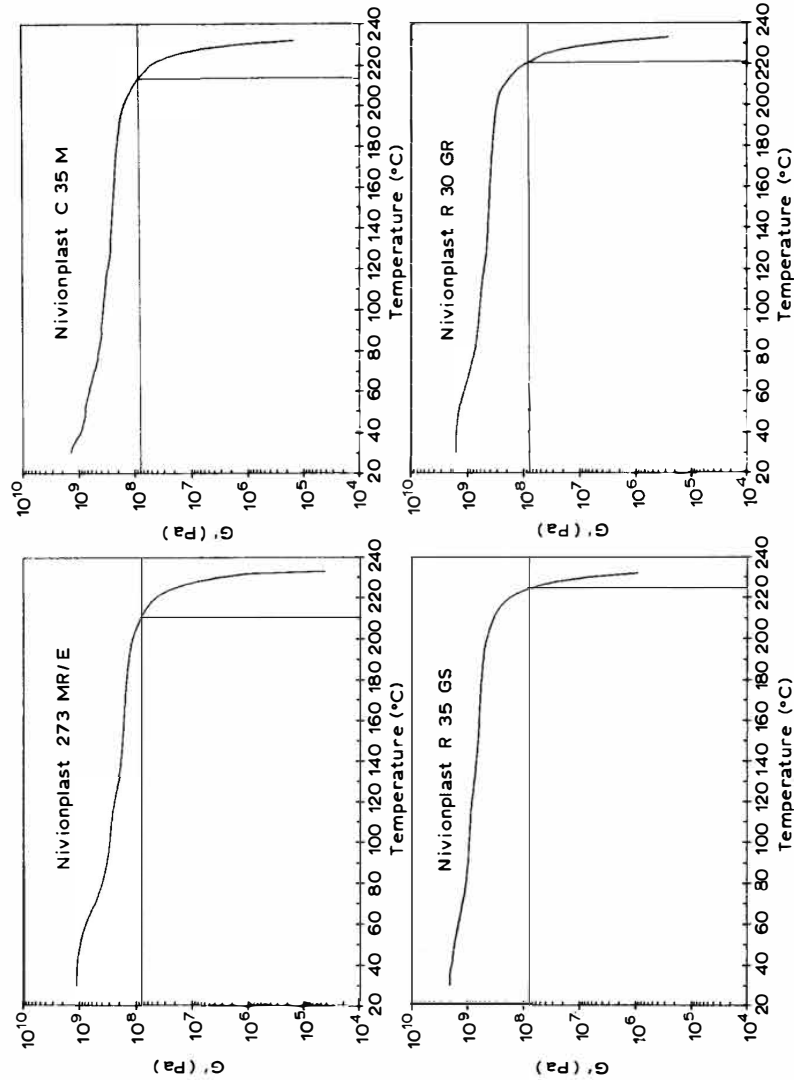


Fig. 1E. Typical G' versus temperature curves for some unfilled and reinforced polyamide-6 materials. (See Table 2.)

deformation is reached under a particular load. So we can simply think of it as the temperature at which the modulus of the material reaches a definite value.

If the above interpretation of Vicat measurement is valid then it should be true for all types of polymeric material, irrespective of their nature.

Guided by this simple interpretation we tried to find out the value of modulus responsible for the Vicat softening point.

From dynamic-mechanical tests, curves of G' as a function of temperature were generated for all the materials as described in Section 2 (Figs 1A, 1B, 1C, 1D and 1E), and the Vicat values are tabulated in Tables 1 and 2.

We have found that the temperature at which G' reaches 1.7×10^8 Pa correlated well with Vicat A values, whereas the temperature at which G' reaches 8×10^7 Pa correlated well with Vicat B values. These correlations are shown respectively in Figs 2A and 2B. They are linear with slope 1, hold good over a wide range of temperature from 70 to 200 °C and are true for a large variety of materials.

It is even more interesting to try to analyse the behaviour of PC/PET

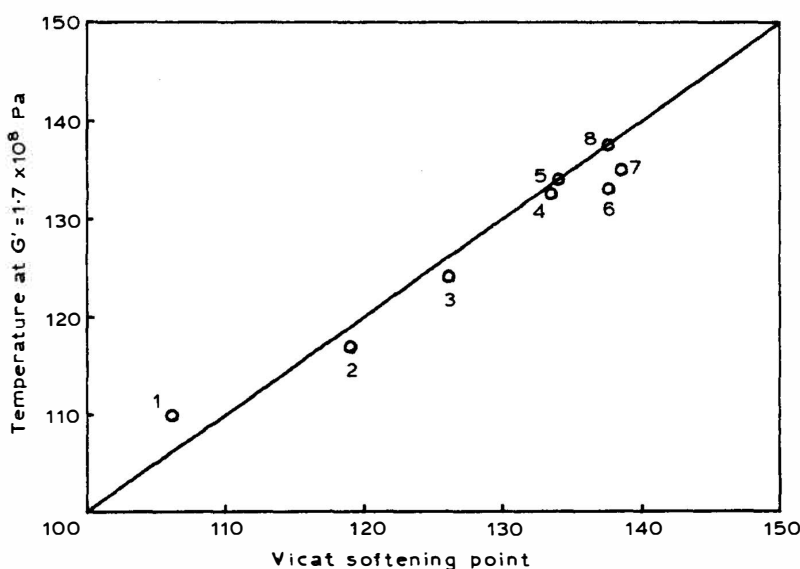


Fig. 2A. Correlation of Vicat softening point (Vicat A) with shear modulus (G'). Heating rate in dynamic mechanical experiment, 120° C/h; Vicat conditions, 1 kg, 50 °C/h. 1, Sample 212; 2, Sample 211; 3, Bayblend T45; 4, Sample 206; 5, Sample 207; 6, Sample 204; 7, Sample 205; 8 Sample 215.

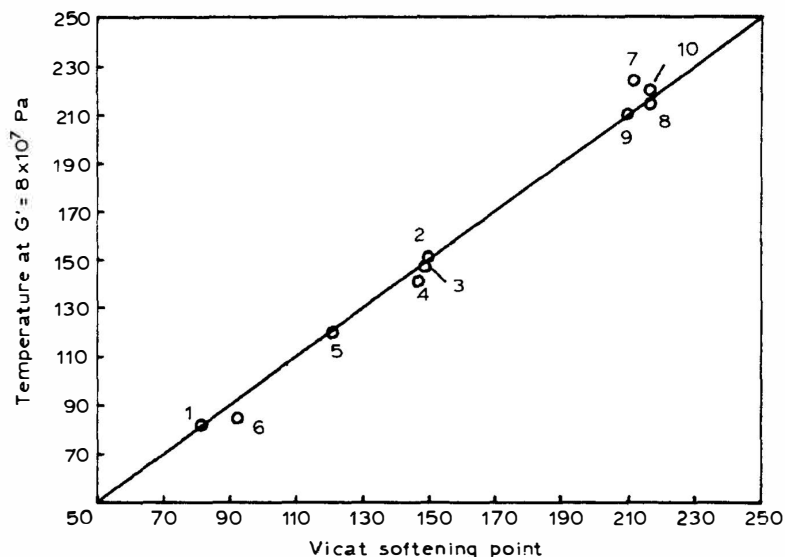


Fig. 2B. Correlation of Vicat softening point (Vicat B) with shear modulus (G'). Heating rate in dynamic mechanical experiment, 120°C/h; Vicat conditions, 1 kg, 120°C/h. 1, Amorphous PET; 2, PP1; 3, PP2; 4, PP3; 5, PP4; 6, PP5; 7, Nivionplast R35GS; 8, Nivionplast C35M; 9, Nivionplast 273MR; 10, Nivionplast R30GR.

blends in the light of the above discussion. The PC/PET blends (PP1, PP2, PP3, PP4, PP5) in the experiment were two-phase partially miscible blends, as observed from the existence of two T_g values and a significant shift of T_g near the polycarbonate T_g .

When the Vicat values of these blends are plotted as a function of composition, a sigmoidal curve (Fig. 3) is generated. This behaviour, particularly the large variation in Vicat values while passing from PP3 (50% PET) to PP4 (65% PET) with little change in concentration, could not be explained satisfactorily from the T_g values of PC and PET. But by taking the above-mentioned modulus value as the determining factor this sigmoidal behaviour could be clearly explained, as shown in Fig. 4.

So, from the above discussion we see that the Vicat softening point can be better understood by consideration of a particular level of modulus. The implication of this simple interpretation is really far-reaching.

In our opinion this information about Vicat can play a very important role in the work of developing blends where the thermal specifications are pre-fixed. It will be clearer if we take an ideal example, which may even arise sometimes in the practical work of blend development.

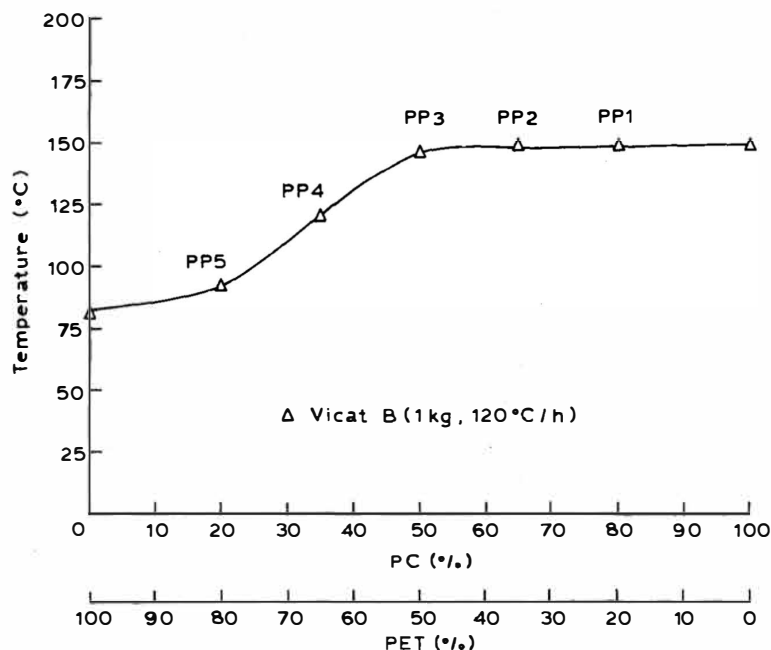


Fig. 3. Vicat values as a function of composition for polycarbonate-polyethyleneterephthalate blends.

In Fig. 5 the moduli of two polymeric materials A and B, having Vicat softening points T_A and T_B , are plotted as a function of temperature. The goal to be reached by blending the two polymers is a pre-specified Vicat softening point T_{sp} . Normally there are a number of possible ways to reach the goal. Figure 6 shows schematically two different situations, both of them having the same Vicat values (T_{AB}) lower than specified although having different dynamic mechanical behaviour. In the case of blend AB_1 the possibility of reaching the goal is quite high, whereas in the case of AB_2 the possibility is more limited.

Another consequence arising from this rationalisation is that we can expect that some blends may be very sensitive to minor variations of production or processing conditions with respect to Vicat values. Practical examples of this condition can be seen in Figs 1A and 1B, for blends of PC and ABS; indeed we expect that formulations similar to those used in samples 203, 204, 205, 206 and 207 are quite critical, because the required modulus level for the Vicat value is present over a wide range of temperature (the modulus with respect to temperature is very flat at this level). On the contrary, sample 212, Nivionplast

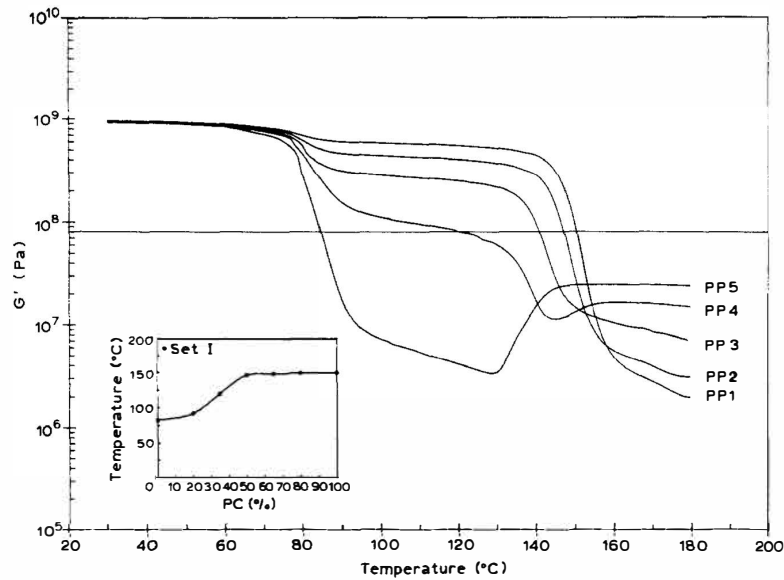


Fig. 4. G' values as a function of temperature for polycarbonate-polyethyleneterephthalate blends. Inset: Vicat values as a function of concentration for PC/PET blends.

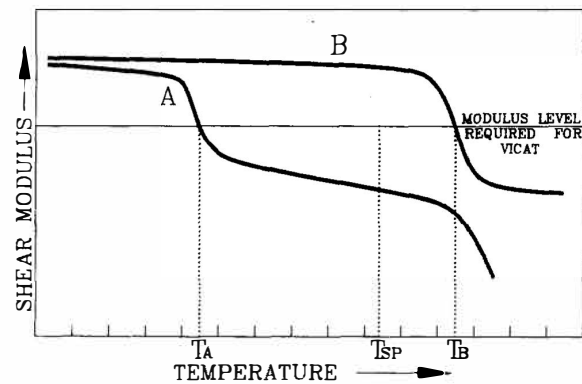


Fig. 5. Schematic representation of the position of Vicat softening point in the shear modulus-temperature diagram for two imaginary polymers A and B.

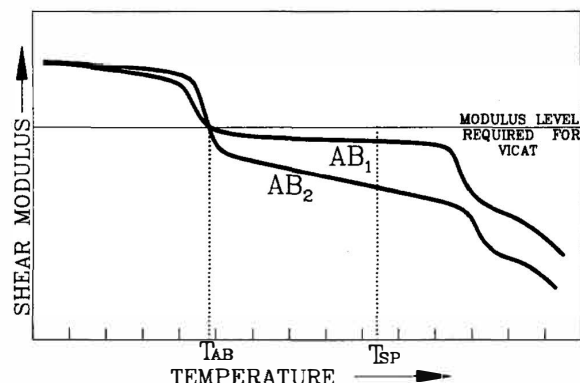


Fig. 6. Schematic representation of the probable position of Vicat softening point for materials AB_1 and AB_2 , which may result from the blending of A and B.

R35GS, Nivionplast C35M, Nivionplast R30GR and all pure materials (PC, PET) are very stable with respect to their Vicat values.

For the same reason, differences in Vicat values for the same materials measured by different laboratories are not impossible when measuring the Vicat value of material such as AB_1 (Fig. 6), where the modulus as a function of temperature becomes very flat.

To avoid all these problems it seems better to use dynamic-mechanical properties for better characterisation of materials. Where this is not possible, Vicat values at different loads may be used as a practical method to obtain a clear idea about the thermal behaviour of the material. This is quite similar to the practice of measuring melt flow index (MFI) under different loads to get an estimate of the shear sensitivity when generation of a complete rheological curve is not possible.

Users of engineering plastics sometimes specify their Vicat values to meet their application requirements. From the above discussion it appears that reliance on dynamic-mechanical characterisation, or on Vicat values under different loading conditions, will definitely give more detailed and quantitative information on thermal behaviour. This practice will obviously enrich the field of polymer technology, where despite all its progress, application of empirical and semi-empirical rules are still enjoying the support of some sectors.

4 CONCLUSIONS

The Vicat values A and B are equal to the temperature which brings the shear modulus of the material to 1.7×10^8 Pa and 8×10^7 Pa respectively.

Using these levels of moduli we can obtain a good rationalisation of Vicat measurements for all types of polymeric materials whether simple polymer, blends or filled. Thermal properties under short-time loading can then be more easily understood and correlated with the polymer structure. Knowing the corresponding modulus of the Vicat values, these values can be used more intelligently for design purposes.