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DETERMINATION OF THE DEFLECTION TEMPERATURE UNDER LOAD, VICAT SOFTENING TEMPERATURE, AND CLASH–BERG T_F OF PLASTICS BY A NEW METHOD*

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ABSTRACT

The American Society for Testing and Materials (ASTM) has developed many tests for determining the temperature at which polymers possess a certain modulus. Included in these methods are the deflection temperature under load (DTUL), the VICAT softening temperature, and the Clash–Berg T_F. Young's Modulus of a material may be determined as a function of temperature by following the penetration of a weighted probe into a sample as the sample is heated at a uniform rate. The duPont 941 Thermal Mechanical Analyzer (TMA) allows the penetration method to be used to determine the DTUL, VICAT softening temperature, and Clash–Berg T_F simultaneously on a small sample.

INTRODUCTION

It has been reported by Gehman[1] that it is possible to determine Young's Modulus as a function of temperature by following the penetration of a weighted probe into a sample as the sample is heated at a uniform rate. The equation used to make this determination is

$$E = \frac{3F}{8RD} (1 - 0.75R/D)$$

where E = Young's Modulus, F = the load on the probe, R = the radius of the probe, d = the depth of penetration of the probe at some temperature, and D = the thickness of the sample. The present work applies this equation to the determination of some of the thermomechanical properties of polymers as defined by the ASTM.

EXPERIMENTAL

The instrumentation used was a duPont 941 Thermomechanical Analyzer (TMA), a plug-in module for the duPont 900 Thermal Analysis System. The sensing

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element of the TMA is a movable core differential transformer. A quartz probe, in contact with the sample, is attached to the movable core of the transformer. As the probe moves, a change in transformer output is sensed and recorded.

Sensitivity of the instrument is 1.5×10^{-4} in. of probe displacement per inch of chart paper in a temperature range from -120 to 500°C. Displacement is plotted on the Y axis and temperature on the X axis by a Moseley X–Y recorder.

The differential transformer was calibrated and checked for linearity by measuring the coefficient of linear expansion of aluminum.

The sample size used in the DTUL and VICAT measurements by means of the TMA was $0.20 \times 0.20 \times 0.10$ in. thick. The programmed TMA heating rate was 5°C/min.

Determination of the deflection temperature under load (DTUL)

The DTUL[2] of a sample can be defined as the temperature at which a sample exhibits a given Young's Modulus. The DTUL is determined using the ASTM method by applying a load to the center of a sample which acts as a beam supported at both ends. The Young's Modulus of this beam can be calculated using the equation[3]

$$E = \frac{FL^3}{4CD^3Y}$$

where C = the width of the sample, L = the length of the sample between the beam supports, Y = the deflection of the sample under load, D = the thickness of the sample, and F = the load on the sample. All of these quantities being standardized by the ASTM[2], DTUL then compares the temperatures at which different samples will exhibit a given Young's Modulus.

The DTUL can be determined at two loading levels, 18.5 and 4.6 kg/cm^2 (264 and 66 p.s.i.). The 18.5 kg/cm^2 loading level, corresponding to a Young's Modulus of 9.7×10^3 kg/cm^2, was selected for these experiments.

The above value of Young's Modulus was substituted into the equation for E to be used with a weighted probe. With this information it was possible to calculate d_r the penetration of the probe at the DTUL temperature.

The penetration necessary, using a duPont 941 TMA equipped with a 0.025-in. diameter penetration probe and a load of 10 g, was calculated and found to be extremely small (5×10^{-6} in.) at the DTUL Young's Modulus. It was concluded therefore that the DTUL temperature would occur at the point where the displacement *vs.* temperature plot of the 941 TMA left the baseline. For purposes of this study, the baseline was defined by the straight line expansion of the material before any detectable penetration.

Four samples of modified and unmodified PVC, on which DTUL data had been obtained, were examined using the probe penetration method. As shown in Table I, there is very good agreement between the ASTM DTUL temperature and the temperature of original deviation from the baseline of the TMA penetration *vs.* temperature plot.

TABLE I

DEFLECTION TEMPERATURE UNDER LOAD FOR MODIFIED AND UNMODIFIED PVC

Sample	*TMA deflection* (°C)	*ASTM DTUL* (°C)
Unmodified PVC	73	75
Service Temp. Mod. PVC	90	91
Impact Mod. PVC	73	73
Service Temp. and Impact Mod. PVC	78	80

Determination of the VICAT softening temperature

The VICAT softening temperature[4] is defined by the ASTM as the temperature at which a circular probe of 1.0 mm^2 cross-section, under a load of 1000 g, penetrates 1.0 mm into a sample 12.7 mm thick.

By substituting this information into the penetration equation, the Young's Modulus at the VICAT Softening Temperature was found to be 66 kg/cm^2.

The plots of penetration *vs.* temperature for the modified and unmodified PVC used to determine DTUL were reexamined, and the temperatures corresponding to a modulus of 66 kg/cm^2 were obtained. This corresponds to a penetration of 6.4×10^{-4} in. with a duPont 941 TMA equipped with a 0.025-in. diameter penetration probe and a load of 10 g. These values are compared in Table II to VICAT temperatures as determined by the ASTM method.

TABLE II

VICAT SOFTENING TEMPERATURE FOR MODIFIED AND UNMODIFIED PVC

Sample	*TMA VICAT* (°C)	*ASTM VICAT* (°C)
Unmodified PVC	86	84
Service Temp. Mod. PVC	106	103
Impact Mod. PVC	84	85
Service Temp. and Impact Mod. PVC	93	92

As can be seen there is again excellent agreement between the two methods.

Determination of the Clash–Berg T_F

It should also be possible to determine Clash–Berg T_F temperatures[5] by using this same method. The ASTM specifications for the Clash–Berg T_F essentially define the temperature at which a material exhibits a torsional modulus or torsional rigidity, G, of 3.16×10^3 kg/cm^2. Assuming a Poisson's Ratio of 0.5, Young's Modulus, E, can be determined using the equation[3]

$$E = 2G(1 + \lambda)$$

where λ = Poisson's Ratio.

After calculation, E for a Clash–Berg T_F was found to be 9.5×10^3 kg/cm^2, which is approximately equal to the Young's Modulus used in the DTUL experiment (264 p.s.i. loading).

DISCUSSION

It would appear that the method based on the TMA is a valid one for approximating ASTM measurements relating temperature and modulus. The method is particularly useful when applied in cases where only a small amount of sample is available and ASTM specimen sizes cannot be molded.

The effect of sample size and heating rates on the measurements made has been neglected for the present. We are aware, however, of the significance of these parameters but chose initially to keep the sample size small enough and the heating rate slow enough to minimize thermal lag in the sample. These parameters will be examined in depth at a later date.

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