

A New Apparatus for Continuously Monitoring the Deformation of a Thermoplastic Material

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Synopsis

A new apparatus is described whereby the thermal behavior, including the deformation temperature, of a thermoplastic material may be obtained. The apparatus is intended to replace ASTM procedure No. D648-56 and has several advantages over that procedure. Thermal behavior of several common thermoplastics under two different stresses and for three different heating rates is described. An application to the determination of optimum cure times for certain thermosetting epoxides is also described.

The deflection temperature of a thermoplastic material is the temperature at which an arbitrary deformation occurs when the specimen is subjected to an arbitrary load. The existing method for determining deflection temperature is the ASTM procedure No. D648-56.¹ It requires that a fairly large sample be subjected to a force transverse to its length at its midpoint while immersed in an oil bath whose temperature is increasing at a specified rate. The deflection temperature is taken to be the point at which the sample has deformed 0.01 in. under the load as read on a dial indicator. The method is well suited for quality control applications during production but suffers from several disadvantages when accurate laboratory data are needed. The large sample size (5 in. long \times 1/2 in. wide \times 1/4 in. thick) is often neither practical nor necessary; the temperature range of the test is limited by the choice of immersion fluid which often swells the sample prior to deformation yielding erroneous data. The arbitrary choice of deformation allows for the sample to begin deforming at a relatively low temperature yet not reach its deformation point until a much higher temperature, again providing misleading information.

The continuous-recording heat deflection apparatus described herein requires that a small, research-size sample be subjected to a stress in the same manner as in the ASTM procedure. The sample is heated in an electric furnace and a continuous record of its deformation behavior under the load as a function of temperature is provided. Figures 1 and 2 show the overall apparatus and the holder and plunger assembly, respectively. The apparatus consists of a cylindrical brass holder (10) which is 1 in. long and 1 in. in diameter, a wedge-shaped brass plunger (20) which fits inside the cylindrical holder with guidepins (22), which fit guides (23) in the

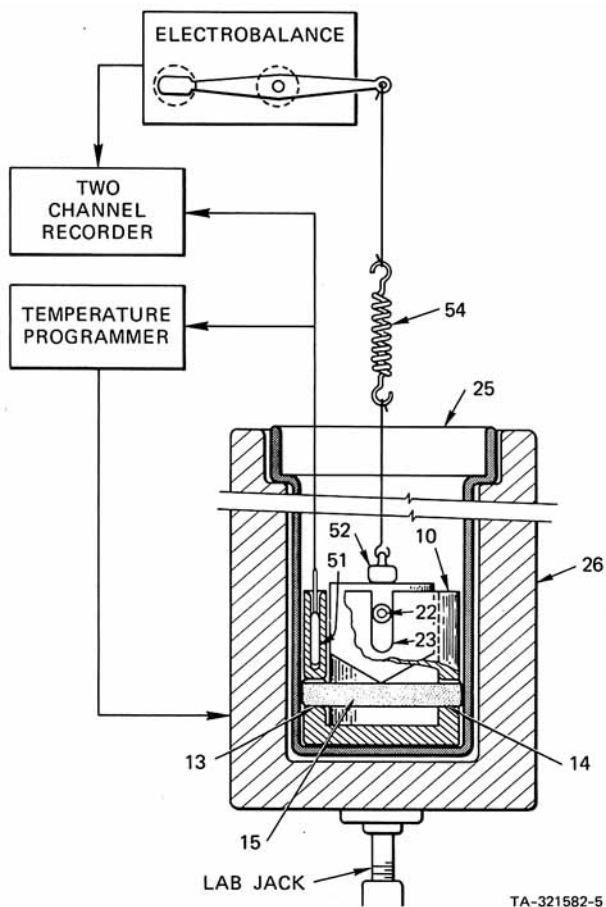


Fig. 1. View of overall apparatus.

holder, and a small (1 g) weight (52) which rests on the plunger and is connected via a low-tension spring (54) to the arm of a Cahn RG recording electrobalance. The sample (1 in. long \times $\frac{1}{4}$ in. wide \times $\frac{1}{8}$ - $\frac{1}{4}$ in. thick) is inserted in the holder at (15) and is supported by the two rounded surfaces (13) and (14). A Chromel-Alumel thermocouple (51) is located in the wall of the holder very near the sample. The plunger is inserted in the guides so that the wedge (21) is transverse to the sample, and the assembly is placed in a Pyrex (or quartz) tube (25) which is in turn inserted in a furnace (26). The furnace is then raised by a lab jack until it contacts the small weight, thus relieving tension on the spring and nulling the balance. Output from the thermocouple is fed to a proportional temperature programmer and to one channel of a two-channel recorder; output from the electrobalance is connected to the other channel. As the temperature of the furnace is steadily increased, any deformation of the sample allows the plunger to move downward in its guides, causing the spring to elongate and register the increase in weight on the recorder. The spring

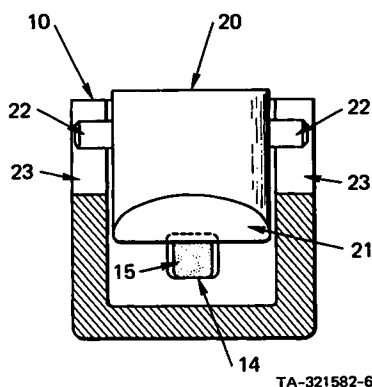


Fig. 2. Detail of holder and plunger assembly.

is wound from 4-mil spring steel and has a force constant of 22.07 mg/sec²; a deflection of 0.025 cm (0.010 in.) in the sample results in a weight change of about 0.5 mg on the recorder. The stress on the sample is calculated according to the equation¹

$$S = 3PL/2BD^2$$

where S is stress in psi, P is the weight of the plunger in pounds, and L , B , and D are the length, width, and thickness of the sample, respectively, in inches. Two different plungers were built which, when using a $1/8$ -in.-thick sample, yielded stresses of 29.5 and 59 psi, respectively. The force due to the spring and small weight was negligible. Heating rates were 2.0, 5.0, and 10.0°C/min.

Deflection temperatures were determined for several polymers, and the results, shown in Table I, are generally consistent with those in the literature.² Polystyrene, poly(vinyl acetate) and Lucite [du Pont's poly(methyl methacrylate)] all exhibited deformations at or near their glass transition temperatures.

Figure 3 shows the recorder trace for Lucite when the temperature program rate was 2.0°C/min and the deflection range was 1.0 mg. The deflection temperature is obtained by constructing a horizontal line through the break in the deflection trace and noting the point at which it intersects the linear temperature trace. Thus, the deflection temperature for Lucite from Figure 3 is 109°C. Any increase in weight causes the balance pen to move upscale; the slight apparent decrease in weight prior to deformation is due to thermal expansion of the sample and brass components of the apparatus. The moderately broad temperature range over which the deformation occurs is also characteristic of the glass transition for polymers.

Figure 4 shows a recorder trace for Delrin (an acetal polymer made by du Pont) which was run at 10°C/min; its deformation occurs over a much narrower temperature range due to both the increased heating rate and the nature of the sample. We have found that deflection temperature is generally independent of heating rate in these cases, allowing for rapid and accurate determinations. The apparatus has also been used in con-

TABLE I
Heat Deflection Temperatures of Various Polymers

Sample	Heating rate, °C/min	Stress, psi	Deflection temp., °C	
			This work	Lit. ^a
Polystyrene	2	29.5	101	95-105
	5	29.5	100	
	5	59.0	100	
Poly(vinyl acetate)	2	29.5	41	-
	2	29.5	34	
	2	59.0	38	
Polypropylene	5	29.5	124	93-110
	5	59.0	111	
	10	59.0	92	
Poly(phenylene oxide)	2	29.5	130	130
	10	29.5	134	
	10	59.0	135	
Lucite	2	29.5	101	99
	2	29.5	109	
	5	29.5	110	
	10	59.0	106	
Lexan ^b	2	29.5	146	143
	5	29.5	143	
	10	59.0	144	
Delrin	10	29.5	173	170
	10	59.0	162	

^a All values are for 66 psi stress.

^b A polycarbonate made by General Electric, Selkirk, N. Y.

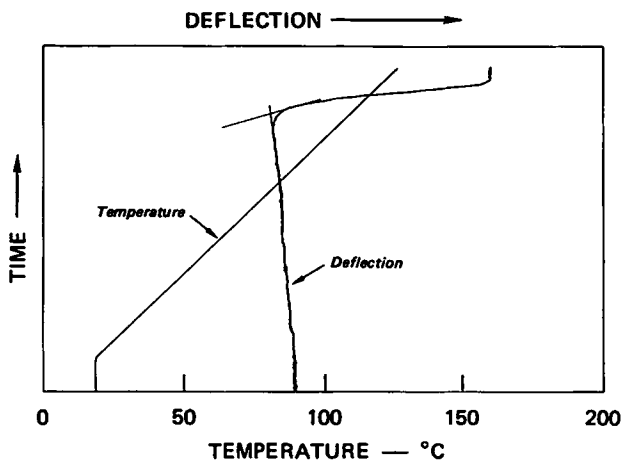


Fig. 3. Temperature-deflection recorder trace for Lucite.

junction with a Cahn Mark II time derivative computer which provides a continuous record of deflection rate and yields better defined, more accurate deflection temperatures.

The apparatus has proved useful in establishing optimum cure times for certain thermosetting epoxides. Figure 5 shows deformation records for

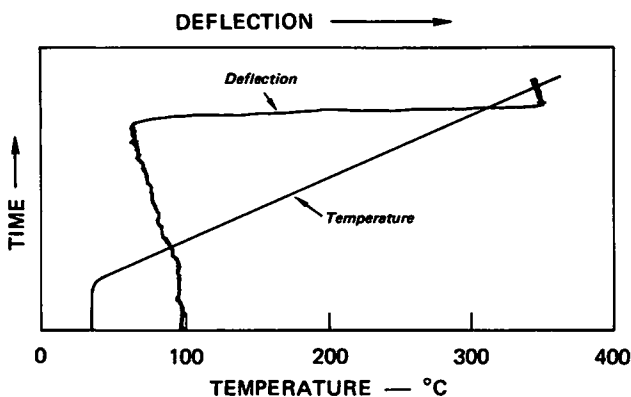


Fig. 4. Temperature-deflection recorder trace for Delrin.

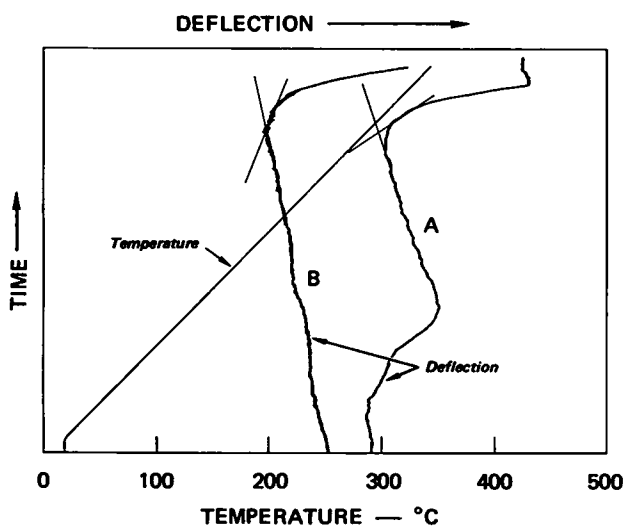


Fig. 5. Temperature-deflection recorder trace for two bisphenol A resins.

two bisphenol A resins cured with methyl nadic anhydride. Sample A was cured for 4 hr and sample B, for 20 hr at 120°C. It can be seen that sample A began to deform at 60°C but stopped after only ≈ 0.0025 in. of deflection. Since this early deformation is absent in sample B, it was concluded that sample A was not fully cured. Apparently, sample A continued to cure during the measurement and finally set, causing deflection to cease; both samples finally deformed at the same temperature.

References

1. American Society for Testing and Materials, *1970 Annual Book of Standards*, Part 27, *Plastics—General Methods of Testing, Nomenclature*, Philadelphia, Pa.
2. *Modern Plastics Encyclopedia*, Vol. 45/No. 1A, McGraw-Hill, New York, 1968.

Received September 29, 1972