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Heat Treatment of Flat Perspex Sheets

By

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The Heat Treatment of Flat Perspex Sheets

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SUMMARY

The heat treatment which has been recommended to improve the craze-resistance of flat methacrylate sheets is discussed. Distortion may be avoided by controlled heating during the initial cycle, and by annealing at a temperature just below the second order transition point during the cooling cycle. To define the latter the isothermal shrinkage of quenched material was measured.

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1 Introduction

Earlier reports^{1,2} have shown that the heat treatment of polymethyl methacrylate improves its craze resistance by removing casting stresses and by completing polymerisation. For curved structures the heat treatment received during the normal shaping process is adequate for this purpose, and the treatment of flat sheet should be considered first. The annealing of complete shapings has a somewhat different objective and will be dealt with later in a separate Note. In the above reports a brief outline was given of some of the chief considerations relating to such processing and the present purpose is to enlarge this information.

2 The heating cycle

In treating flat sheets it is obvious that every endeavour must be made to maintain both flatness and freedom from defects liable to impair the optical properties of the material. To avoid local distortion heating should be uniform. It should therefore be performed either with the material suspended vertically in the oven, or very gradually if it is placed on a flat surface until it reaches a temperature at which it becomes limp. For unplasticized methacrylate this temperature is about 115° - 120°C and for material containing 5% or 8% plasticiser 110° or 100°C respectively. More rapid heating is then permissible. The use of heating media other than air is undesirable in that cleaning difficulties are introduced. Masking paper should be removed before heating and strict attention must be made to the exclusion of dust which might be deposited on the softened plastic and retained on cooling.

A high temperature is necessary to polymerise residual monomer reasonably quickly and the manufacturers have recommended a heating period at 140°C varying in duration t (minutes) with the thickness x (inches) according to the formula

$$t = 10 + 80x.$$

3 The cooling cycle for the avoidance of distortion

On cooling, distortion occurs if the temperature of any portion falls more rapidly than that of the other. This distortion is not merely differential thermal contraction but is of a more permanent character. Fig. 1 represents the cooling curves for the material. ABC is the normal volume-temperature relation obtained on slow cooling; on quenching, the curve AB'C' is observed. Thus the more rapidly cooled portions of a sheet occupy a greater volume than would be the case on slower treatment and the sheet becomes distorted. If for example one side is cooled more rapidly than the other the latter face becomes concave. When a quenched sample is again heated only a portion of the curve C'B' can be realised. In fact slow contraction instead of expansion may occur and the sample assumes the volume it would have taken on slow cooling. The temperature T_m defined as shown in Fig. 1 where there is a change of slope, is designated the second order transition point and is a convenient reference temperature. From the foregoing considerations it will be seen that to avoid distortion two alternative procedures may be used. Either the material may be cooled very slowly and uniformly through T_m , or it may be cooled rapidly to a temperature just below T_m and held in that condition for a sufficient time for uniformity in density to be attained. The former method is obviously the more difficult; the latter will therefore be more fully discussed.

Table I gives the values of the second order transition temperature

T_m of polymethyl methacrylate of varying plasticiser contents, these values were derived from experiments using a rate of heating of $\frac{1}{2}^{\circ}\text{C}$ per minute.

Table I Second order transition temperatures

Plasticiser content - per cent by weight	T_m $^{\circ}\text{C}$
0	101
5	86
8	78

3.1 Period of heating

It is now necessary to determine the time of heating required. For this purpose quenched samples contained in dilatometers of the type previously described were held at a series of constant temperatures. Fig.2 shows a typical shrinkage - time curve and the corresponding logarithmic plot. The linear form of the latter permits its slope to be used for calculating the rate constant of a first order process, correcting for the change to natural logarithms. Values of this constant are given in Table 2 and from them the times were calculated which were needed for the completion of 99.95% of the shrinkage. Direct experiment in the case of the plasticised material confirmed that by this method substantially flat sheet was produced.

Table 2 The isothermal shrinkage of rapidly cooled material

Temperature $^{\circ}\text{C}$	Rate constant $\text{min}^{-1} \times 10^2$	Time for 99.95% completion-minutes
<u>Unplasticised</u>		
80.3	1.75	435
82.9	2.01	380
86.7	2.74	278
93.7	4.06	167
<u>8% plasticiser</u>		
64.0	2.21	344
69.6	2.74	278
74.4	3.69	207

3.2 Recommended procedure

The heat treated sheet is transferred from the air oven at 140°C to a flat plate glass sheet in an oven heated at the temperature given in Table 3 and maintained at that temperature for the stated time. The sheet may then be cooled to room temperature.

/Table 3

Table 3

Sample	Temperature, °C	Time, hours
Unplasticised	93 - 96	3
5% plasticiser	80 - 82	4
8% "	73 - 75	4

4 Conclusions

The heat treatment of flat sheets of polymethyl methacrylate has been discussed with special reference to the avoidance of distortion and other defects. The required conditions of the preliminary heating up to the temperature of 140°C, of heating at that temperature and of cooling are defined.

REFERENCES

<u>No.</u>	<u>Author</u>	<u>Title, etc.</u>
1	E. W. Russell	Report Chem. 441. Studies on polymethyl methacrylate. Parts I and II. June 1948.
2	E. W. Russell	Report Chem. 447. Studies on polymethyl methacrylate Part III. August 1948.

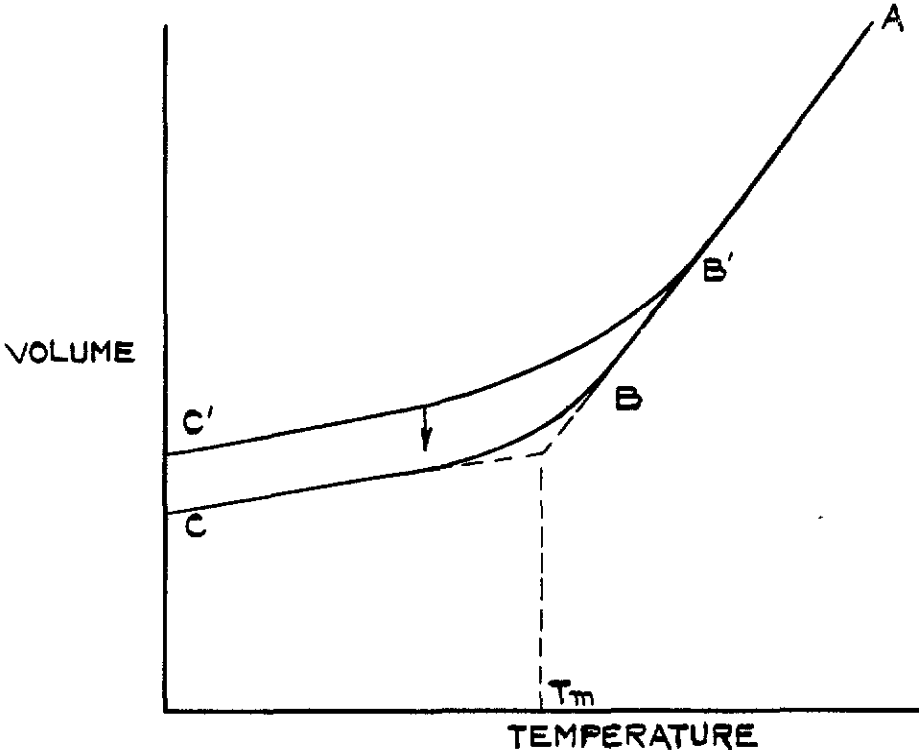


FIG. 1. VOLUME-TEMPERATURE RELATIONS OF POLYMETHYL METHACRYLATE

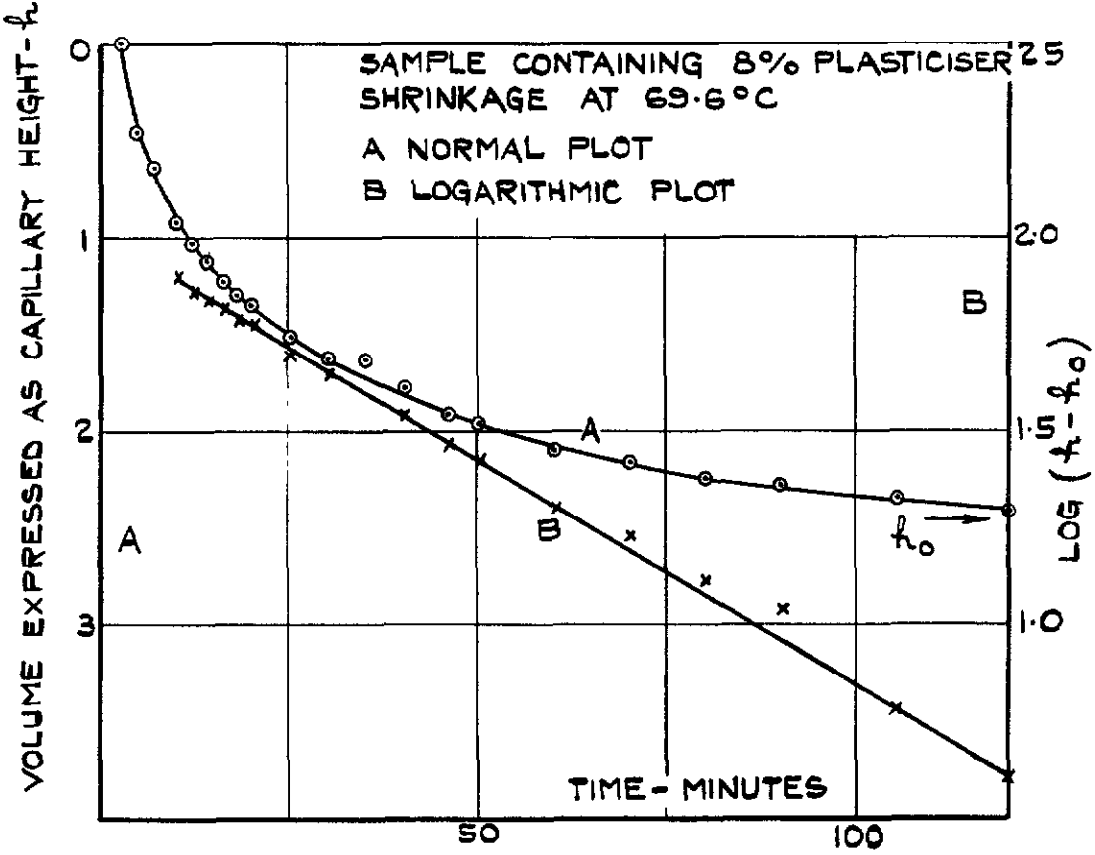


FIG. 2. ISOTHERMAL SHRINKAGE-TIME CURVES FOR POLYMETHYL METHACRYLATE

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