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Structure and properties of injection-moulded nylon-6

Part 2 *Residual stresses in injection-moulded nylon-6*

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The distribution of residual stress through the thickness of injection-moulded nylon-6 has been investigated. It was found that the stress distribution is parabolic with a compressive stress at the surface of the moulding and a tensile stress in the centre of the moulding. The magnitude of these residual stresses is inversely proportional to mould temperature. Exposure of the moulding to boiling water resulted in a reversal of the residual stress distribution, with a tensile stress at the surface and a compressive stress at the centre of the moulding. This effect was attributed to the inhomogeneous volume changes that occurred in the mouldings as a result of water-induced crystallization in nylon-6.

1. Introduction

Residual stresses can be defined as the local mechanical stresses that exist at any point in a moulding. They are generated by non-uniform volume changes in a body and the sum of the forces and moments generated by the stresses must equal zero. If mechanical equilibrium is disturbed by removing some of the stresses, for example by machining the body, then a change in shape of the body occurs until force and moment equilibrium are re-established. An understanding of residual stresses in injection-moulded parts is important because of the effect they have on mechanical behaviour. For instance, secondary crystallization in nylon-6 induced by absorbed water could produce inhomogeneous volume changes in a moulding and generate residual stress.

In Part 2, we describe a technique for measuring residual stress in nylon-6 injection mouldings; discuss the effects of processing variables on the distribution of residual stress in the moulding; and finally, examine the effect of water.

2. A technique for measuring residual stress

The method used by previous workers [1–4] and ourselves is based upon the disturbance of stress equilibrium within a moulding, produced by cutting the moulding or by removing sections from it. A change in shape of the part can be measured as a function of the thickness of section removed, and the original distribution of residual stress in the moulding can be determined.

2.1. Principle of the layer removal technique

Consider a rectangular beam containing residual compressive stresses in the surface and residual tensile stresses in the centre. Removal of a thin layer of material from the surface releases the compressive stresses contained in that layer; the force and moment equilibrium in the moulding is disturbed and the length and curvature of the beam changes in order to re-establish equilibrium. The magnitude of change in length and curvature of the beam depends upon the magnitude of residual stress in the layer of material removed.

The procedure for measuring residual stress is as follows: a layer of known thickness is removed from the surface of a rectangular beam specimen and the curvature assumed by the specimen is

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measured. Additional layers are removed and the corresponding changes in curvature are recorded. These data are sufficient to calculate the residual stress present in each layer before its removal from the specimen. We can build up a quantitative assessment of the distribution of residual stress through the thickness of the specimen.

2.2. Calculation of residual stress

Consider a rectangular bar of thickness $2Z_0$ with residual longitudinal tensile stresses $\sigma(Z)$ (see Fig. 13 in Appendix). If the bar is sectioned to a thickness $(Z_0 + Z_1)$ then the stress that existed in the layer Z_1 is given by

$$\sigma(Z_1) = \sigma_1 - \sigma_2 - \sigma_3, \qquad (1)$$

where σ_1 is the stress in the layer Z_1 at the time of its removal which is calculated from the change of curvature, $d\phi$, upon removal of the layer Z_1. From simple beam theory

$$\sigma_1 = \frac{E}{6(1-\nu^2)}(Z_0 + Z_1)^2, \qquad (2)$$

where E is Young's modulus, ν is Poisson's ratio and dZ is the thickness of the layer removed. σ_2 and σ_3 take into account the stresses introduced into the layer Z_1 by the bending and lengthening of the specimen brought about by the removal of the layers previous to the Z_1 layer.

From simple beam theory

$$\sigma_2 = \frac{2(Z_0 + Z_1)E\phi}{3(1-\nu^2)} \qquad (3)$$

where ϕ is the curvature of the bar. The stress, σ_3, introduced by the change in length of the specimen, is an implicit function of $\sigma(Z_1)$,

$$\sigma_3 = \frac{1}{(Z_0 + Z_1)} \int_{Z_0 + Z_1}^{2Z_0} \sigma(Z_1)\, dZ. \qquad (4)$$

Various attempts have been made to render Equation 1 explicit and permit the calculation of $\sigma(Z)$ from the experimental data. The expression used in this paper is one derived by Treuting and Read [5] and is discussed in the Appendix.

3. Experimental details

3.1. Specimen preparation

Rectangular bars 100 mm x 15 mm x 3 mm were injection-moulded from nylon-6 (Akulon K2 ZG 340) with a melt temperature of 250° C and mould temperatures of 40 and 100° C. ASTM tensile bars with a central portion 70 mm x 10 mm x 3 mm were moulded out of nylon-6 with a melt temperature of 250° C and mould temperatures of 25 and 85° C.

Rectangular bars 50 mm long were cut from the central region of each moulding, and these bars were sectioned to determine the distribution of residual stress.

3.2. Layer removal technique

Each specimen was fixed to a steel block and a layer 0.1 mm thick removed by a milling machine in a single pass. The total thickness removed from a specimen ranged between 0.25 mm and 0.6 mm.

3.3. Measurement of curvature

The instrument used to measure curvature consisted essentially of a dial gauge capable of measuring to within 2 μm. A specimen, with gauge length of 19 mm, is positioned on two knife edges set 19 mm apart. The distance of the specimen below the level of the two knife edges, f, is determined from the dial gauge readings (Fig. 1).

4. Results and discussion

The distribution of residual stresses determined in the prismatic bar specimens are shown in Figs. 2 to 5. These residual stress patterns corresponding to

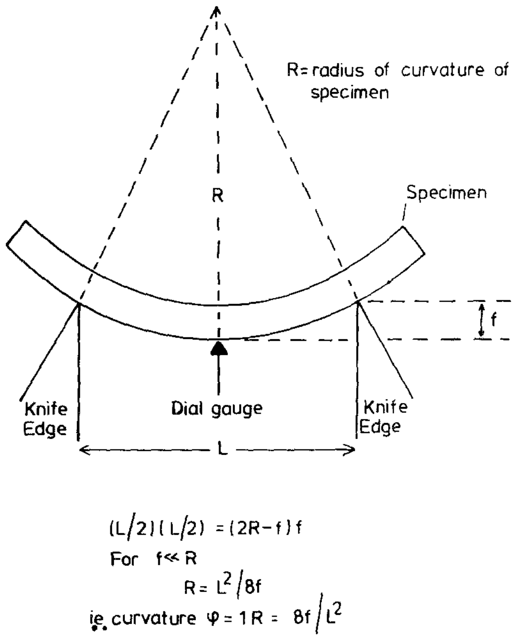

Figure 1 A schematic diagram to show the method for measuring the curvature of rectangular bar specimens cut from nylon-6 injection mouldings.

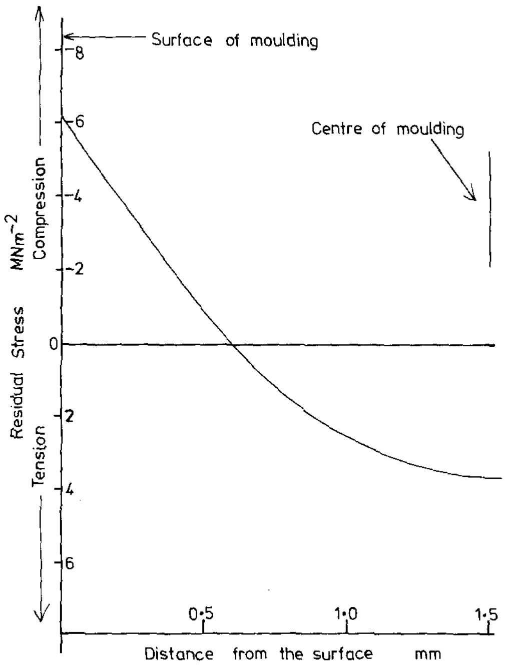

Figure 2 Residual stress distribution in nylon-6 injection-moulded using a mould temperature of 25° C.

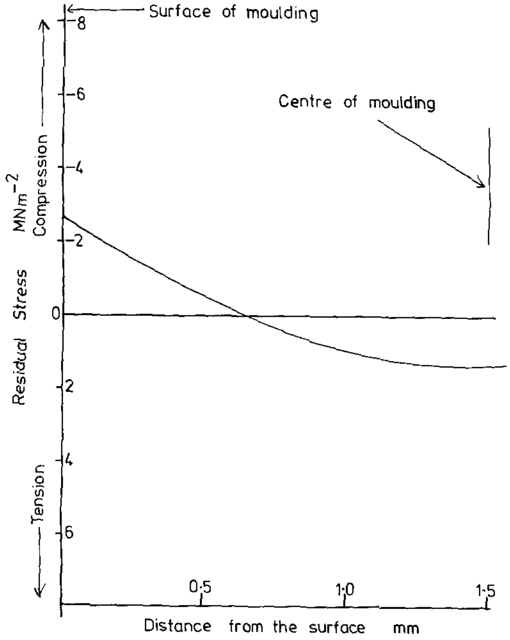

Figure 4 Residual stress distribution in nylon-6 injection-moulded using a mould temperature of 85° C.

the four mould temperatures, 25, 40, 85 and 100° C, have common features; firstly, the surface layer of each moulding is in compression and the centre region is in tension; secondly, the residual stress increases monotonically from the surface, reaching a maximum stress at the centre (we assume compressive stresses to be negative and tensile stresses to be positive); thirdly, the shape of the residual stress distribution curve is approximately parabolic. The maximum compressive stress at the surface is about twice the maximum tensile stress

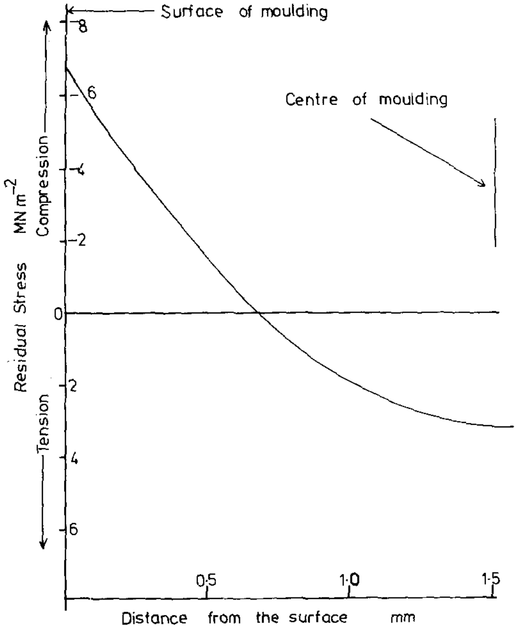

Figure 3 Residual stress distribution in nylon-6 injection-moulded using a mould temperature of 40° C.

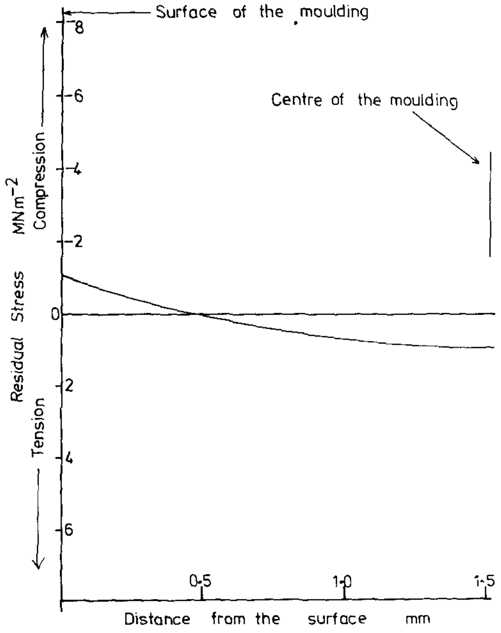

Figure 5 Residual stress distribution in nylon-6 injection-moulded using a mould temperature of 100° C.

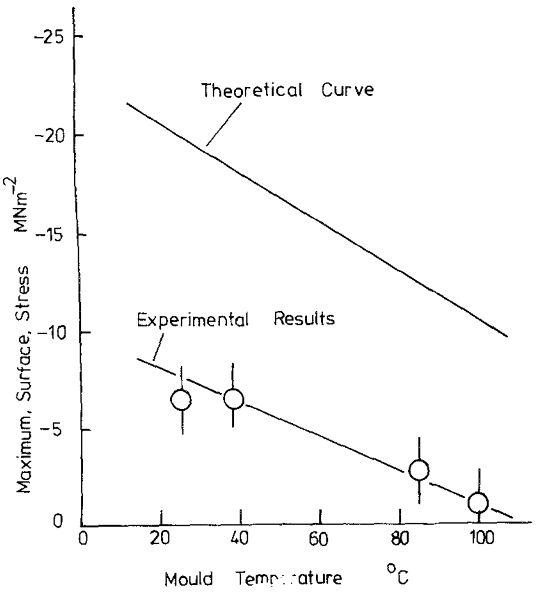

Figure 6 Maximum surface compressive stress as a function of mould temperature. The solid line is determined using Equation 5.

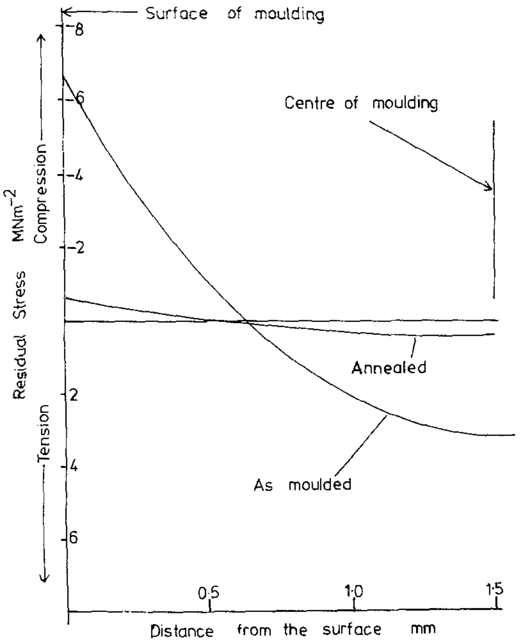

Figure 7 Comparison between the residual stress distribution found in nylon-6 before and after annealing at 180° C for 16 h. (The mould temperature was 40° C.)

at the centre for each moulding examined. It is the magnitude of residual stress which is sensitive to mould temperature, the higher the mould temperature the lower the residual stresses in the moulding; for example, the maximum compressive stress and tensile stress are 6.5 and 3.5 MN m^{-2}, respectively, for a mould temperature of 25° C compared to stresses of 1.2 and 0.8 MN m^{-2} for a mould temperature of 100° C. The magnitude of the surface stress decreases linearly with increasing mould temperature as shown in Fig. 6.

At the point where the centre of the moulding finally solidifies, a temperature gradient will exist through the thickness. On cooling further, the centre of the moulding contracts, the amount of contraction depending upon the difference between melt and mould temperature. The effect is to develop a compressive stress close to the surface and a tensile stress at the centre of the moulding, the magnitude of these stresses depending upon the relative thermal contractions between the surface and central core.

The shape of the residual stress distribution and the magnitude of these stresses can be established if we first consider the temperature profile and cooling-rate in the moulding. A model proposed by Aggarwala and Saibel [6] can be used to predict the distribution of residual stress;

$$\sigma(Z) = \frac{\alpha E \Delta T}{2(1-\nu)}\left(\frac{1}{3} - \frac{Z^2}{Z_0^2}\right). \qquad (5)$$

The equation describes a parabolic stress function where the maximum compression stress is at the surface $\sigma(Z_0)$ and the maximum tensile stress is at the centre $\sigma(0)$ of the moulding which is in qualitative agreement with our experimental results. ΔT is the difference between melting point and mould temperature. Using values of $\alpha = 8 \times 10^{-5}\ ^\circ\mathrm{C}^{-1}$, $E = 2.7\ \mathrm{GN\,m^{-2}}$, $\nu = 0.35$ and $T_\mathrm{m} = 200^\circ$ C, the maximum compressive stress, $\sigma(Z_0)$ as a function of mould temperature can be calculated and compared to experimental measurement (Fig. 6).

The model overestimates the experimental values of residual stress. A possible explanation may be due to the occurrence of stress relaxation in the moulding since the model assumes no relaxation of stresses below the freezing temperature of the polymer.

Subsequent annealing of mouldings may reduce (perhaps eliminate) these residual stresses; a moulding fabricated with a mould temperature of 40° C, for instance, and annealed 180° C under vacuum for 16 h and slowly cooled showed little residual stress (Fig. 7).

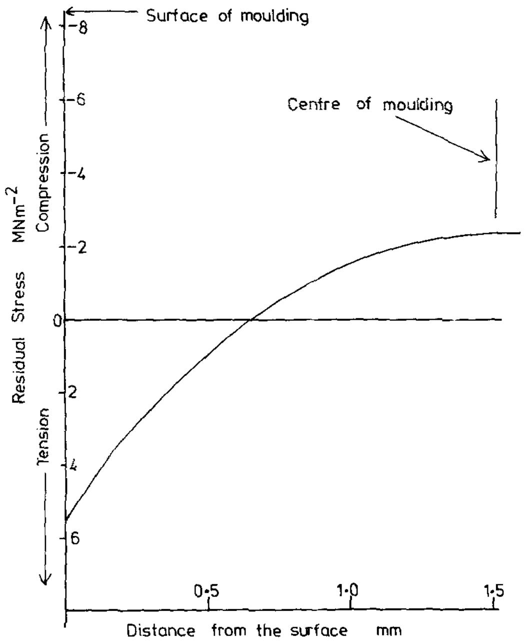

Figure 8 Residual stress distribution in a moulding saturated with water. (The mould temperature was 40° C.)

4.1. Effect of water

Several of the mouldings were boiled in distilled water for various lengths of time. The rate of water pick-up was monitored by weighing each moulding and the residual stress distribution was established using the technique described in Section 2 (Figs. 8 and 9).

Saturating the mouldings with water reverses the sign of the residual stress, the surface layer becomes stressed in tension, for instance. The variation of residual stress with distance from the surface is monotonic, decreasing with increasing distance from the moulding surface. The magnitude of tensile stress and compressive stress in mouldings made at 40 and 100° C is similar; surface tensile stresses are about 6 MN m^{-2} and centre compressive stresses are about 3 MN m^{-2}.

Under non-equilibrium conditions, a concentration of water of 2.6% in nylon-6 results in a compressive stress close to the surface which is less than the compressive stress in a dry moulding fabricated using a mould temperature of 40° C (Fig. 10). A change in shape of the residual stress profile through the thickness of the moulding has resulted in order to accommodate the change in surface stress. About 3% by weight of water is necessary to reverse the sign of the surface stress and the residual stress curve to maintain force and moment equilibrium in the moulding (Fig. 11). A water pick-up of 4% by weight is sufficient to produce a residual stress profile in a moulding which is similar to that in a moulding saturated

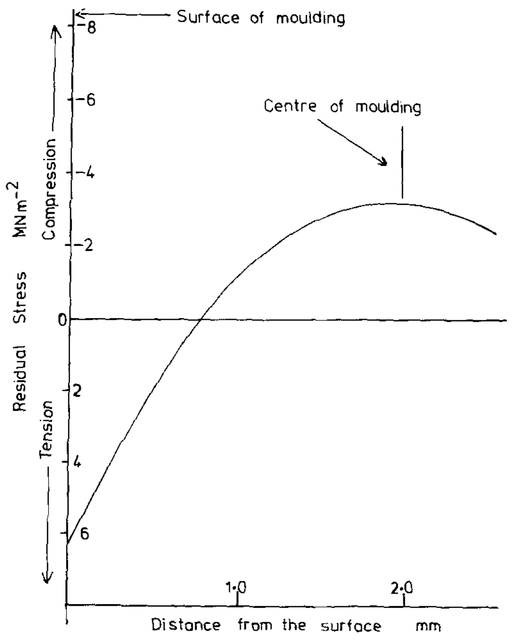

Figure 9 Residual stress distribution in a moulding saturated with water. (The mould temperature was 100° C.)

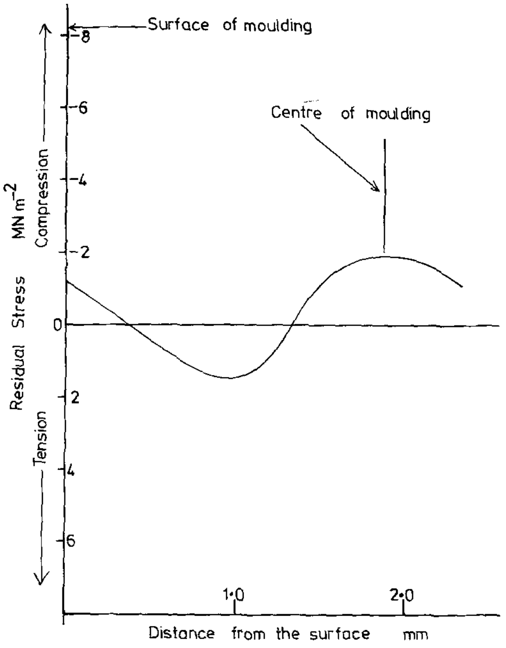

Figure 10 Residual stress distribution in a moulding containing 2.6% by weight of water. (The mould temperature was 40° C.)

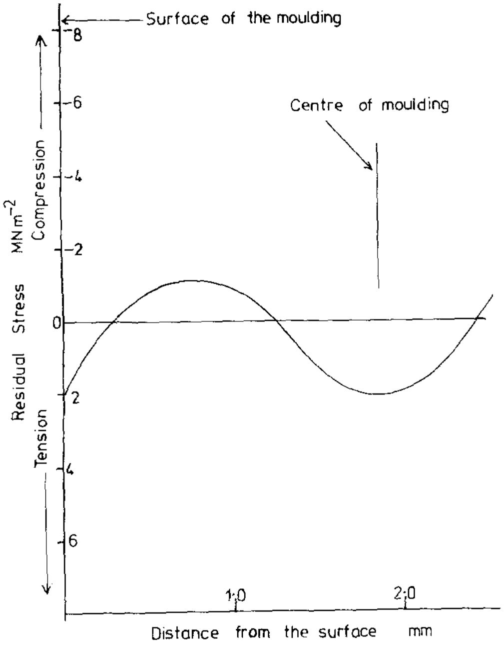

Figure 11 Residual stress distribution in a moulding containing 2.9% by weight of water. (The mould temperature was 40° C.)

with water (~10% by weight), and the magnitude of these stresses is similar (Fig. 12).

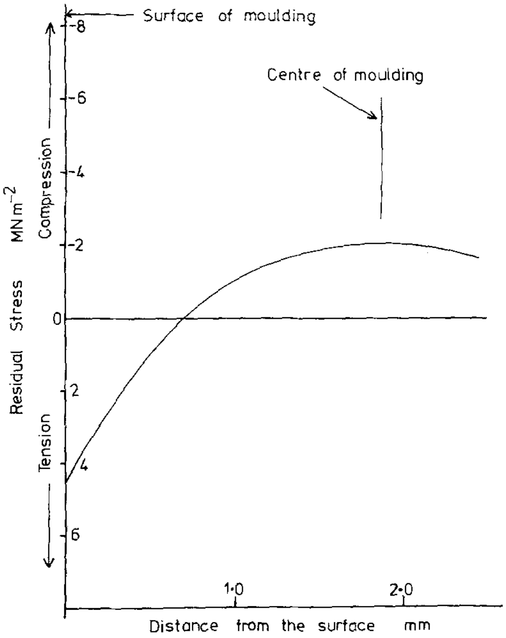

Figure 12 Residual stress distribution in a moulding containing 4% by weight of water. (The mould temperature was 40° C.)

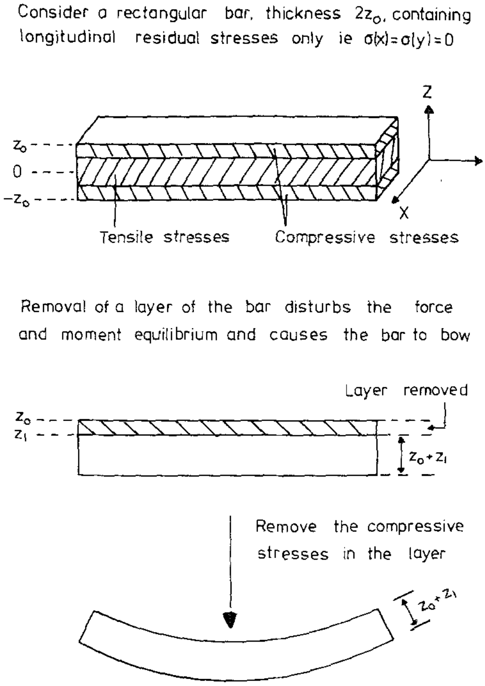

Figure 13 The principle of the technique to measure residual stress distribution in a rectangular bar containing longitudinal residual stresses.

The absorption of water in nylon-6 induces crystallization in the amorphous phase and this results in a decrease in volume of material. This has brought about a reversal of stress due to the concentration of water close to the surface of the moulding. Consider a nylon-6 injection moulding exposed to boiling water until saturation point is reached. The volume change at the surface and centre of the moulding, ΔV_s and ΔV_c, respectively, can be calculated using an equation of the form

$$\frac{\Delta V_s}{V_s} = (1.0X)\left(\frac{\rho_1}{\rho_2}\right) - 1 \qquad (6)$$

where X is the amount of water absorbed at the surface (or centre) of the moulding and ρ_1, ρ_2 (ρ_3, ρ_4) are the densities of the nylon-6 in the surface layer (or central region) of the wet and dry mouldings. From Fig. 2 (Part 1), $\rho_1 = 1.120\,\mathrm{g\,cm^{-3}}$; $\rho_2 = 1.147\,\mathrm{g\,cm^{-3}}$; $\rho_3 = 1.133\,\mathrm{g\,cm^{-3}}$; $\rho_4 = 1.140\,\mathrm{g\,cm^{-3}}$ and X is 10% (by weight) which gives

$$\Delta V_c - \Delta V_s = 1.7\%,$$

i.e. the centre of a moulding exposed to boiling water for 8 h has effectively increased in volume by 1.7% compared to the surface of the moulding.

Now the residual compressive stress in a surface layer of a moulding using a mould temperature of 40° C is 5 MN m^{-2} and after exposure to boiling water a residual tensile stress of $3\,\mathrm{MN\,m^{-2}}$ is produced. The linear contraction of the surface relative to the centre of the moulding is

$$\Delta\epsilon = \frac{\sigma_1}{E_1} + \frac{\sigma_2}{E_2}. \qquad (7)$$

Using values of $E_1 = 2.8\,\mathrm{MN\,m^{-2}}$ for a dry moulding and $E_2 = 0.9\,\mathrm{MN\,m^{-2}}$ for a wet moulding, $\Delta\epsilon = 0.6\%$. Hence the volume contraction at the surface relative to the centre of a moulding is 1.8%, assuming isotropic behaviour of the nylon-6. The volume changes calculated from the residual stress measurements and the volume changes obtained from density measurements are in good agreement.

5. Conclusions

In nylon-6 mouldings, the surface layer is under a residual compressive stress and the central region is subjected to a residual tensile stress. The effect of exposing these mouldings to boiling water is to reverse the residual stress distribution through the thickness of the moulding, a residual tensile stress in the surface layer and a residual compressive stress at the centre.

The reversal of the residual stress distribution can be explained in terms of a crystallization process in the moulding. The potential of the surface layer to undergo crystallization is greater than that of the central region because of the high amorphous content on the outside of the moulding. A decrease in volume of the surface layer compared to the central region of the moulding can be sufficient to produce this reversal of residual stress.

Appendix. Residual stress distribution in a rectangular bar by the method of Treuting and Read

Consider a rectangular bar of thickness $2Z_0$ subjected to a residual longitudinal stress $\sigma(Z)$. If the curvature of the bar after reducing its thickness to $(Z_0 + Z_1)$ was $\phi(Z_1)$, the residual stress, $\sigma(Z_1)$ that existed in the original bar is given by

$$\sigma(Z_1) = -\frac{E}{6(1-\nu^2)}\left[(Z_0+Z_1)^2\frac{\mathrm{d}\phi(Z_1)}{\mathrm{d}Z_1} + 4(Z_0+Z_1)\phi(Z_1) - 2\int_{Z_1}^{Z_0}\phi(Z)\,\mathrm{d}Z\right]. \qquad (\mathrm{A1})$$

The curvature, $\phi(Z)$ of the bar after removing thin layers can be calculated by measuring the sagitta, f, of a chord of known length, L (Fig. 1). $\phi(Z)$ is given by

$$\phi(Z) = \frac{8f}{L^2}. \qquad (\mathrm{A2})$$

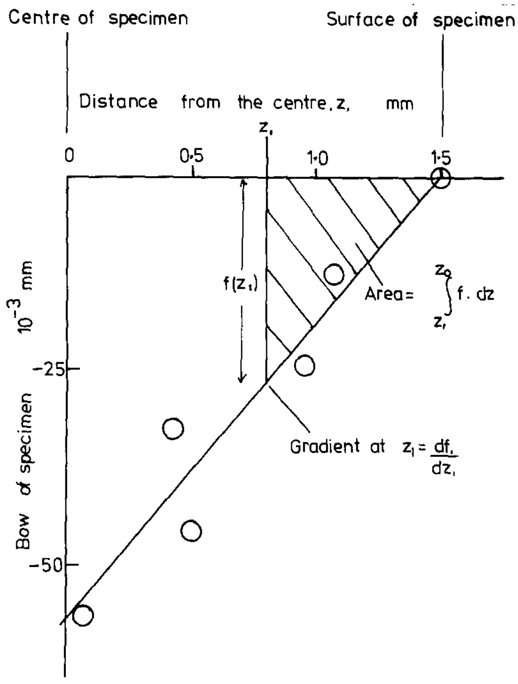

Figure 14 A typical curve to calculate residual stress distribution in a rectangular bar, showing the variation of the bow of the specimen, f, with thickness.

Substituting Equation A2 into Equation A1.

$$\sigma(Z) = -\frac{4E}{3(1-\nu^2)L^2}\left[(Z_0+Z_1)^2\frac{\mathrm{d}f(Z_1)}{\mathrm{d}Z} + 4(Z_0+Z_1)f(Z_1) - 2\int_{Z_1}^{Z_0} f(Z)\,\mathrm{d}z\right]. \qquad (\mathrm{A3})$$

The residual stress distribution is calculated graphically. A curve with deflection, f, as the ordinate and the thickness of the moulding after removing a thin layer as the abscissa, was plotted. A typical curve is shown in Fig. 14.

$\mathrm{d}f\,(Z_1)/\mathrm{d}Z$ is the gradient of the curve at the point $Z = Z_1$; f is the ordinate of the curve at the point $Z = Z_1$; and $\int_{Z_1}^{Z_0} f(Z)\,\mathrm{d}Z$ is the area under the curve between the points $Z = Z_0$ and $Z = Z_1$.

It is assumed in the analysis that the bar behaved elastically in pure bending and the stresses in the specimen were invariant in the xy plane and varied only in the Z direction.

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