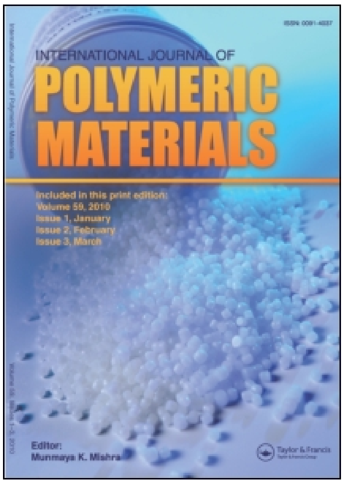

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Internal Stress Assessment of Thick-Section Injection-Mouldings

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Ways of assessing internal stress in injection-moulded bars of thick cross-section (12.5 mm square), have been investigated. The layer removal procedure confirmed that residual stresses were tensile in the interior, but it was not possible to produce an accurate through-thickness stress profile. The Kubát and Rigdahl analysis was found to give a satisfactory straight line extrapolation when applied to uniaxial compressive stress relaxation data, but the parameters so derived could not be correlated with the results of tensile tests. Birefringence measurements were made at various positions within the bars and some tentative deductions are discussed by reference to results obtained from thinner bars.

INTRODUCTION

Injection-mouldings almost always contain residual stresses in addition to molecular orientation. Residual stresses are a result of differential cooling rates and can be minimised, if so desired, by suitable choice of moulding conditions or by post-moulding annealing. They may even be diminished by room-temperature ageing in the case of semi-crystalline polymers with T_g below room temperature. In almost all cases the residual stress in the interior of an injection-moulding is tensile, opposed by compressive stresses near to the surface. In many instances this may be beneficial, inhibiting the growth of cracks from surface flaws. On the other hand the residual stress will add to any externally applied stress so that in tension the interior of the article will

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experience a net stress larger than the mean applied stress and this may cause premature crazing, for instance. In the case of thick-section mouldings the hydrostatic tensile stress generated in the interior on cooling is often sufficient to produce voiding. While this may partially relieve the residual stresses, the introduction of a large internal flaw is extremely detrimental and should be avoided. It is of importance to determine the distribution of residual stresses in thick-section mouldings under conditions in which no voiding is produced and to investigate methods of controlling their magnitude. The present work represents a preliminary study in this area.

Several techniques for assessing residual stresses in polymers have been reported. Those based on the stress–optical effect, in which birefringence measurements are related to the stress, are complicated by the strong birefringence associated with molecular orientation. Although it is fairly easy to demonstrate whether the major contribution to birefringence is due either to residual stress or to orientation,[1] it is not easy to separate their relative contributions when both are strong, though significant progress has been made recently.[2] A further complication occurs with materials having highly polarizable side groups which may re-orient under stress or at slightly elevated temperature without the need for significant main-chain conformational changes.[3] In the layer removal technique successive layers of material are removed leaving behind an unbalanced system of stresses in a straight bar, causing it to bend until force equilibrium is restored; measurements of curvature as a function of the depth of removal enables computation of a residual stress profile through the bar. Finally assessment procedures based upon stress relaxation data have been developed by Li[4] and by Kubát and Rigdahl (KR).[5] The Li procedure produces an "internal stress parameter" that is dependent on both residual stress level and on applied deformation. The results of several stress relaxation tests at different deformations should in principle enable extrapolation to zero deformation and hence to give a value relating to residual stress only.[6] In the KR procedure sets of stress relaxation curves are obtained at different initial stress levels, σ_0, and the values of the slope at the steepest part are plotted versus σ_0. A straight line is normally obtained and σ_i is taken to be proportional to the intercept with the σ_0 axis. This procedure effectively extrapolates to zero deformation so that the σ_i value relates only to the residual stresses,[7] though the intercept also contains a proportionality factor which appears to have been overlooked by KR.

The exact significance of the internal stress parameter remains to be established. It has been found that the slope of the KR plot is very sensitive to changes in fabrication conditions[8] and to ageing[9] and other post-fabrication treatment[6] and this parameter may turn out to be a more valuable method of characterizing injection-mouldings than σ_i.

EXPERIMENTAL

Specimen preparation

Izod impact test-pieces The Izod impact test-piece has a cross-sectional thickness that is greater than that of the vast majority of injection-moulded articles. It was therefore chosen as a convenient configuration with which to examine the properties of thick injection-mouldings. A two-cavity mould was employed, one cavity for moulding a straight bar of dimensions 63 mm × 12.7 mm × 12.7 mm and the other for moulding a bar with the same dimensions but containing in one face a notch corresponding in dimensions to that specified by the Izod impact test (BS 2782: 1970, Figure 306.1). It should be noted here that the exact dimensions and shape of the moulded-in notch were sensitive to moulding conditions; whereas the measurements were often in close agreement with the prescribed dimensions it was often found that post-moulding recovery caused the notch to become shallower and for the root radius to increase. Although both cavities could be fed together all tests reported here were conducted on mouldings produced during runs in which one cavity only was in use (the straight bar with no notch). The cavities are end-gated and can be fed from either end or both ends; in the present work all test bars were produced under double end-gating, Figure 1. A Butler–Smith 100-60 reciprocating screw injection-moulding machine was used and the moulding conditions are shown in Table I.

It was found very difficult to prevent void formation. Voids were always located along the axis, usually several mm long and approximately 2 mm in diameter (Figure 1). The injection-moulding conditions were adjusted to reduce voiding, and as the final conditions were approached it was found that

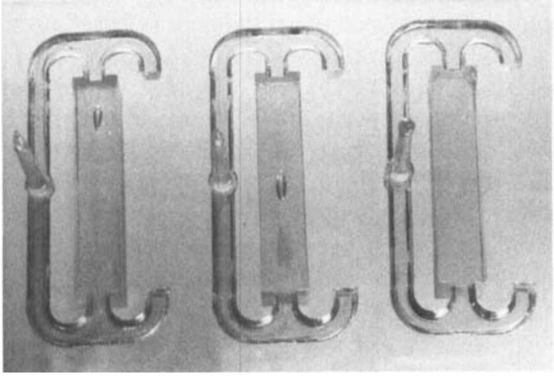

FIGURE 1 Izod bar mouldings with sprue and runner still attached. Left hand moulding contains a void near one of the ejector pins (common fault). Centre moulding contains a void near its centre. Right hand moulding is flaw-free.

TABLE I

Material	Specimens	Injection Pressure (MPa)	Moulding temps. °C Nozzle/Zone 2/Zone 1	Mould Temperature °C
BP KLP 35	Izod Bars	71	230/230/170	~50
BP KLP 35	3 mm Bars	37	280/270/220	~25

voiding was usually confined to those regions along the axis of the moulding that were adjacent to the ejector pins as in Figure 1. Careful observation of the moulding during mould opening revealed that the voids appeared at a quite advanced stage of cooling, and often seemed to be nucleated when the ejector pins first began to push the moulding out of the cavity. The machine was adjusted to give a quite slow retraction of the mould onto the ejector system, so facilitating visual observation. The mouldings were found to be fairly tightly held in the mould, a consequence of having a high moulding pressure and a deep, parallel-sided cavity.

Straight bars Straight bars were moulded on the Butler–Smith injection-moulding machine using a cavity of the following dimensions: 190 mm × 12.7 mm × 3 mm. These specimens were single-end-gated, and moulding conditions are given in Table I.

TESTING AND ANALYSIS

Layer removal procedure

The layer removal procedure[10,11] has been used previously to derive the residual stress distribution in the 3 mm-thick bars.[8–12] High speed milling was used to perform the layer removals and a method based on the reflection of laser light from mirrors attached to the surface was used to obtain the curvature.[9] In the case of the Izod bar the radii of curvature obtained were very small, a consequence mainly of the large cross-sectional area, but the sense of the curvature was quite easily perceived by the time about one half of the bar had been machined away, and indicated that the interior of the bar contained residual tensile stresses with compressive stresses near to the surface. (We estimate that even for an Izod bar possessing tensile residual stresses near to the crazing stress of 40 MN/m^2 the curvature for 0.5 mm removed would be only approximately 0.3/m.)

Stress relaxation testing

Izod bars The runner system was carefully removed and the remnants of the gates at each end of the Izod bar were made flush with the moulded ends using emery paper. Uniaxial compression tests were performed using the stress relaxation machines described previously,[9] operated in the opposite sense to that used for the tensile tests reported before, and using flat-ended cylinders of mild steel in place of the wedge grips, mounting the specimen with its axis vertical and its ends against the parallel flats. This arrangement was permitted because the Pye-Ether UF2 load cells are bi-directional. The specimen constant temperature enclosure was maintained at 40°C.

Straight bars In order to compare the results of uniaxial compression tests with results of uniaxial tensile tests it is desirable to use the same type of specimen to conduct both series of experiments. The Izod test bars were unsuitable for performing tensile tests, while compression tests on the 3 mm bars are rendered difficult by the bending that takes place. Wedge grips were constructed that operated in the opposite sense to normal, so that delivery of a compressive load caused tightening of the wedges (Figure 2), and a channel was made to restrain the bending of the specimen. Tests were again conducted at 40°C.

Birefringence Values of relative retardation were estimated by identifying the characteristic colour corresponding to the optical path difference caused by the double refraction of polychromatic light in the specimen. For this purpose a polarizing microscope was used in conjunction with a quartz wedge compensator. For some specimens a plot of relative retardation versus thickness removed was generated in a manner similar to that described previously by E. F. T. White *et al.*[13] Layers were removed by high speed

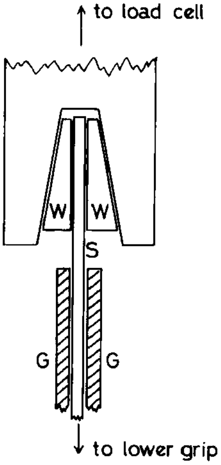

FIGURE 2 Wedge grips for compression testing. GG = guide; S = specimen; WW = wedges.

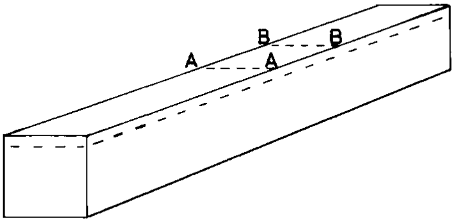

FIGURE 3 Locations of relative retardation measurements (i) centre of bar (AA), (ii) at position of greatest relative retardation (BB).

milling and the machined surface polished prior to conducting the optical measurement. Relative retardation was measured at fixed locations. For the Izod bars the locations chosen were (i) the centre of the bar and (ii) the position at which the overall relative retardation was found to be greatest (see Figure 3). For the straight bars the position at which the overall relative retardation was greatest was again chosen for these measurements and was located close to the gate.[12]

RESULTS

Layer removal procedure

The results of a layer removal test on a 3 mm-thick polystyrene bar moulded at a quite high injection pressure, (143 MPa), are shown in Figure 4. If the points on the curvature versus depth removal plot lie on a straight line, as seems to be a fair approximation, then the computed residual stress distribution is parabolic. This result is similar to that obtained with bars of the same grade of polystyrene moulded at lower injection pressures,[8] and shows that the stresses in the interior are tensile and those near to the surface are compressive. Although the curvatures obtained with the Izod bars were too small to allow accurate measurement the sense of the bending was quite clear and indicated once again that tensile stresses resided in the interior of these specimens.

Stress relaxation analysis

Although most examples of the application of the KR analysis of stress relaxation appearing in the literature have used data obtained in uniaxial tension tests,[5–9,12,14–18] the underlying theory does not discriminate between tension and compression and it should therefore apply equally to the uniaxial compression tests. This is confirmed in Figure 5 in which is shown a KR plot obtained from data using the Izod bars. Compressive stresses have been plotted as positive for convenience throughout so that the sign of the intercept

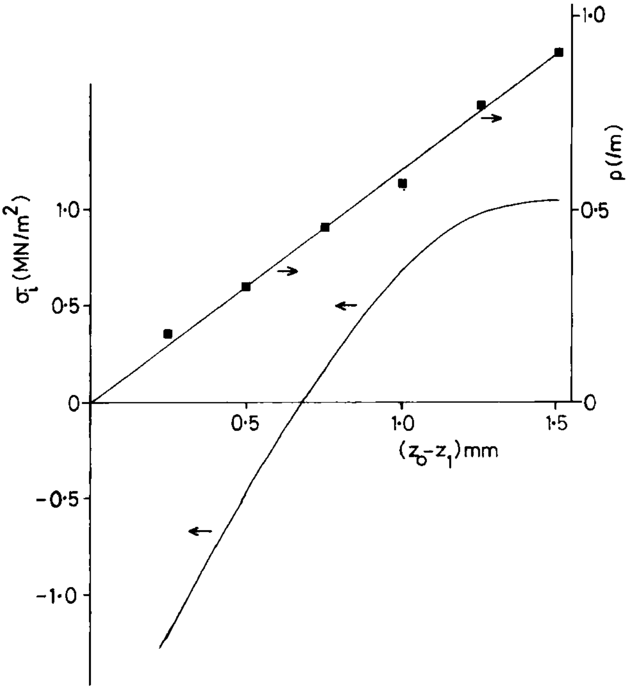

FIGURE 4 Curvature (ρ) versus depth removed (z_0-z_1), and residual stress (σ_i) versus position (z_0-z_1) for 3 mm polystyrene bar moulded at 143 MPa. Results obtained for bars moulded at 37 MPa were very similar.[8]

on the σ_0 axis must be reversed. Hence in Figure 5 the intercept corresponds to a positive σ_i value, $\sigma_i \simeq 1.9$ MN/m^2. The value of the parameter, n, obtained from the slope of the KR plot[5] is 8.9 ± 1.0. The results of tests on 3 mm bars are plotted according to the KR procedure in Figure 6. The scatter is large, but it is evident that the slope is quite large, giving a small value for n (~ 4.8).

Birefringence

Examples of plots of the relative retardation versus position across the width of an Izod bar for locations AA and BB and for various depths removed are shown in Figure 7. Points along section BB were selected for study of through-thickness birefringence, these stations being located (i) 0.25 mm from one edge, (ii) 0.5 mm from the edge and (iii) in the centre of the bar. The relative retardation at each of these positions was read from the plots shown in Figure 7 and those obtained for different machining depths and plotted against the thickness removed (see Figure 8). By differentiating the curves shown in Figure

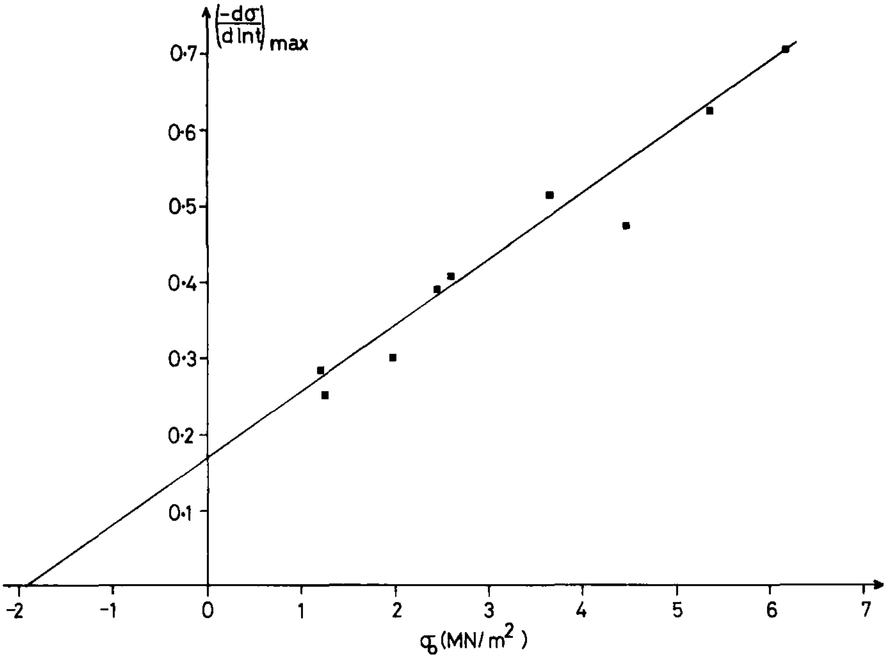

FIGURE 5 KR plot obtained from compressive stress relaxation tests conducted on Izod bars at 40°C. Compressive stress has been plotted as positive for convenience.

8 the birefringence at each location can be estimated and the results of this procedure are shown in Figures 9 and 10. The birefringence is negative near to the surface of the moulding and remains negative at all depths at the centre of the bar with the largest numerical values being observed near to the surfaces of the moulding. At the other locations, negative values are also found in the centre of the bar, but there are positive peaks on either side of centre. An example of the birefringence profile through a 3 mm bar has already been published.[12]

DISCUSSION

In our previous studies of internal stress and orientation in injection mouldings[7–9,12] we have employed specimens 3 mm thick. This is a convenient size both for the layer removal procedure and for tensile testing. For Izod bars of 12.7 mm square cross-section tensile testing is not very convenient. Furthermore, with this geometry sharp curvatures cannot be produced even when the bending moment is quite large and the radii obtained

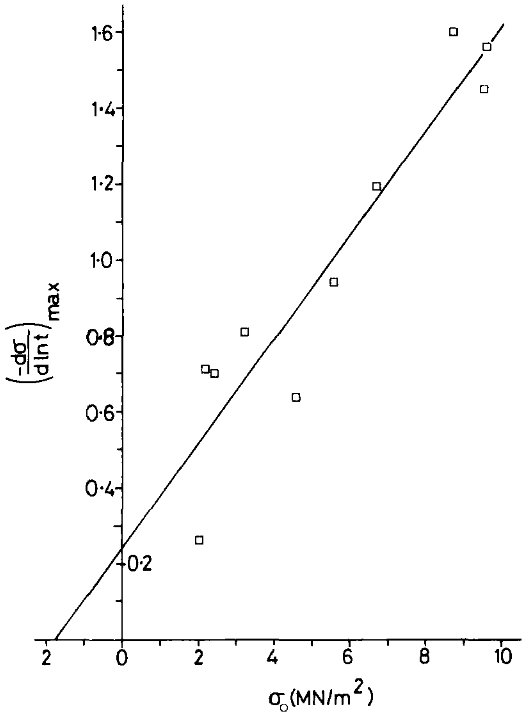

FIGURE 6 KR plot obtained from compressive stress relaxation tests on whole 3 mm polystyrene bars at 40°C. Compressive stress has been plotted as positive. [Note that some tests reported elsewhere[34] were conducted using short specimens cut from 3 mm bars.]

when using the layer removal technique were too large to measure accurately so that residual stress profiles could not be conveniently derived by this method. Information about the Izod mouldings was therefore sought by other means.

Firstly, inspection of the Kubát and Rigdahl analysis[5] revealed no (mathematical) objection to reversing the direction of testing and stress relaxation tests were conducted on the Izod bars in uniaxial compression. Application of the usual KR procedure[5] gave rise to a straight line KR plot with no more than the usual amount of scatter (see Figure 5). Before attempting to interpret this result we considered that it was desirable to compare results in tension with those in compression from a single batch of specimens. A few preliminary tests convinced us that the difficulties (associated with bending) in testing a 3 mm bar in uniaxial compression would be easier to overcome than the problem of testing the Izod bar in tension. A channel was constructed to reduce bending to a minimum and the resulting analysis is shown in Figure 6. This plot is to be compared with Figure 3(a) of Ref. 7, in which is shown the analysis of uniaxial tensile stress relaxation from specimens produced under the same conditions. The value of σ_i was found to be -0.8 ± 0.6 MN/m^2 and $n = 13.8 \pm 1.4$ from the tensile data. There is clearly no compatibility between the parameters obtained from the tensile data and the compressive data respectively. To attempt to explain this apparent anomaly it is useful to follow the analysis presented by Kubát and Rigdahl in

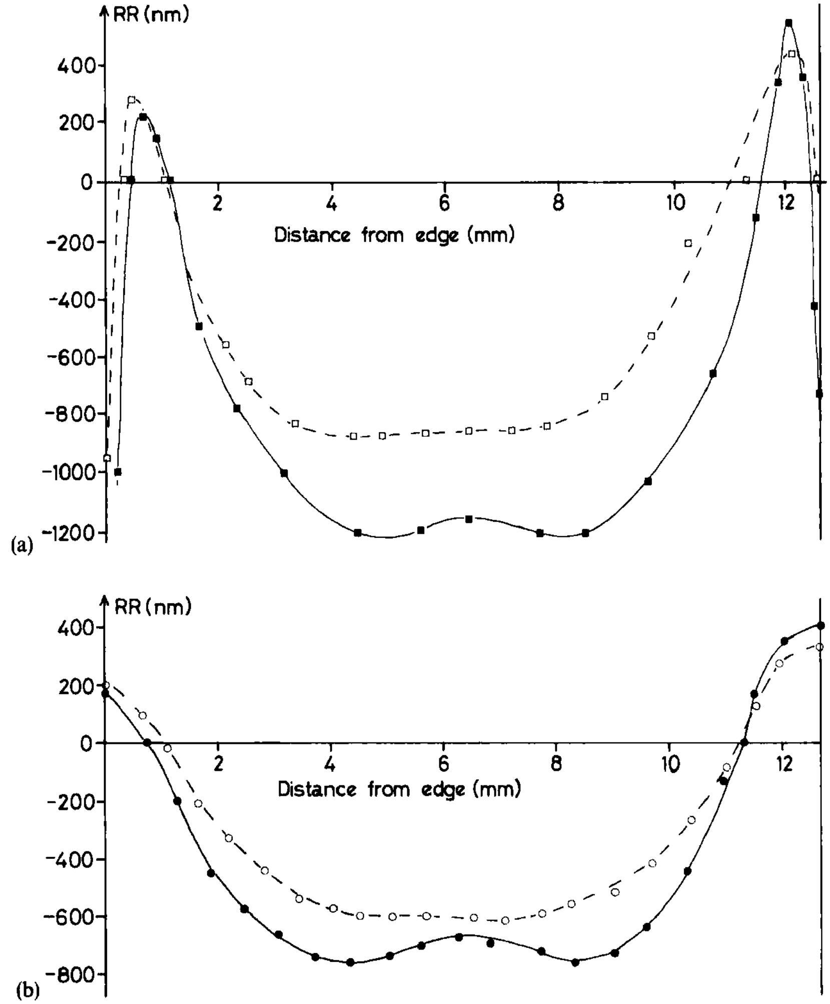

FIGURE 7 Relative retardation (RR) plots across AA (open symbols □ ○ △) and BB (closed symbols ■ ● ▲) for Izod bar from which successive layers were machined from the face parallel to AABB, (a) after removing 1.4 mm, (b) after removing 6.7 mm, (c) after removing 12 mm.

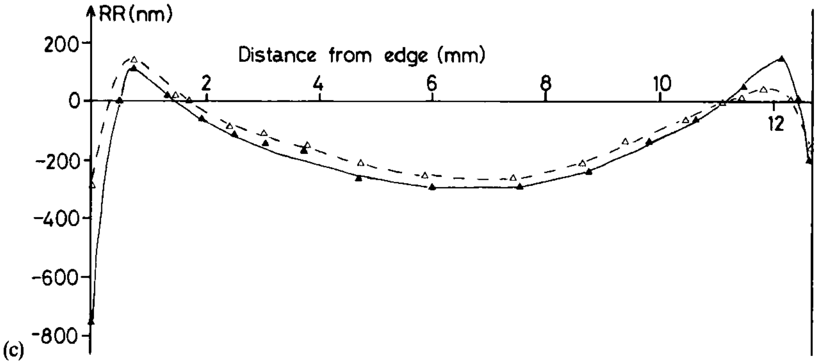

FIGURE 7 (*Continued.*)

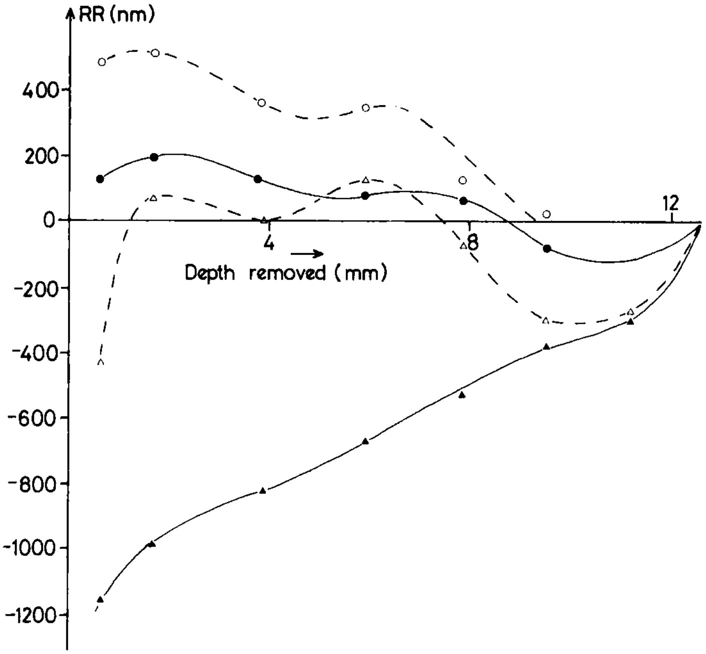

FIGURE 8 RR versus depth removed along BB for stations (i) 0.25 mm from one edge (△); (ii) 0.5 mm from the edge (●); (iii) centre of the bar (▲). Also shown are results for a fourth station (iv) 0.5 mm from the other edge (○).

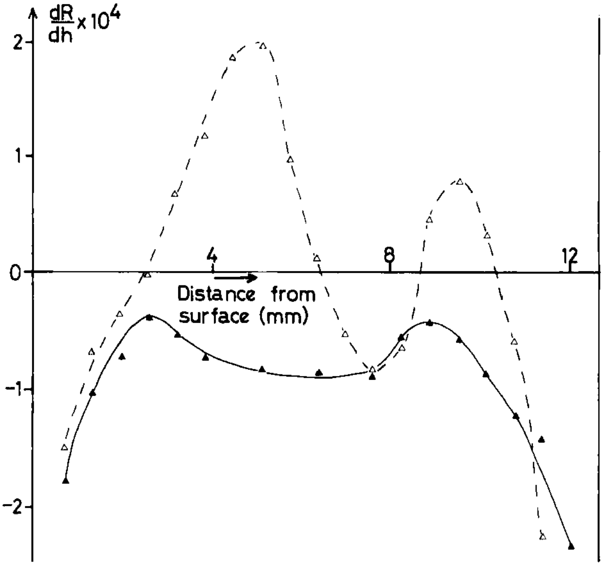

FIGURE 9 Through-thickness birefringence distributions in the Izod bar along BB at stations (i) (△) and (iii) (▲).

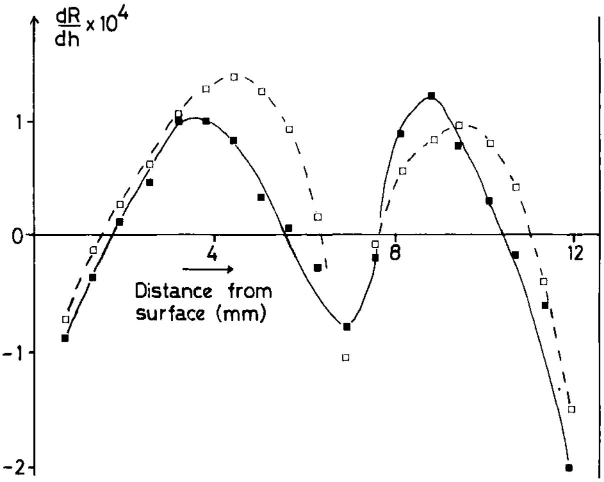

FIGURE 10 Through-thickness birefringence distributions in the Izod bar along BB at stations (ii) (■) and (iv) (□).

another publication.[19] They showed that a non-zero value for σ_i should only be observed when the specimen contains two or more components with different relaxation behaviour. If each component in an injection-moulded bar has a characteristic "n" value the overall value observed is expected to be determined mainly by the component having the largest "n" value.[19] Kubát and Rigdahl took the view that n would be determined by the morphology of each particular component and were guided in their choice of n values used in example computations by experiments conducted on drawn material, (providing a value for the oriented material near to the surface of an injection moulding) and annealed material, (thought to have a morphology similar to the core of an injection moulding).[19] This does not help to resolve the present problem of relating tensile and compressive results, though there is no reason to suppose that the n value would be the same in tension and in compression. Indeed, as the sign of σ_i is believed by Kubát and Rigdahl to be determined by the nature of the stress in the component possessing the lowest n value,[19] then a change in the ranking of n values on switching from tensile to compressive testing would lead to a change in the sign of σ_i. We have attempted similar derivations to those of Kubát and Rigdahl, but based on the exponential law that follows from the site-model theory,[20,21] and found sufficient agreement with the KR analysis to indicate that a simple explanation for the compression/tension results, (reversing the conclusions deduced from the KR approach), is unlikely to be obtained by adopting this alternative analysis. It is, however, well known that relaxation is stress sensitive,[22] possibly through the effect of applied stress on the free volume, and we feel that the depth-dependence of relaxation behaviour is more likely to be caused by the presence of a stress distribution through the specimen than by changes in morphology. At the present time, then, we have to admit that the stress relaxation analysis has not yet provided any tangible information on the thick-section mouldings. In tension, although we do not yet understand the fundamental significance of σ_i and n, we have been able to correlate these parameters with other properties to a limited degree.[6,8,9,12] The equivalent situation is less likely to be obtained for thick mouldings tested in compression because of the severe restriction on the range of moulding conditions that can be used to produce satisfactory, (void-free), mouldings.

The birefringence study reveals marked through-thickness variations of this property in the moulded bars. Uniaxially oriented polystyrene normally displays negative birefringence and this probably accounts for the negative values found near the surface of the bar where the molecules are expected to freeze with molecular backbone conformations that show a preference for the axis of flow. In some regions of the bar positive birefringence has been found and in an earlier study by E. F. T. White *et al.* it was proposed that this was caused by molecular orientation perpendicular to the flow axis ("contra-flow").[13] In the current work we have found some regions of positive birefringence that are more developed than those found by the previous workers,[13] but we are as yet unconvinced by the "contra-flow" explanation. It is to be expected that substantial molecular conformational changes will occur during solidification of the slowly cooling interior of a thick bar and that the flow-promoted orientation of molecules will be largely replaced by random conformation as a result of relaxation during this period. This may cause an axial contribution to the cooling stresses in addition to the hydrostatic tensile stress expected to accompany solidification. If the positive birefringence peaks are the consequence solely of the residual stresses within the solidified bar (polystyrene possessing a positive stress optical coefficient in the glassy state), then a knowledge of the stress optical coefficient for polystyrene would enable an estimation of the local magnitude of the residual stress to be made. Because many of the values for the stress optical coefficient appearing in the literature were obtained on oriented material which has a different stress optical coefficient to isotropic material,[23] or at different temperatures,[24] and because of the effect of stress relaxation,[3,25] we are unable to choose a suitable value with any confidence. If we take a value of 8×10^{-12} m^2/N then a measured birefringence number of 2×10^{-4} (the highest positive value recorded in the work reported here) corresponds to a stress of 25 MN/m^2 which is below the crazing stress of polystyrene ($\sim$40 MN/m^2),[8,13] yet greater than the tensile stresses measured in the thinner bars. Theoretical and experimental studies have not given very detailed guidance on the effect of section thickness on residual stresses in injection mouldings,[26–31] though Knappe[26] reported that thicker mouldings contained higher stresses. The observation that voiding is more common in thicker mouldings is consistent with the development of higher stresses. Our own analyses of 3 mm-thick bars have not revealed any regions with positive birefringence though this is not inconsistent with the results presented by E. F. T. White *et al.*[13] as they found the presence or absence of positive regions to be dependent on moulding conditions. It seems to be indicated, therefore, that in the thinner bars orientation effects often dominate even in the centre of the bar. Finally our studies of the effects of annealing and of deformation on birefringence appear to demand that the highly polarizable phenyl residues have the ability to take up different orientations without the need for backbone conformational changes so that the birefringence does not bear a direct relationship to the state of molecular backbone orientation even when the stress optical contribution has been subtracted.[3,12]

One apparent anomaly that cannot be explained easily is the presence of a region of negative birefringence at the centre of the bar, between the two positive peaks, and we can only speculate that reorientation occurs during the later stages of cooling as a result of the very large stresses that build up before the central region is completely solidified. (The stress optical coefficient is negative in the melt state.)

Although the compressive stress directed parallel to the bar axis near the surface will give a negative contribution to the birefringence, we believe that there is also a contribution from frozen-in orientation in the Izod bars. In the specimens used for the study reported here there were no shear bands visible, and this must set an upper limit on the compressive residual stress, probably between 50 and 70 MN/m^2, though the studies from which these figures were taken were not conducted on polystyrene in injection-moulded form.[32,33]

CONCLUSIONS

This study has shown that the measurement of residual stresses is not easily accomplished in thick injection mouldings. We feel there is scope for improving the layer removal technique but have not yet had the opportunity to attempt to overcome the difficulties experienced during this preliminary investigation. The stress relaxation analysis demonstrated that the Kubât and Rigdahl procedure can be applied to uniaxial compression data but the parameters so found do not correlate with those obtained from tensile tests and are not very helpful in isolation. Birefringence may yet prove to be a useful technique despite the difficulty of separating the contributions of orientation and of stress respectively. An added complication here is the time-dependent change in birefringence of polystyrene when under load, a property thought to be associated with phenyl group re-orientation.[3]

The normal compressive–tensile–compressive stress distribution for the skin–core–skin section was deduced to be present in the thick (Izod) bars with the stress magnitudes possibly large fractions of the values required to produce shear bands (compression) or crazes (tension) respectively.

Acknowledgements

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