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The effect of annealing on fracture behaviour of polycarbonate

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Brittle fracture of polycarbonate (PC) caused by thermal pretreatment, is discussed in this paper. Further results on the thickness effect provide added support for the earlier work on this aspect. These results together with the temperature effect on the fracture of polycarbonate give a complete picture of brittle fracture in a tough material.

1. Introduction

The brittle fracture in polycarbonate (PC) with regards to thickness and temperature has already been investigated [1, 2]. To anticipate the experimentally observed thickness effects, it was postulated that the apparent fracture toughness, K_c, measured in single-edge notched (SEN) tests is made up of contributions from K_{1c}, the plane strain fracture toughness, relevant to flat fracture thickness, $H-2r_y$, and K_{2c}, plane stress fracture toughness relevant to shear lips thickness, $2r_y$, (H is the specimen thickness, $r_y = 1/2\pi(K_{2c}/\sigma_y)^2$ is the extent to which the plane stress effect penetrates the thickness). K_c can then be determined by apportioning the K_{1c} and K_{2c} values to $H-2r_y$ and $2r_y$, respectively, so that:

$$HK_c = (H-2r_y)K_{1c} + 2r_yK_{2c} \tag{1}$$

or

$$K_c = K_{1c} + \frac{2r_y}{H}(K_{2c} - K_{1c}) \tag{2}$$

K_c is the measured value of fracture toughness from a fracture test. K_{1c} is the measured K_c for very large thicknesses which were not commercially available in the UK at the time this work was reported. K_{1c} and K_{2c}, however, were computed from two sets of data from SEN and surface notch (SN) tests (the SN specimen has an effective thickness $H \simeq 4b/3$ much greater than that of the nominal sheet thickness [1]; $2b$ is the surface crack width) by using Equation 2 as $K_{1c} = 2.2\,\text{MN m}^{-3/2}$ and $K_{2c} = 5.7\,\text{MN m}^{-3/2}$. Careful observation of the crack front in SEN specimens showed that the crack started growing* in a bow shape and an initiation value of $K_{\text{init.}} = 2.24\,\text{MN m}^{-3/2}$ was found by taking the load at crack initiation and using the original crack length. This value is almost identical with the calculated K_{1c}. This is expected, since $K_{\text{init.}}$ represents fracture in the central region arrested by the plane stress region.

The thickness dependence governed by Equation 2 has also been observed at different temperatures [2].

In the present work the measurement of K_{1c} using compact tension specimens together with the effect of annealing on the fracture behaviour, are discussed.

2. Determination of K_{1c} using compact tension (CT) specimens

Tensile tests were performed on standard CT specimens with $B = 0.5\,w$ (Fig. 1) in accordance with the suggestion of Brown and Srawley [3]. CT specimens of dimensions 30 mm × 28.8 mm and of 12 mm thickness were machine notched by a very sharp fly cutter. The produced cracks were sharp and as material was simply removed by the fly cutter no crazing of plastic deformations were produced at the crack tip. All the specimens loaded on an Instron Universal Testing Machine at $0.6\,\text{cm min}^{-1}$ cross-head speed exhibited brittle fracture. The K_{1c} were calculated using the boundary collocation solutions of Brown and Srawley [3]

$$K = \sigma\sqrt{a}\,Y \tag{3}$$

*Dr R. Kambour, General Electric Company, Schenectady, New York, believes that this is, in fact, craze initiation.

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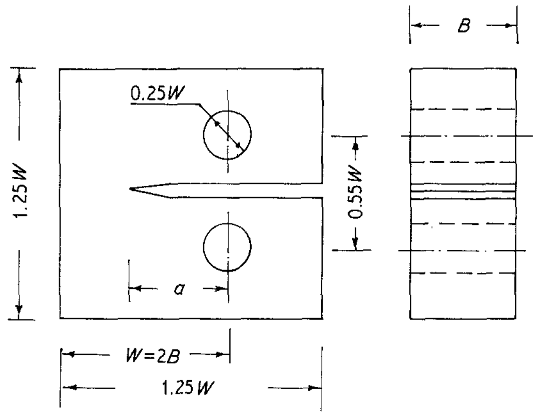

Figure 1 Compact tension specimen – standard proportions.

where

$$Y = 29.6 - 185.5\left(\frac{a}{W}\right) + 655.7\left(\frac{a}{W}\right)^2 - 1017\left(\frac{a}{W}\right)^3 + 638.9\left(\frac{a}{W}\right)^4$$

where σ is the fracture stress and a is the crack length. A $K_{1c} = 2.2\,\mathrm{MN\,m^{-3/2}}$ is indicated in the plot of $\sigma^2 Y^2$ versus $1/a$ for both annealed and unannealed specimens as shown in Fig. 2. This value is in excellent agreement with computed values from Equation 2 [1, 2]. Frazer and Ward [4] have also used CT specimens to measure the overall (apparent) fracture toughness. For 6.48 mm thick specimens at room temperature they have calculated a value of about $1\,\mathrm{MN\,m^{-3/2}}$ for K_{1c} from craze shape and a value of about $7.8\,\mathrm{MN\,m^{-3/2}}$ for K_{2c} from the same model as proposed in [1]. At lower temperatures the values of overall fracture toughness have been plotted versus the inverse specimen thickness and K_{1c} and K_{2c} were found by extrapolating the obtained straight line

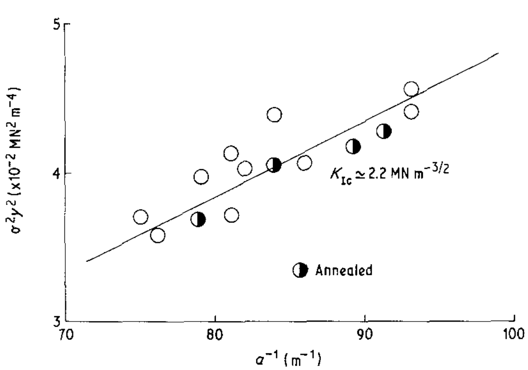

Figure 2 Compact tension specimen results.

to infinite thickness and shear lips width, respectively. The K_{1c} values obtained by Frazer and Ward are lower than expected. For example, K_{1c} at room temperature is very close to those of polystyrene and PMMA which are more brittle and do not show any thickness dependence.

3. Effect of annealing on the fracture behaviour of polycarbonate

3.1. Experimental procedure

Rectangular PC specimens of dimensions 150 mm by 50 mm by 3 mm or 5 mm were annealed in an air-flow oven at 130° C for 5 to 250 h and then cooled at a slow rate ($5°\,\mathrm{C\,h^{-1}}$). Tensile tests on both SEN and SN specimens were performed on an Instron Universal Testing Machine at ambient temperature. Single-edge notches were induced both by razor blades and by machining by a very sharp fly cutter. Surface notches were induced by machining.

3.2. Yield stress results

The tensile yield stress of a dumb-bell shaped specimens was determined at different strain rates, as shown in Fig. 3.

3.3. Stress intensity factor solutions

(a) For a *single-edge notch* the stress intensity factor, K, as described by Brown and Srawley [3], is given by

$$K = \sigma a_0^{1/2} Y, \tag{4}$$

where σ is the applied stress, a_0 is the crack length and Y is the correction factor.

(b) For a *surface notch* the stress intensity factor, K, as described by Irwin [5], is given by

$$K = \sigma d^{1/2} Y, \tag{5}$$

where d is the crack depth and Y is the correction factor.

3.4. Results of 3 mm and 5 mm specimens

Both SEN and SN specimens exhibited brittle fracture at different cross-head rates. Crack initiation in SEN specimens showed the same characteristic as that seen in unannealed specimens. The fracture toughness, K_c, for SEN specimens at final fracture was determined from a plot of $\sigma^2 Y^2$ against a_f^{-1}, as shown in Fig. 4, where a_f includes the slow crack growth, giving $K_c \simeq 4.34\,\mathrm{MN\,m^{-3/2}}$ for specimens of 3 mm thickness and $K_c \simeq 3.87\,\mathrm{MN\,m^{-3/2}}$ for specimens of 5 mm thickness. Then, using Equation 2 with $\sigma_y = 69.75\,\mathrm{MN\,m^{-2}}$, values

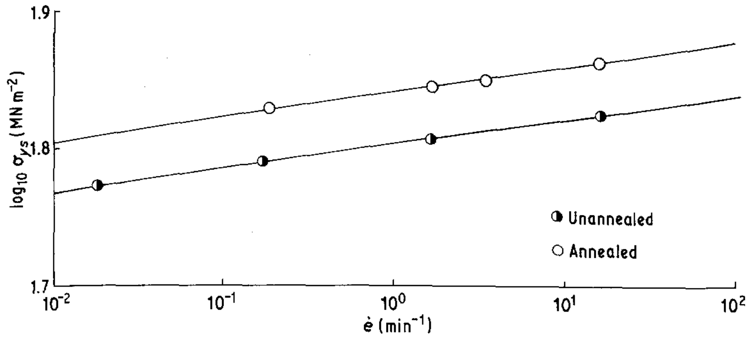

Figure 3 Variation of tensile yield stress with strain rate.

of $K_{2c} = 5.5$ and $5.88\,\mathrm{MN\,m^{-3/2}}$ (from 3 and 5 mm thickness data, respectively) will be deduced. K_{2c} values for both thicknesses are close, indicating that the fracture toughness varies with thickness according to Equation 2.

Brittle fracture in SN specimens occurred even for shallow notches, and ductile fracture occurred only for $d < 0.3$ mm. When data are plotted as $\sigma^2 Y^2$ versus $1/d$ (no slow crack growth was observed during loading), as shown in Fig. 5, an average of $2.65\,\mathrm{MN\,m^{-3/2}}$ is indicated by both thicknesses. The variation of fracture stress versus crack depth is shown in Fig. 6.

It can be shown that the postulated model (Equation 2) responds to thickness variations in the same way that the experimental results do. The form of the curve in Fig. 6 can be predicted from Equation 2 using SEN data and considering the change of SN effective thickness H with d. The broken line in Fig. 6 is the predicted line, and is in good agreement with the experimental results.

3.5. Discussion on annealing

The major conclusions to be drawn from these tests is that annealing increases the degree of

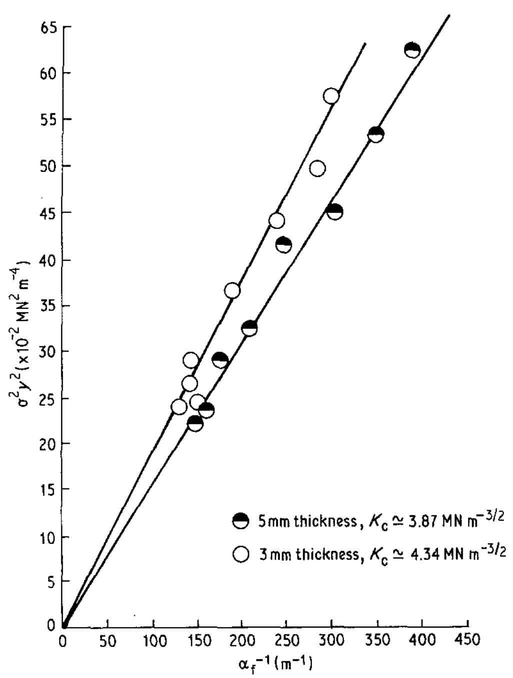

Figure 4 3 and 5 mm SEN annealed PC results at final fracture – cross-head speed = 0.5 cm min^{-1}.

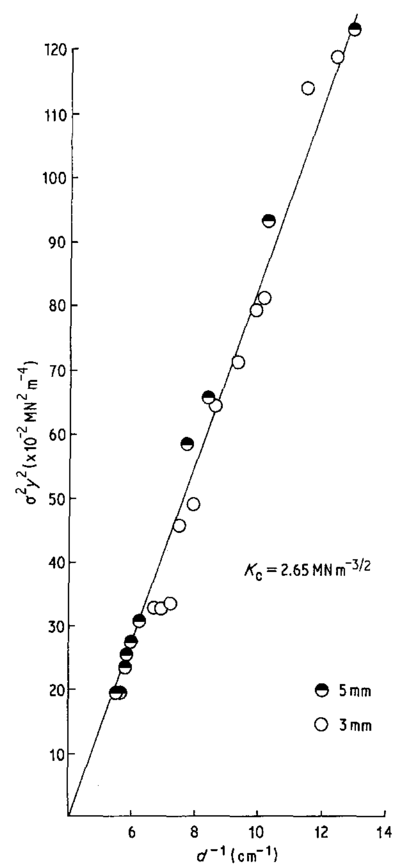

Figure 5 3 and 5 mm SN annealed PC results at a cross-head speed of 0.5 cm min^{-1}.

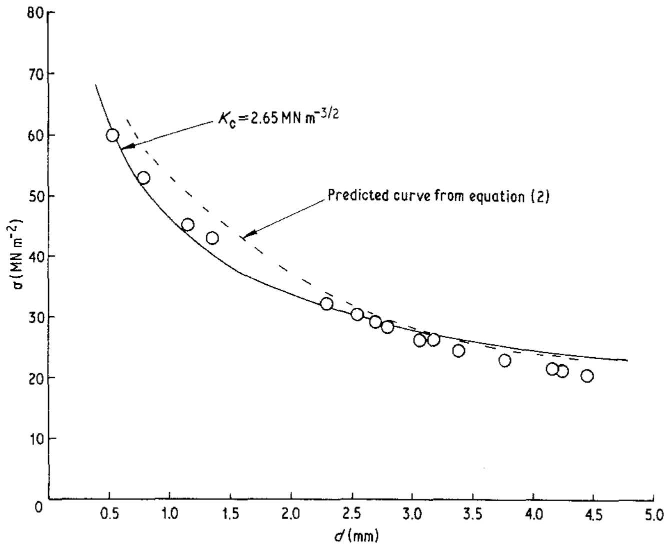

Figure 6 Variation of maximum stress with crack depth for 5 mm SN annealed PC at a cross-head speed of 0.5 cm min^{-1}.

brittleness, and must be considered in any study of the ductile–brittle transition.

The 3 mm single-edge notch specimens of unannealed polycarbonate exhibited ductile failure with gross yielding and necking over the range of the cross-head speed of the testing machine (0.005 to 50 cm min^{-1}) so that no K_c criterion could be considered [1]. However, the 3 mm SEN specimens became completely brittle when annealed below their glass transition temperature. The 5 mm thickness SEN specimens of unannealed polycarbonate exhibited a duplex behaviour (both gross ductile and brittle fracture) [1], while the 5 mm annealed specimens were always brittle with a K_c value lower than that of unannealed specimens (3.87 MN m$^{-3/2}$ compared to 4 MN m$^{-3/2}$ of unannealed specimens). This transition has also been observed in Izod and Charpy impact studies of notched samples of annealed polycarbonate [6–10]. The physical explanation for these transitions requires a complete study of any possible change in material properties. Yield stress of annealed specimens are higher than that of the unannealed ones, as shown in Fig. 3, and this may well have some influence on the change in behaviour. The increase in the yield stress has also been reported by many other investigators [8–13]. Annealing increases the density and produces an endothermic peak at glass transition temperature, T_g, as measured on a differential scanning calorimeter [6, 14, 15]. Golden *et al.* [11] have suggested that the preheat treatment of polycarbonate in the temperature range 80 to 130° C produces a greater degree of order within the amorphous region, resulting in an increase in strength. No effect of cooling rate on the fracture behaviour of PC was observed in the present work. The specimens which were cooled immediately by immersing in iced water showed the same fracture behaviour as those cooled at a slow rate (5° C h^{-1}). Increasing the annealing time from 5 h to 2 weeks has also no effect on fracture behaviour. Annealing temperature was found to be a governing factor since annealing below 100° C produced no change in fracture behaviour.

Although both SEN and SN tests on annealed PC result in apparent brittle fracture, a thickness effect still prevails which can be well explained by the proposed model expression by Equation 2.

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