ASSESSING THE EFFECTIVENESS OF TOUGHENING AGENTS

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A technique has been developed for studying the inherent fracture properties of polymeric materials containing toughening agents. It was important to achieve good isolation between crack initiation and crack propagation in specimens of material that could be precisely loaded and allowed to stress relax before being fractured. In this study, the fracture resistance of acrylonitrile-butadiene-styrene (ABS) is compared with styrene-acrylonitrile (SAN).

INTRODUCTION

Polymers exhibit widely different degrees of stress relaxation and this needs to be considered in evaluating the crack propagation resistance of these materials. It is important, therefore, to be able to control precisely the loading and stress relaxation of specimens before a crack is initiated. It is essential that the established loading conditions are not disturbed by the crack initiation process. Also, the only energy available to the crack tip must be that stored in the specimen. For polymers that have good thermal insulation properties, then, the above requirements can be met using the frozen tongue technique devised by Dear (1). A small tongue of material extends the intended crack path in a rectangular specimen which is loaded normal to the crack path. Liquid nitrogen cooling is used to embrittle the tongue so that it is easy to fracture with a low force and launch a crack into the main section of the specimen. The frozen tongue technique was originally devised to study polyethylene and then developed for researching the fracture properties of polycarbonate, polymethylmethacrylate and similar materials (Dear and Ibru (2)). Further developments of the frozen tongue technique were needed to research the fracture properties of ABS materials. The first part of this research was to make these new developments to the fracture technique and then to move onto compare the fracture properties of ABS with SAN, the latter being the host material for the rubber particles.

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EXPERIMENTAL

The general arrangement of the frozen tongue specimen and the device used to initiate cracks in the tongue section, after it had been freeze-cooled with liquid nitrogen, is shown in Figure 1. A problem with the frozen tongue of the ABS material was that it had a propensity to shatter rather than fracture along the intended crack path. This was overcome by devising a new freezing method which only cooled the material along the crack path in the tongue. Figure 2 shows the new freezing device which consists of two metal blocks with triangular protrusions to fit into the side-grooves of the tongue. The metal blocks were cooled in liquid nitrogen and then lightly clamped into position on the tongue of the specimen. In this way, the crack path in the tongue was quickly frozen without reducing the temperature of the whole of the tongue or the main section of the specimen for more than 5 mm along the crack path. Also, it was verified that when fracturing the tongue, with the three-point bend device, there was negligible disturbance of the loading of the main section of the specimen. Another assessment made was that the disturbance of the stress field in the main section of the specimen, by the presence of the tongue, was confined to a region of 10 mm in extent from the root of the tongue.

For this study, a tongue was provided at both sides of the specimen as shown in Figure 3 so that the crack could be initiated from either side of the specimen. Also, it was convenient for some experiments to arrest the crack after it had severed the main section of the specimen and the second tongue was used for this purpose. To monitor the build up and then constant crack velocity, as the crack length in the main section of the specimen increased, a series of conducting strip monitors were located along the crack path as in Figure 4. It was found important to achieve a sharp and clean breaking of the conducting strips as the crack opened. This required the ability to form thin conductive strips across the crack path which had to be strong enough not to be damaged when handling the specimen and mounting it in the Instron loading machine. Each strip had a graphite resistor and this was also formed on the specimen using a masked spraying technique. Two separate sets of conducting strip monitoring were formed with one on each side of the specimen to obtain confirmatory outputs. The conducting strips and their graphite resistors were incorporated into a Wheatstone bridge network. As each conducting strip was broken by the propagating crack, this changed the balance of the Wheatstone bridge to produce a stair-case stepping of the voltage output. These outputs from the Wheatstone bridge were monitored using a Digital Storage Scope (Gould Instrument Systems - DataSYS 740) which was equipped with facilities for transfer of the captured data to a Personal Computer (PC) for processing. In addition, a completely separate monitoring of the crack tip was achieved by using a high-speed camera (Hadlands - IMACON 468). This was equipped with an optical fibre link for transfer of the photographic images to a PC. These photographic crack velocity data were correlated with those from the conducting strips.

RESULTS

Side-grooves were introduced of depth 1mm on each side of the specimens of thickness (B) 6mm for all the frozen tongue ABS and SAN specimens. Figure 5 shows a loading curve for an ABS specimen and a similar one was obtained for the SAN specimens. The load was applied at a rate of 5mm min^{-1}, in an Instron machine, up to the required load level and then the Instron cross-head was stopped.

After a period of time to allow the required stress relaxation, the frozen tongue technique was used to initiate a crack. Figure 6 shows an example of a high speed photographic sequence of fracture in an ABS specimen to confirm crack velocities and other measurements made by the conducting strip method. Back-lighting of the specimen was used so that the crack appeared as a bright streak of light passing through the specimen from left to right. Figure 7 shows a comparison of the crack velocity, da/dt, versus stress relaxed load, p, for ABS and SAN both having a thickness (B) of 6mm. To these experimental data for ABS and SAN, relationships were fitted of the form:

$$(da/dt)^2 = C_L^2\{1 - (p_0^2/p^2)\} \quad \ldots\ldots\ldots\ldots\ldots \quad (1)$$

where C_L is the limiting crack velocity and p_0 is the threshold stress relaxed load for crack propagation. Figure 8(a) presents the crack velocity, da/dt, versus stress relaxed load, p, for the ABS material in a different way as a plot of $(da/dt)^2$ versus $(1/p)^2$ and Figure 8(b) shows the same plot for the SAN material. The comparison of Figures 8(a) and 8(b) reveals that the two materials do not have a common gradient of $(da/dt)^2$ versus $(1/p)^2$ as is the case for example for different toughness grades of polyethylene as observed by Dear and Williams (3). The toughness, R, can be determined from the following equation as reported in reference (3):

$$R = (\pi p_o^2) / (4EBB_n D) \quad \ldots\ldots\ldots\ldots\ldots \quad (2)$$

where D is the specimen width, B is the overall thickness of the specimen and B_n is the crack thickness after side-grooving. The value of E used was determined by longitudinal and transverse wave-velocity measurements and for an excitation frequency of 2 MHz, the E - value for ABS was 2.7 GPa and for SAN it was 3.3 GPa. The stress intensity factor, K, was determined from the relationship:

$$K = (ER)^{1/2} \quad \ldots\ldots\ldots\ldots\ldots \quad (3)$$

Table I summarises the fracture parameters for ABS and SAN determined by the frozen tongue technique.

TABLE 1 - Frozen tongue fracture parameters for ABS and SAN.

	p_o (kN)	C_L (m s^{-1})	E (GPa)	R (kJ m^{-2})	$K = (ER)^{1/2}$ (MPa m$^{1/2}$)
ABS	8.2	495	2.7	9.1	5.0
SAN	1.6	869	3.3	0.3	1.0

DISCUSSION

The indications are that the dispersed rubber particles are more effective in improving the toughness of ABS at low rather than high crack velocities. This fits in well with the research findings of Donald and Kramer (4) who showed cavitation processes in rubber particles when the ABS material was subject to a high stress concentration. These cavitation and other particle toughening processes in ABS will have finite response times and they will have more time to increase the crack tip toughness of the material at low rather than high crack velocities.

To obtain from small laboratory specimens definitive information about a material's resistance to crack initiation and crack propagation presents many problems. A major difficulty is achieving good isolation between crack initiation and crack propagation conditions. This is particularly so for rubber toughened and other soft particle toughened materials. A feature of the frozen tongue technique is that it also tends to inhibit the toughening effect of these particles in the tongue section making it easier to start a crack in ABS.

CONCLUSIONS

The frozen tongue technique has provided several useful methods of obtaining fast fracture data from small specimens of tough polymeric materials. Fast crack propagation information, which has been obtained from experiments, where there has been a good isolation between crack initiation and propagation, is particularly useful as these fracture data relate more closely to the inherent properties of the material. Obtaining these fracture data, from conveniently small specimens, is very helpful when researching the effect of making small changes to an existing material or developing a new material.

ACKNOWLEDGEMENTS

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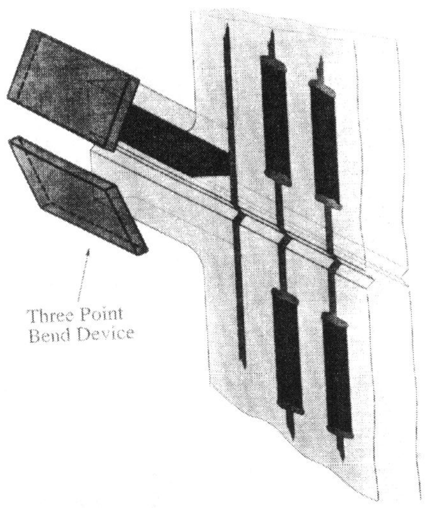


Figure 1 Three-point bend initiation device around the tongue.

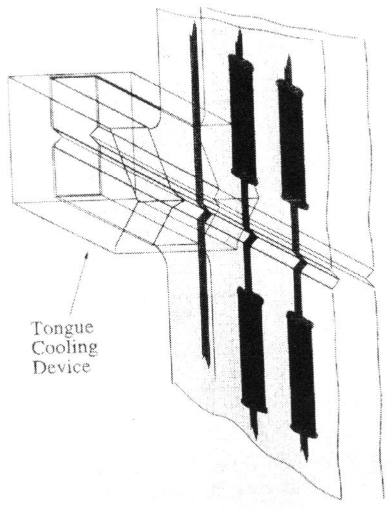


Figure 2 The device used to cool the side-grooved section of the tongue.

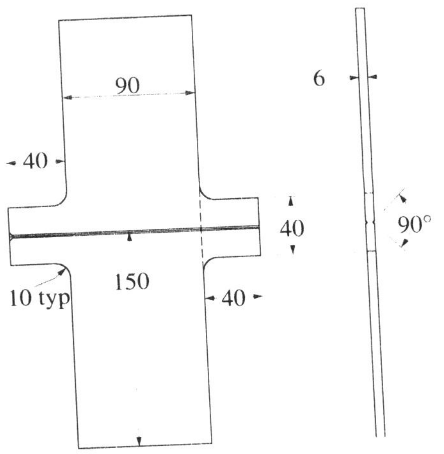


Figure 3 Frozen tongue specimen with dimensions in mm.

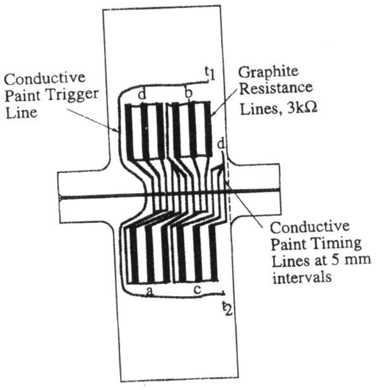
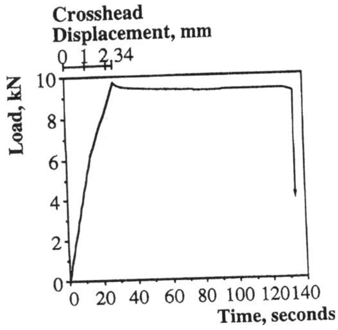


Figure 4 Positioning of conducting strips on frozen tongue specimen.



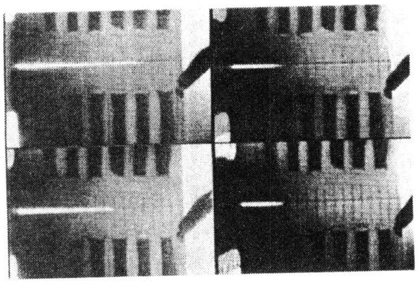
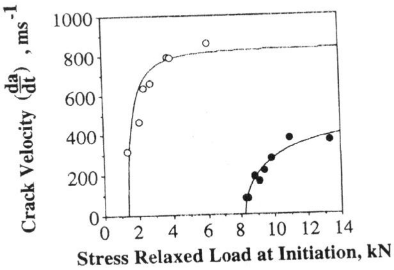


Figure 5 Load-time trace for ABS showing stress relaxation.

Figure 6 IMACON 468 high-speed sequence of fracture of ABS.



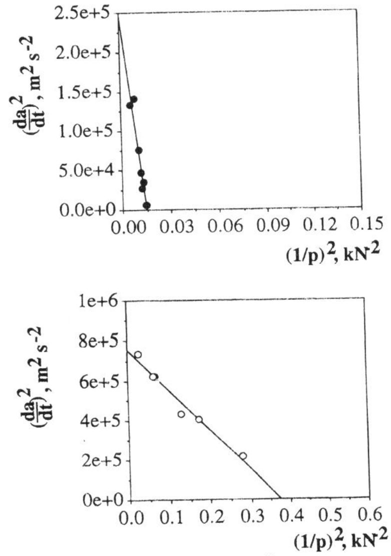


Figure 7 Crack velocity,*da/dt* , versus load,*p*, for ABS(•) and SAN(o).

Figure 8 Plots of *(da/dt)* ^{2} versus *(1/p)* ^{2} for (a) ABS(•) and (b) SAN(o).