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# Effects of pre-cracking methods on fracture behaviour of an Araldite-F epoxy and its rubber-modified systems

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The fracture behaviour of an Araldite-F epoxy and its rubber-modified systems was evaluated using compact tension specimens pre-cracked by three methods, namely, razor blade pressing, razor blade tapping and fatigue pre-cracking. The results show that the razor blade tapping method produces a lowest critical stress intensity factor, $K_{Ic}$, while the razor blade pressing produces an abnormally high $K_{Ic}$, being about five times higher than the former for the pure epoxy. Transmission polarized optical microscopy reveals that the crack tip produced by razor blade pressing in the pure epoxy specimen was completely surrounded by a plastic deformation zone with compressive residual stress, but the crack tip produced by razor blade tapping was free of residual stress and plastic deformation. It was found that the sensitivity of the fracture toughness value to the pre-cracking methods decreases after the pure epoxy was modified by 10% core-shell rubber or 10% liquid rubber.

## 1. Introduction

It is well known that the notch root radius in metal materials has to be much smaller than the half-value of the critical crack-tip-opening displacement (CTOD) in order to obtain a true value of fracture toughness, and such a sharp crack can be normally achieved using a fatigue technique [1]. However, it is often difficult to create a fatigue crack in plastics because unstable crack propagation often occurs and leads to final fracture of specimens before a sufficiently long fatigue crack can be produced. Some other techniques, such as pressing and tapping a razor blade into the notch root of specimens, were applied to aid in creating a pre-crack.

Cayard and Bradley [2] applied four methods to pre-crack Lexan polycarbonate specimens and found that pressing a fresh razor blade into a specimen can produce a high compressive residual stress at the crack tip, which resulted in a high fracture toughness value of 3.8 $\mathrm{MPa\,m^{1/2}}$. The pre-crack tip sawn by a razor blade was free of residual stress and produced a lowest fracture toughness value of 3.35 $\mathrm{MPa\,m^{1/2}}$. Jones and Bradley [3] found for the polyethylene pipe material that the razor notching produced a value of $J_{Ic}$, being 33% higher than that measured using fatigue pre-cracked specimens. Marshall [4] showed for the polystyrene that both slow razor notching and impact razor notching produced multiple craze bundles at the crack tip, resulting in high values of fracture toughness for polystyrene, while fatigue pre-cracking led to a single craze at the crack tip and hence a low value of fracture toughness. Darwish *et al.* [5] and Mandell *et al.* [6] reported that for poly(vinyl chloride) the pre-crack by razor blade pressing produced adequate crack sharpness, compared with fatigue pre-cracks and those grown at low temperatures. Low and Mai [7] have investigated the effect of notch root radius on the impact toughness of GY250 epoxy and its rubber-modified systems. It was found that the impact toughnesses amounted to 14 $\mathrm{kJ\,m^{-2}}$ and 18 $\mathrm{kJ\,m^{-2}}$ for the pure and 15% rubber-modified epoxy specimens, respectively, with a notch root radius of 1.5 mm, while the fracture toughnesses of the pure and rubber-modified epoxies was only 0.2 $\mathrm{kJ\,m^{-2}}$ and 1.78 $\mathrm{kJ\,m^{-2}}$, respectively, for a very sharp notch.

Most previous studies on this topic were conducted using thermoplastic materials which are normally much tougher than brittle thermoset epoxies. In this study, the influences of different pre-cracking techniques on the evaluation of fracture toughness of a standard epoxy and its two rubber-toughened systems were investigated, and differences in fracture mechanisms were identified.

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## 2. Experimental procedures

### 2.1. Materials

The basic material used in this study is a diglycidyl ether of bisphenol A (DGEBA) epoxy resin, Araldite-F, produced by Ciba-Geigy, Australia, and it is a high-viscosity standard base resin for general use. The curing agent was piperidine, mixed with the pure epoxy at a weight ratio of 5:100. Rubber-toughened epoxies were produced by adding core-shell rubber (CSR) in a powder form, supplied by Rohm and Haas Co., USA, or liquid rubber (LR), CTBN 1300 × 13, supplied by BF, Goodrich, USA. The average particle diameter for core-shell rubber is about 0.2–0.4 μm. The CSR was first dried in an oven at 80 °C for 2 h and then mixed with the pure epoxy using a mechanical mixer to achieve a uniform blend. The blend was degassed in a vacuum oven (−100 kPa) at 100 °C for about 2 h. Then the vacuum was removed and piperidine was added to the mixture while stirring slowly, which was cast into a pre-heated mould and cured at 120 °C for 16 h, followed by gradual cooling at ambient temperature. For the LR-modified epoxy, the same procedure was applied except the drying at first step. The thickness of the plates after curing was 12 mm. Standard tensile specimens were cut from the plates to evaluate the strain–stress behaviour and Young's modulus of the materials, as described in American Society for Testing and Materials (ASTM) Standard D 638M-91a. Compact tension specimens, based on ASTM Standard D 5045-93, were machined from these plates, with the specimen width, $W$, and the specimen thickness, $B$, being 50 mm and 12 mm, respectively (Fig. 1). The thickness, which is twice that normally adopted for compact tension specimens of polymers in the literature, was chosen in order to meet the plane-strain condition for the rubber-modified systems, i.e., the thickness must be larger than 2.5 $(K_{Ic}/\sigma_y)^2$ [8].

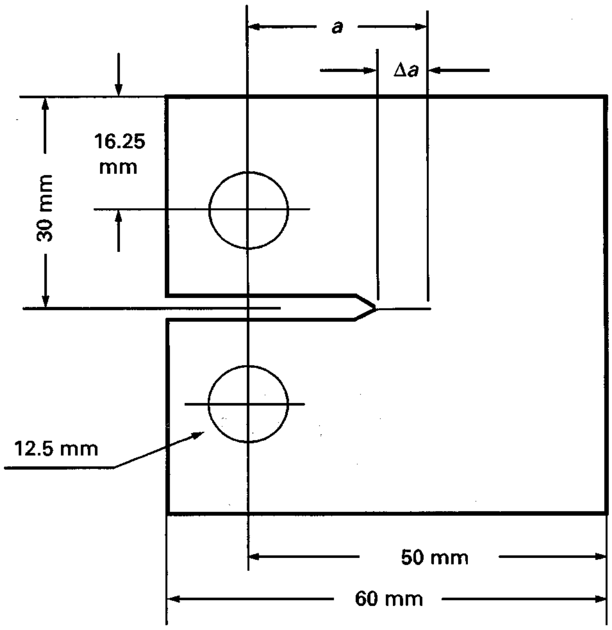

*Figure 1* Schematic diagram of a compact tension specimen.

### 2.2. Razor blade pressing

The pre-crack was made in the specimens using a guillotine-like apparatus with a fresh razor blade driven by a screw with 1 mm pitch. This apparatus can easily control the length of pre-crack driven into the machined notch tip of specimens. The obtained pre-crack length, $\Delta a$, from the machined notch tip in all specimens is approximately 0.5 mm.

### 2.3. Razor blade tapping

A specially designed clip was used to firmly hold two ends of a fresh razor blade; then the machined notch tip of specimens was placed over the edge of the razor blade. A hammer was used to tap the specimen slightly. Great attention has to be paid to avoid forming a long crack or breaking the specimen. Specimens with a long crack, i.e., the value of $a/W$ exceeding 0.55, were discarded.

### 2.4. Fatigue pre-cracking

Fatigue pre-cracking was performed on an Instron 8501 servohydraulic testing machine under load control with the load ratio being 0.2. The maximum load was set to be 250 N, which meets the requirement of $K_{max}$ and $K_{max}/E$ as prescribed by ASTM Standard E 399-90. In order to avoid possible hysteretic heating, which usually exists in plastics, a frequency of 3 Hz was applied. However, the pure epoxy specimens cannot be easily pre-cracked using this method because of unstable fatigue crack growth, although various fatigue testing parameters were tried.

## 3. Results and discussion

The tensile specimens were tested in accordance with ASTM Standard D 638M-91a, and typical stress–strain curves for the pure and rubber-toughened epoxies are shown in Fig. 2. The mechanical properties are summarized in Table I. Both Young's modulus and strength at yield (or maximum strength) clearly

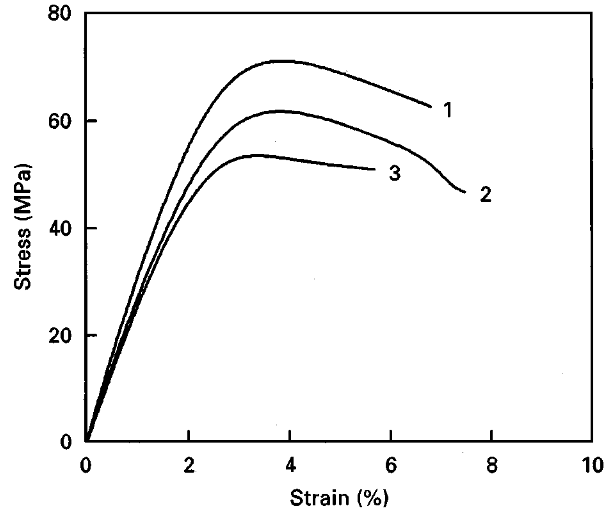

*Figure 2* Typical stress–strain curves for pure and rubber-toughened epoxies. Curve 1, pure epoxy; curve 2, 10% LR modified; curve 3, 10% CSR modified.

TABLE I Mechanical properties of pure and rubber-toughened Epoxies: $\sigma_T$, tesile strength at yield (or maximum tensile strength); $\sigma_t$, tensile strength at break; $\varepsilon_t$, Elongation at break; $E$, Young's modulus

| Resin system | $\sigma_T$ (MPa) | $\sigma_t$ (MPa) | $\varepsilon_t$ (%) | $E$ (GPa) |
|---|---|---|---|---|
| Araldite-F | 72 | 61 | 6.7 | 3.04 |
| Araldite-F + 10% LR | 62 | 47 | 7.5 | 2.67 |
| Araldite-F + 10% CSR | 54 | 48 | 5.6 | 2.54 |

decreased after the pure epoxy was modified with rubber, but all systems clearly illustrate a non-linear stress–strain response before final failure.

The specimens with the pre-cracks produced by three different methods were tested at ambient temperature at a cross-head rate of 0.5 mm $\text{min}^{-1}$ on an Instron 5567 testing machine. All tests were carried out with at least three specimens. Fig. 3 depicts the typical load–extension curves for compact tension specimens with pre-cracks obtained by different pre-cracking methods. One of most essential characteristics induced by the different pre-cracking methods is that the maximum load was remarkably reduced in specimens pre-cracked by razor blade tapping, especially for the pure epoxy specimen. The crack growth behaviour also changed because of the different pre-crack tip states. The crack grew in an unstable manner in the pure epoxy specimens with razor blade pressing but, for the specimens with razor blade tapping, continuous crack growth was observed after the crack finished its first jump. This type of crack growth can be termed transition from brittle unstable crack growth to brittle stable crack growth according to the work by Kinloch *et al.* [9]. For the LR-modified epoxy, brittle unstable crack growth was observed in specimens with razor blade tapping or pressing. For the CSR-modified epoxy, ductile stable crack growth, followed by unstable crack growth, can be clearly seen in specimens with razor blade pressing or fatigue pre-cracking in Fig. 3c, but the crack grew in an unstable manner in the specimens with razor blade tapping.

The crack length was measured using a micrometer with a resolution of 0.01 mm and the average crack length was calculated using a nine-point averaging technique on the post-fracture surface, described in ASTM Standard E 813-90. The critical stress intensity factor, $K_{\text{Ic}}$, was calculated using [8],

$$K_{\text{Ic}} = \frac{P_{\text{Q}} f(a/W)}{BW^{1/2}} \qquad (1)$$

where $P_{\text{Q}}$ is the critical fracture load, which in the present study corresponds to the maximum load, $B$ is the specimen thickness, and $f(a/W)$ is a non-dimensional geometry factor given by

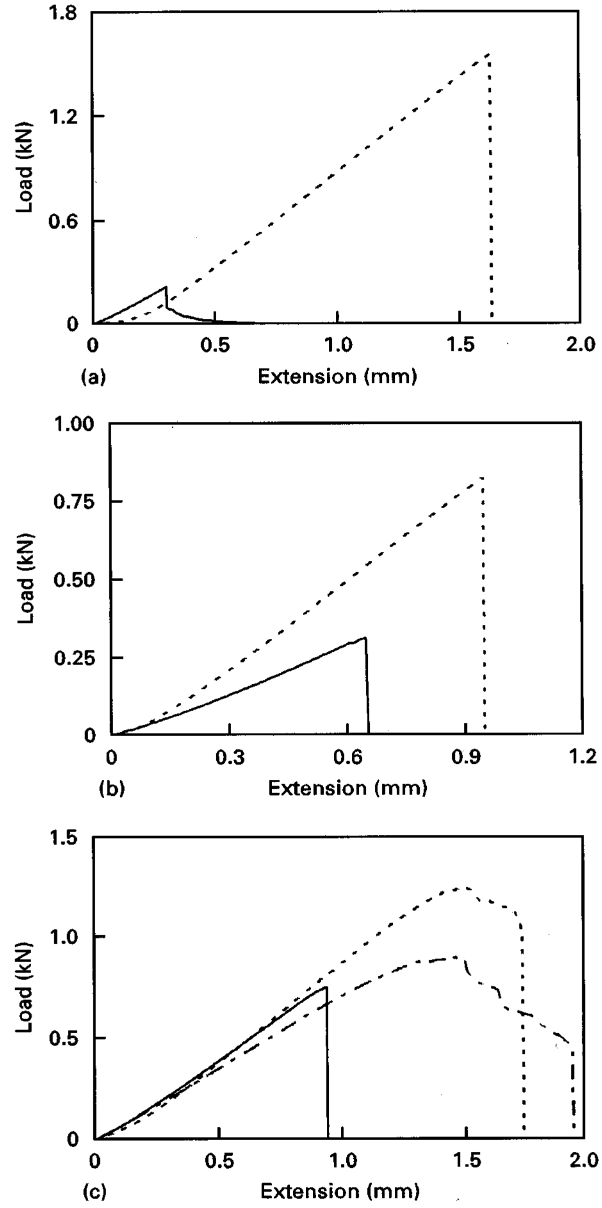

*Figure 3* Typical load–extension curves for compact tension specimens with different pre-cracking methods: (a) pure epoxy; (b) 10% LR modified; (c) 10% CSR modified. (——), razor blade tapping; (----), razor blade pressing; (---), fatigue pre-cracking.

Fig. 4 shows the fracture toughness values for both pure and rubber-modified epoxies. For the pure epoxy specimens, $K_{\text{Ic}}$ with razor blade pressing can be abnormally as high as 4.1 MPa $\text{m}^{1/2}$, which is 4.7 times larger than that with razor blade tapping, as shown in Fig. 4a. This is obviously not a true value since most brittle epoxies usually have $K_{\text{Ic}}$ values not exceeding 1.0 MPa $\text{m}^{1/2}$. No actual value for the pure epoxy with fatigue pre-cracking was obtained owing to the unsuccessful attempts in producing test specimens. For the LR-toughened epoxy, the $K_{\text{Ic}}$ value with razor blade pressing is 100% higher than that with razor blade tapping. The $K_{\text{Ic}}$ value for the CSR-toughened epoxy

$$f\left(\frac{a}{W}\right) = \frac{(2 + a/W)[0.886 + 4.64a/W - 13.32(a/W)^2 + 14.72(a/W)^3 - 5.6(a/W)^4]}{(1 - a/W)^{3/2}} \qquad (2)$$

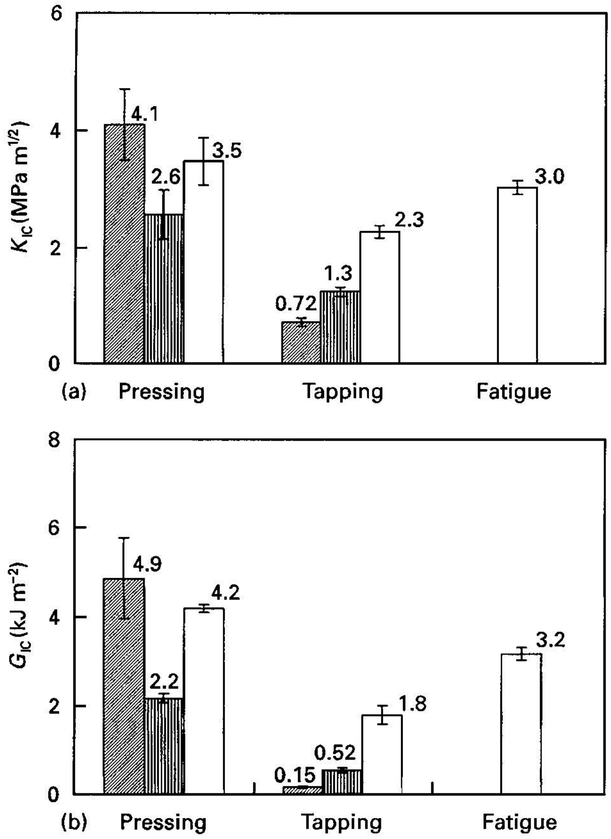

*Figure 4* Fracture toughnesses of pure and rubber-toughened epoxies as functions of pre-cracking methods: (a) fracture toughness, $K_{Ic}$; (b) fracture energy, $G_{Ic}$. (▨), Araldite-F; (▥), Araldite-F + 10% LR; (☐), Araldite-F + 10% CSR. The scatter bars indicate the standard deviations.

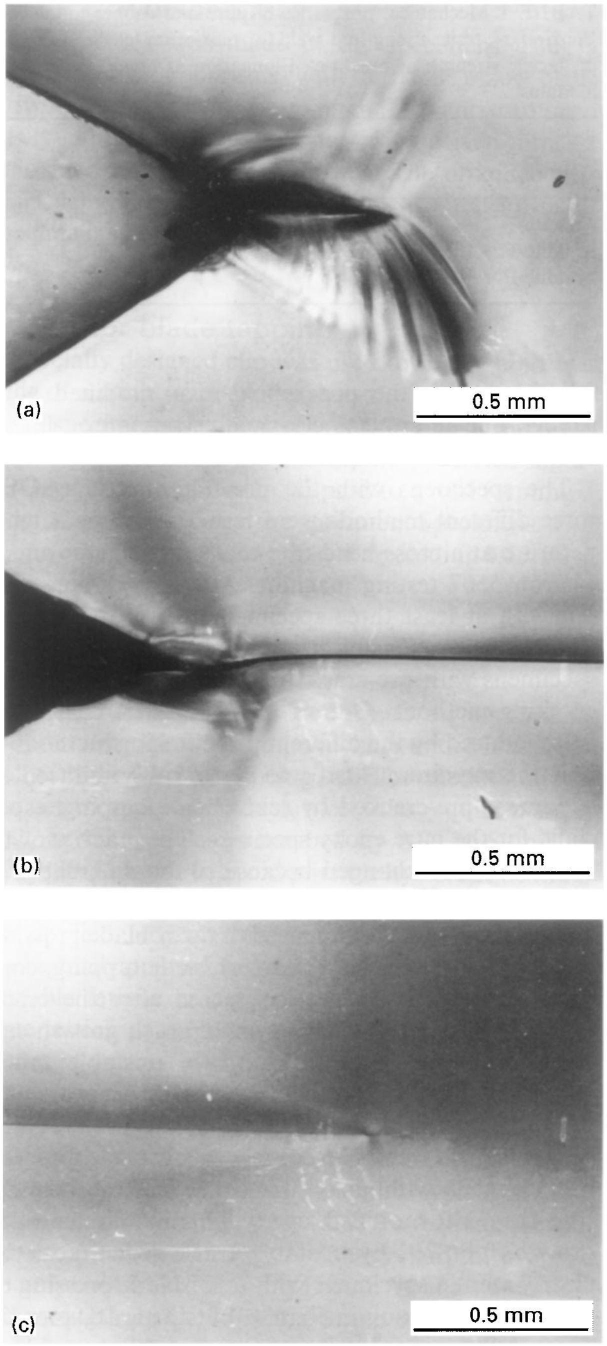

*Figure 5* TOM photographs of precrack in pure epoxy: (a) precrack with razor blade pressing: (b) crack root with razor blade tapping; and (c) crack tip with razor blade tapping.

using razor blade tapping amounted to be 2.3 MPa m$^{1/2}$, being lowest among the three methods used. $K_{Ic}$ with razor blade pressing is about 52% higher than that with razor blade tapping, and 17% higher than that with fatigue pre-cracking. The brittle pure epoxy was much more sensitive to the pre-cracking methods than the toughened epoxies for evaluation of fracture toughness, $K_{Ic}$.

The pre-cracking methods have a more significant influence on the value of critical strain energy release rate, $G_{Ic}$, shown in Fig. 4b. Assuming a plane-strain condition, $G_{Ic}$, is calculated using the following relationship [10]:

$$G_{Ic} = \frac{(1-\nu^2)\,K_{Ic}^2}{E} \qquad (3)$$

where $E$ and $\nu$ are Young' modulus and Poisson's ratio, respectively. In the calculation, $\nu$ is set to 0.35 for all materials. The $G_{Ic}$ value of the pure epoxy with razor blade pressing is about 32 times higher than that with razor blade tapping ($G_{Ic} = 150\ \mathrm{J\,m^{-2}}$). However, the $G_{Ic}$ values with razor blade pressing for the LR- and CSR-toughened epoxies are about 3.2 and 1.3 times, respectively, higher than those with razor blade tapping.

Transmission optical microscopy (TOM) with polarized light provides some useful information about the crack tip status and the residual stress distribution around the crack tip from the birefringence patterns. Thin sections of 20–30 μm thickness for TOM observation were prepared from the plane-strain core zone around the crack tip in the specimens using a similar procedure to that proposed by Takemori and Yee [11]. Fig. 5 shows TOM photographs of the pure epoxy specimens with razor-blade-pressed and razor-blade-tapped pre-cracks, respectively. It can be clearly seen that large butterfly-shaped patterns consisting of plastic deformation lines and fringe patterns completely surround the crack tip in the specimen pre-cracked by razor blade pressing, which means that this method induces not only residual stress but also plastic deformation around the crack tip. On the other hand, the fringe patterns were also observed at the root of the razor-blade-tapped pre-crack, but far away from the actual pre-crack tip which is completely free of both residual stress and plastic deformation.

The post-fracture surface of the pure epoxy specimen pre-cracked by razor blade pressing displayed many stretched lines scattering from the pre-crack front, as shown in Fig. 6a. On the contrary, the fracture surface in the razor-blade-tapped specimen was very smooth and mirror like. Much more energy was dissipated during crack growth in specimens with razor blade pressing, resulting in a high $K_{Ic}$ value.

Cayard and Bradley [2] reported that a compressive stress field existed around the crack tip in razor-blade-pressed Lexan polycarbonate specimens. Lexan polycarbonate is a more ductile material with a $K_{Ic}$ of 3.35 MPa $m^{1/2}$, compared with 0.72 MPa $m^{1/2}$ for the pure Araldite-F epoxy in this work. The corresponding $K_{Ic}$ was 13% larger than that of the specimen with a razor-sawn pre-crack, which is somewhat similar to a razor-tapped crack and produces a low $K_{Ic}$. Concerning the 4.7 times difference in $K_{Ic}$ values for the pure epoxy between razor blade pressing and razor blade tapping in this study, it can be assumed that the large amount of plastic deformation around the crack tip induced during pressing the blade is the main and the most essential cause for the abnormally high value of $K_{Ic}$ for the pure epoxy.

The scanning electron micrographs in Fig. 7 illustrate the crack tip states in 10% CSR-toughened epoxy specimens pre-cracked by three methods. The crack tip radius with razor blade tapping is smaller than with fatigue pre-cracking, because the crack popped-in several millimetres ahead of the razor blade tip at tapping, forming a very sharp crack. The largest crack tip radius was found in specimens pre-cracked by razor blade pressing.

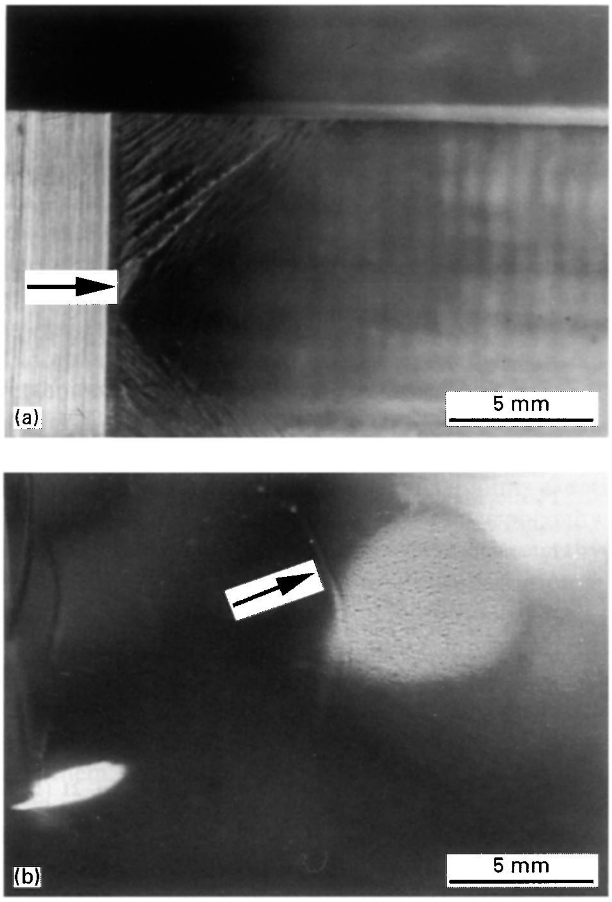

*Figure 6* Fracture surfaces of pure epoxy specimens (the arrows indicate pre-crack fronts): (a) razor blade pressing; (b) razor blade tapping.

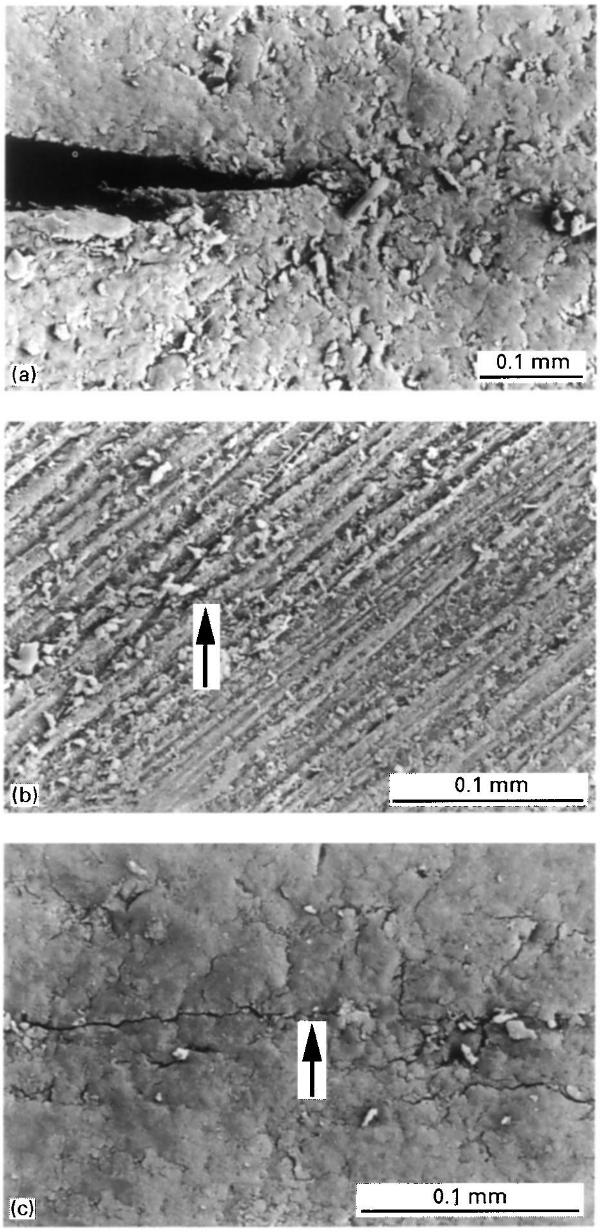

*Figure 7* Scanning electron micrographs of pre-crack tip in 10% CSR-toughened epoxies (the arrows indicate the pre-crack tips): (a) razor blade pressing; (b) razor blade tapping; (c) fatigue pre-cracking.

Reflection optical microscopy (ROM) reveals some microscopic characteristics of fracture surfaces of 10% CSR-toughened epoxy specimens pre-cracked by three different methods, shown in Fig. 8. Two typical regions, namely a slow crack growth region characterized by a stress-whitened zone and a fast crack growth region characterized by a relatively smooth fracture surface, are clearly visible. The stress whitening is considered to be the localized cavitation in rubber-modified epoxies, described in detail by Pearson and Yee [12] and by Garg and Mai [13]. The length of the stress-whitened zone decreases significantly when the pre-cracking method is shifted from razor blade pressing, to fatigue pre-cracking to razor blade tapping,

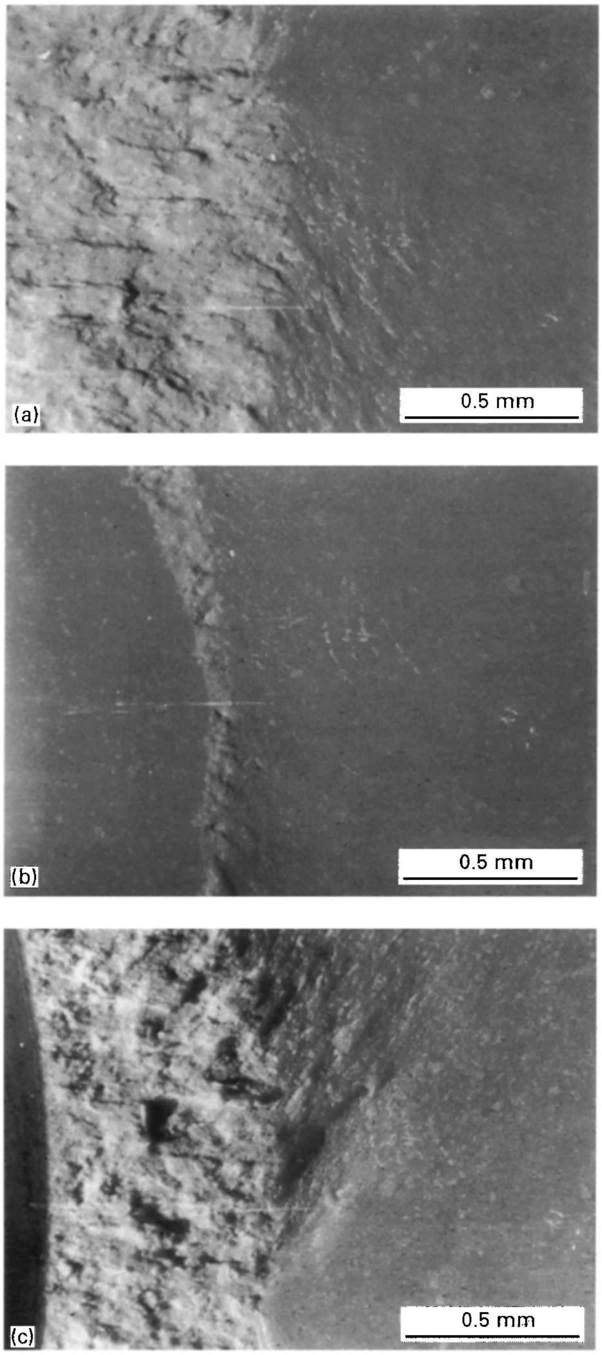

*Figure 8* ROM photographs of fracture surfaces of 10% CSR-toughened epoxy: (a) razor blade pressing; (b) razor blade tapping; (c) fatigue pre-cracking.

which completely coincides with the variations in $K_{\text{Ic}}$ or $G_{\text{Ic}}$ for the CSR-modified epoxy, as shown in Fig. 4. Furthermore, the topography in the stress-whitened zone becomes much finer for the specimen with razor blade tapping, shown in Fig. 8b.

## 4. Conclusions

The fracture behaviour of pure and rubber-toughened epoxies has been evaluated using three pre-cracking methods. From the results, the following points can be addressed.

1. The fracture behaviour of pure and rubber-toughened epoxies is very sensitive to the pre-cracking methods. Significant difference (up to 32 times) in fracture energy values was obtained for the pure epoxy resin. However, the rubber-toughened epoxies are less sensitive to the pre-cracking methods than the pure epoxy.

2. Razor blade tapping is an appropriate method to produce the pre-crack in both pure and rubber-toughened epoxy specimens; it produces the smallest pre-crack tip radius, hardly causes residual stress around the pre-crack tip and gives reasonable values of fracture toughness.

3. Razor blade pressing can induce a large amount of plastic deformation and residual stress around the pre-crack tip. It is believed that plastic deformation around the crack tip is the most essential factor responsible for the abnormally high value of fracture toughness.

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