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Fractography of polyester resins

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The effect of resin flexibility on the fracture surface characteristics of a series of polyester resins has been investigated by scanning electron microscopy. Fracture surfaces were obtained from fracture toughness specimens, un-notched tensile specimens and notched specimens cycled over a constant range of stress intensity factor. Characteristic surface markings have been identified with regions of stable crack growth, slow unstable crack growth and fast unstable crack growth. The three regions were only present on the surfaces of failed fracture toughness specimens. The extent of each region is a function of the resin flexibility. The absence of a given region on the fracture surfaces of other specimens is a function of the loading conditions.

1. Introduction

The effect of matrix properties on failure mechanisms in glass fibre reinforced polyesters has been investigated for conditions of single and repeated loading [1, 2]. The matrices were modified by the addition of various amounts of a flexibilizer. Glass chopped strand mat and fabric laminates made from these matrices were used in a study of the nature of progressive damage leading to failure. Resin cracking, which occurs prior to ultimate failure in tension, was suppressed by increasing the flexibility of the resin. However, resin cracking in fatigue was not suppressed by increasing the flexibility of the resin. To investigate this observation further, experiments on the fracture behaviour of unreinforced resins were undertaken. The tensile strengths of resin casts of differing flexibility were measured [3]. Centre notched rectangular specimens were used to measure the fracture toughness and also the crack extension rates for constant stress intensity factor range cycling [3]. After the crack extension rates had been measured, the specimens were taken to failure by a single application of load. From the tensile, fracture toughness and crack extension specimens, fracture surfaces were obtained.

The present paper is concerned with the morphology of the fracture surfaces. The aim is to correlate surface markings as a function of resin flexibility and loading mode and ultimately, to identify characteristic surface markings in the resin phase of glass-reinforced laminates as a basis for failure analysis. The fracture surfaces were examined by scanning electron microscopy.

2. Materials and procedures

The basic resin was supplied by BIP Chemicals Ltd. It was a typical orthophthalic polyester resin of low viscosity and low reactivity with an alkyd/styrene ratio of 65:35. To obtain a range of resin properties, polypropylene adipate in styrene, 65:35, a low viscosity low reactivity flexible resin, was added to the basic resin in amounts of up to 50% by weight. Resin casts, approximately 70 cm × 55 cm, were made in sheet form. The resin was poured between two clamped sheets of glass separated by shims to give nominal thicknesses of 0.125 and 0.8 cm. 1% MEKP catalyst was used with 0.5% cobalt accelerator to promote cross-linking. An 18 h room temperature cure was followed by post-curing for 3 h at 80° C.

Necked tensile specimens were cut from rectangular blanks using an engraving machine with electro-bonded diamond cutting tools. The specimen shape and dimensions conformed to B.S. 2782 Part 3, 1965 and ASTM D638-64T.

(Fig. 1). Centre notched 30.5 cm × 7.6 cm rectangular specimens were used in the measurement of fracture toughness and crack growth rates in specimens cycled over a constant stress intensity factor range. The specimens were notched, first with a jewellers saw and then with a serrated razor blade to give a sharp tipped notch of 2.54 cm nominal length. The notch tip was examined microscopically and in all cases the radius of the notch tip was less than 0.010 cm. An E-type Tensometer universal testing machine was used for the tests. Single loads were applied at a cross-head displacement of 0.13 cm min^{-1} and cyclic loads at a frequency of 0.166 Hz.

Specimen fracture surfaces were examined by scanning electron microscopy. The specimens were mounted on standard 1.27 cm diameter stubs using an electrically conductive paint as adhesive. To prevent electrostatic charging, the specimens were coated with a thin layer of gold-palladium alloy by vacuum deposition.

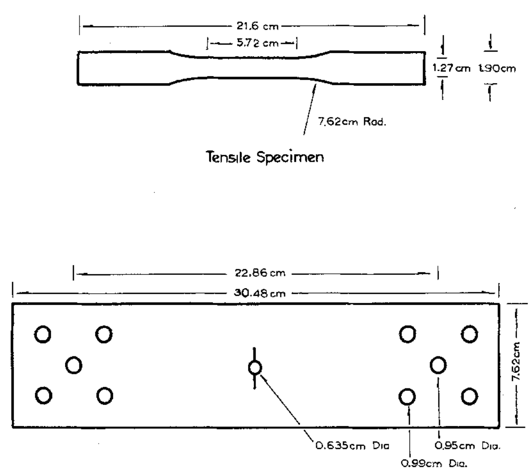

Figure 1 Test specimens.

3. Results

3.1. Fracture surfaces of fracture toughness specimens

Fracture surfaces at the notch tip are shown in Figs. 2 to 4 as a function of resin flexibility. The surface structures of specimens with 0 and 15% by weight of flexibilizer were similar and are shown in Fig. 2. Adjacent to the crack tip there is a small area of river markings which is shown in Fig. 2 and 5. As the crack front moves away from the notch, a smooth fracture surface is formed and then further away from the notch, surface roughness occurs. The two regions of surface roughness can be distinguished by the presence of conical markings. A typical conical marking is illustrated in Fig. 7 and these are found in the region of surface roughness furthest away from the notch.

As the resin flexibility is increased, the region of river markings adjacent to the notch increases in size and the river markings take on a more ordered and regular appearance. This is shown in Figs. 2 to 4 and 6. Fig. 5 is a detail of Fig. 2 and Fig. 6 is a detail of Fig. 4.

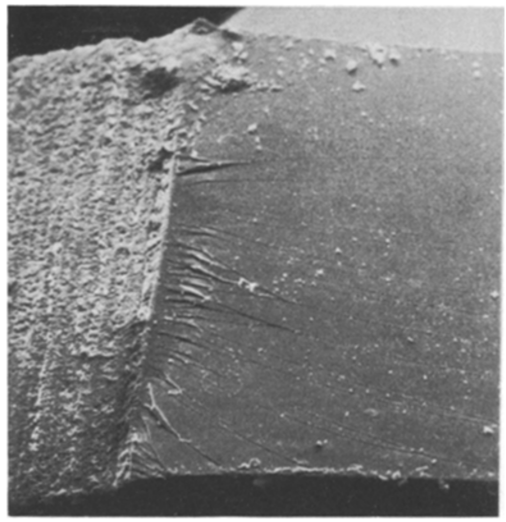

Figure 2 Fracture toughness specimen. No flexible resin addition, × 40.

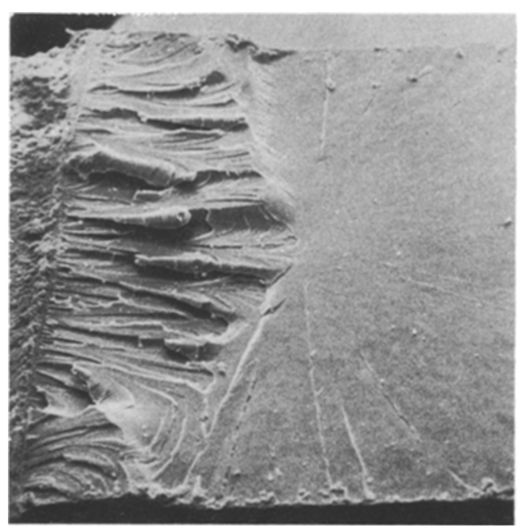

Figure 3 Fracture toughness specimen. 30% by weight of flexible resin addition, × 40.

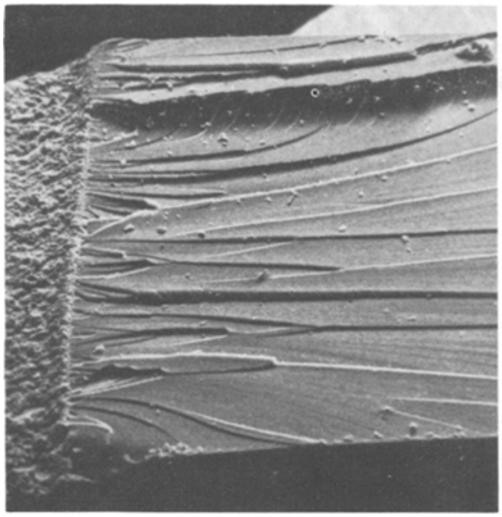

Figure 4 Fracture toughness specimen. 50% by weight of flexible resin addition, × 35.

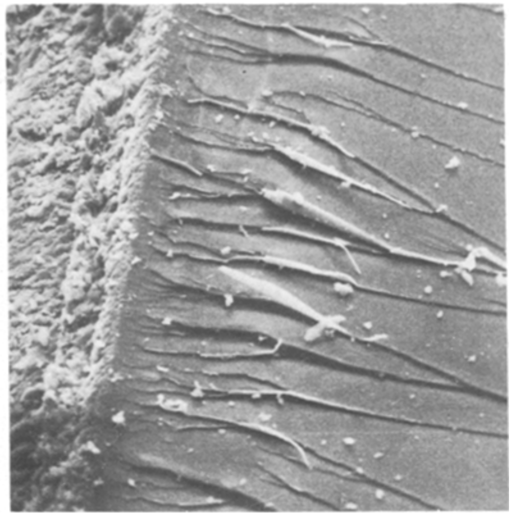

Figure 5 Fracture toughness specimen. No flexible resin addition, × 160.

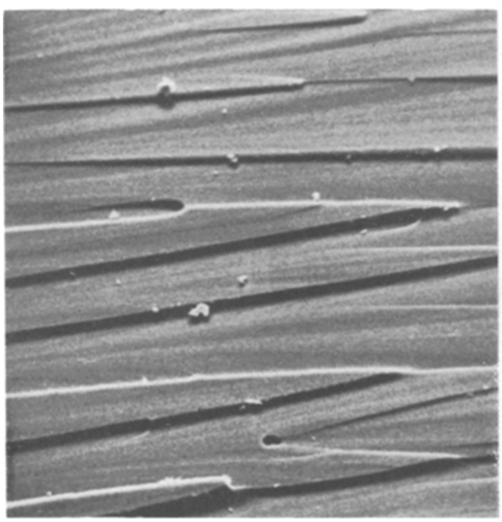

Figure 6 Fracture toughness specimen. 50% by weight of flexible resin addition, × 160.

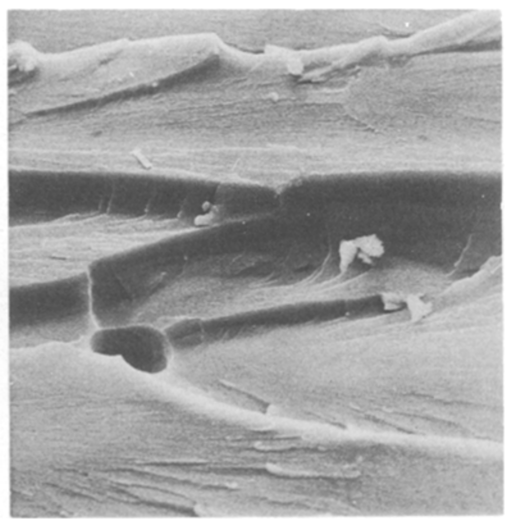

Figure 7 Fracture toughness specimen. 30% by weight of resin addition, × 435.

3.2. Fracture surfaces of standard tensile specimens

Fracture surfaces of standard tensile specimens are shown in Figs. 8 to 11 as a function of resin flexibility. Areas having characteristic river markings are not seen in the region adjacent to the crack initiation site. In the region of the crack initiation site the fracture surfaces are smooth and this smooth area associated with the start of fracture increases with increasing resin flexibility. Away from the crack initiation region, the fracture surfaces are rough. These regions of surface roughness are associated with the formation of multiple secondary cracks ahead of the main crack front and the characteristic conic markings are seen. Examples of the conical markings observed on

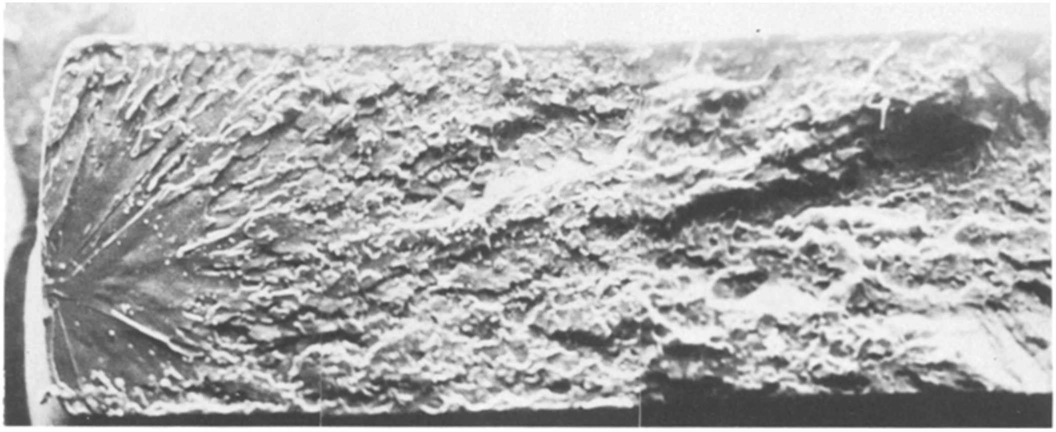

Figure 8 Tensile specimen. No flexible resin addition, × 25.

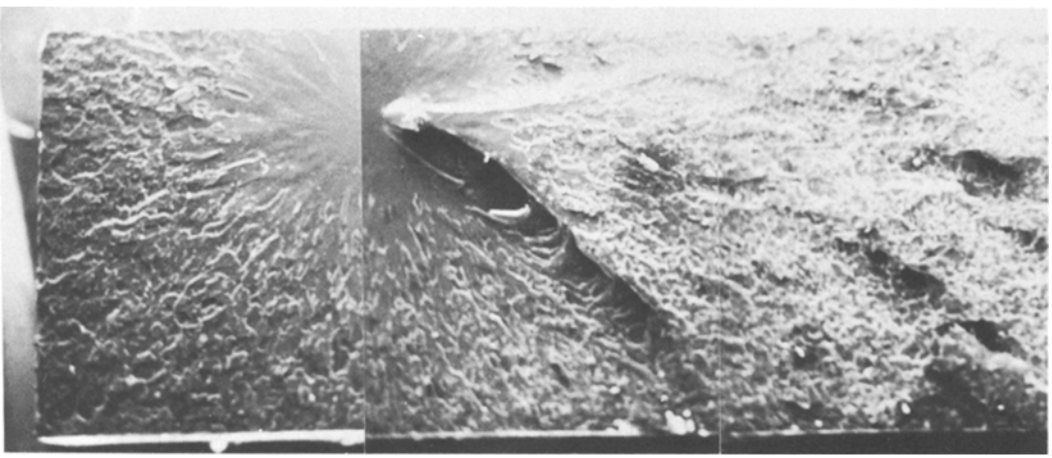

Figure 9 Tensile specimen. 15% by weight of flexible resin addition, × 25.

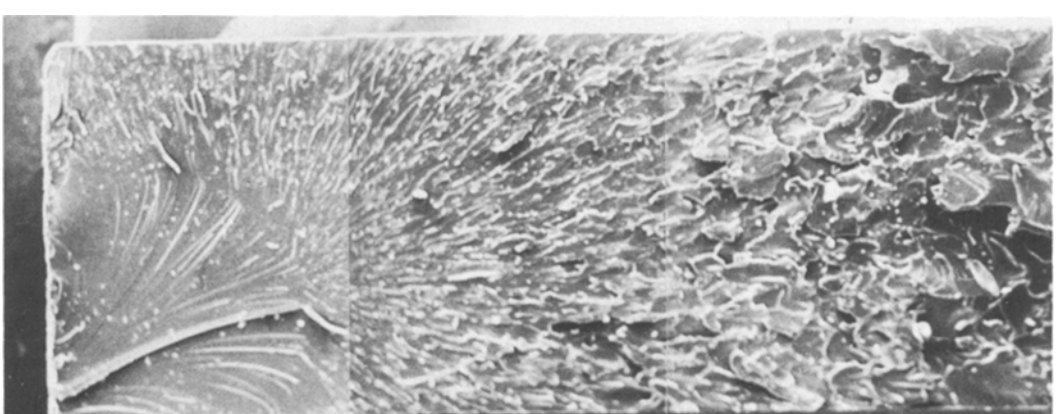

Figure 10 Tensile specimen. 30% by weight of flexible resin addition, × 25.

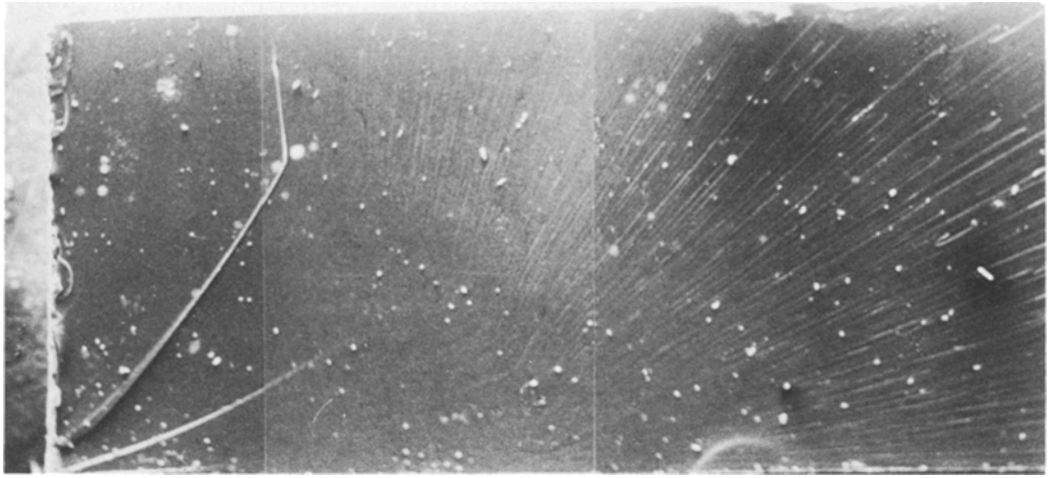

Figure 11 Tensile specimen. 50% by weight of flexible resin addition, ×30.

specimen surfaces shown in Figs. 8 and 11 are given in Figs. 12 and 13. The example shown in Fig. 12 is from the resin with no flexibilizer added and that in Fig. 12, from the most flexible resin.

3.3. Fracture surfaces of cyclically loaded notched specimens

Surface structures in the region of the crack tip are shown in Figs. 14 to 17 as a function of resin flexibility. The surface ridging, which is at right angles to the crack front, becomes more ordered as the resin flexibility is increased. With low flexible resin additions there is a marked contrast between surface structures obtained from cyclic and single loading, but there is little difference at 50% by weight of flexible resin addition, cf. Figs. 4 and 17.

On completion of measurements of crack growth for constant stress intensity factor cycling as a function of the number of cycles, the specimens were loaded in tension to failure. Although the crack tip was formed by constant stress intensity factor cycling, the surface structures due to tensile loading were the same as those found in the mechanically notched fracture toughness specimens.

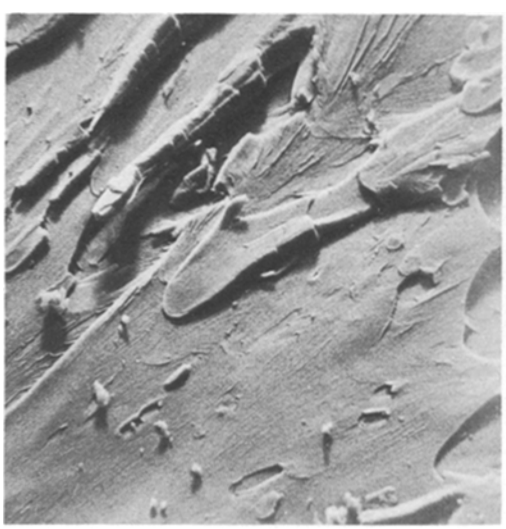

Figure 12 Tensile specimen. No flexible resin addition, ×160.

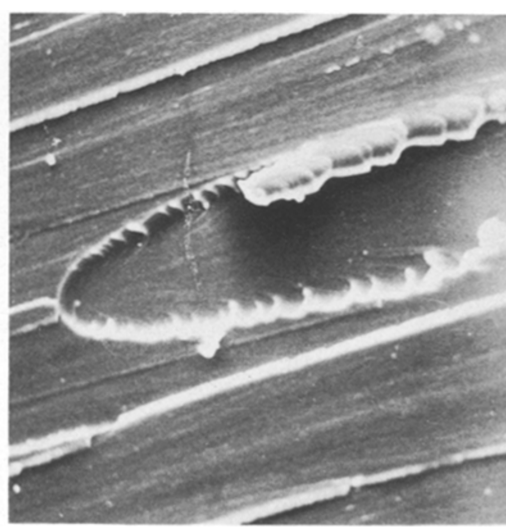

Figure 13 Tensile specimen. 50% by weight of flexible resin addition, ×135.

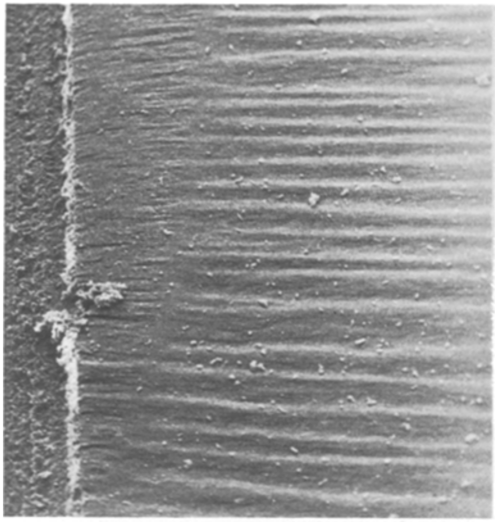

Figure 14 Fracture toughness specimen cycled over a constant range of ΔK. $\Delta K = 0.60\,\mathrm{MN\,m^{-3/2}}$. No flexible resin addition, $\times 45$.

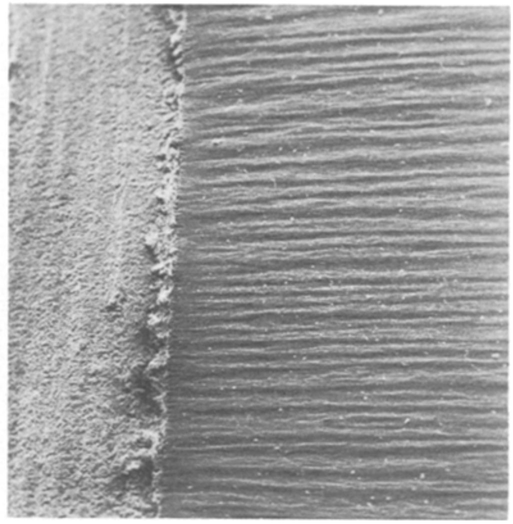

Figure 15 Fracture toughness specimen cycled over a constant range of ΔK. $\Delta K = 0.54\,\mathrm{MN\,m^{-3/2}}$. 15% by weight of flexible resin addition, $\times 45$.

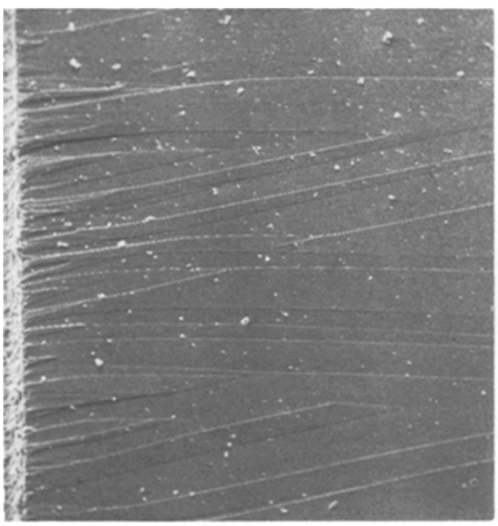

Figure 16 Fracture toughness specimen cycled over a constant range of ΔK. $\Delta K = 0.60\,\mathrm{MN\,m^{-3/2}}$. 30% by weight of flexible resin addition, $\times 45$.

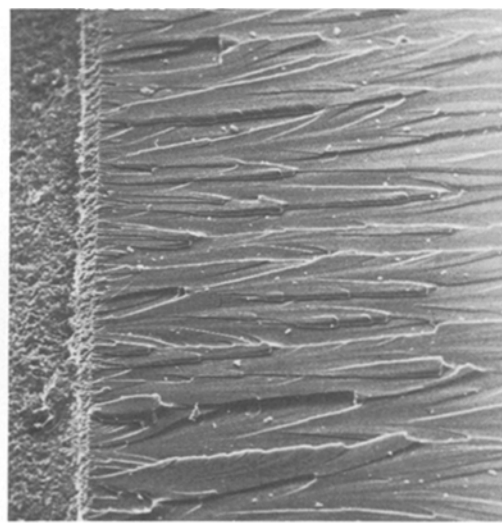

Figure 17 Fracture toughness specimen cycled over a constant range of ΔK. $\Delta K = 0.56\,\mathrm{MN\,m^{-3/2}}$. 50% by weight of flexible resin addition, $\times 45$.

4. Discussion

The features of the fracture surfaces, where direct comparison is possible, are similar to those observed in glass [4] and PMMA [5, 6]; The fracture surface characteristics can be divided into groups. These groups are illustrated by the surface characteristics of failed fracture toughness specimens. The surface markings of un-notched tensile specimens and cyclically loaded notched specimens taken to failure are then compared with those of the fracture toughness specimens. Fracture surface markings of notched fracture toughness specimens can be divided into two main areas. One area is associated with stable crack growth and the other area with unstable crack growth. Stable crack growth is defined when the crack is not self-propagating and the movement of the crack front can be controlled by the experimental test technique. Unstable crack growth is defined when crack growth is self-propagating.

River markings, curls [7] and well-defined stepped features are characteristic of stable crack growth fracture surfaces. Fracture surface markings formed during unstable crack growth can be further subdivided into regions associated with slow and fast propagation of the crack front. A smooth fracture surface is associated with slow unstable crack propagation and a rough fracture surface on which conical markings are found is associated with fast unstable crack propagation. It is possible, and necessary, to distinguish between surface roughness associated with stable crack growth and fast unstable crack growth. The conical markings are only found in the region of fast unstable crack growth.

The three regions found in fracture toughness specimens provide a basis against which the fracture surface markings of un-notched tensile failures and of crack growth, formed by constant stress intensity factor cycling, can be compared. A region of stable crack growth is shown on the left of Fig. 3. The smooth fracture surface on the right of Fig. 3 is where the propagation of the crack front has become unstable. This is a region of slow unstable crack growth and as the crack front velocity increases, the surface roughness increases. A detail of this surface roughness is shown in Fig. 7.

The fracture surface shown in Fig. 3 is for a polyester resin containing 30% by weight of flexibilizer. As the flexibility of the resin is further increased, the region of stable crack growth increases and the surface markings become geometrically defined. This is shown in Fig. 4. In the brittle resin, the region of stable crack growth is smaller and less defined and river markings are seen. These are shown in Fig. 2 [8]. Fracture surfaces of un-notched tensile specimens, shown in Figs. 8 to 11, do not have a region of stable crack growth. However, the crack initiation site can be identified, as in Fig. 9, together with the regions of slow and fast unstable crack growth. As the flexibility of the resin is increased, the size of the smooth area associated with slow unstable crack growth increases.

The absence of a region of stable crack growth in un-notched tensile specimens may be attributed to the difference between the energy required to create and grow a critical sized crack and the energy required to grow a critical sized crack from a defect or crack already present.

The characteristic feature of fracture surfaces produced by cycling notched specimens over a constant range of stress intensity factor is a ridged structure with the ridges aligned at right angles to the crack front. The orientation of the ridges contrasts with that of striations formed during fatigue crack growth in metals and which lie parallel with the crack front.

As the resin flexibility increases the number of ridges decreases, and is shown in Figs. 14 to 16. At 50% by weight of flexible resin addition (Fig. 17), the geometrical form of the surface markings is similar to that found in the stable crack growth region of the fracture toughness specimen. Ridged structures have been observed in PMMA specimens taken to failure in tension or fatigue [6]. A large number of ridges was associated with cyclic loading and a small number with stable crack growth in tension. This distinction is not clearly defined for polyester resins.

5. Conclusions

The fracture surface markings of a series of polyester resins have been classified into three main groups, each group being associated with a crack front displacement – stable crack growth, slow unstable crack growth and fast unstable crack growth. Changes in resin flexibility affect the extent to which a given region of characteristic surface markings is present and in the case of constant range stress intensity factor cycling, the number and geometry of surface ridges. Different loading conditions affect the observed number of characteristic fracture regions. No difference in surface markings was observed between mechanically notched specimens and specimens for which the notch was formed by crack growth in cyclic loading when the specimens were loaded to failure in tension.

The ridges formed during constant range of stress intensity factor cycling are aligned at right angles to the crack front. This is in contrast to the orientation of the striations which are characteristic of fatigue surfaces in metals.

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