Deformation in an epoxy resin revealed by an etching technique

A recent investigation concerning the fracture and mechanical behaviour of an epoxy resin system has stimulated a study of the morphology of epoxy resins in order to explain variations induced by stoichiometric changes. Among the observations reported in [1] was the response of cured epoxies to hot acid etching. At that time there was doubt about the nature of the etched surface. The significance of the features observed after chromic acid etching of Epikote 828* cured with Epikure DDM* has now been more firmly established by means of very simple experiments.

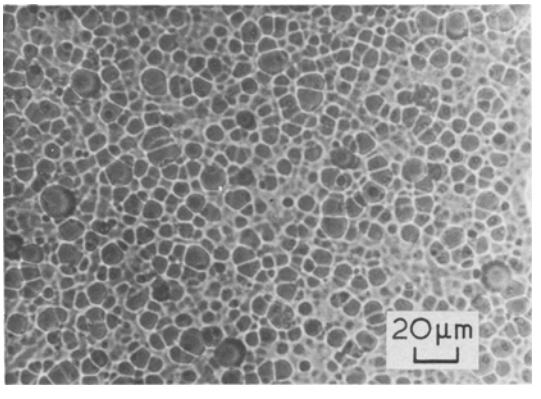

Figure 1 An optical micrograph of a polished epoxy resin surface (stoichiometric composition of Epikote 828/Epikure DDM) after etching in 1 molal CrO_3/H_2O at 70° C.

Fig. 1 shows a typical result of etching the surface of a stoichiometric resin in 1 molal CrO_3/H_2O at 70° C for 8 h. The surface had previously been prepared by careful polishing on silicon carbide papers and diamond impregnated cloths to a 1 μm diamond finish. Fig. 2 shows how the surface regions of a similarly prepared compression test piece (containing 35 parts DDM per 100 of resin) responded to deformation beyond the yield point and subsequent acid etching. It should be emphasized that the deformation had been carried out prior to etching. The surface shown in Fig. 3a and b is a cleavage fracture surface, the plane of which is at right angles to a

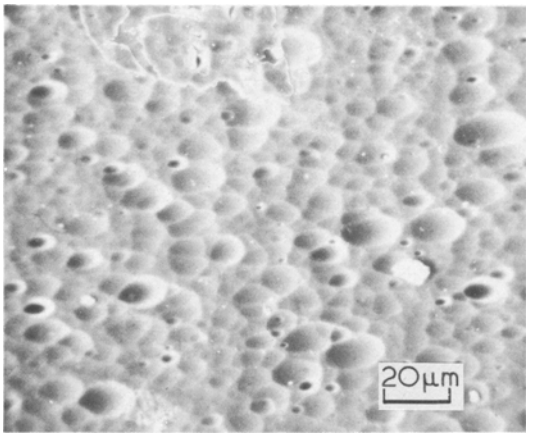

Figure 2 A stereoscan photograph of the surface of a compression test piece after polishing, deforming, and etching (Epikote 828/Epikure DDM : 100/35).

machined surface slot. Examination of the features thus revealed indicated that a microscopically observable response to mechanical forces has occurred during machining operations. The well defined zone of deformation in Figs. 3a and 3b was created during slot-cutting of tapered double-cantilever cleavage fracture-toughness specimens (prior to testing). The possibility that the deformation seen in Figs. 3a and b was the result of a fracture event (e.g. due to a plane stress conditions at specimen edges) can be discounted because the orientations of the lenticular features on each side of the fracture surface were arranged in opposite senses (Fig. 4). The orientation has presumably been determined by the direction of rotation of the cutter used during sample preparation. In any case, the areas of fracture surface chosen for etching were those where the crack would have been moving rapidly between arrest locations, thus minimizing the extent of visco-plastic deformation. Cutter/surface relative speeds during sample preparation were low, hence producing a situation more analogous (in part) to the compression tests at 0.017 mm sec^{-1} cross-head speed than to high-speed cleavage.

Summarizing, it appears that a feature of the type described by Cuthrell [2] and Selby and Miller [1] can respond to mechanical forces. The characterization of this feature may provide quantitative relationships between resin properties

*Shell Chemical Co Ltd trade names for a di-glycidol ether of bisphenol-A resin and diaminodiphenylmethane respectively.

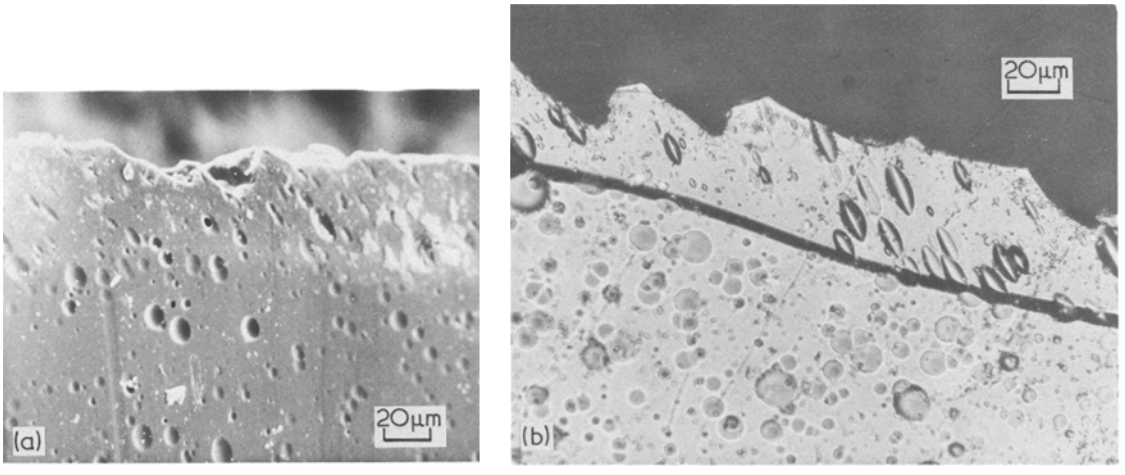

Figure 3 Photographs of a deformed layer at the edge of a cleavage fracture surface (Epikote 828/Epikure DDM stoichiometric composition). (a) Stereoscan; (b) optical micrograph.

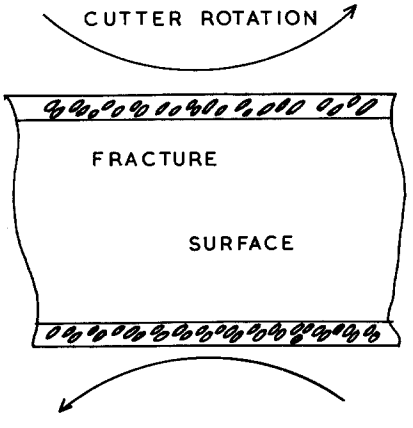

Figure 4 A sketch showing the orientations of deformed, etched, features at the edges of fracture surfaces.

and physical morphology. Such relationships would be of valuable use in the composites field. Also, the etching process could possibly be used to advantage in investigating deformation characteristics at interfaces in epoxy composites. Although Cuthrell [3] has reported some correlation between what he terms "floccule" size and thermal or electrical properties, as far as the authors are aware the mechanical response reported here (albeit with some potential thermal connotations in one case due to friction) has not been previously reported or published. Hopefully this information will be of some interest to those working on the deformation behaviour of resins.

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