

The epitaxial layers grown from SiCl_4 at 1150°C appeared to have a smooth shiny surface under visual inspection. Platinum/carbon replicas confirmed that the surface was free from steps of height greater than 50 \AA . However, XRT and TEM showed that defects were still present, though at low densities.

It is well known [6] that ion implantation creates an amorphous layer just below the surface of the crystal. As the surface is oxidized this layer is partly absorbed into the oxide and partly annealed out. Our work shows that the damage is not completely removed but that dislocation loops propagate into the crystal by a climb process. Most of these loops propagate more than $1 \mu\text{m}$ into the crystal, a finding which is in conflict with Moline *et al.* [1]. These authors reported that the damage in their slices could be eliminated by etching only $0.2 \mu\text{m}$ from the surface.

Under some conditions of epitaxy the ends of these dislocation loops propagate with the layer where they form the majority of defects found ($\sim 10^7 \text{ cm}^{-2}$). The density of epitaxial stacking faults was much less ($\sim 10^4 \text{ cm}^{-2}$). Although the growth from SiCl_4 was much more successful, some defects were still present in those layers.

Work is continuing to elucidate the mechanisms by which the dislocations are formed. In particular we wish to investigate the effect of beam heating as both our results and those of

Moline *et al.* suggest that the defect density may be related to substrate temperature during implantation.

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Crack growth in epoxide resin adhesives

The vast majority of the structural adhesives commercially available are based upon epoxide resins and this has generated interest in the fracture properties of these relatively brittle, thermosetting materials. This paper describes some preliminary results on the application of continuum fracture mechanics to the failure of epoxide resins and although this, in itself, is not novel [1-3], by also measuring rates of crack propagation some unexpected characteristics of crack growth have been observed.

The specimen geometry employed in this investigation was a tapered double cantilever beam joint as shown schematically in Fig. 1. Detailed specimen preparation and fracture test techniques have been reported elsewhere [1, 4] and it is sufficient to note that the substrate was aluminium alloy, to specification British Standard 1476.He 30, which was machined into

cantilever beams, 308 mm long, 12.7 mm thick and with a height, h , varying between 16.0 and 47.8 mm. The surfaces to be bonded were first subjected to a liquid and vapour degreasing bath of dichloro-ethane, then grit blasted with 180-220 mesh alumina, then degreased again and finally allowed to air-dry in a desiccator. The epoxide adhesive employed was a diglycidyl ether of bisphenol A cross-linked with either 9.4 mass per cent of a tertiary amine curing agent (tri-2-ethyl hexanoate of 2,4,6 tris (dimethylaminomethyl) phenol) or 11.0 mass per cent of a primary curing agent (triethyltetramine). In the former case the curing schedule was 23°C for 96 h, followed by 100°C for $1\frac{1}{2}$ h and finally 180°C for $2\frac{1}{2}$ h and, in the latter case, 23°C for 2 h followed by 60°C for 2 h. The specimens were then usually conditioned at 23°C and 56% R.H. for a few days prior to testing. The thickness of the epoxide resin layer was controlled to $0.50 \pm 0.06 \text{ mm}$.

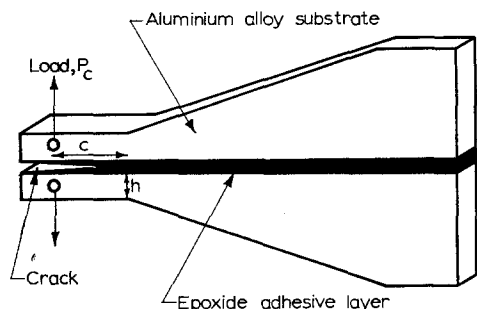


Figure 1 Sketch of tapered double cantilever beam specimen.

The fracture energy, G_{IC} , was determined by first propagating a crack into the adhesive for a short distance, usually about 70 to 100 mm, to act as a natural, starter crack. The arms of the specimen were then separated at a constant rate, using an Instron tensile testing machine, until crack propagation was observed. The fracture energy, G_{IC} , was then ascertained from the relationship [1]:

$$G_{IC} = \frac{4P_c^2 m}{Eb^2} \quad (1)$$

where P_c is the applied load at crack propagation, E is the modulus of the substrate (68.9 GPa), b is the specimen thickness and m is a geometry factor given by [5]:

$$m = \frac{3c^2}{h^3} + \frac{1}{h} \quad (2)$$

where c is the crack length corresponding to a height of substrate beam, h . The initial crack velocity was ascertained either by visual observa-

tion or, for crack velocities greater than about 0.01 m sec^{-1} , by the crack progressively rupturing a conductive grid painted on the side of the specimen and recording the associated voltage change on an oscilloscope [4]. Further, after joint fracture, the nature and topography of the joints fracture surfaces were examined both visually and by optical and scanning electron microscopy.

The relation between the fracture energy, G_{IC} , and the associated initial crack velocity, \dot{c} , for joints using the two different adhesive systems was determined at room temperature and 56% R.H. and the results are shown in Fig. 2. There are several noteworthy features. First, for both types of adhesive the locus of joint failure was cohesive in the epoxide resin and, indeed, the crack propagated along the centre of this layer. Second, the values of the fracture energy, G_{IC} , are far greater than would be expected from theoretical considerations of brittle fracture, which solely involves the rupture of molecular bonds in the material; such considerations lead to values of the order of 500 mJ m^{-2} [6]. The measured fracture energies are orders of magnitude greater than this theoretically calculated value and this discrepancy is presumably due to the energy that must also be dissipated in producing local plastic deformation at the tip of the crack. Present, and previous [7], fractographic examination of epoxide resins clearly confirm that such deformational processes occur during fracture. It is to be expected that, since such deformational energy losses are time-dependent, the fracture energy may well be a function of crack velocity and temperature.

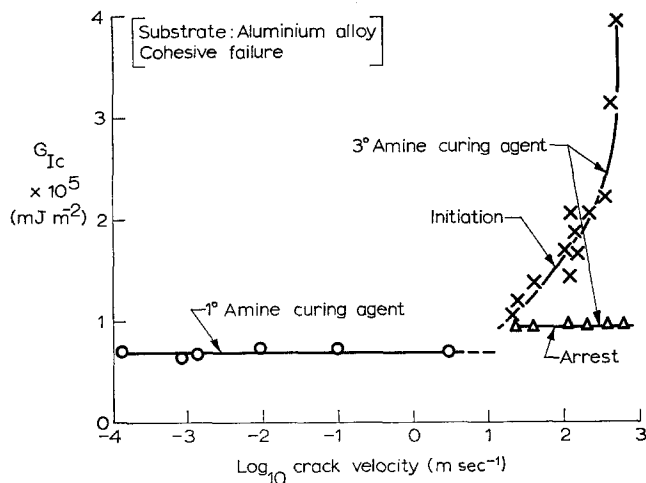


Figure 2 Relationships between fracture energy, G_{IC} , and crack velocity.

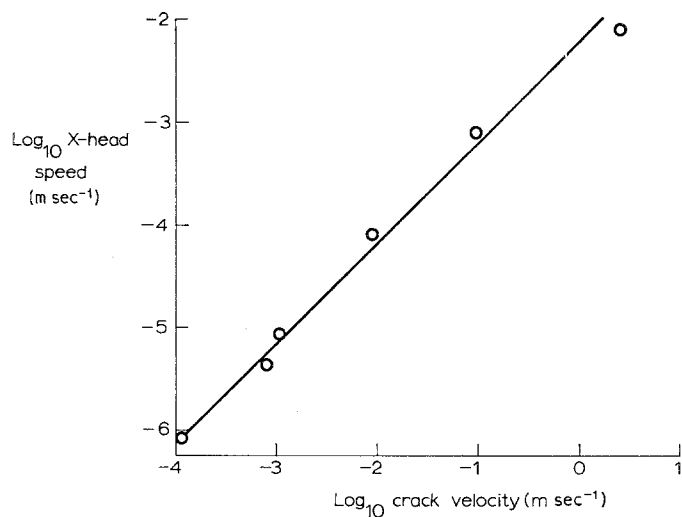


Figure 3 Cross-head speed of testing machine versus resulting crack velocity for the primary amine cured epoxide resin.

Third, if the joints were prepared using the primary (1°) amine curing agent then the crack propagated in a continuous, steady manner through the adhesive and crack velocities within the range from about 10^{-4} to 3 m sec^{-1} were observed and the value of G_{IC} was virtually independent of crack velocity. Further, at these relatively low crack velocities the velocity observed was directly proportional to the rate of separation of the specimen's arms (which, in turn, was determined by the cross-head speed of the testing machine) and this is shown in Fig. 3. A velocity of 3 m sec^{-1} represented the fastest cross-head speed that could be attained; higher crack velocities in this adhesive would probably be achieved with a faster cross-head speed.

However, if instead, the tertiary (3°) amine curing agent was used to cross-link the epoxy adhesive then the crack propagated intermittently in a slip-stick manner and values of G_{IC} for crack initiation and arrest could be ascertained. As may be seen from Fig. 2, the value of G_{IC} (initiation) increases rapidly with increasing crack velocity while G_{IC} (arrest) is independent of initial crack velocity. Another difference in the crack growth behaviour of the tertiary amine cured epoxide adhesive is that the velocity of crack growth was always greater than about 20 m sec^{-1} ; initial crack velocities slower than this were never observed, no matter how slowly the arms of the cantilever beam specimen were separated. Indeed, there was no apparent correlation between crack velocity and the rate of separation of the specimen's arms.

Thus these two epoxide adhesives, which have

similar chemical structures, uniaxial stress-strain properties and glass transition temperatures, show distinctly different types of crack growth behaviour. This is of importance when interpreting, and designing with, the fracture mechanics data that are currently being collected on structural adhesives. Present work is being directed towards elucidating the controlling parameters in the fracture of these materials.

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