markedly greater for the samples produced using lower calcination temperatures. The extensive cracking evident after a creep strain of 0.05 for the sample which had been calcined at 1273 K is shown in Fig. 3. Intergranular cracks therefore seem to form more easily on boundaries present in samples exhibiting low n values. As concluded from the studies of the plasticity of MgO [5, 6], the detailed fabrication processes used to prepare the samples then appear to affect the stress exponent by determining the ability of grain boundaries to resist cracking. In this way, the present observations are consistent with the view [2] that factors which influence the ductility of polycrystalline ceramics also affect the value of the stress exponent for creep.

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A self-toughening mechanism in epoxide resins

Interest has recently flourished in the fracture behaviour and failure mechanisms in epoxide resins, since these thermosetting polymers are being increasingly employed in structural engineering applications both as adhesives and as matrices in composite materials. A few years ago several papers were published [1, 2] concerning the static fatigue of epoxide resins both in bulk and in adhesive joint specimens and a failure criterion was advanced [2] based upon the attainment of a critical plastic zone size at a crack tip. However, more recently it has been reported [3, 4] that certain epoxide resins of different compositions did not appear to suffer from static fatigue, even when stressed to a relatively high level. This letter outlines further interesting aspects of this observation and demonstrates that an epoxide resin adhesive may even be toughened appreciably by subjecting it to an applied load.

The specimen geometry employed for the adhesive joints was a tapered double cantilever beam joint. The substrate material was aluminium alloy, to specification British Standard 1474 NE4, 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 trichloroethane, then grit blasting with 180–220 mesh alumina; then after degreasing again were finally allowed to dry in air. The epoxide adhesive employed was a diglycidyl ether of bisphenol A cross-linked with 10.0 mass per cent of an amine curing agent (tetraethylenepentamine). Immediately prior to joint preparation the aluminium alloy substrates were treated as described above. Adhesive was spread on the treated faces and the two beams pressed lightly together. Small pieces of plastic

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TABLE I Constant displacement rate fracture results before and after subjecting the specimen to a static fatigue test.

Results prior to static fatigue tests			Results after static fatigue tests			
Crack growth mode	Crack velocity (m sec^{-1})	G_{Ic}(kJ m^{-2})	Time under load (sec)	Crack growth mode*	G_{Ic}(kJ m^{-2})*	
					G_{Ic} (initiation)	G_{Ic} (arrest)
Stable	1.8×10^{-3}	0.056	10	Stable	0.058	–
Stable	1.7×10^{-3}	0.058	190	Stable	0.058	–
Stable	1.6×10^{-3}	0.059	500	Unstable	0.066	0.058
Stable	1.6×10^{-3}	0.058	1050	Unstable	0.062	0.058
Stable	1.7×10^{-3}	0.056	3000	Unstable	0.075	0.062
Stable	1.7×10^{-3}	0.054	1.80×10^{4}	Unstable	0.081	0.058
Stable	1.7×10^{-3}	0.058	1.69×10^{5}	Unstable	0.091	0.058
Stable	1.6×10^{-3}	0.054	1.19×10^{6}	Unstable	0.101	0.056
Stable	1.7×10^{-3}	0.054	1.22×10^{6}	Unstable	0.096	0.058
Stable	1.7×10^{-3}	0.067	2.25×10^{6}	Unstable	0.131	0.058
Stable	1.6×10^{-3}	0.054	5.17×10^{6}	Unstable	0.110	0.059

*Refers to first increment of crack growth, thereafter the mode of crack growth and G_{Ic} value are as for initial tests.

sheet, previously inserted in the adhesive at the far ends of the joint, were used to control the thickness of the epoxide resin layer to 0.50 ± 0.06 mm. Further, a piece of Teflon tape, about 30 mm long, 12.7 mm wide and 0.08 mm thick was previously placed, approximately in the centre of the adhesive and at the narrow end of the joint, to assist in propagating a "starter" crack. Excess adhesive on the beam sides was removed and to effect cure of the adhesive the joint was held at 23° C for 22 h followed by 80° C for 6 h.

The testing schedule followed was:

(i) The arms of the specimen were separated at a constant rate of 8.5×10^{-3} mm sec^{-1} using an Instron tensile testing machine. The first few centimetres of crack growth, originating from the Teflon tape, were ignored but thereafter the load, P_c, for crack propagation and the rate of propagation were recorded. When the crack was about 14 cm long the test was halted and the specimen loaded in a creep machine.

(ii) The creep machine (a "Unisteel Stress Corrosion" apparatus, manufactured by W.H. Mayes and Son Ltd) possessed a double lever loading system giving a 30:1 loading ratio. The specimen from (i) above was placed in the machine and a load of 235.4 N gently applied. This load represented about 86% of the failure load required to cause crack growth in (i) above and gave a G_I (applied) of 0.043 kJ m^{-2}. The crack tip was frequently observed using a travelling microscope, fitted with a graticule eyepiece unit which permitted a minimum crack growth increment of 0.01 mm to be detected.

(iii) If, after the required time period, no fracture had occurred under the static fatigue conditions, then the specimen was removed and re-tested as in (i) above.

All the above tests were conducted at 23 ± 2° C and 50% r.h.

The adhesive fracture energy, G_{Ic}, was determined from the relationship [5]:

$$G_{Ic} = \frac{4P_c^2 m}{E_s b^2} \quad (1)$$

where P_c is the load, E_s is the modulus of the substrate, b is the specimen thickness and m is the geometry factor given by [6]:

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

where a is the crack length corresponding to a height of substrate beam, h.

The results obtained from these studies are shown in Table I and in all tests the locus of joint failures was cohesive through the adhesive layer. The initial crack growth studies, conducted at a constant displacement rate prior to loading, confirmed previously reported results [3]. Namely, crack growth occurred in a continuous, steady manner at a given load with the rate of crack propagation being governed by the rate of crosshead displacement used. This is commonly termed stable crack growth and a typical load–displace-

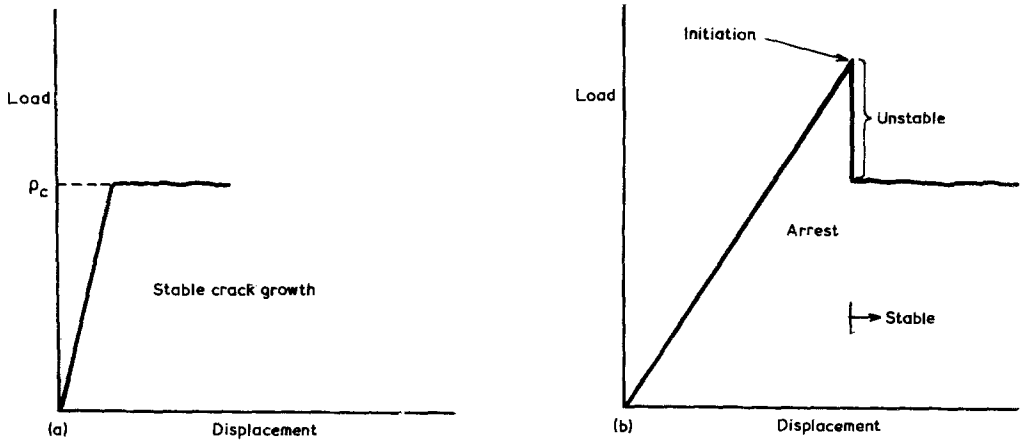

Figure 1 Typical load–displacement curves for constant displacement rate tests conducted (a) prior to subjecting specimen to static fatigue test and (b) same specimen after static fatigue test.

ment trace for a specimen exhibiting this type of behaviour is shown in Fig. 1a.

In the static fatigue experiments none of the specimens failed and, indeed, no crack growth was observed.

After having subjected the specimens to such static fatigue conditions they were re-tested at a constant displacement rate and the results obtained were particularly revealing. For static loading times of a few hundred seconds or less no significant changes in the mode of crack growth or G_{Ic} values were recorded. However, for longer times differences were observed. The first increment of crack growth now occurred in an unstable manner, i.e. the crack jumped forward at a relatively high, indeterminate velocity until, having outpaced the rate of energy supply, it was arrested. Thus load values corresponding to initiation and arrest could be ascertained. After a brief pause the crack began to propagate again at the arrest load, but now in a stable manner. A typical load–displacement trace for this behaviour is shown in Fig. 1b. Now the G_{Ic} (initiation) value for the first, unstable increment of crack growth was much higher than the G_{Ic} value required for stable crack growth prior to loading. However, once the crack had propagated by the initial jump, further propagation occurred in a stable manner at the same G_{Ic} value as recorded prior to loading.

The ratio of G_{Ic} (initiation, after static fatigue test)/G_{Ic} (prior to static fatigue test) is shown as a function of the logarithmic time-under-load

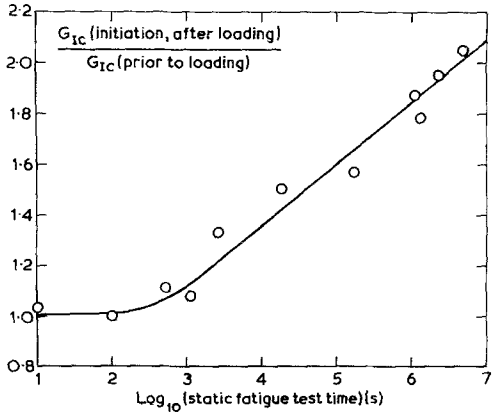

Figure 2 Increase in G_{Ic} value, ascertained from constant displacement rate tests, for a specimen as a function of the time the specimen was subjected to an applied load during the static fatigue experiment.

in Fig. 2 and, above a few hundred seconds, a linear relation exists between these parameters. This self-toughening mechanism was not observed for specimens stored under no applied load.

Considering the mechanisms responsible for these observations then it is of interest to note that the true compressive yield stress of this epoxide resin decreases linearly with increasing logarithmic time-of-test [3]. Thus the formation of a plastic zone at the crack tip should be easier at longer times and hence the degree of crack blunting will increase. It is suggested that it is an increase in the severity of crack tip blunting that is responsible for the rise in adhesive fracture energy and associated change in crack propagation behaviour from stable to unstable. However, once

the crack has propagated through the blunting region, further propagation occurs in a stable manner at the original G_{Ic} value. Furthermore, this tendency for the crack to blunt under static fatigue testing means that although the applied load may initially represent a large percentage of that required for fracture, this percentage rapidly diminishes as the experiment proceeds. Thus the self-toughening mechanism forestalls static fatigue failure. Longer term experiments are required to assess whether static fatigue failure is completely prevented or merely delayed.

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