TABLE I Contribution in dB km<sup>-1</sup> p.p.m.<sup>-1</sup> of transition metal impurites to the loss in alkali lime germanosilicate optical fibres.

	850 nm	1050 nm	1300 nm
Ti	<5	<5	<5
v	150	220	80
Cr	90	40	<5
Mn	<5	<5	<5
Fe	30	40	30
Co	40	370	1460
Ni	430	350	240
Cu	270	140	30

bution of 1 p.p.m. of a transition metal in the glass, to the total loss of the fibre (Table I). The values are given at 850, 1050 and 1300 nm, wavelengths at which it is proposed to use optical fibres for long distance optical communication purposes [3]. Comparable data for other glass systems used for making optical fibres are given by Schultz for fused silica [11], by Ikeda *et al.* for T1 containing sodium borosilicate glass [12] and by Newns *et al.* for sodium lime silicate glass [13]. Concerning Table I the following concluding remarks can be made:

(1) In the case of alkali lime germanosilicate optical fibres a concentration of a few p.p.b. of Ni, Cu, V or Co is sufficient to cause measurable losses in the fibre at the proposed wavelengths. Therefore glasses have to made in which these impurities are present in concentrations below 1 p.p.b.

(2) The contribution of the absorption produced by Cr, Ni and Cu, which together with Fe are the most common impurities, to the loss of the optical fibre, decreases at longer wavelengths.

(3) Fe in alkali lime germanosilicate glasses melted under oxidizing conditions is predominantly present as  $Fe^{3+}$ . Consequently the suscepti-

Direct observations of debonding at crack tips in glass bead-filled epoxy

Glass bead-filled polymers are often much tougher than the unfilled matrix polymer. In studying the origin of the increased toughness various workers [1-4] have examined fractured samples after unloading but there are no reports of direct obserbility of the loss of the optical fibre to the presence of the most common impurity both in the starting material and the environment (such as ambient air and furnace lining), is relatively small.

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Received 31 January

and accepted 23 February 1979. G. A. C. M. SPIERINGS Philips Research Laboratories, Eindhoven, The Netherlands

vations of crack tip processes in these materials during fracture. The results of observing cracks directly as they grow in a glass bead—epoxy resin composite are reported, together with the implications of these observations for the origins of the increase of toughness.

Plate-like specimens  $(35 \text{ mm} \times 35 \text{ mm} \times 3 \text{ mm})$  of an epoxy resin\* containing approximately 25%



Figure 1 Schematic diagram of sample in loading apparatus.

by volume of glass microspheres\* were prepared and a slot about 8 mm long cut in the middle of one edge. A crack about 8 mm long was forced to grow from the bottom of the slot by tapping in a razor blade. The specimen was positioned in the apparatus shown schematically in Fig. 1 and mounted on the stage of a microscope so that the crack tip region could be studied using transmitted light. The crack was slowly opened by driving the wedge into the slot using the hand driven screw.

Figs. 2a to d show a sequence of photographs of a typical crack tip in the material from the beginning of loading, Fig. 2a, to just before the onset of fast fracture, Fig. 2d. The solid line marks



Figure 2 (a) to (d) Sequence of photographs from video-recording showing progressive debonding of glass spheres from the matrix as the crack is loaded to the point of fast fracture. The solid line marks the position of the crack and the arrows indicate the mode of opening. The A and B mark the same particle in each photograph.

\* Ballotini 2429, Plastichem Ltd. Used in as-received condition with no surface coupling agent. 2522 the position of the crack and the arrows show the mode of opening. The letters A and B mark the same particles in successive photographs.

In Fig. 2c the outlines of the glass particles are visible as fine lines. As loading proceeds, segments of the surface of individual particles suddenly pull free from the matrix in a zone surrounding the crack tip; the debonded segment then grows in size, spreading round the particle surface. The zone itself grows in size until (in our experiments) its diameter becomes comparable with a frame of Fig. 2. Finally, the crack advances into the debonded zone.

The sudden nature of the initial debonding is most strikingly seen by direct observation, or on movie or video recordings made through the microscope eyepiece. Fig. 2 shows 4 frames from such a video recording. Debonding is visible as a thickening and darkening of the outlines of the particles as the interface pulls open, scattering more light. The sequence shows an increasing number of debonded particles as the crack tip is slowly loaded. Fig. 2d, taken immediately before fast fracture, shows debonded particles up to 10 particle diameters distant from the crack tip, while closer into the crack tip nearly every particle has debonded from the matrix. There were no indications of crazing or of shear zones originating at particles in this composite.

When a crack propagates through epoxy stiffened with a dispersion of glass spheres, the spheres pull free from the matrix in a zone ahead of the advancing crack. The debonding of a single particle occurs by the sudden appearance of segments of debonding at points on the particle where one would expect the tensile stress to be greatest. These segments then spread more slowly round the surface of the particle. When particles debond, the modulus of the composite falls. The crack tip, therefore, is advancing into a zone of locally reduced modulus. The stress intensity at the crack tip is changed by this - it is lower than it would be if no debonding occurred. It is thought that this is an important contribution to the toughening in filled polymers and we are now developing a model and conducting experiments to examine it more closely.

## Acknowledgements

The author would like to acknowledge the support of the Science Research Council in the form of a research studentship, and to thank Professor M. F. Ashby and Dr P. W. R. Beaumont for helpful discussions on various aspects of the work, and Mr M. Rayner for technical assistance.

This work forms part of a larger programme on the fracture of polymers and composites supported by the Science Research Council.

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Received 8 February and accepted 12 March 1979.

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## A new powder metallurgy method

We wish to report preliminary findings of a new powder metallurgy technique which has some novel features. Parts produced by powder metallurgy are generally made by cold pressing, followed by sintering, or in some cases, if superior properties are sought, by sinter forging. To produce a satisfactory product, a significant time at high temperature is required. Explosive compaction of cold powder generally produces a product similar to statically pressed materials [1-3], although if the powder is heated, much better properties may be obtained [4]. Explosive compaction is, however, dangerous and difficult to control. Compaction in a high speed press can