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Reply to comments on "The velocity dependent fracture toughness of epoxy resins"

In reply to Drs Mai and Atkins' comments concerning the velocity dependence of fracture toughness in epoxy resins, we should like to say that we are grateful for their clarification of the stability aspects of the TDCB specimen. We feel that the analysis provided by them endorses our original view that one should exercise caution when faced with a combination of material and test geometry which involves the "crack-jumping" situation. As they quite rightly point out, knowledge of the rate sensitivity of the fracture process is of paramount importance. In the context of our original investigation, however, the fracture toughness measurement was intended as much as a monitoring device for morphological changes in the resin as it was for generating engineering data. It was found that the TDCB specimen provided a reliable source of information on both these aspects and indeed, its very susceptibility to unstable cracking was instrumental in suggesting other investigations concerning the variation of mechanical properties with changes in stoichiometry.

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Observation of processes of superplasticity with the scanning electron microscope

Tensile tests have been performed in the scanning electron microscope on a eutectic Pb–Sn alloy having super-plastic properties. In this way it was possible to follow the dynamics of the events during deformation at a microscopic level. The specimens were electrolitically attacked in order to remove the superficial layer and to expose single grains. Marker scratches were made on the surface with a 0.5 μm diamond paste. The difference in atomic number between Pb and Sn allowed one to distinguish between grains of different composition, especially when using back-scattered electrons.

From the figures some conclusions can be drawn about the mechanisms acting during the deformation of this superplastic alloy. The grains which originally formed the surface remain at the surface during the entire deformation (grains denoted by 0, 1 ... g, a, h, m, n, in Fig. 1). They do not change their form to a measurable extent. During the initial period of deformation they are shifted against one another but are later separated by new grains coming to the surface (Fig. 1d). This is only possible if the grains slide against one another. Our observations are limited to the

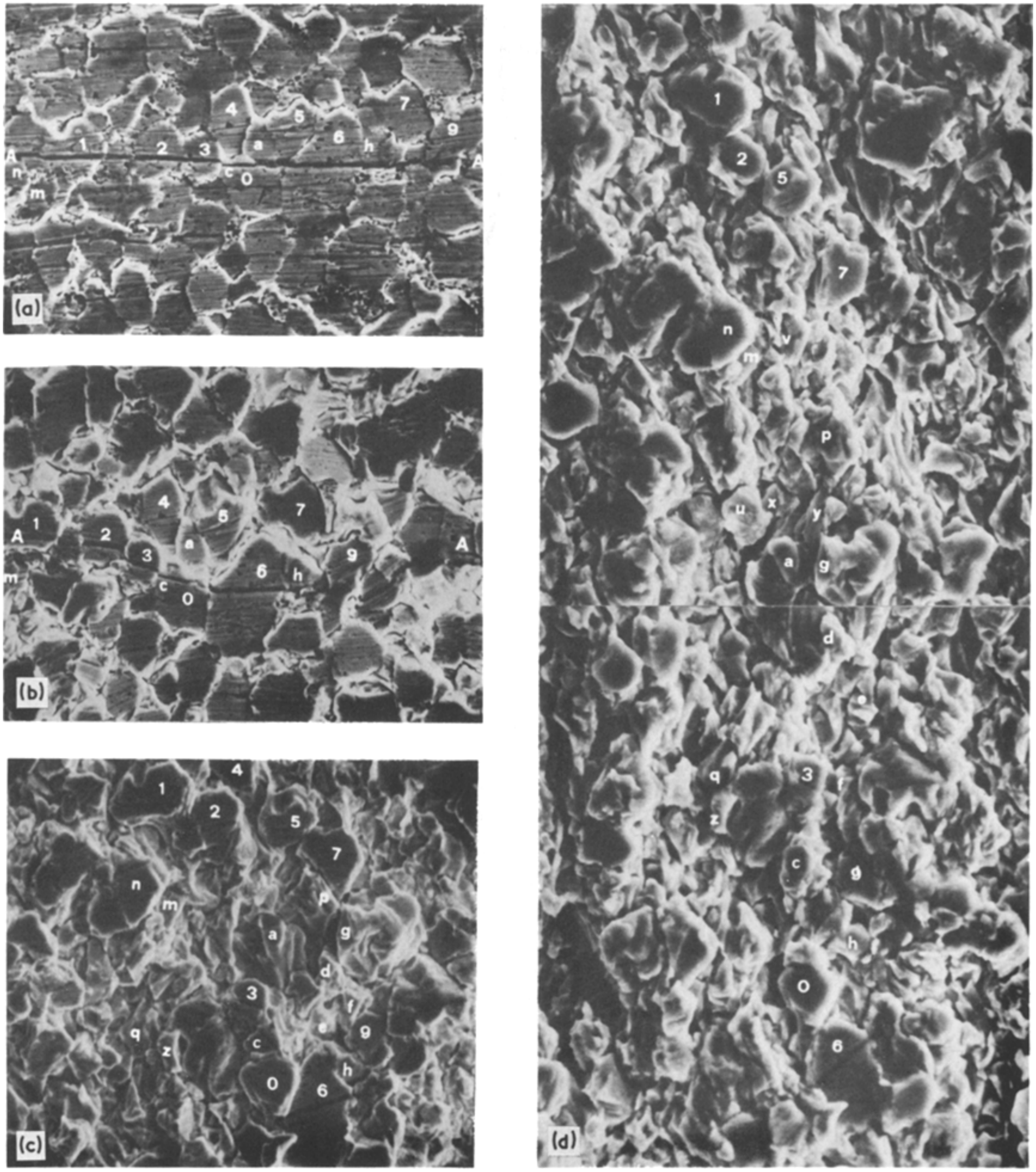

Figure 1 **Sequence of micrographs taken with the SEM of the same region of a Pb–Sn specimen during deformation. It shows the relative displacement of original surface grains and the appearance of new grains on the surface, coming from the interior of the material. The degree of average deformation of the specimen in engineering strain on the different micrographs is: (a) 40%, (b) 90%, (c) 200%, (d) 320%. Secondary electrons; 15 kV; × 1250.**

surface and one may argue that the processes in the bulk are different. However, during deformation new grains appear on the surface from the interior, as can be seen from the photographs (Fig. 1c, d); therefore, grain-boundary sliding must also have been operating in the interior of the specimen.

It is possible to obtain some information about the mechanisms of "accommodation" that allow sliding from some of the grains coming to the

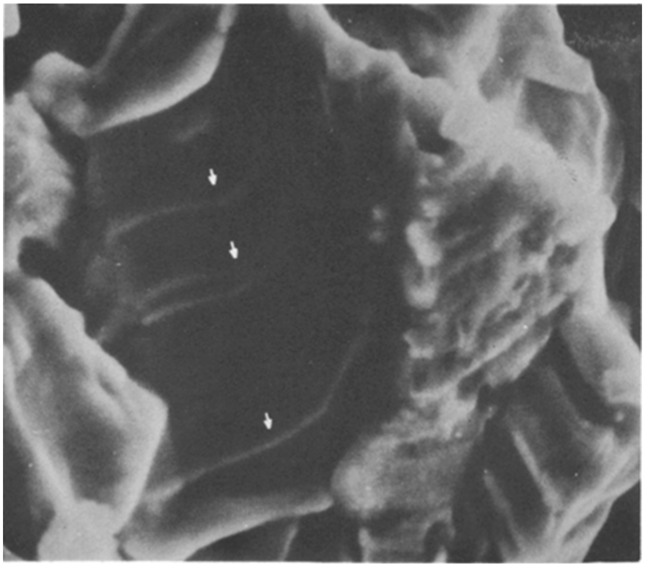

Figure 2 Slip-lines on a Pb-grain coming to the surface from the interior of the material. Secondary electrons; 15 kV; × 900.

surface. In our case slip-lines were noticed on many of these grains (Fig. 2). These slip lines strongly suggest the presence of a mechanism for dislocation motion which acts in parallel to the diffusion mechanism. The grains which were originally on the surface are deformed very little or not at all. This may be due to the fact that, after a certain deformation they stand out of the surface (Fig. 1c) and are therefore under stresses lower and of different type than those in the bulk.

The sequence of photographs shows a great analogy with the simulation made by Ashby and Verrall [1] with a centrifuged emulsion of mineral-oil and dyed detergent, and with the model elaborated by Hazzledine and Newbury [2]. It is evident from our observations that the "accommodation between grains" occurs by diffusion and dislocation motion.

In the photographs it is possible to notice the following facts:

(1) During deformation grains rotate randomly. This can be seen by following the sections of the scratch during deformation through Fig. 1a to d. (Follow, for example, grains 2 and 6.)

(2) There is sliding between the grains. The contrast in Fig. 1a is due to this sliding. In the following photographs, the sliding is shown up by the splitting up of the scratch in the sequence 1a, b, c, d.

(3) The surface in the neighbourhood of the grains is changing. This means that adjacent grains do not normally remain together. New grains migrate to the surface, a further demonstration of grain-boundary sliding. (For example grains 5 and 6.)

(4) The dimensions and the equiaxed form of the grains remain constant. The surface changes and constant form of the grains is characteristic for this material and is not observed in normal plasticity.

(5) In some cases, slip-lines are observed on grains coming to the surface (Fig. 2); an indication that dislocation motion is one of the processes which release internal stresses and make grain-boundary sliding possible.

(6) At high deformation there is a tendency for groups of grains to stick together without relative movement during a certain stage of deformation.

The experiments showed that for the Pb–Sn eutectic alloy a model for superplasticity should include grain-boundary sliding as a main deformation process. Dislocation motion and diffusion may contribute to accommodate the stress-concentrations which build up during sliding.

Acknowledgement

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The initial growth of GaAs layers

The Ga/$AsCl_3$ technique has now become widely used for the production of high-quality layers of GaAs [1]. In this method, $AsCl_3$ vapour is used as the transporting agent in a system in which layers of the semiconductor are grown on some substrate. It is generally assumed that the rate of growth of the GaAs on the substrate does not change during the growth cycle. Shaw [2], for instance, states that in his experiments the rate was uniform up to a growth time of 90 min. He does not give his evidence for this however. Seki *et al.* [3] show results indicating that growth was linear with time on their apparatus over a period of 2 h, but their measurements were taken only at 30 min intervals. In this note we present evidence that although the growth rate is uniform for long-time growths, the rate during the first few minutes is very much higher.

All of the substrates were (1 1 1) slices of GaAs, and the growths were carried out using the gallium face. The growth surface was polished using a 10% solution of NaOCl on a polishing pad [4]. About 20 μm was removed, and a high quality polished surface was obtained. Before mounting in the specimen holder, the substrates were washed in distilled water and a degreasing agent.

A simplified diagram of the apparatus is shown in Fig. 1. The growth technique employs two stages. The gallium in the boat is first saturated. Pure hydrogen is passed through the $AsCl_3$ bubbler where the reaction takes place:

$$2\,AsCl_3 + 3H_2 = 6HCl + \tfrac{1}{2}As_4.$$

The gallium is held at 850° C and it dissolves the arsenic, forming a crust of solid GaAs in the boat on top of a Ga/As liquid. The substrate can now be introduced. The reaction

$$2GaAs + 2HCl = 2GaCl + \tfrac{1}{2}As_4 + H_2$$

then takes place at the boat. The vapours flow to the substrate end of the reaction tube where the As_4 and GaCl react to deposit GaAs. In the experiments described here, the substrates were held at 750° C. In order to time the growths accurately, a specimen holder fitted with a shutter was used. When the shutter was closed, the specimen was cut off from the gas flow. It was operated from outside the apparatus.

The first stage of the experiment was carried out separately, i.e. a ready-saturated boat was put into a clean reaction tube, together with a specimen holder containing a substrate. The furnaces were brought up to temperature with a flow of pure hydrogen passing through the apparatus, and with the shutter open, so that the specimen was in the hydrogen flow. Some time after reaching operating temperature, the shutter was closed and the hydrogen was diverted through the $AsCl_3$. The gallium source had become slightly under-saturated during the initial flushing and it generally took about 4 min to become re-saturated. The shutter was then removed, and growth commenced. At the end of the pre-determined growth time, the shutter was replaced. Growths were carried out for times in the range 1 to 60 min.

Growth rates were determined by weighing the sample before and after growth. The average rate for twelve samples for a growth time of 1 h was 27 μm h^{-1}. It is difficult to compare this with

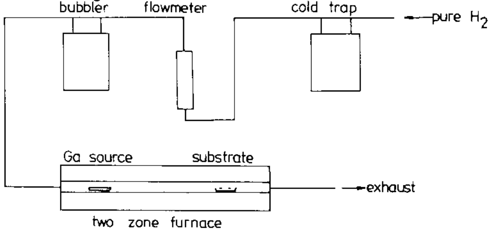

Figure 1. Ga/$AsCl_3$ growth apparatus.