each other due to the shear stresses generated. A dramatic evidence of this phenomenon is seen Fig. 5. Again, one notices that crack started at the boron/core interface. Also to be noted is the separation of the boron fibre/aluminium matrix interface. In conclusion then, all three means enumerated above operate as result of thermal fatigue involving greater than 100 cycles and lead to a much severe microstructural damage in these commercially produced Borsic\*/Al composites.

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# The etching of (1 1 1) As surfaces of GaAs with nitric acid

Chemical etching is a common process used for the study of the properties of semiconductor materials. In the case of GaAs dislocations are usually detected on the (1 1 1) Ga plane using a nitric acid solution (Schell etch). However little attention has been paid to the reaction of nitric acid on (1 1 1) As surface apart from the observation that  $As_2O_3$  crystals are deposited in it [1].

Results are presented here which show that under certain conditions of solution strength and volume a different etching behaviour is found.

The material used for these experiments was Cr doped (111) orientated bulk GaAs with the two (111) surfaces being etch polished using sodium hypochlorite [2] to produce a mirror-like surface finish. The material was then cleaved into samples about  $2 \text{ mm} \times 1 \text{ mm}$  and cleaned in acetone, trichloroethylene and methanol in an ultrasonic cleaner.

The etched surfaces were studied in an SEM (Cambridge stereoscan Mk IIA) and in some case it was found advantageous to coat the samples with a thin ( $\sim 300$  Å) gold film, due to the insulating nature of the surface films.

The dilution ratio of the etchant being the ratio of the volume of water to that of the concentrated acid.

When the samples were etched in 25 ml nitric © 1976 Chapman and Hall Ltd. Printed in Great Britain.

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acid solutions of dilution ratios 1.5 and 2 for times between 4 and 60 min without any agitation at room temperature cubic crystals were produced on their surfaces in a random manner (see Fig. 1). A microprobe analysis of these crystals showed them to contain As and subsequently X-ray diffraction showed these crystals to be Arsenolite  $(As_2O_3)$ . According to Kyser and Millea [1] these adhere to the sample surface by means of a raised pyramidal base of GaAs, although here this was only found when a short etching time (25 sec) and a nitric acid solution dilution ratio of 1 were



Figure 1 Cubic As<sub>2</sub>O<sub>3</sub> crystals, × 1327.



Figure 2 Pyramid of GaAs, × 3260.

used, in which case insufficient  $As_2O_3$  crystals were produced to cover all the GaAs pyramids, which are shown in Fig. 2. The structure of these pyramids is seen to be produced by the preferential etching of various crystallographic planes.

When the above experiments were repeated with the solution being agitated continuously no  $As_2O_3$  crystals were formed on the sample surface. Similarly when 150 ml of the etchant was used with and without agitation, for etching times up to 8 h with solutions of dilution ratio 1.5, no  $As_2O_3$  crystals were formed although other etch



Figure 3 Etch pits on the (111)As surface, × 6530.

features were produced on the sample surface. The absence of any  $As_2O_3$  crystals here is probably due to the larger solution volume and/or agitation of the solution allowing the  $As_2O_3$ , which is only slightly soluble in water, to dissolve more easily in the water of the solution thereby showing up the etching characteristics of the (111)As surface. One sample whose surface was scratched was etched for 10 min in 150 ml nitric acid solution of dilution ratio 1.5 without agitation. A subsequent examination of its surface showed a series of deep etch pits of a characteristic triangular shape and



Figure 4 (a) Dendrites of  $As_2O_3$  produced by etching the (111)As surface of GaAs in concentrated HNO<sub>3</sub>, × 1238; (b) A micrograph of an individual dendrite, × 3090.

orientation at the location of the scratch, as shown in Fig. 3. Since scratches are associated with linear arrays of dislocations and the etching of the above scratches produced characteristic etch pits it is possible that these etch pits are dislocation etch pits. To determine the orientation of these etch pits the sample was cleaved; it was found that the sides of the etch pits were parallel to the (110) cleavage plane.

When concentrated nitric acid was used as the etchant then dendrites of  $As_2O_3$  were produced, as seen in Fig. 4a and b. These crystals were confirmed by X-ray diffraction and microprobe analysis experiments to be crystals of Arsenolite  $(As_2O_3)$ .

The results presented here suggest that the limited solubility of  $As_2O_3$  in water produced different etching characteristics, depending on the solution volume, concentration and amount of solution agitation used. Etch pits, which are believed to be dislocation etch pits, have been identified.

## The effect of triple junctions on grainboundary migration

The rate controlling process in recrystallization and grain growth is usually considered to be the motion of grain boundaries. The necessary adjustment or the motion of triple junctions is not considered important. For two dimensional grain growth in a thin sheet, the effect of grain-boundary grooves [1, 2] has been suggested. While the groove effect is a surface effect, the junction effect is a bulk effect. This note presents experimental evidence of both effects.

For the study of grain growth kinetics, it is convenient to observe boundary motion *in situ* inside a microscope [3]. Camphene was chosen because it recrystallizes at room temperature and it is a b c c globular plastic crystal with a rotational transition temperature of  $-120^{\circ}$  C [4]. Thus at room temperature, camphene may simulate the behaviour of b c c metals at high temperatures.

As-received, practical grade camphene (95% purity) from Eastman Kodak contains an orange-brown liquid. The liquid was removed and the solid recrystallized five times from the melt and one time from alcohol. It was then sublimed five times in a 2 mm Hg vacuum. Camphene thus purified was col-

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ourless, soft and sticky.

A small flat piece of the purified camphene was placed on a microscope slide and inside a small vacuum cell which is covered with another microscope slide as shown in Fig. 1. The assembly was



Figure 1 Microscope stage for observing grain growth in camphene.