of failure equal to the surface energy has been computed by Bayer and Cooper [12] using published values of modulus and surface energy and their temperature dependence. These computations, with appropriate modifications for the present instance, are shown as the dashed line in fig. 1. It is quite evident that the data, even below 350°C, are not consistent with the Griffith mechanism.

Another interesting aspect of the data is that in spite of the large temperature dependence, the strength remains at a level near 100 ksi at  $1325^{\circ}$  C. One of the potential uses of sapphire is in composites for jet engines. In such an application, the stresses on moving parts are proportional to the mass of the material. Thus, the specific strength (fracture stress/density) is often more significant than the actual strength. On this basis, the strength of the sapphire filament is approximately 750000 in. at  $1325^{\circ}$  C.

## Acknowledgements

The author wishes to thank Dr J. T. A. Pollock for many helpful discussions and Mr Richard Todino for assistance with the experimental work. This work was supported by the Air Force Materials Laboratory, Nonmetallic Materials Division, Wright-Patterson Air Force Base, Ohio, under Contract No. F33615-69-C-1369.

## Surface Damage Produced By Sputtering of Silicon

Recently the phenomenon of sputtering has been applied to materials research, for example in the preparation of thin foils for transmission electron microscopy [1]. The sputtering conditions used are generally very similar. A beam of low energy inert gas ions strikes the specimen at a shallow angle, dislodging atoms from the surface. Ion energies vary from 1 to 10 kV, and the removal rate, which depends on ion type, energy, current, angle of incidence, and specimen type, is usually between 0.5 and 5  $\mu$ m per hour.

When using sputtering, it is important that the other effects of ion bombardment should not be neglected, these being the incorporation of a very large number of impurity atoms in the material, and the damage produced by collisions between ions and target atoms. Both impurities and damage will be concentrated close to the surface, and can markedly affect some physical properties of this part of the material.

© 1972 Chapman and Hall Ltd.

## References

- 1. M. L. KRONBERG, Acta Metallurgica 5 (1957) 507.
- 2. H. E. LABELLE, JUN. and G. F. HURLEY, SAMPE J. 6 (Jan. 1970) 17.
- 3. H. E. LABELLE, JUN. 6 (1971) 581 Mats. Res. Bull.
- 4. G. F. HURLEY and J. T. A. POLLOCK, accepted for publication in *Met. Trans.*
- 5. J. B. WACHTMAN, JUN. and L. H. MAXWELL, J. Amer. Ceram. Soc. 37 (1954) 291.
- 6. Idem, ibid 40 (1957) 377.
- 7. Idem, ibid 42 (1959) 432.
- 8. R. J. CHARLES, "Studies of the Brittle Behavior of Ceramic Materials," ASD-TR-61-628, pp 370-404.
- 9. M. L. KRONBERG, J. Amer. Ceram. Soc. 45 (1962) 274.
- 10. H. CONRAD, G. STONE, and K. JANOWSKI, *Trans. Met. Soc. AIME* 233 (1965) 889.
- 11. S. S. BRENNER, J. Appl. Phys. 33 (1962) 33.
- 12. P. D. BAYER and R. E. COOPER, J. Mater. Sci. 4 (1969) 15.
- 13. Idem, ibid, 2 (1967) 233.
- 14. R. L. MEHAN and E. FEINGOLD, Proc. 12th Sagamore Army Materials Research Conf. 12 (1965) 355.

Received 18 October and accepted 29 October 1971.

G. F. HURLEY Tyco Laboratories Inc. Bear Hill Waltham, Massachusetts, USA

In this investigation, silicon has been bombarded with 4kV N<sup>+</sup> ions at an angle of  $25^{\circ}$  to the surface, and the depth of damage measured. using both electrical and electron microscope techniques. The ion-beam etching equipment was specifically designed for the measurement of depth distribution of dopants implanted into semiconductors, and is fully described elsewhere [2]. Briefly, ion-etching is used to successively strip thin layers from the centre region of a van der Pauw pattern, and measurements of the sheet resistivity at each stage enable the bulk resistivity, and hence the carrier concentration profile, to be obtained. (Curve-fitting by computer was used to reduce scatter.) The total amount of silicon removed is measured interferometrically from the height of the step formed between etched and unetched material. The sheet resistivity was measured at depth intervals of 100 Å.

This procedure was followed for silicon implanted with 40 kV B<sup>+</sup> ions to a dose of  $10^{15}$  cm<sup>-2</sup>, and annealed at 900°C for 30 min,

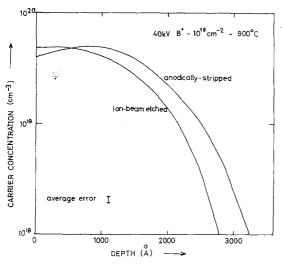


Figure 1 Comparison between ion-beam-etched and anodically-stripped profiles.

the resulting carrier concentration profile being shown in fig. 1. This is compared with the true profile, measured using anodic stripping on a specimen given identical implantation and annealing treatment to the above. It is clear that the only major difference between the curves is a shift of the ion-beam-etched profile about 400 Å towards the surface. This is considered to be a result of the damage produced at the surface by the ion-beam etching. When silicon is ionbombarded, the damage is known to build up to form an amorphous layer at a dose around  $10^{15}$  cm<sup>-2</sup> [3], and such an amorphous layer is known to have a high resistivity. Consequently, any ion-beam-etched silicon sample would be expected to have such a high-resistance surface layer. After a few seconds' etching, an equilibrium is established between the increase in depth penetration of the layer with dose, and the rate of removal of material from the surface by sputtering. The layer thickness will therefore quickly stabilise under constant etching conditions. The contribution of such a high-resistivity layer to the sheet resistance of the sample will be negligible, and therefore from that standpoint it has been effectively removed. Interferometric measurements of the amount of material removed, on the other hand, treat the amorphous layer as an integral part of the silicon, and therefore there should be a difference between the physical and electrical determinations. This will be the discrepancy noted in fig. 1.

The presence of a surface amorphous layer

can be verified using the phenomenon of electron-channelling patterns (ECPs), the quality of which depend strongly on surface crystalline perfection. This dependence has been studied both experimentally and theoretically [4, 5] and the results relate ECP contrast and resolution to amorphous layer thickness.

When the ion-beam-etched silicon specimen described previously was examined in the scanning electron microscope, only a poor quality ECP could be obtained from the etched region. The amount of contrast, relative to the unetched part of the sample, was 30% and the (440) line was just visible. Using these figures in conjunction with the above results [5], the amorphous layer thickness was determined to be  $275 \pm 25$  Å.

Below the amorphous layer there will be damaged regions in an otherwise perfect crystal and these, while not significantly altering ECP contrast, can still have a marked effect on the electrical behaviour. Taking this into consideration, the amorphous layer thickness agrees well with the depth of damage estimated from the electrical measurements to be 400 Å.

It is worth noting that when an ion-beamthinned silicon specimen is examined in the transmission electron microscope, no evidence of damage is generally seen, and it is often assumed that no damage occurs. The present work, using more sensitive methods of detection, shows that surface damage is present, extending to a depth of 300 to 400 Å. The situation may be much worse for other materials, and this should be kept under consideration when sputtering is used for materials processing.

The author gratefully acknowledges useful discussions with Dr G. R. Booker, and financial support from the Science Research Council.

## References

- 1. D. J. BARBER, J. Mater. Sci. 5 (1970) 1.
- 2. S. M. DAVIDSON, J. Phys. E. 5 (1972) 23.
- 3. D. J. MAZEY, R. S. NELSON, and R. S. BARNES. *Phil. Mag.* 17 (1968) 223.
- 4. S. M. DAVIDSON, Nature 227 (1970) 487.
- 5. *Idem*, Proc. 7th Int. Conf. on Electron Microscopy, Vol. I (Grenoble 1970) 235.

Received 23 November and accepted 8 December 1971

> S. M. DAVIDSON Department of Metallurgy University of Oxford, UK