effect of ions rebounding at grazing incidence and sputtering in a second collision has been ignored.

The computer programme has successfully followed the development of a sinusoidal surface to an equilibrium form. The angle at which the sputtering coefficient is again equal to the normal incidence value (θ_n) is of extreme importance; in three-dimensional topography, cones will develop with semi-vertical angle ($\pi/2 - \theta_n$). Experimental observation of such cones is unlikely as the equilibrium does not occur until some 100 mm of surface is eroded. Intermediate stages in the progress to equilibrium will, however, be seen and existing obervations support this model.

Although it is believed that the present calculations present the general details of the topographical development, further simulation with different amplitudes to wavelength values and other forms of the $S - \theta$ function are being investigated to study the development of real surfaces.

References

1. M. J. NOBES, J. S. COLLIGON, and G. CARTER. *J. Mater. Sci.* **4** (1969) 730.
2. G. CARTER, J. S. COLLIGON, and M. J. NOBES. *ibid* **6** (1971) 115.
3. G. K. WEHNER, *J. Appl. Phys.* **30** (1959) 1762.
4. A. D. G. STEWART and M. W. THOMPSON, *J. Mater. Sci.* **4** (1969) 56.
5. V. MARINKOVIC and B. NAVINSEK, Proc. 3rd European Regional Conf. on Electron Microscopy, Prague (1964) pp. 311-12.

Received 13 October
and accepted 18 November 1971

CRISTINA CATANA
J. S. COLLIGON
G. CARTER
Department of Electrical Engineering
University of Salford
Lancashire, UK

Short-term Elevated Temperature Tensile Behaviour in 0° Sapphire Filament

The structure and resulting complicated nature of the slip process in sapphire [1] give this material potential use as a structural material at elevated temperature. At the present time, data exist for the strengths of whisker and bulk forms of sapphire at elevated temperature showing more than an order of magnitude variation in comparative strengths. This note reports studies of the elevated temperature strength of a newer type of bulk material available in very long filament form having room temperature properties approaching the whisker values [2-4].

The first measurements of the high temperature strength of bulk sapphire crystals were carried out in tension, compression and bending by Wachtman and Maxwell [5-7] who demonstrated that macroscopic plastic deformation could be induced above 900° C when the basal plane was favourably oriented for slip. Creep by slip on this system was observed at low resolved stresses, 11 to 12 ksi, at 1000° C. This behaviour was shown qualitatively to be similar to metals at lower temperatures. The same authors [7] also carried out a study of the short term properties in bending of sapphire having the 0 and 45° orientations as a function of temperature up to 1000° C. The strength was shown in both cases to show a minimum value in the range 400 to 600° C. The minimum was lowest in the 0° samples, while the maximum was the greatest in the 45° samples. In the latter case, the 1000° C strength was 90 ksi greater than the room temperature strength. This qualitative behaviour was rationalised by the hypothesis that plastic crack blunting became operative as temperature increased and was most effective in those orientations favouring slip. The minimum strength has been observed by others in experiments carried out in vacuum [8].

More recently, tension tests above 1000° C have demonstrated that sapphire displays a striking yield drop and strain rate effect in tension tests so that, from 1100 to 1500° C, the strain rate as well as the temperature is a significant determinant of fracture stress [9, 10]. The samples in these experiments were 60° rods and were favourably oriented for slip on the basal system.

© *1972 Chapman and Hall Ltd.*

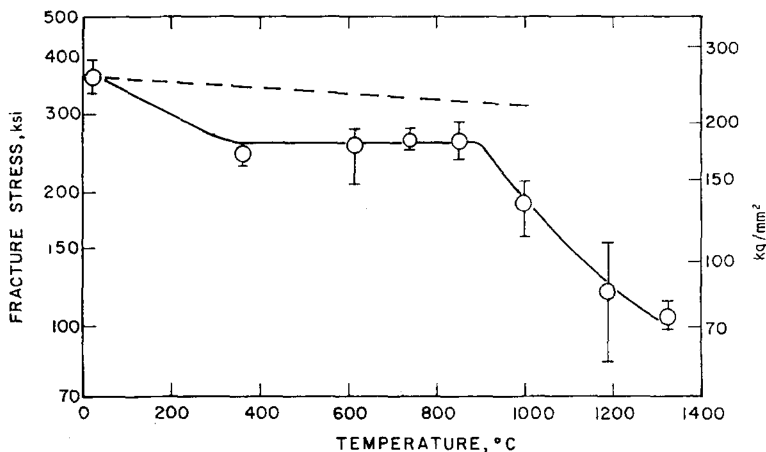

Figure 1 Temperature dependence of the fracture strength of 0° sapphire filament.

The strength of sapphire whiskers is reported to be dependent on temperature [11, 12], size, [13, 14], orientation [13], and probably on strain rate above 630° C [11]. Although there is a large scatter in whisker strength, average performance at 25° C is usually near 1000 ksi at room temperature. Strengths of 300 to 600 ksi at 1100° C and 150 to 300 ksi at 1500° C are given by different authors [11, 12]. No clear evidence of plastic deformation is claimed at any temperature. However, analysis of the detailed temperature dependence of the fracture stress has been used to suggest flow-aided fracture for all orientations at high temperatures and particular orientations at lower temperatures.

In the present work, the strength of *c*-axis sapphire filament was investigated over the temperature range from 25 to 1325° C. The filament was grown from the melt by a process which has been described elsewhere [3]. All the samples were taken from a single continuous length of filament having a diameter of approximately 0.009 in. and room temperature strength of 365 ksi. The samples had beads melted on their ends in an oxy-hydrogen torch and were gripped by laying their ends parallel to the corrugations in cardboard tabs and cementing in place with epoxy resin. The tensile testing apparatus was comprised of a Hounsfield Tensometer and a small RF set which suscepted to a 0.19 in. Pt susceptor, 1.5 in. long. The susceptor was split longitudinally to allow insertion of the sample. With the Hounsfield it is not possible to control strain rate. However, the machine was cranked at a rate so as to give a stress rate equivalent within a factor of 2 of the conventional room temperature stress rate attained at 0.02 min^{-1}. Tests were carried out in air.

Results of thirty-seven test samples are given by the solid line in fig. 1. The range of values for each averaged data point are defined by the vertical bars. The strength declines to 260 ksi near 350° C and is then level to near 850° C. Thereafter, it declines again, reaching 104 ksi at 1325° C.

The level region between 350 and 850° C is almost certainly an artifact of the test method. If, as expected, the strength response to increasing temperature goes through a minimum then maximum stress value, samples tested by gripping outside the heated zone would fail at one end of the furnace giving a level strength curve from the minimum to a temperature above the temperature of maximum stress. Therefore it is concluded that the data of fig. 1 are consistent with the forms of response for other types of bulk sapphire.

The strength levels observed are much higher than those previously noted in bulk sapphire. In bend testing, 0° sapphire had been shown to have a strength of 43 ksi at 1000° C [7]. The minimum strength in that case was reported to be 28 ksi. In the present case, the strengths were 190 and 260 ksi respectively at 1000° C and at the temperature of minimum strength. Whisker strengths reported for the same temperature range vary from 1.5 to 3 times the present values.

The dependence of strength on temperature is far too large to be accounted for by a purely elastic crack propagation mechanism. The dependence of failure stress on temperature in sapphire for the Griffith criterion with the work

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° 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° 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.

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

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 μm 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.

In this investigation, silicon has been bombarded with 4kV N^+ ions at an angle of 25° 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^+ ions to a dose of 10^{15} cm^{-2}, and annealed at 900°C for 30 min,

© *1972 Chapman and Hall Ltd.*