

a capillary, the composition of the solidifying crystal is constrained to be identical with that of the melt, all of the liquid constituents passing upwards through the capillary being incorporated into the solid. Thus, if planar interface growth is achieved under selected growth conditions, this interface will not be modified as solidification continues by impurity rejection, resulting in constitutional supercooling and breakdown into a cellular growth front. Other possible advantages associated with this separation of growth area and melt reservoir are deliberate variation of composition during growth and continuous replenishment of the reservoir.

It is probable that the theoretically highest planar growth rates may be achieved in many eutectic systems using EFG. Ignoring heat losses by radiation from the meniscus surface, Chalmers *et al* [3] showed that, for sapphire, growth by means of a planar interface depended on the crystal being the sole means of extracting heat from the growth region. That is, at the maximum growth rate consistent with planar growth, the thermal conductivity of the solid sapphire and heat loss by means of an optical light pipe effect were the controlling parameters. Assuming zero heat transfer across the liquid growth film from the die surface, they calculated a maximum growth rate of 2.4 cm min⁻¹. One of the present authors [4] obtained an experimentally measured maximum growth rate for light pipe quality sapphire of 3.0 cm min⁻¹. The achievement of this growth rate was made possible by careful control of the growth conditions and the selection of tungsten (which has a substantial thermal conductivity at 2000°C) as die material. If

similarly beneficial growth environments can be arranged for the directional solidification of eutectic systems, there is no obvious reason why near maximum planar growth rates should not be achieved over the entire length of the shape grown.

Acknowledgements

The authors wish to thank B. Chalmers and A. I. Mlavsky for many stimulating discussions.

References

1. H. E. LA BELLE JUN, *Mat. Res. Bull.* **6** (1971) 581.
2. J. T. A. POLLOCK, *J. Mater. Sci.* **7** (1972) 631.
3. B. CHALMERS, H. E. LA BELLE JIN and A. I. MLAVSKY, *Mat. Res. Bull.* **6** (1971) 681.
4. J. T. A. POLLOCK, *J. Mater. Sci.* **7** (1972) 787.
5. *Idem, ibid* **9** (1974) 323.
6. F. H. COCKS and J. T. A. POLLOCK, *J. Appl. Phys.* in press.
7. L. M. HOGAN, R. W. KRAFT and F. D. LEMKEY, "Advances in Materials Research" (edited by H. Herman) Vol. 5 No. 1 (Interscience, New York, 1970).
8. W. A. TILLER, "Liquid Metals and Solidification" (ASM, Cleveland, Ohio, 1958) p. 276.

Received 14 October

and accepted 22 October 1973

J. T. A. POLLOCK
*Australian Atomic Energy Commission,
 Research Laboratories, Lucas Heights,
 Sutherland, New South Wales,
 Australia*

R. STORMONT
*Tyco Laboratories Inc,
 Waltham, Massachusetts, USA*

Fracture strength in tension of *a*-axis filamentary sapphire grown by EFG

The tensile fracture strength of *c*-axis filamentary sapphire grown by the "edge-defined, film-fed growth" technique (EFG) has been extensively investigated and reported in the literature [1-3]. Average room temperature fracture strengths in the range 2.4 to 2.9 GN m⁻² (350 to 425 × 10³ lb f in.⁻²) are commonly measured for filament grown at rates up to 6 cm min⁻¹ and having a nominal 0.025 cm diameter. This *c*-axis fila-

mentary sapphire retains over 30% of its room temperature tensile strength when tested at 1250°C [4].

Compressive strength data obtained from 3 mm diameter rod samples having growth and loading axes parallel to the *a* and *c* directions, respectively, have shown that despite similar room temperature strengths, the *a*-axis rods exhibit only a slight fall in strength with increasing temperature up to 1300°C [5]. At 1300°C *a*-axis samples had retained 80% of their room temperature compressive strength, com-

TABLE I Fracture strength in tension of *a*-axis filament

Growth speed (cm min ⁻¹)	No. of samples	Diameter of samples (cm)		Average fracture strength MN m ⁻² (lb f in ⁻² .)
		Minor axis	Major axis	
2.3	3	0.018	0.024	2.36 (342 × 10 ⁸)
3.2	4	0.015	0.019	2.07 (299 × 10 ⁸)
3.8	6	0.016	0.022	2.46 (357 × 10 ⁸)
4.3	6	0.0165	0.021	2.44 (353 × 10 ⁸)
5.1	5	0.015	0.021	2.33 (338 × 10 ⁸)
3.8	6	0.015	0.019	2.68 (388 × 10 ⁸)
3.8	6	0.016	0.022	2.53 (367 × 10 ⁸)

pared with close to 5% for the *c*-axis samples. This orientation dependent difference in high temperature compressive strength, which may be related to the availability of slip systems for crack nucleation and propagation [6], could be of great importance in applications where a filamentary sapphire reinforcement would be subject to compressive stresses. The aim of the present study was to investigate the growth, room temperature strength in tension and microstructural properties of *a*-axis filament using techniques and experimental procedures previously involved in the characterization of *c*-axis filament.

The filament growth apparatus has been described elsewhere [7]. Molybdenum crucibles and growth orifices (dies) were used to grow nominally 0.025 cm diameter filament. Since a seed crystal of 150 cm minimum length is required to grow filamentary sapphire, a series of growth experiments was carried out to produce a filamentary seed of the correct orientation. A 5-cm long *a*-axis rod bonded to a length of *c*-axis filament was used as seed to grow a few cm of filament. The short piece of filament grown was confirmed to be of *a*-axis orientation by Laué X-ray examination and, after removing the *a*-axis rod, bonded to the *c*-axis length of sapphire filament. It was now possible to consider growing a length of filament, the seed for which would pass through the guiding system, and, more important, the belt puller. Using this bonded seed a 200-cm long filament was grown. Laué examination confirmed that the growth direction was very close to *a*-axis.

The seed length obtained as described above was used to grow continuously several lengths of *a*-axis filament at speeds in the range 2.5 to 5.1

cm min⁻¹. The filament was coated with paraffin before passing through the belt puller and handled with care. Samples were taken from these lengths and, using a 2.5 cm gauge length, tested in tension to fracture at a strain rate of 0.02 min⁻¹. The collected data are presented in Table I. Note that the filament has a distinctive ellipsoidal cross-section. Average strengths were in the range 2.1 to 2.7 GN m⁻² (299 to 388 × 10⁸ lb f in.²), maximum strengths being measured for growth speeds of 3.8 cm min⁻¹. These strengths compare favourably with room temperature values obtained with *c*-axis filament, since improvements are generally obtained with growth procedure experience.

The continuous crystal growth of *a*-axis filamentary sapphire may be summarized as follows:

1. filamentary material may be grown at rates up to 5.1 cm min⁻¹ if a seed of the correct orientation is available and accurately aligned;
2. when the filament is growing on *a*-axis it has a distinctive ellipsoidal cross-section. The minor axis is in the *c*-direction and if during growth the filament is by chance oriented such that this direction is visually observed, a distinctive reflection is noted from the *c*-plane flat which is present. The minor axis is generally measured to be 40 to 60 μm shorter than the major axis, [110], for filament grown from a 0.026 cm o.d. orifice;
3. if the seed is slightly off *a*-axis or, if during growth for some mechanical reason a misorientation develops, it is immediately made apparent at the filament surface by the occurrence of *c*-plane facets;
4. the occurrence of these *c*-plane facets is accompanied by a fall in tensile fracture strength

($<2.0 \times 10^9 \text{ GN m}^{-2}$), due probably to their stress concentrating effect.

A microstructural examination of the a -axis filament grown was carried out. At speeds greater than 2.5 cm min^{-1} a random void dispersion together with the distinctive void free surface zone previously noted in c -axis filament was observed. Unlike c -axis filament no pattern which can be related to crystallographic planes was evident. The only exception to this randomness was noted in the filament grown at the slowest speed, 2.3 cm min^{-1} . When viewed along the major axis, $[\bar{1}100]$ direction, the voids near the mid-plane section arrayed themselves in near longitudinal linear distributions making a shallow angle ($\sim 10^\circ$) with the growth axis.

a -axis filamentary sapphire is presently being tension tested at temperatures up to 1325°C . The results will be reported at a later date.

Acknowledgement

This work was supported by the Air Force Materials Laboratory, Non-Metallic Materials

Division, Wright-Patterson Air Force Base Ohio.

References

1. H. E. LA BELLE JUN. and G. F. HURLEY, *Sampe* 6 (1970) 17.
2. G. F. HURLEY and J. T. A. POLLOCK, *Met. Trans.* 3 (1972) 397.
3. J. T. A. POLLOCK, *J. Mater. Sci.* 7 (1972) 649.
4. G. F. HURLEY, *ibid* 7 (1972) 471.
5. *Idem*, unpublished data.
6. A. H. HEUER, *Proc. Brit. Ceram. Soc.* 15 (1970) 173.
7. J. T. A. POLLOCK, *J. Mater. Sci.* 7 (1972) 631.

Received 14 October

and accepted 22 October 1973

J. T. A. POLLOCK*

*Australian Atomic Energy Commission,
Research Laboratories, Lucas Heights,
Sutherland, New South Wales,
Australia*

J. S. BAILEY

*Tyco Laboratories Inc.,
Waltham, Massachusetts, USA*

*Work carried out while the author was at Tyco Lab Inc, Waltham, Mass., USA.

A method for assessing the quantity and distribution of glass fibre in an opaque matrix

It is well known that the properties of fibre reinforced composite materials are very dependent on the volume fraction of reinforcement, V_f . During fabrication of such a material it is to be expected that a certain amount of local variation in V_f will arise and, since crack initiation and propagation are more likely to occur in regions deficient in reinforcement, this will be reflected in the spread of results obtained on testing nominally identical specimens. It is necessary, therefore, to be able to determine the distribution of fibre in the finished composite, particularly when the material is being made on a large scale. This has been done for various combinations of materials by taking polished sections and photographing under an optical microscope. This procedure is satisfactory when the components are sufficiently dissimilar in their colour and reflectivity. However, it is not satisfactory for the easy examination of some com-

binations, notably glass fibre in cement and plaster [1].

It has been found that commercially available glass fibre embedded in an opaque matrix is capable of transmitting light, by total internal reflection, over a distance of several centimetres. It is thus possible to use a technique of thick section transmission provided that the depth of the section is less than the fibre length. Fig. 1 is a photograph of a section 1 cm thick taken from an aligned glass fibre reinforced cement composite with continuous reinforcement in the form of strands of approximately 200 filaments. The distribution and orientation of the strands is clearly visible. Fig. 2 shows the individual filaments in one of the strands in the same section.

Increasing interest is being shown in the reinforcement of cement with chopped strands of an alkali-resistant glass [2]. This composite material is made at the Building Research Station in the form of large sheets by a spray suction process. The finished material contains about 5% of chopped strand, approximately 30 mm