

($<2.0 \times 10^9 \text{ GN m}^{-2}$), 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.

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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 *Idem*, unpublished data.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.

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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-

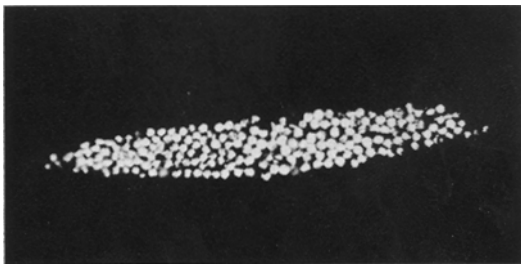
binations, notably glass fibre in cement and plaster [1].

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

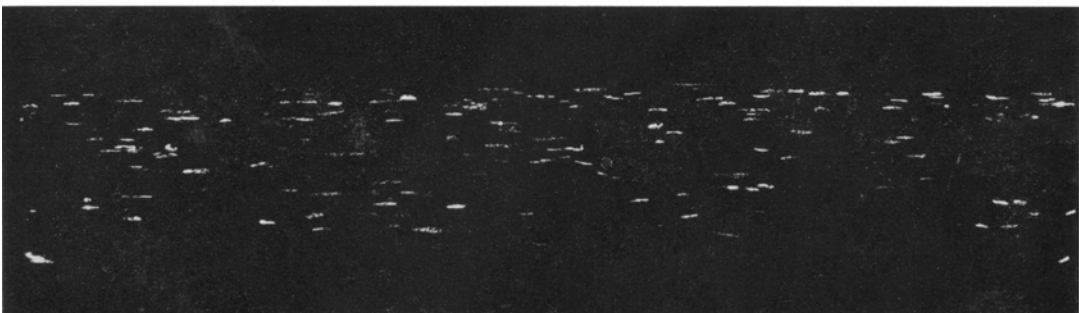


Figure 1 Section of an aligned glass fibre reinforced cement composite taken perpendicular to fibre direction ($\times 5$).



◀ Figure 2 Photograph of a single strand in Fig. 1.

Figure 3 Typical sections of glass fibre reinforced cement with 2-D random orientation. ▼



long, randomly orientated in the plane of the sheet. Specimens for examination by thick section transmission were prepared from test coupons of this composite material which had failed in flexural fatigue. Two parallel cuts were made 6 mm apart, perpendicular to the direction of stress and close to the crack, using a water-fed diamond impregnated cutting wheel. Only a proportion of the strands in the section will be visible by transmitted light but these will be the ones which provide the effective reinforcement in the stress direction. Photographs were prepared by one of three methods, direct contact printing onto a sheet of film, using a camera attached to a microscope with provision for transmitted light; and by photographing the section with an electronic flash gun as the source of illumination. It was necessary in each case to hold the section in a opaque material such a silicone rubber or plasticine but otherwise no further sample preparation was found to be necessary. Some typical photographs are reproduced in Fig. 3.

The technique has proved useful for investigating the number and position of glass fibres in an opaque matrix without the need for elaborate specimen preparation such as is required for thin section work. It also has the considerable advantage of filtering out unwanted information on the matrix structure which would otherwise obscure desired information on fibre distribu-

tion. Sections can be quickly prepared and photographed enabling the distribution of glass to be assessed. This technique has considerable potential for application to the commercial production of materials such as glass fibre reinforced cement and plaster where some inexpensive method of determining the quality of the composite is vitally necessary.

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References

1. A. J. MAJUMDAR and J. F. RYDER, *Glass technology* 9(3) (1968) 78.
2. B. R. STEELE, Prospects for fibre reinforced construction materials, the proceedings of an International Building Exhibition Conference sponsored by the Building Research Station at Olympia, London 1971, pp. 29-39.

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Scanning electron microscopy of reaction-sintered silicon nitride

The microstructure of reaction-sintered silicon nitride is generally agreed to consist of a mixture of phases whose relative abundance, together with the overall density, can be varied with fabrication conditions. The two most important phases, α and β , are morphologically distinct since the α -phase consists of a very fine matte of crystallites while the β -phase is much coarser [1] though individual β -crystals are still much smaller than the initial silicon particles (e.g. [2]). Previous attempts to apply scanning electron microscopy (SEM) techniques to these materials have usually been limited to the examination of fracture surfaces since the fine α -matte obscures details on polished surfaces. However, since failure may have been initiated at some flaw (v, Fig. 1) fracture surfaces may not be truly representative of the bulk microstructure

and thus we have been developing techniques to allow polished surfaces to be examined in the SEM.

The present study has examined both a commercially produced material and some laboratory samples prepared by the technique described by Parr *et al* [2]. In the latter case, commercial grade silicon powder (16 μm mean particle size, 60 μm maximum particle size, purity 98.0%) was isostatically pressed at a pressure of 110 MN m^{-2} and subsequently nitrided in a pure nitrogen atmosphere to a final density of 2.23×10^3 kg m^{-3} .

Cut and polished sections of these specimens were examined both optically and in the SEM. Grain sizes were too small to be distinguished by standard optical techniques and the fine α -matte was found to obscure the coarser features of the microstructure in the SEM image. However, both the fine α -matte and the coarser prismatic β crystals could be readily observed