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 distribution. 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

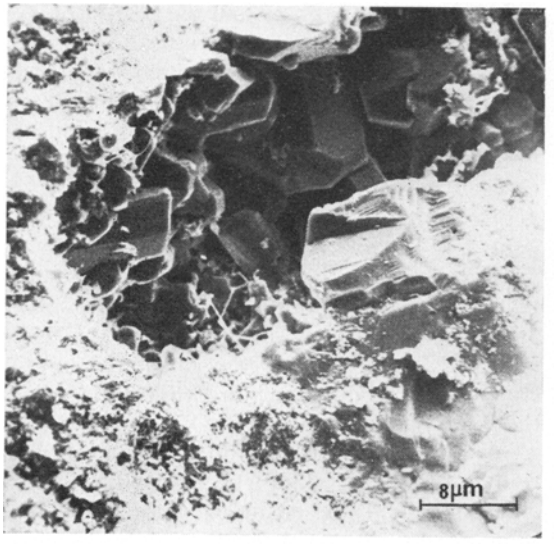

Figure 1 A scanning electron micrograph of a void in a silicon nitride fracture surface. The fine α-matte can be seen in the left foreground, prismatic β inside the cavity and a large unreacted silicon particle in the right foreground. The unreacted silicon may have influenced the fracture path.

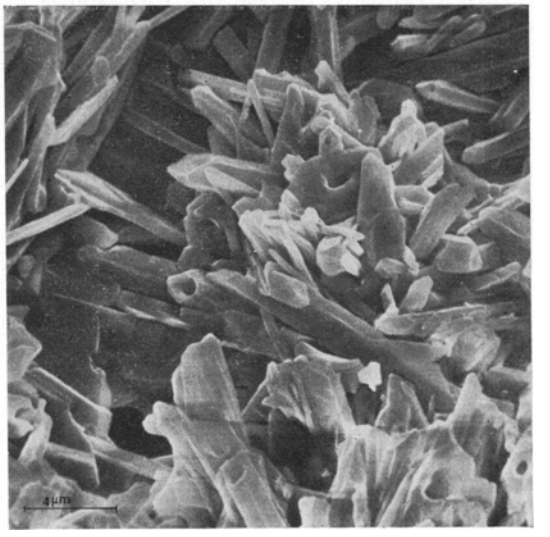

Figure 2 A scanning electron micrograph of an initially polished surface, subsequently etched and ultrasonically cleaned. Many of the hexagonal prismatic β-crystals, which are now clearly visible after removal of the α-matte, display cavities (see text).

in voids in fracture surfaces examined in the SEM (Fig. 1). Similar results have been reported by Barnby and Taylor [1].

Successful studies with polished surfaces have now been achieved by employing further etching and cleaning techniques to remove the surface α-layers. Polished specimen surfaces were etched in 40% HF for 1 h at room temperature and then thoroughly ultrasonically cleaned in an aqueous Teepol solution prior to gold-plating for SEM examination. Fig. 2 shows a micrograph typical of those obtained from both specimen materials using this technique. Well-defined crystals of hexagonal prismatic habit are apparent and these are believed to be the β-phase from a comparison with similar micrographs taken of fracture surfaces of specimens which were known to be predominantly β by X-ray diffractometry.

An immediately interesting feature of Fig. 2 (and all similar micrographs) is the apparent hollow morphology of some of the β crystals. We cannot say whether or not these holes penetrate the whole length of the prisms and absence of similar features on β crystals observed in unetched fracture surface specimens suggests that the effect may be due to etching. It is also noted that the symmetry of the cavity is not the same as that of the external shape of the prism. Whatever the origin of this phenomenon, a through-section inhomogeneity of the β crystals is implied which is difficult to reconcile with any currently proposed growth mechanism.

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References

1. J. T. BARNBY and R. A. TAYLOR, "Special Ceramics 5" (edited by P. Popper) British Ceramics Research Association, Stoke-on-Trent, 1972) p. 311.
2. N. L. PARR, G. F. MARTIN and E. R. W. MAY, "Special Ceramics" (edited by P. Popper) (Academic Press, for British Ceramics Research Association, 1960) p. 102.

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