the third International Conference on Ion Implantation, IBM, Yorktown Heights, USA, 11-14 December, 1972.

References

1. T. R. WILSHAW and R. ROTHWELL, *Nature, Physical Science (Lond.)* **229** (1971) 156.
2. J. WOOLMAN and R. A. MOTTRAM, "The Mechanical and Physical Properties of the British Standard En Steels" (B.S. 970-1955), Vol. 3 (Pergamon Press, Oxford, 1969) 482.
3. N. E. W. HARTLEY and J. F. TURNER, unpublished research.
4. C. MALONEY, private communication.
5. R. HOLINSKI and J. GÄNSHEIMER, *Wear* **19** (1972) 329.
6. M.-A. NICOLET, J. W. MAYER, and I. V. MITCHELL, *Science* **177** (1972) 841.
7. M. J. DEVINE, E. R. LAMSON, J. P. CERINI, and R. J. MCCARTNEY, *Lubrication Engng.* **21** (1965) 16.

Received 4 December 1972
and accepted 29 January 1973

N. E. W. HARTLEY*
W. E. SWINDLEHURST
Applied Sciences Laboratory,
University of Sussex,
Brighton, UK
G. DEARNALEY
J. F. TURNER
Atomic Energy Research Establishment,
Harwell, Berks, UK

*Now at Nuclear Physics Division, AERE, Harwell, Berks.

The microhardness of composite materials

The Vickers and Knoop indenters are the two most commonly used for determining the hardness of materials. The essential difference between them is that the strain field under the Vickers indenter is spherically symmetrical [1], whereas that under the Knoop indenter is not. The latter has a shape anisotropy which makes the strain under it non-symmetrical even in a fully isotropic medium. Recent studies of Knoop hardness anisotropy in single crystals [2-4] have given a clearer insight into the deformation mechanism under the indenter. Discussion has been in terms of shear stresses developed from compressive [5] or tensile [2, 6] forces parallel or normal to the indenter facets, respectively. This means that the principal strains are in directions approximately perpendicular to the long axis of the Knoop indenter. In single crystals, the hardness is a reflection of the stresses developed on active slip planes, when these are small the material does not deform appreciably and so appears hard.

In the case of a highly anisotropic material such as a unidirectional composite, the degree of material anisotropy plays a dominant role in the deformation pattern produced by the indenter. Nonetheless the hardness of the material again reflects its response to the strain field applied by the indenter, as in the case of single crystals.

In order to examine such effects in highly anisotropic materials, we have made Vickers and Knoop indentations at loads ranging from 100 to 500 g in copper-Cu_5Zr and copper-CuZrSi unidirectionally solidified lamellar eutectics (42 and 90.8 vol % copper, respectively) and a 60 vol % carbon fibre-reinforced plastic (CFRP). Studies have been made of the dependence of hardness on relative orientation between the indenter and the fibre (or lamellar) direction. In Fig. 1 is shown the variation in hardness of a copper-Cu_5Zr eutectic as a function of indenter orientation, taking the condition as 0° when the long diagonal of the Knoop indenter or either diagonal of the Vickers is parallel to the lamellae and 90° when it is perpendicular to them. The arithmetic means of sets of ten indentations were taken at each angle and the root mean square error σ calculated. The error bars represent $\pm 3\sigma$. In the case of Vickers indentations, the diagonals are of unequal length (Fig. 2a) because the material is anisotropic. Therefore, we have calculated Vickers hardness values from the individual diagonals rather than averaging pairs as is usual. The exception to this separate treatment occurs at 45° when the diagonals are in an equivalent symmetrical position. In both cases, the fitted curves are sinusoidal but if the Vickers hardness values were to be derived from the mean of the diagonal lengths then no orientation dependence would be observed.

It was clearly shown in all samples that the

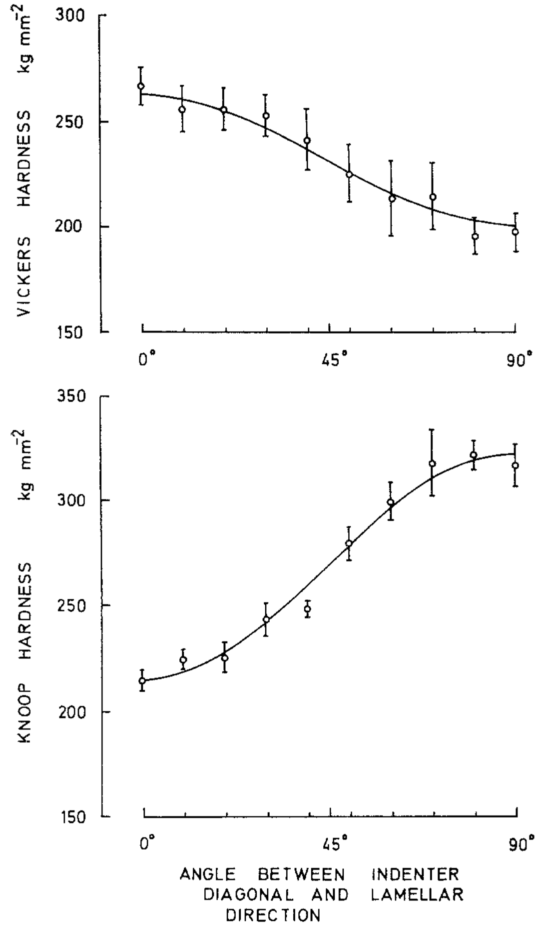

Figure 1 Variation of hardness with indenter orientation.

Knoop hardness is a minimum when the long diagonal lies parallel to the fibres or lamellae (0° position) and maximum when perpendicular to them (90° position), whereas the opposite type of behaviour is shown by the Vickers indentations as seen in Fig. 1. The difference in diagonal length at 0° and 90° can be seen in the example given in Fig. 2a. In Fig. 2b the sides of the indentation which lie parallel to the lamellae are likewise longer than those which lie perpendicular to them, although the diagonals are of equal length. Some Knoop indentations in CFRP are shown in Fig. 3, where the effect of the different orientations of the indenter on the behaviour of the material is quite evident.

A possible explanation of the effects rests upon the shape difference between the two indenters and also upon the highly anisotropic nature of composite materials, in particular their deformation and recovery behaviour. In the case of the Vickers indenter the imposed strain is spherically symmetrical, whereas the stress-field developed in response to it is not, because the material is anisotropic. The stresses are greatest parallel to the fibres because the material is strongest in this direction. While the load is applied the shape of the indentation is assumed to conform to the shape of the diamond. Therefore any irregularities in the shape of the impression must arise during or shortly after the removal of the load. Comparing the length of the diagonals in the 0°-90° case (Fig. 2a), the

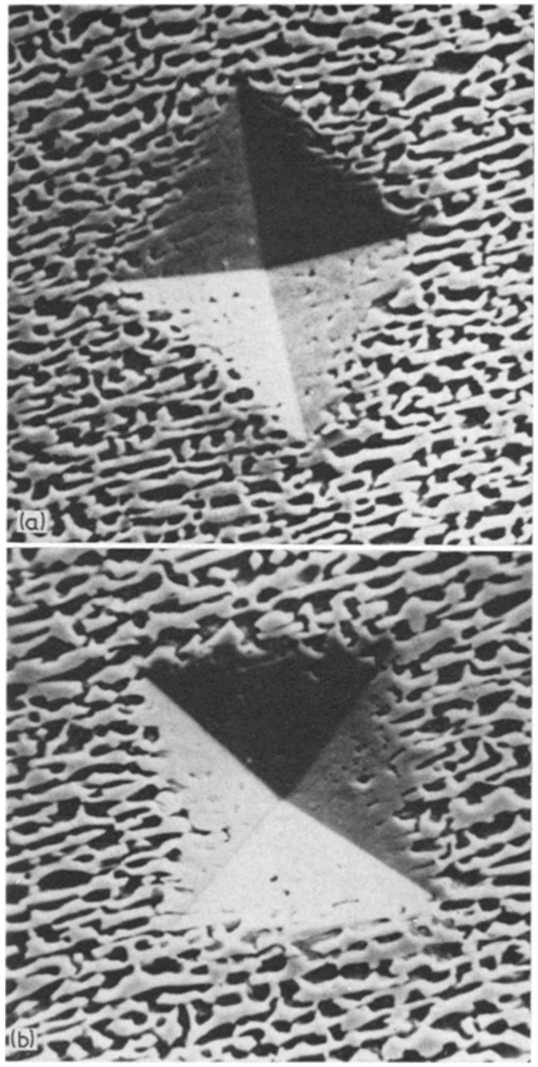

Figure 2 Scanning electron micrographs of (a) 0° – 90° and (b) 45° Vickers indentations in the copper-Cu_5Zr eutectic (500 g load) (× 830).

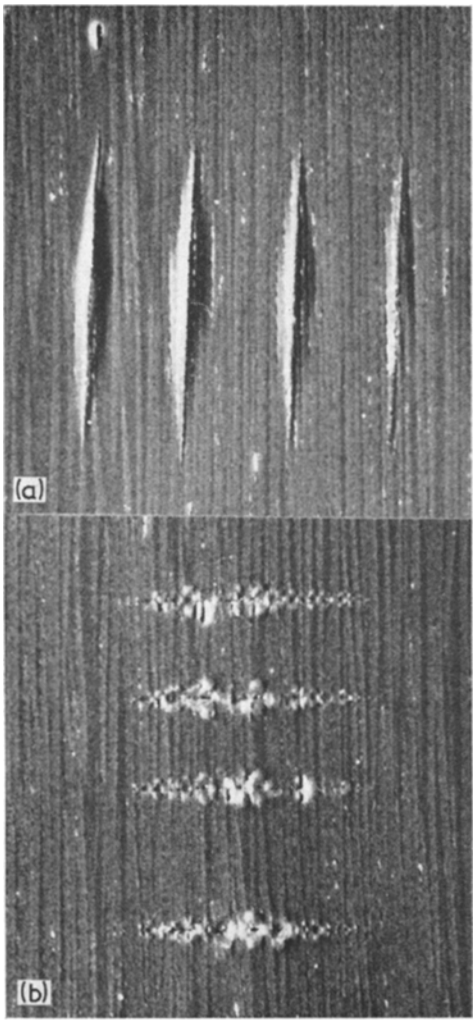

Figure 3 Scanning electron micrographs of (a) 0° and (b) 90° Knoop indentations in CFRP (300 g load) (× 150).

difference is of the order of 15%. We suggest that this effect arises because of greater recovery in the direction parallel to the fibres where the stresses were greatest. Now it has been found in compression tests [7-9] on composite materials that the fibres undergo buckling. The large differences in dimensions seen here could well result from the recovery of lamellae which had buckled under the indenter. Extensive recovery in the diameter of indentations is not seen in single-phase materials where the principal recovery is in the depth [1, 10].

In the case of the Knoop indenter, shape anisotropy leads to strains which are greatest in directions perpendicular to the long axis.

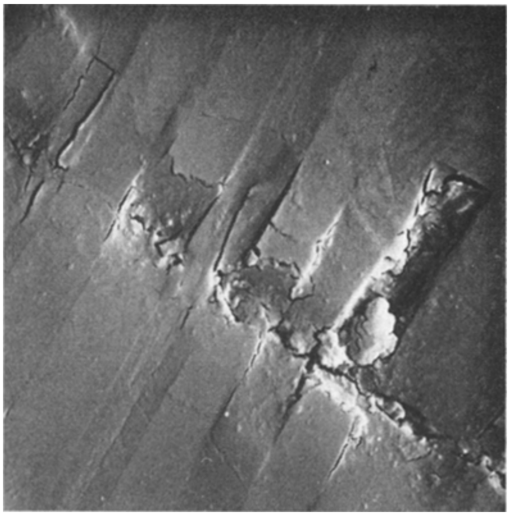

Figure 4 Scanning electron micrograph showing delamination and extensive cracking in a 90° Knoop indentation in CFRP (300 g load) (× 1350).

This shape anisotropy is additional to the high anisotropy of the material being indented. If we consider first the 0° case, then the maximum strain imposed by the indenter is perpendicular to the lamellae, i.e., parallel to the direction of lowest strength. Thus the material appears relatively soft. In the 90° case the maximum strain lies parallel to the direction of greatest strength of the composite; the resistance to deformation is high so that the indentation is small. It is noted that in both cases recovery is perpendicular to the long axis of the indenter and, therefore, does not affect the Knoop hardness values. Hence, we conclude that the hardness variation shown in Fig. 1 is related to the difference in shape anisotropy between the two indenters and their deformation patterns coupled with the high degree of material anisotropy.

The indentations also show interesting deformation effects. In the copper-Cu_5Zr eutectic the intermetallic matrix phase, which is not usually ductile, has clearly been deformed plastically (Fig. 2) during the indentation process. Plastic deformation as opposed to the brittle failure observed under high tensile stress [11], arises because of the high hydrostatic stress component under the indenter.

Very different effects are seen in CFRP (Fig. 3). In the 0° case delamination occurs either as the indentation is made, or as a result of elastic recovery of the fibres which were displaced during the indentation process. At 90° (Figs. 3b and 4) the fibres break. In this case they are bent to much smaller radii of curvature over the sharp intersections of the facets of the Knoop diamond.

Acknowledgements

The authors wish to thank Mrs B. Höhn and Mrs G. Keser for making the numerous indentation measurements involved in this work. They are also grateful to Miss J. Folprecht for the scanning electron microscopy and to Dr B. Bürgel for useful discussions. The CFRP was kindly supplied by Dr P. G. Rose.

References

1. F. P. BOWDEN and D. TABOR, "The Friction and Lubrication of Solids", Ch. 16, Oxford University Press, (1964) p. 320.
2. C. A. BROOKES, J. B. O'NEILL, and B. A. W. REDFERN, *Proc. Roy. Soc.* **A 322** (1971) 3.
3. D. J. ROWCLIFFE and G. E. HOLLOX, *J. Mater. Sci.* **6** (1971) 1261.
4. *Idem, ibid* **6** (1971) 1270.
5. M. GARFINKLE and R. G. GARLICK, *Trans. Met. Soc. AIME* **242** (1968) 809.
6. F. W. DANIELS and C. G. DUNN, *Trans. ASM* **41** (1949) 419.
7. A. S. YUE, F. W. CROSSMAN, H. E. VIDOZ, and M. I. JACOBSON, *Trans. Met. Soc. AIME* **242** (1968) 2441.
8. J. LAGER and R. R. JUNE, *J. Comp. Mat.* **3** (1969) 48.
9. B. J. SHAW, *Acta Metallurgica* **15** (1967) 1169.
10. M. BRAUNOVIC and C. W. HAWORTH, *J. Mater. Sci.* **7** (1972) 763.
11. A. J. PERRY, *ibid* **8** (1973) 443.

Received 2 January and accepted 5 January 1973

A. J. PERRY
D. J. ROWCLIFFE
Brown Boveri Research Center, CH—5401 Baden, Switzerland

Creep strength of single-crystal sapphire filaments

In this note we report the results of investigations of the creep strength of commercially available single-crystal sapphire filaments at temperatures up to 1480°C. The sapphire filament was "Saphikon" obtained from Tyco Laboratories, Waltham, Mass, USA. The material was 250 μm (0.010 in.) diameter and supplied in lengths of 30 m or more (produced by pulling from the melt [1]). The cross-section was nearly circular. With one exception, when the alignment was close to [1$\bar{1}$00], the orientation of the material chosen for examination was *c*-axis, [0001], along the length so that the chance of basal slip was minimized. Although this was the nominal orientation, X-ray examination showed that the true orientation frequently varied by a few degrees from this: in one instance a deviation of 9° was measured. Additionally, the orientation was not always constant along the length. The room temperature tensile strength was typically about 2000 MN m^{-2} (285 × 10^3 lbf in^{-2}) when measured at a strain rate of 0.5 mm min^{-1}. Samples from several batches of the filament were analysed spectrographically in this laboratory and the impurities detected are listed in Table I. The impurity levels are similar to those found previously for flame-fusion grown single-crystal sapphire [2].

Our work followed some preliminary investigations carried out on similar material by workers at the National Gas Turbine Establishment [3, 4]. They used a specimen length of about 20 cm heated in air in a horizontal furnace and determined the time to rupture under stress. Their investigations spanned the temperature range 900 to 1300°C. They did not attempt to measure creep strain, and since when failure

TABLE I Spectrographic analysis of Tyco Sapphire

		Sample no.			
		1	2	3	4
Analysis (ppm by weight)	Fe	3	3	3	5
	Si	3	1.5	10	1.5
	Cr	1	< 1	< 1	< 1
	Cu	2	1	0.5	< 0.2
	Mg	3	3	3	3

The following elements were sought but not detected (with limits): B < 3, Ni < 2, V < 5, Mn < 2, Mo < 3, Zn < 10, Pb < 5, Na < 20, Be < 2, Ca < 1, Ti < 1.