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.

took place it did so rather explosively, it was difficult to recover the broken ends to detect how failure had occurred. In the few instances where recovery was possible, failure appeared to have happened in a brittle manner. The results NGTE obtained showed that, although there was a tendency for failure to occur after shorter times for higher stresses, failure was a rather random event. This fact suggested that failure might be initiated from a flaw or defect, possibly on the surface. Although such flaws might well be present initially in the surface of the filaments, since the specimens were tested horizontally it was always possible that failure could have been initiated by a particle falling from the furnace liner on to the specimen. For these reasons we decided to investigate the matter further at NPL. We used an apparatus in which the samples were loaded in tension and heated in air in a vertical Crusilite tube furnace. Temperatures were measured with a Pt/13% Rh-Pt thermocouple. An attempt was made to detect any creep strain with a linear variable differential transformer: the limit of detection was about 0.05%. The specimen length was about 350 mm of which 50 mm was in the hot zone of the furnace.

Our results are shown in Table II. Again, quite a wide spread can be seen in the time to failure and in no instance was any creep strain recorded. It was rather surprising to us that no creep was detected even in the sample that came from a batch found to be 9° off nominal *c*-axis orientation. We would have expected that the resolved shear stress on the basal planes (calculated to be about 50 MN m^{-2}) should have been large enough to produce basal slip, at least according to the earlier findings of Wachtman and Maxwell [5, 6]. (The results of Wachtman and Maxwell indicate that a resolved stress greater than about 20 MN m^{-2} would produce creep in flame-fusion grown sapphire at temperatures in the range 1100 to 1300° C.)

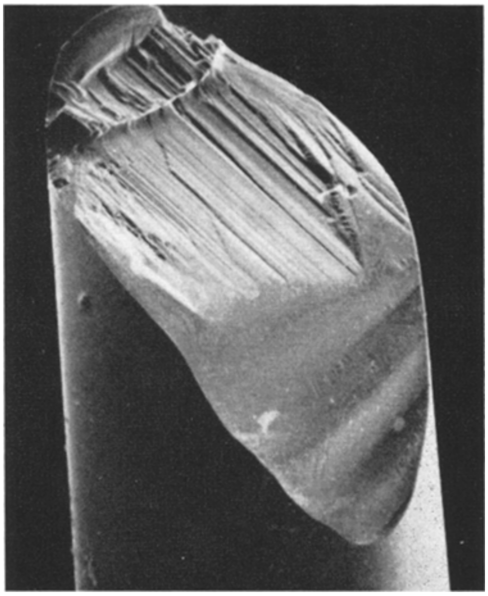

Figure 1 Scanning electron micrograph of sapphire filament after delayed brittle fracture (× 200).

Since our specimens were tested vertically, it is thought unlikely that failure was initiated by particles of material falling on to the surface of the filament. Chemical attack of the surface in air at temperatures in the range 1000 to 1480° C also seems unlikely. However, since the rupture strength measured at these elevated tempera-

TABLE II Stress-rupture results on Tyco sapphire

Specimen	Load (kg)	Stress		Temp. (°C)	Orient. off [0001]	Time to rupture (h)
		(lbf in^{-2} × 10^3)	(MN m^{-2})			
a	1.832	53	363	1000	A	14
b	1.832	53	363	1200	9° ± 1½°	23
c	1.832	53	363	1400	9° ± 1½°	0.3
d	1.832	53	363	1400	1½° ± 1°	25
e	1.832	53	363	1480	1½° ± 1°	1.5
f	0.941	28	190	1480	1½° ± 1°	>216
g	2.760	80	550	1200	½° ± ½°	(0)
h	2.760	80	550	1200	½° ± ½°	(0)

(0) = broke on loading.
A = 1° ± 1° off $[1\bar{1}00]$ orientation.

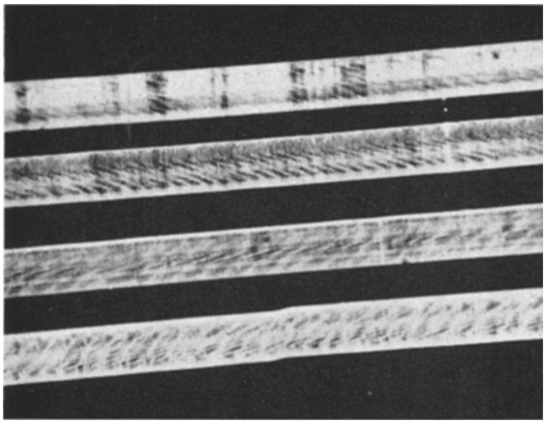

Figure 2 Optical micrograph of sapphire filaments, 9° off *c*-axis orientation (after creep test) (× 27).

tures are very much below those measured at room temperature, a thermally activated mechanism would seem to be responsible.

We have made some examination of the fracture to see if there is any evidence as to how they were initiated. A typical fractured end as seen in the scanning electron microscope is shown in Fig. 1. Failure has obviously occurred in a brittle fashion but there is no internal flaw apparent which could have initiated it. (The appearance of the fracture is somewhat reminiscent to that observed by Stofel and Conrad [7] in 0° sapphire broken in bending at room temperature: they found that fracture followed $[10\bar{1}1]$ planes over most of the surface with the remainder being conchoidal.) With all the specimens we tested, failure was by delayed brittle fracture.

We have also examined filaments along their length and Fig. 2 shows their typical appearance under certain reflected light conditions (the filaments shown were examined after creep test). Markings possibly resulting from the growth process are evident, somewhat similar to those recently reported by Pollock [8], together with irregularly spaced transverse bands. It would seem at least plausible that failure could have started from one such feature although it has not been possible to establish this definitely. When attempts were made to examine such fibres in the scanning electron microscope, no evidence of these features could, however, be seen: the external surface of the fibre also appeared smooth and no defects were detected. One filament in Fig. 2 shows a kink and appears similar to the effect observed by Groves and Gooch [9, 10] after creep at high temperatures in sapphire filaments several degrees off *c*-axis orientation.

In an attempt to analyse the experimental data presented in Table II, it may perhaps be assumed that, to a first approximation, the value of the strain, ϵ, is the same for each specimen at the time, t, when the specimen ruptures. (ϵ was too small to be measured.) It is further assumed that deformation took place either by slip on the basal plane so that $\dot{\epsilon}$ is a function of the resolved shear stress τ of the form

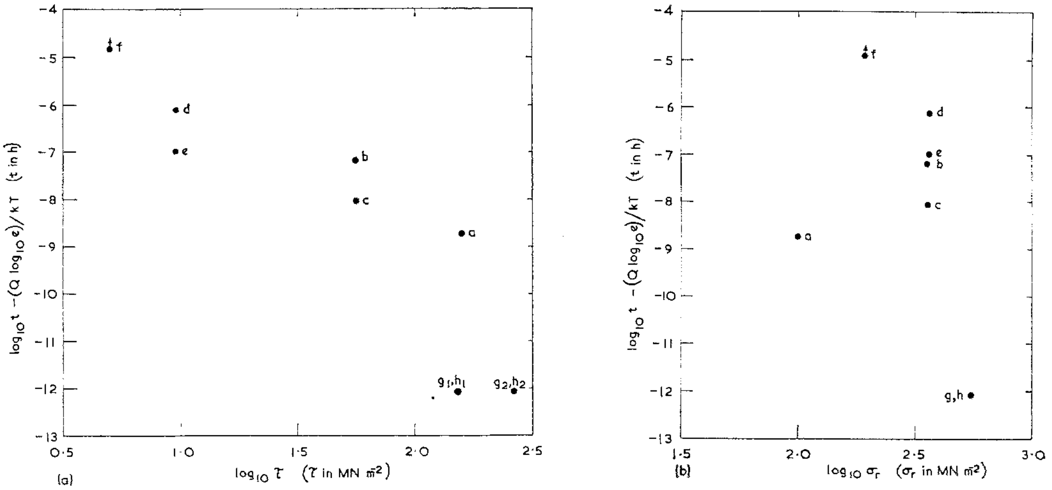

Figure 3 Graph of $\log_{10}t - (Q \log_{10} e)/kT$ against (a) $\log_{10}\tau$, and (b) $\log_{10} \sigma_r$.

$$\dot{\epsilon} = A\, \tau^n \exp(-Q/kT)\,,$$

or by dislocation climb normal to the basal plane so that $\dot{\epsilon}$ is a function of the resolved tensile stress σ_T of the form

$$\dot{\epsilon} = A\, \sigma_T{}^n \exp(-Q/kT)\,.$$

In the above equations, Q is the activation energy for the creep process (assumed independent of the stress), T is the absolute temperature, k is Boltzmann's constant and A and n are other constants. To test these two possibilities for the present data, $\log_{10} t - (Q \log_{10} e)/kT$ has been plotted against $\log_{10} \tau$ in Fig. 3a and against $\log_{10} \sigma_T$ in Fig. 3b. In the two graphs a value of 57.6 kcal mol^{-1} (242 kJ mol^{-1}) has been taken for Q from the results of Oishi and Kingery [11] for self diffusion of oxygen in unannealed specimens of single-crystal Al_2O_3 at temperatures below 1600° C, although the degree of fit of the experimental points to a straight line was found on testing to be rather insensitive to the particular value of Q adopted.

For specimens a to f the stresses were resolved, as indicated above, with respect to the basal plane. As a result, these points are much more nearly colinear in Fig. 3a than in Fig 3b, indicating that slip in the basal plane is more likely to be occurring than dislocation climb. (The scatter in the experimental points is due in part at least to the error in determining the orientation of the specimens.)

For specimens g and h, which broke on loading and in which the tensile axis was very close to the c-axis, slip on the basal plane seems very unlikely due to the very low value of the resolved shear stress (although dislocation climb might be responsible: the points g, and h refer to this possibility in Fig. 3b). Possibly slip occurs in a structural rhombohedral direction for which Groves and Gooch [9, 10] found evidence in sapphire subjected to bending at higher temperatures. If this is so and the time to rupture is assumed to be 0.1 sec, the points g_1, h_1 result in Fig. 3a. Another possibility, as suggested by McLean [12] is that glide occurs in a $c + a$ direction. Such an assumption, with a time to rupture of 0.1 sec, gives the points g_2, h_2. Although the second hypothesis gives a somewhat better fit to a straight line for the complete set of points a to h in Fig. 3a, the mechanism seems a little unlikely due to the large Burgers vector that would be involved. In any case, taking the results as a whole, dependence on τ appears to be stronger than on σ_T, so that glide seems more likely than climb to be responsible for the deformation.

Acknowledgements

The authors thank Dr D. E. Henn, Dr P. T. Clarke and Mrs J. N. Evans for crystallographic orientation determinations, Mr H. R. Whitehead for spectrographic analyses and Mr B. J. Keene for room temperature strength determinations. The authors gratefully acknowledge helpful discussions with Dr D. McLean, and with Dr G. W. Groves of Oxford University.

The work formed part of the research programme of the National Physical Laboratory.

References

1. H. E. LABELLE JUN and A. I. MLAVSKY, *Mat. Res. Bull.* **6** (1971) 571.
2. J. A. CHAMPION, B. J. KEENE, and J. M. SILLWOOD, *J. Mater. Sci.* **4** (1969) 39.
3. A. BURWOOD-SMITH, private communication.
4. J. E. RESTALL and C. R. GOSTELOW, *Proc. Brit. Ceram. Soc.* (in press).
5. J. B. WACHTMAN JUN and L. H. MAXWELL, *J. Amer. Ceram. Soc.* **37** (1954) 291.
6. *Idem, ibid* **40** (1957) 377.
7. E. STOFEL and H. CONRAD, *Trans. Met. Soc. AIME* **227** (1963) 1053.
8. J. T. A. POLLOCK, *J. Mater. Sci.* **7** (1972) 631.
9. G. W. GROVES and D. J. GOOCH, *J. Mater. Sci.* **8** (1973) in press.
10. D. J. GOOCH, D. Phil. Thesis, Oxford University (1972).
11. Y. OISHI and W. D. KINGERY, *J. Chem. Phys.* **33** (1960) 480.
12. D. MCLEAN, private communication.

Received 15 January and
accepted 22 January 1973

J. A. CHAMPION
J. M. SILLWOOD
Division of Inorganic and Metallic Structure,
National Physical Laboratory,
Teddington, Middlesex, UK