Stress rupture of aluminium-coated carbon fibres

The effect of prior elevated temperature anneals on the room temperature ultimate tensile strength (UTS) of individual aluminium-coated carbon fibres was determined in a previous investigation [1]. This research demonstrated that annealing treatments in vacuum above 475° C (for high tensile (HT) fibres) and above 550° C (for high modulus (HM) fibres) produced a reaction between carbon and aluminium to form aluminium carbide (Al₄C₃), with a resultant degradation of the room temperature UTS. In this note the effect of stress application on individual aluminium coated carbon fibres at elevated temperature is discussed.

The apparatus, which has been described previously [2], can be used to test six individual fibres simultaneously in a vacuum of $< 10^{-5}$ Torr. Each fibre was stressed by a selected weight applied to a beam, which transferred the load to a pull-rod linked to the fibre with nimonic alloy adaptors. The creep strain and time to rupture of each fibre were measured by displacement transducers and elapsed time indicators, while temperature was controlled by a Eurotherm controller at temperatures from 400 to 650° C.

Courtaulds HT-S (high tensile-surface treated) and HM-U (high modulus-untreated) fibres were coated by vapour deposition with 180 nm of 99.999% pure aluminium and mounted on the adaptors with Brimor U529 cement. Two other sections from each fibre were tested in an Instron testing machine to obtain a room temperature UTS. The fibres on their adaptors were then transferred to the stress rupture apparatus, loads chosen to give various fractions of the room temperature UTS and the chamber evacuated. The selected loads were applied to the fibres and the furnace heated to the required temperature. Tests were continued to fracture, when the time to rupture was recorded, or stopped after 100 or 500 h. Many fibres survived these tests, but fractured on subsequent cooling to room temperature, when the fracture temperature was also noted.

Five as-received HT-S fibres were tested at stress levels between 55 and 95% of their room temperature UTS at 625°C for 500 h. No creep strain greater than the variation due to thermal fluctuation was detected and the fibres remained intact on cooling to room temperature. Three



Figure 1 The temperature fall required to cause fracture of HT-S carbon fibres coated with 180 nm of Al on cooling from temperatures of 450 to 600° C, after 100 h anneals at various stress levels (represented as a percentage of the room temperature ultimate tensile strength).



Figure 2 Time to fracture of HT-S carbon fibres coated with 180 nm of Al at 600 and 625° C for various applied stress levels (given as a percentage of the room temperature ultimate tensile strength).

similar fibres coated with 180 nm of aluminium and tested at stresses up to 85% of their room temperature UTS at 400° C for 500 h showed the same behaviour. However, at temperatures between 450 and 550° C the aluminium-coated fibres stressed at levels $> \sim 25\%$ of their room temperature UTS generally survived 100 hours at stress, but then fractured during cooling to room temperature. The results are presented in Fig. 1, with the temperature fall to cause fracture plotted against applied stress, as a percentage of the fibres' room temperature UTS. Fibres stressed at less than 25% of the room temperature UTS were not fractured during cooling. At 600° C some fibres fractured during the 100 hour test period, while others failed during the cooling process, as shown in Figs. 1 and 2. At 625° C all but one of the fibres fractured during the 100 hour test, as in Fig. 2.

Some aluminium-coated HM-U fibres were tested for 100 h at 625° C at stress levels between 45 and 90% of their room temperature UTS. One fractured in less than 1 hour, but all the others remained intact, even after subsequent cooling to room temperature.

The resistance to fracture of the coated HT-S fibres tested at 400° C is as expected [1] in the absence of a carbon-aluminium reaction, but contrasts with previous work on bulk composites at this temperature [3].

The coated HT-S fibres tested between 450

and 550°C fractured during cooling after 100 hours, which is attributed to cracks formed in the coating due to the differential contraction of the fibre and the reaction coating containing aluminium carbide. At 450° C, the coating still contained unreacted aluminium [1] and the carbide platelets had not coalesced to form a sheath round the fibre [4]. Hence a large temperature fall was required to open a crack between the ductile aluminium and a brittle carbide platelet, or possibly to cleave a carbide platelet, which under suitable critical conditions of crack length and stress [1], propagated across the carbon fibre. As the temperature increased, the reaction rate increased, the platelets thickened and the percentage of unreacted aluminium decreased. Cracks were then formed more easily and the temperature fall required to produce fracture-initiating cracks in the coating decreased.

At 600° C there is apparently a transition between cooling fracture and stress rupture fracture, which is related to the formation of a complete and continuous sheath of aluminium carbide, when carbon fibre surfaces were perhaps also eroded by preferential diffusion at grain boundaries [4] of the aluminium carbide. It is possible that sufficient erosion occurred either to reduce the cross-sectional area of the fibre or to produce a stress concentration at the erosion site sufficient to fracture the fibre. In contrast to these results on coated HT-S fibres, the aluminium-coated HM-U fibres did not rupture either during tests at 625° C for 100 h or during cooling to room temperature, which is associated with the fibrillar structure of these fibres and their greater resistance to degradation by the aluminium carbide [4].

Acknowledgements

The provision of a research grant and studentship (SJB) by the Science Research Council is gratefully acknowledged.

References

1. S. J. BAKER and W. BONFIELD, J. Mater. Sci. 13 (1978) 1329.

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Electrical anisotropy of asbestos: a fibrous tremolite

The recent reviews [1, 2] and the data collected by Keller [3] show that very little has so far been reported on the electrical properties of asbestos minerals, particularly on their anisotropy which is also dependent on the structural form of the mineral [3]. In view of this, we extended our previous work on the electrical properties of a tremolite asbestos [4] to measure the anisotropy of the electrical properties along the longitudinal and transverse directions. The frequency variations of the dielectric constant, dielectric loss, a.c. conductivity and d.c. conductivity have been measured. The present note reports the results of this investigation.

The same specimen of fibrous tremolite [4] was also selected for the present study. Samples were prepared by carefully packing the fibres parallel to each other to form flat bundles which were cut to suitable sizes with a microtome knife. The samples were then hot-pressed under a pressure of about 3.5×10^8 Nm⁻² to maximum compaction in order to avoid moisture and air gaps. Small pellets of two different forms were thus prepared and the end faces silver painted for measurement along the two directions (Figs. 1a and b).

The dielectric measurements were carried out on a precision capacitance bridge GR 716 in the

- 2. Idem, ibid. 10 (1975) 105.
- 3. P. W. JACKSON, D. M. BRADDICK and P. J. WALKER, Fibre Sci. Technol. 5 (1972) 219.
- 4. G. BLANKENBURGS, J. Aust. Inst. Metals 14 (1969) 236.

Received 7 November and accepted 16 December 1977.

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frequency range 10^2 to 10^5 Hz by applying the resonance curve method [5]. Temperature variations of d.c. conductivities along the longitudinal and transverse directions were measured by adopting a method similar to that of Bhuniya [6].

Results are illustrated in Figs. 2 to 5. Figs. 2 and 3 show that the variation of the dielectric constant (K) follows more or less the same pattern, but the values of K at all frequencies are much higher in the longitudinal than in the transverse direction. Both the curves flatten out to almost the same values beyond 10^4 Hz showing that the anisotropy of the dielectric constant practically disappears in this higher frequency range. The



Figure 1 (a) and (b). Direction of measurement of the samples.