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Structural modifications of a SiC–SiC material exposed to high temperatures in air

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SiC–SiC specimens were exposed to high temperatures in air (from room temperature to 1400° C). A pronounced modification of the interphase between the fibres and the matrix appeared as a consequence of the oxidation of the carbon at the interface.

1. Introduction

This work is the first step of a general approach dealing with microstructural evolution in SiC–SiC material when exposed to high temperatures in a given gaseous environment. This paper reports work on specimens exposed in air. It must be emphasized that this material is not designed to be used at high temperatures in air. In fact, the chemical nature of the Nicalon SiC fibres [1, 2] is such that a strong degradation of the bare fibres, particularly due to oxidation, is observed as early as 1200° C in air [2–4]. Thus, the use of such fibres in structural parts for exposure to temperatures above 1200° C is of no interest, unless they are coated. However, experiments in air and in vacuum are the extreme conditions available for checking the effects of temperature on the interphase located between the fibres and the matrix.

2. Material and experimental methods

The SiC–SiC material comprises bundles of Nicalon SiC fibres woven in two-dimensional layers (40 vol %) embedded in a chemical vapour impregnated (CVI) SiC matrix. The final material had a density of 2.4 to 2.6, with a Young's modulus $E = 230$ GPa and a tensile strength $\sigma_R = 200$ MPa for an elongation $\varepsilon_r = 0.3$. Due to the difference between the thermal expansion coefficients of the fibres and the matrix, the interface is submitted to a compressive stress.

For annealing experiments the furnace was first heated to the desired test temperature, and the specimen was then introduced into it. This procedure may be used because of the good thermal shock resistance of the SiC–SiC material.

Thin slices (400 μm) were cut with a diamond saw and were polished to 100 μm. The samples were then ion-milled (Ar^+, 6 kV) for TEM observations using a Jeol 100 CX microscope.

3. Results and discussion

To improve the mechanical behaviour of SiC–SiC material, the fibres are coated with a thin pyrocarbon layer (50 to 100 nm thick) before processing the CVI SiC matrix. This layer is thought to increase the fibre sliding during deformation, thus the fracture energy is increased. The effectiveness of this mechanism explains the good mechanical behaviour of this material compared to similar composites.

Figs 1 and 2 show examples of the microstructure and the fibre–matrix interphase in two specimens annealed respectively, at 700° C (Fig. 1) and 1300° C (Fig. 2) for 30 min. Fig. 1a is an example of the interphase area between a fibre and the surrounding matrix. The fibre is amorphous while the matrix is crystallized with elongated crystals (of mean size 50 nm) due to the CVI process. Fig. 1b is an enlarged part of (a) in a region located between two fibres, free of matrix. The magnification of the interphase is shown in Fig. 1c where a very good contact is noted between the two fibres and the interphase. Fig. 1d is another example of the interphase region between two fibres which also exhibits good contact of the carbon phase with the fibres.

From 700 to 1300° C no clear change is seen in the contact between the fibres and the interphase (except at 900° C when "clusters" were observed in the interphase region) although a modification of the mechanical parameters (Young's modulus and flexural strength) is observed. Figs. 2a to 2c show micrographs of samples annealed to 1300° C. In Fig. 2a and 2b a decohesion between the fibres and the interphase is clearly seen. The reasons for this decohesion are twofold: (i) the original carbonaceous interphase is oxidized at high temperature in air; (ii) the difference in thermal expansion coefficients between the fibres and the interphase may also explain the fitting of the two regions.

Figs 2c and 2d confirm the above observations in an area defined by the contact zone of two fibres separated only by the carbon layer. The decohesion length is about 0.25 μm. In contrast to Fig. 1c where the layered structure of the pyrocarbon interphase appears, Figs 2c and 2d illustrate the modification of the interphase microstructure where discontinuities such as clusters ($\simeq$ 25 to 50 nm) are seen.

Following these observations, several points remain to be investigated.

(i) The precise temperature range where decohesion between fibres and interphase appears should be

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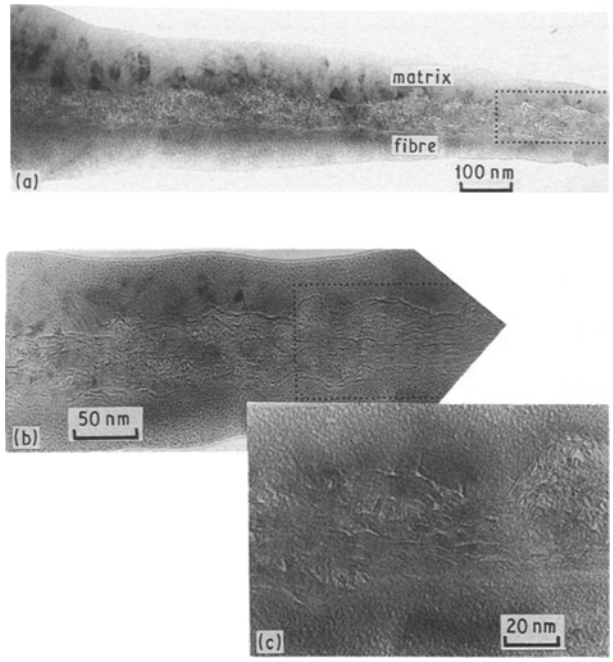

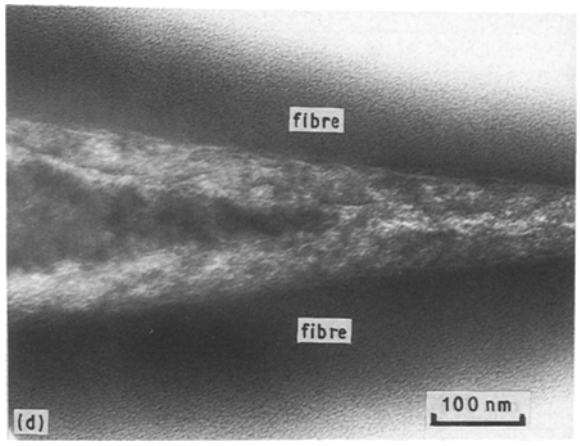

Figure 1 (a) Transmission electron micrograph of the interphase between the CVI SiC matrix (top) and the Nicalon SiC fibre (bottom) in a specimen annealed at 700° C, 30 min in air. Enlarged parts of (a) showing no cracks in the interphase. (d) The contact zone located between two fibres.

found. This temperature differs from that corresponding to the change in mechanical parameters (700 to 800° C). In fact the specimen annealed at 900° C does not exhibit decohesion. Decohesion is essentially due to the oxidation at high temperature. However, the absence of decohesion at 900° C could also be explained by a statistical effect: the measured mechanical parameters could be sensitive to a small amount of decohesion which cannot easily be seen in TEM. Such experiments must be performed below 1300° C.

(ii) The exact nature of the "clusters" inside the interphase region should be determined.

(iii) The effect of the stress during the annealing must be checked to investigate its influence on the decohesion process.

(iv) It will then be interesting to apply the same procedure under different gaseous environments.

4. Conclusions

The present work focused on the effect of the exposure to high temperatures in air on the pyrocarbon interphase in an SiC–SiC material. The observed decohesion localized at the fibre–interphase contact point may impose a limitation in the use of this material in an oxidizing atmosphere; the fibre itself will be damaged due to oxidation. Consequently, parts made of this material must be coated when designed for use at high temperatures in the presence of oxygen.

Acknowledgement

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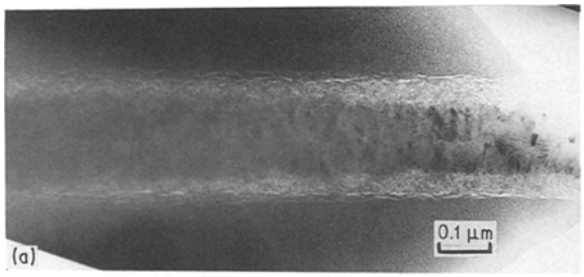

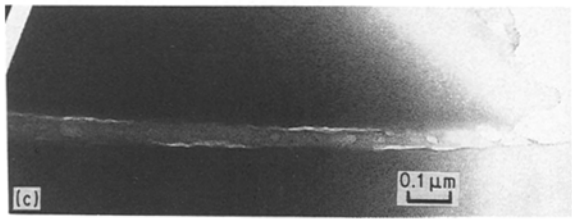

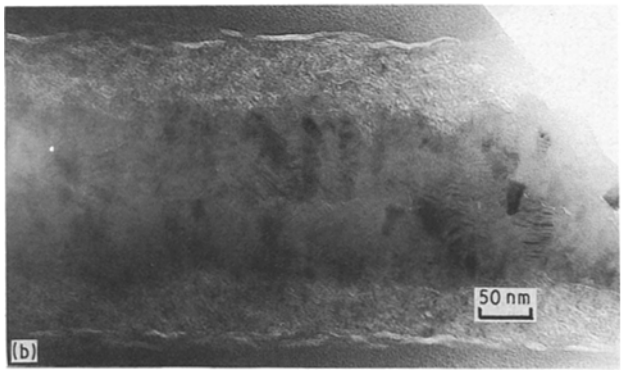

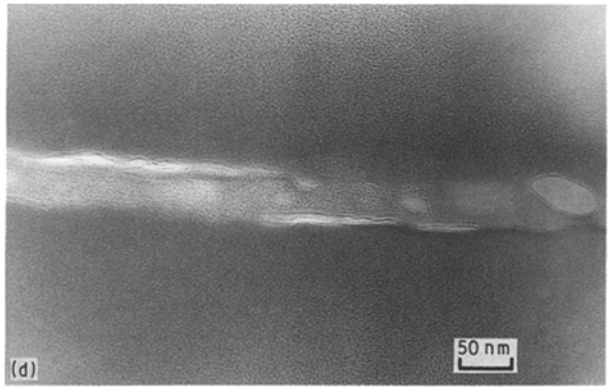

Figure 2 Transmission electron micrographs of the interphase between two adjacent fibres. Debonding cracks are seen in a specimen annealed at 1300° C, 30 min in air at the fibre–interphase junction (2a, and 2b). Along with these cracks ($\simeq 0.25\,\mu$m long) "clusters" are seen in the interphase (2c, and 2d).

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