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INTERFACES IN SiC FIBER-REINFORCED GLASS-CERAMIC COMPOSITES

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Résumé - Les interfaces entre des fibres de SiC Nicalon et une matrice vitrocéramique LAS (lithium aluminum silicate) ont été étudiées par microscopie électronique en transmission associée à une analyse locale par dispersion d'énergie X. La mise en évidence de carbure de niobium à l'interface permet d'expliquer les différences de propriétés mécaniques observées suivant les éléments mineurs ajoutés dans la matrice.

Abstract - Interfaces between SiC Nicalon fibers and a lithium aluminum silicate (LAS) glass-ceramic matrix were investigated by transmission electron microscopy and energy-dispersive X-ray spectroscopy (EDS). Minor additive elements in the matrix, like niobium, can modify the mechanical properties of the composite because of the formation of niobium carbide at the fiber-matrix interface.

1 - INTRODUCTION

Ceramic-ceramic composites have interesting applications due to their high strength and fracture toughness. In particular, materials consisting of a glass-ceramic matrix reinforced by SiC Nicalon fibers have been prepared and investigated by various authors (1-6). The bonding between fibers and matrix at interfaces is a critical parameter in controlling the fracture behavior of the composite. We report here results of an electron microscopy study of interfaces in two types of SiC-LAS composites.

2 - EXPERIMENTAL METHODS

The crystalline phases present in the composites are revealed from X-ray diffraction powder diagrams. The microstructures are studied on a ZEISS DSM950 scanning electron microscope. TEM observations are achieved using a JEOL 2000FX on transverse sections of unidirectional composites thinned by argon ion milling. Each microscope is equipped with a TRACOR energy-dispersive X-ray spectrometer using a SiLi detector with a beryllium window. The analyses are limited to elements with $Z > 10$. Therefore, we cannot detect carbon, oxygen and lithium contained in the composites, but the quantitative analysis of the other elements (aluminum, silicon, magnesium, niobium and zirconium) has been achieved. The spatial resolution in SEM is about 1 μm and a spot of about 20 nm can be selected for analysis in TEM at 200 KV.

3 - RESULTS AND DISCUSSION

Experiments are carried out on two unidirectional composites prepared by a s route and containing a same volume fraction of SiC Nicalon fibers. The starting g (LAS n° 7271 for composite I and LAS n° 6861 for composite II) have the composition in major constituents (aluminum, lithium and silicon), but the nu additive in n° 7271 is only niobium pentoxide whereas n° 6861 contains zirconium and niobium oxide. Previous measurements of mechanical properties shown that these additions significantly influence the fracture behavior of composites (7).

XRD analyses indicate that the main crystalline constituent is a β -spodumene phase in both cases. Careful examination of the spectra of composite I allows to detect add very weak lines corresponding to d spacings of a cubic face centered lattice parameter $a=0.4455$ nm. This value corresponds to that of a NbC_{1-x} phase.

Chemical analyses in SEM indicate a higher content in niobium around SiC fiber in composite I. This is illustrated on Figure 1 by the X-ray mapping of niobium as v those of the major elements silicon and aluminum.

The TEM study confirms the occurrence of niobium carbide at the matrix interface of composite I and not in the other composite. Figure 2 shows fiber-matrix debonding in composite I and the presence of a coronary zone with a thickness 100 nm around a SiC fiber. This intermediate zone is not observed in composite II (Figure 3). Observations of the interface in composite I at higher magnification show crystals which are oriented perpendicularly to the fiber. Diffraction patterns can be indexed with the previously mentioned cfc NbC_{1-x} unit-cell and crystals are elongated along a [001] growth direction (Figure 4). The selective analysis of the larger crystals possible using a small electron probe, it indicates that niobium is the only element detected except traces of silicon which likely comes from the surrounding LAS matrix (Figure 5).

In conclusion, the formation of niobium carbide in composite I results from an interface reaction between carbon coming from the fiber and niobium oxide contained in the LAS matrix. The different behavior of composite II can be explained by the observation of precipitates of a mixed zirconium-niobium oxide compound in the ceramic matrix.

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Secondary electron image

X-ray image of silicon
(K_α line)

X-ray image of aluminum
(K_α line)

X-ray image of niobium
(L_α line)

Fig. 1 : X-ray mapping in SEM of the transverse section of composite I.

Fig. 2 : TEM image of the fiber-matrix interface in composite I.

Fig. 3 : TEM image of the fiber-matrix interface in composite II.

Fig. 4 : TEM image of the fiber-matrix interface (composite I). The electron diffraction pattern corresponds to the largest niobium carbide crystal.

Figure 5 : Dispersive energy X-ray spectrum of the largest crystal on Figure 4. Lines relative to copper come from the specimen-holder grid.