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Structure and Composition of Interlayers in Joints Between SiC Bodies

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Abstract

Joints between SiC bodies (sintered α-SiC and SiC/SiC$_f$ composites) which are intended for thermonuclear fusion reactor applications were produced by coating their surfaces with polycarbosilane (PCS) or a silicone resin (GE SR350) alone, or mixed with SiC or Ti powders, followed by heating in an argon flux at temperatures between 900 and 1200°C applying no pressure or a very moderate one (< 0·1 MPa). The joint quality was determined by microstructural and microchemical analyses (TEM, AEM) and by fracture shear tests for comparative evaluations. For SR350, the joining material was a silicon oxycarbide glass, with no oxygen diffusion occurring between this and the SiC joined bodies. The flat interface structure and the lack of any reaction layer suggest that the joining mechanism involves the direct formation of chemical bonds between the SiC bodies and the joining material. Adding SiC or Ti powders does not improve the joining strength. Contrary to silicone resin, polycarbosilane does not serve as an effective joining material, possibly because of its different decomposition behaviour upon pyrolysis. © 1997 Elsevier Science Limited.

Durch Beschichtung ihrer Oberfläche mit Polycarbosilan (PCS) bzw. Silikonharz (GE SR350) — pur oder mit SiC, oder Ti Pulver, gemischt — wurden Verbunde zwischen SiC-Körpern (gesintertes α-SiC und SiC/SiC$_f$-Composite) hergestellt, die für Verwendung in thermonuklearen Fusionreaktoren bestimmt sind. Die anschließende Keramisierung der Polymere erfolgte im Ar-Strom bei 900–1200°C drucklos oder bei sehr geringen Drucken (< 0·1 MPa). Zur Bestimmung der Verbundqualität wurden Mikrostruktur- und Mikrochemieuntersuchungen sowie Schertests durchgeführt. Im Falle von SR350 besteht die Verbundschicht aus Siliciumoxycarbidglas. Das Fehlen von Reaktionsschichten oder Sauerstoffdiffusionszonen läßt eine direkte chemische Bindung im Grenzbereich Schicht/SiC-Körper vermuten. Die Beimischung von SiC oder Ti-Pulvern wirkt nicht festigkeitssteigernd. Im Gegensatz zu SR350 liefert Polycarbosilan aufgrund seines ungünstigeren Pyrolyseverhaltens keine brauchbaren Verbunde.

1 Introduction

Monolithic SiC ceramics and SiC-based ceramic composites are promising structural materials because of their excellent heat resistance and mechanical properties. Moreover, silicon carbide fibre-reinforced composites are of specific interest for future thermonuclear fusion reactor applications as they exhibit a low radioactivity by neutron transmutations, a high stability after the reactor shut-down, good high-temperature properties, a low plasma contamination and a low specific weight.[1] In order to fabricate large pieces or complicated structures, suitable joining techniques have to be developed. While the joining of monolithic SiC has been accomplished by using different techniques as, for example, active metal brazing, solid state joining and reaction bonding,[2] the joining of SiC/SiC$_f$ composites has not yet been extensively reported in the literature.[3,4] To join these composites in fact implies additional challenges as most of the techniques developed for sintered or reaction-bonded SiC are not suitable here. These techniques often require conditions which are not compatible with the upper temperature of about 1200°C, which currently limits the operation of SiC/SiC$_f$ composites owing to the degradation of the SiC fibres used so far.[5] Furthermore, the major drawback of conventional

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joining techniques with respect to the requirements of fusion technology is that brazing with metallic fillers is not applicable because of the heavy nuclear transmutability of the fillers.[1]

Therefore, we developed an alternative joining method using preceramic polymers like polycarbosilane or polysiloxane, which yield a SiC or SiOC ceramic, respectively, when heated in an inert atmosphere.[6,7] Their use for obtaining ceramic joints was already suggested in the early eighties,[8] but, except for our recent investigations,[9,10] only one case was reported later in the literature.[11,12]

The present paper reports the results of microstructural and microchemical investigations of the interlayers in the newly developed joints between SiC and SiC/SiC$_f$ bodies as revealed by transmission (TEM) and analytical (AEM) electron microscopy in addition to some data which characterize their mechanical properties. The study was performed in order to elucidate the mechanisms determining the joining process.

2 Experimental

2.1 Processing of the joints

Monoliths of sintered α-SiC (diameter 13 mm, thickness 4 mm, containing 0·5–1% of either Al or B as sintering aid and having a porosity of 3%) were provided by ENEA while the two-directionally reinforced SiC/SiC$_f$ composite material (which was cut into 10 × 8 × 3 mm bars for the joining experiments) was from SEP (Societé Européenne de Propulsion, France).

The surface structure of the latter material was rather complex (total roughness R_a ~25 μm, average peak-to-valley height R_z = 130 μm). To improve the surface quality all specimens were mechanically polished on one side, using a 5 μm SiC paper, and ultrasonically cleaned in acetone prior to the joining. After this procedure the total roughness (measured on an area a few millimetres wide) of both materials was reduced to 0·35 and 6 μm, respectively. However, it has to be pointed out that after mechanical polishing, the SiC/SiC$_f$ composite's surface is composed of flat areas (approximately 800 × 800 μm wide) and of deep 'valleys' where the fibres are interwoven.[10] The surface roughness measured only on the flat areas, where joining mainly takes place, was 0·25 μm.

Another feature of the SiC/SiC$_f$ composite material is that it possesses a thick (>100 μm) over-coat of CVD SiC which was not completely removed by the mechanical surface preparation.[10] Thus the fibres were not exposed and did not participate in the joint formation.

The preceramic polymers used for the joining experiments were a methyl-hydroxyl-siloxane (SR350, General Electric Silicone Products Div., Waterford, NY) and polycarbosilane (PCS, Dow Corning X9-6348). The SR350 resin was dissolved in absolute ethanol to yield solutions of concentrations between 2000 and 4500 g/litre (the latter value is close to the solubility limits of the resin in ethanol). PCS was dissolved in monochlorobenzene yielding solutions of concentrations between 700 and 2000 g/litre. In some experiments, commercial titanium (Cerac Inc. T-1241, average grain diameter 15 μm) or β-SiC (H. Starck B10, average grain diameter 0·3–0·5 μm) powders were added to the polymers, in a weight ratio of 0·5 or 0·7 to 1 to act as fillers to lower the polymer shrinkage during the pyrolysis.

The viscous solutions were homogeneously applied to the surface of the specimens to be joined, and the samples were overlapped to establish the joint. Subsequently, the α-SiC and SiC/SiC$_f$ samples were loaded with an axial pressure of 100 to 150 g/cm^2 ($9{\cdot}8 \times 10^{-3}$ to $1{\cdot}47 \times 10^{-2}$ MPa) and 200 to 400 g/cm^2 (2×10^{-2} to 4×10^{-2} MPa), respectively, and heated at 200°C for 2 h in order to achieve the complete crosslinking of the silicone resin or to cure the polycarbosilane.

The samples were then heated in an argon (99·99%) flux for 1 h at temperatures between 900 and 1200°C. The heating and cooling rates were very slow (1°C/min) as to minimize possible residual stresses due to the thermal expansion mismatch.

2.2 TEM preparation

The microstructural and microchemical investigations comprised on the one hand joints between sintered polycrystalline α-SiC bodies and on the other hand joints between SiC/SiC$_f$ composites, processed with SR350 preceramic polymer and applying optimum fabrication conditions (cf. Section 3.1). The joints produced with polycarbosilane did not show adequate strength to withstand the procedure of specimen preparation without seriously affecting the interlayers.

The examinations of the interfaces between composites and joining material were carried out by using a Philips CM 20 FEG (TEM/STEM) electron microscope operated at 200 kV and equipped with a Voyager EDX system, which enables an atomic lattice plane imaging (HREM-mode) and a chemical analysis (element mapping) with a spatial resolution of a few nanometers.

The TEM cross-section specimens were prepared as follows: thin slices (300 μm) were cut by a diamond saw, with the joining interface of interest oriented perpendicular to the final specimen surface, i.e. parallel to the electron beam. After grinding to

100 μm, discs of 3 mm in diameter were cut by using an ultrasonic tool. Finally, the specimens were mounted on a supporting ring, mechanically dimpled down to 10 μm and thinned to electron transparency by Ar-ion milling. These specimens allow an observation of the composites, the joining layers, possible reaction layers and interfaces side by side in one and the same specimen. For details concerning the special problems of TEM investigations of interfaces in ceramics and composite materials including the elucidation of structure/property relations, see, for example, Refs 14–17.

3 Results

First of all, in order to better understand the results that will be presented, a brief discussion on preceramic polymers must be inserted at this point. Preceramic polymers are organoelement polymers, generally containing silicon, which undergo a polymer-to-ceramic conversion when heated in an inert atmosphere at temperatures ranging from 800 to 1400°C. The pyrolysis of cross-linked polymers provides the desired ceramic material accompanied by the formation of gaseous reaction products, high volume shrinkage and a pronounced density increase.[18] This behaviour thus constitutes a serious challenge to obtaining dense and crack-free large dimensioned bodies or even continuous films with a thickness (after pyrolysis) higher than few microns. This is also the reason why, using this novel approach, it is difficult to realize thick joints (i.e. with a thickness of several microns) or to join surfaces possessing a high roughness. A possible solution to this problem is the use of inert (i.e. SiC, SiO_2) or 'active' filler powders (i.e. Ti, Si, which react with the decomposition products evolving from the polymer during pyrolysis yielding a ceramic phase, i.e. TiC, SiC).[18] Both type of fillers were employed in our experiments, with the main aim of testing if it was possible to realize thick joints or to join rough surfaces (as-received SiC/SiC_f composite parts).

3.1 Mechanical characterization

The quality of the joints was determined by fracture shear stress tests following the ASTM D905-89 test procedure, using a crosshead speed of 1 mm/min. Even if a pure shear stress field does not necessarily occur in this testing technique, it is, nonetheless, a suitable means of comparative evaluation.

Figure 1 represents the fracture stresses obtained for the joining of α-SiC monoliths. It is obvious that using only pure silicone resin (SR 350) as joining material results in a sufficient mechanical strength of the structures obtained.

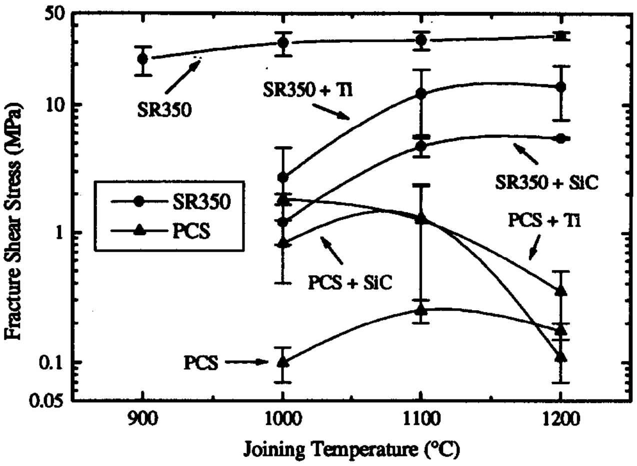

Fig. 1. Fracture shear stresses of α-SiC monoliths joined at different temperatures.

Based on previous investigations,[9,10] the joint thickness was optimized, reaching about 20 μm after the crosslinking (curing step at 200°C). The shear strength increased with the joining temperature, and for the samples heat treated at 1200°C, most of the failures did not occur in the joint region but in the α-SiC monolith. The other samples exhibited a cohesive mechanism of failure, since traces of the pyrolyzed polymer occurred on both surfaces of the jointed specimens.[13]

PCS was not effective as a joining material. At all joining temperatures tested, the specimens exhibited a mechanical strength almost not measurable, independent of the thickness of the joint layers (ranging from about 20 μm up to 200 μm after curing) or of the varying solution concentration.

Because of the considerable shrinkage and density change to which preceramic polymers are subject due to the polymer-to-ceramic conversion,[6–8] it was expected that the introduction of fillers would have helped to reduce this volumetric variation, which could have adverse effects on the joint and its strength. In fact, for pure SR350 resin, the joint thickness is reduced by about a five- to seven-fold factor during the heat treatment at 1200°C (i.e. after pyrolysis the joint thickness ranged between 2 and 5 μm). However, using inert (SiC) or active (Ti) fillers degraded the mechanical strength of the joints. A possible explanation is that with the introduction of these powders the joint thickness (measured after crosslinking) increased from about 20 μm to about 32 μm (for SR350 + SiC powder) and to about 57 μm (for SR350 + Ti powder). Attempts to optimize the deposition procedure to obtain thinner but still continuous and homogeneous joining layers are currently in progress. For PCS-joined specimens, the limited increase in strength with the introduction of powders into the joining material could be due to a more homogeneous layer present there.

Table 1. Fracture shear stress (MPa) for SiC/SiC$_\mathrm{f}$ composites joined at different temperatures

Joining conditions	*1000°C*	*1200°C*
SR350, no applied pressure, polished	1·4 ± 0·8	6·3 ± 1·4
SR350, axial pressure = 200 g/cm^2, polished	9·9 ± 3·4	18·0 ± 3·2
SR35O, axial pressure = 400 g/cm^2, polished	13·5 ± 3·5	21·6 ± 6·5
SR350 + Ti (1/0·7), polished	—	3·5 ± 1·3
SR350 + SiC (1/0·5), polished	—	2·2 ± 0·4
SR350, unpolished	—	<0·2
SR350 + Ti (1/0·7), unpolished	0·7 ± 0·2	3·1 ± 1·2
PCS, no applied pressure, polished	<0·1	<0·2

Table 1 lists the fracture stresses obtained of the SiC/SiC$_\mathrm{f}$ joints. The increase of the joint strength with temperature is again obvious. Moreover, applying pressure during the formation of the joint is certainly advantageous. As for monolithic α-SiC, PCS does not afford the formation of proper joints. Mechanically polishing the SiC/SiC$_\mathrm{f}$ composite was proved necessary, as this probably helped to create flat areas where joining could take place.[10] The introduction of powders improved the joint strength only for unpolished specimens, because of the more effective filling of the surface roughness by the joining material.

3.2 Microstructural characterization

All the images reported are to be considered typical and representative of the interfacial region of each sample.

As the TEM image of Fig. 2 shows, in case of polycrystalline α-SiC joining, a ~2·7 μm thick interlayer forms, having a uniform, amorphous structure with some tangled pores. The interfaces between the SiC part (left and right) and the joining layer (middle) were found to be surprisingly flat within a surface area of some micrometers, with no reaction layer visible. The only features observed are diffraction contours, extending up to ~30 nm from the interface into the bulk SiC material. Increasing the magnification to 3 million shows the $\{111\}$-SiC atomic planes (d = 0·253 nm) to terminate abruptly at the interface to the amorphous joining layer, as the middle part of the HREM image of Fig. 3 demonstrates: again no precipitates or reaction zones are indicated.

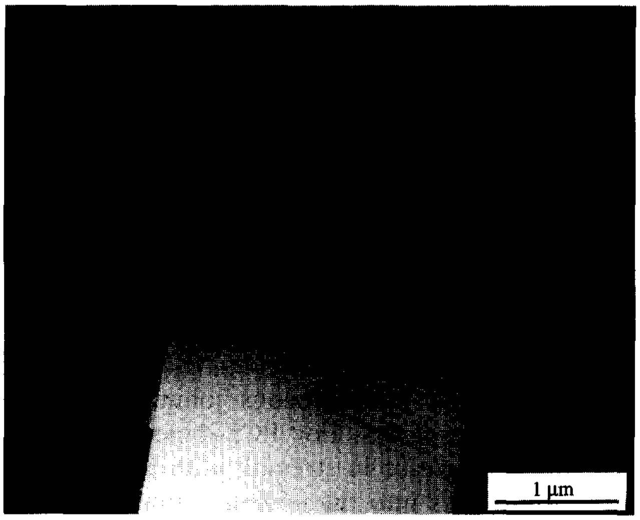

Fig. 2. Joining interlayer in α-SiC, left and right joining parts.

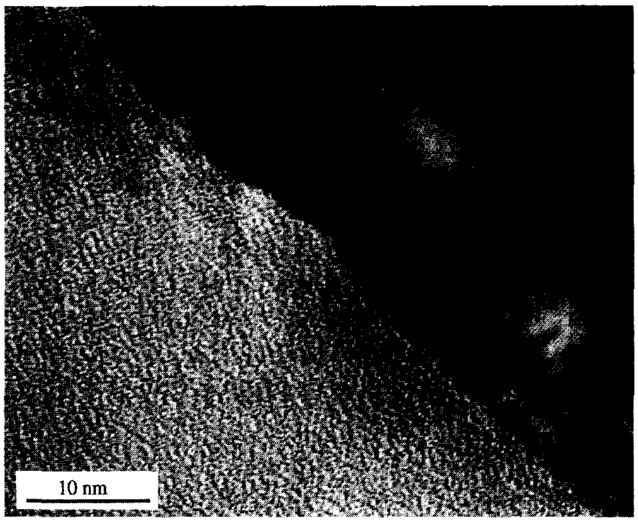

Fig. 3. HREM image of the interface between SiC (upper right) and joining layer in α-SiC joints.

The amorphous joining layer of SiC/SiC$_\mathrm{f}$ composites investigated is free of pores and has a thickness of 5–6 μm. As shown in Fig. 4, the interface between the layer and the SiC matrix of the composite is very flat again, with a 25–40 nm thin zone of a destroyed SiC structure next to the interface. The latter is demonstrated in the high resolution lattice plane image of Fig. 5; while in the interior of SiC (right) the $\{111\}$ atomic planes of SiC regularly propagate over large areas, a lot of crystal defects (stacking faults and/or microtwins, characterized by an interruption and a slight change in the direction of the atomic planes)

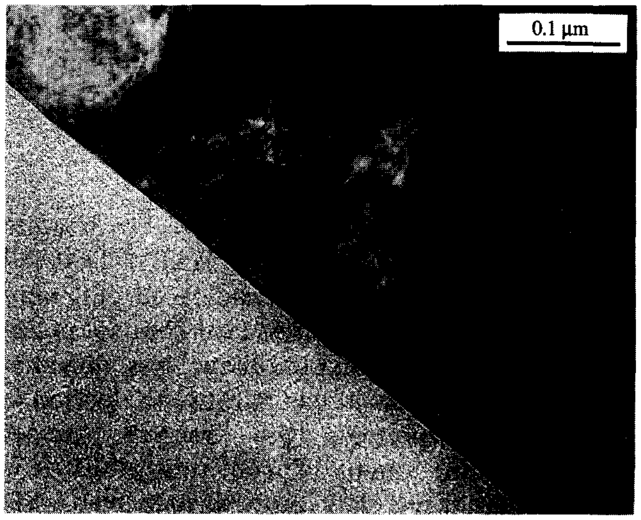

Fig. 4. Interface between SiC (upper right) and joining layer in SiC/SiC$_\mathrm{f}$.

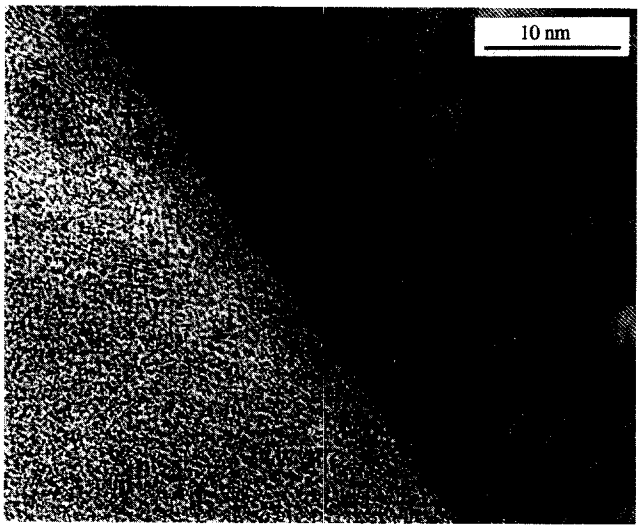

Fig. 5. HREM image of the interface between SiC (right) and joining layer in SiC/SiC$_f$, crystal defects in SiC.

have formed near the interface to the joining layer (left). Contrary to this, the SiC is free of defects at a distance of 25 nm away from the interface. While the joining layer is amorphous in its interior, the high resolution image reveals a precipitation of small amounts of carbon near the interface (cf. the 0·34 nm graphite atomic planes in the almost amorphous joining layer of Fig. 5). In the present stage of investigation it is difficult to infer that the SiC/SiC$_f$ composite surface has any effect on the development of a weak graphitic boundary layer,[13] as it is well known that silicon oxycarbide glasses contain some turbostratic carbon.[7]

3.3 Microchemical characterization

Together with the CM 20 electron microscope the Voyager II EDXS equipment allows the recordings of X-ray spectra, element line scans and element-specific mappings from selected specimen areas with a resolution of some nanometers depending on the specimen thickness. For a proper evaluation of the following X-ray measurements it should be considered, however, that fluorescence phenomena and beam spreading effects in thicker specimen regions or at contamination hillocks will degrade the real spatial resolution of the chemical analysis although the electron probe has an actual diameter of only 1·5 nm. A further uncertainty in recording the elemental profiles may be due to a minor possible overlapping of the materials at the interface.

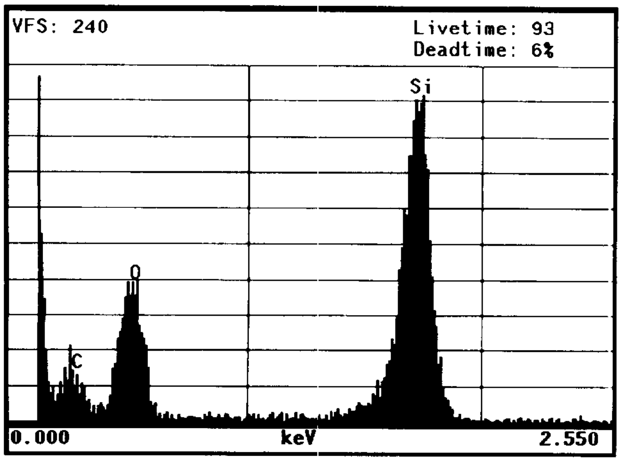

Fig. 6. EDX spectrum of the SiOC glass interlayer.

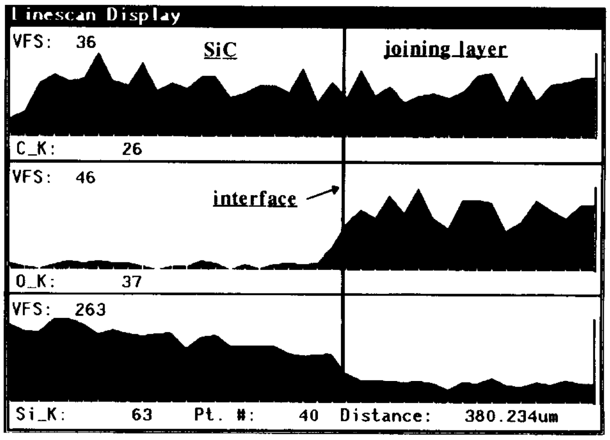

Fig. 7. X-ray linescans of C, O, Si across the interface in α-SiC joints, left: SiC, right: SiOC glass, length: 380 nm.

Regarding this, in all the specimens investigated the chemical composition of the joining interlayer proved to be nearly the same: as an example, the average EDX-spectrum of Fig. 6 (measuring spot 1 μm in diameter) only reveals silicon, oxygen and a small amount of carbon, i.e. a silicon oxycarbide glass has formed.

Figure 7 demonstrates a linescan across the interface of an α-SiC joint presenting a situation similar to that of Fig. 3: a small electron probe was stepped along a definite line of 40 discrete points, with each recording a complete EDX-spectrum. The corresponding X-ray emission of carbon, oxygen and silicon from SiC (left) to the joining layer (right) is shown, with an overall line length of 380 nm (in all the linescans the position of the

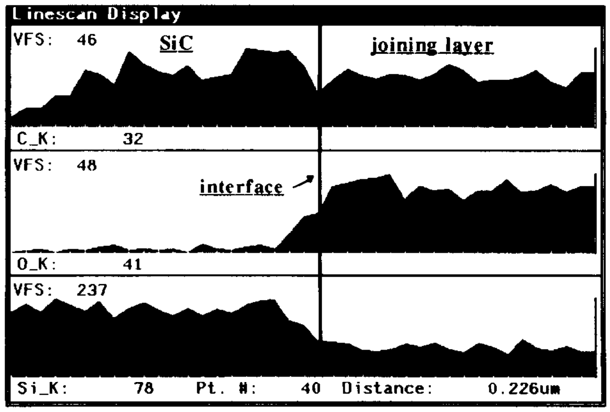

Fig. 8. X-ray linescans of C, O, Si across the interface in SiC/SiC$_f$ joints, left: SiC, right: SiOC glass, length: 226 nm.

interface, as visible by TEM, is marked by a perpendicular line). While most of the carbon signal results from contaminations in the microscope, the silicon X-ray intensity roughly represents the specimen thickness decreasing from SiC toward the interlayer owing to preparation. The oxygen linescan, however, clearly reveals almost no diffusion from the joining layer into the SiC (except within a range of 5 to 10 nm). The small O signal in the SiC region (left) probably results from surface oxidation of SiC during the preparation procedure.

Linescans of carbon, oxygen and silicon X-ray emission across the interface of interest (overall length 226 nm) of a SiC/ SiC_f joint are shown in Fig. 8: again, virtually no oxygen diffusion into the SiC matrix could be observed as is indicated by the oxygen signal abruptly vanishing at the interface to the SiC.

4 Discussion

Besides revealing the mechanisms determining the joining process, we have obtained valuable information from microstructural and microchemical investigations, performed on selected samples, that would enable us to understand the reasons for the differences in the samples observed in performing mechanical tests. In particular, we were interested in understanding why SR350 and not PCS was effective and why joining was successful for α-SiC and not for SiC/SiC_f parts.

The microstructural and microchemical characterizations show that the pyrolytic decomposition of silicone resin produces a rather homogeneous silicon oxycarbide glass layer, the thickness of which seems to depend on the type of substrate joined. The investigations of the interfaces between the SiOC glass and the various SiC bodies indicate that the joining material is absolutely non-reactive, at least at the processing temperatures used, as there are neither reaction layers visible nor a chemical attack of SiC near the joints. Furthermore, the mechanical polishing of the substrates produced very flat interfaces. All these particulars allow one to conclude that here the joining process is similar to adhesive bonding.[2,13] The joining mechanisms do not seem to involve any mechanical interlocking. They rather imply a chemical bonding with the direct formation of bonds between the SiC bodies and the inorganic adhesive material.

The most typical microstructural feature of the interfaces is a 30–100 nm extended zone of enhanced diffraction contrast in SiC near the interface resulting from a destruction of the SiC lattice by mechanical strain and/or crystal defects. According to results of micro-indentation tests at the joint interfaces we can exclude the presence of residual stresses in the materials so that this zone can be attributed to the mechanical grinding/polishing of the SiC parts prior to the joining. The lack of residual stresses is certainly related to the low coefficient of thermal expansion (CTE) mismatch between SiC and SiOC materials.[10]

It has to be pointed out that all studied interfaces appear smooth on the scale observed, but a smoothing of the interfaces during the bonding process seems quite improbable, considering the comparatively low temperature used for joining. Furthermore, as clearly shown by the microchemical analyses, no compositional gradient was observed in the glassy layer and in the SiC material in proximity to the interface. Moreover, no growth or dissolution of the SiC material in the glassy interlayer was observed. The interfacial region itself thus appears to be extremely narrow (see Figs 2 to 5).

The difference of the mechanical properties between joints of α-SiC and joints of SiC/SiC_f bodies obviously is primarily not due to different microstructural or microchemical interfacial features. It might rather be due to the variation in the thickness of the joining layers: the SiC/SiC_f joint is thicker than the α-SiC joint. It should be mentioned, however, that other investigations[10] report on ceramic composite joints with relatively low strength even if the joint thickness was lower than here. Since the SiC/SiC_f composite surface still retains areas which are not completely flat even after mechanical polishing, especially where the fibres in the cloth are interwoven,[10] it is more probable that these regions could be the origin of microdefects from which cracks propagate in the glass joining layer.

Since oxygen does not seem to play an important role in the joining mechanism, the difference in the bonding ability between silicone resin and polycarbosilane preceramic polymers might be explained by their different decomposition behaviour upon pyrolysis. First of all, the weight loss during pyrolysis for pure PCS is considerably higher than for SR350 (PCS ceramic yield ~55%,[6] SR350 ceramic yield ~75%,[7]), possibly resulting in the formation of voids in the joint. It has also been reported in the literature that PCS is less effective for joining SiC bodies[11,12] or as a binder[19] than poly-borodiphenylsiloxane (an oxygen-containing preceramic polymer), despite the fact that both polymers have a similar ceramic yield.[20] Moreover, another aspect which could influence the bonding mechanism is the different thermoplastic behaviour of the preceramic polymers used: while silicone resin is a thermosetting polymer, which completely crosslinks at 200°C, polycarbosilane, when uncured, is a thermoplastic polymer, crosslinking above 500°C. As noted in Ref. 21, the pyrolysis residue is inhomogeneously distributed if the greater part of the polymeric decomposition proceeds prior to crosslinking, as in the case of uncured PCS. If annealing in air at 200°C did not succeed in curing the polycarbosilane, the joining layer would thus probably contain many defects.

5 Conclusions

Joints between SiC bodies were successfully produced using a silicone resin. In the case of joining monolithic α-SiC parts a much higher strength was attained than for joints of SiC/SiC$_f$ composites. Microchemical investigations showed that the joining material consisted of a silicon oxycarbide glass, and that no oxygen diffusion occurred between the glass and the joined SiC bodies. The flat interface structure and the lack of any reaction layer suggest that the joining mechanism involves the direct formation of chemical bonds between the SiC bodies and the joining material. The interfaces between joint and SiC material were similar for α-SiC monoliths and SiC/SiC$_f$ composites. The lower strength of the SiC/SiC$_f$ composite joints is probably due to the uneven surface morphology of the composites. Polycarbosilane was not effective as a joining material, possibly because of its different decomposition behaviour upon pyrolysis. The introduction of SiC or Ti powders did not improve the mechanical strength of the joints.

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