Joining of reaction-bonded silicon carbide using a preceramic polymer

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Ceramic joints between reaction-bonded silicon carbide (RBSiC) were produced using a preceramic polymer (GE SR350 silicone resin) as joining material; samples were heat treated in an argon flux at temperatures ranging from 800–1200 °C without applying any pressure. The strength of the joints was determined by four-point bending, shear and indentation tests. Microstructural and microchemical analyses were performed by optical microscopy, SEM, TEM and AEM. The room-temperature strength of the joints increased with the joining temperature. Maximum values as high as 220 MPa in bending and 39 MPa in shear tests were reached for samples joined at 1200 °C. No detectable residual stresses were observed both in the joining material and the joined parts, and the fracture mechanism was nearly always cohesive. The joint thickness was shown to depend on the processing temperature, and ranged from about 2–7 μm. The joining material was a silicon oxycarbide amorphous ceramic, with no oxygen diffusion occurring between this and the RBSiC joined parts. The lack of compositional gradients, precipitates or reaction layers indicate that the SiOC ceramic acted as an inorganic adhesive, and that the joining mechanism involved the direct formation of chemical bonds between the RBSiC parts and the joining material. © *1998 Chapman & Hall*

1. Introduction

Reaction-bonded silicon carbide RBSiC (also called siliconized silicon carbide SiSiC) is an important engineering ceramic because of its high strength and stability at elevated temperatures, and it is currently fabricated using reasonably cheap manufacturing processes, some of which have been used since the 1960s [1]. In order to use ceramics as structural materials, a suitable joining technique has to be developed, and the realized joints must be able to withstand severe operating conditions, like high temperatures and corrosive environments. For joining RBSiC, brazing and diffusion bonding techniques have been used so far with various degrees of success [2–4]. Generally, diffusion bonding requires elevated temperatures or pressures, while active metal brazing tends to create complex reaction layers which sometimes exhibit poor thermomechanical properties. In this paper we propose the use of a preceramic polymer for the realization of ceramic joints between reaction-bonded SiC parts.

Preceramic polymers are organoelement polymers, generally containing silicon, which undergo a polymer-to-ceramic conversion when heated at temperatures ranging from 800–1400 °C. In order to maintain the shape of the preformed body, the polymer, if not thermosetting itself, must be cross-linked, either by oxidation, electron-beam or radiation curing. The pyrolysis of cross-linked polymers is accompained by the formation of gaseous reaction products, high volume shrinkage and a pronounced density increase [5, 6]. When the pyrolysis temperature is below about 1400 °C, the produced material can be classified as an amorphous covalent ceramic, which usually transforms into a nonostructured crystallized ceramic at higher temperatures [7, 8]. When these novel materials, whose microstructure, composition and general properties are still under investigation, are in the

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amorphous state, they either do not exhibit the presence of a glass transition temperature or, when they do T_g is located at temperatures higher than 1300 °C [9, 10]. Therefore, polymer-derived materials show a brittle–elastic behaviour up to elevated temperatures [11, 12].

Until recently, only very few papers dealing with the use of preceramic polymers in joining have been available in the literature [13–23], despite the advantages that such a method could offer. The most important advantages are that joining is possible at relatively low temperatures (1000–1200 °C), processing of the joints is simple and the joining material is generally microstructurally and compositionally stable up to high temperatures. Moreover, it should be possible to tailor the composition and properties of the joining interlayer by adding suitable inert or reactive filler powders to the preceramic polymer.

2. Experimental procedure
2.1. Processing of the joints
Reaction bonded SiC bars $76 \times 7 \times 6$ mm³ in size (HD530, Norton Company, Worcester, MA) were used for the joining experiments. This material consisted mainly of alpha-silicon carbide crystals with a bimodal grain-size distribution (approximately 10 and 100 µm) embedded in a free silicon phase (23 vol%), with a density of 3.02 g cm⁻³ and an open porosity less than 1%. The specimens were polished on one side using a 5 µm grit SiC paper and ultrasonically cleaned with acetone prior to the joining; the surface roughness, measured using a stylus profilometer, was about 0.1 µm.

The preceramic polymer used for the joining experiments was a methyl-hydroxyl-siloxane (SR350, General Electric Silicone Products Division, Waterford, NY). The SR350 resin was dissolved in absolute ethanol to yield a 4300 g L⁻¹ solution, the viscous solution was homogeneously applied using a spatula to the surface of the specimens to be joined, and the samples were overlapped obtaining a sandwich structure. Subsequently, the specimens were loaded with an axial pressure of 130 g cm⁻² (12.7 kPa) and heated at 200 °C for 2 h in air, in order to achieve the complete cross-linking of the thermosetting silicone resin and to try to form a polymer layer of uniform thickness. The joint thickness, evaluated using a micrometer at this processing stage when the preceramic polymer is not yet transformed into a ceramic material, was about 18 ± 4 µm. The samples were then heated in an argon (99.99%) flux for 1 h at temperatures ranging from 800–1200 °C. The heating and cooling rates were very slow (1 °C min⁻¹) in order to minimize possible residual stresses due to the thermal expansion mismatch. Measured coefficient of thermal expansion (CTE) for RBSiC samples was 4.5×10^{-6} K⁻¹ (20–1200 °C), while CTE of the amorphous silicon oxycarbide ceramic deriving from the pyrolysis of the silicone resin was reported to be 3.14×10^{-6} K⁻¹ [10].

2.2. Mechanical tests
The strength of the joints processed at different temperatures was measured by four-point bending tests.

Butt joint specimens with nominal dimensions $1.5 \times 3 \times 15$ mm³ were obtained from original joined bars. Care was taken in order to maintain the interface in the middle of the specimen. The prospective tensile face (3 mm wide) was polished and the edges were chamfered using diamond paste with decreasing size down to 3 µm. Four-point bending fixture with inner and outer span equal to 6 and 12 mm, respectively, was used. These tests were performed at room temperature using an Instron 1121 UTM. The cross-head speed was 0.2 mm min⁻¹. The room-temperature four-point bend strength of the bulk RBSiC material was determined to be 250 ± 10 MPa, which is considerably lower than literature values, but is probably a result of machining flaws.

Fracture shear stress tests were performed at room temperature according to a modification of the ASTM D905-89 test procedure, using a cross-head speed of 1 mm min⁻¹. Though a pure shear stress field does not necessarily occur in this testing method, it is, however, a suitable means of comparative evaluation. Typical specimen size was $15 \times 7 \times 6$ mm³. The measured shear strength of the bulk RBSiC material was 110 ± 13 MPa. Fracture surface were observed by optical and scanning electron microscopy.

In order to detect the presence of any residual stress field within the joint and in the region surrounding the interface, Vickers indentations were performed using a maximum contact load of 39.4 N. Some indentations were placed at a certain distance from the joint and the length of the four cracks developed was measured. Other indentations were performed right on the joint taking care that one of the indentation diagonals was parallel to the interface. In this case the attention was focused on the path of the crack developed within the joint material.

2.3. TEM preparation
Only the specimens exhibiting the highest strength (i.e. joints performed at 1200 °C) were subjected to detailed microstructural and microchemical investigations.

The examinations of the interfaces between RBSiC parts 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 nanometres.

The transmission electron microscope (TEM) cross-section specimens were prepared as follows: thin silces (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 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 argon-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 further details see references reported elsewhere [18]).
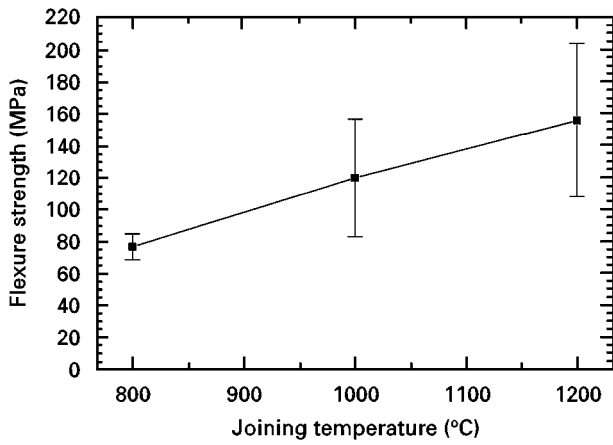
Figure 1 Four-point flexure strength of butt joint specimens as a function of joining temperature.
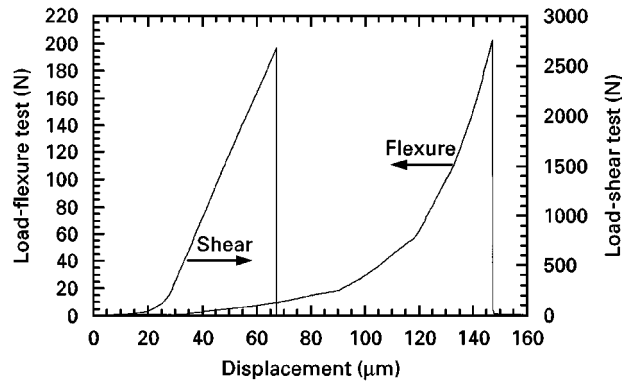

Figure 3 Load–displacement curves for four-point bending test and shear test performed on samples joined at 1200 °C.
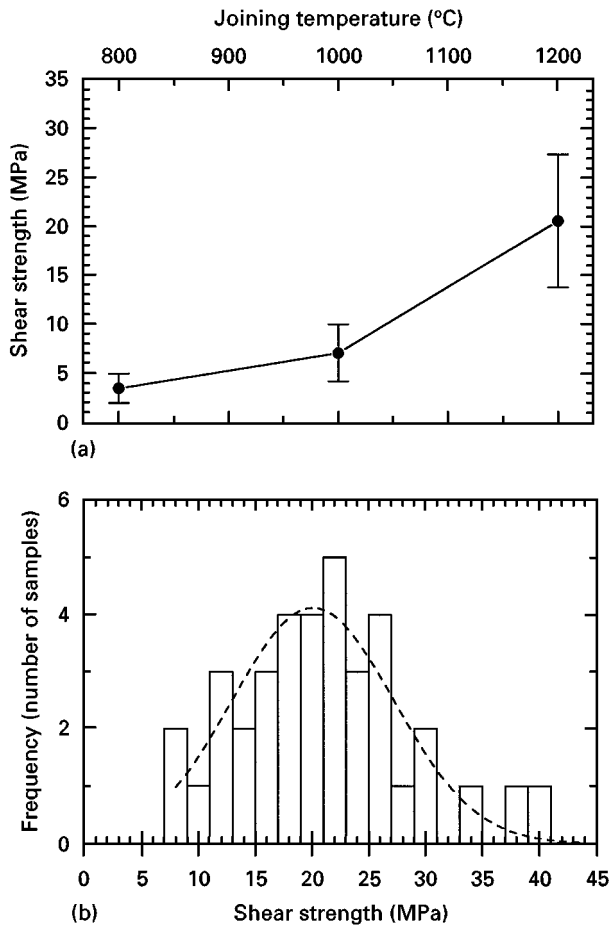

Figure 2 (a) Shear strength of joints as a function of joining temperature; (b) variation of the shear strength for RBSiC samples joined at 1200 °C; (- - - -) Gaussian fit, mean = 20.6 MPa, S.D. = 6.8 MPa.

Figure 4 Scanning electron micrographs of typical fracture surfaces samples joined at (a) 800 °C, (b) 1000 °C and (c) 1200 °C.

3. Results
3.1. Mechanical characterization

Flexural strength as a function of the joining temperature is shown in Fig. 1. The average strength increased from about 70 MPa to 150 MPa with increasing processing temperature. For joints processed at 1200 °C, level strength values as high as 220 MPa were measured. The data represent the average value of five to eight individual tests.
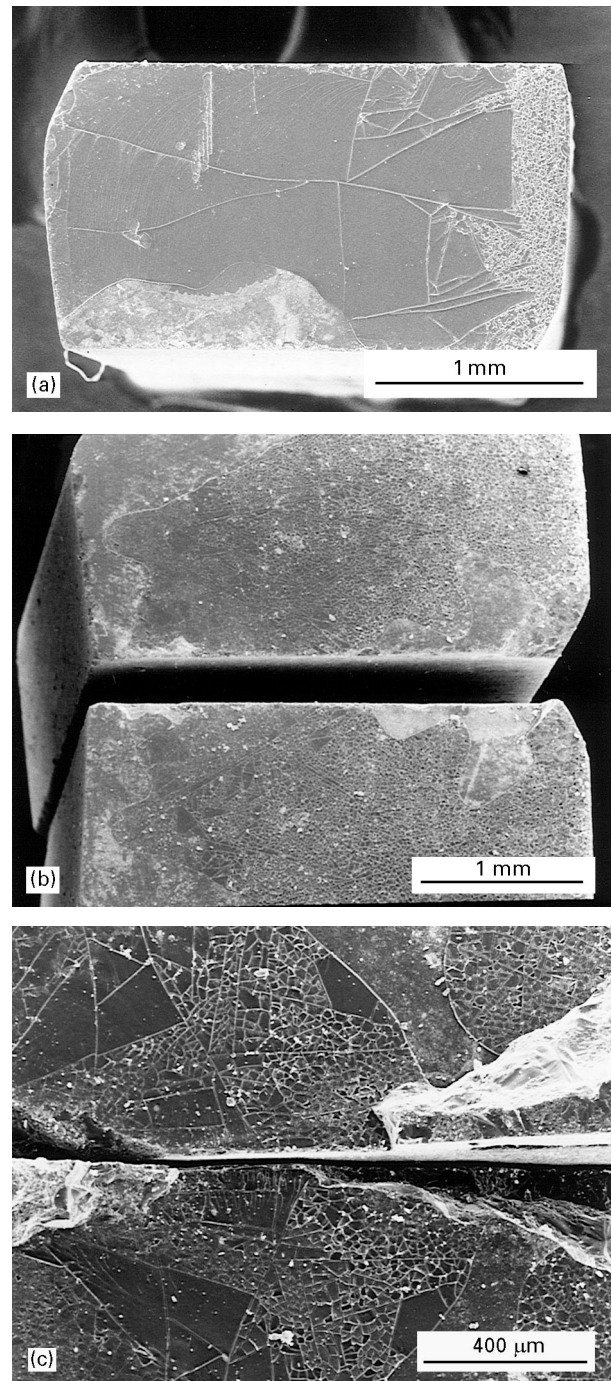
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Fig. 2a shows the shear strength, which averages at least 20 individual tests for samples processed at each temperature. The shear strength strongly increased with increasing annealing temperature, and a maximum value of 39 MPa was measured. In Fig. 2b, a histogram of the variation of the fracture shear stress values for a set of 37 samples joined at 1200 °C, is shown as an example.
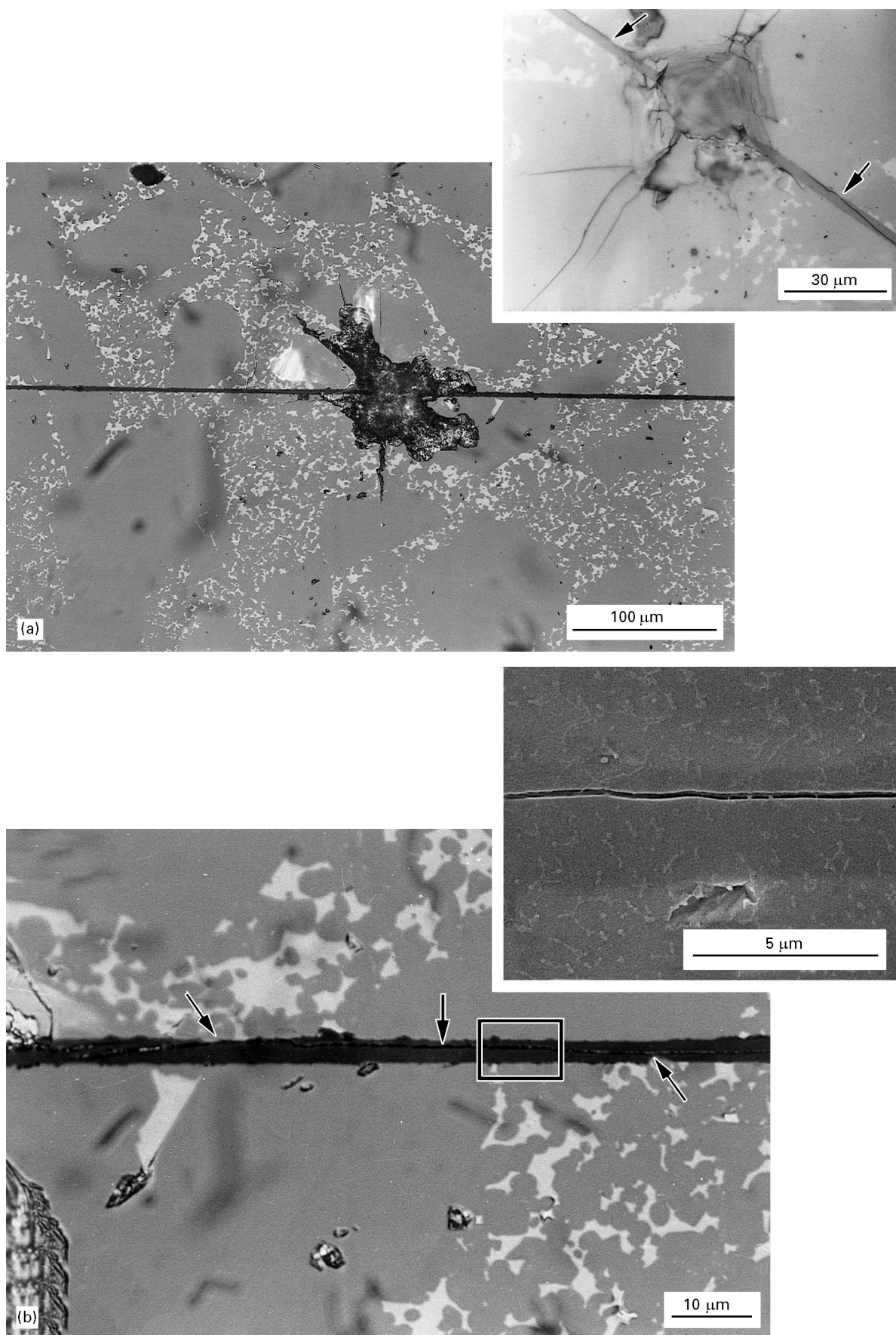

Figure 5 Indentation cracks (see arrows) in the joining layer of a sample joined at 1200 °C. (a) Optical micrograph, ×200 (inset: ×500); (b) optical micrograph, ×1000 (inset: scanning electron micrograph of the region marked by the rectangle).
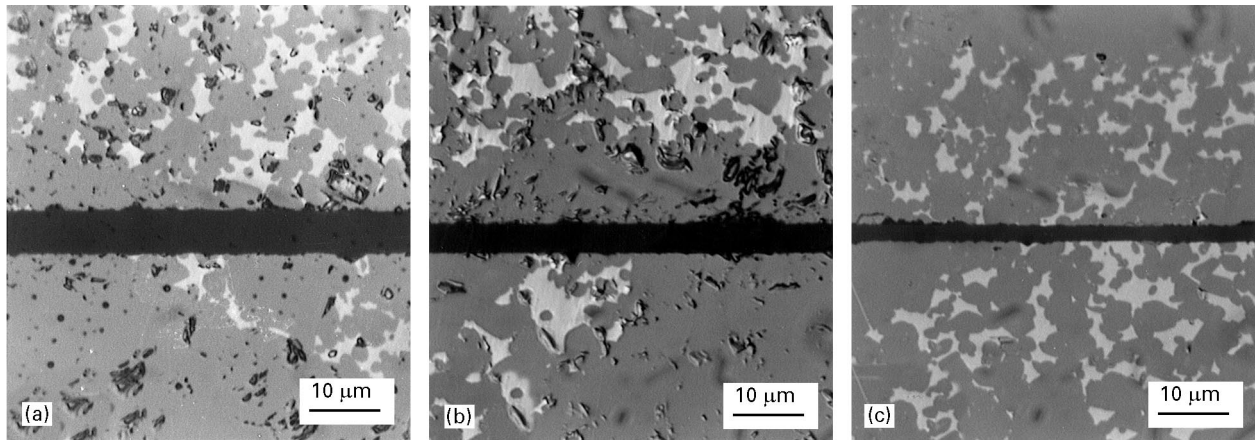
Figure 6 Optical microscope microgrpahs (× 1000) of the joint region for samples joined at different temperatures: (a) 800 °C, (b) 1000 °C, (c) 1200 °C.

The load–displacement curves corresponding to four-point bending test and shear test performed on joints processed at 1200 °C are shown in Fig. 3. The behaviour is linear elastic up to failure. The specimens concerned showed a bending strength of 218.7 MPa and a shear strength of 26.6 MPa.

Fig. 4 shows typical fracture surfaces. Observation of fracture surfaces by optical microscope and scanning electron microscope (SEM) revealed that failure was nearly always cohesive, and displaying mirror-like features.

Fig. 5 shows cracks emanating from indentations obtained on a specimen joined at 1200 °C. Silicon and SiC in RBSiC appear in the optical micrographs light grey and dark grey, respectively.

3.2. Microstructural and microchemical characterization

In Fig. 6 optical and scanning electron micrographs of the joint region for samples processed at different temperatures are shown. The joint material appeared to be fairly continuous, dense, homogeneous and well adherent to the RBSiC parts. Some scattered voids were, however, observed in the joining layer.

An example of the microstructure of the joints between two RBSiC parts joined at 1200 °C is shown in the TEM image of Fig. 7. In this case, ~ 0.5 μm thick interlayer (middle part of the figure) formed, having an almost uniform, amorphous structure. Some microcracks near the RBSiC surface possibly resulted from the polishing process prior to joining. The interfaces between the alpha-SiC crystals in the RBSiC (lower left and upper right) and the joining layer appear to be very flat within a surface area of some micrometres, with no obvious reaction layers, visible. In Fig. 8 a typical HREM image of the interface obtained at a magnification of about 3×10^6 is shown. As can be seen, the SiC atomic planes abruptly terminate at the interface to the joining layer and no precipitates or reaction zones are found.

To determine the microchemical composition of the joining layers between SiSiC parts, X-ray spectra and element-specific line scans from selected specimen areas were performed with a resolution of some
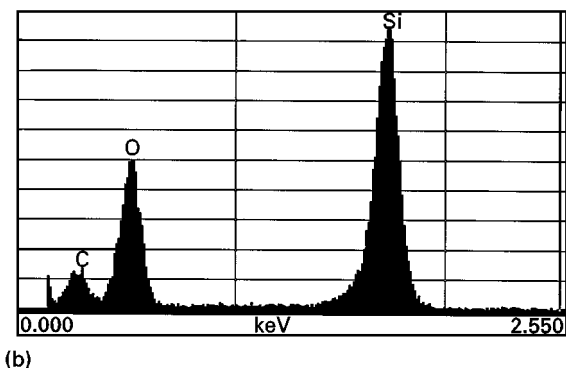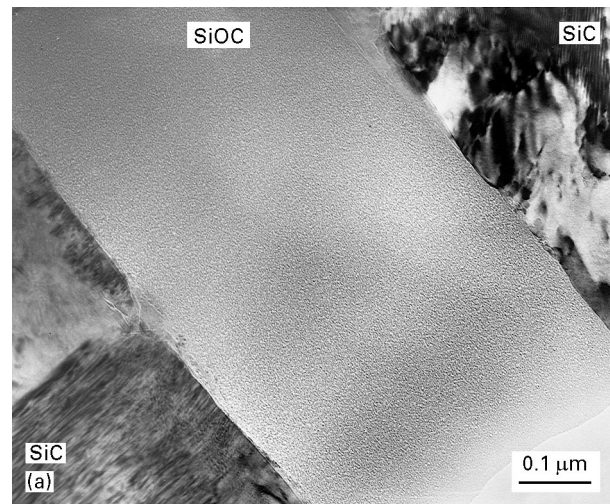

Figure 7 (a) TEM image of the joining interlayer in RBSiC parts joined at 1200 °C (left and right RBSiC joining parts); (b) EDX-spectrum of the SiOC amorphous ceramic interlayer.

nanometers depending on the speciment 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 deteriorate 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 overlapping of the materials at the interface.

The chemical composition of the joining interlayer proved to be very homogeneous: as an example, the
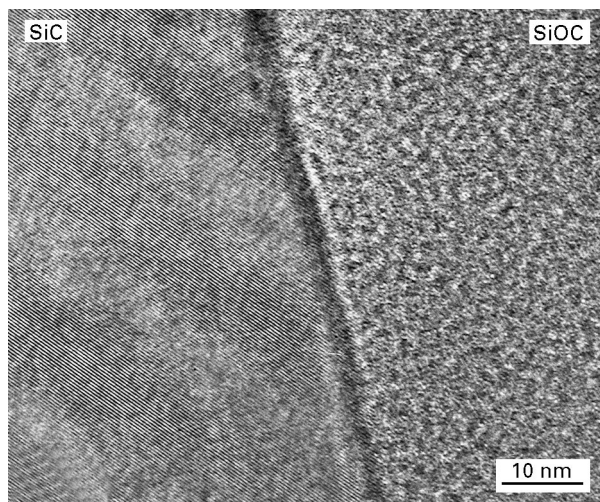
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Figure 8 HREM image of the interface between alpha-SiC (left) and joining layer (right) in RBSiC joined at 1200 °C.
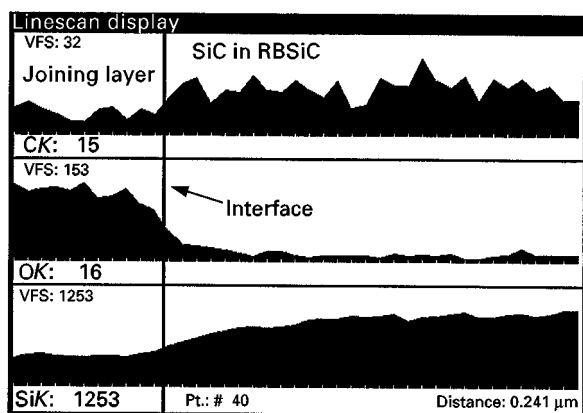

Figure 9 X-ray linescans of carbon, oxygen and silicon across the interface of RBSiC joined at 1200 °C (right, alpha-SiC; left, joining layer; linescan length = 241 nm).

average EDX spectrum of Fig. 7b (measuring spot of 0.1 μm diameter) only reveals silicon (23.6 at%), oxygen (44.4 at %) and carbon (32.0 at%), i.e. a silicon oxycarbide glass (or amorphous ceramic) formed from the decomposition of the silicon resin. In this material, in which part of the silicon atoms in the silicate network share bonds with both oxygen and carbon atoms [9, 10], carbon can be present also as a separate phase (turbostratic carbon). HREM investigations revealed, in fact, the presence of some turbostratic carbon ("free" graphitic C) as well as small β-SiC crystallites in the SiOC layer, as is often found in polymer-derived silicon oxycarbide glass [5, 9, 10].

Fig. 9 shows a linescan across the interface between alpha-SiC crystal in the RBSiC and the joining layer representing a situation similar to that of Figs 7a and 8; a small electron probe was stepped along a definite line of 40 discrete points and a complete EDX-spectrum was recorded from each point. The corresponding X-ray emission of carbon, oxygen and silicon along the line going SiC (right) to the joining layer (left) is shown, with an overall line length of 241 nm

(the position of the interface, as visible by TEM, is marked in the figure by a perpendicular line).

4. Discussion
The joint thickness generally decreased with increasing processing temperature, going from about 6.5 μm at 800 °C, to about 4.5 μm at 1000 °C and to about 2.5 μm at 1200 °C (see Fig. 6). In some places values as low as 0.5 μm were measured (cf. Fig. 7a). Correspondingly, shrinkage of the joining layer during transformation from the polymer to the ceramic state ranged from about 60% up to 85%. This is due to the volume changes occurring during the pyrolysis of the preceramic polymer, which tend to continue up to temperatures around 1200 °C [9, 10]. While the polymer-to-ceramic conversion goes to completion at about 800 °C with the loss of the majority of organic groups (CH_3, etc.), in the 800–1200 °C temperature range further weight loss and denification (density increase as well as reduction of porosity) still occur, with the release of CH_4 and H_2 species [5–12]. Moreover, the nanostructure of the pyrolysis product varies, with an increase in the amount of Si–C bonding with increasing temperature due to a redistribution of the Si–O and Si–C bonds [16, 24, 25]. All these facts might explain why both flexure and shear strength increased with processing temperature (Figs 1 and 2). Another aspect to consider is that the joint thickness influences the level of residual stresses within the joint [26], and a decrease of strength was already observed with joints of increasing thickness when joining sintered SiC using the same preceramic polymer [15]. However, it must be pointed out that an additional factor affecting the strength of the polymer-derived ceramic is the size of the product which strongly influences the amounts of defects in the pyrolysed material [5]. It is difficult, in fact, to obtain thick layers without cracks or macroporosity.

It is evident from Figs 1 and 2 that, though the surface of the specimens subjected to tensile stress was carefully polished and edges chamfered, large scatter in the strength data was measured. This can be correlated to the wide distribution of the initial defects, that was observed on the fracture surfaces. As shown in Fig. 4, the preceramic polymer did not completely cover the joint area, regardless of the processing temperature. Therefore, strength values have to be correlated to large initial defects and the intrinsic strength of the joining material is higher than values shown in Figs 1 and 2. These defects arise mainly from the melting of the preceramic polymer prior to the complete cross-linking at 200 °C [9, 10], resulting in its tendency to flow out of the sandwiched RBSiC parts. This wettability problem, which was not observed to this extent when joining SiC ceramics with a higher surface roughness than the present specimens [18], can be reduced by lowering the cross-linking temperature or by adding chemical cross-linking agents (amines) to the silicone resin solution.

A further factor contributing to the scattering of data is that manual alignment and subsequent 200 °C setting treatment sometimes resulted in joints with

variable thickness across the sample, thus resulting in a non-uniform stress distribution at the joint.

The analysis of the fracture surfaces (see Fig. 4) reveals an evolution of the crack patterns in the joining layer, going from a few long cracks to a minute array of flaws, suggesting an increase in the bonding strength or an evolution in the SiOC amorphous ceramic.

Fracture surfaces were relatively planar, and fracture was, in general, cohesive (note the step features at the fracture surface in Fig. 4c), indicating that the adhesion strength of the glassy phase with reaction-bonded silicon carbide was larger than the strength of the joining material itself. Similar considerations can be made by taking into account the path of cracks developed by Vickers indentation (see Fig. 5). The cracks never propagated along the exact joint/RBSiC interface, and this demonstrates that the interface bonding is relatively strong [27, 28]. The cracks did not follow a preferential or straight path, indicating the lack of residual stresses perpendicular to the joint. The analysis of cracks generated by indentations placed at a certain distance from the interface allowed the absence of residual stresses acting parallel to the joint to be pointed out. Measurement of the crack length revealed that cracks were neither "attracted" nor restrained by the joint. In fact, the lengths of cracks whose tips were closer to the interface were neither longer nor shorter than those of cracks propagating in other directions. This absence of residual stresses is due to the low CTE mismatch between the RBSiC and SiOC materials.

The microstructural and microchemical investigations (Figs 7–9) showed that the pyrolytic decomposition of silicone resin produced a rather homogeneous silicon oxycarbide amorphous layer. The investigations of the interfaces between the SiOC glass and the RBSiC parts indicated that the joining material is absolutely non-reactive, at least at the processing temperatures used, as no compositional gradients, precipitates or reaction layers were observed in the joining material and in the RBSiC parts in the proximity of the interface. In particular, Fig. 8 shows alpha-SiC atomic ($d = 0.253$ nm) parallel to $(0\,0\,1)$ in RBSiC to abruptly terminate at the interface with the amorphous joining layer (the dark line along the interface is a diffraction effect, called a Fresnel line, resulting from changes of the mean inner potential across the interface). In Fig. 9, 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 (in RBSiC) towards the joining layer due to preparation. The oxygen linescan, however, clearly reveals practically no diffusion from the joining layer into SiC (except within a range of ~ 10 nm) as is indicated by the oxygen signal abruptly vanishing at the interface with SiC. The small oxygen signal in the SiC region (right) probably results from surface oxidation of RBSiC during the preparation procedure. The interfacial region itself thus appears to be extremely narrow. Similar investigations performed at the Si (in RBSiC)/SiOC interface showed no differences with the reported data from the SiC (in RBSiC)/SiOC interface.

From all these data we can conclude that the SiOC amorphous ceramic acts as an inorganic adhesive [3], and that the joining mechanism involves the direct formation of chemical bonds between the RBSiC parts and the joining material.

We can expect silicon oxycarbide glass (or amorphous ceramic) joints also to perform well at high temperatures, as the material is oxidation resistant and microstructurally and thermally stable up to 1400 °C [9, 10]. Furthermore, oxycarbide materials have better mechanical properties [11, 12] and lower creep rates [29] than oxide glasses.

It has to be pointed out, moreover, that an inert processing atmosphere is not a strict requirement for obtaining an oxycarbide phase. Even by using an air-containing atmosphere, some carbon will be retained in the structure as the oxygen access of the joining layer in the present case is strongly restricted. Thus this method permits field joining or repairing of parts using, for instance, a propane torch. It should finally be mentioned that the strength of the samples after the thermal setting treatment at 200 °C is high enough to withstand rough handling [15], thus allowing for a very easy processing of the joints.

5. Conclusion
By using a preceramic polymer (GE SR350 silicone resin) it was possible to realize sound ceramic joints between RBSiC parts. Despite some scattering, the measured strength values were comparable with those obtained by other joining methods reported in the literature. In order to produce joints with high flexure and shear strength, processing has to be carried out at 1200 °C, but pressure does not need to be applied. The joining material, consisting of a silicon oxycarbide amorphous ceramic, was proved to be totally unreactive towards the RBSiC parts, thus acting as an inorganic adhesive.

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