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Potential of SiC multilayer ceramics for high temperature applications in oxidising environment

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Abstract

Multilayered ceramics seem very promising for applications at very high temperatures in an oxidising environment. Actually, they present lower cost and better oxidation resistance than many conventional ceramic composites.

The multilayered SiC oxidation and shock resistance has been investigated on tubular specimens processed by tape casting and pressureless sintering. Microstructure, oxidation and mechanical behaviour were investigated by micro-XRD, SEM, TGA–DTA-MS, indentation and radial compressive tests.

The mechanical characterization showed that weak interfacial bonds are present between the layers. Together with the residual stresses left after the preparation phase, they caused crack deflection and improved toughness with respect to traditional ceramics. These mechanisms persisted even after long-term oxidation at 1600 °C or repeated thermal shock tests. The strength was found to depend on the thickness of the single SiC layer, however it was only slightly affected by thermal treatments.

Keywords: A. Tape casting; D. SiC; Multilayers; Thermal shock resistance

1. Introduction

Monolithic ceramics show a catastrophic fracture behaviour under applied stress due to the lack of energy absorbing mechanisms in the failure process. Several tough composites and multilayer ceramics were, however, developed in recent years. The key factor improving the toughness of these materials is the presence of weak interfaces between fibres and the composite matrix or between the ceramic layers. These interfaces allow for energy dissipation before fracture through mechanisms of crack deflection, crack bridging, fibre pull out and interface delamination.

On fibre-based composites, debonding and pull out are frequently achieved by putting a thin interphase layer on the fibre surface. For instance, interphases, generally less than 1 μm thick, made of carbon or boron nitride, were successfully used in SiC/SiC$_f$ composites [1,2].

Multilayered ceramics can be obtained by several methods: tape casting, slip casting, rolling, extrusion, followed by sintering or hot pressing. The most used method is however tape casting [3–9] that consists in casting a thick film of a slurry containing the ceramic powders on a polymeric support. Then the tape is dried and the organic substances are removed by slow heating. Finally, the sintering can be realised without pressure or by hot pressing. In any case, these materials result cheaper than fibre reinforced composites.

Several multilayer ceramics have been investigated in the past [4–22]; the most studied materials have been alumina or alumina–zirconia [4–6,10–11], silicon nitride [12,13], silicon carbide [14–19], even if other composites have been tested [7,20–22].

In the case of multilayers, two methods have been used to enhance toughness over conventional ceramics, namely the introduction of weak interfaces or the presence of residual stresses.

In the first case porous interlayers can be used [7,11,12,14,18,19,23], where the porosity is given by layers not wholly sintered, generally of a different material with

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respect to the main multilayer component [7,11,12,14] or by the addition of pore forming agents in specific layers [18,19]. When a crack approaches a sufficiently weak interface, it deviates, moving along the interface itself; in this way the propagation of cracks from a layer to another is more difficult, and the fracture energy increases. A significant debonding of the interface between layers can be achieved with this method. If the interface is strong, on the contrary, the crack passes over the interface like in a monolithic material, without significant toughening effect.

The other possibility to increase the toughness of multilayers is to exploit the residual stresses either at the surface or at the interface between layers of different composition [10,21,22,24–28]. The residual stress is present due to differential sintering or to the difference in thermal expansion coefficient of the components of the composite, and can be tailored in order to optimize the resistance of the material to the growing of a crack.

Regarding the effect of oxidation on SiC, the phenomenon is well-known in the literature [29–39]. The oxidation reaction can bring to SiO_2 and CO/CO_2 (passive oxidation), with an increase in weight and the passivation of the surface, or to SiO and CO (active oxidation), both gaseous. In the latter case there is no passivating layer forming on the surface of the SiC and there is continual loss of material. The active oxidation however is possible only at low oxygen pressures or very high temperature. Depending on the literature source of data used, the temperature for the transition to active oxidation at 200 mbar of oxygen partial pressure is from 1650 to 1950 °C [29–33]. In the case of presence of water vapour the reactions are different, and an oxidation with continual removal of the oxidised layer is possible [34–38]. In this case it is the SiO_2 formed from the oxidation of SiC that reacts with the water bringing to volatile species.

This paper deals with the processing of a multilayered SiC ceramic fabricated by tape casting and sintering without pressure. The effect of layer thickness and thermal cycling on SiC multilayer oxidation resistance at high temperature was investigated by thermo-analytical techniques as well as by comparing microstructure and mechanical behaviour before and after long-term oxidation treatments.

2. Materials and methods

2.1. Sample processing

Multilayered SiC tubular specimens were fabricated by F.N. S.p.A. Nuove Tecnologie e Servizi Avanzati (Boscomarengo, Italy) [9]. The processing method involved several steps: slurry preparation, tape casting, solvent evaporation, specimen forming, debinding and sintering. The slurry was obtained by dispersing α-SiC powder (Starck UF-10, 15 m^2/g, with a mean particle size of 0.7 μm) in a mixture of ethanol, butanol and tetrachloroethylene; then polyvinyl butyral and polyethylene glycol were added, respectively, as binder and plasticizer. Boron and carbon (about 2 wt.%) were also added, in order to aid the final sintering treatment. Thin sheets were produced by casting the slurry on a moving Mylar support (the tape casting apparatus is presented in Fig. 1).

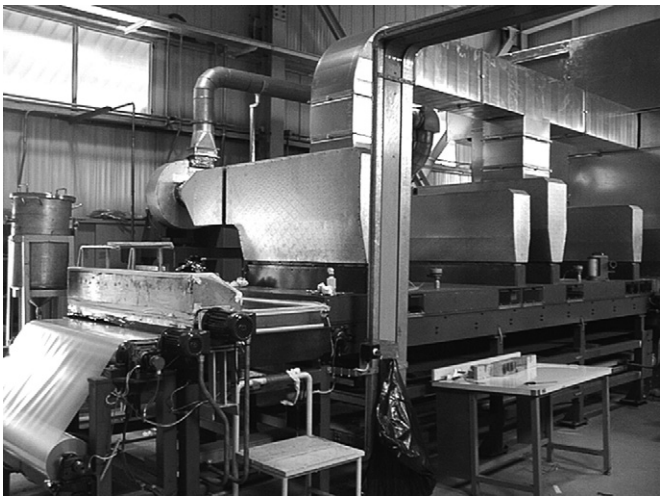

Fig. 1. Tape casting apparatus.

The layer thickness was controlled by the height of the blade (from 0.4 to 0.8 mm) and by the speed of advancement of the Mylar support (100 mm/min), obtaining layers of different thickness. The organic solvents were then slowly removed by controlled evaporation in air at ambient temperature. The SiC green tape was carefully detached from the plastic support and wrapped on a mandrel to obtain tubular specimens (Figs. 2 and 3).

Due to the presence of the Mylar film, the surface roughness of the two tape sides (Fig. 4) are rather different. During tape wrapping, there will always be a rough surface in contact with a smooth one, that could be the cause of tape delamination during fracture. Tubular specimens were submitted to a debinding treatment, carried out by slow heating up to 500 °C under an argon atmosphere. The final pressureless sintering step was performed at 2180 °C under argon.

The bulk green density, after the debinding treatment, was 1.51 g/cm^3, 48% of the theoretical value; after sintering an apparent density of 3.13 g/cm^3 was measured with an hydrostatic balance on a single layer, suggesting an almost complete densification of the ceramic tape; on a multilayer a lower value was measured (2.89 g/cm^3, around 91% of theoretical density), due to the presence of non-accessible porosity between the layers. A severe shrinkage (about 20%)

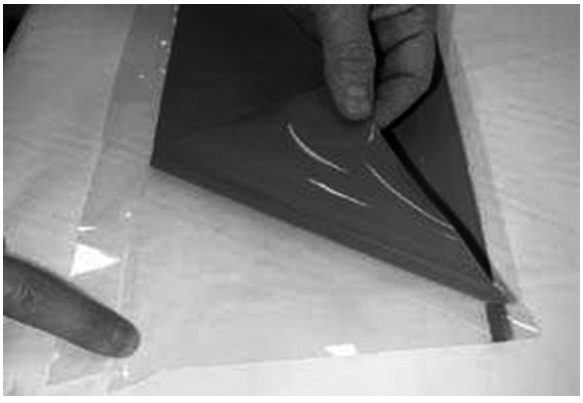

Fig. 2. Detachment of SiC green tape from Mylar support.

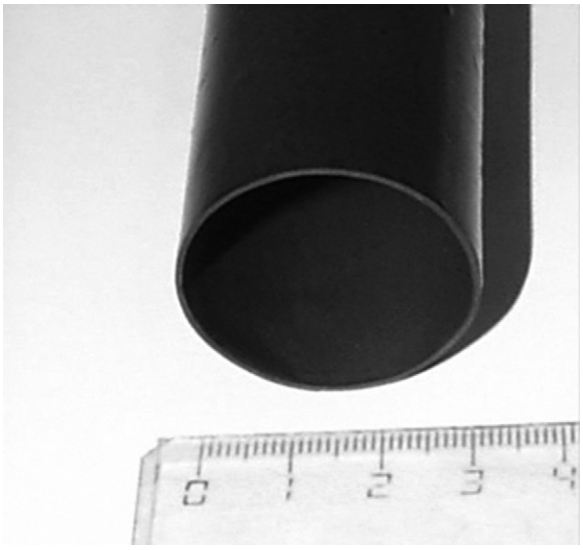

Fig. 3. Tubular SiC specimen after sintering.

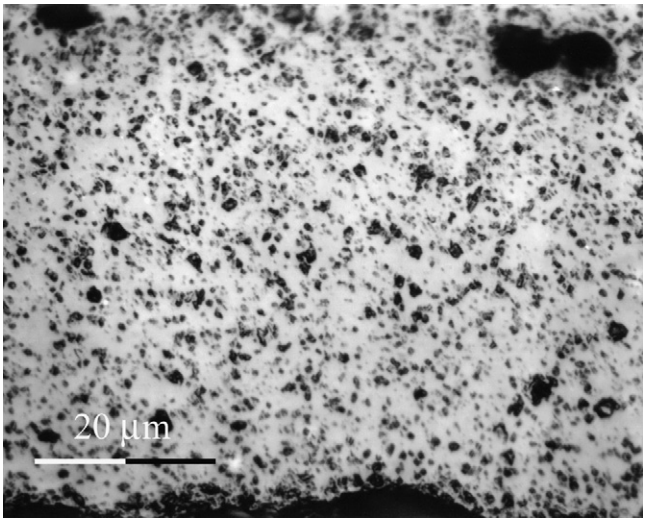

Fig. 5. Microstructure of sintered SiC multilayer; are visible the external surface of the multilayer (on the bottom) and an interface between layers (on the top).

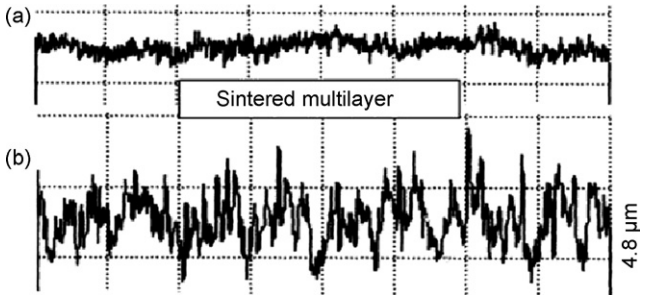

Fig. 4. Roughness profile of fired SiC monolayer: (a) smooth face of green foil (lower-side, in contact with Mylar film) and (b) rough face of green foil (upper-side).

occurred during sintering; ceramic tubes, 100 mm long with an external diameter of 34 mm were obtained. The final thickness of the tube wall, made of the lay up of several SiC layers, was ranging between 0.6 and 1.0 mm (depending on the green tape thickness). Buckles (rings) 10 mm long were machined from the tubes by using diamond tools.

Microstructure, mechanical strength, oxidation and thermal shock resistance of the multilayered material were investigated. A typical microstructure of the sintered silicon carbide ceramic multilayer, observed at the optical microscope, is presented in Fig. 5.

2.2. Oxidation and thermal shock tests

The oxidation conditions were always chosen in order to have a passive oxidation on SiC, with the formation of a SiO_2 scale. For this reason in all cases air at atmospheric pressure was used, in order to have an oxygen partial pressure around 200 mbar.

Firstly, the material oxidation resistance was studied by thermal gravimetric analysis (TGA). A Mettler-Toledo TGA analyser, equipped with a mass spectrometer (Balzers Quadstar 422), was used for the thermal stability tests. Samples of ceramic material (100–200 mg in weight, obtained by cutting sectors of the multilayered SiC rings) were treated in the TGA up to 1500 °C at a constant rate of 20 °C/min in flowing chromatographic air (50 ml/min). The gases generated during the thermal analysis were sent to the mass spectrometer that measured the content in the gaseous flow of various species, in particular carbon and silicon oxides.

Long-term oxidation treatments were also carried out in calm air at high temperatures. Buckles 10 mm long produced by sintered tapes of several thicknesses (the tape thicknesses, referred to the blade height, were 0.4, 0.6 and 0.8 mm) were kept at 1600 °C for 100 h. After the treatment, both the specimen microstructure and the mechanical strength were investigated.

Thermal shock tests were performed by realising the following thermal cycle: the buckles were inserted in an oven kept at 1070 °C, left there 20 min and then extracted and let cool in calm air. After 20 min they were re-inserted in the oven and so on.

2.3. Material characterization

The microstructure of the ceramic samples was studied by microscopy and micro-X-ray diffraction. A Philips 515 scanning electron microscope equipped with an energy dispersive spectrometer (PV9900) and a Rigaku D/MAX Rapid microdiffractometer were used. The XRD patterns of both buckle surface and buckle core were compared before and after the oxidising treatments.

The mechanical strength of the buckles was investigated by radial compression tests, carried out according to the ISO 2739 [40] specification. This test is not specifically designed for ceramic buckles, so that no absolute values for toughness or strength can be extracted from buckles compression tests; nevertheless it can be used to compare the mechanical

properties of the materials under investigations [39], before and after oxidation and thermal shock tests.

At least three samples for each kind of specimens were examined and the corresponding compression test results were averaged. Compression tests were performed by pressing the specimens between two flat plates at ambient temperature, using a Sintech 10D equipment. These experiments, performed at a constant displacement rate of 0.5 mm/min, made it possible to obtain the stress/displacement curve as well as to calculate the radial compression strength [39].

The fracture surfaces were examined by SEM. Indentation tests were carried out in order to investigate crack propagation inside the multilayer ceramic. Vickers indentations were performed on buckle sections taken both in the parallel and transversal direction with respect to the buckle axis. The crack propagation and the residual stress effect on the crack length were studied by both SEM and optical microscopy. Residual stresses were measured by microdiffraction using the DRAST (Debye Ring Analysis for STress measurement) method [41] on the section of the SiC multilayers.

3. Results and discussion

3.1. Characterization of as-processed composite materials

TGA analyses of as-prepared and heat treated multilayer are presented in Fig. 6. The curves show that the ceramic material undergoes a significant progressive loss of weight during heating in air in the temperature range between 700 and 1100 °C (curve a). This weight loss can not be ascribed to silicon carbide oxidation, since the passive oxidation mechanism involves a weight gain, and the conditions of oxidation are very far from those typical of active oxidation. The fact that after 4 h at 1600 °C no weight loss is observed (curve b) suggests that the phenomenon is due to the oxidation of carbon residues contained in the material (carbon was added to the slurry in order to help sintering and an additional amount of carbon also forms owing to the pyrolysis of plasticizers and binders).

This interpretation is confirmed by mass spectrometry, since only mass-to-charge ratios linked to carbon are present (C, CO, CO_2). Moreover, weight loss and CO_2 emission are maximum next to 900 °C, which is a temperature corresponding to rapid air combustion of carbon.

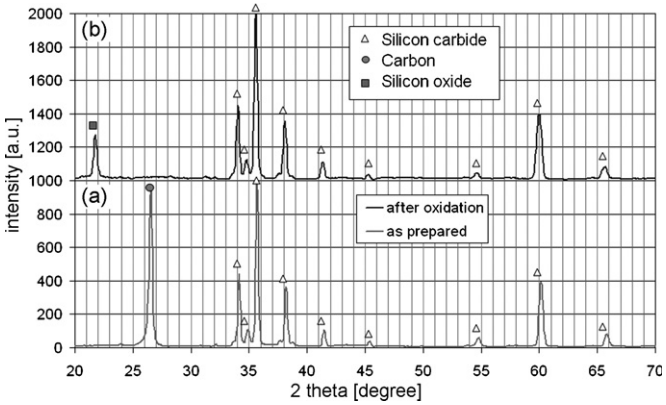

Fig. 7. XRD patterns of the buckle surface, as-prepared (a) and after heat treatment in air at 1600 °C for 4 h (b).

The thermal characterization has been followed by XRD analysis (Fig. 7). Pattern of the as-processed material shows reflexes belonging to carbon and SiC, while after thermal treatment in an oxidative environment only SiC and silica presence is observed. However, it is chiefly the buckle surface which undergoes this reaction, since in the core of the multilayer no oxidation is present; this is confirmed by SEM observations on oxidised samples (Fig. 8), where is seen the passivating layer due to oxidation of silicon carbide.

The mechanical behaviour of multilayers was analysed on materials with different layer thickness. The stress–displacement curves have the particular trend shown in Fig. 9. The stress/displacement curve rises up to a maximum, occurring at the failure of any SiC layer, then the stress abruptly falls, but without resulting in the specimen breaking. The multilayer ceramic can still sustain stresses after the onset of fracture. The SiC layers not yet damaged support a further stress increase, as confirmed by the progressive decreasing of the Young's modulus of the structure. The delamination phenomena allow for significant sample deformation before the final breaking. The delamination mechanism, which provides a toughening

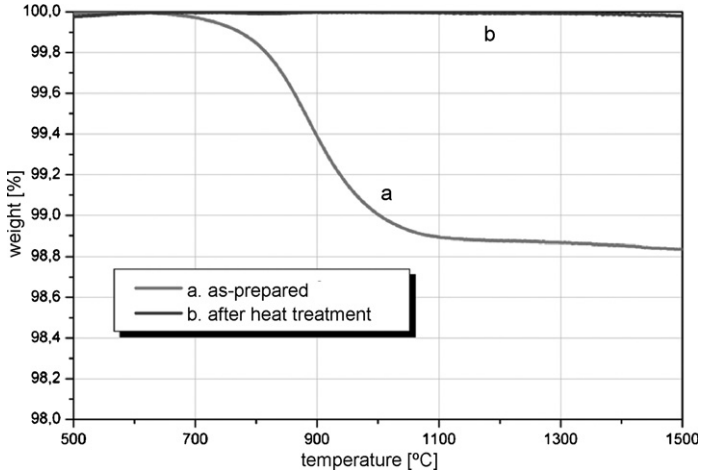

Fig. 6. TGA analysis of SiC multilayer, as-prepared (a) and after heat treatment in air at 1600 °C for 4 h (b).

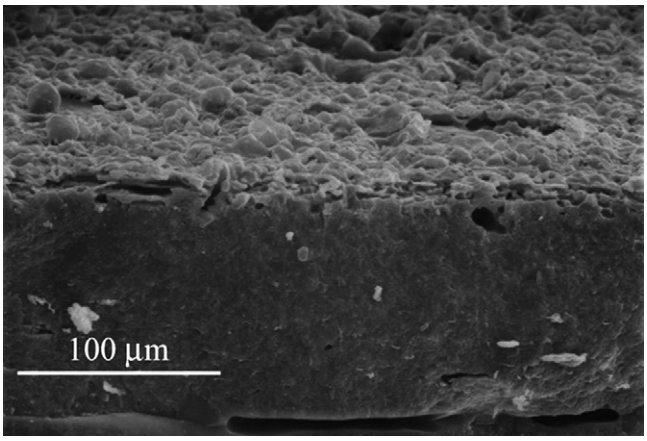

Fig. 8. Silica layer on the surface of the oxidised samples.

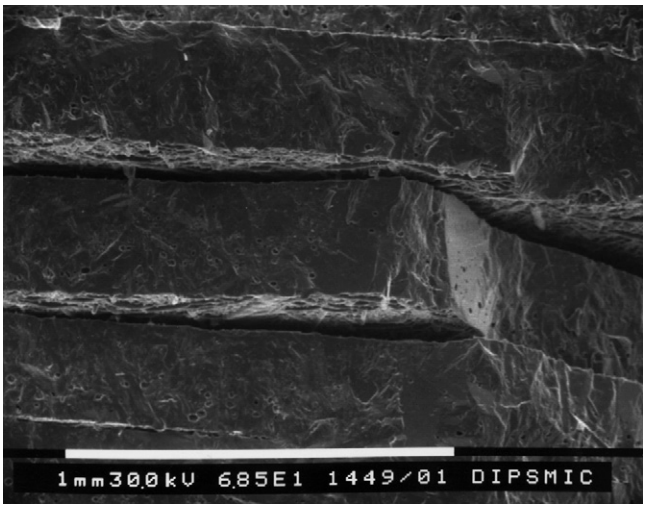

Fig. 10. Fracture surface of as-prepared SiC buckle.

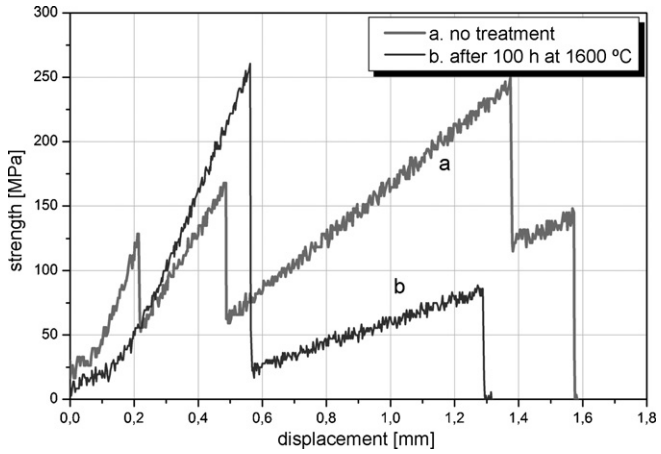

Fig. 9. Stress/displacement curves of SiC multilayer buckles as-prepared (a) and after heat treatment in air at 1600 °C for 100 h (b).

effect, is most evident when the fracture surfaces are examined (Fig. 10).

It is evident from Fig. 9 that the oxidation treatment (100 h at 1600 °C) does not appreciably change the failure mode of multilayers.

The layer thickness on the other side has a rather marked influence on the mechanical properties of such materials. Table 1 shows the radial compression strength of samples obtained from layers of different thickness (tape thickness, as measured from the blade height during tape casting, 0.4, 0.6 and 0.8 mm).

These results shows that thinner layers give better mechanical properties to the multilayers, probably due to a higher number of interfaces and a better residual strength distribution. The best materials are the one with 0.6 mm layers, since 0.4 mm layers are so thin that are more prone to suffer damage during the preparation phase. On the contrary the oxidation has a more marked effect on mechanical properties of thinner layers, even if the overall mechanical strength after oxidation remains significantly greater for thinner layers than for thicker ones.

Crack deflection is another interesting issue that helps to explain the mechanical behaviour of multilayers. Vickers indentation tests were performed on the polished section of the multilayers, and a typical result is presented in Fig. 11: the radial cracks formed during the indentation are very short, while tangential ones are free to move along the layer; the crack deflection is also visible for radial cracks. This suggests the presence of residual stresses. A confirmation of their presence was given by performing residual stress measurements by microdiffraction XRD with DRAST method. Compressive stresses from 600 to 1200 MPa were measured on the multilayer section.

These results corroborate the expected fracture mechanism: the cracks cannot easily propagate from one layer to another, so that each layer fails singularly and, rather than sudden fracture, a structured curve is observed.

Thermal shock tests were carried out on samples with 0.6 mm thick layers. The system described in Section 2.2 was used, and the results are shown in Table 2.

No significant differences were observed after 10 or 50 thermal shock cycles, thus suggesting that thermal shock from

Table 1
Buckles compression tests results: compression strength for different layer thickness samples

	0.4 mm tape thickness		0.6 mm tape thickness		0.8 mm tape thickness	
	As-prepared	Heat treated	As-prepared	Heat treated	As-prepared	Heat treated
Compression strength (MPa)	235	184	356	210	143	123

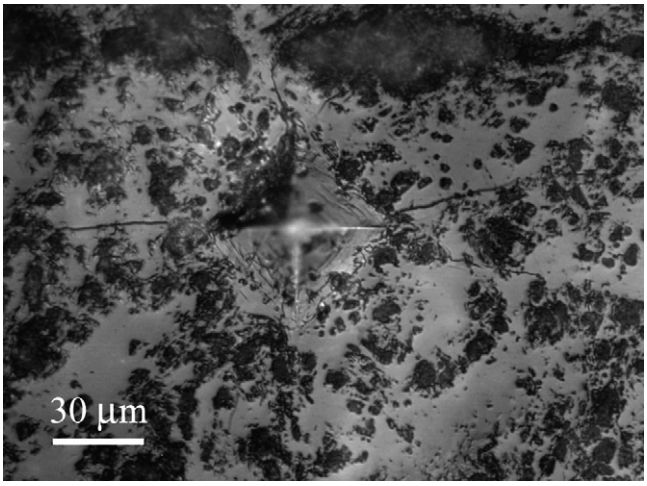

Fig. 11. Indentation cracks on an as-prepared sample, showing cracks moving preferentially in tangential direction and radially deflected.

Table 2
Buckles compression tests after thermal shock cycles

	No thermal cycling	10 cycles	50 cycles
Compression strength (MPa)	287	311	269

1070 °C does not have a profound effect on the mechanical properties of such composites.

Indentation tests were carried out on cycled samples in order to verify if the crack deflection mechanism was yet active. A typical result is presented in Fig. 12, where it is possible to see that the cracks travel only in the tangential direction while are stopped in the radial one. This confirms that residual stress is still present even after 50 thermal shock cycles, and guide the crack path.

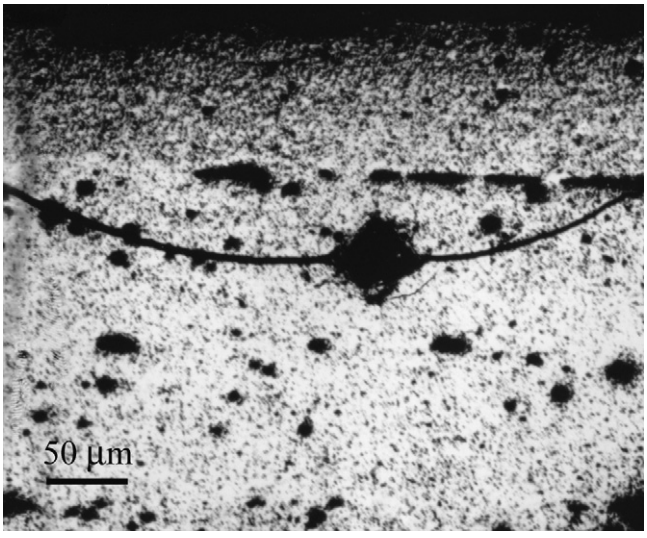

Fig. 12. Indentation cracks on a thermally cycled sample, showing long tangential cracks deflected toward the external surface. Radial cracks are absent or very short.

4. Conclusions

Multilayered ceramics can be a suitable and low expensive way to obtain components apt for working at high temperature. Tubular components of silicon carbide with a multilayer structure were produced by tape casting and sintering without pressure. Compression tests indicated the presence of delamination phenomena that increase the multilayer toughness over that of a conventional ceramic. The layer thickness influences both the material strength and the oxidation resistance. Buckles containing thinner layers show an increased strength, even if their strength slightly decreases after oxidation at 1600 °C. Indentation and microdiffraction tests showed that residual stresses control the crack path. During long-term oxidation a continuous silica coating, which acts as a barrier for a further oxygen penetration, forms. In spite of the oxidative reactions, fracture behaviour of the multilayer ceramic was found unchanged, even after oxidation treatments carried out in very severe conditions (100 h at 1600 °C). Thermal shock tests showed that no loss in mechanical strength was observed and that the mechanism of crack deflection is still working even after 50 cycles from 1070 °C.

Acknowledgements

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