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Degradation at 1200°C of a SiC coated 2D-Nicalon/C/SiC composite processed by SICFILL® method

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Abstract

The thermal stability of a 2D-Nicalon/C/SiC composite was studied through the variation of both mechanical properties and microstructure occurring during heat treating. The composite was processed by infiltration of SiC preforms according to SICFILL® method. The material toughness was enhanced by a carbon interphase put between the fibers and the matrix. In order to improve the thermal stability a CVI layer was deposited on the carbon interphase and the specimen surfaces were CVD covered by an external SiC seal coating about 165 μm thick. The aging tests were carried out at 1200°C in air or in non oxidizing environment (vacuum). Other specimens were thermally cycled between 25 and 1150°C. Three point bending tests and Charpy impact measurements were performed before and after these treatments. The composite microstructure was investigated by scanning electron microscope (SEM), electron probe microanalysis (EPMA), X-ray diffraction (XRD), reflectance infrared spectroscopy (FTIR) and surface area BET measurements. The as-processed material showed a modulus of rupture (MOR) of 483 MPa and appreciable toughness. These characteristics were retained after aging (200 h at 1200°C) under vacuum. Air thermal treatments caused heavy loss of strength and increase of brittleness. Strong oxidation occurred during these last treatments at both the carbon interlayer and the matrix, while the SiC external sample coating was not oxidized. The oxygen needed for composite bulk oxidation flowed through the SiC coating due to the occasional presence of very few structural defects. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: Composites; Interfaces; SiC; Thermal shock resistance

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1. Introduction

Long fiber-reinforced ceramic matrix composites have been proposed as advanced materials suitable for structural applications. In particular, in the last years many efforts have been devoted to the development of SiC_f/SiC composites. These composites show some attractive properties and advantages over traditional ceramics: higher tensile and flexural strength (provided by the continuous fiber reinforcement), enhanced fracture toughness and impact resistance (chiefly achieved by tailoring the fiber/matrix interface characteristics). Furthermore, their specific strength and modulus are greater than those of many other structural materials (metal alloys or ceramic) because both fiber and ceramic matrix are made of SiC (theoretical density for SiC about 3.2 g/cm^3). Finally, the mechanical properties of SiC/SiC composite can be retained at high temperatures and under severe service environments. Generally speaking, most of ceramics and ceramic/ceramic composites show better oxidation resistance than metal alloys. However, many industrial applications (i.e. in the aerospace field) require the development of new oxidation resistant materials able to work in extreme conditions (temperature above 1000°C and oxidizing atmosphere).

In these conditions also the SiC/SiC composites can suffer degradation. In fact, at temperatures higher than 1000°C both the polymer-derived (Nicalon) and the chemically vapor-deposited (CVD) SiC fibers undergo damaging through decomposition and consequent evaporation of gaseous species. The vaporizing compounds responsible for degradation chiefly are CO, in the case of CVD fibers, and SiO plus CO for Nicalon fibers.[1,2] The oxygen needed for the formation of SiO and CO

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may come from both the fiber itself (which contains a non negligible percentage of this element) and an oxidizing environment. The heat treatment of Nicalon fibers performed over 1200°C in air also causes a further SiC fiber oxidation, which results in the formation of a SiO_2 surface layer. On the other hand, according to Mah et al.,[1] this silica layer prevents the reaction product evaporation and slows down the loss of fiber strength, which, for this reason, occurs more quickly under vacuum than in air. Furthermore, a SiC grain growth happens at the fiber core during the thermal treatment and concurs to decrease the fiber mechanical strength.

The thermal stability of a SiC_f/SiC composite greatly depends on that of the SiC fibers, even though the degradation mechanism of this kind of composites is affected by some changes occurring at the fiber/matrix interface and by the presence of protective sealing coatings deposited on the composite surface as well.

In order to understand the phenomena happening at the fiber/matrix interface, it is to be considered that the SiC fibers are frequently coated with a CVD carbon layer, referred to as the interphase, prior to the infiltration of the matrix, with the purpose of allowing fiber/matrix debonding under stress. Debonding is one of the mechanisms responsible for an increase of fracture work, which results in an higher strain and in an enhanced toughness. However, Filippuzzi et al.[3] put in evidence that this interphase of carbon is prone to undergo oxidation, giving gaseous carbon monoxide and leaving the fiber surface free to react with oxygen and to form a SiO_2 layer.

The thermal stability in the temperature range of 1000–1300°C of a 2D-Nicalon/C/SiC composite under different aging environments (vacuum, atmosphere of argon or carbon monoxide) has been well investigated by Labrugère et al.[4,5] These authors studied the behavior of a composite produced (by SEP) according to three steps: preparation of a 2D preform by pressing together fabrics of Nicalon fibers; deposition of a pyrocarbon layer (less than 1 μm thick) on the fiber surface; isothermal/isobaric chemical vapor infiltration (ICVI) of the preform to obtain an in-situ SiC matrix.

During the aging treatment of this composite performed under argon or vacuum, several phenomena occur. Firstly, fibers undergo decomposition through the reaction:

$$SiO_{2x}C_{1-x} \rightarrow SiO_{(g)} + (2x-1)CO_{(g)} + (2-3x)C_{(g)}$$

A subsequent reaction causes the destruction of the pyrocarbon layer and the growth of large SiC crystals on the fiber surface:

$$SiO_{(g)} + 2C_{(s)} \rightarrow SiC_{(s)} + CO_{(g)}$$

Both these reactions, involving the formation of gaseous carbon monoxide, can be slowed down if an atmosphere of CO or a SiC seal-coating (deposited by CVD on the surface of the composite specimen) are adopted. Anyway, a progressive weakening of the interfacial bond between fiber and matrix (and the consequent strength decrease) was also observed during aging carried out in the conditions described above. The SiC coating is able to retain, after a treatment of 80 h at 1200°C, about the 70% of the untreated material tensile strength. More prolonged thermal treatments at 1200°C or at higher temperatures result in a more marked degradation due to either grain growth of SiC inside the fibers or formation of a $SiO_2 + C$ layer according to the following reactions:

$$2CO_{(g)} + SiC_{(s)} \rightarrow SiO_{2(s)} + 3C_{(s)}$$

$$CO_{(g)} + SiO_{(g)} \rightarrow SiO_{2(s)} + C_{(s)}$$

According to literature,[6] an external coating of SiC is also suitable for avoiding the oxidative degradation of a SiC/C/SiC composite caused by a thermal treatment at 1200°C carried out under air.

In conclusion, literature data put in evidence that an external SiC coating enhances the SiC_f/SiC thermal stability, hindering both composite oxidation and decomposition reaction of SiC fibers.

This paper deals with the thermal stability of a 2D-Nicalon/C/SiC composite processed by a new infiltration way (SICFILL® method developed by F.N. S.p.A., Boscomarengo, Alessandria, Italy)[7] and coated with CVD (or CVI)-SiC protective layers.

The changes occurring in both microstructure and mechanical features during composite aging, performed in different conditions, have been investigated.

2. Experimental

2.1. Materials and methods

The composite material under investigation was fabricated by a hybrid method involving CVI, CVD and polymer impregnation-pyrolysis (PIP), starting from a 2D-preform (obtained by lay-up of 100×100 mm fabrics of CG Nicalon fibers). The production process included the following steps:

- deposition of a pyrocarbon layer (about 0.3 μm thick) on the fiber surface by chemical vapor infiltration (CVI), carried out at 1100°C using a CH_4/H_2 gaseous mixture;
- deposition (by CVI) of a second surface layer of SiC (about 2 μm thick), performed at about 1200°C using a methyltrichlorosilane/hydrogen (MTS/H_2) mixture;

- preform liquid infiltration under vacuum by a slurry composed of polycarbosilane (PCS), xylene and crystalline β-SiC nanopowders produced by laser assisted synthesis at ENEA;
- solvent evaporation at room temperature and pyrolysis of PCS at 1100°C under inert atmosphere;
- further six densification steps (carried out by PIP process again, but without the addition of SiC particles to the slurry) suitable for achieving a density of the composite plate of about 2.2 g/cm^3.

The composite plates were then machined by a cutting machine equipped with a diamond saw in order to obtain $50\times10\times4$ mm^3 samples. These samples were coated by CVD (MTS/H_2 precursors, temperature of 1200°C) obtaining a seal layer of SiC about 165 μm thick and a final density of 2.56 g/cm^3.

Both this last coating and the previously CVI-deposited on the carbon interphase were placed with the aim of avoiding the composite oxidation as well as the evaporation of fiber decomposition products.

The composite samples were submitted to the following different thermal treatments:

- thermal aging at 1200°C in air for periods up to 200 h;
- thermal aging for 200 h at 1200°C under vacuum (to this purpose the samples were sealed under vacuum in a silica tube);
- thermal cycling (1000 cycles) between 25 and 1150°C (each cycle was performed by keeping the samples in a tubular furnace for 20 min, taking the samples out of the furnace and leaving them to cool in stationary air)

Bars of as-processed composite were cut in several slices (in the parallel and transversal directions with respect to the bar major axis) and the section surfaces were observed by SEM in order to test the homogeneity of the external SiC coating.

The mechanical behavior of the as-prepared composite was compared with that of the treated samples by three point bending tests. The flexural strength was measured, as average of three tests, using a Sintech 10D equipment, with support span of 40 mm and crosshead speed of 1 mm/min.

Some specimens, of the as-processed material and of the composite aged in extreme conditions, were also submitted to Charpy test. An instrumented Izod-Charpy equipment (ATS-FAAR) with a 3.5 kg hammer was used to obtain the impact-load/time curves and to calculate the work of fracture. The microstructure of untreated and aged samples was studied by different techniques: X-ray diffraction (XRD) and grazing-angle XRD (Philips diffractometer equipped with a PW3020 goniometer for grazing angle measurements, Cu K_α), optical microscopy, scanning electron microscopy (SEM) and electron probe microanalysis (EPMA, Jeol-Superprobe JXA-8600), infrared spectroscopy, BET measurements.

XRD measurements were carried out on the sample surface and repeated after progressive mechanical removal of external parts (with definite thickness) of the samples. Grazing angle XRD were performed on the sample surfaces and repeated after mechanical removal of a surface layer about 40 μm thick.

EPMA analyses were done on the transversal sections of the composite specimens: silicon, carbon and oxygen were analyzed. Wavelength dispersion spectrometers (WDS), equipped with thallium acid phthalate crystal (2d = 25.757 Å) for Si, layered dispersion element 1 multilayer (2d = 60 Å) for O and layered dispersion element C multilayer (2d = 99.1 Å) for C, were used to detect the K_α line of these different elements. Standards of pure SiC (Sigma-Aldrich) and pure quartz were used for calibration. After calibration the analysis of each of these standards gave reproducible results, differing no more than 1 at% from the nominal standard composition. The sample analyses were repeated several times (and the results were averaged) in each of the different specimen parts: fiber core, external part of the fibers, dual C/SiC interphase, matrix and external coating of SiC. Also, the thin fiber coatings were well distinguishable in the sample sections of the untreated samples, however, the electron probe (about 1 μm in size) cannot be completely contained inside the thin layer of pyrocarbon. For this reason, the results of the analysis performed on this fiber coating were affected by the presence of the fiber and the SiC interphase, neighbouring with the analyzed area.

Diffuse reflectance Fourier transformed infrared spectroscopy (DR-FTIR; Bruker IFS66 instrument, equipped with MCT-Cryodetector) was used with the aim of checking the presence of Si–O bonds (arising from SiC oxidation) on the sample surface; this analysis was repeated after removal of a 40 μm thick layer from the external SiC sample coating.

Following the results obtained with the methods described above, it was considered necessary to perform further experiments aimed at assessing the capability of the SiC coating of avoiding the gas penetration inside the material. Indirect indications about this coating characteristic were obtained by measuring the surface area of the composite bars by BET adsorption isotherms of nitrogen at 77 K (Carlo Erba Sorptomatic 1800 instrument).

2.2. Experimental results and discussion

2.2.1. Mechanical tests

The flexural strength and the Charpy impact resistance of the composite samples in the thermal treated and untreated conditions are compared in Table 1.

Table 1
Mechanical properties of untreated and thermal treated composite samples

Material	Bending strength		Charpy fracture work
	MOR [MPa]	E [GPa]	[kJ/m^2]
Untreated	483	58	81
After 200 h at 1200°C under vacuum	469	58	–
After 200 h at 1200°C in air	71	60	3.6
Cyled between 25 and 1150°C (1000 cycles)	44	59	4.2

A characteristic stress/displacement curve obtained by bending the untreated composite is reported in Fig. 1 (curve A). This graph shows that the composite underwent a severe deformation during flexural test. In the first part of the test, the stress increase caused a progressive crosshead displacement (up to about 0.6 mm), afterward, the load suddenly fell to about 150 MPa and remained practically constant meanwhile the displacement increased up to more than 1.2 mm. Then the test was interrupted because deformations as large as these were not compatible with the sample-holder geometry. At the test end the specimens were heavily bent, but not shared in two parts, the pull-out of fibers providing the connection of the composite bar at the bending center.

The specimens aged under vacuum showed a similar behavior (Fig. 1, curve B), while the samples thermal treated in air gave a very different response (Fig. 1, curve C). In this last case, the material failed in a brittle mode, after much lower deformation and stress (crosshead displacement of about 0.1 mm and stress of 71 MPa only).

The thermal treatments performed in air result in a marked decrease of modulus of rupture (MOR), but do not cause Young's modulus variations.

After 200 h at 1200°C, the residual flexural strength was about only the 15% of that showed by the as-fabricated composite. Thermal cycling carried out between 25 and 1150°C (1000 cycles) caused a similar effect on the composite mechanical behavior.

The decrease of MOR strictly depended on the progress of oxidation phenomena, because samples aged for 200 h at 1200°C under vacuum maintained a high MOR value (469 MPa).

The embrittlement of the composite samples aged in air at 1150–1200°C was confirmed by Charpy tests. The work of fracture measured by Charpy method greatly depended on the sample orientation with respect to the impacting hammer: the fracture energy was greater when the composite bar was put with its major section thickness (10 mm) perpendicularly to the falling hammer. The results reported in Table 1 refer to this sample orientation. The untreated composites showed an average work of fracture (81 kJ/m^2) much greater than that of the samples thermally treated under air (ranging between 3.6 and 4.2 kJ/m^2). Furthermore, the untreated composite specimens underwent a deformation process before fracture (for a period of 0.9 ms), contrary to the

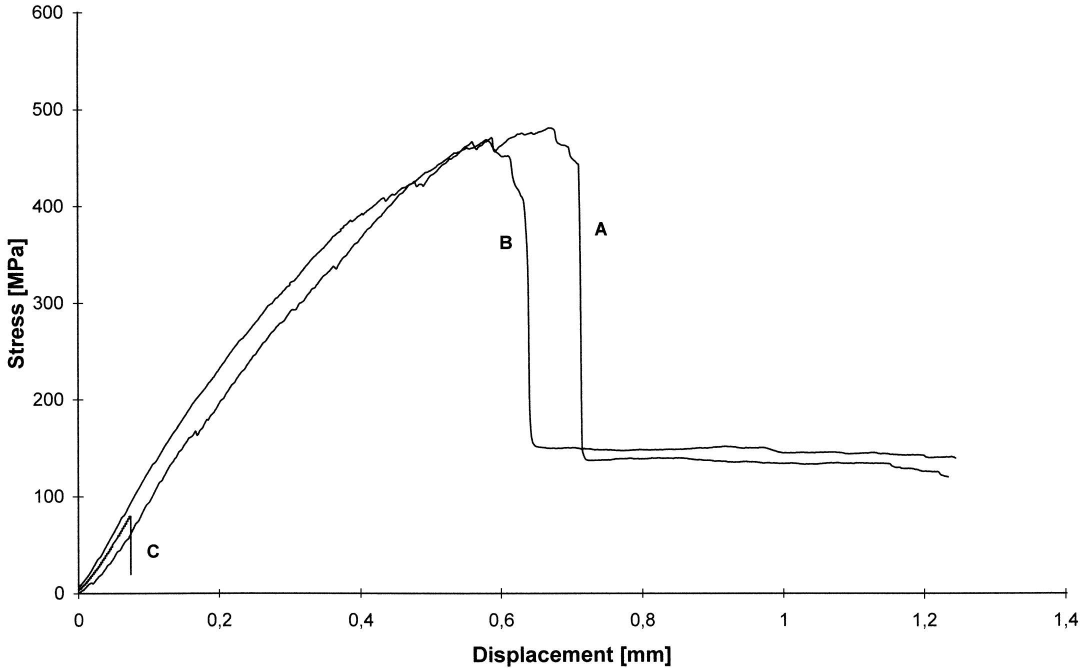

Fig. 1. Three-point bending curves of composite specimens: A = untreated sample; B = sample after 200 h of aging under vacuum at 1200°C; C = sample after 200 h of aging in air at 1200°C.

air aged samples which immediately broke in a brittle manner (Fig. 2).

The Charpy fracture surface of this two kinds of composite specimens was observed by scanning electron microscope. The fracture of untreated samples was characterized by important phenomena of fiber debonding and pull-out (Fig. 3), while the surface fracture of samples aged at 1200°C was completely flat (Fig. 4). On the other hand, the comparison of the transverse sections of these specimens, obtained cutting them some mm apart from the fracture surface, put in evidence that aging affects the interphase morphology (Figs. 5–7). This treatment also caused the fiber damage in several points (Fig. 6). This fiber damage, which likely can occur either during mechanical test or sample cutting also, is a clear evidence of the fiber embrittlement.

2.2.2. Microstructure characterization

2.2.2.1. X-ray diffraction. The XRD patterns of the as-fabricated composite show that all the stronger peaks pertain to silicon carbide. However, after ablation of the more external part of the specimens, the XRD spectra present some changes. In Fig. 8A the spectrum of the sample surface (pattern "a") and those recorded after progressive removal by polishing of surface layers 200

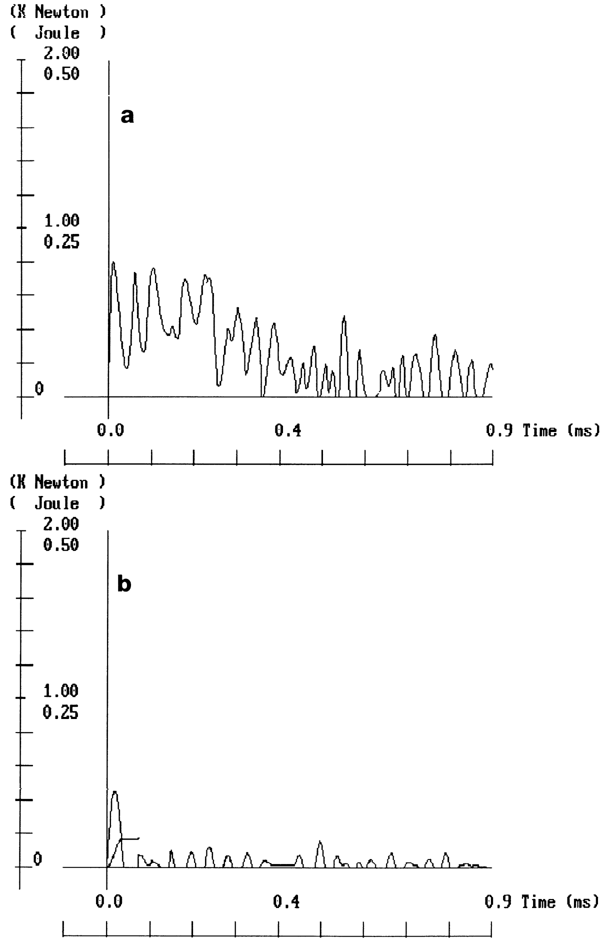

Fig. 2. Charpy load/time curves of composite specimens: a = untreated sample; b = sample aged in air for 200 h at 1200°C.

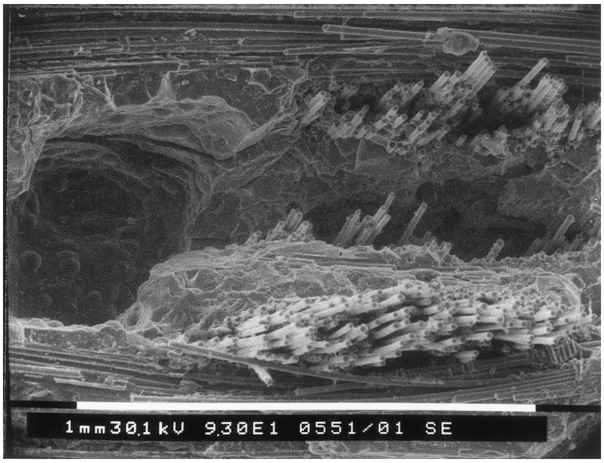

Fig. 3. Charpy fracture surface of untreated specimen: fiber pull-out.

Fig. 4. Flat Charpy fracture surface of a specimen after thermal treatment (200 h) at 1200°C in air.

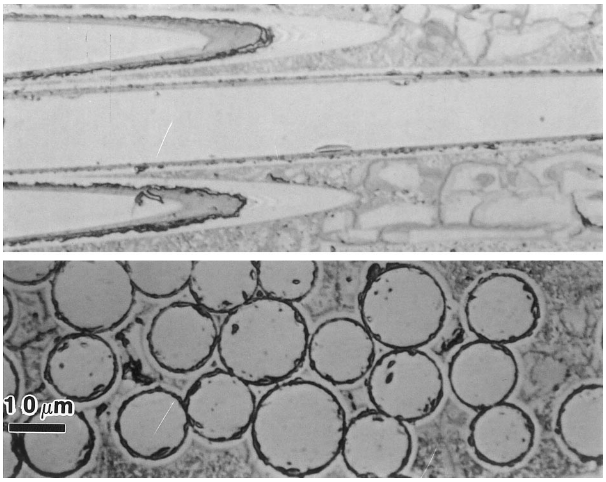

Fig. 5. Section of untreated composite bar.

and 400 μm thick (patterns "b" and "c") are compared. These XRD patterns show that the SiC surface coating processed at 1200°C by CVD is better crystallized than the SiC matrix produced by polycarbosilane pyrolysis at 1100°C. In fact, the intensities of the more important peaks of β-SiC (JCPDS-ICDD card 29-1192) progressively become weaker and their shape widens with the increase of the distance from the sample surface; in the meantime faint peaks likely belonging to α-SiC (ICDD card 42-1091) grow in the XRD pattern. The XRD of the sample surface shows few small, not identified reflexes, that disappear as soon as the surface is polished. Fig. 8B shows the XRD patterns of the samples treated in air for 200 h at 1200°C. In these spectra a new peak placed just below $2\theta = 22$, which can be reasonably attributed to cristobalite (ICDD card 39-1425), was observed. The intensity of this peak decreases moving from the surface toward the inner of the specimen, becoming close to zero at a distance of about 200 μm from the surface. Afterward, its intensity grows in the specimen core, at 400 μm from the surface or more. This particular trend (observed for samples thermally cycled too) was confirmed by grazing-angle XRD measurements. The grazing-angle XRD patterns of the sample aged for 200 h at 1200°C are reported in Fig. 9. Pattern "a" refers to the sample surface and pattern "b" to the same specimen after removal of a layer 40 μm thick: the ablation resulted in a slight decrease in the intensity of the peak placed at $2\theta = 22$. Furthermore, if the XRD measure of the polished specimen (Fig. 9, pattern "b") is repeated adopting a Bragg-Brentano geometry, that is using more penetrating X-rays, the spectrum "c" is obtained. This last spectrum shows that the peak at $2\theta = 22$ is almost vanished.

On the basis of XRD measurements, it can be inferred that oxidation occurred both on the sample surface and in the sample core, but not (or not so much) in the internal part of the SiC coating deposited on the composite bar surface.

2.2.2.2. Infrared spectroscopy. In order to confirm the outcomes of XRD measurements regarding the formation of silica inside the composite samples aged in oxidative environment, infrared spectroscopy was used. The infrared spectrum of the untreated sample surface did not show bands between 1000 and 1250 cm^{-1} , that is in the characteristic range for silica bands. However, the spectra of samples treated at 1200°C or cycled in air showed bands in this range only when the analysis was carried out on the sample surface, while these signals disappeared after sample polishing (removal of a material layer about 40 μm thick). These IR measurements strengthen the XRD results, putting in evidence that the SiC coating oxidation occurs close to the sample surface only.

2.2.2.3. Electron probe microanalysis. The SEM-WDS analyses allow us to compare the chemical composition of composite samples showing a non-brittle behavior (composite specimens untreated or treated under vacuum) with that of composite bars aged in oxidizing environment (kept at 1200°C or cycled between 25 and 1150°C).

The microanalysis results collected in Table 2 show that the atomic percent of C, Si and O, measured in the different part of the composite (fibers, matrix, interphases and external coating), may appreciably change after thermal treatment.

Some of these composite parts display a non-homogeneous composition; for this reason, the composition of both core and external part (labeled with the symbol "*") of Nicalon fibers and SiC bar coating are separately reported in Table 2. The changes in the material composition can be better appreciated by considering the C/Si and O/Si atomic ratios, reported as histograms for untreated and thermal aged samples in Figs. 10 and 11.

The SiC fiber coating and the sample coating of the untreated composite show a C/Si atomic ratio close to

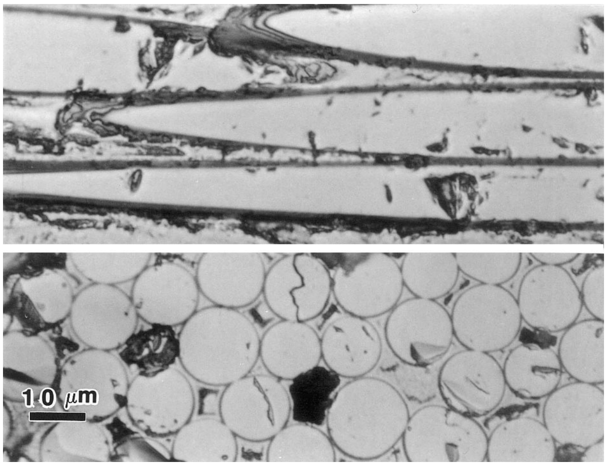

Fig. 6. Fiber damage in the composite aged in air for 200 h at 1200°C.

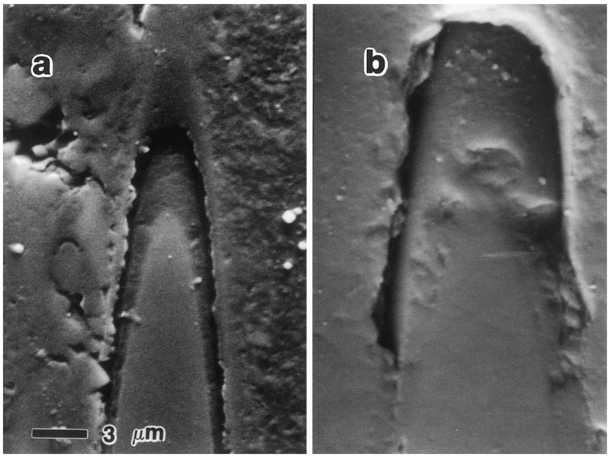

Fig. 7. Fiber coating (a) before and (b) after thermal treatment.

1:1 and less than 1% of oxygen, while the composition of Nicalon fibers is consistent with the presence of β-SiC, SiO_xC_y and free carbon.[8]

The analysis of the sample aged under vacuum at 1200°C puts in evidence only small composition changes with respect to the as-fabricated composite. The vacuum treated composite shows a more uniform oxygen content in the Nicalon fibers and a maximum oxygen percentage in the matrix of 4%. Furthermore, a carbon depletion was observed in the more external part of the SiC sample coating.

The aging in air at 1200°C and the thermal cycling cause much more significant variations in the sample composition. In these last samples the interphases (originally of C and SiC) close to the fiber surface are no more well distinguishable, even though an intermediate zone between fibers and matrix can be seen by SEM (Fig. 7).

In Table 2 as well as in Fig. 11, we have labeled the zone closer to the Nicalon fiber surface as "inner reaction layer" and the adjacent more external zone as "outer reaction layer". Actually, the intermediate zone at the fiber/matrix interface is strongly oxidized, its oxygen atomic percent ranging between 45 and 6%. Conversely, the carbon content decreases here to low values: down to 26% in the inner reaction zone grown on the primary carbon interphase. The oxygen atomic percent also increases in the matrix of the air treated samples (between 7 and 9%). The surface of the SiC coating sealing the composite bars is oxidized, showing

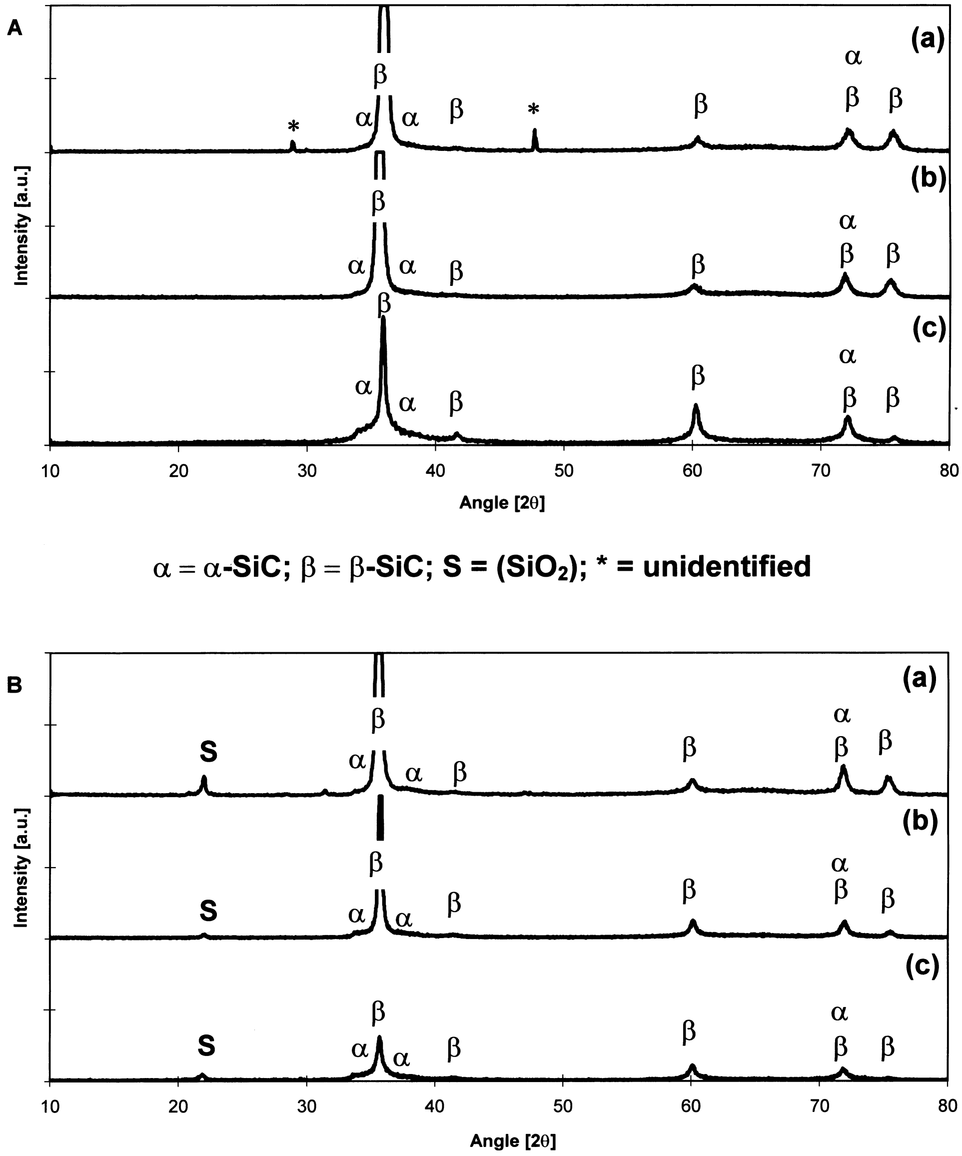

Fig. 8. XRD patterns of (A) the untreated material and (B) of composite specimen after 200 h in air at 1200°C. Patterns: a = sample surface; b = after removal of a surface layer 200 μm thick; c = after removal of a surface layer 400 μm thick.

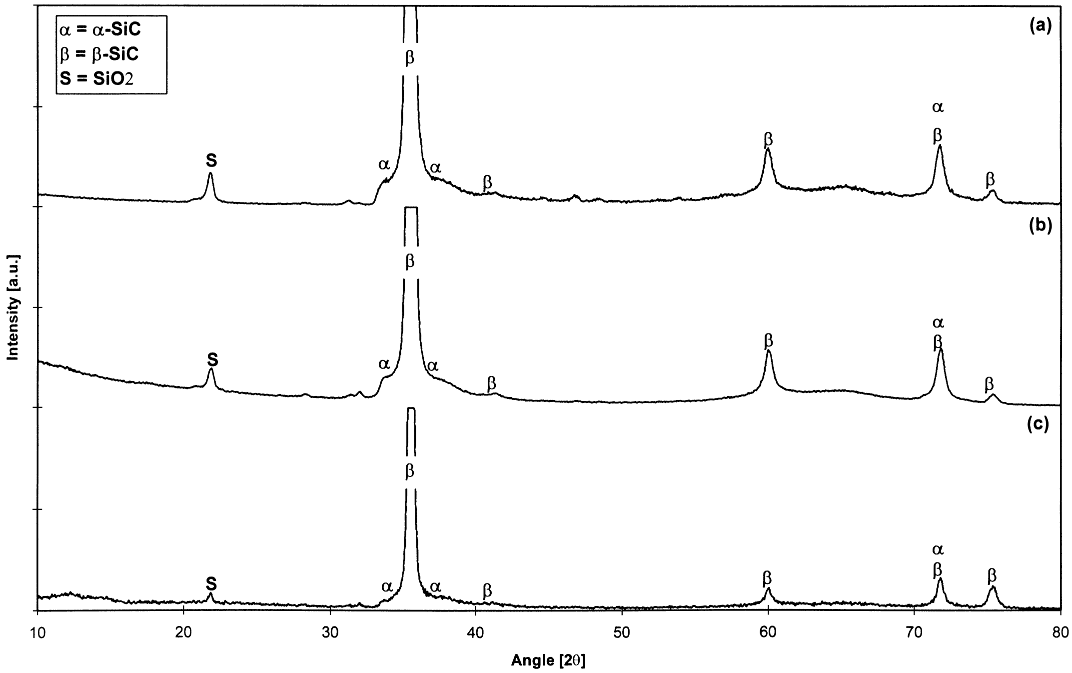

Fig. 9. Grazing-angle XRD patterns ("a" and "b") and Bragg–Brentano XRD pattern ("c") of a sample aged in air for 200 h at 1200°C: a = grazing-angle XRD of the sample surface; b = grazing-angle XRD after removal of a surface layer about 40 μm thick; c = Bragg–Brentano XRD after removal of a surface layer about 40 μm thick.

an oxygen percentage greater than that of the coating core. However, it is surprising that only near the coating surface the oxygen content is greater than 1% at and that it quickly decreases with depth inside this coating. In conclusion, it is clear that the external coating is less oxidized than the composite matrix or the fiber interfaces.

Table 2
Electron probe microanalysis

	Fiber	Interphase of C	Interphase of SiC	Matrix	SiC coating
Untreated composite (at%)					
C	54–55*[a]	63	48	50	51
Si	45–40*	36	51	48	48
O	< 1–5.5*	< 1	< 1	2	< 1
Composite thermal treated for 200 h at 1200°C under vacuum (at%)					
C	51–54*	64	51.5	49	44-41*
Si	47–46*	35	48	47	55–57*
O	~1	< 1	< 1	4	< 1
	Fiber	Inner reaction zone	Outer reaction zone	Matrix	SiC coating
Composite thermal treated for 200 h at 1200°C in air (at%)					
C	54	26	47	47	54–44*
Si	46	29	47	44	45.5–55*
O	< 1	45	6	9	< 1–2*
Composite cycled between 25 and 1150°C (1000 cycles) (at%)					
C	51	33	41	40	53–47*
Si	48	48	46	53	47–53*
O	< 1	18.5	13	7	< 1*

[a] * = composition of the more external part of the zone.

2.2.2.4. Microstructure and gas permeability of SiC seal coating. The sections of the as-processed composite (observed by SEM) show that the external SiC coating is free from cracks, while only occasionally pores can be detected inside this protective layer. However, the thickness of this layer is well far to be constant, because it becomes very thin in some sample parts. Conversely, cracks placed across the SiC layer were observed after thermal treatment at 1200°C and, more frequently, after cycling between 25 and 1150°C.

These results show that the SiC coating looses its integrity during rapid temperature changes, becoming unsuitable for the prevention of oxygen diffusion towards the composite bulk. This behavior is likely foreseeable in the case of samples submitted to thermal cycling, but not for specimens treated in isothermal conditions which experience only one heating step up to 1200°C. Furthermore, for these last composite samples, cracks could arise from cooling at the test end or from sample cutting and polishing (due to the increased material brittleness).

As the results reported above seemed quite puzzling, the gas permeability of the as-processed SiC coating, apparently free from both cracks running through this SiC layer and open porosity, was further investigated by BET measurements. This test gives an indication about the material porosity through the measurement of the total amount of gas (nitrogen) that can be adsorbed on the pore surface.

BET measurements show that the as-fabricated composite bars have a high specific surface area (up to about 10 m^2/g), which is not explainable with their geometrical area and the presence of close surface porosity only. Probably the gas penetrates inside the composite bars where it is adsorbed on the walls of the internal pores. On the basis of BET analyses the presence of few coating defects, not easy to find by SEM, has to be inferred.

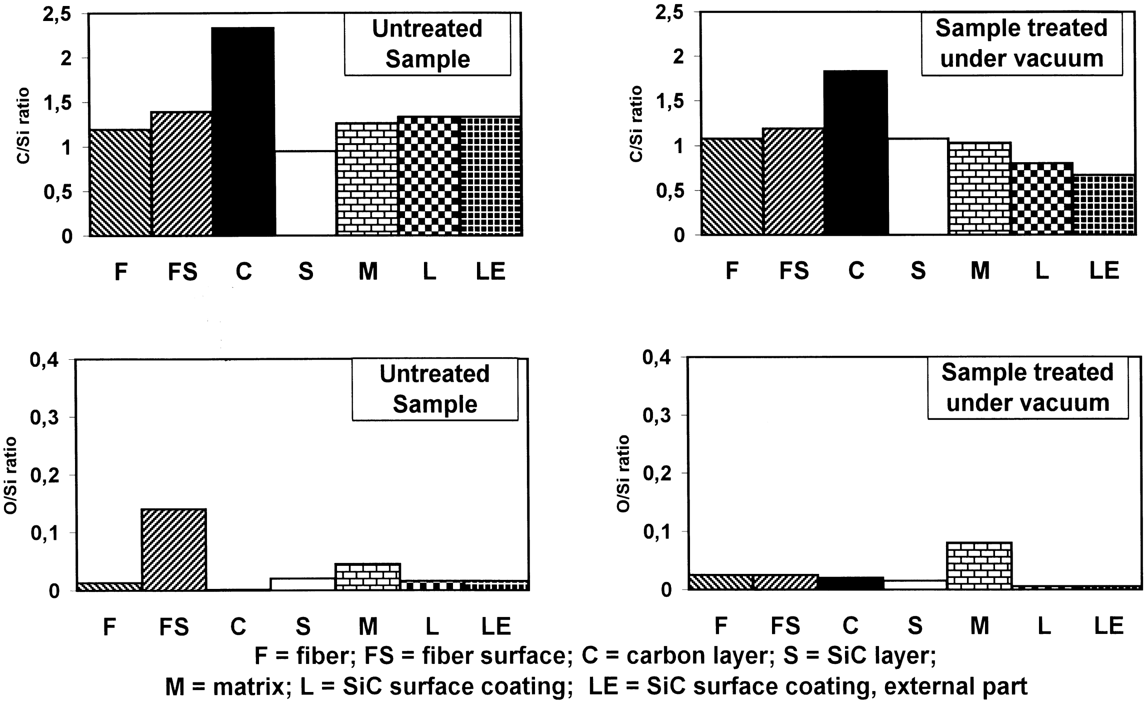

Fig. 10. C/Si and O/Si ratios measured by WDS inside different parts of composite specimens untreated or aged at 1200°C under vacuum.

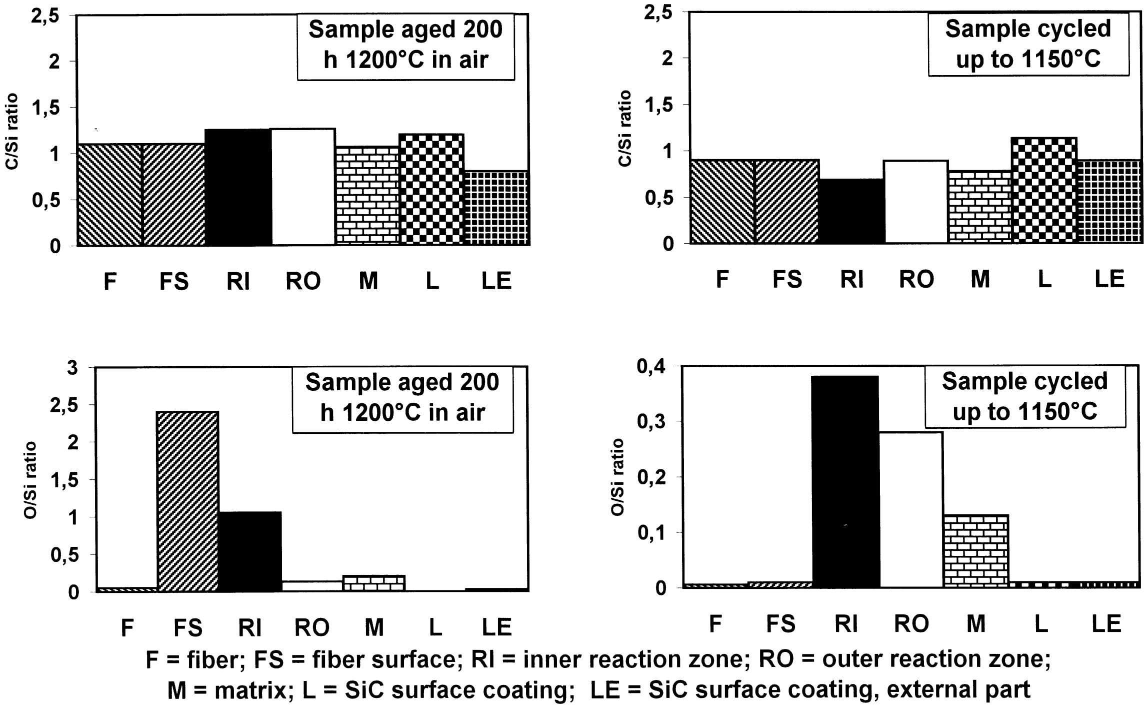

Fig. 11. C/Si and O/Si ratios measured by WDS inside different parts of composite specimens aged at 1200°C in air (200 h) or thermally cycled.

These defects make the composite core prone to undergo oxidation, even though oxygen would diffuse very slowly towards a compact SiC protective layer.

3. Conclusions

A 2D-Nicalon/C/SiC composite, cheaper than other composites of this class because of the use of common ceramic grade fibers and due to the adoption of infiltration process instead of CVI for the matrix fabrication, was produced and investigated. This material shows high flexural strength and appreciable toughness. The composite has a high thermal stability at 1200°C in non-oxidizing environment, but suffers from degradation when aged in air at this temperature.

The study of the degradation mechanism allows to draw the following conclusions:

- the oxidation of the carbon interphase (put between fibers and matrix) and the formation of silica are responsible for the embrittlement and the strength decrease of the composite;
- the oxygen contained in the Nicalon fibers scarcely reacts with the carbon interphase (as shown by the aging tests under vacuum), probably also due to the presence of a second SiC interphase which prevents the emission of gaseous reaction products;
- the oxygen needed for the detrimental process comes from the environment, flowing towards the SiC barrier deposited by CVD on the sample surface;
- this SiC coating in the as-processed specimens seems to be free of cracks, while these last form after thermal aging or after thermal cycling;
- BET measurements suggest that the as-deposited SiC layer shows a non-negligible gas permeability, probably caused by very few defects only occasionally present and, for this reason, difficult to be observed by SEM;
- furthermore, as after thermal treatments the oxygen percent in the more internal part of the SiC coating is near to zero and, generally, very lower than that observed in the composite bulk, the occurrence of a mechanism for oxygen migration (different from solid state diffusion inside the SiC lattice) has to be inferred.

Anyway, the results concerning the oxidation mechanism put in evidence the difficulty to obtain a surface coating suitable for avoiding oxygen diffusion in the working conditions that this class of composite can experience in service (thermal shocks coupled with mechanical stresses). Further difficulties in carrying out effective oxidation barriers should arise from the joint points between SiC/SiC components and other materials. Probably the improvement of SiC/SiC oxidation resistance would be more easily pursued through a suitable modification of the fiber/matrix interfaces.

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