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# SiC<sub>f</sub>/SiC Composite: Attainment Methods, Properties and Characterization

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## 1. Introduction

Silicon carbide exists in several polymorphic forms (over 150) and in each case, the bond between the Si and C is always tetrahedral. The simplest form is silicon carbide (SiC) in a cubic zinc blend structure, also called 3C-SiC or  $\beta$ -SiC. The other polymorphs are a hexagonal network and are known as 2H-SiC, 4H-SiC, 6H-SiC shown in Figure 1, and all are listed as  $\alpha$ -SiC (Ching et al., 2006; Camassel, 2000).

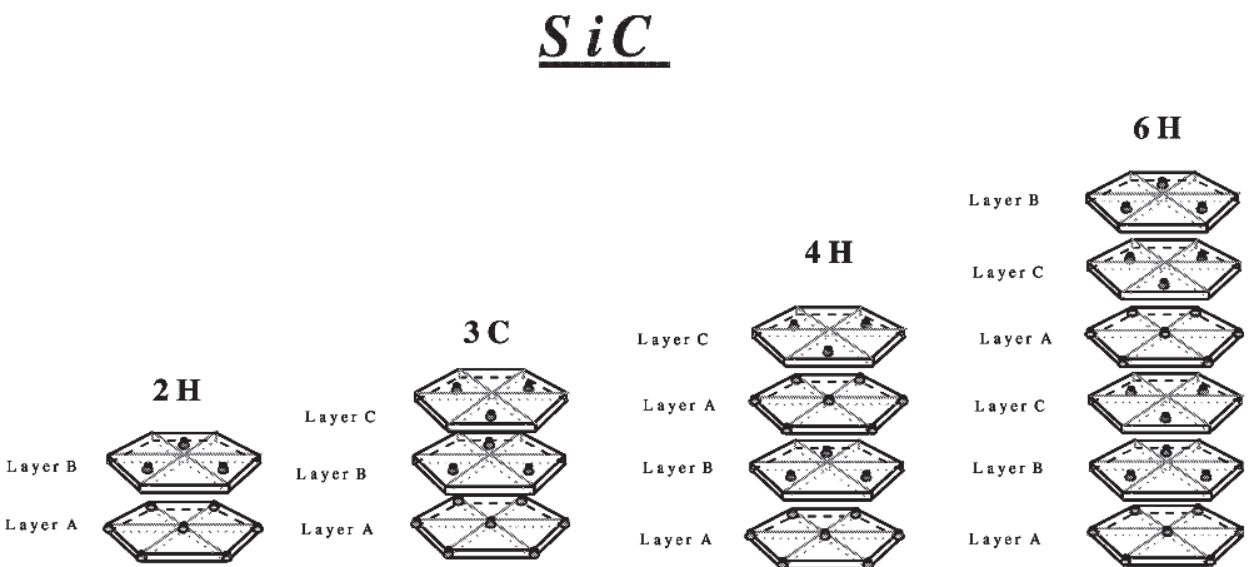


Fig. 1. Illustration of the stacking of successive layers of Si and C to represent the polytypes of SiC (Ching et al., 2006).

The four polytypes shown are the most widely used: 2H and 4H-SiC in the electronics area; 6H-SiC as a nitride substrate for optoeletronics; and 3C-SiC for use at high temperatures (Cassel, 2000).

## 2. SiC<sub>f</sub>/SiC composite

Ceramic silicon carbide (SiC) has received wide attention because of its excellent oxidation resistance, corrosion resistance, and low density even at high temperatures. These materials have been widely used in the engineering industry, chemistry, energy resources and military projects (Xu, 2001). Thus, this material has been used in advanced ceramics, as it combines the advantages of traditional ceramics, such as high hardness, heat resistance, and chemical inertness, with the ability to withstand a considerably tensile strength (Kubel Jr, 1989; Roman & Stinton, 1997) together with high specific hardness and chemical inertness at high temperatures (Kubel Jr, 1989).

Ceramic matrix composites (CMC) materials, based on SiC, containing continuous or woven fibers show potential for many applications such as structural materials at high temperatures in the aerospace (Davies et al., 2001; Ferraris et al., 2000) and automotive fields, as well as, high-performance machines and turbines (Davies et al., 2001).

The SiC<sub>f</sub>/SiC silicon carbide fiber reinforced silicon carbide composite studied in this chapter is part of the class of ceramic matrix composites in which a SiC fiber preform is infiltrated and densified by a matrix of SiC, thus improving its properties. Compared to monolithic SiC, SiC<sub>f</sub>/SiC composite exhibits a high increase in fracture toughness, making it non-catastrophic. (Ortona et al. 2000; Goto & Kagawa, 1996). Therefore, SiC<sub>f</sub>/SiC composite is being considered as a structural material (Young et al., 2000), with potential applications in a wide spectrum of activities, ranging from aerospace and fusion reactors up to filters for pollution control for high temperature and corrosive environment because it is lightweight, tough, and maintains antioxidant stability even at high temperature (Interrante et al., 1997).

The first SiC fibers developed were obtained by deposition via chemical vapor on a tungsten or carbon support. Its large diameter, more than one hundred microns, prevented the weaving of preforms, and only in the early 80's, did small diameter ( $\approx 10\mu\text{m}$ ) "ex-polymer" SiC fibers appear, obtained from polycarbosilane (PCS). This polymer of linear formula -  $(\text{CH}_2\text{SiHMe-CH}_2)_n$ - is reliable in the molten state ( $\approx 200^\circ\text{C}$ ), after being crosslinked in three directions, before finally being converted into ceramics by pyrolysis under nitrogen, argon and hydrogen. The methyl groups (Me) show an excess of carbon (and hydrogen), which is not prejudicial where the mixture of SiC/C is stable up to  $2500^\circ\text{C}$ . Its great nanostructural homogeneity gives "ex-polymer" SiC fibers good mechanical properties. (Gouadec, 2001).

### 2.1 Mechanical properties

When ceramic fibers are embedded in the ceramic matrix composite, mechanical properties are quite different from monolithic ceramics because of the reinforcement fibers, which act so that the mechanical stress received by the matrix is transferred to the fiber, increasing the flexural resistance and fracture toughness. For example, the fracture toughness and thermal shock resistance of the composites are superior when compared to monolithic materials. The fracture toughness of monolithic SiC is close to  $5\text{ MPa}\cdot\text{m}^{1/2}$ , while the SiC<sub>f</sub>/SiC composite is in the order of  $20\text{--}30\text{ MPa}\cdot\text{m}^{1/2}$ . Moreover, the properties of ceramic matrix composites (CMC) can easily be adapted, varying, for example, the architecture of the fibers, fiber types, interfacial layers of materials and thickness of composites.

Due to the efficiency of the CVI process to fill between the fibers, and the purity and crystallinity of the matrix material, it is expected that the mechanical properties of composites obtained by CVI are better than those of composites obtained by other techniques. However, no major difference in the values of flexural strength and fracture is

observed. The average flexural strength of SiC composite with Nicalon fiber obtained by different methods is 300 MPa with a fracture toughness of 15 MPa.m<sup>1/2</sup> (Roman & Stinton, 1997).

## 2.2 Chemical properties

SiC<sub>f</sub>/SiC composite is considered a structural material when in contact with liquid materials, such as Li<sub>16</sub>Pb<sub>84</sub> and Li<sub>2</sub>BeF<sub>4</sub>, which are used as refrigerants, tritium producers (<sup>3</sup>H<sub>1</sub>) and protective materials for fusion reactors whose compatibility of SiC<sub>f</sub>/SiC with molten materials is studied. According to thermodynamic assessments, the β-SiC crystalline phase is stable against lithium metal saturated with oxygen. For the alloy Li<sub>16</sub>Pb<sub>84</sub>, SiC can be more stable because the activity of lithium in the alloy is 4-5 times lower than lithium pure (Yoneka, et al., 2001). The predominant chemical reactions between SiC and liquid metals are those which result in carbon exchange. SiC is quickly eroded when in contact with liquid lithium ( $2\text{SiC} + 2\text{Li} \rightarrow 2\text{Si} + \text{Li}_2\text{C}_2$ ) at temperatures above 600 °C. At high temperatures, SiC<sub>f</sub>/SiC composites are stable when exposed to corrosion of Li<sub>17</sub>Pb<sub>83</sub> at 800 °C for 1500 hours.

Corrosive factors found in the atmosphere (H<sub>2</sub>O, NO<sub>x</sub>, NaCl), or derived from kerosene (sulfides, Na<sub>2</sub>SO<sub>4</sub>, K<sub>2</sub>SO<sub>4</sub>) will necessary accelerate the corrosion process. This phenomenon is difficult to anticipate, since the combustion atmospheres vary strongly according to the kind of the fuel and temperatures, typically in the order of 1400 °C in combustion chambers. SiC is inert under N<sub>2</sub>, H<sub>2</sub> or in the H<sub>2</sub>-H<sub>2</sub>S mixture but particularly vulnerable to oxidation by hot steam and corrosion by molten alkali salts. SiC fibers have good resistance to oxidation at temperatures up to 1300 °C for the Hi-Nicalon fiber and even higher temperatures for the quasi-stoichiometric fibers. However, the ceramic matrix component of the CMC, for which are designed for extreme conditions, can limit its lifetime (<10,000 hours required for civil construction) (Gouadec, 2001).

## 2.3 Thermal properties

The thermal conductivity of ceramics is strongly influenced by structural defects, impurities, grain size and porosity; in the specific case of ceramic matrix composites (CMC) the fiber-matrix interface also significantly influences the thermal conductivity. The SiC single crystal with high purity has a thermal conductivity of about 5000 W.m<sup>-1</sup>.K<sup>-1</sup> at 50 K and decreases to about 500 W.m<sup>-1</sup>.K<sup>-1</sup> at room temperature (298K). SiC<sub>f</sub>/SiC composite, because of its high porosity, inherent in the manufacturing process has low thermal conductivity in the direction perpendicular to the fibers. SiC<sub>f</sub>/SiC composites exhibit advanced thermal conductivity of 73 W.m<sup>-1</sup>.K<sup>-1</sup>, at room temperature and 35 W.m<sup>-1</sup>.K<sup>-1</sup>, at 1000 °C, approaching the sintered monolithic SiC. The thermal conductivity varies with the orientation of fibers within the composite (Sharafat et al., 1995).

## 3. SiC<sub>f</sub>/SiC composite fabrication

The most common methods for the production of SiC matrix reinforced with high strength SiC are: chemical vapor infiltration, chemical vapor deposition, impregnation and liquid pyrolysis with reaction sintering and chemical vapor reaction, the latter being, the method used in this study (Roman & Stinton, 1997).

### 3.1 Chemical vapor infiltration

The chemical vapor infiltration appears to be one of the most promising techniques for preparing CMC currently available, for several reasons: the SiC crystal matrix with high purity and density, can be introduced into complex preforms at relatively low temperatures. Damage to the fiber and the interdiffusion between the matrix and fibers are avoided and densification requires no additional mechanical load, and finally, the application of a layer at the interface is part of the process. The first information about the CVI technique for the preparation of ceramic materials was patented in 1964. In the CVI process, a porous preform is heated and reactive gases are passed through its pores. Most commonly, this preform is made of woven carbon or fibers SiC.

There are reports of the use of tungsten carbide substrate, where the material is infiltrated into thin  $\beta$ -SiC crystals, preferably oriented in the tungsten parallel plane (111). The problem is that the SiC formed reacts with the substrate after a prolonged use at temperatures above 1000 °C, forming  $W_2C$  and  $W_5Si_3$ , affecting and consequently degrading their properties (Matthews & Rawlings, 2000). One of the most common precursors of silicon carbide is methyltrichlorosilane. The reaction via chemical vapor infiltration to form the SiC matrix is given in Equation 1.



At first, a variety of matrices can be produced using other gaseous precursors. The modification process via chemical vapor infiltration differs in process conditions, that is, temperature and pressure (around 850 – 1200 °C and between 1 to 1000 mbar). Ortona (Ortona et al., 2000), Interrante (Interrante et al., 1997) and Nechanicky (Nechanicky et al., 2000) have suggested that this process requires, complex and expensive equipment and typically produces gaseous hydrochloric acid as a byproduct, in addition to the difficulty of infiltrating into thick layers ( $\geq 4$ mm). Pochet (Pochet et al., 1996) also studied some limitations of the chemical vapor infiltration technique: exhaust from the gas phase, which results in a high growth rate of the substrate layer, nodular deposits and variation of the deposit thickness in the substrate.

### 3.2 Infiltration and polymer pyrolysis

Another way to manufacture  $SiC_f/SiC$  composite can be by the route of infiltration of polymer precursors or the sol-gel method. The advantages of this method are related to low processing temperatures, effective mixture of the components of the composite, therefore, greater homogeneity and potential for the formation of multiphase matrices. The liquid precursor for the formation of the ceramic is infiltrated into fibers or particles to produce the matrix. The precursor is converted by hydrolysis in the sol-gel and thermal decomposition of the polymer precursor method. The result is a densified matrix and the process can be repeated several times to achieve an increase in the density. The major disadvantages of the sol-gel process are: low yield of the reaction, high shrinkage and low rate of gelation of polymers. (Nechanicky et al., 2000).

The alternative to this process is polymer precursor infiltration and subsequent pyrolysis of matrix (Nechanicky et al., 2000), making the technique economically attractive (Ortona et al., 2000). For the infiltration process and pyrolysis of polymer to be successful, the following parameters must be optimized:



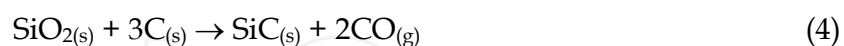
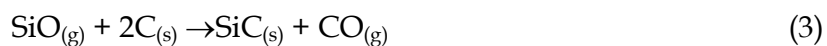
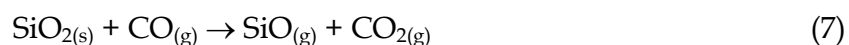
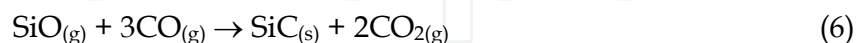
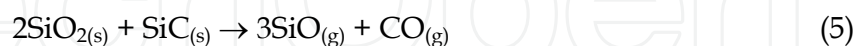
- High efficiency for impregnation;
- Applicable to manufactured components with complex sizes (Ortona et al., 2000);
- Quality of the precursor (high yield, chemical purity after the pyrolysis and controlled microstructural evolution) (Nechanicky et al., 2000);
- Porosity (minimum porosity or completely open in the green compact and during processing);
- Microstructure (controlled densification with minimal grain growth during sintering) (Nechanicky et al., 2000).

For the manufacturing of SiC matrix, two routes were developed using polycarbosilane (PCS), which maintains the stoichiometry Si:C 1:1, when pyrolysis is carried out. The first route involves the synthesis of linear PCS  $[\text{SiH}_2\text{CH}_2]_n$ , which has all the desirable characteristics for an ideal matrix of SiC, but presents high costs and low yield. The precursor of this study allows a detailed understanding of cross-links and pyrolytic conversion processes occurring in the PCS system containing Si-H to produce a high yield of SiC. The second route employs a different organosilane, chloromethyltrichlorosilane ( $\text{ClCH}_2\text{SiCl}_3$ ) as starting material (lower cost) and obtains a final product which differs in terms of structure with, however present approximately the same compositional formula  $[\text{SiH}_2\text{CH}_2]_n$ . In this case, either the properties of the precursor as well as projected costs are calculated in the application of SiC matrix (Interrante et al., 1997).

One drawback of this technique is that the matrix of SiC obtained after pyrolysis, has lower purity and an amorphous glassy phase, resulting in decreased thermal and mechanical properties compared to composites obtained by chemical vapor infiltration. (Lin et al., 1995). Kotani (Kotani et al., 2001) proposed a process for fabrication of SiC<sub>f</sub>/SiC composite using PIP, which provides highly efficient impregnation and control of the microstructure, with reaction sintering to obtain a dense SiC matrix without hot pressing. The impregnated fibers were prepared by polymeric densification intra-fiber with four options in the process: (a) pyrolysis of the SiC precursor, (b) pyrolysis of carbon precursor and subsequent reaction sintering of polymer-derived carbon and silicon particles; (c) reaction sintering between particles of silicon and carbon and (d) reaction sintering between particles of carbon and impregnation of liquid silicon. Process optimization was performed by adjusting the ratio in the mixture and formation conditions in order to reduce porosity.

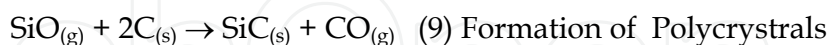
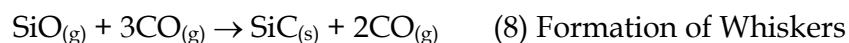
### 3.3 Chemical vapor reaction

The chemical vapor reaction method was first introduced in 1985 and patented in 1992. In this process, particles or grains of SiC are formed from gaseous precursors via chemical vapor deposition and deposited on a hot graphite substrate, resulting in a coating. Having attained sufficient thickness, the graphite is removed by oxidation (Roman & Stinton, 1997). The chemical vapour reaction method is based on the carbothermal reduction process, in which an intermediate phase of SiC is formed during the reduction process of silica and carbon. The chemical reaction between silica and carbon is applied to the formation of a SiC layer on carbon materials and synthesis of powders of SiC and Si. The carbothermal reduction process can be classified into two steps. The first step is the reduction of silica by carbon, this step consists of the formation of SiO, CO, and the formation of SiC (intermediate phase) between SiO and carbon. The second step is the reduction of silica by SiC, this step consists of the formation of SiO and CO from SiC and silica, and the formation of SiC from SiO and CO (Yun et al., 2005).

*First Step**Second Step*

The transformation of carbon into  $\beta$ -SiC (zinc blende structure) can be driven by the following steps: at high temperatures, graphitic materials show an appreciable expansion of the C-axis, in which case the prismatic planes have a great reactivity. Moreover, it is thought that conversion of the graphite substrate is obtained by the interaction of SiO gas and planes of crystalline carbon (or atomic sites), which are more reactive than other planes. Probably, the process includes the decomposition of SiO and the insertion of Si in the structure between the carbon layers. As a result, carbon crystalline units are converted into SiC tetrahedral units.

During the conversion of carbon into SiC, some SiC whiskers could be formed. This occurs because the formation of SiC leads to a decrease in the SiO/CO ratio during the conversion process. It is suggested that the two reactions may occur following competition with one another in the conversion process. The notable difference in the two reactions is the type of phase reactant, a vapor-vapor reaction (Equation 8), in contrast to a solid-vapor reaction (Equation 9). It is known that the reactant SiO and CO should be provided for the production of SiC whiskers, so the formation of SiC whiskers is likely (Yun et al., 2005), according to the equations:



Kowbel (Kowbel, 1997, 2000) transformed only the carbon fibers into SiC fibers from a carbon cloth through the process of chemical reaction via vapor at high temperatures, which used SiO gas as the reactive gas. The conversion of carbon to SiC was controlled by the gas generation and temperature of operation. The level of conversion was measured by direct oxidation of the converted fibers and by scanning electron microscopy of a cross section of fibers converted. After observation, it was found that 100 per cent of the fibers were converted into SiC after being subjected to this treatment.

In order to obtain the composite the chemical vapour infiltration technique was used following four different routes: (a) SiC powder consolidation and densification by chemical vapor infiltration, (b) SiC powder consolidation and densification via hot-pressing; (c) PCS consolidation and densification by chemical vapor infiltration and (d) consolidation and densification via chemical vapor infiltration, achieving high purity crystalline  $\beta$ -SiC.

Ohsaki (Ohsaki et al., 1999) produced the SiO gas, using a powder mixture of Si and SiO<sub>2</sub>, with a ratio of 1:1 (%weight), which was heated from 1200 to 1400 °C, varying 1 to 10 hours (in a vacuum of 1.5 x10<sup>-2</sup> Torr) to obtain β-SiC from activated carbon.

With this reaction mixture, it is necessary to heat the system at temperatures below the eutectic point to cause a process of oxidation-reduction in the mixture, where silicon oxides and SiO<sub>2</sub> reduce, according to the reaction shown in Equation 10:



Tang (Tang et al., 2000) grew SiC nanotubes from carbon nanotubes in an oxidizing atmosphere of SiO. From this growth, it was expected that SiC nanotubes would have a diameter have diameter equal to carbon nanotubes, however, nanotubes have an epitaxial growth on the surface of SiC due to the reaction between gaseous SiO and CO as shown by Equation 6. Furthermore, the CO<sub>2</sub> gas generated can react with carbon nanotubes still present, reducing the initial diameter of carbon nanotubes, thus mitigating the SiC nanotubes, consequently, presenting a more widespread diameter, as shown in Equation 11.



Rogers (Rogers et al., 1976) covered the C/C composite with a silicon carbide layer using the “*pack-process*” technique which was used in a powder mixture consisting of 60% SiC, Si 30% and 10% Al<sub>2</sub>O<sub>3</sub>, in which the first stage is controlled by the liquid phase, where the molten metallic silicon reacts with carbon to form SiC and the second stage is controlled by the vapor phase, where silicon vapors react with carbon. The SiC formed on the carbon surface is presented in cubic form (β-SiC).

#### 4. Conversion of C/C composite into SiC<sub>f</sub>/SiC composite

Powders and materials utilized in this conversion process are:

- Carbon fiber twill, T-10 EKHO (Ural, Ukraine), obtained by carbonization of a PAN precursor;
- Phenolic resin Resafen 8121, manufactured by Reichhold- Resana Ind. Quim. S/A (Mogi das Cruzes, SP, Brazil), in the form of a liquid resin soluble in water, used as carbon matrix precursor;
- Silicon powder from Elektroschmeltzwerk Kempten GmbH, (Kempten, Germany), 99.9% purity and mean size particle 10 μm;
- SiO<sub>2</sub> powder manufactured by Mineração Jundu, (Descalvado, SP, Brazil), 99% purity and mean size particle <2 μm;
- α Al<sub>2</sub>O<sub>3</sub> SG A-16 manufactured by Alcoa, 99% purity and sub-micron particles.

The first step in the preparation is the fabrication of a primary C<sub>f</sub>/C composite by a PIP method. A first carbon/resin (C<sub>f</sub>/R) composite is made out of eight carbon fabric layers impregnated with phenolic resin. The resulting laminate was heated in an autoclave with a heating rate of 5°C/min and a pressure of 0.3 MPa up to 130 °C. The C<sub>f</sub>/R composite was carbonized at 1000 °C in an argon atmosphere, with a heating rate of 60 °C/h. The resulting C<sub>f</sub>/C material was dried for 12 h at 180 °C.

The second step involves the transformation of the C<sub>f</sub>/C into SiC<sub>f</sub>/SiC by Chemical Vapor Reaction (CVR). The source of gaseous silicon monoxide was produced by two different



powder mixtures. First by a reaction among 60%SiC + 30%Si + 10%Al<sub>2</sub>O<sub>3</sub> (%weight) (Rogers et al., 1976) (called Mixture 1) and the second by the reaction of 50% SiO<sub>2</sub> + 50% Si (%weight) (called Mixture 2), both of which were prepared by co-milling in a planetary mill for 30 min and drying for 12 h at 180 °C. The mixture 1 powder was put in a carbon crucible and the C<sub>f</sub>/C was placed inside the powder mixture. The mixture 2 powder was placed in an alumina crucible and the C<sub>f</sub>/C composite was located above the mixture without contact with the powder. Mixture 1 was placed in a furnace in vacuum atmosphere and heated at temperatures from 1400 °C to 1800 °C for 3 h in a with a heating rate of 10 °C/min. Mixture 2 was placed in a furnace in a vacuum atmosphere and heated at 1400 °C for 3 h in a with a heating rate of 10 °C/min. Total conversion of the material was verified by X-ray diffraction and scanning electron microscopy.

## 5. Results

### 5.1 Results from Mixture 1 (60%SiC, 30%Si e 10%Al<sub>2</sub>O<sub>3</sub>)

Figure 2 shows the photomicrographs obtained by scanning electron microscopy of the cross section of converted SiC<sub>f</sub>/SiC composites. At the temperature of 1400 °C, the composite was not fully converted, because a difference can be observed a difference in the fiber color. Since the images are taken by electron backscattering, is possible to obtain contrast due to the atomic weight of elements (the heaviest appear brighter), we can say that the light outside corresponds to the carbon converted into SiC and the inside dark carbon not converted. This partial conversion can be proven by an X-ray diffractogram of the composite surface, which reveals a band of carbon, as shown in the Figure 3.

At the temperature of 1600 °C, the composite is fully converted as shown in the Figure 4. Because the fiber and matrix of the composite show a homogeneous contrast (carbon converted into SiC). This conversion is shown by an X-ray diffractogram of the composite surface not revealing the presence of carbon as shown in Figure 5, indicating a complete conversion.

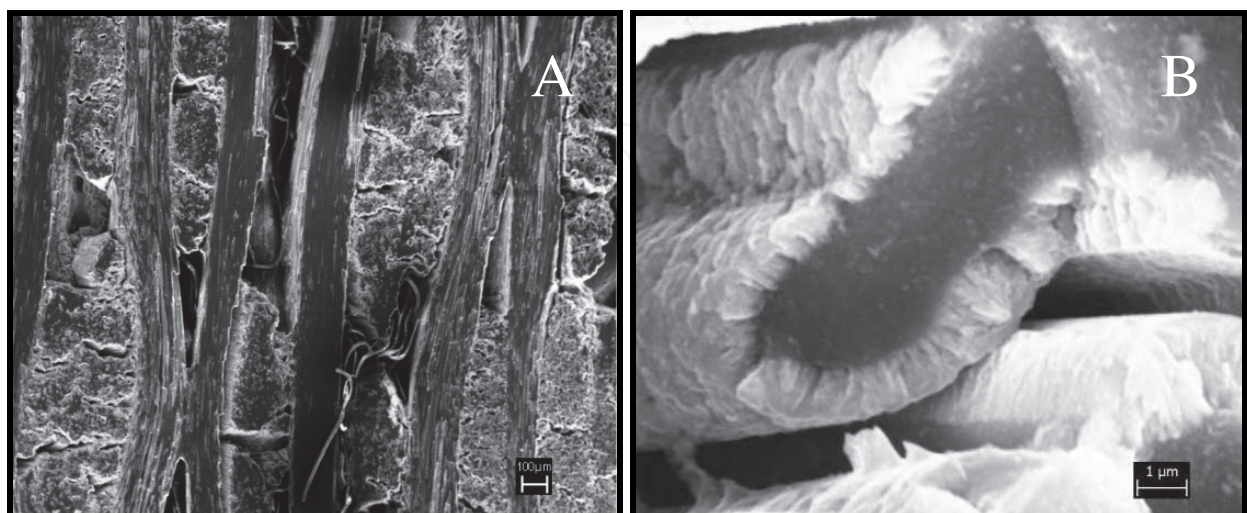


Fig. 2. (a) Scanning electron micrographs of SiC<sub>f</sub>/SiC composite converted at 1400°C for 3 hours and (b) Fibers not fully converted into SiC.

At the temperature of 1800 °C, the composite, in addition to being totally converted, showed a growth of SiC grains, altering the shape of the fiber and causing cracks in the grain boundaries as shown in the Figure 6. This conversion is shown by X-ray diffractogram of the composite surface. The presence of silicon is also observed, from the silicon melting of the mixture, as shown in Figure 7, because the composite is in contact with the powder mixture during the conversion. Transformation at this temperature is harmful to the integrity of the composite, despite the increase in crystallinity of the β-SiC phase, as shown by the decreased background and increased intensity of characteristic peaks in the X-ray diffractogram.

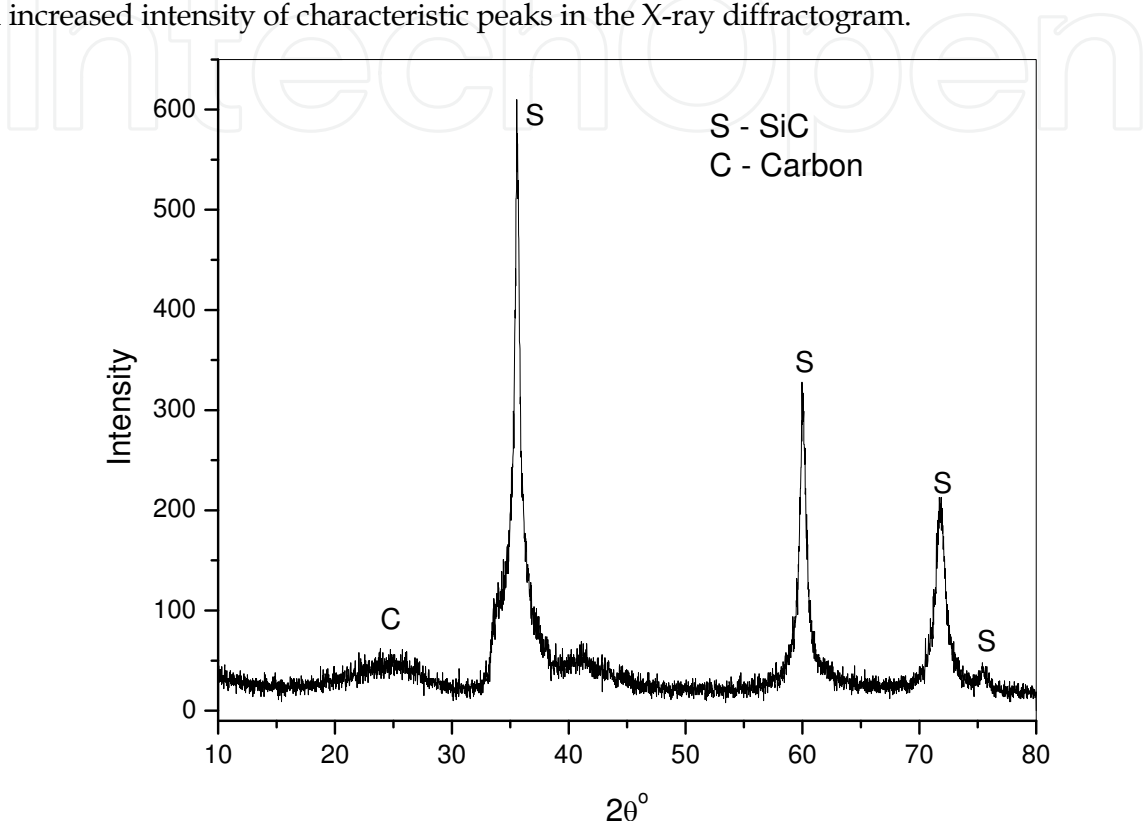


Fig. 3. X-Ray diffractogram of phase transformation of carbon into SiC at 1400°C for 3 hours.

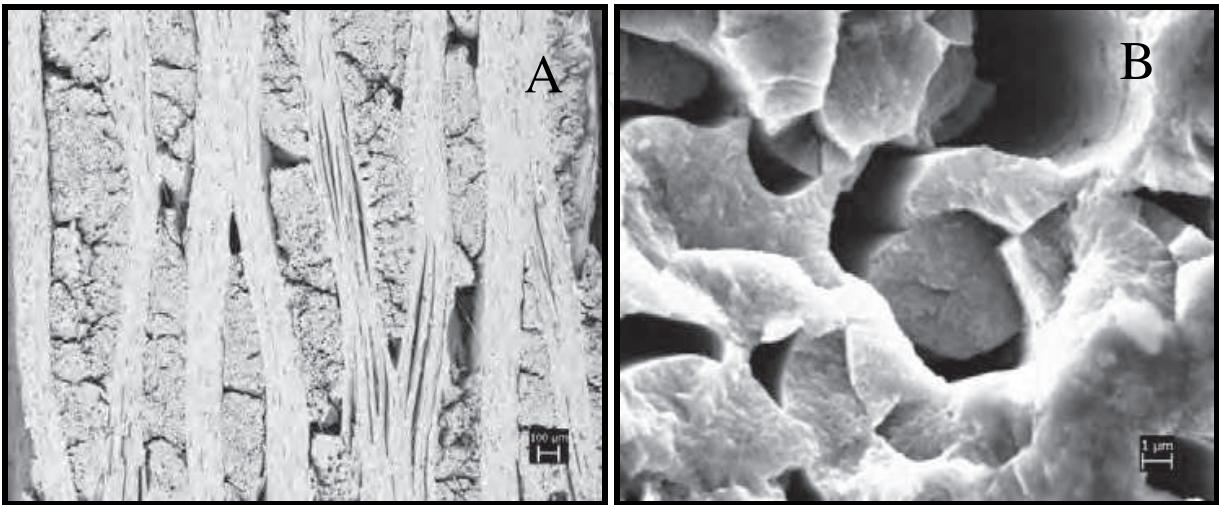


Fig. 4. (a) Scanning electron micrographs of SiC<sub>f</sub>/SiC composite converted at 1600°C for 3 hours and (b) Fibers fully converted into SiC.

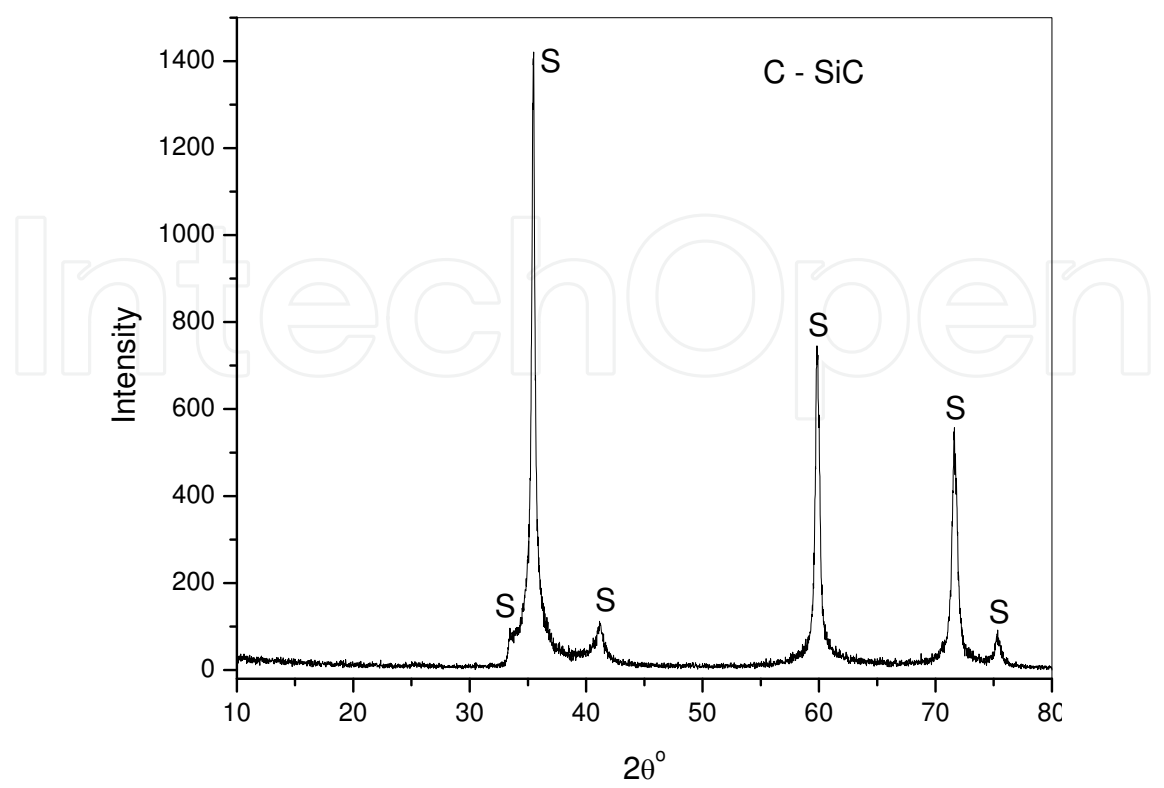


Fig. 5. X-Ray diffractogram of phase transformation of carbon into SiC at 1600°C for by 3 hours.

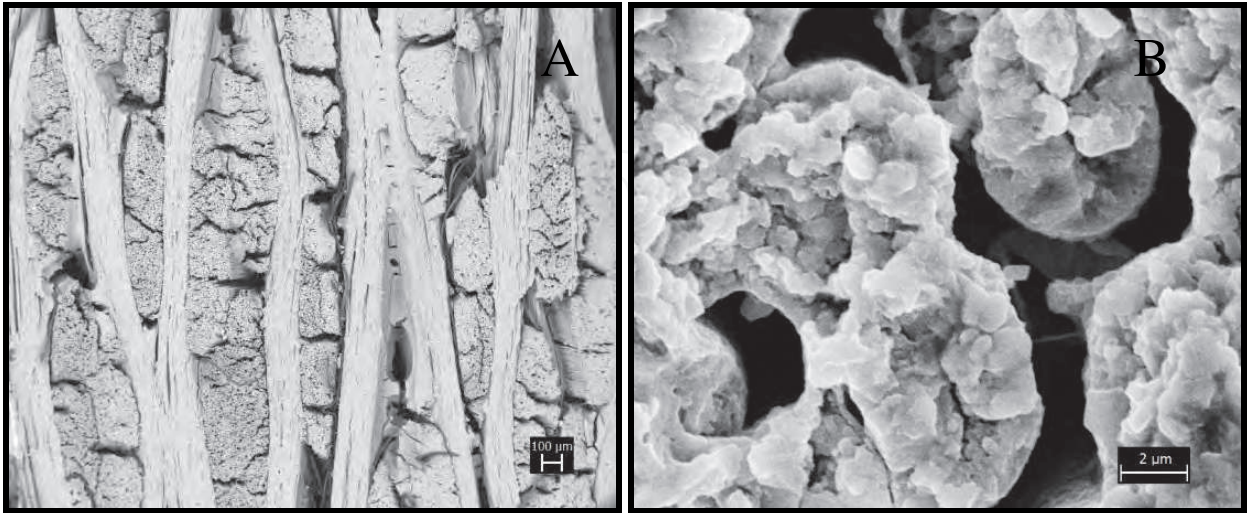


Fig. 6. (a) Scanning electron micrographs of SiC<sub>f</sub>/SiC composite converted at 1800°C for 3 hours and (b) Fiber totally converted into SiC with the grain growth.

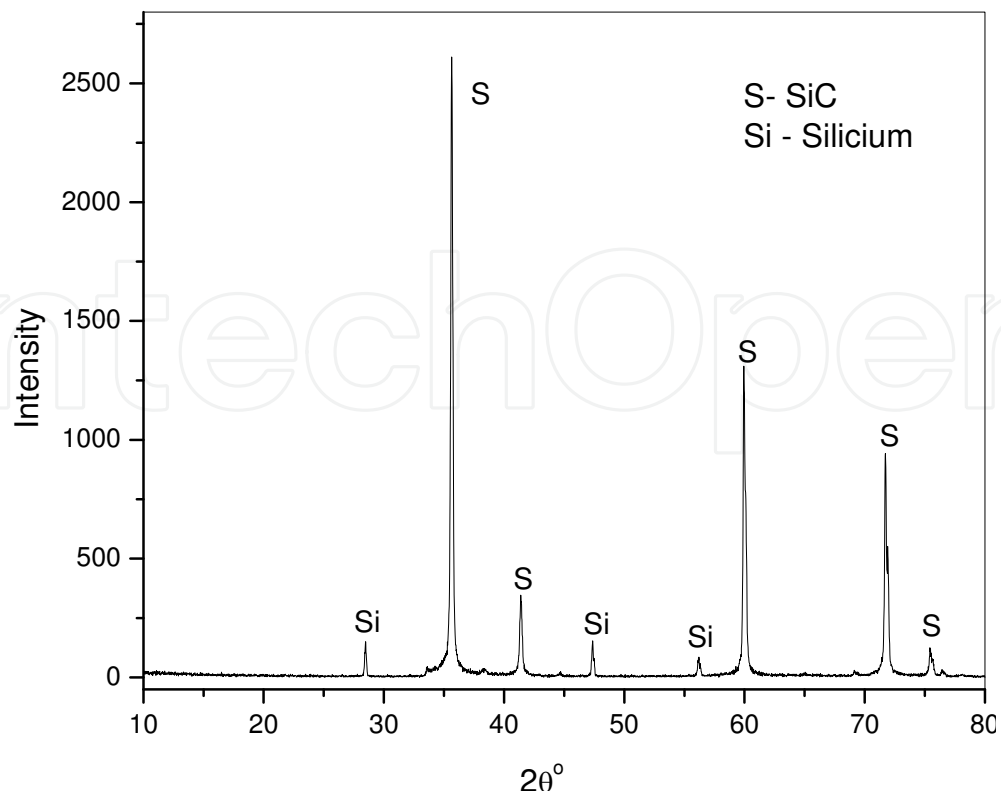


Fig. 7. X-Ray diffractogram of phase transformation of carbon into SiC at 1800°C for 3 hours.

From the results presented of the microstructure analyzed by scanning electron microscopy (SEM) and X-ray diffractogram (XRD), the best fit to convert the composite was at a temperature of 1600 °C, since we obtained a SiC<sub>f</sub>/SiC composite fully converted and intact without fracture of the fibers. The analysis by energy dispersive spectroscopy (EDS), performed to determine the constituents of the composite, revealed the presence of aluminum from alumina used in the mixture, as shown in Figure 9.

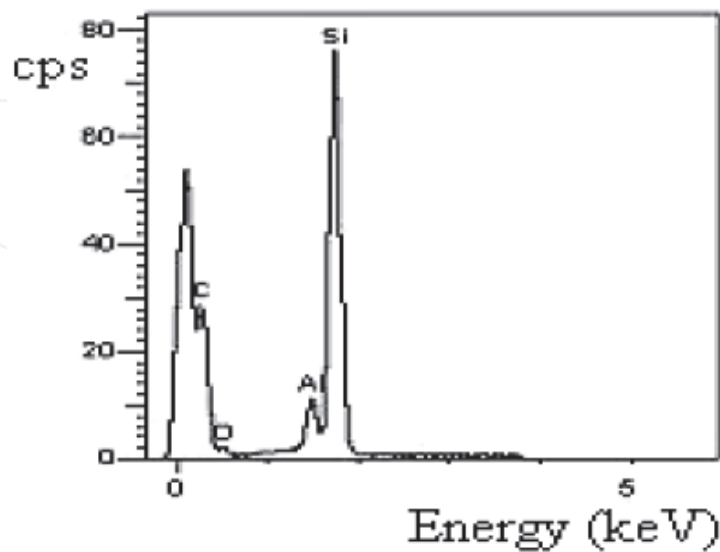


Fig. 8. Elemental analysis by energy dispersive spectroscopy in the SiC<sub>f</sub>/SiC composite processed at 1600 °C, showing the presence of Si, C and Al.



The presence of aluminum in the  $\text{SiC}_f/\text{SiC}$  composite is harmful because it reduces the values of mechanical strength at high temperatures, and increases the values of conductivity/thermal diffusivity (Itatani et al., 2006).

**5.2 Results from Mixture 2 (50%  $\text{SiO}_2$  + 50% Si)**

Figure 9 presents photomicrographs by scanning electron microscopy of the  $\text{SiC}_f/\text{SiC}$  composite converted by the Mixture 2.

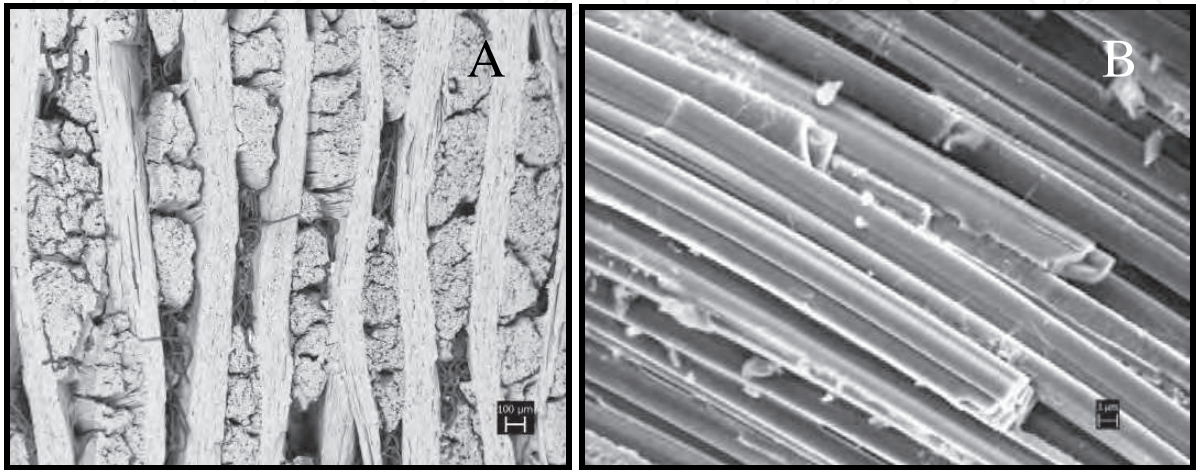


Fig. 9. (a) Scanning electron micrographs of  $\text{SiC}_f/\text{SiC}$  composite converted at  $1400^\circ\text{C}$  for 3 hours. (b) Fibers fully converted into SiC.

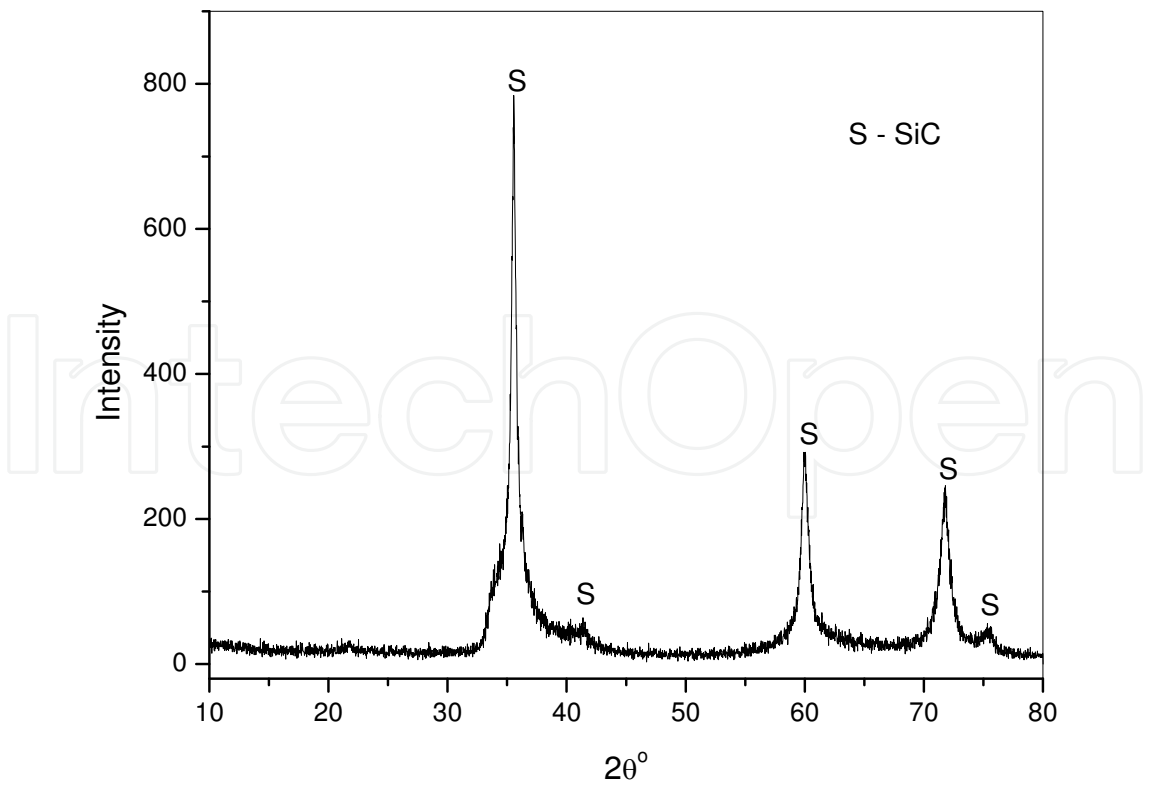


Fig. 10. X-Ray diffractogram of phase transformation of carbon into SiC at  $1400^\circ\text{C}$  for 3 hours.



The composite was fully converted into SiC. The fibers showed a smoother texture than those obtained in the conversion at 1600 °C using mixture 1. Only the  $\beta$ -SiC phase was identified by X-ray diffraction in the composite, as is shown in Figure 10. In the energy dispersive spectroscopy analysis, the converted composite shows only the peaks corresponding to silicon and carbon, as illustrated in Figure 11.

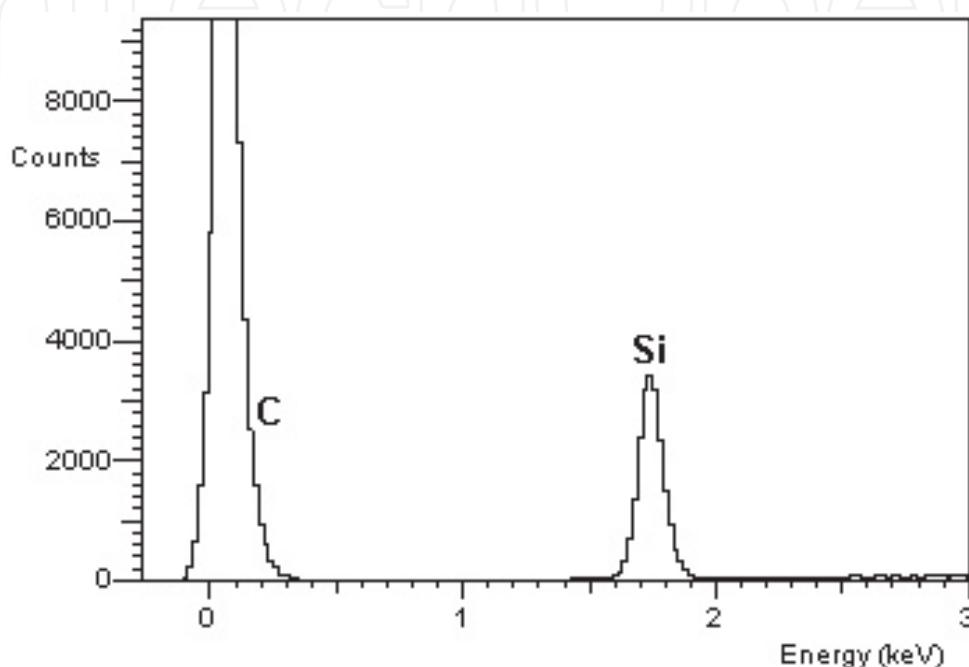


Fig. 11. Elemental analysis using energy dispersive spectroscopy in the SiC<sub>f</sub>/SiC composite processed at 1400 °C, showing the presence of Si and C.

Since the conversion method utilizing the Mixture 2 was carried out at a lower temperature, and avoided the contamination by aluminum with a mixture containing only the powders of SiO<sub>2</sub> and Si, this process was chosen to obtain samples of SiC<sub>f</sub>/SiC in order to carry out a study using the plasma torch test.

### 5.3 Plasma torch test

Figure 12 shows the sequence of the test procedure performed in plasma torch.

Figure 13 shows the mass variation versus time of exposure to the plasma torch. The temperature of the plasma attack was 1450 °C with a distance of 8 cm between the nozzle and the sample. The larger decrease in mass of the composite, the greater was the exposure time of the plasma.

As exposure time increases a reduction in the mass of the composite occurs. This mass variation is associated with mechanical erosion caused by the flow of plasma over the surface of the composite, but this value is low compared to the total mass. The mass was decreased by 0.017% per 100 seconds.

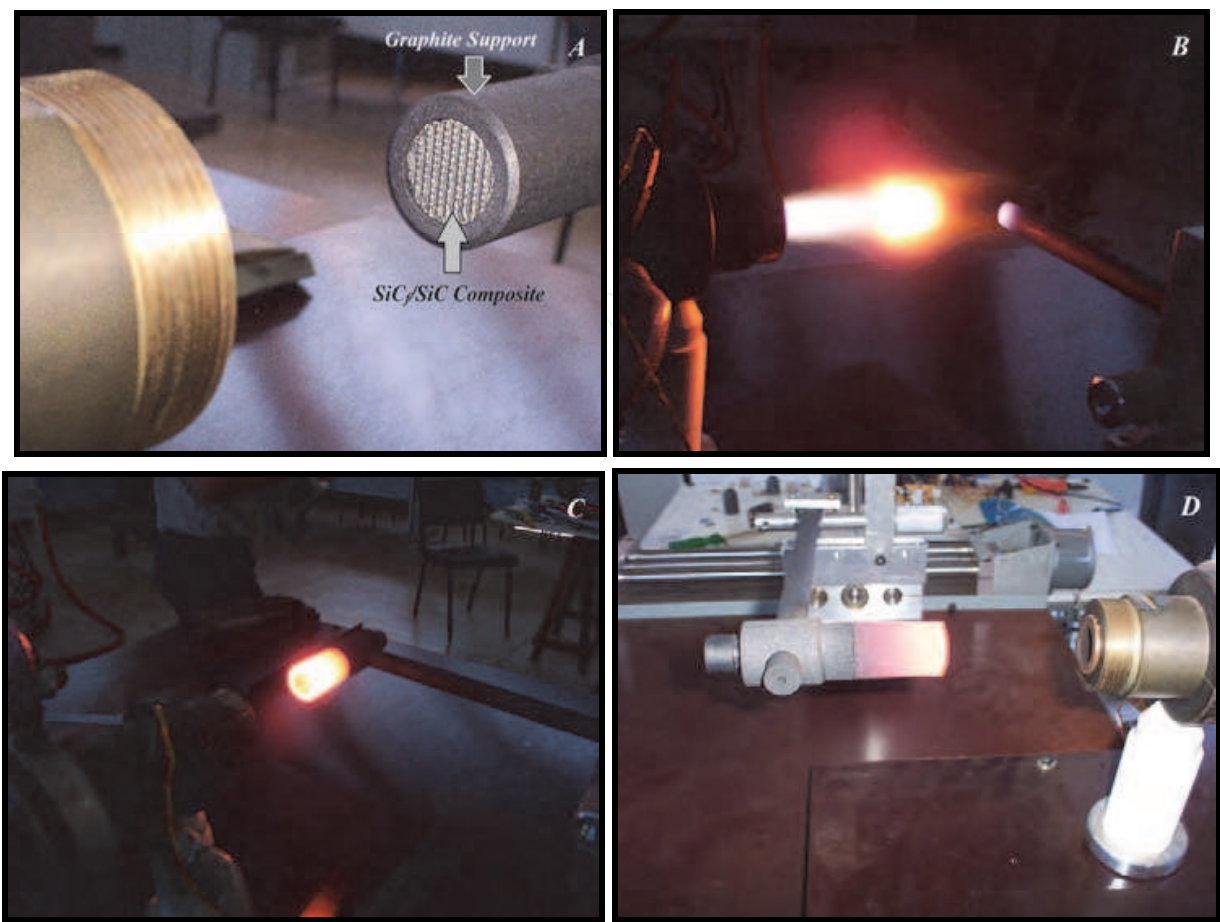


Fig. 12. (a) View of the SiC<sub>f</sub>/SiC composite before the plasma attack, (b) Beginning of the plasma attack on the SiC<sub>f</sub>/SiC composite, (c) End of plasma attack, the SiC<sub>f</sub>/SiC composite and (d) Cooling support assembly of SiC<sub>f</sub>/SiC composite and graphite.

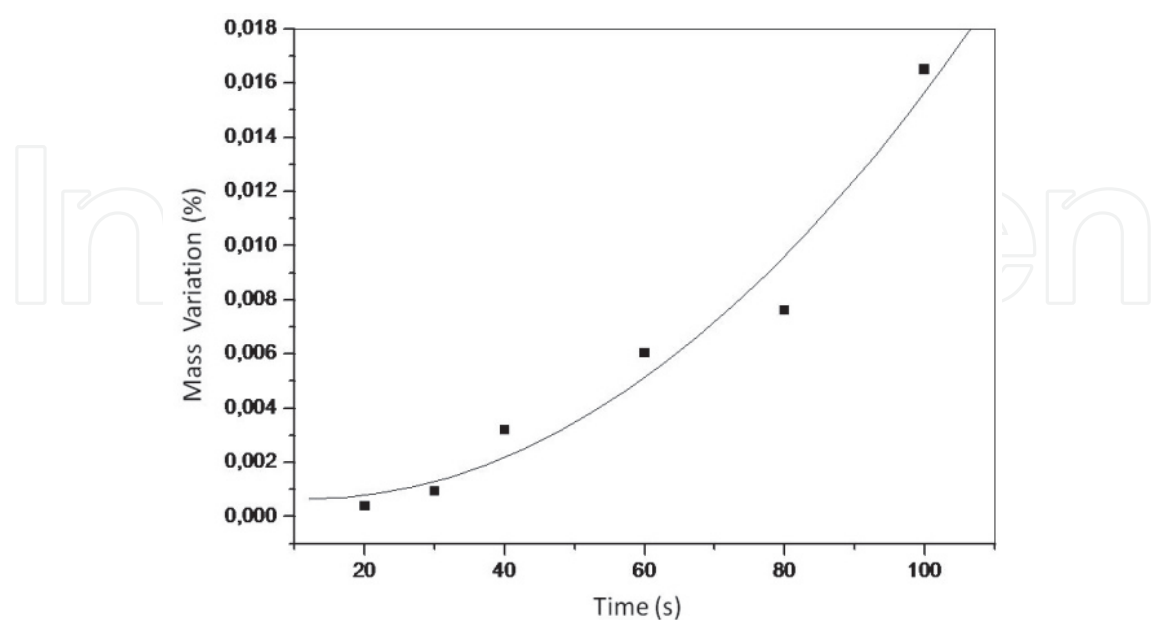


Fig. 13. Mass variation of the SiC<sub>f</sub>/SiC composite as a function of exposure time to plasma at a temperature of 1450 ° C.

Figure 14 shows an overview of the surface of the SiC<sub>f</sub>/SiC composite before and after the plasma attack.

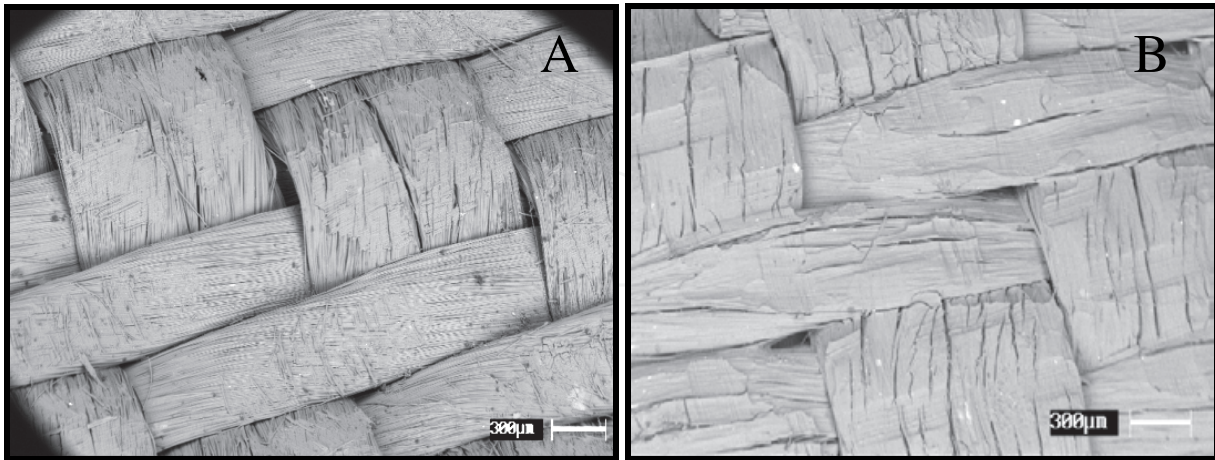


Fig. 14. (a) General view of the SiC<sub>f</sub>/SiC composite before the plasma attack and (b) General view of the SiC<sub>f</sub>/SiC composite after a 60 seconds plasma attack.

It can be observed that the composite showed no microstructural difference, keeping the same arrangement and morphology of the fibers before and after the plasma attack. In Figure 15, it is possible to see that in the fractured surface, after the plasma attack, only the ends of the fibers that were exposed SiC are oxidized with a glassy aspect, with the formation of SiO<sub>2</sub>.

This is possible because at temperatures above 600 °C, SiC begins to suffer oxidation, transforming itself into SiO<sub>2</sub>, as shown in Equation 12 (Opila & Jacobson, 1995). This new phase forms a thin film on the surface, which acts as a protective layer in the composite, preventing further penetration of oxygen into the composite avoiding higher oxidation of the composite. This can be observed by X-ray diffraction carried out on the matrix surface after the plasma attack, indicating the presence of both the β-SiC phase and the SiO<sub>2</sub> phase, as shown in Figure 16.

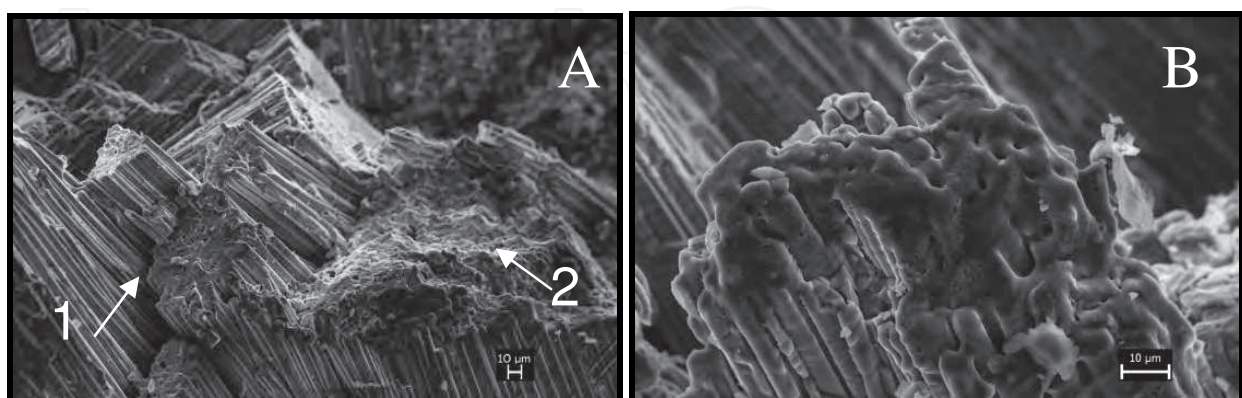


Fig. 15. View of the fiber bundles attacked by the plasma beam. (1): Region oxidized with formation of SiO<sub>2</sub> and (2): Region fractured by mechanical erosion. (b) Detail of a fiber bundle attacked by the plasma beam.

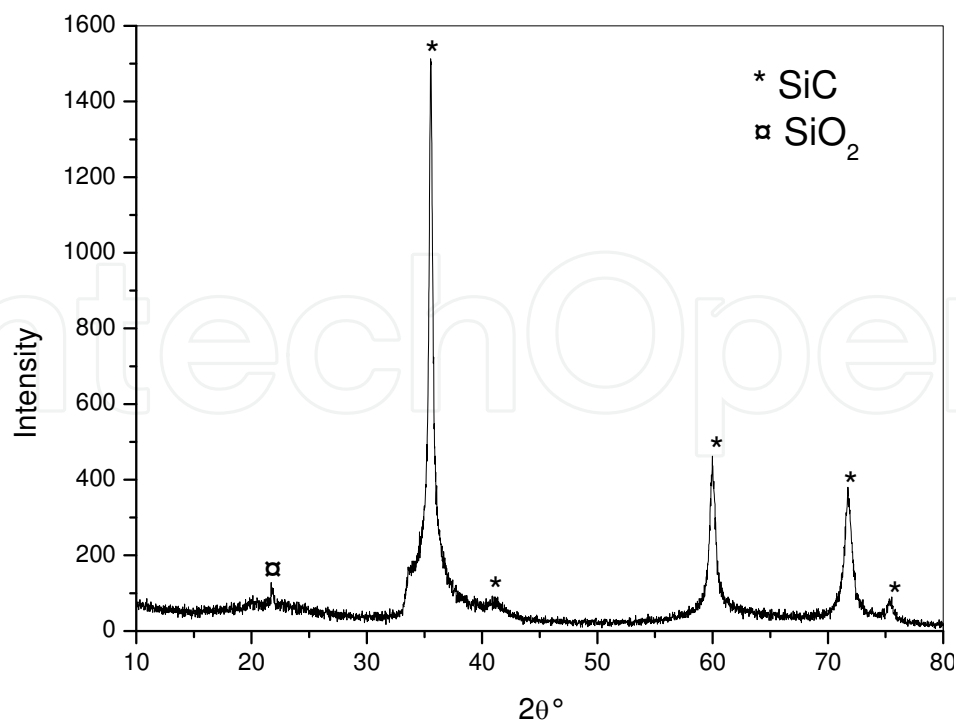


Fig. 16. X-ray diffractogram of the SiC<sub>f</sub>/SiC composite after plasma attack.

## 6. Conclusion

1. It is possible to obtain SiC<sub>f</sub>/SiC composite by conversion reactions at high temperatures, starting from C/C composite, by the reaction between carbon and SiO<sub>(g)</sub> from pack mixtures composed of 60 SiC + 30 Al<sub>2</sub>O<sub>3</sub> + 10 Si and 50 Si + 50 SiO<sub>2</sub> (wt%). Using the mixture 50 + 50 Si SiO<sub>2</sub>, the appropriate temperature for conversion is 1400 °C, lower compared to the former 1600 °C, which produces a composite with fibers of a fine texture, with submicrometric grains and purity of the β-SiC phase.
2. The method makes it possible to obtain CVR composite SiC<sub>f</sub>/SiC with the same microstructure of C/C precursor and avoids any dimensional variation or changes in the original distribution voids.

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## 8. References

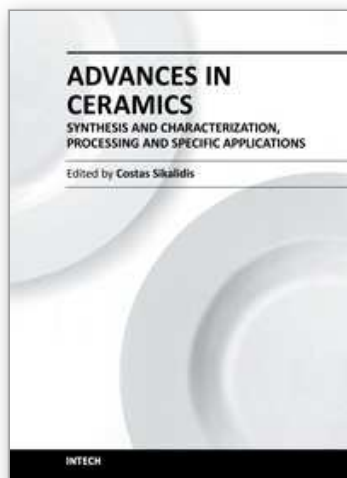
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