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Spectroscopic properties of carbon fibre reinforced silicon carbide composites for aerospace applications

Davide Alfano
*Italian Aerospace Research Centre
Italy*

1. Introduction

The use of Ceramic Matrix Composites (CMCs) such as carbon fibre reinforced silicon carbide composites (C/SiC), carbon fibre reinforced carbon composites (C/C) but also silicon carbide fibre reinforced silicon carbide composites (SiC/SiC) is mandatory within the aerospace sector whenever the transfer of mechanical loads at high temperatures (up to 1900 K in air) is required and any metallic material (e.g. refractory metals) or intermetallic materials cannot be employed.

CMCs are constituted by the coupling of long fibres reinforcement (usually carbon fibres) and a refractory ceramic matrix (i.e. carbon, or silicon carbide) and represent a class of ceramic materials characterized by good mechanical properties, thermo-mechanical stability and fracture toughness: their fracture behaviour sets them apart from conventional monolithic ceramics, allowing for a variety of uses in which damage tolerance is the main requirement.

The extremely good high temperature fracture toughness of CMCs is provided by the crack bridging effect of the carbon fibres: stress concentrations, e.g. notches or holes, are reduced by stress redistribution and inelastic deformation. In case of overloading, monolithic ceramics break immediately, while CMC materials are still able to carry load even if the elastic mechanical load range is exceeded. Such a damage tolerant behaviour constitutes an important point for the safety issues in particular for space re-entry vehicles.

Typical applications for CMCs within aerospace sector include thermo stable satellite structures for optical components, ultra lightweight scanning mirrors (Harnisch et al., 1998), hot structures for atmospheric re-entry vehicles (Laux et al., 2001), and very high temperature parts for hypersonic and rocket engines (Mühlratzer & Leuchs, 2001 Naslain & Schneider, 2002).

One of the most relevant application fields of carbon fibre reinforced silicon carbide composites (C/SiCs) concerns the manufacturing of structural thermal protection systems (TPSs) for planetary re-entry vehicles, where good thermo-mechanical properties at high temperature associated with oxidation resistance are required. During the re-entry phase in Earth's atmosphere nose tips and leading edges are the structural parts of spacecrafts subject

to high heat fluxes and then high temperatures which, in any case, depend on re-entry trajectory as well as the shape and dimension of the structural part.

The design of shape and dimensions of that element must consider a lot of parameters such as the temperatures, pressures and thermal fluxes reached at the stagnation point, but also surface properties. In particular, if the stagnation pressure depends on the re-entry trajectory, the surface temperature, and the heat flux are also greatly influenced by the emissivity and recombination efficiency (catalycity) values of the employed material. Consequently the choice of the material is not only the starting point for the design of TPSs but it is also a strategic point since it can strongly influence the value of a series of parameters that define the re-entry conditions.

This chapter aims to present the results of emissivity and catalytic measurements carried out reproducing the experimental conditions, in terms of pressure and temperature values, typical of a re-entry phase in atmosphere for a carbon fibre reinforced silicon carbide composite produced by polymer vapour infiltration and then coated with SiC by chemical vapour deposition. In particular, the tested samples are produced by MT Aerospace and marketed under the brand name Keraman®. Moreover microstructural investigations performed on post-test samples are illustrated in order to discuss about the resistance to oxidation processes of C/SiC in terms of active and passive oxidation of silicon carbide.

2. Aerospace applications for silicon carbide

Silicon carbide is defined by the Engineered Materials Handbook (Reinhart, 1987) as “reinforcement, in whiskers, particulate, and fine or large fibre, that has application as metal matrix reinforcement because of its high strength and modulus, density equal to that of aluminium, and comparatively low cost”. For aerospace applications SiC can be employed as matrix in reinforced composites (CMCs) or as particulate filler in massive ceramic composites also indicated as Ultra High Temperature Ceramics (UHTCs).

2.1 SiC-based Ceramic Matrix Composites

Silicon carbide is often combined with carbon fibres in order to obtain Ceramic Matrix Composites (CMCs) with non-oxide matrix materials for high temperature applications. Carbon fibres show no degradation up to temperatures over 3000 K in non oxidizing atmosphere. So that if carbon fibres are protected from oxidation reactions, they become a useful material in space vehicle applications where temperatures up to 2000 K occur (for instance during the re-entry phase in atmosphere). The main role of SiC matrix in CMCs is to protect the carbon fibres from oxidation processes, which already become active starting from about 800 K, by the formation of a protective silica-based glassy layer. SiC can be also employed as protective coating in the case of carbon fibre reinforced carbon composites (C/C). Reinforced Carbon-Carbon (RCC) have been used as Thermal Protection System (TPS) for wing leading edges and nose cap of the Space Shuttle where the temperature values can increase up to 1800-1900 K during the re-entry phase in atmosphere. In order to provide oxidation resistance for reuse capability, the outer layers of the RCC are converted to silicon carbide.

The Institute of Structures and Design of DLR (German Aerospace Centre) has been developing fibre reinforced ceramic matrix composites via the liquid silicon infiltration process for more than a decade. The materials manufactured using this processing technique

are suitable for a broad range of applications. In general, this material exhibits excellent thermal shock resistance, high mass specific values and possess a dense matrix. In the framework of the project EXPRESS (realised within the scope of a German-Japanese cooperation), DLR developed and produced the tile called CETEX (Ceramic Tile Experiment) which is a fibre reinforced ceramic component made of C/C-SiC, a special ceramic material, applying the liquid silicon infiltration process. CETEX was attached to the stagnation point of a capsule's ablative heat shield. Qualification tests with samples and components took place in several plasma wind tunnels: in the PWK 2 wind tunnel of the Institute for Space Systems of the University of Stuttgart the maximum applied temperature was reportedly 3000 K. Although the Japanese launcher did not work as intended, the (uncontrolled) re-entry of the capsule was successful, leading to about 2500 K in CETEX without apparently revealing any problems (Hald & Winkelmann, 1995; Hald & Winkelmann, 1997; Hald, 2003).

MT Aerospace (Augsburg, Germany) produces a carbon reinforced silicon carbide for aerospace applications and markets it under the name Keraman®. This materials was qualified during NASA X-38 project, in the form of CMC body flaps and leading edges for the X-38 vehicle (Coperet et al., 2002; Dogigli et al., 2002a,b; Pfeiffer & Peetz, 2002; Steinacher et al., 2007). On the same space vehicle the nose cap was manufactured in C/SiC provided by DLR. Nose cap and body flaps were produced by a Chemical Vapour Infiltration process (CVI). Also Snecma Propulsion Solide (Bordeaux, France) has developed a CMC shingle TPS (Pichon et al., 2006); a flat panel was tested in arc-jet reaching the maximum temperature of 1500 K.

In the frame of the European eXPERimental Re-entry Testbed (EXPERT) project conducted by the European Space Agency (ESA), SiC-coated C/C composite manufactured by DLR has been chosen as material to produce the nose cap of the vehicle whose goal will be to collect data of different physical phenomena during the re-entry phase in atmosphere (Reimer & Laux, 2005).

A load bearing aeroshell in C/SiC for hypersonic flight was developed in the project Sustained Hypersonic Flight Experiment (SHyFE) financed by UK Ministry of Defence (Dadd et al., 2006; Goodman & Ireland, 2006). The aeroshell was fabricated by MT Aerospace utilizing CVI.

In the framework of the Sharp Hot Structures project, a technology project within the Italian Unmanned Space Vehicle (USV) program, the Italian Aerospace Research Centre (Capua, Italy) has studied and developed, during last ten years, a nose cone ceramic demonstrator for re-entering Low Earth Orbit (LEO) space vehicles, whose structure is divided into a conical/hemispherical part (nose tip) manufactured in massive UHTC and a layered conical part (dome) in C/SiC (Russo & Marino, 2003; Scatteia et al., 2005; Del Vecchio et al., 2006). Moreover a ceramic composite containing SiC particles dispersed in a ZrB₂ matrix was deposited on the dome surface by plasma spraying in order to improve its oxidation resistance at high temperature (Bartuli et al., 2002; Tului et al., 2006).

2.2 SiC as additive for Ultra High Temperature Ceramics

Ceramic compounds based on metal borides, such as zirconium diboride (ZrB₂) and hafnium diboride (HfB₂) have been commonly referred to as Ultra High Temperature Ceramics (UHTCs). UHTCs represent a class of promising materials for use in extreme applications because of their high melting point and relatively good oxidation resistance in

re-entry conditions. UHTCs are characterized by high melting temperatures (ZrB_2 3518 K, HfB_2 3653 K), solid state stability, good thermo-chemical, and thermo-mechanical properties (Schneider, 1991). These extremely promising high performance materials are also characterized by hardness above 20 GPa, high wear resistance, high emissivity, high electrical conductivity, excellent corrosion resistance, and good thermal shock resistance (Mroz, 1994; Fahrenholtz et al., 2007a). Leading applications are currently found in aerospace, more specifically in the possibility to employ them to realize sharp-shaped hot structures like wing leading edges and nose caps able to withstand the severe thermal requirements of next generation of hypersonic re-entry vehicles. The highly thermal demanding trajectories foreseen for future spaceplane-like winged re-entry vehicles dictate the need for base materials able to sustain operating temperatures approaching 2500 K, to resist evaporation, erosion and oxidation in the harsh re-entry environment.

The research on this class of materials began in the 60's in the frame of Air Force contracts (Kuriakose, & Magrave, 1964; Tripp & Graham, 1971). The early works were devoted to the production of dense materials by mean of pressure assisted sintering, and to investigate the influence of a variety of additives, including carbon and silicon carbide, on the processing and oxidation resistance of Hf and Zr diborides. These works showed that the addition of SiC as secondary reinforcing phase provides significant enhancements to the oxidation resistance of UHTCs (Tripp, et al., 1973). Moreover the SiC addition was also found to improve the processing by lowering sintering temperatures (Monteverde et al., 2003; Chamberlain et al., 2004; Monteverde, 2006). Then, when combined with SiC, ZrB_2 and HfB_2 -based composites exhibit indeed excellent refractoriness, high oxidation resistance, and are as such good potential candidates for the above-mentioned application. An important parameter such as the upper limit of the service temperature is strongly related to the characteristics of secondary phases. For example, above 1500 K the oxide scale formed in air on the surface of pure MB_2 , with $\text{M}=\text{Zr}$ or Hf , is unstable and non-protective due to intensive volatility of B_2O_3 (Kuriakose, & Magrave, 1964; Tripp & Graham, 1971; Opeka et al., 1999; Ban'kovskaya & Zhabrev, 2005; Chamberlain et al., 2005; Fahrenholtz, 2005; Fahrenholtz et al., 2007) while the SiC-containing MB_2 showed enhanced resistance to oxidation up to 1900 K. In fact for temperature higher than 1500 K, the addition of SiC promotes, on the exposed surface, the formation of borosilicate glass which gives much more oxidation protection than B_2O_3 alone. Several studies have dealt with the thermal stability and physical properties of ultra-refractory MB_2 -based ceramics in oxidizing environments, and highlighted the role of composition and microstructure on the mechanisms governing the materials response to hostile environments (Opeka et al., 1999; Levine et al., 2002; Fahrenholtz et al., 2004; Gasch et al., 2004; Opeka et al., 2004; Opila et al., 2004; Ban'kovskaya & Zhabrev, 2005; Chamberlain et al., 2005; Monteverde & Bollosi, 2005; Rezaire et al., 2006; Fahrenholtz, 2007b; Rezaire et al., 2007; Han et al., 2008; Zhang et al., 2008; Carney et al., 2009; Hu, et al., 2009; Karlsdottir & Halloran, 2009).

NASA started in 1990 a research program on UHTCs and ended up in 1997 and 2000 demonstrating the use of ZrB_2 and HfB_2 for sharp leading edge in the Sharp Hypersonic Aero-thermodynamic Research Probe Ballistic experiments (SHARP-B1 and B2) (Rasky et al., 1998). UHTCs were also tested by the flights of Delta Clipper (DC- X and DC-XA) in order to evaluate their potential application on new entry vehicles (Smith et al., 1997).

During the 90s a wide range research activity on UHTC materials was conducted in Italy, mainly by the Italian National Research Council Institute of Ceramic Materials (CNR-

ISTEC). The CNR-ISTEC investigated new processing routes based on pressure assisted sintering, on the adoption of sintering aids and secondary reinforcing phases in order to obtain dense bodies characterized by superior oxidation resistance and mechanical properties.

Since 2000, the Italian Aerospace Research Centre (CIRA) has studied, developed, and tested massive UHTCs in the frame of the Unmanned Space Vehicle (USV) National Program (Russo & Marino, 2003; Savino, et al., 2005; Scatteia et al., 2005; Del Vecchio et al., 2006; Monteverde & Scatteia, 2007; Monteverde et al., 2008; Scatteia et al., 2010).

The poor fracture toughness of UHTCs can be still considered the main limitation of this class of materials for aerospace applications. In these last years the activities of several research groups on UHTCs have been focused on the improvement of the fracture toughness by using SiC whiskers or SiC chopped fibers as reinforcing adds (Chen et al., 2009; Zhang et al., 2009; Guicciardi et al., 2010; Silvestroni et al., 2010).

3. C/SiC employed in emissivity and catalycity tests

Emissivity and catalycity tests were carried out on a SiC-coated two-dimensional C/SiC ceramic matrix composite produced by MT Aerospace and marketed under the name Keraman® (Fig.1a).

The C/SiC composite is produced by Polymer Vapour Infiltration process (PVI) while the SiC-coating, applied by Chemical Vapour Deposition method (CVD), is characterised by a thickness of about 25 μm . In Fig. 1b one example of cross-section SEM micrograph used to estimate the SiC-coating thickness is reported. In the same image the two orthogonal directions of carbon fibres are also evident.

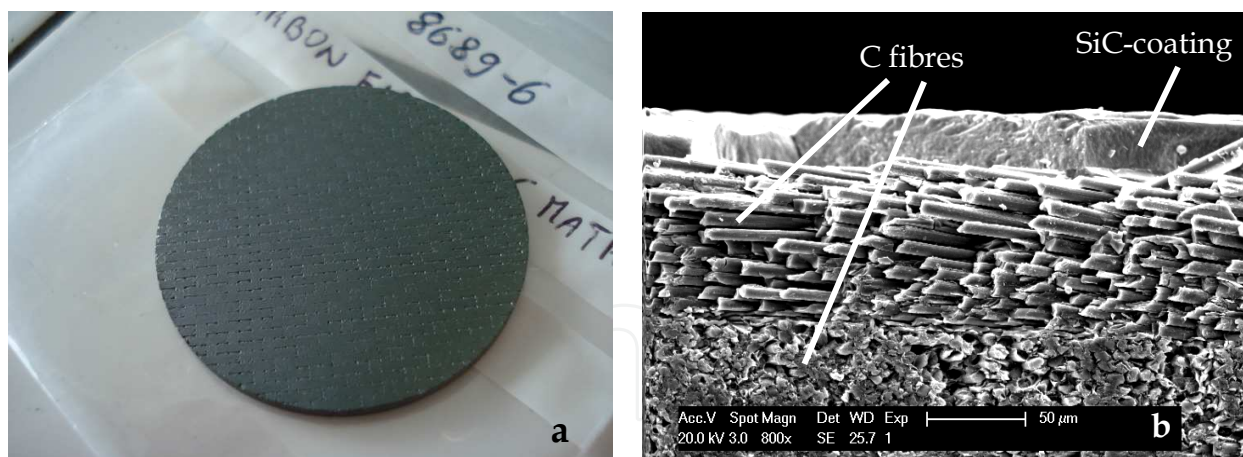


Fig. 1. CVDed SiC coated C/SiC: a) picture of specimen employed to perform emissivity and catalycity tests, b) cross-section SEM micrograph of a specimen wherein SiC-coating and 2D orthogonal carbon fibres are indicated.

4. Emissivity

The heat transfer by radiation from the surface of such space vehicle becomes a significant part of the total heat transferred when the surface temperature is high, and when the convective heat transfer is low as at high altitudes. Since radiative heat transfer is an

important method of cooling under such conditions, a knowledge of the emissivity values of a surface is required whenever theoretical simulations involving radiant heat are to be made. At each given temperature and wavelength, there is a maximum amount of radiation that a surface can emit which is known as a blackbody radiation, and can be theoretically predicted by Planck's law. However, most surfaces are not blackbodies, and emit some fraction of the amount of thermal radiation that a blackbody would. This fraction is known as *emissivity*. Then emissivity of a body is the rate between the energy emitted and an ideal emitter or blackbody at the same temperature. Hence emissivity (ϵ) may be expressed as follows:

$$\epsilon(\lambda, \theta, \varphi, T) = i_s(\lambda, \theta, \varphi, T) / i_b(\lambda, T) \quad (1)$$

where $i_s(\lambda, \theta, \varphi, T)$ is the energy emitted by the sample per unit time, per unit area, per solid angle per wavelength interval, at temperature T , and $i_b(\lambda, T)$ (given by the Planck's law) is the energy emitted by a blackbody per unit time, per unit area, per solid angle, per wavelength interval at the same temperature T of the sample. $\epsilon(\lambda, \theta, \varphi, T)$ is known as *spectral directional emissivity*. By integrating over the angle variables θ and φ one can define the *spectral hemispherical emissivity* while integrating only on wavelength the *total directional emissivity* is obtained. *Total hemispherical emissivity* (ϵ^0) is obtained by integrating both on angle variables and on wavelength range.

4.1 Experimental set-up for emissivity measurements on C/SiC samples

Total hemispherical emissivity, as a function of temperature, have been measured in the range 1000-1800 K with the *Moyen d'Essai et de Diagnostic en Ambiance Spatiale Extrême* (MEDIASE) set-up developed at PROMES-CNRS laboratory in France (Paulmier et al., 2001; Balat-Pichelin et al., 2002; Paulmier et al., 2005).

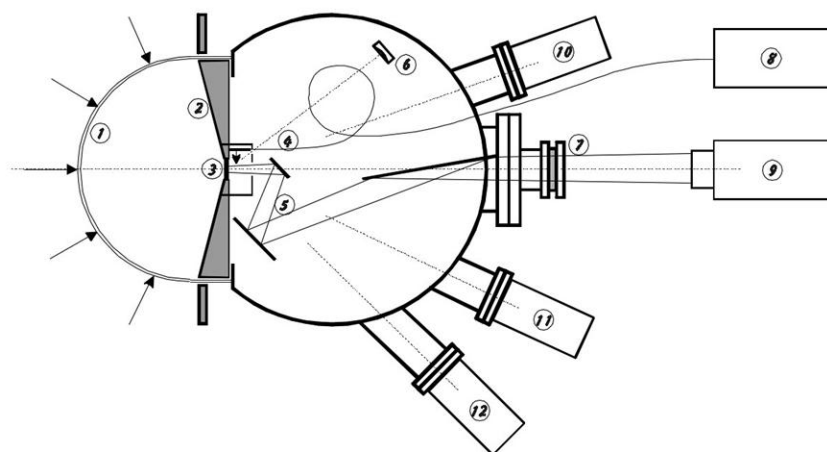


Fig. 2. MEDIASE test facility: 1) hemispherical silica window, 2) water-cooled front shield and sample-holder, 3) sample, 4) optical fibre, 5) 3-mirrors goniometer, 6) quartz crystal microbalance, 7) viewport, 8) pyro-reflectometer, 9) radiometer, 10) UV source position, 11) and 12) ion gun positions.

The device in Fig. 2 is composed of a vacuum chamber equipped with a hemispherical silica glass window (35 cm in diameter) for solar irradiation, a water-cooled front shield surrounding a sample holder. The specimen, in our experiments 40 mm diameter and 2 mm thick, is heated by concentrated solar energy at the focus of the 1 MW solar furnace. On the back face of the sample, total (wavelength range 0.6-40 μm) directional (0 to 80° by 10° step) radiance is measured by mean of a moving three-mirrors goniometer that collects the radiation emitted from the sample at different angles. The total directional radiance $i_s(\theta, \varphi, T)$ is measured with a radiometer calibrated against a reference blackbody. The total directional emissivity is then given by:

$$\varepsilon(\theta, \varphi, T) = i_s(\theta, \varphi, T) / i_b(T)$$

(2)

where $i_b(T)$ is the theoretical blackbody radiation at temperature T . The surface temperature is measured with a pyro-reflectometer, developed at PROMES-CNRS laboratory (Hernandez, 2005), collecting radiation from the centre of the sample. The total hemispherical emissivity (ε^h) is readily obtained by angular integration of the directional ones. Emissivity experiments have been performed in air at the pressure of 4 and 200 Pa.

4.2 Emissivity values of C/SiC samples

Emissivity values of C/SiC composites obtained in the temperature range 1000-1800 K and at 4 and 200 Pa are listed in Table 1 and plotted in Fig. 3 (Alfano et al., 2009).

Total Pressure [Pa]	Temperature [K]	Total Hemispherical Emissivity (ε^h) (0.6-40 μm)
200	1019±53	0.57±0.03
	1220±37	0.71±0.04
	1391±40	0.73±0.04
	1627±59	0.73±0.04
	1695±66	0.72±0.04
	1897±92	0.74±0.04
4	1114±41	0.59±0.03
	1220±32	0.69±0.03
	1417±38	0.71±0.04
	1620±68	0.72±0.04
	1714±68	0.70±0.03

Table 1. Values of total hemispherical emissivity measured at 4 and 200 Pa on CVDed SiC coated C/SiC samples produced by MT Aerospace (from Alfano et al., 2009).

In the examined temperature range, the observed differences between the emissivity values measured at 4 Pa and at 200 Pa lie in the experimental uncertainty. In both cases the emissivity values show a similar trend increasing from ~0.6 to ~0.7 in the temperature range 1000-1300 K then staying almost constant in the temperature range 1300-1800 K with an average value of about 0.7.

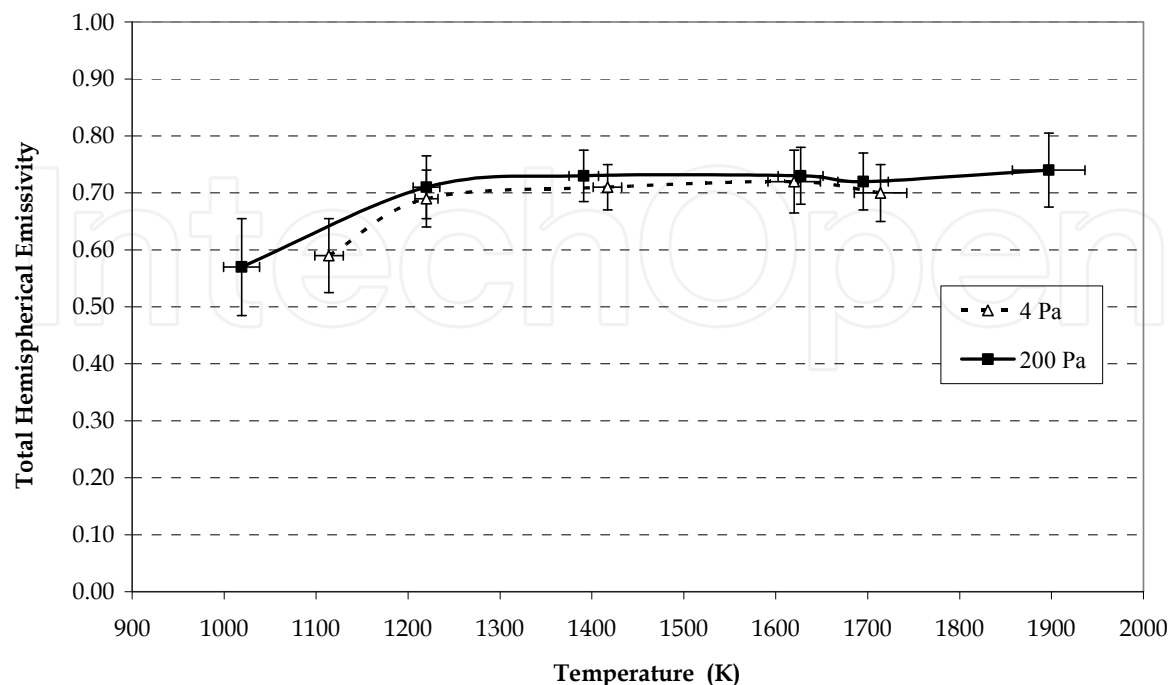


Fig. 3. Total hemispherical emissivity values of CVDed SiC coated C/SiC samples measured at 4 Pa (open triangles) and at 200 Pa (filled squares) from Alfano et al., 2009.

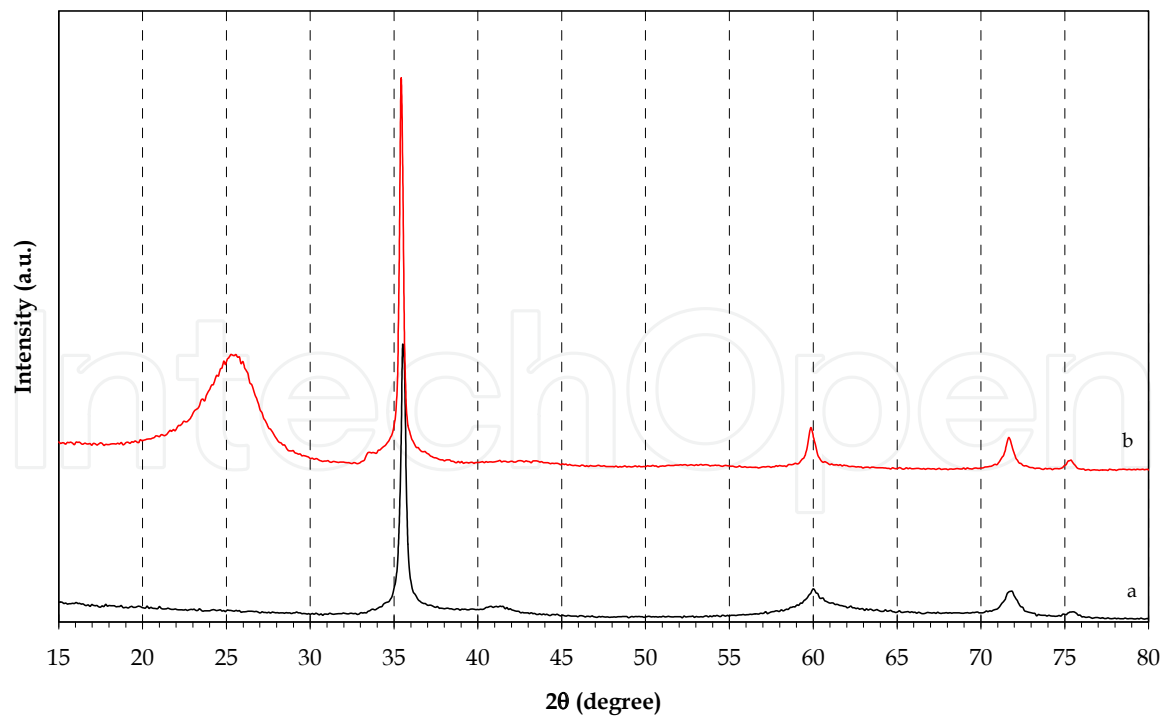


Fig. 4. X-ray diffraction patterns of the CVDed SiC coated C/SiC sample a) before and b) after the emissivity measurements performed at 200 Pa.

In Fig. 4 X-ray diffraction (XRD) patterns of the pristine specimen and tested one in the MEDIASE set-up are compared. In the XRD pattern of the tested sample the presence of the peak at $2\theta=26.2^\circ$ related to the reflection [101] of quartz (SiO_2) is evident as well as the characteristic signals of $\beta\text{-SiC}$ at $2\theta=35.5^\circ$, 60.0° , 71.9° , and 79.9° related to [111], [220], [311], and [222] reflections, respectively. SiO_2 is the result of oxidation processes occurring during the emissivity measurements.

At the testing conditions the oxide layer is not thermodynamically stable: actually, the partial pressures of both gaseous SiO_2 and SiO are not negligible. In fact, as Heuer and Lou theoretically demonstrated by means of the volatility diagrams of $\beta\text{-SiC}$ (Heuer & Lou, 1990), at 1800 K and at total air pressure of 200 Pa silicon carbide exhibits active oxidation with formation of oxides SiO and CO . This result is confirmed by several experimental data obtained by tests performed under standard air on massive and CVDed $\beta\text{-SiC}$: at 200 Pa the threshold temperature for the transition passive/active oxidation of CVDed samples is at about 1600-1700 K (Balat et al., 1992; Balat, 1996; Morino et al., 2002; Charpentier et al., 2010). By Fig. 5, wherein the optical picture of the post test sample is illustrated, the surface corrosion is evident in particular if compared to the picture of the virgin specimen shown in Fig. 1a. These observations are also confirmed by SEM analysis: by comparing between the SEM cross-section micrograph carried out on the tested sample and shown in Fig. 6a to the analogous SEM image recorded on the pristine specimen and reported in Fig. 1b, the absence of the SiC-coating after emissivity tests becomes evident. Moreover SEM analysis also confirms that the fraction of SiO_2 remaining on the sample surface and detected by XRD measurements is not thermodynamically stable since a uniform protective silica-based glassy layer has not been observed.



Fig. 5. Picture of the emitting surface of the sample CVDed SiC coated C/SiC after the emissivity experiment carried out at air pressure of 200 Pa increasing the temperature from 1000 K up to about 1900 K.

In Fig. 6b fibre cracks can be observed probably due to high temperatures reached during the test and expansion mismatch between the SiC matrix and the carbon fibers. Therefore during emissivity tests, the sample surface undergoes ablation process due to formation of SiO and CO by active oxidation of SiC and contemporary partial evaporation of SiO_2 .

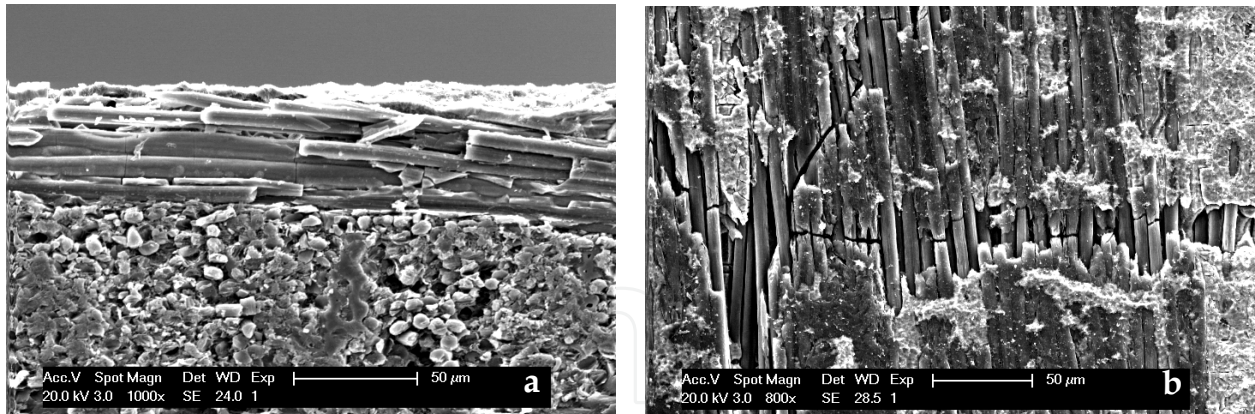


Fig. 6. SEM micrographs of a) cross-section and b) surface of the sample CVDed SiC coated C/SiC after the emissivity experiment carried out at air pressure of 200 Pa increasing the temperature from 1000 K up to about 1900 K.

5. Catalytic efficiency (catalycity)

Catalycity could be defined as the catalytic efficiency shown by a material with respect to the recombination on the surface of atomic species due to the chemical reactivity of unsaturated valences of surface atoms.

The vehicle entering in a planet atmosphere by hypersonic trajectory creates a shock wave leading to very high temperatures. The created excited species (ions, atoms, molecules, electrons) diffuse in the boundary layer and react with the materials of the vehicle. Atomic oxygen (major species) and nitrogen, present for terrestrial entries, can strike the surface of the vehicle and recombine to form molecular species (O_2 , N_2 , NO) by exothermic reactions which have the effect to produce an increase of the surface temperature and to damage the integrity of the material. Then during the phase of development of a material as TPS, the contribution due to the recombination of atomic species on surfaces to estimation of thermal fluxes must be taken into account to predict heat rates on the hot parts of a re-entry spacecraft (Scott, 1985; Carleton & Marinelli, 1992; Barbato et al., 2000). Furthermore considering that atomic recombination reactions are typical surface reactive processes usually described by means of models for heterogeneous catalysis (Kovalev & Kolesnikov, 2005), the surface molecular structure and the surface morphology of structural part of aerospace vehicle involved in atomic recombination processes play a major part on the catalytic efficiency.

5.1 Experimental set-up to evaluate the atomic oxygen recombination coefficient of C/SiC samples

The catalytic efficiency of the material for the recombination of atomic oxygen was studied by means of a direct method for the measurement of the recombination coefficient γ_O , defined as the ratio of the flux of atomic oxygen that recombines on the surface to that of the total atomic oxygen impinging the surface of the sample. The *Moyen d'Essai Solaire d'Oxydation* (MESOX) experimental set-up, developed at French PROMES-CNRS laboratory, was used for this kind of experiments. In this apparatus, the atomic oxygen recombination coefficient γ_O is determined by measuring the concentration gradient of atomic oxygen in

proximity of the sample surface by means of actinometry and Optical Emission Spectroscopy (OES) techniques.

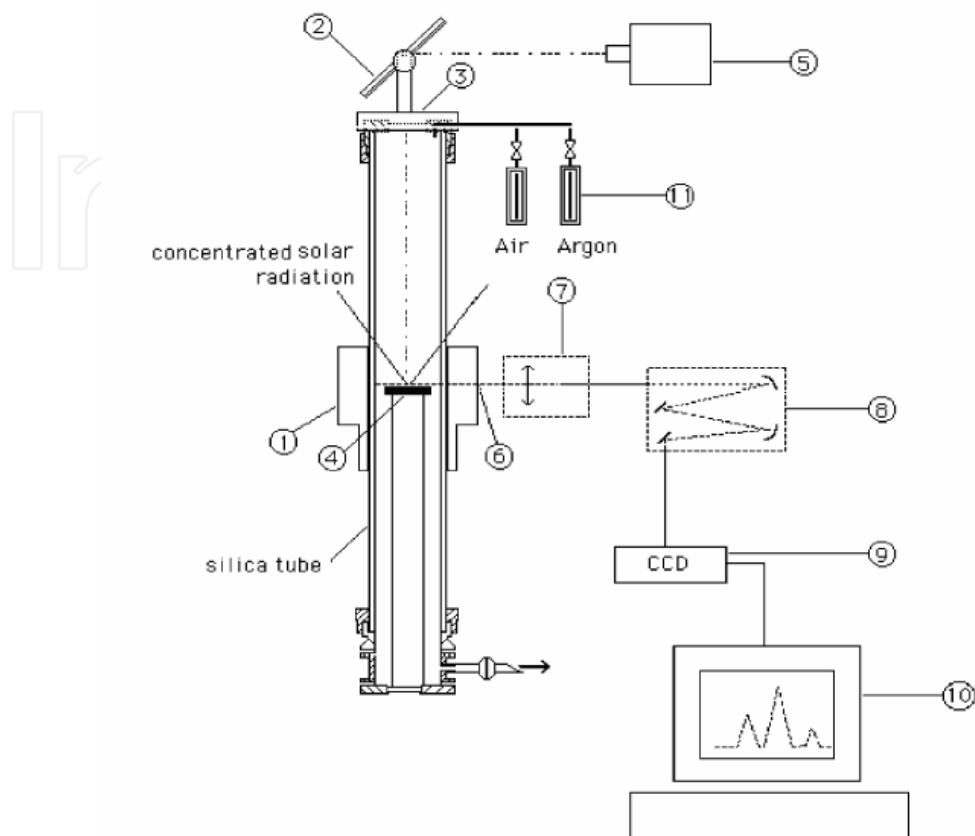


Fig. 7. MESOX set-up for optical emission spectroscopy measurements :1) waveguide, 2) mirror, 3) viewport, 4) sample, 5) pyrometer, 6) aiming slit, 7) lens, 8) spectrometer, 9) CCD-3000, 10) computer, 11) mass flowmeters.

The MESOX apparatus, shown in Fig. 7, is described in detail elsewhere (Balat et al., 2003): here we will only outline its main features. The ceramic composite sample (25 mm diameter and 2 mm thickness) is put in a plasma reactor consisting of a silica tube (quartz), 50 cm length and 5 cm diameter with CaF_2 viewports. The air plasma is generated by a 300 W microwave discharge surrounding the sample. A regulator, a gauge and a vacuum pump allow the precise control of the total pressure during experiments. The airflow, coming from the top of the reactor and then pumped downward, is fixed at 4 L/h. The sample is placed on a sample-holder at the centre of the plasma discharge. The reactor is positioned in such a way to let the sample be at 25 mm above the theoretical focus of the 6 kW solar furnace equipped with a variable opening shutter. Due to this shutter, the available incident concentrated solar flux can reach 5 MW m^{-2} .

The relative atomic oxygen concentration in the reactor is determined by means of OES combined with actinometry. The actinometry technique allows to follow the relative atomic oxygen concentration profile along the discharge. A small amount of argon (5%) is introduced in the flow and the evolution of the ratio $I_{\text{O}}/I_{\text{Ar}}$, where I_{O} and I_{Ar} are the intensities of oxygen and argon emission spectral lines respectively, is measured along the

discharge zone. The transitions at 844.6 nm and at 842.4 nm are chosen for atomic oxygen and argon respectively. Under well-defined conditions, the ratio I_O/I_{Ar} is proportional to the oxygen concentration in a wide range of temperature and in a broad region of the reactor. The spectroscopic bench is composed of an optical sampling system including a lens and a mirror, and a monochromator (spectrometer Triax 550 Jobin-Yvon) equipped with an Optical Multichannel Analyzer (OMA). The spectral resolution is 0.2 nm and the spatial resolution is around 270 μm .

Once the concentration profile of the atomic oxygen has been measured, the recombination coefficient is obtained according to the relation:

$$\gamma_O = \left(\frac{\frac{I_O}{I_{Ar}} \Big|_{x=L}}{\frac{I_O}{I_{Ar}} \Big|_{x=0}} \cdot \frac{T_S}{T_L} - 1 \right) \cdot \frac{4D_{O,air}}{VL} \quad (3)$$

where $D_{O,air}$ is the binary diffusion coefficient of atomic oxygen (O) in air calculated for each gas temperature by the Chapman-Enskog theory, V the mean square velocity of atomic oxygen calculated by the gas kinetic theory (rarefied gas), L the thickness of the recombination boundary layer which enters in the diffusion-reaction model and which is measured experimentally, $(I_O/I_{Ar})_{x=L}$ and $(I_O/I_{Ar})_{x=0}$ are the measured intensities ratios at distance L from the surface and on the surface respectively, T_s the surface temperature and T_L the gas temperature at the edge of the layer L .

The uncertainties $\Delta\gamma/\gamma$ have been calculated taking into account the errors on I_O/I_{Ar} (10%) and L (5%) but also on the flow parameters: the diffusion coefficient $D_{O,air}$ and the mean square atomic velocity V . The accuracy on these two last values is due essentially to that of the gas temperature (5%), measured by OES (N_2 rotational temperature) giving a total accuracy on the recombination coefficient γ of $\pm 30\%$.

5.2 Recombination coefficient of atomic oxygen on surface of C/SiC samples

Two identical C/SiC specimens (labelled as A and B) were employed to perform catalytic tests in MESOX facilities. In Fig. 8 the logarithm of γ_O for the two samples with respect to the reciprocal of the absolute temperature is plotted in accordance with the well-known Arrhenius' equation (Arrhenius, 1889):

$$\gamma_O = A \exp(-E_a/RT) \quad (4)$$

where A is the pre-exponential coefficient, E_a is the activation energy of the atomic oxygen recombination process, R the universal gas constant, and T is the absolute temperature.

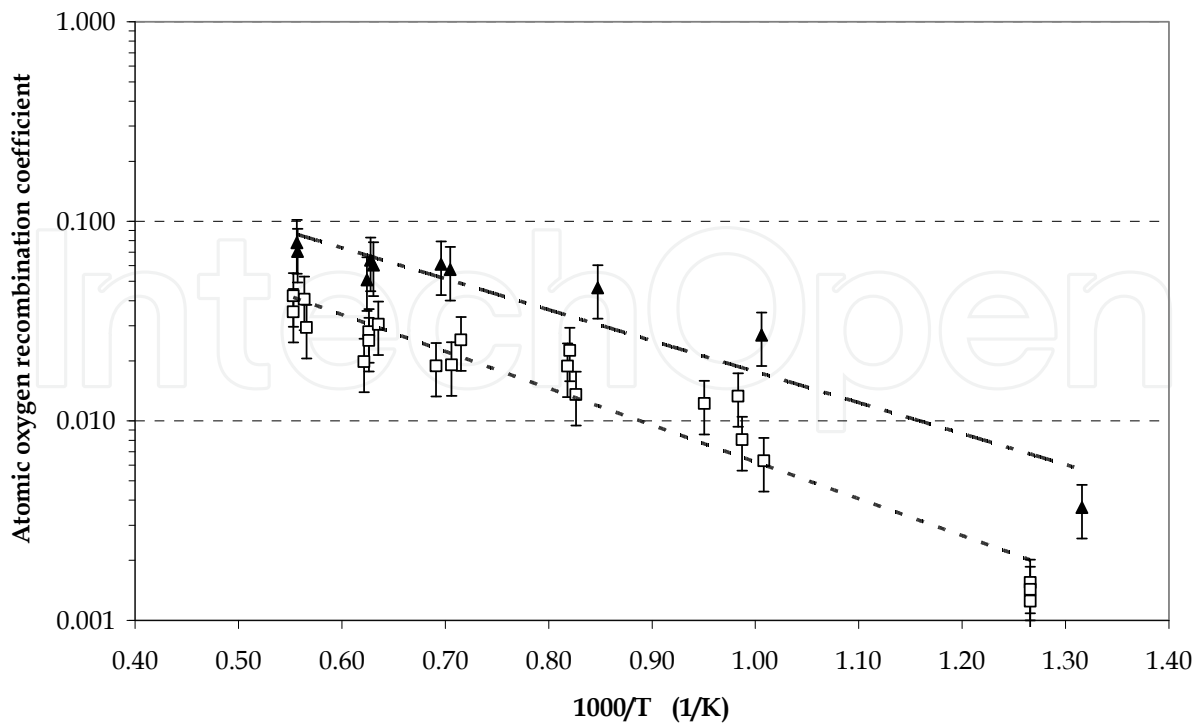


Fig. 8. Recombination coefficient for atomic oxygen versus reciprocal absolute temperature for the two CVDed SiC coated C/SiC samples A (open squares) and B (filled triangles). Both the measurements were performed at 200 Pa of total air pressure (from Alfano et al., 2009).

Both samples conform approximately to the Arrhenius law that predicts a linear behaviour in the examined temperature range. The Arrhenius parameters (A and E_a) used to fit by the equation 4 and the values of γ_0 measured at the maximum temperature reached during the tests are summarized in Table 2.

Sample	Pre-exponential coefficient (A)	E_a/R [K]	Correlation coefficient	Activation Energy [kJ/mol]	Mean γ_0 measured at 1800 K
A	0.436	4.251	0.913	35.3	0.0367
B	0.629	3.574	0.899	29.7	0.0744

Table 2. Parameters of the Arrhenius type expression (Eq. 4) used to fit the measured recombination coefficients (γ_0) plotted on Fig. 8, and mean measured values of γ_0 at 1800 K related to the two tested samples of CVDed SiC coated C/SiC shown in Fig. 9 (Alfano et al., 2009).

The values of γ_0 coefficients exponentially increase with the temperature starting from about 0.001 at 800 K to about 0.07 at 1800 K. Although the two series of γ_0 coefficients differ more for low temperatures, the fitting curves are characterised by comparable slope values confirming the reproducibility of the catalycity experiments. This behaviour could be explained considering that each tested sample has a different surface roughness which strongly depends on the manufacturing process. In Fig. 9 the pictures of the two samples manufactured by PVI and employed to perform emissivity tests are shown: the different

roughness structure of the two samples is evident by the simple macroscopic and optical examination. The activation energy for the atomic oxygen recombination process on C/SiC surface has been estimated equal to about 30 kJ/mol.



Fig. 9. Pictures of two CVDed SiC coated C/SiC specimens obtained by physical vapour infiltration.

The experimental conditions applied during the catalycity tests lie on the boundary line between the active and passive oxidation process (Balat, 1996; Morino et al., 2002). Then the plasma flux activates the passive oxidation process of the C/SiC samples associated with the formation of a silica glassy oxide scale (Fig. 10), and, at the same time, the partial ablation of the samples due to the loss of silicon as SiO_2 and SiO . The final results are the net weight loss that for the two tested samples has never been higher than 0.6 wt% and the formation of a glass coating with the thickness of 25-35 μm .

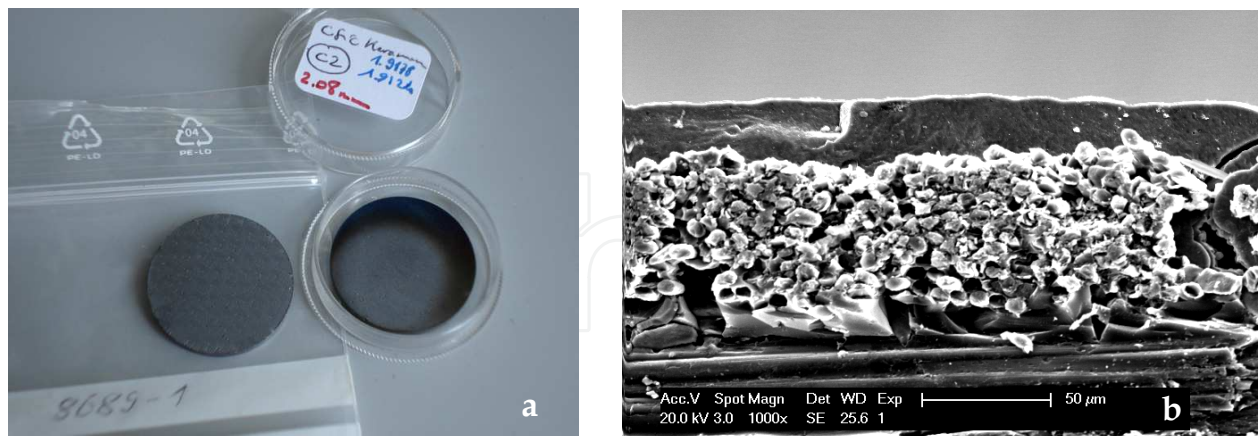


Fig. 10. a) Picture of post test C/SiC sample labelled as A and a virgin one, b) cross-section SEM micrograph after the catalycity test at 1800 K and 200 Pa.

6. Conclusions

The evaluation of emissivity and catalycity efficiency of materials employed in aerospace applications is necessary to estimate heat fluxes and surface temperature values reached by spacecraft vehicles during the re-entry phase in a planet atmosphere. The target is to design and then employ materials characterised by high emissivity values and low catalycity efficiency. In this chapter the measurement of hemispherical emissivity and atomic recombination coefficients of carbon fibre reinforced silicon carbide composites, used as thermal protection system for space vehicles, have been illustrated.

The C/SiC samples tested in MEDIASE facility in the temperature range 900-1900 K and both at 4 than 200 Pa are characterised by relatively high average emissivity values of about 0.7. This value is in line with data retrieved on silica-covered surfaces on different ceramic materials, confirming that the radiative behaviour of the SiC-coated C/SiCs is mainly dictated by the surface glassy oxide layer.

The catalycity measurements carried out using the MESOX facility have on the other hand shown a low oxygen recombination coefficient at high temperature (about 0.07 at 1800 K). The tests performed on two samples have also shown the strong dependence of the recombination coefficient on the surface morphology, which may slightly vary from sample to sample due to manufacturing issues. While samples of the same production batch have indeed shown different values of the recombination coefficient, the overall catalycity trend is the same allowing, moreover, to evaluate the activation energy of atomic oxygen recombination reaction that is about 30 kJ/mol.

The low catalycity and high emissivity exhibited by the investigated C/SiC further confirm its suitability for the intended application in the manufacturing of hot structures for re-entry vehicles. In any case the control of the manufacturing process becomes really mandatory in order to obtain surfaces characterised by a specific morphology which is able to guarantee defined emissivity and catalycity values.

Degradation effects due to the emissivity and catalycity tests on surface of the samples have been also evaluated by electron microscopy analysis and XRD analysis. The characterisation of post-test surface modifications becomes particularly important whenever the reuse capability of a material must be verified. To this end a lot of research groups are making many efforts to improve the resistance to oxidation of C/SiC composites in particular by deposition of ceramic coatings.

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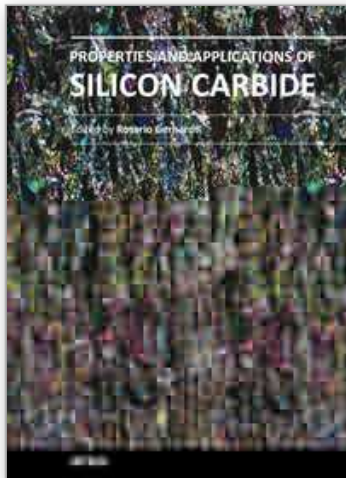
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In this book, we explore an eclectic mix of articles that highlight some new potential applications of SiC and different ways to achieve specific properties. Some articles describe well-established processing methods, while others highlight phase equilibria or machining methods. A resurgence of interest in the structural arena is evident, while new ways to utilize the interesting electromagnetic properties of SiC continue to increase.

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Phone: +86-21-62489820
Fax: +86-21-62489821

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