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Oxidation behavior of SiC ceramics synthesized from processed cellulosic bio-precursor

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Abstract

Oxidation behavior of Si/SiC ceramic composite synthesized from processed cellulosic bio-precursor was studied in dry air over the temperature range 1200–1350 °C. The material was synthesized from processed bio-precursors (bleached bamboo kraft pulp in the form of flat board of bulk density 0.58 g cm^{-3}) and had a bulk density of 2.66 g cm^{-3}, porosity of 0.6 vol% and contents of Si and SiC phases of 39.1% and 60.3% (v/v) respectively. The process of oxidation could be described closely by a parabolic oxidation equation. An activation energy of 141.4 kJ/mol was obtained. Both the SiC and Si phases oxidized and the oxidation was mainly controlled by the transport of molecular oxygen through the growing oxide layer. Pre-oxidation at 1300 °C for 24 h in ambient air increased the strength of Si/SiC ceramics by around 46% because of the healing of the surface defects created during surface preparation by the oxide layer.

Keywords: D. Mechanical property; Processed bio-precursor; SiC ceramic; Oxidation behavior

1. Introduction

Siliconized silicon carbide (Si/SiC) or reaction bonded silicon carbide (RBSC) is a well known industrial ceramic composite produced by infiltrating a porous carbonaceous preform with molten silicon at a temperature in the range of 1500–1600 °C. Various carbonaceous preforms are used such as carbon/SiC powder compacts, carbon felts, carbon fiber–carbon matrix composite preforms, and carbon powder compacts [1–5]. Si/SiC ceramics are also produced by replication of naturally occurring plant bio-structures (bio-preforms or bio-precursors), for example, woods. The obvious aim is to mimic the biological cellular morphology in a ceramic microstructure. Compared to the technically manufactured ceramic materials, the biomorphic Si/SiC ceramics are expected to provide superior combination of properties. In a two step processing, bio-preforms are first converted to carbon templates (C-templates) which on subsequent infiltration with liquid silicon produce Si/SiC ceramics. A major problem of this processing technique arises out of the variation of microstructure of bio-preforms. Depending on the plant age, locality, climate, etc. lot-to-lot variation of the microstructures of the starting bio-preforms can occur which ultimately affects the properties of the final ceramics. Processed bio-preforms are used to produce industrial goods of acceptable uniform properties (e.g. paper). Recently we used processed bio-preforms made from bamboo pulp to synthesize Si/SiC ceramics [6]. The aim was to develop a process of synthesis of bio-morphic Si/SiC ceramics with the avoidance of structural inhomogeneity and related problems. This paper reports the oxidation behavior of Si/SiC ceramics synthesized from the processed bio-precursor; the effect of oxidation on mechanical properties of biomorphic Si/SiC ceramics will also be discussed.

2. Experimental

Pulp stock was made from well-matured Bamboo (*Bambusa tulda* Roxb. collected from Jorhat district of Assam, India) following kraft pulping method. Pulp slurry was prepared by

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Table 1
Post-ceramization properties of C-templates and its bio-preform formulation.

Characteristics of processed bio-preform			Characteristics of C-templates					Characteristics of siliconized C-templates			
Bulk density[a] (g cm^{-3})	Flexural strength[b] (MPa)	Young's modulus[c] (MPa)	Dimensional shrinkages (%)			Pyrolytic weight loss (%)	Bulk density (g cm^{-3})	Bulk density (g cm^{-3})	Porosity (vol%)	Flexural strength (MPa)	Young's modulus (GPa)
			Length	Width	Thickness						
0.58	4.4 ± 0.8	165.8 ± 26.9	20.26	21.33	21.43	71.21	0.60	2.66	0.60	207.7 ± 7.8	199.5 ± 5.1

[a] Determined by measuring weight and dimension.
[b] Determined in an Universal Testing Machine (Instron Bluehill-2, UK) in 3 point mode.
[c] Determined from load–deflection curve obtained during strength testing.

beating the bleached pulp stock followed by sizing using chemical agents and used for making rectangular boards by casting and drying in pressed condition (for processing details see [6]). The pulp boards had a very high content of cellulose of ~78.1% (w/w) and were reasonably strong. The properties of the pulp fiber boards are presented in Table 1. The cast pulp boards were subsequently converted to C-templates by pyrolysis at 800 °C under flowing nitrogen. The dimensional shrinkages, pyrolytic weight changes and other properties of the C-templates are listed in Table 1. C-templates were further infiltrated and reacted with molten Si at 1600 °C under vacuum for conversion to SiC ceramics. The properties of the Si infiltrated C-templates are also summarized in Table 1. The density and porosity of the Si-infiltrated specimens were measured by standard water immersion technique. Rectangular slices were cut from the infiltrated specimen for the preparation of samples for oxidation behavior study and mechanical property evaluation. These samples were properly ground and polished using diamond paste up to 1 μm finish. Oxidation behavior was studied by thermogravimetric method (STA 409C, Netzsch-Geratebau, GmbH, Germany) in flowing dry air ($P = 100$ kPa, flow rate = 1 L/h) at 1200, 1250, 1300 and 1350 °C. In each case the sample (rectangular polished chips of 10 mm × 10 mm × 3 mm) was heated at a constant rate (20 K/min) and an isothermal hold was given for 7 h at the peak temperature. The mass variation (% mass retained) was automatically recorded as a function of time. Rectangular polished bars (45 mm × 3.5 mm × 2.5 mm, edges of the tensile surface were rounded off) were tested at room temperature for bending strength measurement (3-point mode, span 40 mm, crosshead speed 0.5 mm/min) and Young's modulus was obtained from the load–deflection data using a standard software (Instron Bluhill-2, UK). Average values of five readings were taken for strength and modulus determination. In a separate experiment bar samples were heated in air at 1300 °C for 24 h and subsequently tested for measurement of 3-point flexural strength. The fracture surfaces of failed samples were examined under microscope.

3. Results and discussion

3.1. Material preparation

Despite a vast loss in weight during pyrolysis, C-templates showed structural integrity without any sign of visible cracks. Nearly uniform pyrolytic shrinkages were obtained in all major dimensions. Liquid silicon infiltration produced almost dense samples. XRD-analysis showed the presence of only β-SiC and Si as the major ceramic phases (Fig. 1). In our earlier work we have carried out detailed XRD- and microstructure analysis of C-templates and Si-infiltrated C-templates [6]. Microstructure examination of the C-templates showed that the structural morphology of cellulosic fibers was retained in the pyrolyzed samples. The contents of the SiC and Si-phases were estimated from the density and porosity data and found to be 39.09 and 60.31 vol% respectively. Microstructure examination revealed that the fibrous carbons were converted to the fibrous SiC structures which were seen to be embedded in the solidified residual Si phase. All the details of results were described in our published work [6].

3.2. Oxidation behavior

The Si/SiC ceramic samples exhibited similar trends during oxidative heating at four different temperatures (Fig. 2). They showed an initial weight loss followed by a progressive weight gain. The maximum loss in weight occurred at temperatures between 967 and 1017 °C; the weight loss was found to vary in the range of 0.01–0.04%. Also a change was noticed in the weight gain trend. Following a rapid gain in weight, the gain

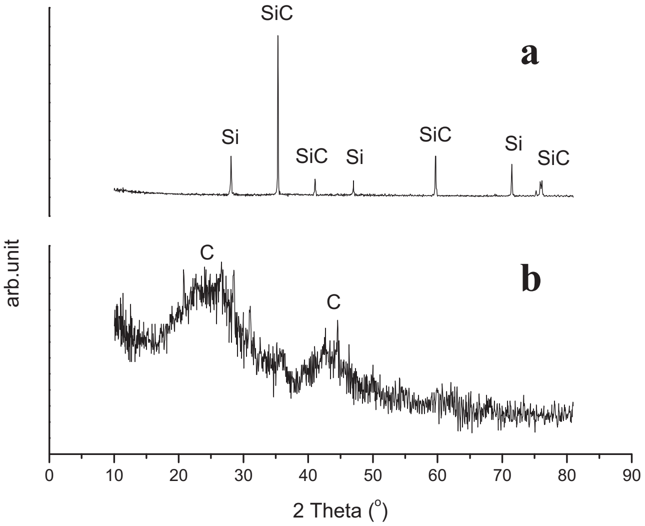

Fig. 1. XRD-profiles of (a) the silicon infiltrated C-template and (b) the C-template prior to infiltration.

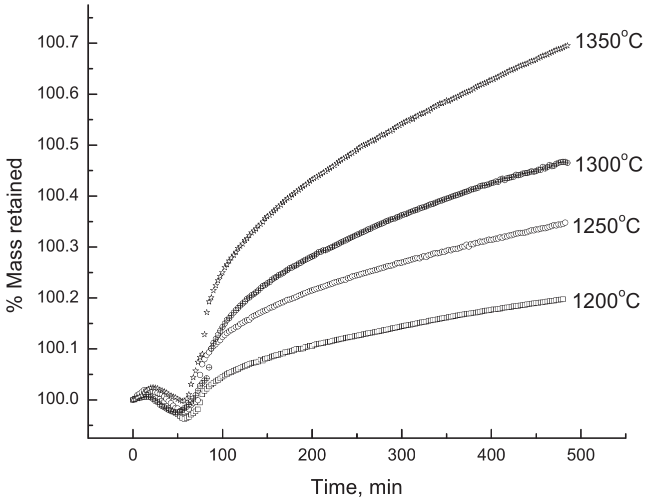

Fig. 2. Oxidation mass change vs time.

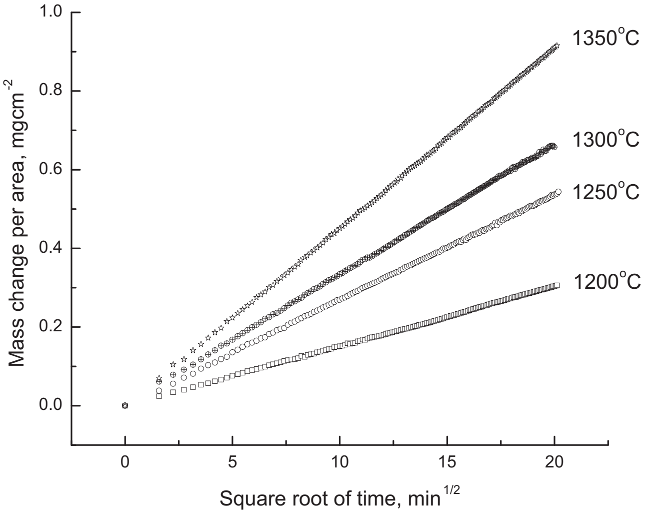

Fig. 4. Oxidation mass change vs time$^{1/2}$.

became slow and finally the weight gain curves flattened out. This change became more pronounced at higher temperatures. Isothermal heating was continued for a period of 7 h at each temperature (1200, 1250, 1300 and 1350 °C). The weight gain recorded was converted to weight gain per unit area and was plotted against time (Fig. 3).

The initial weight loss indicated that residual carbon was likely to be present in the infiltrated specimens. During investigation on the microstructure of dense biomorphic Si/SiC ceramics synthesized from wood precursors by orienting imaging microscopy, our group detected the presence of unreacted carbon [7]. The oxidation process is assumed to begin with oxidation of residual carbon, evolving gaseous CO or CO_2; the mass loss observed can be assigned to the oxidation of carbon. At low temperature oxidation of carbon is controlled by the rate of accompanying chemical reaction whereas at high temperatures (for example, $T > 700$ °C) mass transfer by diffusion controls the process of oxidation of carbon [8]. By the evolution of gaseous products pores are formed and the reacting species have to diffuse along the pore prior to reacting with the carbon lying at the pore bottom. At the low temperatures, the chemical reaction rate is likely to be low and the oxygen consumption along the pore is also low; at high temperatures chemical reaction rate corresponding to oxidation of carbon is high and the oxygen consumption is also high making diffusion mass transfer to control the process of oxidation of carbon. As long as carbon is present and reacts with oxygen, the length of the diffusion path for the gaseous reactant will increase, and, as a result, the consumption rate of carbon will decrease as the function of time. This is evident in the weight loss trend as shown in Fig. 2. The increase in weight is related to the oxidation of both the Si and SiC phases giving rise to the formation of silica. The growth of the silica on the pore wall likely results in a progressive decrease in the pore size [9]. A time eventually comes when the pores are sealed with the silica near the sample surface. The reactant has to diffuse through a coherent layer of the silica deposited on the sample surface for reaction to occur. We observed a change in the weight gain trend in the TG curves likely because of the onset of the protective scale formation. Finally the weight gain curve flattens out. This feature is very much similar to the oxidation weight gain trend of reaction bonded silicon carbide, a dense duplex ceramic composite containing Si and SiC phases [10]. For the entire period of isothermal heating at each of the four peak temperatures the weight gain data were found to be best fit by a parabolic oxidation rate curve of the type: $w^2 = kt$, where w is the weight gain per unit area, k the oxidation rate constant and t the time (Fig. 3). The oxidation rate constant k at each temperature was determined from the slope of the linear plot of w against $\sqrt{t}$ (Fig. 4). The activation energy was calculated from the Arrhenius equation $k = A\mathrm{e}^{-E/RT}$, where A is the pre-exponential factor independent of temperature, E the activation energy, R the gas constant and T the absolute temperature. The Arrhenius plot of $\log k$ vs $1/T$ is shown in Fig. 5. All the points corresponding to the test temperatures fit to a straight line

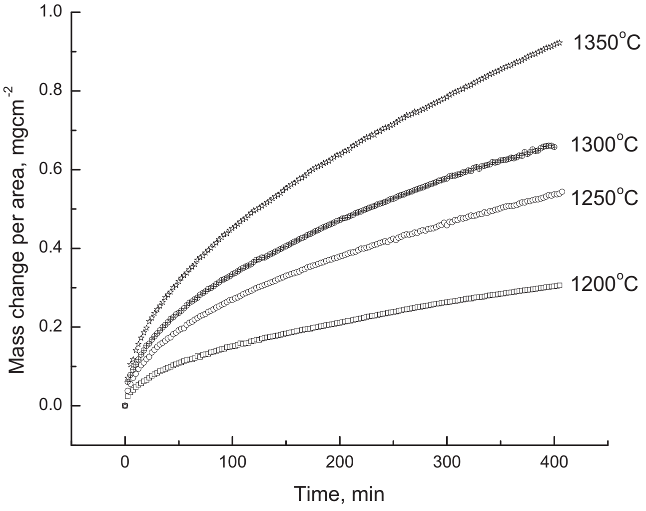

Fig. 3. Oxidation mass gain vs time.

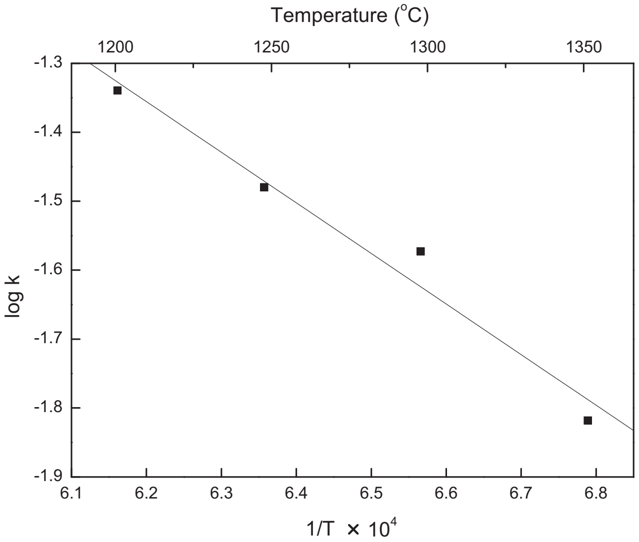

Fig. 5. Plot of log k vs 1/*T*.

where the gradient of the line gives the value of the activation energy. The value of *E* obtained in the present work was 141.4 kJ/mol.

In case of oxidation of Si it is widely accepted that the process of oxidation is controlled by diffusion of diatomic oxygen through the growing silica film [11]. The activation energy for oxidation of Si agrees well with value of activation energy 112.5 kJ/mol for molecular oxygen diffusion through fused silica [12]. At low temperatures the activation energies for oxidation of sintered and hot pressed SiC are reported to be 133.5 and 154.4 kJ/mol, whereas at high temperatures high values are obtained. This suggests that oxidation of SiC at low temperatures may be controlled by inward diffusion of oxygen molecules, similar in process occurring in the case of oxidation of Si. High activation energies obtained for oxidation of SiC at high temperatures are not in favor of oxygen transport mechanism. Many investigations are reported which revealed that several factors are responsible for higher oxidation activation energy at higher temperatures for SiC [13–19]. The activation energy is dependent on many factors, such as crystallographic plane, dopant concentration, crystallization of oxide films, and grain orientation on the surface of oxidation. Harris examined the influence of crystallographic plane ({0 0 0 1} planes) on the oxidation of single crystal SiC (6H polytype) [20]. According to the author the $(0\,0\,0\,\bar{1})$c face exhibited parabolic oxidation with the activation energy of 197 kJ/mol. The opposite face exhibited reduced oxidation rates and the author observed linear kinetics and measured the activation energy to be 356 kJ/mol (it corresponds to a chemical reaction at the interface). Difference in oxidation behavior for the two {0 0 0 1} faces was also reported by Von Munch and Pfaffender [21], similar to the observations made by Harris [20]. Ramberg et al. examined the oxidation behavior of the cubic CVD SiC and observed that the oxidation rates of the (1 1 1) face of cubic CVD-SiC were the same as those of the (0 0 0 1) face of the single crystal SiC, the opposite faces of the two materials, $(\bar{1}\,\bar{1}\,\bar{1})$ and $(0\,0\,0\,\bar{1})$, also oxidized at the same rates, but much faster than their opposite (0 0 1)/(0 0 0 1) faces [22]. Many investigations reported extremely high activation energies (>400 kJ/mol) for the oxidation process of polycrystalline SiC materials above 1400 °C. Several studies have shown that appreciable lattice diffusion of oxygen occurs during oxidation of polycrystalline SiC materials at high temperatures (1300 °C and above) [23]. The activation energy for lattice diffusion in fused silica was reported by Sucov to be ~300 kJ/mol [24]; other studies indicated this value could be as high as 450 kJ/mol [25]. Lattice diffusion of oxidant may be one of the reasons for high activation energies for high temperature oxidation of SiC. The decrease in oxidation rate at and above 1400 °C exhibited by the sintered polycrystalline SiC materials was found to be related to the crystallization of the oxide film. It was indicated that the transport of oxidant through the crystalline phase was considerably slower than through the amorphous phase. Decreases in oxidation rate of the sintered material were correlated with the formation of a continuous layer of the crystals over the SiC surface [14]. Singhal suggested outward desorption of CO molecules from the growth interface as the reason behind the high activation energy for the oxidation process of SiC at high temperatures [13]. In our material both the Si and SiC phases oxidize and the activation energy obtained in the present study indicates that the oxidation process occurs by inward oxygen transport mechanism. The oxidation rate constants obtained in the present work over the temperature range of 1200–1300 °C are listed in Table 2. Coppola et al. studied the oxidation behavior of pressure-less sintered α-SiC ceramics (98–99% dense, sintered with boron and carbon-based sintering additives) at temperatures between 1200 °C and 1500 °C in static air and obtained rate constant values of 7.08×10^{-13}, 5.09×10^{-12}, 7.64×10^{-11} kg^2/m^4 s at 1200, 1350 and 1500 °C respectively [26]. Oxidation behavior of reaction sintered silicon carbide having a bulk density of 3.00 g cm^{-3} and unreacted Si-content of ~23% (v/v) was studied in air over a temperature range of 1200–1350 °C and parabolic oxidation rate constants of 3.4×10^{-11}, 4.3×10^{-11}, 1.03×10^{-10} and 1.45×10^{-10} kg^2/m^4 s were reported at temperatures 1200, 1250, 1300 and 1350 °C respectively [27]. Reaction sintered SiC has higher oxidation rate constants than pressure-less sintered SiC because of the presence of residual silicon in the material. The biomorphic SiC has higher residual silicon content than reaction sintered SiC which is likely related to

Table 2
Values of oxidation rate constant at different temperatures.

Temperature (°C)	Oxidation rate constant (kg^2/m^4 s)
1200	3.85×10^{-10}
1250	1.19×10^{-9}
1300	1.51×10^{-9}
1350	3.31×10^{-9}

the higher oxidation rate constants obtained in the present study.

3.3. Flexural strength

The effects of pre-oxidation at 1300 °C for 24 h in air on the strength of the Si/SiC ceramics were examined. The failure of the specimen in bending occurred without any deformation and a mean fracture strength value of 303.9 ± 38.3 MPa was found. Pre-oxidation at 1300 °C substantially increased the strength by nearly 46%. Limited information is available on the effect of pre-oxidation on the strength of biomorphic SiC ceramics. In case of conventional Si/SiC ceramic composites synthesized following the powder route conflicting information is available. Trantina observed that the strength of the siliconized SiC could be increased by about 25% by a heat-treatment at 1200 °C for 1 h [28]. Tomlinson et al. showed that the oxidation of siliconized silicon carbide always decreased the strength, the extent of which could go up to 50% depending on period of oxidation at 1350 °C [29]. For strengthening due to oxidation it is generally accepted that, after oxidation the silica scale tends to fill in or blunt surface flaws, resulting in increasing the strength. Tomlinson et al. observed that oxidation of siliconized silicon carbide specimens at 1350 °C resulted in formation of an oxide layer following a parabolic oxidation law and preferential inter-grain oxidation roughened the surface and formed deep pits. Extensive cracking was observed on the surface of the oxide layer that could result in decrease in strength. Our sample is a duplex ceramic composite synthesized from processed cellulosic precursor that contained both the SiC and Si phases. During microstructure examination no cracks were evident on the surface of the specimen oxidized. A number of fractured surfaces were examined. The typical fracture surface of pre-oxidized specimen tested at room temperature for flexure strength measurement is shown in Fig. 6. Hardly any initiation site could be located or crack path could be identified. It indicated that cracking could not occur near the surface after oxidation. It, therefore, appeared that, in the present case pre-oxidation at 1300 °C could improve the room temperature strength of the Si/SiC ceramics by healing or rounding off the crack or flaws by the flow of the silica scale. Retention of strength of Si/SiC ceramics in service conditions involving high temperature ambient atmosphere is a very important issue. The experimental results obtained in the present study indicated the suitability of the Si/SiC material synthesized from processed bio-precursors for such uses.

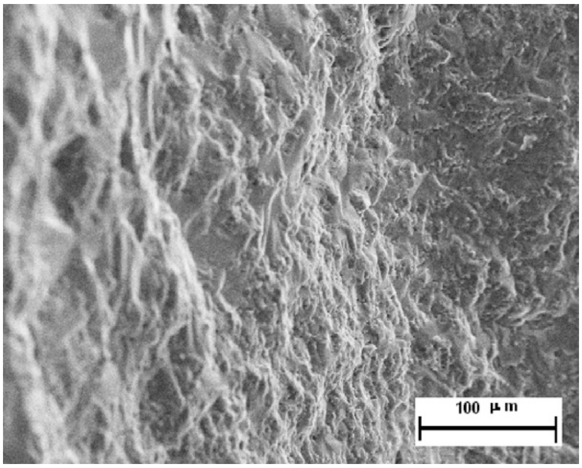

Fig. 6. Fracture surface of pre-oxidized Si/SiC specimen.

4. Conclusions

Oxidative heating of Si/SiC ceramics synthesized from processed cellulosic bio-precursors in dry air indicated initial loss of weight of the samples likely because of burning of any residual carbon present in them. Maximum weight loss varied in the range of 0.1–0.4% and it was found to occur at temperatures in the range of 978–1016 °C. Afterwards, the samples were found to gain in weight and following rapid weight gain, the weight change curves flattened out indicating formation of a protective oxide scale on the surface. The weight gain data obtained during the isothermal heating at temperatures in the range of 1200–1250 °C were found to be best fitted to a parabolic rate equation. Activation energy of 141.4 kJ/mol obtained in the present study indicated that oxidation might occur via transport of molecular oxygen through the growing oxide layer. Si/SiC samples pre-oxidized at 1300 °C for 24 h in ambient air showed an increase of flexure strength by 46%, probably because of the healing of the surface cracks created during sample surface preparation by the flow of oxide scale and it further indicated suitability of the material for high temperature application in challenging atmospheres.

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