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High-temperature Compressive Strength of Reaction-formed Silicon Carbide (RFSC) Ceramics

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Abstract

High-temperature compressive strength of reaction-formed silicon carbide ceramics, fabricated by the melt infiltration of silicon, and silicon-5 at% niobium alloy into microporous carbon preforms, have been evaluated. The values of maximum strength ranged from 1000 MPa at 1250°C to 500 at 1400°C. These maximum strengths were reached for the ceramic fabricated by silicon infiltration into a low silicon yielding carbon preform. The ceramics fabricated by silicon infiltration into a higher volume fraction carbon preform showed a decrease on the strength. The infiltration of silicon-5 at% niobium alloy into a carbon preform also resulted in a decrease of strength compared to the ceramic fabricated by silicon infiltration into the same carbon preform. The dependence with temperature and composition of the high temperature compressive behavior is discussed. Published by Elsevier Science Limited.

1 Introduction

Silicon carbide based advanced ceramics are leading candidate materials for a number of applications in the aeronautics, energy, electronics, nuclear, and transportation industries.[1–3] These materials are being considered for a variety of engine components, radiant heater tubes, heat exchangers, heat recuperators, and components for land based turbines for power generation. Other applications include the first wall and blanket components of fusion reactors, furnace linings, and bricks, and components for diffusion furnitures (boats, tubes) in the microelectronics industry. Reaction formed silicon carbide based ceramic materials have many attractive properties for a number of these applications.[3–12] In this process a carbon preform is infiltrated with molten silicon or silicon-refractory metal alloys to reduce, in the second case, the amount of residual silicon and improve the toughness at room temperature[7] without degradation of the high temperature thermomechanical properties. The final products are low cost, fully dense and have tailorable microstructure and mechanical properties. The work reported here is part of a program aimed at developing low cost reaction-formed silicon carbide-based ceramics with tailorable microstructure and thermomechanical properties. A number of high temperature properties, i.e. strength and toughness, creep, and environmental durability of these ceramics have been discussed in earlier publications.[7–12] In this paper, high temperature compressive strengths of three types of reaction-formed silicon carbide materials will be discussed. Effect of silicon and niobium disilicide phases on the mechanical behavior will be presented.

2 Experimental Procedures

The fabrication details of the reaction formed silicon carbide based ceramics (RFSC) have been discussed elsewhere.[3–10] The materials used in this study were fabricated by the reactive infiltration of molten silicon or silicon-5 at% niobium alloy into microporous carbon preforms. The final product was $SiC + Si$ when molten silicon was used and $SiC + Si + NbSi_2$, when silicon-5 at% niobium alloy was used. After infiltration, samples were cross-sectioned and polished for metallographic studies.

For mechanical property measurements, rectangular samples of $2{\cdot}3 \times 2{\cdot}3 \times 4$ mm were machined. The specimens were tested in compression at a

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constant strain rate of $2\times10^{-5}\,s^{-1}$ for temperatures ranging from 1250 to 1400°C, in air. Testing was performed using a screw driven Instron universal testing machine model 1185 with a furnace mounted on its frame. Alumina rods with SiC pads were used. The load/time behavior was monitored on a chart recorder. The deformed samples were studied with a Philips XL20 scanning electron microscope (Microscopy Service, University of Seville, Spain).

3 Results and Discussion

Figure 1 (a) and (b) shows the microstructure of the reaction formed silicon carbide (RFSC) materials fabricated by silicon infiltration with approximately 8–10% and 20–22% free silicon (designated as RFSC–LS and RFSC–HS, respectively). This microstructure indicates the uniform distribution of phases. The higher free Si content is correlated with the higher pore volume fraction of the carbon preform used. The RFSC material with $NbSi_2$ was fabricated by the infiltration of silicon-5 at% niobium alloy into low silicon yielding carbon preform (designated as RFSC–$NbSi_2$). The RFSC–$NbSi_2$ material contains 4–5% free Si, and 5–6% $NbSi_2$. The lower amount of free Si was due to the presence of $NbSi_2$.

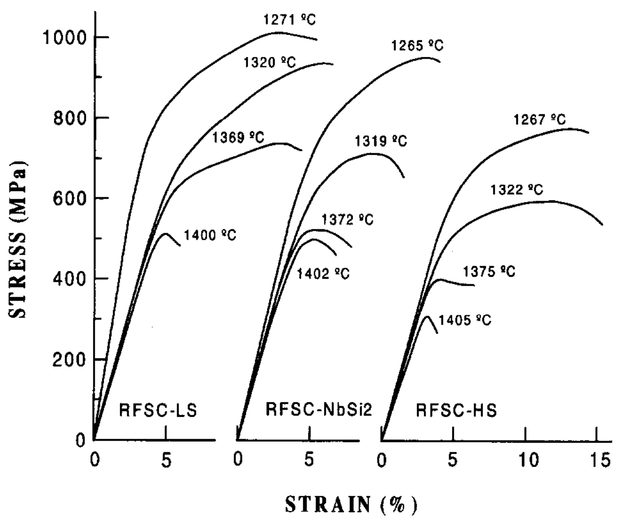

Fig. 2. Plot of stress versus strain for the three materials studied.

The stress–strain curve of the constant strain rate experiments are shown in Fig. 2. The stresses on the sample decreased after a maximum was reached. The experiments were stopped at strains ranging from 4 to 17% once the stress on the sample started to decrease. The strains at which this decrease in strength starts to occur are considerably larger at the lower temperatures (Fig. 2). The deformed samples showed microcracking [Fig. 3(a)] but catastrophic crack propagation did not occur. A plot of the maximum stress as a function of temperature for the three materials is shown in Fig. 4. The strength of the RFSC–LS material was considerably higher than the strength of RFSC–HS material for the whole temperature range studied. The strength of the RFSC–$NbSi_2$ reached values between the RFSC–HS and RFSC–LS, although at 1400°C it had the same value than the RFSC–LS (Fig. 4). SEM observations indicate that the amount of free silicon on the specimen surface increases with temperature, creating a superficial silica layer at 1400°C [Fig. 3(b)].

The stress–strain curves of Fig. 2 show three well defined stages: a linear elastic region, a non stationary region of plastic deformation where a continuous increase of stress is necessary in order to progress the deformation of the material at the

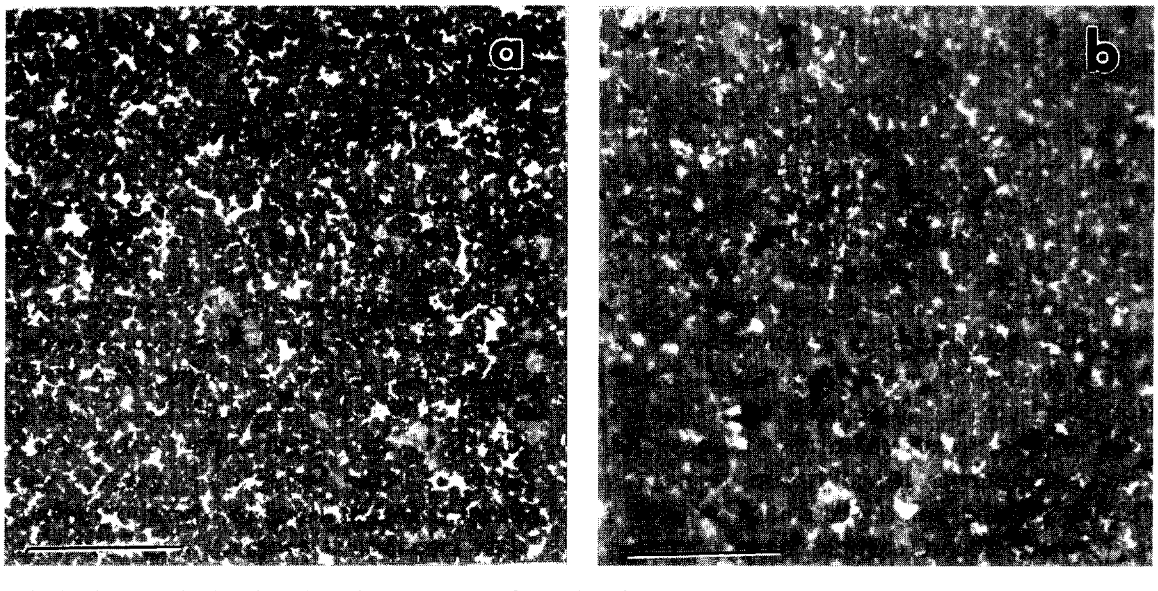

Fig. 1. Optical micrograph showing the microstructure of reaction formed silicon carbide; (a) RFSC–HS (high silicon) and (b) RFSC–LS (low silicon). The length of the scale lines in each micrograph is 100 μm.

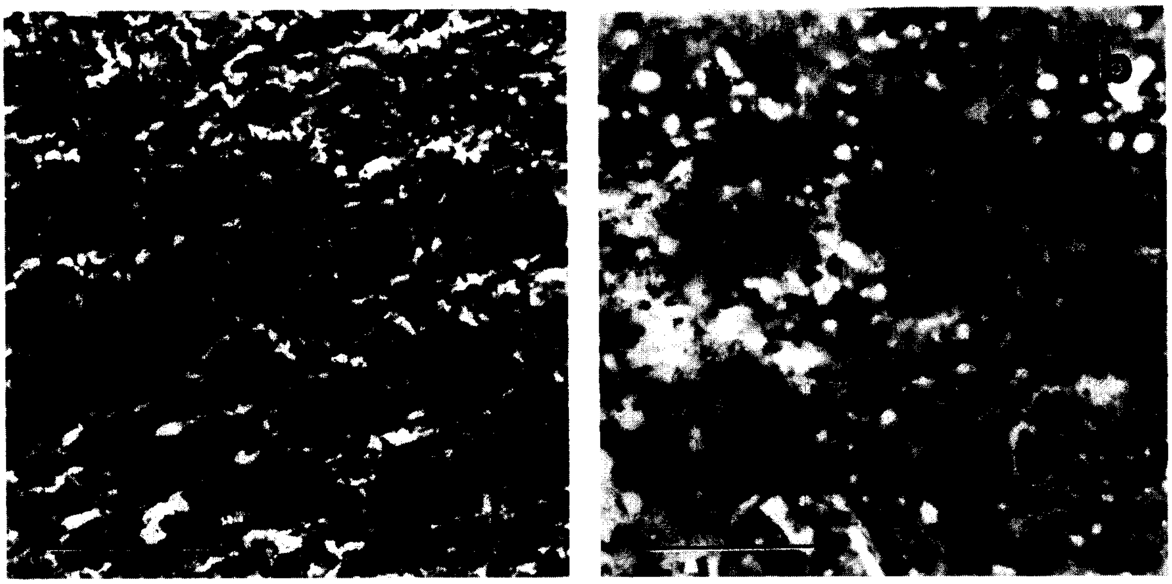

Fig. 3. Scanning electron micrographs of RFSC–HS specimens after deformation at (a) 1267°C and (b) 1405°C. The length of the scale lines in each micrograph is 50 μm.

strain rate imposed by the experiment, and a third region in which the material accommodate plastic deformation by formation and coalescence of cavities with the subsequent decrease in strength. The system could reach a short stationary stage between the second and third region but, independent of this fact, the maximum stress reached must be approximately the stress at which significant deformation damage starts to occur.

The reaction formed silicon carbide (RFSC) can be considered as composed of a continuous silicon carbide (SiC) network with $NbSi_2$ and/or Si filling up the pores of such a network. This microstructure cannot be deformed without the deformation of both components, so the total strength to plastic deformation will have contributions of all the phases present. The lower strength of the RFSC–HS over the RFSC–LS material at all the temperatures studied is related with the lower amount of SiC present on the first one (different initial carbon preform density), indicating clearly that the strength is dominated by the SiC, which plays the main role in the deformation behavior. It is important to point out the high deformation resistance of the RFSC–LS material, which reaches a value over 500 MPa at temperatures very close to the melting point of silicon.

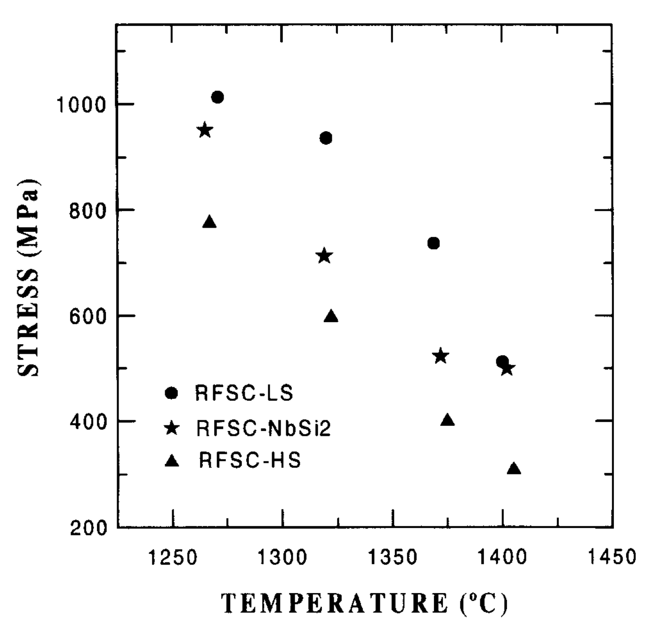

Fig. 4. Plot of the maximum stress versus temperature for three RFSC materials studied.

The RFSC–LS and RFSC–$NbSi_2$ materials have the same amount of SiC (similar carbon preform) but the strength of the first one is significantly higher at the lower temperatures studied. This fact indicates that the contribution of the $NbSi_2$ and/or Si can not be neglected at these temperatures and that the differences in strength are related with differences on the deformation of the Si and $NbSi_2$ phases. If the deformation of these phases were dominated by a dislocation mechanism the opposite effect would be observed because, although no data are available for the $NbSi_2$, the Si is expected to have a higher dislocation mobility than the $NbSi_2$. The values of the melting points for Si and $NbSi_2$ (1410°C and 1930°C, respectively) and studies of the brittle–ductile transitions on another refractory disilicide $(MbSi_2)^{13}$ and Si^{14} support this statement. The lower strength must be associated with the different nature of the SiC/Si and SiC/$NbSi_2$ interfaces and/or a finer microstructure associated with the introduction of $NbSi_2$. TEM observations of the Si/SiC interfaces on reaction bonded SiC^{15} showed smooth interfaces that could be stronger than the SiC/$NbSi_2$ interfaces due to the structural differences between SiC and $NbSi_2$. A finer microstructure can accommodate the SiC deformation more easily, accounting also for a decrease in strength. As the temperature increases the Si strength decreases faster than for the other phases as the melting point for Si is 1410°C, and the differences between the RFSC–LS and the RFSC–$NbSi_2$ materials decreases, having the same strength at 1400°C (Fig. 4). There is another effect to be considered; the thermal expansion coefficient of $NbSi_2$ ($11{\cdot}7\times10^{-6}$/°C) is higher than for SiC ($4{\cdot}4\times10^{-6}$/°C). Thus for temperatures below the processing temperatures, the SiC/$NbSi_2$ interface is under tensile stress. This could facilitate interface debonding and cavitation. This effect should decrease as the test temperature approaches the processing temperature.

Finally, it should be pointed out that although cavitation deteriorates the materials strength as showed in Fig. 2, this deterioration is not catastrophic for deformations up to 17% at strain rates and stresses considerably higher than the threshold values for deformation damage of reaction bonded SiC [$10^{-8}\,s^{-1}$ (Ref. 15) and 132 MPa[16] at 1300°C]. The smaller maximum strains at the higher temperatures must be related with a time dependent cavity formation and coalescence associated with the deposition of Si on the surface of the sample [Fig. 3(b)].

4 Conclusions

1. The maximum strength of the reaction formed SiC ceramics studied range from 500 MPa at 1400°C to 1000 MPa at 1250°C when a deformation strain rate of $2\times10^{-5}\,s^{-1}$ is imposed.
2. The strength decreases with the increase of the porous volume fraction in the carbon preforms used, indicating that the strength is dominated by the SiC. For the same volume fraction, the strength decrease with the presence of $NbSi_2$. This decrease should be related with differences on the interfaces.
3. At 1400°C, the strength of the RFSC–LS and RFSC–$NbSi_2$ are the same due probably to the decrease in strength of Si. The reaction formed silicon carbide (RFSC) containing $NbSi_2$ could have higher strength over the melting point of silicon due to this fact.

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