

Thermal shock resistance and fracture toughness of liquid-phase-sintered SiC-based ceramics

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Abstract

The effect of the heat treatment on the toughness and thermal shock resistance of the silicon carbide–silicon nitride composites prepared by liquid-phase-sintering was investigated. The fracture toughness has been estimated using the indentation method and the thermal shock resistance was studied using the indentation-quench method. The results were compared to those obtained for a reference silicon carbide material, prepared by the same fabrication route. The indentation toughness increased from 2.88 to 5.39 MPa m^{1/2} due to the toughening mechanisms (crack deflection, mechanical interlocking and crack branching) occurring in the heat-treated materials during the crack propagation. Similarly the thermal shock resistance increased after the heat treatment of the experimental materials.

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1. Introduction

Silicon carbide and silicon nitride have been recognised as important structural ceramics because of their good combination of mechanical and thermal properties. SiC ceramics show good wear, creep and oxidation resistance at high temperatures, but relatively low fracture toughness. On the contrary, Si₃N₄ ceramics exhibit higher fracture toughness and good flexural strength, but lower resistance to oxidation at high temperatures.¹ Silicon carbide is a promising material for high temperature applications (heat engines, heat exchangers, and many other devices), however it is difficult to densify without sintering additives because of the covalent nature of Si–C bonding and low self-diffusion coefficient. Components to be properly applied in high temperature need to have a high resistance to thermal shock, thermal fatigue, corrosion, and resistance to creep deformation. Si-based ceramics such as silicon carbide and silicon nitride due to their covalent bonding exhibit a high strength at elevated temperature (up to 1000 °C), as well as low thermal expansion coefficient and high thermal conductivity, and therefore a high resistance

to thermal shock.^{2–4} Because of the lower fracture toughness of silicon carbide ceramics, the possibility of an in situ toughening has been investigated by many authors.^{5–9} Toughening is obtained through the development of large elongated or platelet-shape grains that has been related to the $\beta \rightarrow \alpha$ SiC transition occurring at 1800–2000 °C. Elongated grains have been shown to increase fracture toughness by crack bridging or crack deflection due to weak interface boundaries, but coarsening leads to an increase of the size of the critical flaw which degrades flexural strength.^{6,9–13} The Vickers indentation fracture toughness test has been applied for estimation of the fracture resistance of brittle ceramics for the past three decades.^{14–19} The techniques rapidly achieved popularity because their simplicity (only small volume of material, short preparation of samples and low financial costs). However, Quinn and Bradt²⁰ concluded that the Vickers indentation fracture toughness test is fundamentally different than the standard fracture toughness technique. This technique is not reliable as fracture toughness test for ceramics or for other brittle materials, because the crack initiation and propagation is not the same as the sequence of crack processes in the standardized fracture toughness tests. They also recommended that these tests should not be used for fracture toughness testing of ceramics, even if just for a comparative basis. The ceramic scientific community agrees with the opinion

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of Quinn and Bradt²⁰ with regards to the fracture toughness values mainly for the unsuitability of the indentation lifetime and probability prediction of ceramic samples/parts. On the other hand it could be imagined that the measured “indentation toughness” using indentation method can offer some basic information with regards to the toughness of newly development ceramics. Also the cracks created by Vickers indenter could be useful for the study of the possible toughening mechanisms occurring also during the crack propagation under the applied load required for determination of K_{IC} .

During thermal shock, transient thermal stresses build up in the material which can become large enough to induce damage, such as microcracking or macrocracking.²¹ The thermal shock properties of a material depend on the number of parameters such as tensile strength, fracture toughness, Young's modulus and thermal expansion coefficients. In addition to these materials properties, which can be tabulated, the microstructure character is also of importance and influences the thermal shock behaviour of the material.²² The investigation of the behaviour of Vickers indentation cracks under quenching condition has raised interest during the last years. An indentation-quench method has been developed by Andersson and Rowcliffe.²³ Compared to the Hasselman quench-strength method,²⁴ the evaluation procedure is simple, the sample preparation is easy and only a small number of samples are needed for a series of measurements at different temperatures. The indentation-quench method can be used for the characterization of materials and as a diagnostic tool to predict the thermal shock damage occurrence in a component.²⁵ To evaluate the thermal stress crack initiation and propagation behaviour of ceramics, two thermal shock resistance parameters are usually used.²⁶ First is the resistance to initiation of crack, expressed by parameter R :

$$R = \frac{\sigma_c(1 - \nu)}{\alpha E} = \Delta T_c \quad (1)$$

where σ is the tensile strength, E the Young's modulus, α the coefficient of thermal expansion and ν is the Poisson's ratio.

Higher R represents a greater resistance to the initiation of fracture during rapid quenching and during steady-state heat flow down a steep temperature gradient. The second is the resistance to propagation of crack expressed by the parameter R'''' :

$$R'''' = \left(\frac{K_{IC}}{\sigma_r} \right)^2 (1 + \nu) \quad (2)$$

which dedicates the resistance to catastrophic crack propagation of ceramics under a critical temperature difference, dT_c .

From the above two equations, it is clearly visible that the thermal shock resistance can be improved by the increased flexural strength and fracture toughness and by decreased Young's modulus and coefficient of thermal expansion. Simultaneous increase of strength and toughness in ceramics is not always possible.² Usually ceramics with higher strength are more brittle and materials with more stable crack propagation exhibit a lower strength.

The thermal shock resistance of SiC + Si₃N₄ composites has not yet been reported. Takeda and Maeda²⁷ showed that the

thermal shock resistance of hot pressed SiC with BeO and AlN depends on the thermal conductivity. At 100 W m⁻¹ K⁻¹, $\Delta T_c = 680^\circ\text{C}$ and at 65 W m⁻¹ K⁻¹, $\Delta T_c = 450^\circ\text{C}$. Wang and Singh²⁸ measured for HP SiC $\Delta T_c = 500^\circ\text{C}$ at thermal conductivity of 87 W m⁻¹ K⁻¹. Pettersson et al.²² studied the best parameters for measuring the thermal shock resistance of Si₃N₄-based materials with an indentation-quench method. The best resolution was obtained with a sample diameter 12 mm, height 4 mm, initial crack length 100 μm and water temperature 90 $^\circ\text{C}$. Many others authors have studied thermal shock resistance of different ceramics materials by water quenching or indentation tests (sintered alumina/silicon carbide nanocomposites,²⁹ alumina/zirconia functionally graded material,³⁰ Al₂O₃-TiC composites,³¹ SiC-BN composites,³² porous silicon carbide³³).

The aim of this work is to investigate the influence of the heat treatment on the microstructure, indentation toughness and indentation thermal shock resistance of SiC and SiC + Si₃N₄ composites.

2. Experimental procedure

β -SiC powder (HSC-059, Superior Graphite) was mixed with Al₂O₃ (A 16 SG, Alcoa), Y₂O₃ (grade C, H.C. Starck) and Si₃N₄ (AIY-3/54, Grade C, Plasma & Ceramic Technologies Ltd.). The Si₃N₄ powder contains Y₂O₃ and Al₂O₃ sintering additives in weight ratio 6:3. The weight ratio of nonoxide matrix to oxide sintering additives SiC (+Si₃N₄): Y₂O₃ + Al₂O₃ was kept constant, 91:9. The weight ratio of particular oxides Y₂O₃:Al₂O₃ was 6:3 for all compositions. The final chemical compositions of the investigated materials are listed in Table 1.

The powder mixtures were ball milled in isopropanol with SiC balls for 24 h. The suspension was dried and subsequently sieved through 25 μm sieve screen in order to avoid hard agglomerates. The samples were sintered by hot pressing at 1850 $^\circ\text{C}$ /1 h under mechanical pressure of 30 MPa in N₂ atmosphere. The hot pressed samples were subsequently annealed under various temperature conditions given in Table 1. After sintering and annealing the specimens were cut, polished to a 1 μm finish and plasma etched. The microstructures were then studied using an SEM (JEOL JSM-7000F).

The densities of the sintered and annealed specimens were measured according to Archimedes' principle. Mechanical properties were investigated using indentation methods. Hardness was determined by Vickers indentation (hardness testers LECO 700AT) under a load of 49.05 N with a dwell time of 10 s. In order to determine the indentation toughness at least 15 Vickers indentations per specimen were introduced with the load of 49.05 N. The indentation toughness was calculated from the lengths of radial cracks and indents diagonals using a formula valid for semi-circular crack systems as proposed by Anstis et al.¹⁴:

$$K_{IC} = 0.016 \left(\frac{E}{H} \right)^{1/2} \left(\frac{P}{c^{3/2}} \right) \quad (3)$$

where K_{IC} , indentation toughness (MPa m^{1/2}); 0.016, material-independent constant for Vickers-produced radial cracks; E ,

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