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Porosity and Oxide Layer Dependence of Thermal Shock Behavior of Porous Silicon Nitride Ceramics

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A water-quenching technique has been adopted to evaluate thermal shock fracture and fatigue behaviors of porous Si_3N_4 ceramics in an air atmosphere. The high-porosity Si_3N_4 ceramics exhibit a higher strength retention and a better resistance to thermal shock fatigue because of its role of the pores as crack arresters. A dense and coherent surface oxide layer leads to a significant benefit in residual strength during thermal fatigue, however, an increased fatigue number to 30th cycle cannot cause a further influence although a thicker oxide layer presents, which is attributed to holes defect and disappearance of part intergranular phase.

KEY WORDS: Porous silicon nitride ceramics; Oxide layer; Thermal shock behavior; Thermal fatigue

1. Introduction

The perfect combination of microstructures and mechanical behaviors is expected because it provides a mechanism for designing materials used under harsh conditions and for improving their performance. Over the past few decades, porous Si_3N_4 ceramics as functional components have been extensively employed in various industrial fields such as catalyst supports, antenna windows, radiant burners, hot insulators, and filters for hot gases due to their excellent dielectric properties, good oxidation resistance, high bending strength at room and elevated temperature, and remarkable thermal shock resistance[1−5]. In the majority of applications, porous Si_3N_4 ceramics often encounter strong heat flow and/or abrupt temperature shock, which can lead to instantaneous thermal stresses and subsequently make ceramics become sensitive to crack or harm. The thermal shock behavior of porous ceramics in service has been widely researched and developed on many occasions[6−8], and it is well established that the residual strength after thermal shock was discovered to decrease gradually or interruptedly over a critical thermal shock temperature difference and the thermal shock resistance was observed to increase with increasing porosity, independent of whether the shape of pore in matrix is fully regular or ordered. The conclusions have been verified in a variety of experiments for single-phase porous ceramics accompanying the efforts of researchers[9,10].

Apart from this, the investigations of thermal shock behavior of Si_3N_4 composite ceramics also have been conducted with some remarkable progresses. For instance, the critical temperature difference of 30 wt% BAS/Si_3N_4 is over 1100 °C and the retained strength at a quenching-temperature difference of 1100 °C is hardly affected by the quenching cycles[11]. The highest critical temperature difference was obtained from 15 wt% TiN/Si_3N_4 nano-composite, and the addition of TiN particles can also improve the resistance to thermal shock damage[12]. Porous BN−SiO_2−Si_3N_4 composites were successfully obtained with good critical thermal shock temperature of 800 °C[13].

In general, the thermal-shock experiments are performed by means of measuring the residual flexural strength in an air atmosphere after quenching heated specimens from successively higher temperatures into a water bath. Before quenching, the samples are first kept at expected high temperatures for some time in order to eliminate any temperature gradient within them and achieve thermal equilibrium prior to quenching, which may give rise to some oxidation on porous Si_3N_4 ceramic surface. It has been reported that such an oxidation can improve the ability of materials to endure external forces by sealing surface flaws[14−16], and follow by the increase of mechanical strength. This reveals that the measured strength after thermal shock should embody the comprehensive influences of surface oxidation and thermal shock on materials performance.

However, while increased thermal shock resistance by way of increasing porosity has often been reported, only a few studies have

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Table 1 Relevant physical and mechanical properties of porous samples after sintering

	Porosity (%)	Fracture strength (MPa)	Fracture toughness (MPa $m^{1/2}$)	Young's modulus (GPa)	Poisson's Ratio	Thermal expansion coefficient ($\times 10^{-6}$/°C)	R (°C)	R''''' (μm)
Sample 1	32	250 ± 10	2.11 ± 0.05	270 ± 5	0.24	2.75	252	46
Sample 2	37	230 ± 8	3.53 ± 0.03	220 ± 3	0.23	3.28	245	153
Sample 3	42	115 ± 11	1.84 ± 0.04	120 ± 5	0.22	3.54	210	164

examined the positive effect of surface oxidation on residual strength of porous Si_3N_4 ceramics preceded by thermal shock, especially for multiple cycles of thermal shock. Thus, understanding such influence mechanism is critical for reliability and optimization of a wide range of porous Si_3N_4 ceramics. In view of the above description, in this paper, the porous Si_3N_4 ceramics with porosities of 32%−42% were obtained through die-pressed under different pressures and gelcasting followed by gas-pressure sintering, and then were used to evaluate the thermal shock behavior with emphasis on the influence of surface oxidation on mechanical property. Our goal is twofold. One of the objectives is to investigate how the porosity affects both the crack initiation related to thermal shock fracture resistance and crack propagation concerning thermal shock damage resistance. Another objective is to determine the influence of oxide layer on the residual flexural strength of porous Si_3N_4 ceramics after multiple cycles of thermal shock with the aim of collecting some information on the thermal shock applications of porous Si_3N_4 ceramics.

2. Experimental

The initial raw materials employed in the present work were Si_3N_4 powders (average particle size: 0.37 μm, α phase > 95 wt%). Al_2O_3 (1 μm, 99% purity, 1 wt%) and Y_2O_3 (5 μm, 99.9% purity, 2 wt%) were used as the sintering additives. A mixture of α-Si_3N_4, Al_2O_3 and Y_2O_3 was ball-milled in alcohol for 24 h. After being dried and sieved through a 100-mesh screen, the resultant powder mixture was die-pressed at 35.7 and 10.7 MPa and then pressure-sintered for low and middle porosity Si_3N_4, respectively, or by gelcasting and subsequently gas-pressure sintering for high-porosity Si_3N_4. In all cases, sintering was conducted at 1800−1900 °C for 4 h with a heating rate of 10 °C/min under a nitrogen pressure of 0.4 MPa, utilizing Si_3N_4 powders as protecting particles to inhibit the decomposition and deformation. Testing specimens with dimensions of 3 mm × 4 mm × 40 mm were obtained through diamond cutting and ground mechanically with the tensile surface perpendicular to the axis direction. Flexural strength of samples was determined by three-point bending tests at room temperature with a span of 16 mm and a crosshead speed of 0.5 mm/min. The porosity of samples was determined by means of Archimedes displacement method, employing distilled water. Fracture toughness was measured by the single-edge notch beam (SENB) technique with a notch span of 3 mm and a height of 2.3 mm at a loading rate of 0.1 mm/min by precracking. The linear coefficient of thermal expansion was calculated from thermal expansion that measured by dilatometer up to 1000 °C at a heating rate of 5 °C/min in vacuum. Young's modulus and Poisson's ratio were determined by the uniaxial compression and pulse-echo methods, respectively. Table 1 lists the physical and mechanical properties of the investigated porous ceramics. Fracture surfaces of the samples were observed by scanning electron microscopy (SEM, Model JSM-7000F, JEOL, Japan). As shown in Fig. 1, fine elongated rod-like grains with the length of 10 μm and diameter of 1 μm are randomly oriented and jointed to form pores.

Thermal shock resistance experiments were performed by quenching the specimens (3 mm × 4 mm × 40 mm) from a resistance furnace into 20 °C water bath. The specimens were heated in air at a rate of 10 °C/min to a preset temperature and held at this temperature for about 20 min prior to quenching. The specimens were dropped parallel to their long axes into the water. Then, the residual flexural strength of the quenched specimens was measured under the same conditions as those of unquenched specimens. The strength results of specimens in this work are given as the average values of three measurements. The critical thermal shock temperature difference can be determined by ASTM standard C1525-04 to be the temperature at which 70% of the room temperature strength was still retained. Thermal shock damage resistance experiments were conducted by repeating the heating and quenching procedure, and the assessment of which can be realized via comparing the extent of damage of porous specimens after repeated thermal shock experiments.

3. Results and Discussion

3.1. Thermal shock behavior

Considerable efforts about thermal shock behaviors of porous Si_3N_4 ceramics have been devoted. In general, several thermal

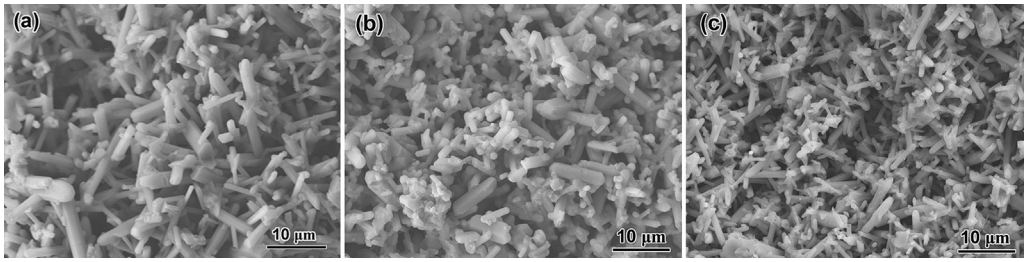

Fig. 1 SEM images of porous Si_3N_4 ceramics with the porosities of 32% (a), 37% (b), and 42% (c).

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