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Crack-healing ability and strength recovery of different hot-pressed TZ3Y20A–SiC ceramics by heat treatment in air

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ABSTRACT

Crack-healing ability and strength recovery of different $ZrO_2(Y_2O_3)$–Al_2O_3–SiC (TZ3Y20A–SiC) ceramics have been investigated by tailoring heat treatment temperature, heat treatment time, SiC content and crack size. Heat treatment in air enhances distinctly the flexural strength of TZ3Y20A–SiC ceramics. A complete crack-healing of TZ3Y20A–10 vol%SiC ceramic can be realized by optional heat treatments at 1073 K for 30 h, 1273 K for 10 h or 1373 K for 5 h, respectively. With increasing the content of nanosize SiC particles from 10 to 20 vol%, the crack-healing process of TZ3Y20A–SiC ceramics speeds up at identical temperature levels. The pre-existing cracks with a width up to 0.45 μm are healed completely under the optimum heat treatment conditions, and the flexural strengths of crack-healed TZ3Y20A–SiC ceramics are completely recovered to the value of smooth specimens. Crack closure and rebonding of crack wall due to the formation of SiO_2 glassy phase by oxidation reaction between SiC and the oxidizing atmosphere are dominant healing mechanisms in TZ3Y20A–SiC ceramics.

1. Introduction

Advanced ceramics have mechanical properties superior to metals at high temperatures. However, they are very brittle and susceptible to cracks. As a result, the structural reliability is low, which limits their industrial applications. It is well known that a flawed ceramic can recover its strength to some extent by suitable heat treatment. The strength recovery is caused by one of the following reasons: (1) by relieving the residual tensile stress, (2) by resintering the flaws, and (3) by crack-healing [1].

Generally, crack-healing sacrificial particles are divided into three categories as low-temperature, moderate-temperature, and high-temperature sacrificial particles according to the requirement of crack-healing temperature. Boron and boron-containing compounds have a low crack-healing temperature of about 973–1273 K. Oxides of boron bond strongly to the ceramic matrix and easily cause the crack blunting. However, boron oxide has a high volatilization rate above 1273 K, and is sensitive to moisture, which is unacceptable for high-temperature structural applications [2]. High temperature crack healing additives are $MoSi_2$ [3], $ZrSiO_4$ [4] and Y_2SiO_5 [5]. However, high thermal expansion coefficient of $MoSi_2$, high fabrication temperature and narrow healing temperature range of $ZrSiO_4$ and Y_2SiO_5 limit their applications as sacrificial particles.

Moderate-temperature crack-healing materials include SiC [6–9] and Si_3N_4 [10]. The oxidized products of SiC and Si_3N_4 contribute to the healing of pre-cracks, and have a good compatibility with ceramic matrix. In addition, the healing temperature of about 1273–1973 K is suitable for crack healing and strength recovery of ceramic matrix composites incorporated with SiC and Si_3N_4 particles.

Adding SiC particles into YSZ obtains high elastic modulus, fine grains, and enhanced toughening based on crack deflection and crack bridging [11–13]. However, there is no detailed report on the crack healing effect of SiC in YSZ ceramics. SiC has an excellent crack-healing ability since the oxidized product of SiO_2 fill the crack completely, and exhibit high strength as contrasted with the ceramic matrix. In addition, a large heat generation of 943 kJ/mol is beneficial for crack healing [14].

Strength recovery of specimens with a complete crack-healing ability is closely related to healing conditions on the strength of the healed zone, including chemical composition and crystalline state of the intergranular phase [7–9,15], heat treatment temperature and time [16–18], oxygen partial pressure [19–21], the crack size that can be completely healed [22,23], the self-healing particle size [14,24], threshold stress during crack healing treatment [14], high-temperature strength of crack-healed zones [25–29].

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In order to use the crack-healing ability of SiC effectively, the following topics should be studied: (1) the effect of healing condition on the strength of the crack-healed zone, (2) the effect of SiC content in matrix, and (3) the maximum size of pre-existing crack, which can be healed completely. For this purpose, a variety of composite ceramics were fabricated by hot pressing. The machined specimens were crack-healed under different heat treatment conditions. The flexural strengths of these crack-healed specimens were then evaluated at room temperature.

2. Experiment procedure

In the present work, the starting materials used are as follows: TZ3Y20A ceramic powder is a mixture of 80 wt.% YSZ (3 mol.% Y_2O_3 partially-stabilized zirconia) and 20 wt.% α-Al_2O_3 (Tosoh Corporation, Japan). Table 1 lists the chemical compositions of TZ3Y20A ceramic powder. Different volume fractions of β-SiC powder were then added into TZ3Y20A to form the composite powder. The mean particle diameters of both TZ3Y20A and β-SiC powders were about 100 nm. Alcohol was added into the powder mixture and was blended for 24 h using ZrO_2 grinding balls. After the ball-milled slurry was dried, the mixture was subsequently hot-pressed in vacuum for 1 h under 30 MPa at sintering temperatures of 1773 and 1873 K, respectively, to form dense TZ3Y20A–SiC ceramics with different SiC contents. Chemical compositions and processing conditions are presented in Table 2.

The hot-pressed samples were cut into 3 mm × 4 mm × 20 mm rectangular bar specimens. The specimens were polished to a mirror finish on one face and the edges of the specimens were beveled to prevent fracturing from edge cracks. For strength testing, a Vickers hardness tester (HVS-30 type) was used to make the indents with different surface cracks at the center of the polished face of a bar specimen under the loading conditions of 49 N/15 s, 49 N/60 s, 98 N/15 s and 196 N/15 s, respectively. The indentation will introduce a crack of 120–350 μm in length and 0.25–0.61 μm in the corresponding width at the center of the polished surface, as shown in Fig. 1 and Table 2.

Heat treatment experiments of TZ3Y20A–SiC specimens with pre-existing cracks were performed at different temperatures of 1073–1373 K for 1–30 h in air for crack-healing, respectively, followed by spontaneous furnace cooling. As contrasted with Al_2O_3 or mullite based ceramics, the sintering temperature of ZrO_2 based ceramic is relatively low, and therefore, crack-healing of ZrO_2–SiC based ceramic occur at relatively low temperatures. ZrO_2–SiC–TiO_2 ceramic could completely recover at 1073–1173 K [17], while the crack-healing of ZrO_2–SiC ceramic occurs at even 873–1073 K [30]. Therefore, the crack healing temperature as low as 1073–1373 K in the present work is beneficial for high-temperature structural applications of self-healing TZ3Y20A–SiC ceramics. The crack-healed specimens were subsequently tested on three-point bending tester (Instron-5569 universe tester, USA) at a crosshead speed of 0.5 mm/min, by using a fixture with a span of 16 mm. To investigate the mechanism of crack healing, the specimen surfaces were analyzed by EMS (SEM Helios Nanolab600i, FEI, USA).

Table 1
Chemical compositions of nanoscale TZ3Y20A ceramic powder (wt.%).

ZrO_2	Y_2O_3	Al_2O_3	SiO_2	Fe_2O_3	Na_2O	Other
73.97	3.97	21.27	0.005	0.002	0.019	0.70

3. Results and discussion

3.1. Effect of heat treatment temperature and time on crack healing

Fig. 2 shows the variations in flexural strength of TZ3Y20A–10 vol.%SiC ceramics with a pre-existing crack length of 120 μm and a width of 0.25 μm as a function of heat treatment time at different temperatures. For comparison, the strength data of smooth specimens without pre-existing cracks and the crack specimens without heat treatment are also shown in Fig. 2. For a complete strength recovery, flexural strength of pre-cracked samples after heat-treatment is recovered up to that of the corresponding smooth samples, in addition, crack closure and rebonding of crack surface occur along the entire length of cracks. The average strength of smooth specimens is 995 MPa, however, the strength of the cracked specimens is only one third of the former. After suitable heat treatments at temperatures of 1073–1373 K, the specimen with pre-existing cracks recovers its strength similar to that of the smooth specimen, and in some cases surpasses it, as shown in Fig. 2. Moreover, the crack healing time for a complete strength recovery depends mainly upon the healing temperature. Based on the room temperature strengths of both smooth specimens and the crack specimens after heat treatments in Fig. 2, the optimum self-healing condition of the crack specimens is by heat-treatment at 1073 K for 30 h, 1273 K for 10 h and 1373 K for 5 h, respectively. However, further prolonging the crack-healing time at temperatures of 1273 and 1373 K will cause the grain coarsening and leads to the performance degradation.

Fig. 3 shows the XRD patterns of hot-pressed TZ3Y20A–10 vol.%SiC ceramics after heat treatments at 1373 K for different time. The oxidized products of β-SiC include two phases of a glassy phase and a crystalline SiO_2. The glass phase that form at relatively low temperature or short healing time during heat treatment contributes to the healing of pre-cracks. However, excess crack-healing time over the optimum condition will cause the performance degradation. The glassy phase transforms to the crystalline phase α-SiO_2, and then partial crystalline α-SiO_2 reacts with Y_2O_3 to generate γ-$Y_2Si_2O_7$ with further enhancing temperature or prolonging the healing time during heat treatment. This is confirmed by XRD analysis of self-healed specimens as shown in Fig. 3.The crack-healing reactions of SiC in TZ3Y20A–SiC ceramics at elevated temperatures can be achieved by the following chemical reactions:

$$SiC + 3/2O_2 = SiO_2 + CO(g) + 943\ kJ/mol \tag{1}$$

$$SiC + SiO_2 + Y_2O_3 + 2O_2 = Y_2Si_2O_7 + CO_2(g) \tag{2}$$

Therefore, the heat treatment temperature and time have a significant influence on the degree of crack healing and strength recovery.

From the results of flexural strength of the crack-healed samples, the lowest healing temperatures (T_H) are obtained for each healing time (t_{HMin}), in short, it is 1073 K for 30 h, 1273 K for 10 h and 1373 K for 5 h, respectively. The Arrhenius plots of five kinds of self-healing ceramics are shown in Fig. 4. The symbol (☆) indicates the results of TZ3Y20A–10 vol.%SiC ceramic, while the symbols (□), (○), (△) and (◇) show the results of ZrO_2/SiC [30], Si_3N_4/SiC/Y_2O_3 [8], mullite/SiC [8] and Al_2O_3/SiC [8], respectively.

The activation energy of TZ3Y20A–10 vol.%SiC ceramic calculated using Eq. (1) is given in Fig. 4. The Q_{aH} values of ZrO_2/SiC, Si_3N_4/SiC/Y_2O_3, mullite/SiC and Al_2O_3/SiC composites are 116 kJ/mol, 277 kJ/mol, 413 kJ/mol and 334 kJ/mol, respectively. Clearly, TZ3Y20A–10 vol.%SiC ceramic has a Q_{aH} value of 96 kJ/mol, which is much lower than other self-healing composites as shown in Fig. 4. Therefore, it is considered that the TZ3Y20A–10 vol.%SiC ceramic has a high crack-healing ability as compared with other