ENGINEERED CERAMICS . . . [II]

Y-PSZ/Al_2O_3 (Composites between Yttria Partially Stabilized Zirconia and Alumina)......Mechanical Property

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The temperature dependence of bending strength, fracture toughness and Young's modulus of the composite materials fabricated in $ZrO_2(Y_2O_3)$-Al_2O_3 system was examined. The addition of Al_2O_3 led to enhancement of high temperature strength. The composite material, 60wt% ZrO_2 (2 mol% Y_2O_3)/40wt% Al_2O_3, fabricated by hot-isostatic pressing exhibited extremely high strength of 1000MPa at 1000 °C.

I. Introduction

Y_2O_3-partially stabilized ZrO_2(Y-PSZ) has attracted considrerable interest as the new strautural material, because it exhibits high toughness and high strength[1,2]. These features are attributed to the stress-induced transformation of tetragonal phase. However, the strength and toughness of Y-PSZ decrease remarkably with increasing temperature. The decrease of strength and fracture toughness at high temperature was also observed for zirconia toughened ceramics such as Mg-PSZ and Al_2O_3/ZrO_2[3~5]. According to the theory of transformation toughening, the low values of high temperature strength are due to the decrease of contribution of stress-induced transformation toughening. Some approaches were proposed to improve the high temperature mechanical properties of zirconia toughened ceramics[6]. We thought that one way to improve high temperature strength is to reinforce the toughened zirconia with addition of another ceramic compenents. We reported previously that the room temperature strength of Y-PSZ was greatly enhanced by the large amount of Al_2O_3 addition and the application of hot-isostatic pressing. We report here the temperature dependence of strength and fracture toughness of these composite materials.

II. Experimental Procedure

The submicron $ZrO_2(+Y_2O_3)$ powder* and the composite powders $ZrO_2(+Y_2O_3)/Al_2O_3$** were used as a starting material. The compositions of these powders were as follows, ZrO_2+2 mol% Y_2O_3(2Y), 90wt% 2Y+10wt% Al_2O_3(2Y10A), 80wt% 2Y+20wt% Al_2O_3(2Y20A)and 60 wt% 2Y+40wt% Al_2O_3(2Y40A). These powders were uniaxally pressed into plate at 40MPa, and then isostatically cold-pressed at 300MPa.

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The green compacts were sintered at 1400–1450°C for 2 h in air to obtain the pre-sintered bodies. The hot-isostatic pressing was performed using the pre-sintered bodies at 1500°C and 100MPa for 0.5 h in Ar gas. The sintered bodies used for bending tests bar fired at 1500°C for 1 h.

The measurements of three point bending strength were performed using a 30mm span and a crosshead speed of 0.5mm/min. The size of samples was 3 mm height, 4 mm width, and 40 mm length.

The temperature dependence of bending strength was measured in temperature range from room temperature to 1000°C. The bending test was carried out after the sampeles were kept for 0.5–5 h at each temperature. Four to six bars of each sample were tested at each temperature. The temperature dependence of Young's modulus and internal friction were measured by the flexural resonant frequency technique. The arrangement of apparatus was described in previous report⁷⁾. The size of test bar was 1 mm thick, 10 mm width and 43 mm length. The speed of heating was 4°C/min. The resonant frequency was 3.6 KHZ. Young's modulus was calculated from the resonant frequency on the basis of the formula reported by Föster⁸⁾. The internal friction was determined by the free-decay method.

Fracture toughness was evaluated by single edge notched beam technique. Single notches were machined with a 0.1 mm thick diamond saw. The notched specimens were annealed at 1000°C in air in order to relieve the residual stress introduced by the saw. Fracture toughness measurements were performed by three point bending test using a cross head speed of 0.1mm/min. In high temperature range, the tests were carried our after the specimens kept for 0.5 h at each temperature.

III. Results and Discussion

The temperature dependence of bending strength of hot-isostatic pressed materials, 2Y and 2Y/Al_2O_3 is shown in **Fig. 1**. The hot-isostatic pressed 2Y/Al_2O_3 exhibited extremely high bending strength more than 2000MPa at room temperature, which has been described in previous report⁹⁾. As shown in Fig. 1, Al_2O_3 addition was also effective to enhance strength at high temperature. The bending strength of all samples decreased drastically when temperature increases up to 400°C, and showed a little increase of strength at 600°C. This feature in temperture dependence of strength was characteristic for hot-isostatic pressed materials, and was also observed for Y_2O_3-rich composite materials, 3Y and 3.85Y containing 20–40wt% Al_2O_3. In temperature range 600–1000°C, the strength of samples

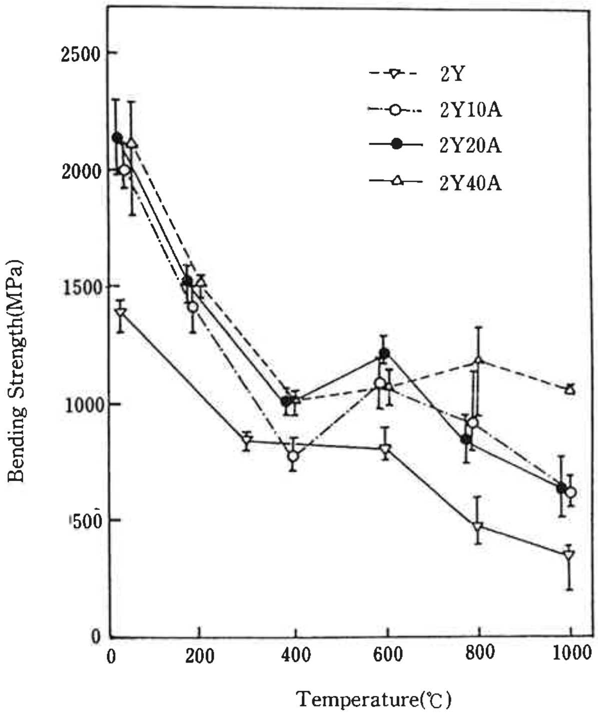


Fig. 1 Temperature dependence of bending strength of hot-isostatic pressed $ZrO_2(2mol\%Y_2O_3)/Al_2O_3$ composite materials.
(Error bars indicate the maximum and minimum value of strength.)

without and with small ammount of Al_2O_3 such as 2Y, 2Y10A and 2Y20A decreased gradually with increasing temperature. Whereas, The sample containing large amunt of Al_2O_3(2Y40A) showed nearly constant and high strength. The strength of this sample remained 1000MPa at 1000°C. This result indicated that the large amount of Al_2O_3 addition contributes to the enhancement of strength at temperature 600–1000°C. Temperature dependence of fracture toughness was examined for hot-isostatic pressed material, 2Y20A. The result is shown in **Fig. 2**. The fracture toughness decreased drastically with temperature increases up to 400°C, and increased a little at 400°C to 600°C. Above 800°C, the fracture toughness decreased gradually with increasing temperature. It was found that fracture toughness has a similar temperature dependence of strength.

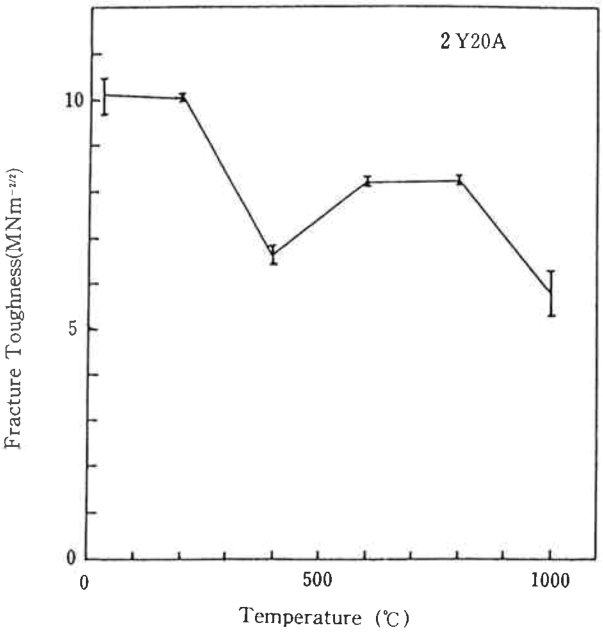


Fig. 2 Temperature dependence of fracture toughness of hot-isostatic pressed composite material, 80tw% ZrO_2(2mol% Y_2O_3)/20mol% Al_2O_3(2Y20A).
(Error bars indicate the maximum and minimum value of fracture toughness.)

It is expected that the drastic decrease of strength and fracture toughness observed at room temperature to 400°C is due to the reduction in the contribution of phase transformation toughening. According to the theory of transformation toughening[53], the energy absorbed by phase transformation decreases with increasing temperature because of reduction of differential free energy between tetragonal and monoclinic phase. It is considered that the minimum

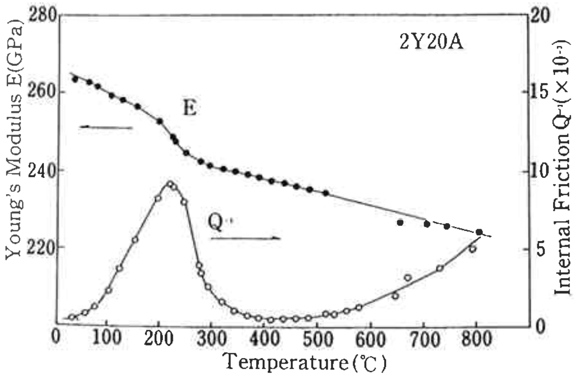


Fig. 3 Temperature dependence of Young's modulus and internal friction of hot-isostatic pressed composite material, 80wt% ZrO_2 (2mol% Y_2O_3)/20wt% Al_2O_3(2Y20A).

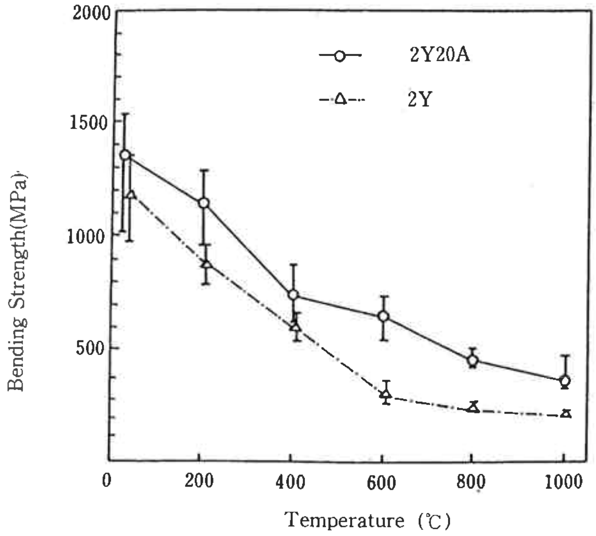
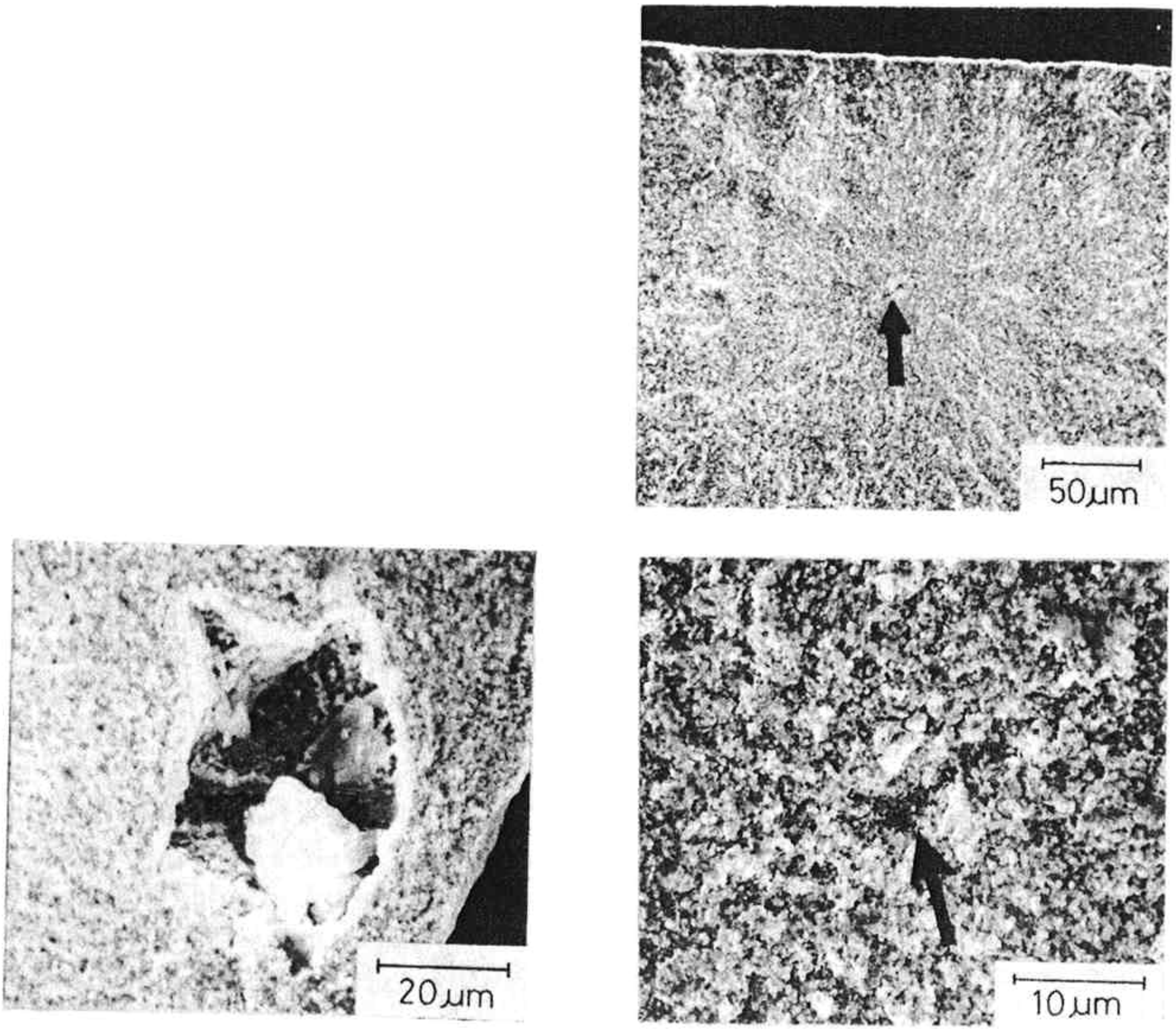


Fig. 4 Temperature dependence of bending strength of sintered materials, ZrO_2(2mol% Y_2O_3) (2Y) and 80wt% ZrO_2(2mol% Y_2O_3)/20wt% Al_2O_3(2Y20A).
(sintered; 1500°C, 1h)

strength and fracture toughness at 400°C are attributed to the disappearance of phase transformation. However an increase of strength and toughness observed at 600°C can not be understood by the concept of transformation toughening. It is not clear why strength and toughness increase at temperature between 400°C and 600°C. Ingel et al.[10)] reported on the strength and toughness of Y_2O_3-partially stabilized ZrO_2 single crystal. The strength of single crystal decreased drastically up to 500°C, and remained well above 500°C. This result is similar to the present one of $ZrO_2(Y_2O_3)/Al_2O_3$ composite materials.

The temperature dependence of Young's modulus and internal friction of hot-isostatic pressed material, 2Y20A is shown in **Fig. 3**. Young's modulus decreased with temperature increases at room temperature to 1000°C. It is noticeable that the precipitous drop of Young's modulus appeared at room temperature to 400°C. The increase of internal friction was observed at



(A) (B)

Fig. 5 Fracture origins in the sintered and hot-isostatic pressed materials, 80wt% ZrO_2 (2mol% Y_2O_3)/20wt% Al_2O_3(2Y20A).
((A); the sample sintered at 1500°C for 1h.
(B); the sample hot isostatically pressed at 1500°C and 100 MPa for 0.5h.
Arrows indicate the position of fracture origin.)

room temperature to 400°C and above 600°C. The previous studies reported that the steep drop of Young's modulus and internal friction peak were observed up to 400°C for Y_2O_3-partially stabilized ZrO_2 ceramics[7] and Praseodymium stabilized ZrO_2 and HfO_2 ceramics[11]. It is considered presumably that this phenomena is resulted from the interaction between the stabilizer cation defects and the oxgen vacancies. It is probable that not only disappearance of phase transformation but also the steep drop of Young's modulus is responsible for the drastic decrease of strength and fracture toughness.

The temperature dependence of bending strength of sintered materials, 2Y and 2Y20A is shown in **Fig. 4**. The strength of both materials decreased gradually with increasing temperaturre. The strength of sintered materials was not so susceptible to temperature up to 600°C as that of hot-isostatic pressed materials.

The Al_2O_3 addition gave the enhancement of strength of sintered material at room temperature to 1000°C. However, the strength obtained for sintered sample 2Y20A was considerably lower than that of hot-isostatic pressed material. The fracture origins of both sintered and hot-isostatic pressed materials, 2Y20A are shown in **Fig. 5**.

The large pore, as shown in Fig. 5 (A), was often observed at fracture origin of the sintered body, but on the other hand, the fracture origin of hot-isostatic pressed body did not include large pores. The typical fracture origin of hot-isostatic pressed materials was illustrated in Fig. 5 (B). In many cases, the size of defect was small less than about 10μm. Fractograpy observations revealed that the largely enhanced strength in hot-isostatic pressed materials is resulted from the decrease in size of defect.

IV. Conclusions

As a result of the study of ZrO_2 (2 mol% Y_2O_3)/Al_2O_3 composites, it was found that the addition of Al_2O_3 results in the enhanced strength at room temperature to 1000°C. Especially, hot-isostatic pressed composite materials exhibited extremely high strength at high temperature. At room temperature to 400°C, the abnormally decrease in strength, fracture toughness and Young's modulus were found for the materials fabricated by hot-isostatic pressing.

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