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Fracture strength and damage resistance of slip cast Y-TZP/Ce-TZP layered composites

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Asymmetric, three- and symmetric five-layer Y-TZP/Ce-TZP composites have been prepared by sequential slip casting and pressureless sintering at 1400–1600°C in air. The three-layer material sintered at 1500°C showed the maximum fracture strength (534 MPa), measured by a diametral compression test and failed by the triple-cleft mode of fracture. Contact damage resistance was superior in three-layer composite compared with five-layer, possibly due to the development of relatively large residual compressive stress.

1. Introduction

Ceria-doped tetragonal zirconia polycrystals (Ce-TZP) when compared with yttria-doped tetragonal zirconia (Y-TZP), have been shown to exhibit superior toughness together with good thermal and chemical stability, but low fracture strength [1, 2]. In order to improve mechanical properties of ceramics for structural applications, a great deal of attention has recently been concentrated on preparation of layered composites [3–10]. Layered TZP composites can demonstrate enhanced mechanical properties compared with monolithic material, resulting from the presence of residual compressive stress generated not only by crack deflection at interface but also by the t-ZrO_2 phase transformation to m-ZrO_2. Layered ceramic composites containing Y-TZP or Ce-TZP, as a matrix or second phase material, have been widely explored [5, 6, 11]. However, very little literature on Y-TZP/Ce-TZP layered composites has been reported. In the present work, asymmetric three- and symmetric five-layer Y-TZP/Ce-TZP composites have been fabricated by sequential slip casting, and their mechanical properties investigated.

2. Experimental procedure

Two ZrO_2 starting powders produced by Tosho Co. (Japan) were used: 3 mol% Y_2O_3-ZrO_2 (Y-TZP) with $d_{50} = 0.5$ μm, $S_{BET} = 7.2$ m^2/g and 12 mol% CeO_2-ZrO_2 (Ce-TZP) with $d_{50} = 0.4$ μm, $S_{BET} = 7.7$ m^2/g.

Aqueous Y-TZP, Ce-TZP and 50/50 vol% Y-TZP/Ce-TZP slips with a solids loading of 15 vol% were made using a colloidal processing technique. This involved the control of pH and apparent viscosity using 0.3 wt% DARVAN-C (R. T. Vanderbilt) and NH_4OH (28%, Yakuri Pure Chemicals Co., Japan).

The asymmetric three-layer and symmetric five-layer stacks were fabricated by sequential slip casting in the sequence of Ce-TZP (layer 'A'), Y-TZP/Ce-TZP (layer 'B') and Y-TZP (layer 'C') slips, into two different shaped plaster of Paris molds, a cylindrical section (12.7 mm dia.) and a rectangular section (50 mm × 25 mm). The green bodies were then dried for seven days in air, followed by 3 days at 40°C until there was no longer a change in weight at 100°C. The layered green compacts were fired at 1400–1600°C for 2 h, with a heating and cooling rate of 3°C/min.

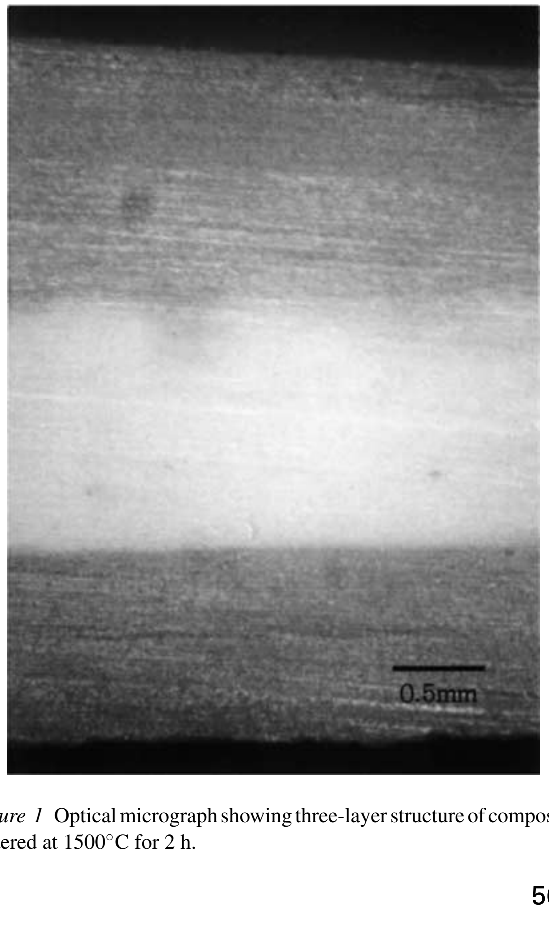

Figure 1 Optical micrograph showing three-layer structure of composite sintered at 1500°C for 2 h.

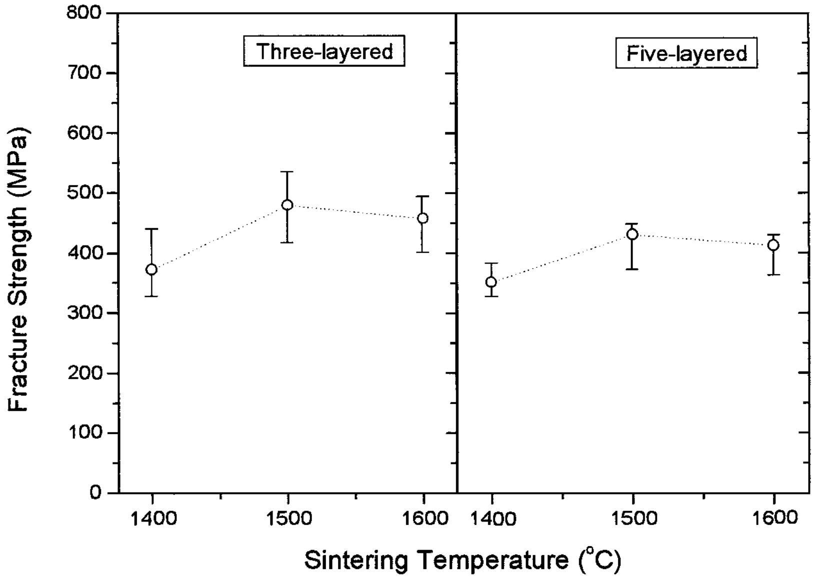

Figure 2 Fracture strength measured by diametral compression disc test for three- and five-layer composites as a function of sintering temperature.

The layered structure of the densified body was optically examined with a stereoscopic microscope (WILD M10, Leica). The sintered bulk density was measured by water immersion method and crystalline phases present were identified by XRD (D-MAX 1400, Rigaku). The microstructures of fracture surfaces were examined using SEM (JSM-840A, Jeol). The fracture strength was measured for five cylindrical specimens using a Universal Test Machine (model 6025, Instron) with a constant crosshead speed of 0.5 mm/min, and calculated using the appropriate equation for the diametral compression disc test:

$$\sigma_f = 2P/\pi dt \qquad (1)$$

where σ_f is the fracture strength (MPa), P is the load at fracture, d and t are the diameter and thickness of the specimen respectively. To evaluate the damage resistance of sintered monolithic and layered specimens, indentation strength was measured with three ground and polished bars (3 mm $\times$ 4 mm $\times$ 40 mm) by four-point bending, using an inner span of 10 mm, outer span of 20 mm and crosshead speed of 0.5 mm/min. The specimens were indented at the center of the layer 'C' side in the three-layer material and the layer 'A' side in five-layer material using a Vickers indenter under a load ranging from 9.8 to 294 N, and with the contacting time of the indenter to the specimen surface being kept constant at ~25 s. The fracture toughness of each monolithic material sintered at 1500°C was evaluated by the indentation technique [12] (136° Vickers indenter, 196 N, 10 s).

3. Results and discussion

Y-TZP/Ce-TZP composites consisting of layers with thickness values in the range 1 to 1.4 mm were fabricated (Fig. 1). The layer 'C' consisted of small grains (<0.3 μm), compared with the layer 'A' (<2.2 μm) [13]. By means of XRD analysis, tetragonal and minor

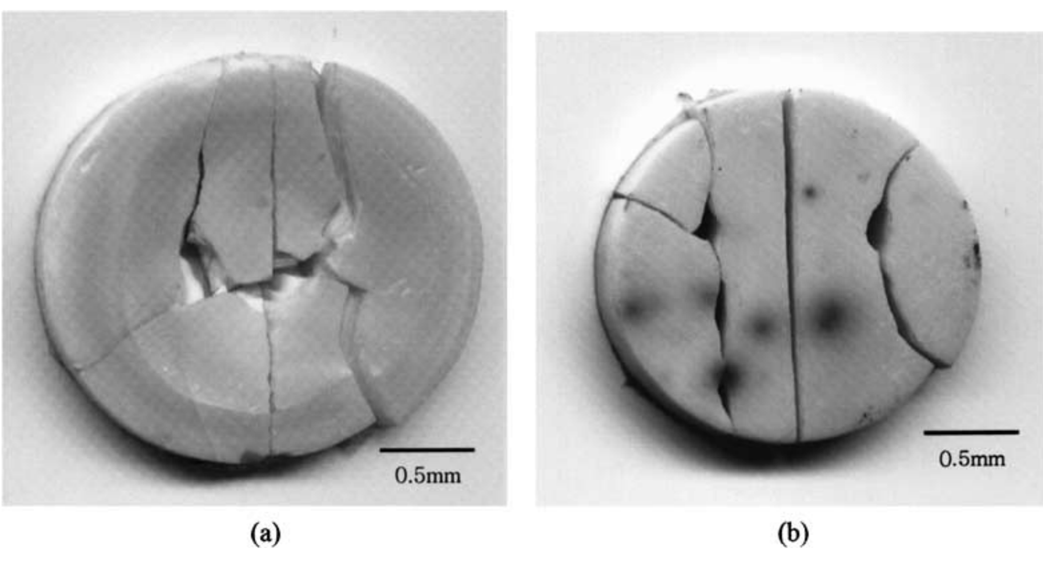

Figure 3 Fractures observed in (a) five-layer composite sintered at 1400°C and (b) three-layer composite sintered at 1500°C.

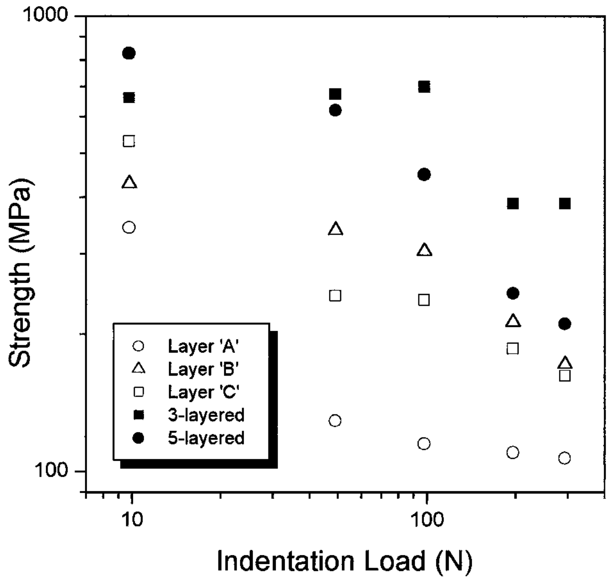

Figure 4 Bend strength of monolithic and layered materials sintered at 1500°C after indentation at different loads.

monoclinic peaks were confirmed in the specimens regardless of the sintering temperature. The sintered bulk densities were 94.7 (1400°C), 98.7 (1500°C) and 98.1% (1600°C) of the theoretical density. Fig. 2 shows the mean fracture strength for sintered three- and five-layer materials, measured by the diametral compression disc test. The specimens sintered at 1500°C exhibited high fracture strength (>400 MPa), due to high densification and small grain size (<0.7 μm). At the same sintering temperature, the fracture strength of three-layer material was somewhat higher (20–50 MPa) than that of five-layer material. The typical fracture pattern of layered materials was triple-cleft [14], as can be seen from the cracking that occurred on samples that had split in half in the tensile failure (Fig. 3).

The average indentation bend strength of the monolithic and layered materials, sintered at 1500°C is shown in Fig. 4. In monolithic indented with a low load (9.8 N), layer 'C' material exhibited higher strength (531 MPa) than layer 'A' and 'B' material. With increasing indent load to 98 N, however, the degree of strength reduction was small in layer 'B' material (29%), compared with layer 'A' (66%) and layer 'C' material (55%). After indentation with a load of 49 N, the strength of monolithic specimen was only 129–339 MPa whereas that of layered specimen was 620–674 MPa. At low indent load (9.8 N), the strength of five-layer specimen was higher than that (661 MPa) of three-layer composite. When indentation load was increased to 98 N, however, five-layer specimen exhibited a sudden reduction (~56%) in strength but a corresponding alteration of strength in three-layer was not observed. At even higher (294 N) indent loads, the three-layer specimen still retained a high strength (388 MPa). A continuous reduction of strength in the monolithic specimen with increasing indentation load strongly implies that the crack introduced by the

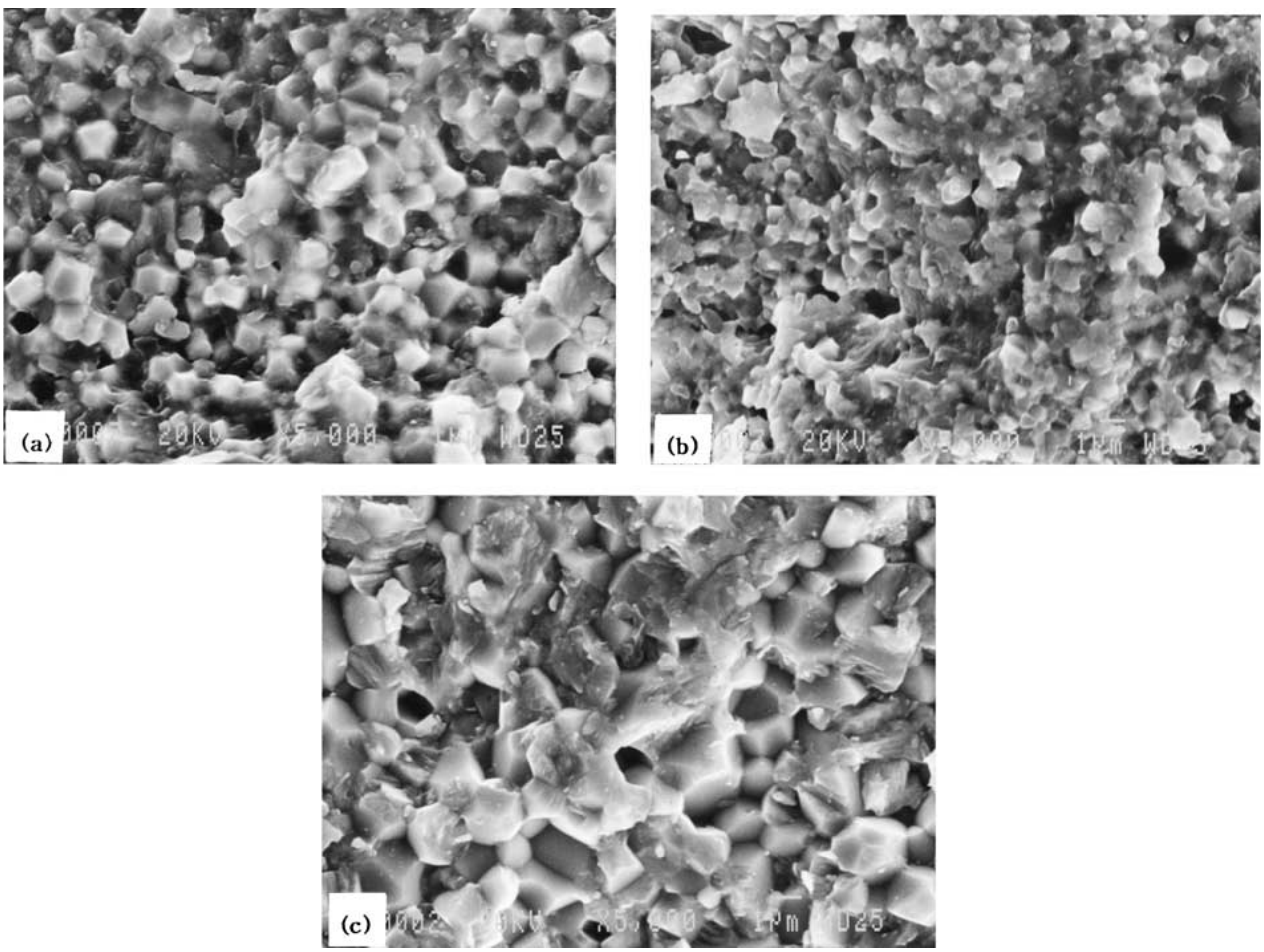

Figure 5 Fracture surfaces of (a) layer 'A' in three-layer composite indented at 49 N load, after sintering at 1500°C, (b) layer. 'C' in five-layer indented at 294 N load, after sintering at 1500°C and (c) layer 'B' in five-layer indented at 196 N load, after sintering at 1600°C.

indent is acting as the origin for failure. However, the retention of high strength in the layered specimens even at high indent loads suggests the possibility of strength-limiting flaws other than those introduced by the indents existing or being developed, and acting as the fracture origin [10]. An alternative explanation is that the layered structure acts to neutralize the indentation flaw, for example by redirecting it along the layer interface, thus allowing a different population of flaws to act as the failure source.

The strength of an indented, surface stress-free material (σ_f^o) is given approximately by [15]

$$\sigma_f^o = 2K_{IC}^{4/3}(H/E)^{1/6}P^{-1/3} \quad (2)$$

where K_{IC} is the fracture toughness, H the hardness, E the Young's modulus, and P the indentation load. Equation 2 implies that the strength reduction associated with contact damage is less sensitive to the damage in high toughness material. In spite of the lower toughness of layer 'C' material (7.7 $MPa \cdot m^{1/2}$) compared to layer 'A' material (13.1 $MPa \cdot m^{1/2}$), the damage resistance of the asymmetric three-layer specimen is higher than the symmetric five-layer body. It is assumed that this higher damage resistance is due to the residual compression stress that is produced in the outer layer 'C' (Y-TZP) during cooling from sintering temperature. Y-TZP ($\alpha = 10.6 \times 10^{-6}\ K^{-1}$) has a lower coefficient of thermal expansion than Ce-TZP ($\alpha = 11.5 \times 10^{-6}\ K^{-1}$) [16, 17] and Y-TZP/Ce-TZP.

Intergranular fracture was observed in the sintered layered materials (Fig. 5). After sintering at 1600°C, the five-layer specimen indented at 196 N consisted of relatively large grains which were seen to be strongly interlocked, and it was these which exhibited the partial intragranular fracture and drop out of grains (Fig. 5c).

4. Conclusions

An asymmetric three-layer Y-TZP/Ce-TZP composite fabricated by sequential slip casting and having an indented Y-TZP surface layer, exhibited superior damage resistance when compared with a symmetric five-layer composite with an indented Ce-TZP surface layer and also with monolithic blocks of each material. The diametral compression test for the layered composites confirmed the occurrence of the triple-cleft fracture mode and the three-layer material exhibited higher fracture strength compared to the five-layer material.

Acknowledgements

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