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Correlation between Microstructure, Phase Transformation during Fracture and the Mechanical Properties of Y–TZP Ceramics

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Abstract

The mechanical properties of Y–TZP(yttria-stabilized tetragonal zirconia polycrystalline) materials with different microstructures have been studied and related to the phase transformation capability during fracture. It has been found that the microstructure of the materials affects the phase composition and also the phase transformation volume (tetragonal to monoclinic) on the fracture surface during fracture. Higher densities lead to higher phase transformation volume; and both of them contribute to higher fracture toughness and strength.

Introduction

The relation between mechanical properties and microstructure is fundamental for structural ceramics, and also one of the most interesting and extensively studied themes.[1-4] The presence of large defects (pores, flaws, etc.) in the microstructure degrades the mechanical properties of the materials, for example, the strength. Although many relations have been suggested between the fracture strength (σ_f) and the relative density (ρ) of a ceramic, commonly this relation is written as:[5]

$$\sigma_f = \sigma_o \exp[-b(1-\rho)] \qquad (1)$$

where b is a constant, σ_f, σ_o are the material strength at $\rho = \rho$ and $\rho = 100\%$, respectively.

For phase transformation toughened Y–TZP ceramics, the phase transformation in the front (or vicinity) area of a crack tip is also a critical factor for the enhancement of the fracture toughness. A higher phase transformation volume from tetragonal to monoclinic will lead to higher fracture toughness.[6-8] Factors affecting the phase transformation volume are the grain size of the tetragonal phase,[8-11] the amount of stabilizer,[12-14] and the restraining conditions.[15-16] However, in investigating the effect of these parameters on the phase transformation, dense materials (often manufactured by hot-pressing or hot isostatic pressing) have commonly been used, and the effect of density has been surprisingly ignored. This paper investigates the relation between density and phase transformation behavior and the effects of both of them on the mechanical properties of pressurelessly sintered Y–TZP ceramics.

Experimental Procedure

Materials preparation

The Y–TZP powders were first prepared via coprecipitation. For the preparation of the mixed solutions of zirconium oxychloride ($ZrOCl_2.8H_2O$) and yttrium chloride ($YCl_3.8H_2O$), the Zr^{4+}/Y^{3+} ratio was controlled to give an yttria content of 3 mol% in the final powders. The mixed solutions of different concentrations were added to ammonia solution of concentration of 12 vol%, and the coprecipitates were obtained. The pH value was maintained above 9·5 during the coprecipitate. The coprecipitates were washed with distilled water to remove Cl^- until Cl^- contents in the filtrates was lower than 2–40 ppm. The coprecipitates were then dried in an oven overnight at 110°C, and finally calcined at 750°C for 120 min. The resulting powders were composed mainly of tetragonal zirconia with no monoclinic phase.

PVA(polyvinyl alcohol) was used as a binder. Powders were compacted by uniaxial pressing at 50 MPa and then by a cold isostatic pressing at 250 MPa. The compacts were presintered at 750°C for 120 min to burn out the binders and then sintered at 1450 or 1550°C for 120 min with a heating rate of 3°C/min. To maintain the homogeneity of the temperature distribution and to prevent evaporation of components from the sample surface, the green bodies were embedded for sintering in powders of the same composition.

Property measurement

The densities of the sintered samples were measured by the Archimedes method in distilled water. The fracture strength was determined by the three point bend method with a load speed of 0·5 mm/min with samples of 2·5 × 5 × 26 mm, and a testing span of 20 mm. Fracture toughness was measured by the single edge notched beam (SENB) method with a loading speed of 0·05 mm/min. The notch width was 0·2 mm and notch depth was 2·5 mm on 2·5 × 5 × 26 mm. The strength and toughness measurement was made on an Instron 1195 materials tester. The Vickers hardness of the sintered bodies was measured on an Akashi-AVK-A micro-hardness tester with a constant punch load of 98 N.

Determination of the phase compositions

The phase compositions of the sintered bodies were determined by X-ray diffraction (Rigaku Denki, RAX-10) with the tetragonal content being given by:

$$t(\%)=I_{t(111)}/[I_{t(111)}+I_{m(111)}+I_{m(11\bar{1})}] \qquad (2)$$

where $I_{t(111)}$, $I_{m(111)}$ and $I_{m(11\bar{1})}$ are the diffraction line intensities of the tetragonal (111), monoclinic (111) and monoclinic (11$\bar{1}$) lattice places. The samples were ground to remove a surface layer of 0·5–1 mm in depth, and then polished with 0·5 μm diamond particles. The phase compositions of the sintered samples were determined on the polished surface. X-ray diffraction was also conducted on newly fractured surfaces. The difference between the tetragonal contents of the polished surfaces and the fractured surfaces is taken as a measure of the phase transformation volume during fracture.

Results

Relation between the mechanical properties and the sintered densities

Figures 1, 2 and 3 show the dependence of fracture strength (σ_f), fracture toughness (K_{Ic}) and Vickers hardness (H_v) on sintered density. The relation between Vickers hardness and relative density is approximately linear:

$$H_v\,(\mathrm{GPa}) = 11{\cdot}43-0{\cdot}439(1-\rho) \qquad (3)$$

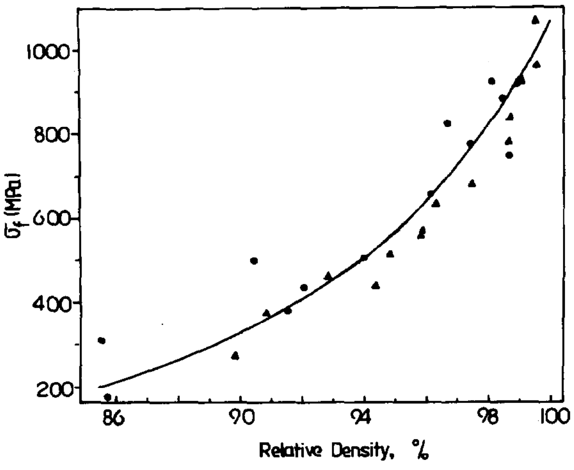

Fig. 1. Density dependence of the fracture strength (σ_f) of sintered 3Y–TZP bodies at (●) 1450°C, and (△) 1550°C.

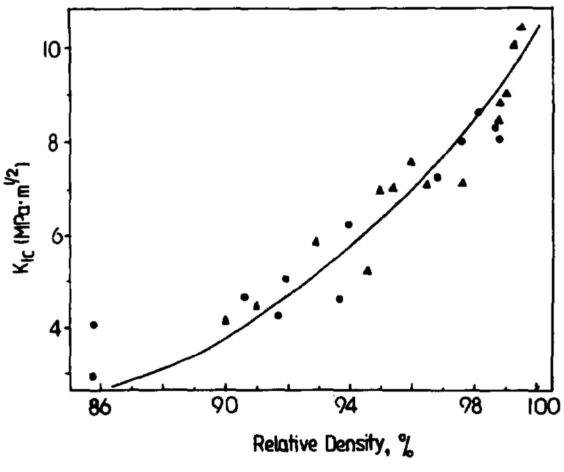

Fig. 2. Density dependence of the fracture toughness (K_{Ic}) of sintered 3Y–TZP bodies at (●) 1450°C, and (△) 1550°C.

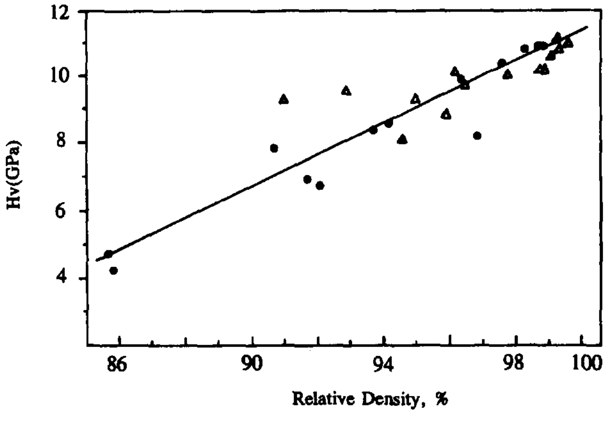

Fig. 3. Density dependence of the Vickers hardness (H_v) of sintered 3Y–TZP bodies at (●) 1450°C, and (△) 1550°C.

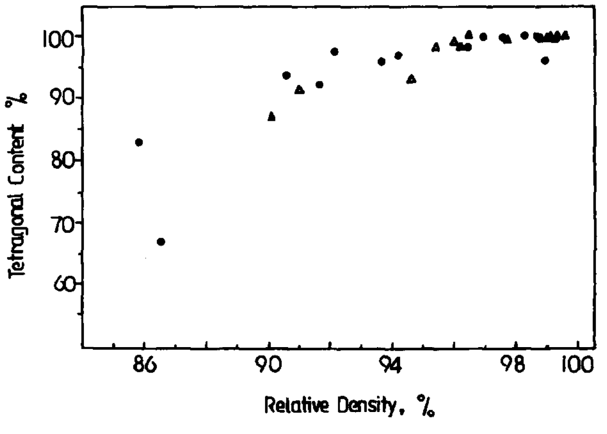

Fig. 4. Content of tetragonal phase (t%) on polished surfaces of sintered 3Y-TZP bodies versus density at (●) 1450°C, and (△) 1550°C.

The fracture strength obeys eqn (1) with:

$$\sigma_f(\text{MPa}) = 1045\exp[-0{\cdot}116(1-\rho)] \qquad (4)$$

The results for fracture toughness follow eqn (1) also satisfactorily with:

$$K_{Ic}(\text{MPa m}^{1/2}) = 10{\cdot}4\exp[-0{\cdot}10(1-\rho)] \qquad (5)$$

The relation fits least well in the lower density region.

The effect of density on phase composition and phase transformation volume

Generally there is no monoclinic phase in high density 3Y–TZP ceramics pressurelessly sintered at or below 1550°C; however, if the sintered density is low, the sintered bodies may contain some monoclinic phase, as illustrated in Fig. 4. The sintered density also affects the phase transformation volume during fracture, as shown in Fig. 5. In addition, sintering temperature also affects the phase composition and the phase transformation volume: at densities of ≥96%, higher sintering temperature leads to lower tetragonal content and higher phase transformation volume. This is due to the larger grain size at the higher sintering temperature, which results in greater instability of the tetragonal phase.[12]

Once chemical compositions and grain size are fixed, the main factor affecting the tetragonal stability is the restraint imposed on the grains by the surrounding material. Lower sintered densities result in lower restraining force and the tetragonal phase content is consequently lower.

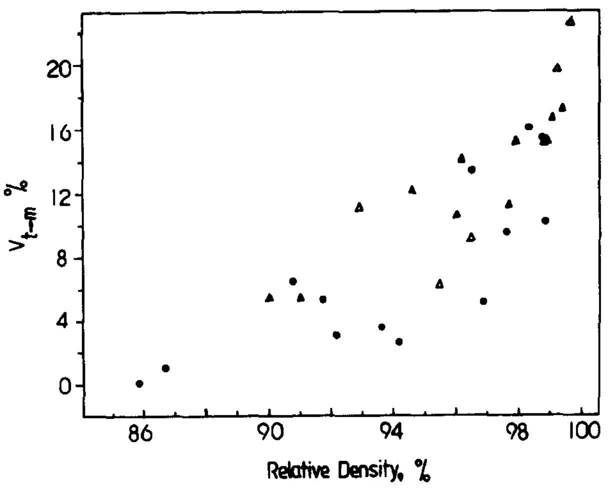

Fig. 5. Volume ($V_{t\to m}$) of phase transformation from tetragonal to monoclinic during fracture of sintered 3Y–TZP bodies versus density at (●) 1450°C, and (△) 1550°C.

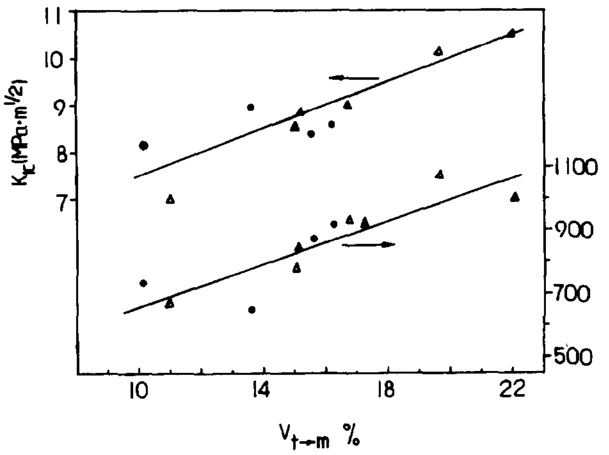

Fig. 6. Dependence of fracture toughness and strength on fracture phase transformation volume, (●) 1450°C, and (△) 1550°C.

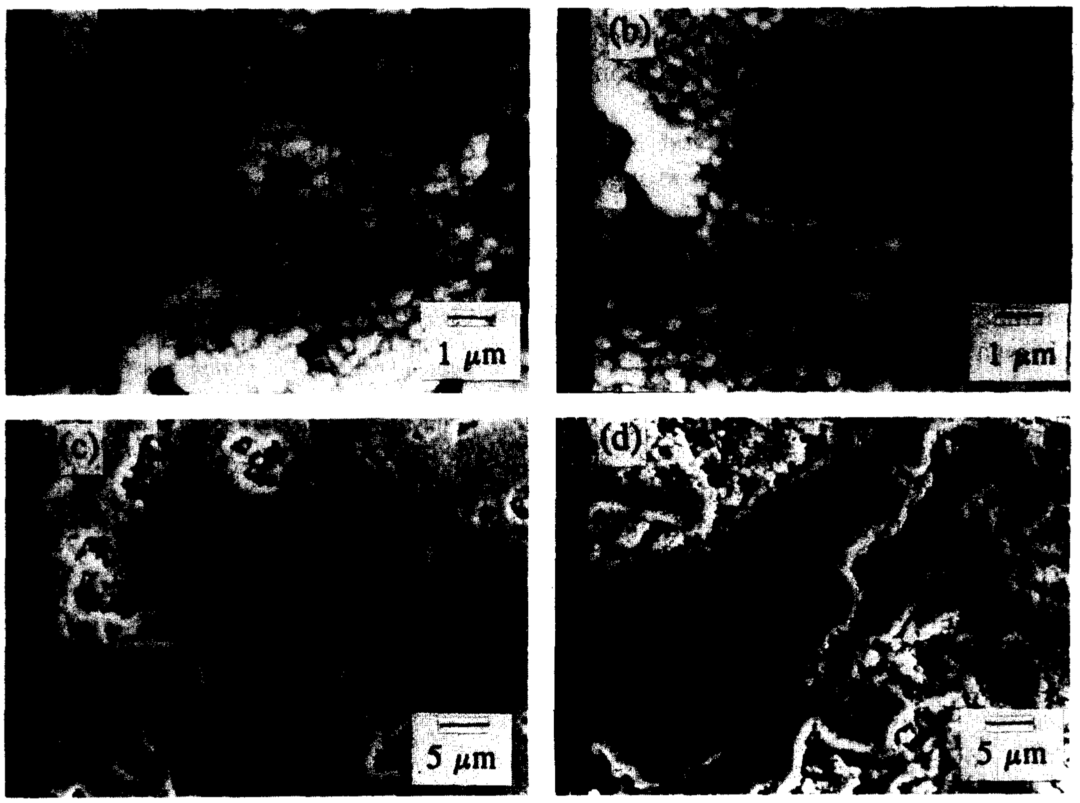

Fig. 7. SEM Micrographs of sintered 3Y–TZP bodies at 1550°C for: (a) 99·0%; (b) 97·4%; (c)90·6%, and at 1450°C for (d)85·8%.

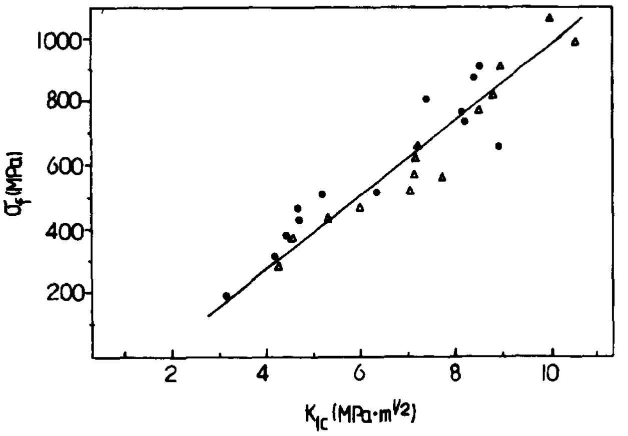

Fig. 8. Relations between fracture strength and fracture toughness of the sintered Y–TZP bodies at (●) 1450°C, and (△) 1550°C.

Discussion

For phase transformation toughened materials, the toughening contribution of stress-induced phase transformation volume $V_{t\rightarrow m}$ can be formulated as:[6,8]

$$K_{1c} = K^{o}_{1c} + A\,V_{t\rightarrow m} \qquad (6)$$

or[7]

$$K_{1c} = (K^{o}_{1c}{}^{2} + Bv_{t\rightarrow m})^{1/2} \qquad (7)$$

where K_{1c} and K^{o}_{1c} are the fracture toughness with and without the phase transformation toughening effect, $V_{t\rightarrow m}$ is the phase transformation volume and A and B are constants related to materials properties and the phase transformation process.

Figure 6 shows the relations between the fracture strength, fracture toughness and the phase transformation volume when the relative sintered densities are higher than 97%. The two relations are approximately linear.

According to the Griffith equation,[17] the relation between fracture strength and fracture toughness can be written as:[18,19]

$$K_{1c} = Y(\pi C)^{1/2}\sigma_f \qquad (8)$$

where C is one half of a critical defect (e.g. crack) length and Y is a geometrical factor. Figure 7 shows the microstructures of the Y–TZP materials with different densities. Materials of lower density contain larger pores, i.e. defects of larger size, so the change of fracture toughness with decreasing density will be less than that of fracture strength, as found in eqns (4), (5), and Figs 1 and 2. Figure 8 demonstrates the relation between the fracture strength and the fracture toughness for 3Y–TZP ceramics with various densities. At higher densities, where the strength and toughness are also higher, the calculated critical defect length, if the geometric factor Y is assumed to be one, is around 60 μm; at lower density where the strength and toughness were also lower, the defect length is up to 160 μm; both of them are much greater than the grain sizes of the pressureless sintered materials.

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