

## Hydrothermal Stability of (Y, Nb)-TZP/Al<sub>2</sub>O<sub>3</sub> Composites

Deuk Yong Lee, Dae-Joon Kim\* and Seung-Jae Lee\*\*

Department of Metallurgical and Materials Engineering,  
Daelim College of Technology, Anyang 431-715, Korea

\*Division of Ceramics, Korea Institute of Science and Technology, Seoul 136-791, Korea

\*\*Department of PWR Fuel Development, Korea Nuclear Fuel Co., Ltd., Taejeon 305-353, Korea  
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Y<sub>2</sub>O<sub>3</sub> and Nb<sub>2</sub>O<sub>5</sub> co-doped tetragonal zirconia polycrystals ((Y,Nb)-TZP) containing 10 to 30 vol% Al<sub>2</sub>O<sub>3</sub> were prepared and hydrothermal stability of the composites was evaluated after aging for 5 h at the temperature range of 150 °C to 250 °C under 4 MPa H<sub>2</sub>O vapor pressure in an autoclave. The (Y,Nb)-TZP/Al<sub>2</sub>O<sub>3</sub> composites showed excellent phase stability under the hydrothermal conditions, as compared with the 3Y-TZP/Al<sub>2</sub>O<sub>3</sub> composites, due to the combined effects of the Y-Nb ordering in the t-ZrO<sub>2</sub> lattice, the reduction of oxygen vacancy concentration, and the Al<sub>2</sub>O<sub>3</sub> addition. The strength and fracture toughness of the (Y,Nb)-TZP/Al<sub>2</sub>O<sub>3</sub> composite, containing 20 vol% of 2.8 Al<sub>2</sub>O<sub>3</sub> particles, were 700 MPa and 8.1 MP · am<sup>1/2</sup>, respectively.

**Key words:** Tetragonal zirconia polycrystal, Al<sub>2</sub>O<sub>3</sub>, Oxygen vacancy, Autoclave, Hydrothermal stability

### I. Introduction

Y<sub>2</sub>O<sub>3</sub>-stabilized tetragonal zirconia polycrystals (Y-TZPs) possess excellent mechanical properties such as fracture strength and toughness. Nevertheless, Y-TZPs experience low-temperature strength degradation (LTD) because of the spontaneous tetragonal (t) to monoclinic (m) phase transformation during annealing over the temperature range of 100 to 400 °C for a long period of time in air or water.<sup>1-7)</sup> The LTD of Y-TZP is more detrimental in the presence of water vapor because the t → m phase transformation, which results in severe cracking on the surface of Y-TZP, occurs at lower temperatures and in shorter time, as compared with aging in air.<sup>4,7)</sup>

Considerable attempts have been made to elucidate a cause of LTD in water atmosphere, but it is generally agreed that an exact mechanism is not fully understood. Recently, it has been proposed that oxygen vacancies present inside the specimen play an important role in LTD.<sup>7)</sup> Hughes *et al.*<sup>5)</sup> showed that the destabilization of t-phase during aging was related to the annihilation of oxygen vacancies by the substitution of O<sup>2-</sup> anions. Kim *et al.*<sup>8-8)</sup> proposed that LTD of Y-TZP was governed not by the existence of H<sub>2</sub>O but the amount of residual stress in t-ZrO<sub>2</sub> during aging, which was probably accumulated by the diffusion of oxygen vacancies. The residual stress facilitated the t → m phase transformation and the reaction between Zr-O-Zr bond and H<sub>2</sub>O.

The moisture assisted degradation of Y-TZPs can be suppressed by adding additional stabilizer, such as CeO<sub>2</sub><sup>9-12)</sup> and TiO<sub>2</sub>,<sup>5,13)</sup> or the formation of composites with Al<sub>2</sub>O<sub>3</sub>.<sup>12)</sup> Boutz *et al.*<sup>10)</sup> reported that the phase stability of 2Y-TZP,

aged at 185 °C in hot water, was maintained by alloying with 46 mol% of CeO<sub>2</sub>. They rationalized that the segregation-surface-layer of cerium acted as a protective coating for the yttrium sites and then caused no LTD. Hughes *et al.*<sup>5)</sup> observed that the addition of TiO<sub>2</sub> into 3Y-TZP retarded the t → m phase transformation during the autoclave treatment at 170 °C due to the Ti<sup>4+</sup> enrichment on the surface. Lee *et al.*<sup>14,15)</sup> reported that Y-TZP doped with Nb<sub>2</sub>O<sub>5</sub> ((Y,Nb)-TZP) exhibited no LTD after aging at 220 °C for 1000 h in air due to local Y-Nb ordering<sup>14,16)</sup> in t-ZrO<sub>2</sub> into a scheelite-like arrangement,<sup>17)</sup> which resulted in a relief of the internal strain in the t-ZrO<sub>2</sub> lattice since the internal stress caused the degradation.<sup>7)</sup> Although (Y,Nb)-TZP shows superior phase stability and fracture toughness, the strength of (Y,Nb)-TZP is about 500 MPa,<sup>13)</sup> which is far lower than 850 MPa of 3Y-TZP.<sup>8,19)</sup>

It has been reported<sup>12,19)</sup> that the dispersion of rigid Al<sub>2</sub>O<sub>3</sub> particles in a zirconia matrix can form a new class of ceramics with improved strength and toughness, which also shows much better resistance to aging even under hydrothermal conditions.<sup>4,12)</sup> Accordingly, in this study, the mechanical properties and the hydrothermal stability of (Y,Nb)-TZP/Al<sub>2</sub>O<sub>3</sub> composites were investigated and then the mechanism of LTD was evaluated.

### II. Experimental Procedure

Powder preparation procedure of (Y,Nb)-TZP, having the composition of 90.24 mol% ZrO<sub>2</sub>-5.31 mol% Y<sub>2</sub>O<sub>3</sub>-4.45 mol% Nb<sub>2</sub>O<sub>5</sub>, was reported elsewhere.<sup>14)</sup> (Y,Nb)-TZP/Al<sub>2</sub>O<sub>3</sub> and 3Y-TZP/Al<sub>2</sub>O<sub>3</sub> powder mixtures were prepared by adding 10, 20, and 30 vol% of Al<sub>2</sub>O<sub>3</sub> into the TZPs and mixing in a

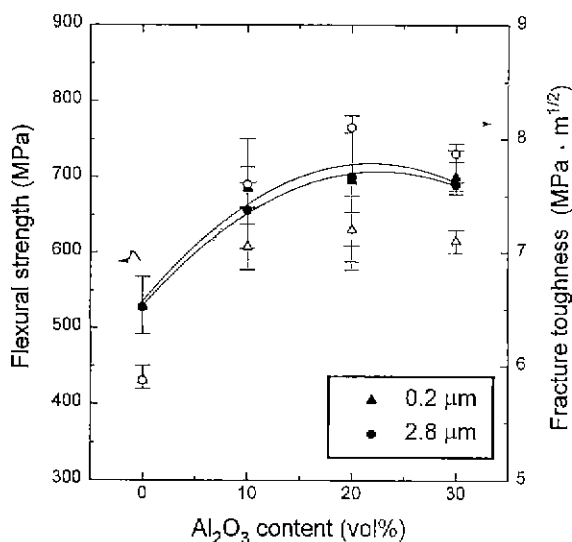
PE jar with zirconia balls for 24 h, using ethyl alcohol as the liquid. 3Y-TZP (Tosoh Inc., Japan) and two different particle sizes (0.2  $\mu\text{m}$  and 2.8  $\mu\text{m}$ ) of  $\text{Al}_2\text{O}_3$  powders (Sumitomo Chemical Co., Japan) were used as the starting powders. The milled slurries were dried, sieved through a 100-mesh screen, die-pressed into disks, and then isostatically pressed at 140 MPa. The green compacts were sintered for 2 h at 1550  $^\circ\text{C}$  in air. The typical dimension of the sintered specimens was approximately 18 mm in diameter and 1.8 mm in thickness. The sintered density was measured by the Archimedes method, using distilled water as the immersion medium.

For mechanical property measurements the disk specimens were polished to a 1  $\mu\text{m}$  diamond finish. The disks with a diameter of 18 mm and a thickness of 1.8 mm were broken using a flat-on-three-ball biaxial-fixture at a stress rate of 23  $\text{MPa} \cdot \text{s}^{-1}$  for the strength determination.<sup>19,20</sup> The fracture toughness was determined by the indentation-strength method.<sup>21</sup> A Vickers indent of 490 N was placed on the center of the tensile face of 5 test specimens. A drop of silicon oil was applied to minimize moisture assisted sub-critical crack growth. For the calculation of toughness, the hardness to modulus ratio was estimated from the measurements of the diagonal dimensions of 49 N Knoop indentations.<sup>21</sup>

A hydrothermal stability of the specimens was evaluated after aging for 5 h at temperatures from 150  $^\circ\text{C}$  to 250  $^\circ\text{C}$  under 4 MPa  $\text{H}_2\text{O}$  vapor pressure in an autoclave. The extent of the phase stability was estimated from the m-ZrO<sub>2</sub> fraction on the aged specimen according to Garvie and Nicholson.<sup>29</sup>

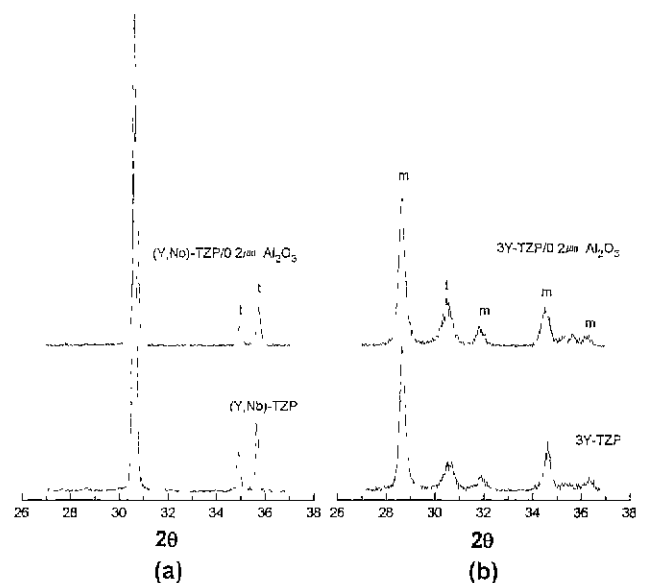
### III. Results and Discussion

Flexural strength and fracture toughness of (Y,Nb)-TZP/



**Fig. 1.** Strength and fracture toughness of (Y,Nb)-TZP, prepared by sintering for 2 h at 1550  $^\circ\text{C}$ , as a function of  $\text{Al}_2\text{O}_3$  content.

$\text{Al}_2\text{O}_3$  composites containing 0 to 30 vol%  $\text{Al}_2\text{O}_3$  are shown in Fig. 1. The strength and the fracture toughness of (Y,Nb)-TZP were 530 MPa and 5.9  $\text{MPa} \cdot \text{m}^{1/2}$ , respectively. The maximum value of strength and toughness was obtained for the composites containing 20 vol%  $\text{Al}_2\text{O}_3$ . The flexural strength and the fracture toughness of both composites having the fine (0.2  $\mu\text{m}$ ) and the coarse (2.8  $\mu\text{m}$ )  $\text{Al}_2\text{O}_3$  particles rose as the  $\text{Al}_2\text{O}_3$  content increased to 20 vol% and then fell slightly, which was consistent with the results reported earlier.<sup>24,25</sup> The gradual rising trend may be attributed to the enhanced phase transformation of TZP and the crack bridging effect with the secondary  $\text{Al}_2\text{O}_3$  particle addition.<sup>19,24-27</sup> Toughness difference between the composites caused by the phase transformation and the grain bridging was likely due to the fact that the grain size of transformable t-ZrO<sub>2</sub> depends on the  $\text{Al}_2\text{O}_3$  particle size according to the Zener's relationship, indicating that the ultimate grain size of matrix phase scales with that of inclusions.<sup>19</sup> The strength of the composites containing the coarse  $\text{Al}_2\text{O}_3$  particles was consistently higher than that of the composites containing the fine  $\text{Al}_2\text{O}_3$  particles due to the toughness dependence of the strength as a result of Griffith relationship.<sup>19</sup> The reduction in strength with an  $\text{Al}_2\text{O}_3$  content above 20 vol% may be ascribed to an increase in defect population and size and a decrease in constraint on the t-ZrO<sub>2</sub> as a result of low bulk density.<sup>24</sup> The relative density of (Y,Nb)-TZP/20 vol%  $\text{Al}_2\text{O}_3$  composites containing the fine and coarse  $\text{Al}_2\text{O}_3$  particles was above 98.5%, but it decreased to 98% as  $\text{Al}_2\text{O}_3$  particles were added more than 30%. The optimized flexural strength and fracture toughness for the coarse  $\text{Al}_2\text{O}_3$  particle containing composites, sintered for 2 h at 1550  $^\circ\text{C}$ , were 700 MPa and 8.1  $\text{MPa} \cdot \text{m}^{1/2}$ , respectively.



**Fig. 2.** XRD patterns of (a) (Y,Nb)-TZP and (Y,Nb)-TZP/20 vol% of 0.2  $\text{Al}_2\text{O}_3$  composites; (b) 3Y-TZP and 3Y-TZP/20 vol% of 0.2  $\text{Al}_2\text{O}_3$  composites, after aging for 5 h at 200  $^\circ\text{C}$  and 4 MPa  $\text{H}_2\text{O}$  vapor pressure in an autoclave.

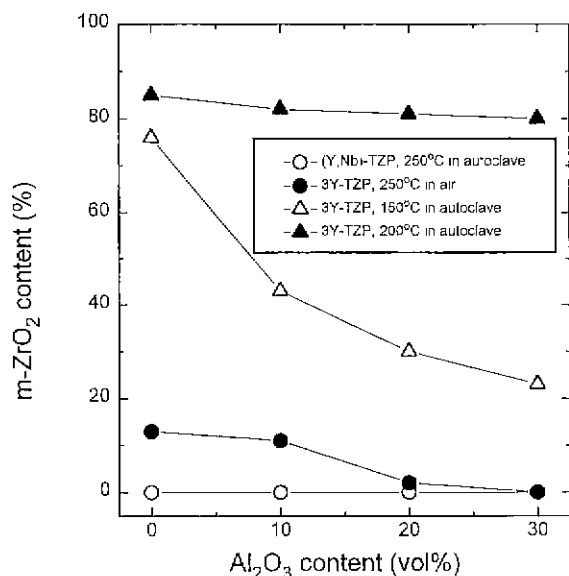
Both monolithic (Y,Nb)-TZP and (Y,Nb)-TZP/20 vol% of 0.2  $\mu\text{m}$  Al<sub>2</sub>O<sub>3</sub> composites showed only t-ZrO<sub>2</sub> after aging for 5 h at 200 °C under 4 MPa H<sub>2</sub>O vapor pressure in an autoclave, as shown in Fig. 2. However, 3Y-TZP and 3Y-TZP/Al<sub>2</sub>O<sub>3</sub> composites, prepared under the identical condition, exhibited about 80% m-ZrO<sub>2</sub> content during the aging procedure. The fraction of m-ZrO<sub>2</sub> in 3Y-TZP/Al<sub>2</sub>O<sub>3</sub> composites, aged for 1000 h at 250 °C in air and for 5 h at 150 °C under 4 MPa H<sub>2</sub>O vapor pressure in the autoclave, decreases with the Al<sub>2</sub>O<sub>3</sub> addition due to the hindrance of a relaxation of the strained t-ZrO<sub>2</sub> lattice as a result of the presence of rigid Al<sub>2</sub>O<sub>3</sub> particles as shown in Fig. 3. However, the fraction of m-ZrO<sub>2</sub> is not influenced by the Al<sub>2</sub>O<sub>3</sub> addition as the autoclave temperature is raised from 150 °C to 200 °C because thermal energy for the t  $\rightarrow$  m phase transformation is enough. At 250 °C in the autoclave, the 3Y-TZP/Al<sub>2</sub>O<sub>3</sub> composites were all shattered to parts due to the nearly complete phase transformation to m-ZrO<sub>2</sub> regardless of the Al<sub>2</sub>O<sub>3</sub> content and the Al<sub>2</sub>O<sub>3</sub> particle size. This Al<sub>2</sub>O<sub>3</sub> addition into 3Y-TZP is not effective to the phase stability of t-ZrO<sub>2</sub> when the autoclave temperature is above 200 °C.

The absence of the hydrothermal degradation in monolithic (Y,Nb)-TZP and (Y,Nb)-TZP/Al<sub>2</sub>O<sub>3</sub> composites under the low-temperature conditions, as shown in Fig. 3, is attributed primarily to the t-ZrO<sub>2</sub> phase stability due to the Y-Nb ordering in the t-ZrO<sub>2</sub> lattice<sup>14,16</sup> because (Y,Nb)-TZP maintains the phase stability even after aging for 5 h at 250 °C under 4 MPa H<sub>2</sub>O vapor pressure in the autoclave. In addition, the alloying of pentavalent oxide to Y-TZP may suppress the number of oxygen ion vacancies, which are formed by the substitution of Y<sup>3+</sup> for Zr<sup>4+</sup>, as a result of the substitution of Nb<sup>5+</sup> for Zr<sup>4+</sup>.<sup>6,8,14,18</sup> The reduction of oxygen

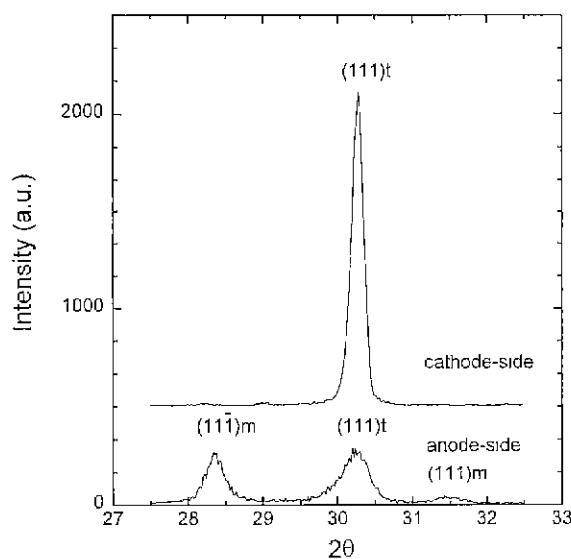
vacancy concentration in t-ZrO<sub>2</sub> suppressed the t  $\rightarrow$  m phase transformation due to a low vacancy diffusion rate during aging at low temperatures.<sup>8,81</sup> Furthermore, the addition of the rigid Al<sub>2</sub>O<sub>3</sub> particles to Y-TZP may also contribute to the stability of the composite because the secondary particles impedes a relaxation of the strained t-ZrO<sub>2</sub> lattice.<sup>21,25,27</sup>

In an effort to rationalize the role of oxygen vacancy diffusion on degradation, the activation energies for the bulk ionic conduction of 3Y-TZP and 3Y-TZP that contained Nb<sub>2</sub>O<sub>5</sub> (0 to 1.5 mol%) were measured by one of the authors,<sup>81</sup> along with the enthalpy for the t  $\rightarrow$  m phase transformation of the corresponding specimens during aging in air.<sup>61</sup> The average value of activation enthalpy for the ionic conduction and that for LTD in 3Y-TZP doped with Nb<sub>2</sub>O<sub>5</sub> is 83 $\pm$ 5 kJ/mol and 83 $\pm$ 2 kJ/mol, respectively. The similarity suggests that the diffusion of oxygen vacancies, which are the movable charge carrier in this system, is involved in both processes. Therefore, LTD of TZPs is probably a relaxation process of internally strained lattice by a thermally activated oxygen vacancy diffusion.<sup>6,81</sup>

The belief that the migration of oxygen vacancies is involved in LTD was further fortified by the observation of m-ZrO<sub>2</sub> content in 3Y-TZP after aging for 2 h under the applied field of 1.1 kV/mm in a silicon-oil bath at 120 °C as shown in Fig. 4. About 60% of m-ZrO<sub>2</sub> was observed only on the surface in contact with the anode and no phase transformation was detected on the surface in contact with the cathode. The selective degradation on the anode-sided surface is likely due to migration of oxygen vacancies toward the cathode-sided surface under the applied field.<sup>28</sup> Thus, the vacancy-depleted surface is overcrowded by oxygen ions, which results in a strained t-ZrO<sub>2</sub> lattice. As the residual stress is accumulated with the sufficient lapse of the



**Fig. 3.** Influence of aging temperature on the extent of m-ZrO<sub>2</sub> of 3Y-TZP containing various amount of 0.2  $\mu\text{m}$  Al<sub>2</sub>O<sub>3</sub> particles. The specimens were aged for 1000 h at 250 °C in air or for 5 h at 4 MPa H<sub>2</sub>O vapor pressure and different temperature in an autoclave.



**Fig. 4.** XRD patterns of 3Y-TZP specimen surfaces, that are faced to the node and the cathode after aging for 2 h under an applied field of 1.1 kV/mm in a silicon-oil bath at 120 °C. 3Y-TZP prepared by sintering for 4 h at 1550 °C.

aging time to overcome the nucleation barrier related to the  $t \rightarrow m$  phase transformation, the degradation occurs.

#### IV. Summary

The migration of oxygen vacancies may be responsible for LTD of TZPs. The phase stability of the (Y,Nb)-TZP/Al<sub>2</sub>O<sub>3</sub> composites under the low-temperature hydrothermal conditions was achieved by: 1) the Y-Nb ordering in the t-ZrO<sub>2</sub> lattice; 2) elimination of oxygen ion vacancies in Y-TZP as a result of Nb<sub>2</sub>O<sub>5</sub> co-doping, which leads to a low vacancy diffusion rate during annealing at low temperature; 3) suppression of a relaxation of the strained t-ZrO<sub>2</sub> lattice by the rigid Al<sub>2</sub>O<sub>3</sub> particles during aging at low temperatures.

Both the strength and the toughness of (Y,Nb)-TZP (530 MPa and 5.9 MPa · m<sup>1/2</sup>) was improved by the addition of Al<sub>2</sub>O<sub>3</sub> particles. The optimized flexural strength and fracture toughness for the coarse Al<sub>2</sub>O<sub>3</sub> particle containing composites, sintered for 2 h at 1550 °C, were 700 MPa and 8.1 MPa · m<sup>1/2</sup>, respectively.

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