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The aging effect on the low temperature mechanical strength of 3.2YSZ single crystals manufactured by the Skull melting method

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Abstract

Single crystals of ZrO_2 containing 3.2 mol% of Y_2O_3 were manufactured by the Skull melting method. The single crystals were composed of the t′-ZrO_2 phase. For a comparison of the mechanical strength, 3 mol% of Y_2O_3 added tetragonal zirconia polycrystal (3Y-TZP) (Tosoh TZ-3Y®) samples were also prepared. The biaxial flexure strengths of the single crystal and 3Y-TZP ceramics were 1328 ± 537 MPa and 1227 ± 312 MPa, respectively. After autoclave treatment at 200 °C for 24 h, the strength of the single crystals and polycrystalline ceramics changed to 1345 ± 251 MPa and 281 ± 31 MPa, respectively. This result shows that the t′-ZrO_2 single crystals have excellent resistance in moisture-enhanced mechanical degradation. Since the martensitic transformation of the tetragonal (t) and monoclinic (m) has crystallographic correspondences between the lattices, the relationship will lead to low interface energy at the t–m interface and high binding energy between the t and m phases. As far as the crystallographic relationship is maintained in the t′-ZrO_2 single crystals, different from polycrystalline 3Y-TZP, the partial coherency seems to retard low temperature degradation – monoclinic phase pull out or crack generation – of single crystals.

Keywords: Zirconia; Single crystal; Skull melting method; Aging; Mechanical strength

1. Introduction

Interest in strong and tough ZrO_2 based ceramics has increased greatly since it was discovered that ZrO_2 ceramics containing fine tetragonal particles are tougher and stronger than either single phase or monoclinic containing ZrO_2 ceramics. Particularly, in the system of ZrO_2–Y_2O_3, various mechanical properties could be engineered with different Y_2O_3 compositions and heat treatments [1–3]. The Y_2O_3 stabilized tetragonal ZrO_2 polycrystal (usually called 3Y-TZP) ceramics have very good mechanical properties at room temperature around 1200 MPa [4]. However, extended exposure of toughened ceramics to low temperatures (around 200 °C) in the presence of water has been reported to lead to a slow t–m transformation from the metastable tetragonal phase to the more stable monoclinic phase in the surface grains which induces microcracking and severely degrades the mechanical strength [5,6]. This phenomenon – low temperature degradation (LTD) – is well documented in the literature [7–10]. In order to suppress the aging phenomena in Y-TZP, the effect of Mn, Ge and Ce doping on isothermal degradation has been studied [11,12]. In addition to humidity at low temperatures, the grain boundary in polycrystalline tetragonal ZrO_2 is another weakness from a mechanical point of view. Therefore, a single crystal ZrO_2 could be one of the alternative means that can compensate for the weakness.

According to the phase diagram of the ZrO_2–Y_2O_3 system [7,13], a phase decomposition was anticipated during cooling from a melt into a high-yttria cubic matrix and a low-yttria tetragonal precipitate between 2300 and 1000 °C, and the phase decomposition accompanies yttria diffusion. With further cooling to room temperature, this

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equilibrium low-yttria tetragonal phase was expected to transform martensitically into a monoclinic structure. Under certain conditions, particularly when yttria-partially-stabilized zirconia (Y-PSZ) is quenched (relatively fast cooling) from a melt [14], another kind of tetragonal phase can be retained at room temperature, called the t′ phase. As in t′-ZrO_2, the tetragonal domains filled the whole volume, so their spatial arrangement must have been highly symmetrical in order to minimize the coherency strain energy [15]. The tetragonal t′-ZrO_2 contains a higher yttria concentration than the metastable low-yttria tetragonal phase. Therefore, unlike the metastable low-yttria tetragonal phase, the martensitic transformation to monoclinic had been assumed unavailable in the t′ phase [14–17], and it has been reported that the t′ phase is highly resistant to stress-induced transformation to the monoclinic phase [16,18–23]. However, it has been shown that the transformation is possible depending on the domain size [16,17,24].

Even though there have been a lot of studies on the mechanical properties of polycrystalline zirconia ceramics and on partially- and fully-stabilized ZrO_2 single crystals [25–28], the moisture-enhanced aging effect on the mechanical strength of the t′-ZrO_2 single crystals has not yet been studied. In this study, single crystals of 3.2 mol% of Y_2O_3 added ZrO_2 were manufactured by the Skull melting method. The mechanical strength of single crystals before and after the aging treatment (i.e., autoclave treatments at 200 °C for 24 h) was compared with those of the 3Y-TZP specimens.

2. Experimental procedure

High purity ZrO_2 (High Purity Zirconia, KCM Co., Japan) and Y_2O_3 (99.99%, Kojundo, Japan) were used as raw materials. The ZrO_2 and Y_2O_3 were weighed with a 93.6:6.4 atomic ratio. The weighed powders were wet mixed for 20 h in a jar with zirconia balls and ethanol. Then, the slurry was fully dried in an oven. Single crystals of 3.2YSZ were obtained by Skull melting the mixed powders, and the total amount of mixed powders for a batch was 40 kg. The temperature for Skull melting reached 2900 °C, and the growth rate of the crystal was around 5.0 mm/h. The total cooling process to room temperature took around 48 h.

The microstructure of the specimens was analyzed with a SEM (JEOL, JSM6701F). TEM observations of the single crystals were performed in an instrument equipped with a field-emission gun operating at 200 kV (Tecnai G2 F20 S-TWIN). The crystals were cut into coin-shapes for the analysis, and commercial grade 3Y-TZP (Tosoh TZ-3Y®) polycrystalline specimens were also prepared for comparison with the single crystals. After cold isostatic pressing under 40,000 psi, the 3Y-TZP specimens were sintered at 1400 °C for 2 h. Autoclave treatment for the coin-shape single crystals and the polycrystalline specimens was carried out at 200 °C (1.55 MPa) for 24 h. This is a very cruel condition corresponding to at least 60 years (simulated) of aging time for *in-vivo* conditions [7].

To evaluate the mechanical property, the biaxial flexure strength (Shimadzu AG-500E, rate: 0.1 mm/min) method (ISO6872:2008E) was used based on the equation $S = -0.2387P(X-Y)/d^2$, where S is the maximum center tensile stress (MPa), P is the total load causing fracture (N), $X=(1+\nu)\ \ln(B/C)^2+[(1-\nu)/2](B/C)^2$, $Y=(1+\nu)[1+\ln(A/C)^2]+(1-\nu)(A/C)^2$, ν is Poisson's ratio (0.25), A is the radius of the support circle (5 mm), B is the radius of the loaded area or ram tip (0.7 mm), C is the radius of the specimen (mm), and d is the specimen thickness at fracture origin (mm).

3. Results and discussion

Fig. 1 shows a typical feature of the 3.2 mol% of Y_2O_3 added ZrO_2 single crystals prepared with the Skull melting method. The size of the crystals was about 10 cm in height and 2–4 cm in width. The color of the single crystals was translucent milky, which suggests that there were some kind of light scattering sources in the crystal. When the amount of yttria went over 10 mol%, it was generally assumed that transparent cubic zirconia single crystals were obtained.

Fig. 2 shows the microstructure of the fracture surface of a single crystal. 'Herringbone'-shaped and fine regular stripe structures (marked with arrows), which are the typical features of the polydomain structure of the t′-ZrO_2 phase, were visible. The longitudinal domains extended approximately hundreds of nanometers in width and 1–2 μm in length.

Fig. 1. Single crystals manufactured by the Skull melting method.

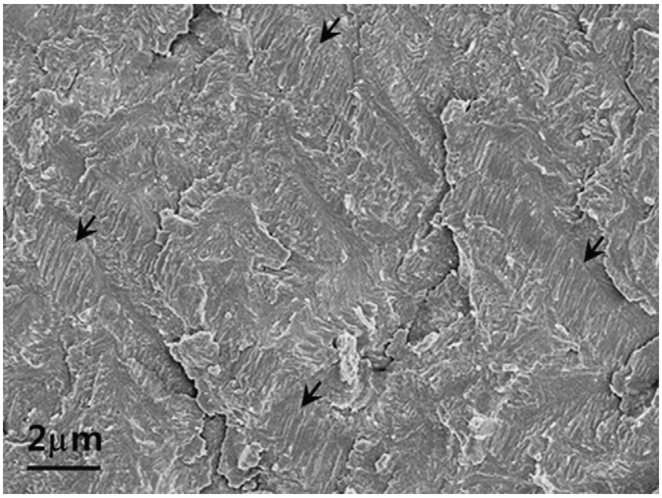

Fig. 2. SEM microstructure for the fracture surface of the single crystal.

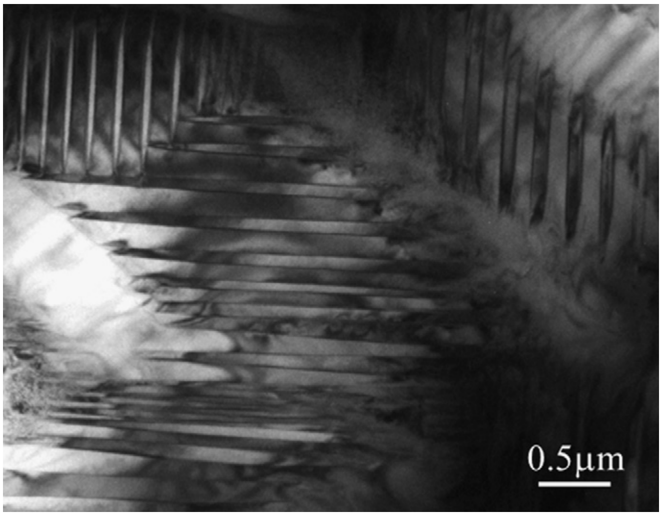

Fig. 3. Bright-field TEM images of the single crystals.

The TEM of the bright-field image of the single crystals is shown in Fig. 3. A polydomain structure, which is arranged in very regular pattern with a bright and dark longitudinal domain, was clearly observed. Thus, the domain colonies are sometimes described as 'herringbone' domain structures as mentioned in Fig. 2. The ferroelastic domain switching phenomenon, which is the deformation process of the herringbone domain, is different from the matensitic transformation of the tetragonal into monoclinic phase because it arises from a symmetry-lowering ferroic phase transition (i.e., reorientation of ferroelastic domains) without a change in the crystal structure [15]. The excellent resistance of the t′ materials to low-temperature aging is explained by the ferroelastic domain structure of these materials [16], and the ferroelastic transformation is known for its potential toughing mechanism [17]. The translucent milky color of the single crystal shown in Fig. 1 can be explained by light scattering at the boundaries of the colonies formed by the twinned tetragonal structure (t′-phase) in the crystals.

High-resolution TEM micrographs of the domain boundary areas are presented in Fig. 4(a)–(c). In the cases of (a) and (b), the left upper area of the images corresponds to the domain matrix area, and the lower right area corresponds to the domain boundary (DB) area, while the right upper is the domain matrix in (c). The thickness of the domain boundary was about 15–20 nm. The lattice fringes of the matrix and domain boundary, which were parallel, were clearly seen in both areas, but the intervals of the fringes in each area were different. In (a), the domain boundary was composed of parallel superlattice-type fringes, and the widths of the fringes were about 0.274 nm and 0.518 nm for the domain matrix and the parallel superlattice (domain boundary area), respectively. In the cases of (b) and (c), however, perpendicular superlattice-type fringes were observed together with the parallel superlattice-type fringes. The perpendicular superlattice-type fringes are marked with white arrows. In the case of (b), the widths of the fringes were about 0.262 nm and 0.529 nm for the domain matrix and the perpendicular superlattice-type fringes, respectively. Since the domain boundaries are coherent, it makes the nucleation of the monoclinic phase difficult [20,29]. Therefore, high strength and fracture toughness of single crystals can be attained through the ferroelastic transformation of the tetragonal polydomains when approximately 3 mol% Y_2O_3 is added to ZrO_2 [17].

In t′-ZrO_2 with a Y_2O_3 content of 3 mol%, its tetragonality was very small at about half the value of pure ZrO_2 with a c/a ratio of 1.013–1.02 [16,30]. Therefore, in a macroscopic point of view, the crystal structure of the t′-ZrO_2 phase was regarded as a pseudocubic crystals [15]. Even though it has a small c/a ratio, however, tetragonality determined the mutual orientation of the c-axes of the twined domains. Since domains share their boundaries coherently [28], a large amount of mechanical stress might have been piled up at the domain boundaries, which may have resulted in the compression and expansion of the lattices. Therefore, the almost doubled lattice widths in the superlattice-type fringes near the domain boundary area observed in Fig. 4(a)–(c) might have been caused due to the mechanical stress between the adjacent domains as the domain configuration alters with different tetragonal axes.

Fig. 4(d) is the selective area diffraction pattern (SADP) of the matrix composed of polydomains. The analysis of dark spots in the reciprocal lattice showed that the axial length from the origin to respective 200 and 020 was identical, and the angular relationship between $|g_a|$ and $|g_b|$ formed a 90° angle, which supports the observation that the matrix had a pseudocubic structure. However, weak diffraction spots were also observed in the middle of clear spots. Since the half-distance of vectors between diffraction spots in the reciprocal plane corresponded to the double-distance in real plane, the parallel and perpendicular superlattice-type fringes observed near the domain boundary area may be attributed to the generation of weak diffraction spots in Fig. 4(d).

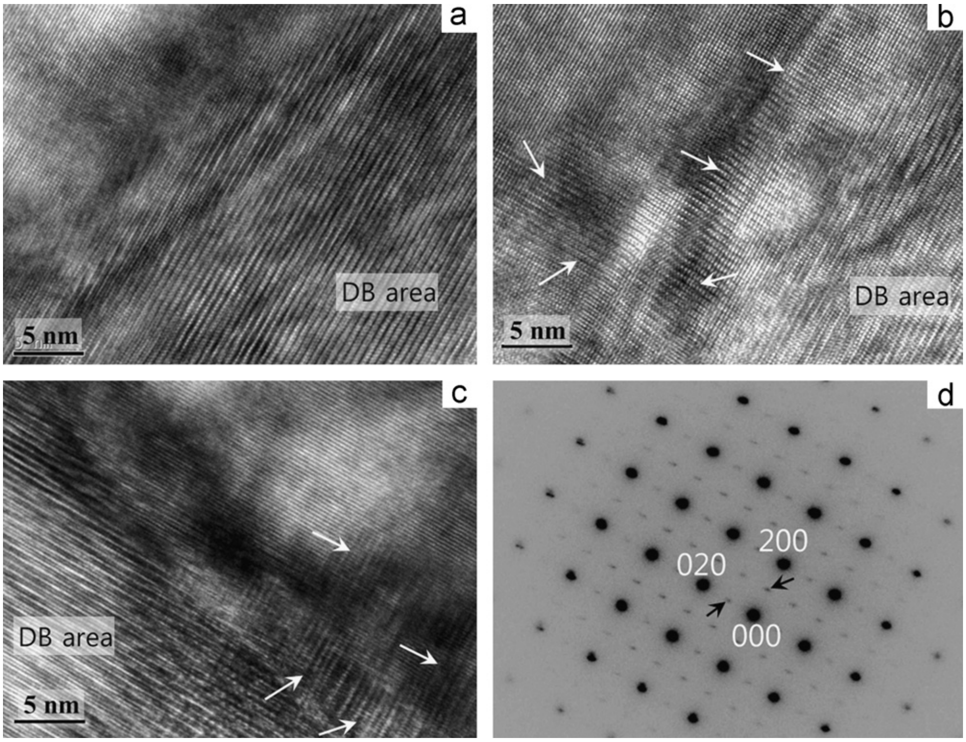

Fig. 4. High-resolution TEM micrographs near the domain boundary (a)–(c) and the selective area electron diffraction pattern of the single crystal (d).

Fig. 5 presents the SEM images of the t′-ZrO_2 single crystal and the polycrystalline specimen. Fig. 5(a) and (b) is the respective surfaces of the single crystal and the polycrystalline specimen after autoclave treatment. Fig. 5(c) and (d) is the fracture surfaces of the single crystal and the polycrystalline specimen after autoclave treatment, respectively. The surface of the single crystal was relatively clean and some scratches formed by the machining process could be seen. Concerning the fracture surfaces of the single crystal (Figs. 2 and 5c)), there were no great differences in the fracture images of the single crystals regardless of the autoclave treatment, i.e., the Herringbone domain structures were still maintained (Fig. 5(c)). In the case of Fig. 5(b), some bumpy large grains were observed. When some t-zirconia transforms to m-zirconia by the aging treatment, this transformation is accompanied by a volume increase of about 9%. If a grain transforms to monoclinic on the top surface, it is free to expand, provoking an uplift of the surface itself. Since the aging process includes surface degradation with grain pull out and microcracking, the large grains and the irregular protruding parts on the surface in (b) is thought to be formed by moisture-enhanced low temperature degradation (t–m transformation). Once grain pull out and/or microcracks are formed on the surfaces, those can be immediate at the moment of the crack propagation [31] and can provide high diffusion pathways of water molecules. In the polycrystalline fracture image of Fig. 5(d), a transgranular fracture dominant microstructure can be seen and this seem to be associated with the transformed grains [32].

The biaxial flexure strength of the single crystal and the Tosoh TZ-3Y® polycrystalline ceramics was 1328 ± 537 MPa and 1227 ± 312 MPa, respectively. After the autoclave treatment at 200 °C, the biaxial flexure strength of the single crystal and the polycrystalline ceramics changed to 1345 ± 251 MPa and 281 ± 31 MPa, respectively. The aging by autoclave treatment could not degrade the strength of the single crystals, but it severely reduced the strength of the polycrystalline specimens.

Concerning the low temperature degradation, the diffusion of moisture-related isotopical species into both tetragonal (3 mol Y_2O_3) and cubic (9 mol Y_2O_3) single crystals at temperatures characteristic of moisture enhanced low temperature degradation is reported using secondary ion mass spectrometry (SIMS) [33]. From the experiment, it was found that both H and D diffuse into the tetragonal crystal when immersed in isotopically labeled water. However, it is not identified whether the diffusion of H and D resulted in the phase transformation to monoclinic in the bulk of the single crystals, and the effect of the diffusion on mechanical characteristics has not been analyzed. According to the literature [33], after exposure to the isotopically enriched water, however, the surface of the tetragonal single crystal was partially transformed to monoclinic. However, the partially transformed monoclinic regions were perfectly recovered (healing) to the original tetragonal surface after annealing at 1200 °C. This result suggests a fact that the transformed monoclinic crystals are aligned to a specific orientation with respect to the tetragonal crystal so that perfect healing is available by heat treatment.

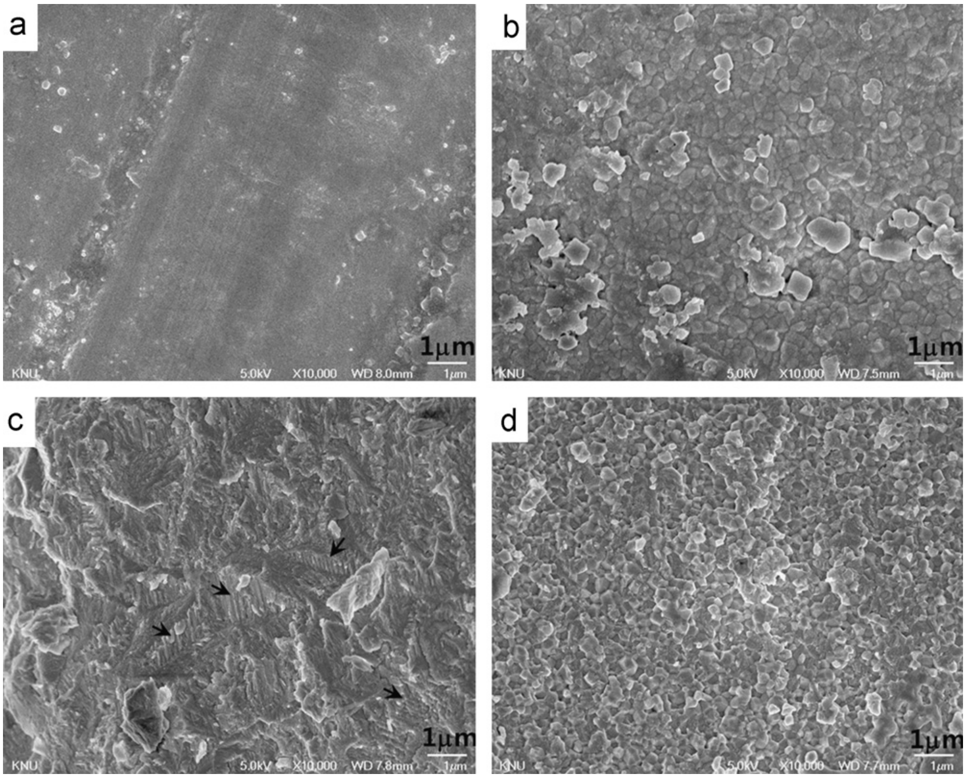

Fig. 5. SEM images of the surfaces for (a) t′-ZrO_2 single crystals and (b) 3Y-TZP, and the fracture surfaces for (c) t′-ZrO_2 single crystals and (d) 3Y-TZP after autoclave treatment.

The tetragonal to monoclinic phase transformation of zirconia is commonly recognized as being of martensitic nature, i.e., first order and diffusionless, involving shape change determined by shear [7,34,35]. The previous study on the tetragonal to monoclinic transformation in 2 mol% of Y_2O_3 added ZrO_2 also showed that there are crystallographic correspondences with habit planes and invariant shear directions between the tetragonal and monoclinic lattices [7,24,36,37]. Therefore, when the t–m transformation occurred, the very regular orientation of the planes of monoclinic phase on the surfaces could be seen [7,34]. In this case, the interface energy between the tetragonal and monoclinic phases will be decreased compared to the case of an interface without crystallographic correspondences, which will prevent detachment of the monoclinic phase (particles) from the parent phase. Furthermore, since the habit plane is regarded as a semi-coherent interface in martensite transformation [38], the binding energy between the phases will have quite a large value. As a result, the monoclinic phase pull out or crack generation may not occur easily and a relatively smooth surface is expected to be formed. In the case of polycrystalline 3Y-TZP, however, diffusion of the water species is much easier and faster through the grain boundaries. Since the grain boundaries do not have coherent interfaces, monoclinic grain pull out and crack generation are easy. Therefore, the surface of the polycrystalline will have high roughness which will lead to a lower strength than the single crystals.

The previous study [33] on the diffusion of isotopical water species in single crystals showed some surface modifications and detected oxygen and hydrogen ions in the crystals. However, the t′-ZrO_2 single crystals are not affected by aging in terms of strength, whereas for such aging polycrystalline 3Y-TZP is highly degraded.

4. Conclusion

PSZ single crystals manufactured by the Skull melting method were composed of the t′-ZrO_2 phase with polydomains. The mechanical strength of t′-ZrO_2 single crystals before and after autoclave treatment was almost unchanged while the 3Y-TZP polycrystalline specimens experienced severe mechanical degradation. Consequently, the t′-ZrO_2 single crystals revealed an exceptional resistance to moisture-enhanced low-temperature degradation within the extent of our experiment. When the tetragonal to monoclinic martensitic transformation occurred on the surface of single crystals, the crystallographic relationships between the two phases could be maintained. This seems to retard low temperature degradation of single crystals. However, in the case of polycrystalline ceramics, the adjacent grain boundaries (interface) cannot hold the monoclinic grains tightly, which results in grain pull out and crack generation.

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