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Short communication

Evaluation of effects of crack deflection and grain bridging on toughening of nanocrystalline SiO₂ stishovite

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ABSTRACT

Nanocrystalline SiO₂ stishovite has a fracture toughness higher than 10 MPa m^{1/2} due to the toughening mechanism by fracture-induced amorphization. In order to identify other toughening mechanisms which may operate simultaneously, we evaluated effect of crack deflection on fracture toughness. The median deflection angle of nanocrystalline stishovite was lower than polycrystalline ceramics such as silicon nitride and Y-TZP. While the crack deflection can contribute to fracture toughness to some extent, it cannot be the major origin of high fracture toughness of nanocrystalline stishovite. We discussed also the role of grain bridging from the relation between the fracture toughness and grain size.

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1. Introduction

Stishovite is a high-pressure phase of silica (silicon dioxide, SiO₂) [1], and has the highest hardness (33 GPa) among any oxides under ambient temperature [2]. Hard materials tend to be brittle, so that the fracture toughness of a single crystal of stishovite is only 1.6 MPa m^{1/2} [3]. Recently, Nishiyama [4] found that the nanocrystalline stishovite had a fracture toughness K_{IC} of 10–13 MPa m^{1/2} measured by the indentation fracture method (IF method). This discovery lead to a proposal of a new type of transformation toughening by the direct transition from crystal to amorphous state, i.e., fracture-induced amorphization [5]. Transformation of metastable stishovite to amorphous phase [6] can be induced by large tensile stress at the crack tip in fracture. Actually, amorphous phase with thickness of a few tens of nanometers was found on the fracture surface by X-ray absorption near edge structure (XANES) spectroscopy. Fracture toughness increased with the amount of amorphous silica near the fracture surface [5]. The fracture resistance curve (R-curve), which was measured by using micro-cantilever beam specimens, rose steeply with crack extension of only a few μ m

[7], and reached to a plateau value of 10.9 MPa m^{1/2} [8]. The sharply rising R-curve was explained as a result of narrow transformation zone width in nanocrystalline stishovite according to the theory of transformation toughening in zirconia-based ceramics [9]. The martensitic transformation of zirconia occurs rapidly catching up with crack propagation, because it is diffusionless in that no thermally activated diffusion is required. On the other hand, the amorphization is a melting in the thermodynamic sense. The quantum molecular dynamics simulation revealed that the fracture-induced amorphization could occur quickly on the order of picosecond at a fast moving crack tip [10].

While these results confirm that the fracture-induced amorphization is the major toughening mechanism of nanocrystalline stishovite, it is still possible that several other mechanisms, such as crack deflection and crack bridging [11], operate simultaneously. The crack deflection reduces the stress intensity at the crack tip. The increase in toughness by deflection depends on deflection angle and twist angle [12,13]. Faber and Evans [14] analyzed the crack profile, and showed the correlation between the toughness and the median deflection angle for glass ceramics and hot-pressed silicon nitrides (Si₃N₄). Alternatively, Mai and Lawn [15] derived a R-curve model based on grain bridging. Swanson [16] presented direct microscopic evidence to explain R-curve behavior in alumina by grain-localized bridging across the crack interface.

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The purpose of the present report is to evaluate the effects of crack deflection and grain bridging on fracture toughness of nanocrystalline stishovite, if they exist.

2. Experimental procedure

Nanocrystalline stishovite was synthesized by a Kawai-type multi anvil high pressure apparatus (LPR 1000-400/50, Max Voggenteiter GmbH, Germany). The starting materials was pure bulk silica glass, whose dimension was 2.5 mm in diameter and 0.6 mm in height. All the impurities were less than 0.1 ppm except OH (about 800 ppm). The pure bulk glass was heated rapidly from 723 K to different synthesis temperatures (1473–2073 K) under high pressure, 15 GPa. After holding for 0.5 h, temperature was decreased to 723 K, and decompression was started [4]. The average grain size was evaluated by TEM observation and optical microscopy, and it increased with the synthesis temperature; 128 nm, 253 nm, and 18.2 μm for synthesis temperatures of 1473 K, 1873 K, and 2273 K, respectively. The fracture toughness was measured by IF method [17].

The crack deflection was studied for a nanocrystalline stishovite synthesized at 1473 K (grain size, 128 nm) which had the maximum fracture toughness of $10.1 \pm 1.6 \text{ MPa m}^{1/2}$. Vickers indentation cracks were introduced on a mirror finished surface with the indentation load of 196 N. Cracks produced at the corners of Vickers indentation provide an outline of a propagating crack front as shown in Fig. 1. The lengths, ℓ , and deflection angles with respect to the direction of macroscopic crack propagation, θ , of crack segments were measured.

For comparison, 3Y-TZP was sintered by spark plasma sintering technique by using commercial zirconia powders stabilized with 3 mol% Yttria (TZ3Y, Tosoh Co. Ltd., Japan) at 1673 K under a uniaxial pressure of 50 MPa. The average grain size of the 3Y-TZP was determined to be 450 nm by analyzing the SEM images of thermally etched surfaces. The indentation cracks were introduced by

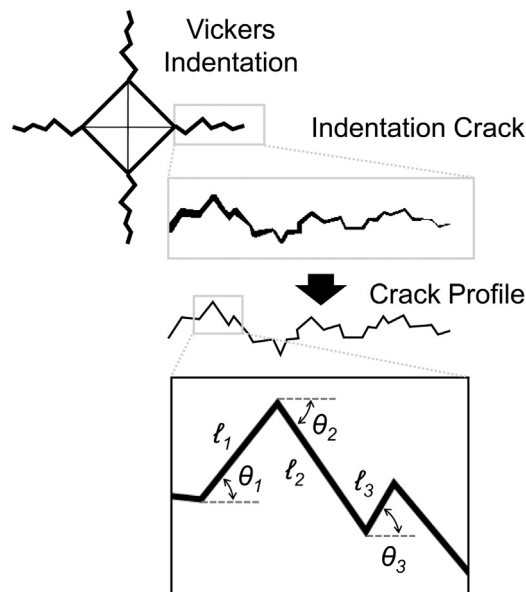


Fig. 1. Schematic illustration of analyzing method for crack geometry. A length, and angles with respect to the direction of crack propagation, of each crack segments were measured.

an indentation load of 19.6 N for 3Y-TZP. The fracture toughness was $4.2 \pm 0.2 \text{ MPa m}^{1/2}$.

3. Results and discussion

3.1. Crack deflection

Fig. 2(a) and (b) show SEM micrographs of the fracture surfaces of nanocrystalline stishovite and 3Y-TZP, respectively. The fracture surface of nanocrystalline stishovite was characterized by

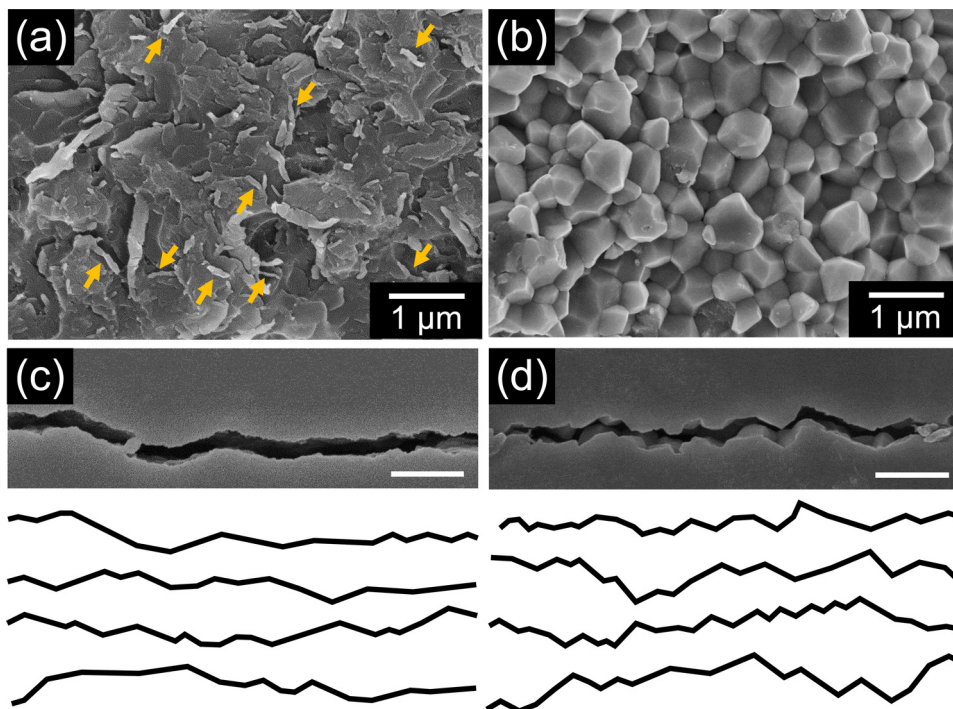


Fig. 2. SEM micrographs of fracture surface of (a) nanocrystalline stishovite and (b) 3Y-TZP. On fracture surface of stishovite, there were several worm-like structure indicated by arrows. Indentation cracks observed by SEM and crack profiles are shown in (c) nanocrystalline stishovite and (d) 3Y-TZP. Cracks propagated from left to right. The bar in SEM image is 1 μm .

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