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# Revealing Transformation Toughening Size Effects in Ceria-Stabilized Zirconia via micro-Pillar Splitting combined with Raman spectroscopy

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## Abstract

Fundamental applications of Yttria-stabilized zirconia (YSZ) include biomedical implants such as dental crowns and hip replacements. YSZ's enhanced toughness results from yttria stabilizing the metastable tetragonal phase of zirconia, which induces a martensitic transformation to the monoclinic phase under stress, increasing volume and closing cracks due to compressive stress. However, low-temperature degradation (LTD) reduces the material's ability to undergo this transformation, causing structural failures due to volume increase. Recent studies focus on ceria-stabilized zirconia (CeSZ) ceramics, which are being investigated to address the LTD issue. This study examines CeSZ with 10 mol %, 11 mol %, and 12 mol % ceria content to understand the toughening behavior, including R-curve behavior and the impact of ceria content and microstructural conditions. In this work, micro-pillar-splitting method is adopted for spatially resolved measurement of fracture toughness in CeSZ ceramics. Pillars ranging from 7  $\mu\text{m}$  to 49  $\mu\text{m}$  in diameter were fabricated via focused ion beam (FIB), in progressive increments of 7  $\mu\text{m}$ , and tested using various nanoindentation tips. The method reveals a significant dependence of toughness on pillar diameter in CeSZ, with an increase from 2.5 to 4.0  $\text{MPa}\sqrt{\text{m}}$  for the 12 mol % ceria content linked to the activation energy for phase transformation and crack deflection. Raman spectroscopy is used to understand the development of phase transformation within the volume of the pillars before and after splitting. This research provides a novel framework for faster determination of contributions to toughening mechanisms in composite ceramic materials, paving the way for developing materials with superior strength and toughness.

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