MICROPILLAR COMPRESSION OF ANISOTROPIC AI₂O₃-BASED EUTECTIC COMPOSITE

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Unidirectionally solidified oxide eutectic ceramics such as Al_2O_3 -GdAlO₃ (GAP) were developed as a candidate for next-generation high-temperature structural materials in the late 20th century [1]. In the early studies, they produced coarsened microstructure to improve high-temperature creep resistance, but later finer microstructure has been fabricated by controlling the solidification condition. The eutectic ceramics with finer microstructure was successfully fabricated at higher solidification rates in recent researches, resulting in higher mechanical strength. For example, Medeiros et al. fabricated Al_2O_3 -GAP eutectic ceramics at different solidification rates, and achieved the highest fracture strength of 1780 MPa with the finest interphase spacing of 0.4 μ m at the highest solidification rate of 240 mm \cdot h⁻¹ [2]. Here, we propose a new method for fabricating fine Al_2O_3 -GAP eutectic ceramics employing a flash event, in which specimen temperature can be abruptly changed. On the present study we successfully produced Al_2O_3 -GAP eutectic ceramics with a submicron interphase spacing by usage of a flash event under AC field. Also we performed the micropillar compression test for systematic observation of the mechanical response.

Commercially available high-purity Al₂O₃ and Gd₂O₃ powders were ball-milled in a eutectic composition of Al₂O₃-23 mol% Gd₂O₃. This powder mixture was cold-isostatically pressed and calcined at 1450°C × 2 h in air. The dimension of the calcined sample was 1.4 mm × 3.8 mm × 23 mm. The calcined sample was heated up to 1400°C in air, and a sinusoidal voltage of 1000 V/cm at a frequency of 1 kHz was applied from an AC power supply with the current limit of 110 mA for 40s, and then the external power supply was turned off. As a result, we successfully obtained anisotropic fine eutectic microstructure. The microstructure was observed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). From the eutectic region of the sample, micropillars with a diameter of 1 µm and a height of approximately 2.5 µm were fabricated by focused ion beam (FIB). Micropillar compression tests were performed at room temperature at a constant strain rate (1 × 10^{-3} s⁻¹) using a nanoindenter equipped with a flat-end diamond tip. Anisotropy in the mechanical behavior was investigated as a function of compression axis relative to the crystal-growth direction.

The flash event resulted in partial melting of the calcined sample along the current path, wherein an anisotropic eutectic microstructure was formed with rod-like GAP phases aligned in Al_2O_3 single crystals. The TEM observation revealed that the orientation relationship of both phases in this area were [0001] Al_2O_3 // [010]GAP for the growth direction. The average interphase spacing was approximately 170 nm in the finest area, which was comparable to the finest one reported in the previous study [3].

In the micropillar compression tests, brittle fracture occurred at around 20 GPa when the angle between the compression axis and the crystal growth direction was lower than 15°, while plastic deformation with work hardening occurred with the yield stress less than 16 GPa when the angle was greater than 20°. The yield stress tended to decrease with the increasing angle, suggesting the crystallographic orientation dependency in the deformation behavior of this eutectic structure. The observed plastic deformation behavior was different from that observed in the micropillar compression tests of Al_2O_3 single crystals in the previous study [4]. This indicates that characteristic deformation behavior can be raised by introducing fine anisotropic microstructure in Al_2O_3 -GAP eutectic ceramics. The deformation mechanism and the effect of multi-phase microstructure on the deformation behavior will be discussed in the presentation.

References

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