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(高速超塑性に向けたナノ結晶 Si_3N_4 の微構造設計)

Superplastic forming is an attractive method for complex-shaped components of silicon nitride (Si_3N_4)-based materials which are quite hard and brittle due to their nature of strong covalent bonding. Superplasticity is defined as an ability of polycrystalline materials to exhibit large tensile elongation. For industrial applications, the high-strain-rate superplasticity (HSRS) at strain rates higher than 10^{-2} s^{-1} is desirable to increase the production efficiency. However, superplastic deformation in Si_3N_4 ceramics has been restricted to relatively low strain rate around 10^{-6}-10^{-3} s^{-1}. Because the microstructural evolution such as grain growth, elongation and alignment along the tensile direction increased flow stress (strain hardening), and the unusual increase of flow stress at higher strain rates often occurred in compression, which is referred to shear thickening. Furthermore, the glassy phase existing at grain boundaries and multigrain junctions plays an important role on deformation. The grain-boundary sliding of neighboring grains is facilitated by the viscous flow of the glassy phase. The changes of glass chemistry due to their vaporization during high-temperature deformation is important on the flow behavior.

In this work, the superplastic behavior of nanograined Si_3N_4 ceramics containing Y_2O_3-Al_2O_3-MgO (YAM) has been studied in the terms of microstructural development and the changes of glass chemistry during deformation. YAM samples were fabricated by spark plasma sintering (SPS) at a temperature 1600 ºC under pressure 50 MPa in nitrogen atmosphere. The superplastic elongation of >300% has been achieved in YAM ceramics at a strain rate 5x10 s^{-1} at temperature of 1650 ºC. The initial microstructure of as-sintered YAM sample consisted equiaxed and fine grains with an average size of 64±16 nm. Si_3N_4 grains noticeably grew and elongated, and then the elongated grains highly aligned along tensile direction leading to the unusual stress increment (strain hardening) at the later stage of deformation. TEM/EDX analysis revealed that additive cations (Mg, Al and Y) segregated along grain boundary (thin glass film) and interface between Si_3N_4 grain and glass pocket ($\text{interface}_{\text{grain-pocket}}$) in as-sintered YAM sample. After large deformation at strain of 1.4 (300% elongation), Al and Y cations accumulated at grain boundary, and also in glass pocket with strain because of reduction of glass phase and interface area owing to grain growth. Although the major cause of strain hardening is microstructural evolution, the changes in glass chemistry is also suggested to affect the flow behavior.

In addition, the influence of MgO (YAM) and CaO (YAC) addition to Y_2O_3-Al_2O_3 glass in Si_3N_4 ceramics on superplastic flow behavior at 1700 ºC with various strain rates ranging from 10^{-4} – 10^{-3} 10 s^{-1} under compression. The strain hardening was more significant at higher strain rates, suggesting that the origin of the strain hardening was not the grain growth. The shear thickening was observed in the YAC sample at the critical stress of 10~20MPa, which coincided with the stress where strain hardening occurred in stress-strain curves. These results suggests that the strain hardening in compression tests is caused by the shear thickening. Since CaO addition causes the shear thickening, it is necessary to avoid CaO addition to promote superplastic deformation in compression.

Base on the obtained results, it revealed that the conventional manner of superplastic forming, i.e. the strain rate ranging of 10^{-5}-10^{-3} s^{-1}, changes the microstructure and the glass chemistry and hinders superplastic deformation. High-strain-rate superplasticity (HSRS) is key technique which can suppress the grain coarsening and its vaporization of glass phase due to very short forming time. It is considerable interest for industrial applications. To attain HSRS in Si_3N_4 ceramics, the further refinement of grains is necessary. The nanograined Si_3N_4 ceramics with the average diameter of 56±13 nm successfully densified at extremely low sintering temperature 1300 ºC with high pressure 300 MPa under nitrogen atmosphere. In this study, The HSRS in Si_3N_4 nanoceramics have been achieved at strain rate of 10^{-3} -10^{-2} s^{-1} with an absence of cavity damage and cracking. Their microstructure still remained the fine grains even after large deformation until strain 1.1 (33% of its initial height) under compression. Little strain hardening was observed due to the suppression of the grain coarsening and vaporization of glassy phase for short deforming time. Such achievement of HSRS in Si_3N_4 ceramics in this study can be utilized for the industrial application of the superplastic forming.