

Soaking method for fabrication of alumina-based nanocomposites

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Abstract

Previously published research proposed strengthening and toughening mechanisms of nanocomposites based on dislocation activities even in brittle ceramics. We reported that an intra-type nano-structure could only have the potential to improve strength and fracture toughness due to dislocations formed around the dispersed particles. In this research, nickel dispersed alumina nanocomposites were fabricated using a novel soaking method, based on the developed strengthening and toughening mechanisms. Commercially available γ -alumina powder with high porosity was used as the starting materials. Secondary, particles were introduced into the nano-pores of the porous γ -alumina powder using a soaking method, where the alumina powder was soaked in a nickel nitrate solution under vacuum. During pre-calcination, nickel oxide particles were generated inside the nano-pores. The alumina powders were then reduced under hydrogen atmosphere to obtain nano-sized metallic nickel inside the γ -alumina grains. The alumina–nickel composite powders were sintered by pulse electric current sintering (PECS) technique with α -alumina seeds. The mechanical properties of specimens were investigated, such as density, three-point strength, and fracture toughness. The maximum strength of the alumina–nickel nanocomposites was 984 MPa after being sintered at 1450 °C with α -alumina seeds, where the specimen size was 2 mm \times 2 mm \times 10 mm. The maximum fracture toughness was 5.5 MPa m^{1/2} after being sintered at 1350 °C with seeds measured by the single edge V-notched beam method.

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1. Introduction

Structural ceramics have low fracture toughness because of their ionic and covalent bonds, severely limiting the plastic deformation of ceramics due to dislocation activities. It is, therefore, hypothesized that a frontal process zone (FPZ) ahead of a crack tip is composed of nano-cracks rather than dislocations as is the case with metals. To overcome the inherent brittleness of ceramics, a new material design concept must be developed. The microstructure of nano-composites developed by Niihara [1] is constructed by dispersing second-phase nano-size particles within the matrix grains (intra-type nanostructure) and on the grain boundaries (inter-type nanostructure). The intra-type nano-structure produces thermal expansion mismatch between the

matrix and second-phase particles and provides a marked improvement in several mechanical properties such as fracture strength, fracture toughness, creep resistance, thermal shock resistance, and wear resistance.

Ceramics-based nanocomposites have been fabricated using high speed sintering techniques, such as hot pressing and pulse electric current sintering (PECS) [2,3] because of their low sinterability. Intra-type nano-structure is particularly difficult to fabricate using a pressure-less sintering technique. In this paper, first, we summarize the toughening and strengthening mechanisms of nanocomposites introduced in our previous papers [4,5], and second, a newly developed soaking method for constructing an intra-type nano-structure will be proposed for alumina-based nanocomposites. Finally, experimental results for alumina–nickel nanocomposites will be shown and discussed.

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2. Toughening and strengthening mechanisms

2.1. Dislocation activities

Intra-type nanocomposites consist of nano-particles dispersed within matrix grains. The characteristics of the intra-type nanocomposites result in the generation of thermally induced residual stresses after sintering. To clarify the role of the residual stresses around the dispersed particles in nanocomposites, Awaji and coworkers [4,6] analyzed residual stresses using a simplified model that consisted of a spherical particle within a concentric matrix sphere with axial symmetry, shown in Fig. 1, where we restrict our attention to the alumina-based nanocomposites to simplify the following discussion. Residual stresses numerically calculated on the particle-matrix boundary for alumina–nickel nanocomposites are shown in Table 1, where we assumed that the temperature difference between the sintered temperature and room temperature was 1570 °C and that the ratio of the particle/matrix radii was 1/5. In the table, symbols with suffix p indicate the properties of the particle (nickel) and symbols with suffix m are the properties of the matrix (alumina). It is noted that there is a large maximum shear stress on the particle/matrix boundary.

Lagerlöf et al. [7] reported that the temperature dependence of both basal and prism plane slips in an α -alumina single crystal could be described by a simple logarithmic law over a wide range of temperature:

$$\ln \tau_{cb} = \ln \tau_0 - 0.0052T, \quad (1)$$

and

$$\ln \tau_{cp} = \ln \tau_0 - 0.0026T, \quad (2)$$

where τ_{cb} and τ_{cp} represent the critical resolved shear stresses (CRSS) for basal and prism plane slips, respectively, T is temperature (K), and τ_0 is 109 and 9 GPa for basal and prism plane slips, respectively. Fig. 2 shows the temperature dependence of the CRSS for basal and prism plane slips in an

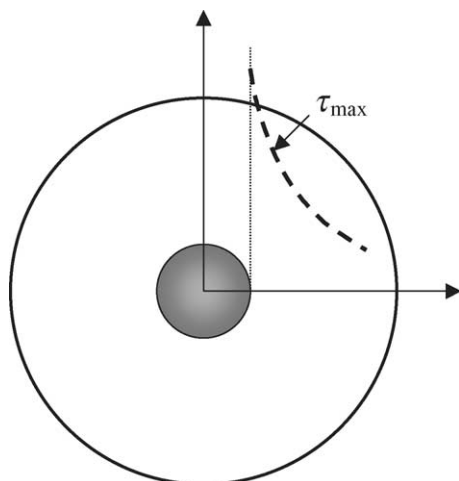


Fig. 1. A model of intra-type nano-structure.

Table 1

The maximum shear stress along the particle–matrix boundary in alumina–nickel nanocomposites

System	Al ₂ O ₃ /Ni
$\alpha_m/\alpha_p \times 10^{-6}$ (K ⁻¹)	8.8/13.7
E_m/E_p (GPa)	380/207
ν_m/ν_p	0.21/0.31
τ_{max} (GPa)	1.06

At room temperature under the assumption of $\Delta T = 1570$ °C and the ratio of the spherical particle and matrix radii is 1/5.

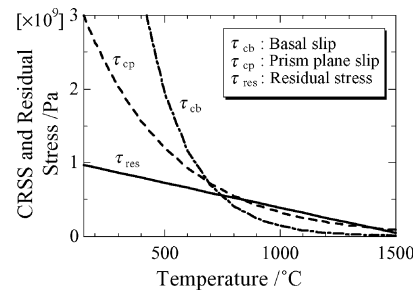


Fig. 2. Critical resolved shear stresses for α -alumina single crystal and residual stresses in alumina–nickel nanocomposites.

α -alumina single crystal and the residual shear stress τ_{res} on the alumina–nickel boundary in the nanocomposites. It is noted that dislocation activities are possible in the alumina grains at temperatures ranging from about 800 to 1400 °C.

Mismatches in thermal expansion and Young's modulus between the matrix and the dispersed particles will yield highly localized residual stresses. These stresses are reduced quickly as distance from the boundary increases because of the nano-sized particles, which can generate only small defects such as dislocations in close proximity to the particles, as shown in Fig. 3A. Large-scale cracks or other large defects will be difficult to create in the nanocomposite system and only dislocations can disperse in matrix grains at high temperatures. These dislocations are believed to become nano-crack nuclei at room temperature because the CRSS at room temperature for prism plane slip is estimated to be 4.1 GPa from Eq. (2), which is higher than the theoretical strength of the α -alumina of 2.6 GPa. This fact suggests that further annealing is important in

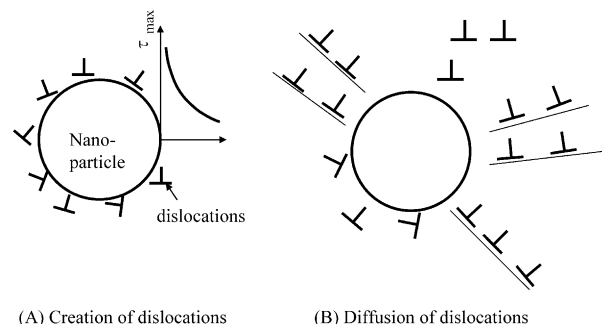


Fig. 3. Dislocation activities after sintering (A) and after annealing (B).

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