Sintering and Mechanical Properties of SiC-Whisker/ Al_2O_3 Composites with Two-Layered Structure

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2 層構成 SiC ウィスカー/ Al_2O_3 複合体の焼結と機械的特性

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Two-layered hybrid composites, the Al_2O_3 layers containing different amounts of SiC whisker, were prepared by hot-pressing the two layers simultaneously. Outstanding improvement was observed in the strength and fracture toughness of composites when one of the layers is reinforced by 10 vol% whisker addition. The difference of the fracture toughness between the layers in the composite is also observed. The effects of residual stresses resulting from the thermal expansion mismatch between two layers were discussed.

Key-word : Al_2O_3 , SiC, Whisker, Composite, Hybrid

1. Introduction

Currently there are many investigations on SiC-whisker/ Al_2O_3 composites.¹⁾⁻⁴⁾ For instance, a substantial increase in fracture toughness has been attained in SiC-whisker reinforced Al_2O_3 ceramics. These ceramics have been applied to cutting tools for their high fracture toughness.⁵⁾ Although, few applications have been found as structural materials, till now.

Two-layered hybrid composites, the Al_2O_3 layers containing different amounts of SiC-whisker, were prepared by hot-pressing the two layers simultaneously as illustrated in Fig. 1. In the whisker enriched layer, enhancement in fracture toughness and flexural strength are expected, whereas improvement in thermal behavior is expected in the whisker poor layer.

In this paper, mechanical properties of these composites were examined and effects of residual stresses resulting from the thermal expansion mismatch between two layers were also discussed.

2. Experimental procedures

As starting materials, $\alpha\text{-Al}_2\text{O}_3$ (AKP-30, Sumitomo Co., Ltd.) of 0.4 μm in average particle size and SiC-whisker (Tateho Chem. Co., Ltd.) of 0.7 μm in average diameter were used. Improved dispersion of SiC whiskers with Al_2O_3 powders was attained by the ultrasonication and ball mill mixing in ethylalco-

hol as a dispersing agent. Whisker contents were 10, 20, 30 and 40 vol%, respectively.

The mixed powders were hot-pressed in graphite dies as shown in Fig. 1. The compositions of composite were as follows: pure Al_2O_3 /10 vol% whiskers denoted as 0/10, and 10 vol% whiskers/20, 30 and 40 vol% whiskers denoted as 10/20, 10/30 and 10/40, respectively. They were hot-pressed in flowing nitrogen atmosphere at 1700°C for the 0/10 composite and at 1800°C for other composites at a pressure of 30 MPa for 45 min.

The mechanical test specimens were machined from the hot-pressed billets (50 mm ϕ by 3 mm) into rectangular bars of 4 mm by 3 mm by 40 mm. The surfaces were ground with 1000-grit diamond wheel. Hardness was measured by Vickers hardness with an indentation load of 196 N. The flexural strength was determined in three point flexure with a 30 mm span for these specimen. The fracture toughness was obtained on the polished surfaces of the flexure tested bars by IF method.⁶⁾ Indentations were made by means of Vickers hardness testing (indentation load of 196 N). These mechanical tests were carried out on both layers of each composite.

Fig. 1. Schematic illustration of the two-layered composite by hot-pressing procedure.

The microstructure of the composites samples was characterized by scanning electron microscopic (SEM) observations on the fracture surfaces.

3. Experimental results

Figures 2 and 3 show the hardness and flexural strength of composites. The values of the 10/40 composite are excluded, because it was fractured when cooled to room temperature after hot-pressing. The substantially higher value of hardness (Fig. 2) is obtained on the whisker containing layer of the 0/10 composite compared to that on the pure Al_2O_3 layer. For the 10/20 and 10/30 composite, the hardness difference decreased, probably because the hardness of composite increased with increasing SiC whisker content.

For the flexural strength, no difference is observed between the whisker enriched and pure Al_2O_3 layer in the 0/10 composite. Moreover, the obtained value of 300 MPa is lower than these of the other composites, probably because of occurrence of abnormal grain growth mentioned later. The flexural strengths of the whisker enriched layer and the poor layer of

the 10/20 composite were 650 MPa and 550 MPa, respectively. As expected, whisker addition reinforces the composite. The flexural strength of the whisker enriched layer of the 10/30 composite increased to 750 MPa, while in the whisker poor layer was maintained to 550 MPa. The 10/40 composite fractured before the mechanical tests, owing to the resulting large thermal expansion mismatch stresses discussed later.

Difference in the fracture toughness increase by the whisker reinforcement (Fig. 4) between the layers in all composites is observed, as in the flexural strength.

Figures 5 and 6 show the microstructures of the fracture surfaces of the 0/10 and 10/20 composites. In Fig. 5, the abnormal grain growth in pure Al_2O_3 layer occurred. This coarsening of grain may result in decreasing the strength of the composite as mentioned earlier. Whisker addition suppressed completely the coarsening of grain, as observed in 10 vol% whisker added layer.

Figure 6 reveals that the bonding at the interface between the layers is perfectly attained.

Fig. 2. Hardness vs. composition of composite.
● whisker enriched layer, ○ whisker poor layer.

Fig. 3. Flexural strength vs. composition of composite.
● whisker enriched layer, ○ whisker poor layer.

Fig. 4. Fracture toughness vs. composition of composite.
● whisker enriched layer, ○ whisker poor layer.

Fig. 5. Microstructure of the fracture surface of the 0/10 composite.

Fig. 6. Microstructure of the fracture surface of the 10/20 composite.

4. Discussion

As already mentioned, the 10/40 composite has been fractured during cooling to room temperature due to resultant thermal stresses owing to the thermal expansion mismatch between the two layers. Then, the thermal stresses are discussed in detail.

Table 1 shows the values of Young's modulus (E), Poisson's ratio (ν) and thermal expansion coefficient for Al_2O_3 , SiC and Al_2O_3 -SiC composites. Ordinary, in hot-pressed composite, whiskers are randomly oriented in a plane perpendicular to the pressing direction. In the plane, composite is assumed to be homogeneous mechanically and thermoelastically. Hence, approximate values of the composites are calculated with parallel model of mixtures law.

Table 1. Numerical values of thermoelastic constants of composite.

Materials	$E(\text{GPa})$	ν	$\alpha (10^{-6})$
Al_2O_3	380	0.2	8.8
SiC	414	0.14	4.7
Al_2O_3 -10%SiC	383	0.19	8.4
Al_2O_3 -20%SiC	386	0.18	8.0
Al_2O_3 -30%SiC	389	0.18	7.6
Al_2O_3 -40%SiC	392	0.17	7.2

Fig. 7. Coordinate of the composite for thermal stress analysis.

culated with parallel model of mixtures law.

As illustrated in Fig. 7, owing to the cylindrical symmetry of the composite, we choose the center axis of the composite as z axis and its vertical direction as r . No bending of the specimen was observed

(A)

(B)

(C)

Fig. 8. Distribution of thermal stresses in the composite: (A) shows axial distribution of σ_θ and σ_r at the central axis of the disc ($\sigma_\theta = \sigma_r$). (B) shows radial distribution of σ_θ for various z . (C) shows radial distribution of σ_z .

$$p = E/(1-\nu) \cdot \xi \quad \xi = (\alpha - \beta) \cdot (t - t_0)$$

a = radius of disc

despite its thin thickness. Hence, the analytical results obtained for the cylindrical joint with infinite length⁷⁾ can be applicable to this specimen except axial stress σ_z . It might be diminished somehow since the upper and lower surfaces of the composite are stress free. Figure 8 illustrates distribution of thermal stresses σ_r , σ_θ , σ_z , where the maximum value of stresses is given by the following equation,

$$\sigma_{\max} = \frac{E}{2(1-\nu)}(\alpha - \beta)(t - t_0) \quad (1)$$

where, α and β denote the thermal expansion coefficients for the whisker poor and enriched layers of the composite, respectively. And, t and t_0 denote the sintering temperature (1800°C) and room temperature (20°C), respectively. As seen in Figs. 8(A) (B) (C), the tensile stresses σ_r and σ_θ occur in the whisker poor layer with high thermal expansion coefficient. σ_θ is generally larger than σ_r , though, their maximum value coincides. Hence, it suggests that the fracture of the composite occurs mainly owing to this stress. The tensile σ_z occurs in the whisker enriched layer of composite which is enough reinforced by whisker addition. Therefore, this stress is ineffective to weaken the composite.

In Fig. 9, the calculated values of the maximum thermal stress is compared with the flexural strength of composites. The flexural strengths of 0/10, 10/20 and 10/30 composites are higher than stresses estimated from Eq. (1). Consequently, no crack or fracture has been occurred in these composites. In the 10/40 composite, the calculated maximum stress value is comparable with the flexural strength of whisker poor layer (10 vol%). Indeed, the whisker

Fig. 9. Comparison of the calculated value of the maximum thermal stress with the flexural strength of composites. ● whisker enriched layer, ○ whisker poor layer, □ The value of maximum thermal stress calculated from Eq. (1).

Fig. 10. The surface of the whisker poor layer in the 10/40 composite showing cracks resulted by thermal stress.

poor layer of the 10/40 composite (Fig. 10) shows that several cracks are running axially. This suggests that cracks are mainly caused by the tangential stress σ_θ .

5. Conclusions

The two-layered hybrid composites were successfully prepared by hot-pressing. Improvement was observed in the strength of the composites which whisker poor layer is reinforced by 10 vol% whisker addition. Cracking and failure have been occurred in the composite when thermal stress is comparable with the strength of whisker poor layer. Namely, the flexural strengths of the 0/10, 10/20 and 10/30 composites are higher than residual thermal stress estimated theoretically, while that of the 10/40 composite is lower. These facts agree with the fracture behaviors of these composites experimentally observed.

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