PREPARATION AND MECHANICAL PROPERTIES OF CARBON NANOFIBER/ ALUMINA COMPOSITE MATERIALS

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1. Introduction

Recently, there have been increasing researches on carbon nanotubes (CNTs) reinforced ceramic matrix composite materials because CNTs have high strength and are expected to improve the toughness of ceramics. The difficulty in dispersion of CNTs in matrix poses a problem for fabricating such composite materials. The results obtained are diversifying [1-3]. On the other hand, carbon nanofibers (CNFs) with a diameter of about 100 nm and straight shape are commercially fabricated. These CNFs may be good candidate for the reinforcement of ceramics, but few researches have been focused on it. In this paper, CNFs reinforced alumina composite materials (CNF/Al_2O_3) were prepared by pressure-less sintering and hot-pressing, the porosity, microstructure and fracture toughness of CNF/Al_2O_3 samples were measured to evaluate the effect of CNFs on the densification process and mechanical properties of alumina.

2. Experimental

2.1 Preparation of CNF/Al_2O_3 composite powders

The preparation process of CNF/Al_2O_3 mixing powder is shown in Figure 1. CNFs (Showa Denko) were treated in HNO_3 (69%) at 110ºC for 1 hour to improve their dispersion in water. Alumina slurry was prepared by mixing Al_2O_3 powder (60g) and H_2O (30g) and dispersant 0.91g (1.5wt%) and ball-milling for 24 hours. The acid-treated CNFs were added to the alumina slurry with 3 vol.% of CNFs and followed by mixing and grinding.

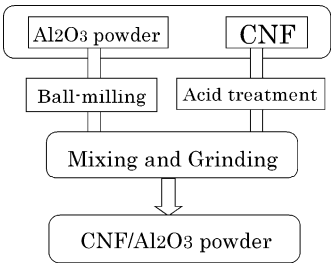

Figure1 Preparation process of CNF/Al2O3 composite powder

2.2 Preparation of alumina and CNF/Al2O3 composite materials by pressure-less sintering and hot-pressing process

The alumina powder and CNF/Al2O3 composite powder were formed to the size of $50\times4\times4mm^3$ and then pressure-less sintered in argon atmosphere at 1300, 1400, 1450ºC for 2 hours, respectively. The samples were denoted as PL-Al1300, PL-CNF1300, PL-CNF1400, PL-1450, respectively. Both the powders were also hot-pressed in a graphite module in argon atmosphere at 1300, 1400, 1450ºC under a pressure of 30 MPa for 1 hour, respectively. The samples were denoted as HP-Al1300, HP-CNF1400, HP-CNF1450, respectively.

2.3 Evaluation method

The morphology of CNFs and alumina powders was observed by TEM. The density and porosity of sintered samples were measured by Archimedes method. Fracture surfaces of the samples were examined using SEM. Fracture toughness (KIC) of the samples were measured using indentation fracture method (IF methods) with diamond indentation angle of 136º under a load of 100 N with a speed of 0.1mm/min, and the K_{IC} values then were calculated from length of the cracks according to the following equation,

$$K_{IC} = 0.026\times\frac{E^{0.5}P^{0.5}a}{c^{1.5}} \qquad \ldots (1)$$

where E is Young's module (GPa), P the load (N), a : half of the length of diagonal (m), c : half of the length of crack (m).

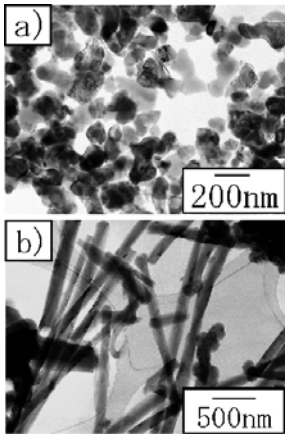

Figure 2 The TEM micrographs of a)Al2O3 powder and b)carbon nanofiber

3. Result and discussion

TEM micrographs of the acid-treated CNFs and alumina raw powder are shown in Figure 2. It can be seen that the diameter of CNFs was about 145 nm, and the size of alumina powders was about 200 nm. Table 1 shows the densities,

porosities and fracture toughness of the samples fabricated by pressure-less sintering and hot-pressing. The porosity of PL-Al1300 was 1.32%, suggesting the sintering at 1300ºC was adequate for monolithic alumina. PL-Al1300 has a porosity of about 14%, significantly higher than PL-Al1300. The sintering at 1400ºC and 1450ºC reduced porosity to about 10% and 8%, respectively. The difficulty of sintering of CNF/Al_2O_3 is because CNFs limited the shrinkage

Table 1 Results of Pressureless sintering and Hot press process

Sintering Method	Pressureless sintering				Hot-pressing			
Samples	PL-Al1300	PL-CNF1300	PL-CNF1400	PL-CNF1450	HP-Al1300	HP-CNF1300	HP-CNF1400	HP-CNF1450
Bulk density($g \cdot cm^{-3}$)	3.91	3.36	3.50	3.58	3.94	3.75	3.87	3.90
Open porosity(%)	0.30	9.28	7.12	2.02	0.03	1.74	0.03	0.02
Closed porosity(%)	1.04	4.55	3.24	6.09	0.82	2.33	0.78	0.01
Total porosity(%)	1.32	13.82	10.36	8.11	0.86	4.07	0.80	0.03
K_{IC} ($MPa \cdot m^{0.5}$)	2.9	3.6	3.6	3.1	2.8	3.4	3.4	3.1

of the samples during sintering. Therefore, hot-pressing technique was used to fabricate the composite materials. The porosity of HP-Al1300 was 0.8%, slightly lower than that of PL-Al1300. HP-CNF1300 had a porosity of about 4%, much lower than that of PL-samples. The HP-CNF1400 and HP-CNF1450 showed porosity of 0.8% and 0.03%, indicating the CNF/Al_2O_3 composite materials were almost completely densified under these conditions.

Figure 3 shows the SEM micrographs of fracture surfaces of the samples. It is seen that CNFs were almost homogeneously distributed in the matrix. The pull-out of CNFs from alumina matrix was also observed. This phenomenon suggested weak interfacial bonding between alumina and CNFs. In pressure-less sintered samples, the fracture toughness of CNF/Al_2O_3 composite materials was 20% higher than that of monolithic alumina. It is thought that the increase of the fracture toughness value may not accurate because pores in CNF/Al_2O_3 might block progress of cracks. The fracture toughness of CNF/Al_2O_3 fabricated by hot-pressed at 1300 and 1400ºC was 20% higher than monolithic alumina samples. Since the porosity of HP-CNF1400 was only 0.8%, its fracture surface value was trustworthy. However, the fracture toughness of HP-CNF1450 decreased in comparison to HP-CNF1400. It is thought that the strong bonding between alumina grains resulted in transgranular fracture and thus a lower fracture toughness. This phenomenon was also observed in the pressure-less sintering samples at 1450ºC.

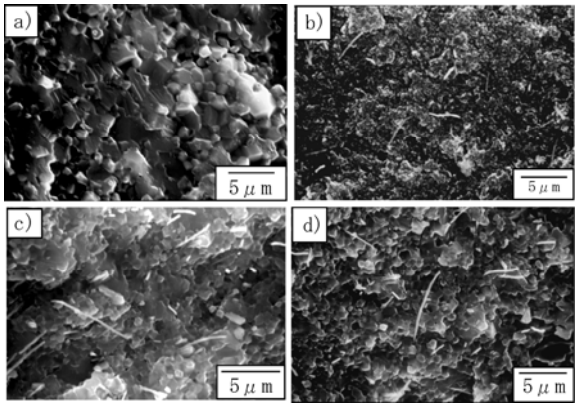

Figure 3 The SEM micrographs of fractured surface of a)PL-Al1300, b)PL-CNF1300, c)PL-CNF1400, d)PL-CNF1450.

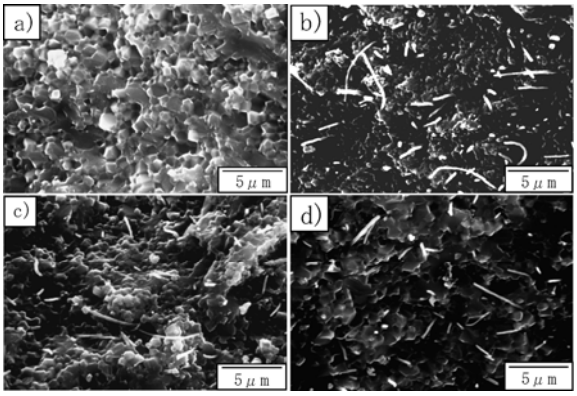

Figure 4 The SEM micrographs of fracture surface of a)HP-Al1300, b)HP-CNF1300, c)HP-CNF1400, d)HP-CNF1450.

4. Conclusions

1). CNFs were almost homogeneously distributed in alumina matrix by mixing and grinding process.
2). The optimum sintering condition of CNF/Al_2O_3 is hot-pressing at 1400ºC.
3). The fracture toughness has been improved by about 20% for addition of 3 vol% CNFs in alumina matrix.

References

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