

Effect of Powder Synthesis Processing on the Microstructure and Electrical Conductivity of Sintered CNTs/Fe/Al₂O₃ Nanocomposites

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Abstract

The microstructure and electrical conductivity of CNTs dispersed Al₂O₃ nanocomposites depending on the powder processing and CNTs content were demonstrated. The composite powders with homogeneous dispersion of CNTs could be synthesized by a catalytic route for direct formation of CNTs on nano-sized Fe dispersed Al₂O₃ powders. The sintered nanocomposite using the composite powder with directly synthesized CNTs showed homogeneous microstructure and enhanced electrical conductivity. The influence of powder processing on the properties of sintered nanocomposites was discussed by the observed microstructural features.

Keywords : Carbon nanotubes, Al₂O₃ nanocomposites, Microstructure, Electrical conductivity

1. Introduction

Carbon nanotubes (CNT) have recently emerged as reinforcement materials of ceramics, because of their unique and excellent properties [1]. They are highly rigid with a Young's modulus of about 1 TPa and strength of about 30 GPa. In addition, it has been reported that CNT can give an electrical conductivity to insulating materials [2]. However, the extraordinary properties of CNT have not been successfully used in composites. This is basically attributed to the difficulty in homogeneous mixing between CNT and matrix powders as well as their full densification with sound microstructure [3].

In the early work, we suggested that the Al₂O₃ composite powders with homogeneous dispersion of CNTs can be synthesized by a catalytic route for the in-situ formation of CNTs on nano-sized Fe dispersed Al₂O₃ powders [4]. In this study, we demonstrate the densification of CNTs/Fe/Al₂O₃ powders and their microstructural characteristics. In addition, the electrical conductivity of the composites was measured at room temperature and analyzed based on the observed microstructural characteristics of sintered composites dependent on the powder synthesis processing.

2. Experimental and Results

High-purity α -Al₂O₃ powder with particle size less than 0.2 μ m (Sumitomo Chem. Co., Japan) and Fe-nitrate (High Purity Chemicals Lab., Japan) were used as the matrix material and the source material for the Fe catalyst, respectively. The preparation of Al₂O₃ composite powders with nano-sized Fe catalyst was described in previous

papers [4]. Two methods (Methods A and Methods B) of CNTs addition were used. Method A was the synthesis of CNTs on the Fe/Al₂O₃ powder mixture by thermal CVD and further addition of Al₂O₃ powders to the CNTs/Fe/ Al₂O₃ composite powder by ball milling. In this process, the final CNTs content were 5 vol% and 20 vol%. Method B was the direct synthesis of 4 vol% and 8 vol% of CNTs on the powder mixture without further addition of matrix powder. Sintering was accomplished via spark plasma sintering at the temperature of 1400°C for 10 min under a vacuum and an applied pressure of 30 MPa. Electrical conductivity was obtained by 4-probe point method.

Typical SEM images of composite powders with 5 vol% CNTs prepared by Method A and 4 vol% CNTs by Method B are shown in Fig. 1(a) and (b), respectively. As clearly shown in Fig. 1(a), the composite powder, prepared from the synthesis of CNTs by thermal CVD and further addition of Al₂O₃ powders to the synthesized CNTs/Fe/Al₂O₃ composite powder by ball milling, exhibited an inhomogeneous microstructure with the agglomeration of Al₂O₃ powders. Conversely, homogeneous dispersion of CNTs in the composite powder, prepared by Method B, was observed. Thus, this result indicated that the composite powder with homogeneous distribution of CNTs can be synthesized by Method B.

To analyze an effect of initial powder mixtures on the composite properties, the powder mixtures prepared by different processing were sintered at 1400°C for 10 min. In comparison with 5 vol% CNTs/Fe/Al₂O₃ nanocomposite prepared by Method A, the composite with 20 vol% CNTs exhibited relatively fine grains of Al₂O₃. Also, the microstructure of the sintered nanocomposites prepared by Method B was characterized by refinement of matrix

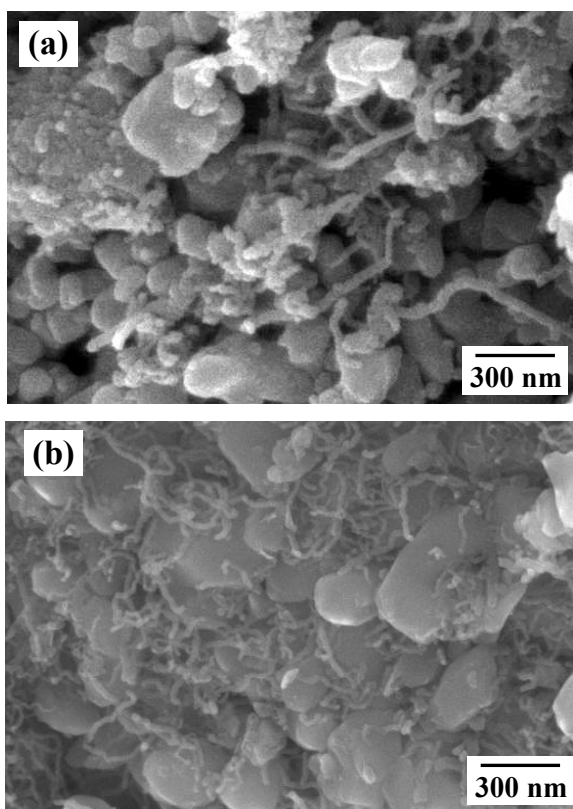


Fig. 1. SEM micrographs of composite powders, (a) 5 vol% CNTs prepared by Method A and (b) 4 vol% CNTs prepared by Method B.

Table 1. Relative density and electrical conductivity for sintered nanocomposites.

Powder processing	CNTs cont. [vol%]	Rel. density [%]	Electr. Conduct. [S/cm]
Method A	5	99.0	-
	20	93.4	1.37
Method B	4	93.0	6.32
	8	81.3	16.08

grains with the increase in CNTs content.

Table 1 summarizes the relative density and electrical conductivity of the sintered composites depending on powder processing and CNTs content. Increasing CNTs content produced a decrease of relative density regardless of processing method. The decrease in relative density can be explained by the resistance of the $\text{Al}_2\text{O}_3/\text{CNTs}$ interface to the diffusion process, as suggested by Stearns et al in $\text{Al}_2\text{O}_3/\text{SiC}$ nanocomposite systems [5]. On the other hand, it is interesting to note that the relative density of the composites prepared by Method A showed higher value than

that prepared by Method B. However, considering the densification retardation by the presence of CNTs, this result shows opposite tendency with the grain growth behavior of conventional composite.

The electrical conductivity variance of composites with different processing and CNTs content is also shown in Table 1. The electrical conductivity of nanocomposites prepared by Method B is in the range of 6.32-16.08 S/cm whereas monolithic Al_2O_3 is insulating ($<10^{-9}$ S/cm). Moreover, the values are fairly well correlated to the CNTs content. However, the electrical conductivity of the composites prepared by Method A is not measured (5 vol% CNTs) or low value (20 vol% CNTs). It was observed that the microstructure of composites prepared by Method B was characterized by a homogeneous dispersion of CNTs within the matrix compared to that by Method A for which the CNTs agglomerations were formed. This probably leads to a better connectivity of the CNTs in composites prepared by Method B [6], and thus the composites show high electrical conductivity as summarized in Table 1.

3. Summary

The composite powders, prepared by the direct synthesis of required amount of CNTs using thermal CVD, showed the homogeneous distribution of CNTs. With increasing CNTs content the sintered nanocomposites exhibited decrease in matrix grain size and relative density. The relative density of the composites prepared by Method B showed lower value than that prepared by Method A due to the formation of CNTs agglomerations. The composites prepared by Method B showed increased electrical conductivity. The behavior of electrical conductivity depending on powder processing was explained by the formation of CNTs agglomerations and their influence on the connectivity of the CNTs in composites.

4. References

1. S. Iijima, *Nature*, **354**, 56(1991).
2. R.S. Ruoff and D.C. Lorents, *Carbon*, **33**[7], 925(1995).
3. S. Rul, F. Lefevre-schlick, E. Capria, Ch. Laurent and A. Peigney, *Acta Mater.*, **52**[4], 1061(2004).
4. C.D. Park, H.J. Jeon, H.J. Wang, Y.H. Choa, S.T. Oh, K.M. Kang and S.G. Kang, *Mater. Sci. Forum*, **449-452**, 797(2004).
5. L.C. Stearns, J. Zhao and M.P. Harmer, *J. Europ. Ceram. Soc.*, **10**[6], 473(1992).
6. E. Flahaut, A. Peigney, Ch. Laurent, Ch. Marliere, F. Chastel and A. Rousset, *Acta Mater.*, **48**[14], 3803 (2000).