Characteristics of Al$_2$O$_3$–SiC Nanocomposite Prepared by Sol-Gel Processing

Yueping XU, Atsushi NAKAHIRA and Koichi NIIHARA
The Institute of Scientific & Industrial Research, Osaka University, 8-1, Mihogaoka, Ibaraki-shi, Osaka 567

ソル・ゲル法により作製したAl$_2$O$_3$–SiCナノ複合材料の特性
徐 躍萍・中平 敦・新原皓一
大阪大学産業科学研究所，567茨木市美濃ヶ丘8-1

[Received December 22, 1993; Accepted January 27, 1994]

Al$_2$O$_3$–SiC nanocomposite powders containing small crystalline SiC powders have been prepared by sol-gel processing. Less than 50 nm ultrasonic dispersion and sedimentation techniques were used in the presence of an organic solvent to remove the agglomerates of SiC. Homogeneous SiC powders with narrow size distributions were mixed with an Al$_2$O$_3$ sol. The characteristics of the nanocomposite powder as well as microstructure of the sintered Al$_2$O$_3$–SiC nanocomposite were investigated by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Sol-gel processing was shown to produce Al$_2$O$_3$–SiC nanocomposites with homogeneous distribution of SiC in the Al$_2$O$_3$ matrix.

Key-words: Al$_2$O$_3$–SiC, Nanocomposite, Sol-gel processing, Microstructural characteristics

1. Introduction

It is well known that the incorporation of small amount of nano-sized SiC particles into Al$_2$O$_3$ matrix could significantly enhance the mechanical properties compared to monolithic Al$_2$O$_3$. Many impressive results have been obtained using the conventional ball milling methods.$^{1-4}$ Even so there are still many problems remaining to be solved, e.g. how to make a uniform dispersion of ultrafine particles within the fine grain ceramic matrix. Sol-gel processing has a potential to make the very fine second phase homogeneously dispersed in the matrix by tailoring a wide range of processing parameters.$^{5-7}$

In this paper, sol-gel processing is used for the preparation of fine grained alumina nanocomposite containing 5 vol.% of SiC. By controlling the parameters of sol-gel processing after removing the SiC agglomerates, the homogeneous dispersion of SiC into the Al$_2$O$_3$–SiC nanocomposite was obtained. The microstructural characteristics of this nanocomposite were investigated by scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

2. Experimental procedures

Commercially available high purity boehmite with BET surface area of 240 m$^2$/g (Japan. Abrasive Co., Ltd., Osaka) was used as the source of Al$_2$O$_3$ sol. The SiC particles were obtained from Ibiden Co. with a mean particle size <0.3 μm. In order to enhance the transformation and the densification of the alumina matrix, a small amount of α-Al$_2$O$_3$ (2 wt%) was added (99.99%, Sumitomo AKP-53, Osaka, Japan).$^8$ Before mixing the Al$_2$O$_3$ sol and SiC particles, it is very important to peptize boehmite and remove the hard agglomerates of SiC. The SiC particle size was measured by centrifugal particle size analyser (Shimadzu Corporation). Clear hydrosols were prepared by dispersing 14 wt% boehmite powder in distilled water and adjusting the pH with nitric acid. The optimum pH and mixing time conditions to mix transparent Al$_2$O$_3$ sols with non-hard agglomerate SiC are 3.5 and 6h, respectively. After mixing, the mixture was dried at 70°C for 40h in an electric oven. Calcining was performed at 600°C for 2h in air to burn off any residual organics and water from powder processing. The dried ultrafine powders were hot pressed in N$_2$ atmosphere at 1600°C/1h with an applied pressure of 30 MPa. The microstructural characteristics of this nanocomposite were examined by SEM as well as TEM. SEM was performed on the polished surface after thermal etched at 1400°C/40 min in vacuum and the grain size was measured using the linear intercept method with at least 400 intercepts for each measurement. TEM was used to observe the distribution of SiC and the microstructure of nanocomposite.

3. Results and discussion

3.1 Peptization and dispersion of Al$_2$O$_3$ sol and SiC particles

The structure of boehmite is like linked cardhouse structure and its surface contains many OH groups. In order to stabilize the Al$_2$O$_3$ sol suspension for preparing Al$_2$O$_3$–SiC nanocomposites, it is necessary to peptize boehmite and breakdown the cross link-force between the unit layers. An elegant way to achieve deflocculation would be to reverse the negative surface charges and to create well-developed positive surface charges by adjusting pH value in the
boehmite suspension. Then the strong surface to surface repulsion would result in a breakdown of the weak chemical or physical bonds between the linked card-house structures. The experimental results show that the mean particle size of boehmite decreases with the decrease of pH value, because of the reverse of the surface charges making the Al₂O₃ sol stabilized. The transparent Al₂O₃ sol can be obtained while the pH=3.5.

For the SiC powder, ethanol was chosen as a dispersant solvent. The dispersion properties of SiC powders are strongly dependent upon their surface characteristics. The experimental results show that the particle size of SiC changes as a function of pH value. It's evident that the optimum condition for mixing Al₂O₃ sol and SiC particles is pH about 3.0-3.5. In order to remove the SiC agglomerates, ultrasonic dispersion and sedimentation techniques were used. After removing the agglomerates in the sediment, SiC was chosen as the second particle in the Al₂O₃-SiC nanocomposite. Figure 1 shows the particle size distribution of SiC before and after treatment. It is evident that the large, hard agglomerates of SiC have been removed and the particle size distribution of SiC become much smaller and narrower. It is also observed by TEM that the primary particle size is about 30-50 nm, while the secondary particle size is 0.17 μm which was measured by centrifugal particle size analyser.

On the basis of the results above, the optimum condition for the mixing of Al₂O₃ sol and SiC ultrafine particle was chosen. Figure 2 shows the nanocomposite powder obtained using this process. We observed the presence of the phase with high contrast within the Al₂O₃, and the EDX analysis verifies that the dark particle is the SiC as shown by the arrow in Fig. 2. This means that SiC can be well dispersed within the Al₂O₃ matrix powders by sol-gel processing. Further analyses of microstructure of Al₂O₃-SiC nanocomposite were made using SEM and TEM. Figure 3 shows that the grain size distribution of
nanocomposite obtained by sol-gel processing and ball-milling after heat treatment at 1600°C and 30 MPa. It is evident that much smaller and narrower of Al₂O₃ matrix grains can be obtained by the sol-gel processing. This is caused by well dispersed SiC particles which inhibit the grain growth of Al₂O₃ matrix. Figure 4 shows that most of the SiC particles are well dispersed and located within Al₂O₃ grains, and these ultrafine particles cause many dislocation because of the mismatch of thermal expansion between Al₂O₃ and SiC. As we had reported that such dislocation network can make the refinement of matrix Al₂O₃ grains and have good effects on the mechanical properties. The relationship between the structure of the dislocation and the mechanical properties requires further studies.

4. Conclusion
(1) By sol-gel processing, the Al₂O₃–SiC nanocomposite powder could be prepared such that the SiC particles are dispersed within the Al₂O₃ powder.
(2) Sol-gel processing results in a homogeneous microstructure of the Al₂O₃ matrix after sintering compared to the ball milling methods.
(3) In the Al₂O₃–SiC nanocomposite, most of the SiC particles are well dispersed within the Al₂O₃ grain. Many dislocations were observed within the Al₂O₃ grains.

References