Integrated Composite Particles for Designing of Microstructural Controlled Nanocomposite

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Introduction

The fabrication of composite with nano-size particles dispersed in ceramic matrix often improves not only the mechanical properties such as failure strength, fracture toughness, fatigue and ware resistances, etc. of structural ceramics but also the electrical, magnetic, and optical properties of functional ceramics. In composite processing, in general, to properly design a desired microstructure/property, it will be very essential that the second phase nano particles must be well dispersed in the matrix. In the conventional fabrication technique of nanocomposites, mechanical mixing of raw powders in a ball mill has often been conducted. However, this technique might be insufficient to produce nanocomposites with homogeneous microstructures due to the aggregation of nano-sized raw powder. The processing through an aqueous solution such as coprecipitation method has also been extensively studied to fabricate nanocomposite particles. However, these techniques are still in its developing stages and containing some of practical disadvantages and difficulties.

The layer by layer (LbL) adsorption method has been recognized as one of powerful techniques to fabricate an ultrathin nano film coated onto a solid substrate. The most of studies on LbL techunique have been conducted to produce multilayered nano films of polyelectrolytes, and then utilize them to adsorb various kinds of chemical species, such as protein, nucleic acid, nanoparticles, etc. Recently, an LbL technique was successfully applied not only to flat surfaces such as flat substrates, but also to spherical grains, to fabricate alternatively multilayered composites on colloidal particles[1]. LbL-method will be one of the simplest and the most versatile processing techniques for fabricating nanocomposite powders with homogeneous microstructure.

In this study, we propose a novel processing technique to fabricate nanocomposite powders. The composite powders obtained in this study, *i.e.*, the integrated composite powders by coating nano-sized additive particles on the surface of matrix particles.

Experimental

Materials

The starting powder for the matrix is a high-purity granulated spherical alumina (Taimicron alumina; the mean grain size of about 50μm comprising with nine alumina particles of 100nm, Taimei Chemical Co. Ltd., Japan). A suspension of nano-sized carbon sphere (Carbon Nano Spheres, Tokai carbon Co. Ltd., Japan) with the mean particle size of about 260nm was used as the additive to the matrix. The suspended carbon nanotube was also used as another additive. Details of the preparation of this carbon nanotube was given in literatures. Poly(sodium 4-styrene sulfonate) (PSS; Mw 70,000) and poly(diallyldimethylammonium chloride) (PDDA; Mw 100,000 - 200,000) were supplied from Aldrich as the polyelectrolytes used in the present technique.

Fabrication of composite powder

The first stage of fabrication of integrated composite powder involves the sequential adsorption of oppositely charged polyelectrolytes, i.e., PSS and PDDA on the surface of spherical Al_2O_3 matrix grain in order to produce a uniformly as well as stably charged surface to enhance the adsorption of carbon nanoparticles. The Al_2O_3 particles were dispersed in PSS using a homogenizer to alter their surface charge from positive to negative, and then incubated for 15min with a magnetic stirrer. The suspended particles were separated by centrifuging followed by washing with distilled water three times to remove the excess PSS. To enhance the density of electrical charges induced on the surfaces of Al_2O_3 particles, the PSS adsorbed Al_2O_3 particles thus prepared were repeatedly coated with PDDA and the PSS to produce a multilayered structure, i.e., Al_2O_3-(PSS/PDDA/PSS) having the charge density sufficient enough for adsorbing the carbon particles. Along with the formation of multilayers coated on Al_2O_3 particles, the carbon particles having multilayered structure, i.e., C-(PDDA/PSS/PDDA) were also produced. Finally, the Al_2O_3 particles, Al_2O_3-(PSS/ PDDA/PSS) having positively charged outer surfaces, were mixed with the carbon particles, C-(PDDA/PSS/PDDA) having negatively charged surfaces, to fabricate the composite powder, i.e., the alumina particles coated with carbon nanoparticles.

The carbon nanotube having negatively charged surface was suspended in the water. No additional surface treatments were needed for LbL technique due to the surface charge was large enough to fabricate integrated composite particle. Al_2O_3- (PSS / PDDA / PSS) having positively charged outer surfaces, were simply mixed with the suspension of carbon nanotube.

A quartz crystal micro-balancer (QCM) was used for quantitatively monitoring the mass changes associated with the sequential LbL processes. The ζ-potential was also measured by a ζ–potential analyzer (ELS-8000, Ohtsuka Electronics, Japan) to examine the transitional change of charge density at each step of layering. The surface morphology of the integrated composite powder was studied by a scanning electron microscope (SEM, S-4800, Hitachi, Japan).

Results and Discussion

From the results of the change in ζ–potential as a function of the LbL-number of polyerectrolyte adsorptions, it was confirmed that the Al_2O_3-(PSS / PDDA/ PSS) and the C- (PDDA/PSS/PDDA), *i.e.*, N=3, are sufficient enough to attain a good absorption of the carbon particles on the Al_2O_3 surface via the LbL-induced electrostatic attraction.

A SEM image of the fabricated Al_2O_3-C composite particles is shown in Fig. 1, indicating that nano-sized carbon particles and carbon nanotube are successfully adsorbed around Al_2O_3 spherical particles, respectively.

It was concluded that the LbL technique examined in this study provides an efficient tool for fabricating the nanocomposite powders.

References

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Acknowledgements:

A part of this study is supported by Toukai Foundation for Technology, JGC-S Scholarship Foundation (Grant for young researcher) and The New Energy and Industrial Technology Development Organization (NEDO) (Grant for Industrial Technology Research Program No. 09A19002a).

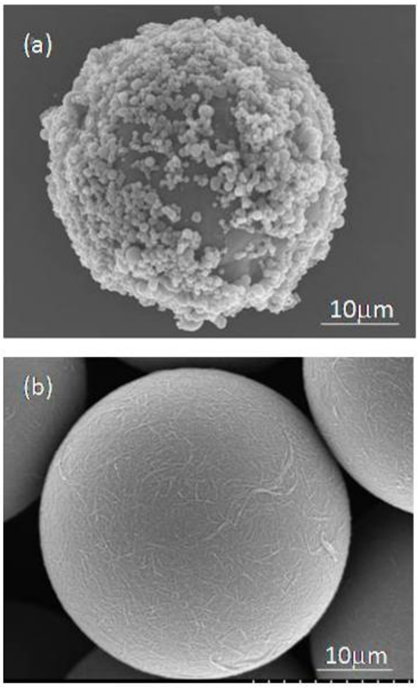

Fig. 1 Integrated composite particles obtained by LbL technique. (a) carbon nano particle coated- and (b) carbon nanotube coated-alumina.