

Fabrication of ultrafine Nd-Fe-B powder by a modified reduction-diffusion process

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(Received 2006-08-15)

Abstract: In order to obtain ultrafine Nd-Fe-B powder, a spray-dried precursor was treated by reduction-diffusion (R/D) process. And, unlike the conventional R/D process, calcium reduction that is a crucial step for the formation of Nd₂Fe₁₄B was performed without conglomerating the precursor with Ca powder. By adopting this modified process, it is possible to synthesize the hard magnetic Nd₂Fe₁₄B at the reaction temperature as low as 850 °C. The average size of Nd₂Fe₁₄B particles that are uniformly distributed in the optimally treated powder was << 1 μm. Most Nd₂Fe₁₄B particles were enclosed with thin layers of Nd-rich phase. Typical magnetic properties of such powder without eliminating impurity CaO were $iH_c = \sim 5.9$ kOe, $B_r = \sim 5.5$ kG, and $(BH)_{\max} = \sim 6$ MGOe.

Key words: NdFeB powder; spray drying; reduction-diffusion process; Nd₂Fe₁₄B; magnetic powder

1. Introduction

Currently, most Nd-Fe-B magnetic powders for bonded or sintered magnets are prepared by crushing Nd-Fe-B melt-spun flakes or ingots that are subsequently annealed or HDDR treated, which needs additional energy and increases production cost because of using high-purity metals as starting materials. On the other hand, it is possible to obtain the Nd-Fe-B powder inexpensively, without the pulverizing sequence, by employing reduction-diffusion (R/D) process[1-2] in which cheaper raw materials are used. In the reduction-diffusion process, Nd-oxides are usually reduced by Ca in the presence of Fe or Fe-oxide and hard magnetic Nd₂Fe₁₄B is formed simultaneously by solid-state diffusion. It has been shown that the size of final Nd-Fe-B product of the R/D process depends on the size of starting materials[3-4]. We have also successfully prepared fine Nd-Fe-B powder (~ 1 μm) by adopting the

spray-drying as an initial stage of the R/D process[5-7]. However, it is still not easy to obtain the powder consisting of finely distributed Nd₂Fe₁₄B and possessing reasonable coercivity.

As an attempt to prepare such fine powder, the calcium reduction that is a crucial step for the formation of Nd₂Fe₁₄B in the R/D process was tried without conglomerating a sprayed-dried precursor with Ca powder in this study. And effect of this process modification on phase formation, morphologies, microstructures, and properties of Nd-Fe-B powder was investigated.

2. Experimental

Nd(NO₃)₆·H₂O, Fe(NO₃)₃·9H₂O, and H₃BO₃ were dissolved into deionized water, adding neodymium hydrate 10% more for the compensation of Nd loss during reactions, to make spray-drying solutions for the target composition of Nd₁₅Fe₇₇B₈. Procedures of spray drying, debinding, milling, and hydrogen reduc-

tion in the R/D process were described in detail in Refs. [5-6]. The H₂-reduced precursor was well mixed with Ca powder (Ca : powder = 2:1), and then the mixed powder itself, not as a conventional compact, was reduced for 3 h at 850-1100 °C under Ar atmosphere. The reacted powder was subsequently washed in distilled water and dried in a vacuum. Characterization of the powder was performed by using Cu K α X-ray diffraction, scanning electron microscopy, transmission electron microscopy, and thermo-magnetic analysis. Magnetic properties were measured with a VSM with a maximum applied field of 15 kOe.

3. Results and discussion

As shown in Fig. 1, the calcium-reduced powders were all composed of Nd₂Fe₁₄B and CaO regardless of the reduction temperature. It tells us that hard magnetic Nd₂Fe₁₄B can be readily formed through a solid-state reaction even though the Ca reduction is carried out in a mixed powder state, i. e., as the hydrogen-reduced precursor is not consolidated, but just mixed with Ca powder. It is also found that this reduction method has an advantage of lowering the reduction temperature as the Nd₂Fe₁₄B was fully obtained even at 850 °C.

All calcium-reduced powders, as typically shown in Fig. 2 (a), were spherical clusters with the particle size 1 μ m more or less al-

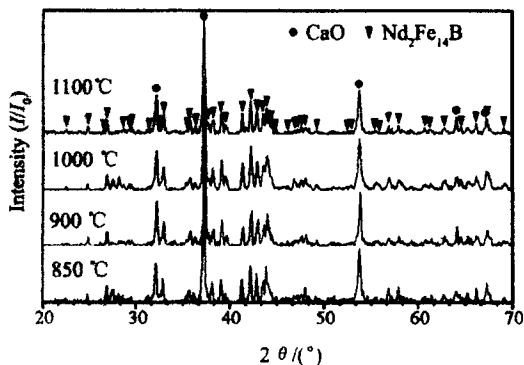


Fig. 1. XRD patterns of Nd-Fe-B powder calcium reduced in the powder state for 3 h at 850, 900, 1000, and 1100 °C. (Ca : powder = 2:1).

though the particles tended to grow as the reduction temperature increased. In fact, they were much smaller than those obtained by conventional Ca reduction[6]. When the powder in Fig. 2 (a) was washed in water and dried in vacuum, most spherical particles in the powder became much smaller than 1 μ m as shown in Fig. 2 (b). Moreover, long rod-type particles that were not found in the calcium-reduced state were often found after washing in water. It indicated that a certain change in shape or phase of the particles occurred during washing[7-8]. In fact, the thermo-magnetic analysis revealed that Nd₂Fe₁₄B was partially decomposed to soft magnetic Nd₂Fe₁₇B_x due to the thermal energy generated by severe exothermic reaction occurred in washing[8]. EDX results revealed that both the spherical and the rod-type particles were Nd₂Fe₁₄B. Such rod-type particles were also found in the Nd-rich Nd-Fe-B alloy[9].

As shown in Fig. 3, Nd₂Fe₁₄B particles in the above mentioned powders were enclosed

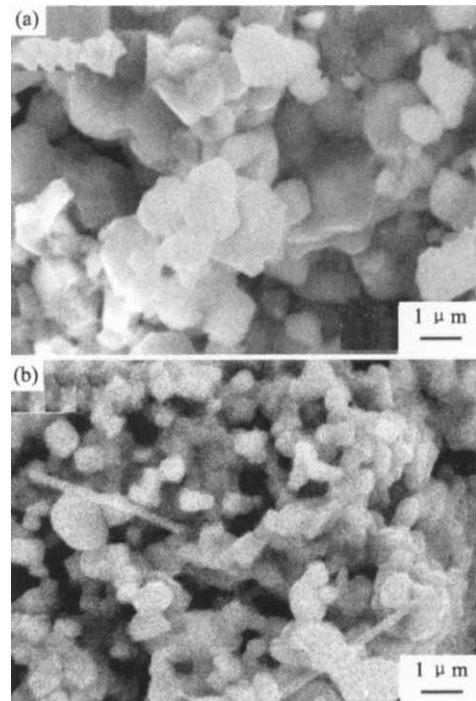


Fig. 2. SEM images of Nd-Fe-B powder calcium reduced in the powder state for 3 h at 1000 °C (a) and subsequently washed in distilled water (b).

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