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습식공정에 의한 Nd-Fe-B 분말 제조에 관한 연구

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Since Nd-Fe-B magnet was first discovered by Sagawa et al [1], many kinds of methods have been developed to fabricate, such as powder metallurgical, rapidly quenching and reduction-diffusion (R-D) processes. Nowadays, powder metallurgical and rapidly quenching methods are commonly used. But both of them need several processes for achieving fine particles and increase the production cost due to the use of high purity metals as raw materials. Compared with above methods, main ad-vantages of our proposed process are the use of a relatively inexpensive Nd oxide as raw material and the direct production of fine alloy powder suitable for further procedures. In this study, a novel route to prepare Nd-Fe-B magnetic particles by utilizing mechanochemical and R-D process was proposed.

Precursors were prepared by spray drying method using the aqueous solutions containing Nd salt, Fe salt and boric acid with stoichiometric ratio. The spray dried powders were desalted at 800 °C for 2 h in air, followed by ball milling for 20 h. To reduce iron oxides, heat treatment of the milled powders was performed under H_2 atmosphere at 800 °C for 2 h. The amount of Calcium (Ca) as a reducing agent in R-D process was mixed with powders obtained by H_2 reduction in appropriate ratio. The R-D of the compacts was carried out at 1000 °C for 3 h in Argon (Ar) atmosphere. For the effective washing, the compact was pulverized to fine powder and the powders were washed with water several times to achieve $Nd_2Fe_{14}B$ powders. The phases and the magnetic properties of the particles were examined by X-ray diffractometer, Scanning electron microscopy and vibrating sample magnetometer.

XRD patterns of each step in this procedure as shown in Fig. 1, depicted that precursors obtained by spray drying was amorphous structure due to volatile compounds and physical adsorption of elements. They were crystallized into oxides of Nd and Fe through desalting at 800 °C that was performed previously by Dong et al. [2]. And then ball milling was performed to triturate the aggregates after desalting. As shown in Fig. 1, Fe oxides were reduced to α-Fe by heat treatment in H_2 atmosphere. After mixing and compacting with reduced powders in H_2 and exceeded Ca granules, R-D process was carried out. $Nd_2Fe_{14}B$ particles were formed and CaO and unreacted Ca were remained. This result came from the reactions as following equations and was almost the same as references by Dong et al. [2] and Jang et al. [3].

$$Nd_2O_3 + 3Ca \rightarrow 2Nd + 3CaO$$

$$Nd + 13Fe + FeB \rightarrow Nd_2Fe_{14}B$$

The final step of washing was performed using water to wash out CaO for 1-3 h. The washed powders of (BH)max reached 15.5 MGOe after washing for 1 h with de-ionized water.

To reduce non-magnetic phase, ball milling process under an ethanol was added in washing process. The final magnetic property was enhanced to 16.7 MGOe of (BH)max with a rectangular demagnetization shape, as shown in Fig. 2. This demonstrates that our process is a promising route for fabrication of Nd-Fe-B magnetic powders, especially for recycling of the Nd magnets. In this paper, we will present the change of phases, morphologies and magnetic properties in $Nd_2Fe_{14}B$ powders in R-D process and discuss the potential application to industry.

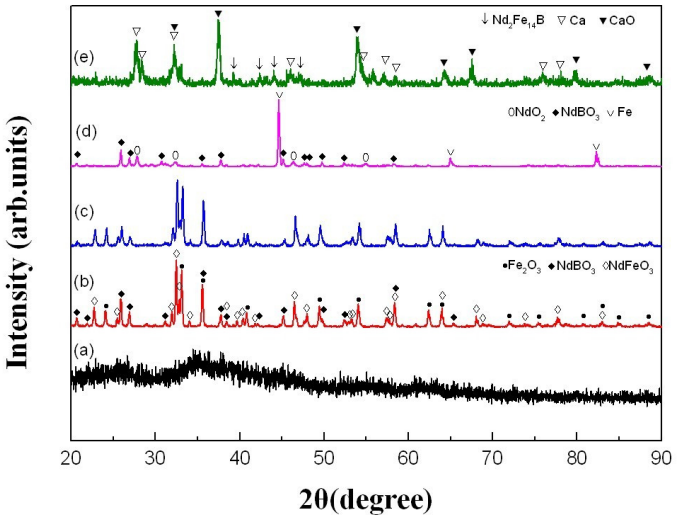

Fig. 1. XRD patterns of powders after
(a) spray-drying, (b) desalting, (c) ball milling, (d) H_2 reduction and (e) Ca reduction.

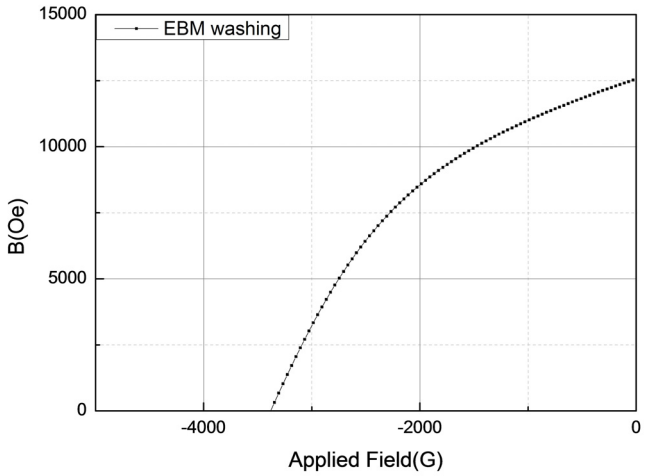

Fig. 2. Demagnetization curve of $Nd_2Fe_{14}B$ powder after ethanol balling milling and washing process.

References

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