Fabrication of hard magnetic Nd-Fe-B powders by a thermochemical process

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Modern permanent magnets, whether they are sintered or bonded, are produced by using magnetic powder. Accordingly, magnetic properties of the magnets primarily depend on the nature of the powder such as particle shape, size, purity, and composition. In order to develop high performance magnets, therefore, these powder parameters should be optimized. In this work, we fabricated Nd-Fe-B powder by thermochemical process, and investigated effect of Ca reduction and washing step on the magnetic properties of the powder.

The Nd-Fe-B powder with the target composition of Nd₁₅Fe $_{7}$ B₈ was prepared by thermochemical process including spray dring, ball milling, H₂ reduction, Ca reduction, washing, and drying. Phase identification of the powder was performed by Cu $_{6}$ x-ray diffraction. Morphologies and microstructures of the powder were examined with a SEM. Magnetic properties of the final powder were measured with a VSM with a maximum applied field of 15 kOe.

In the thermochemical process, the Ca reduction process was mostly essential process due to the formation of hard magnetic Nd₂Fe₁₄B phase from various oxides such as NdFO₃, Nd₂O₃, Fe₂O₃, etc. Therefore, the magnetic properties of the final powders were significantly effected by the phase formation, particle size, and shapes of the Ca reduced powder.

The H_2 reduced powder was reduced at $800 \sim 1100$ °C for 3 hours with the mixing ratio of Ca:powder = 2:1. It was found by SEM observation that the distribution and size of the Ca reduced powder became uniform and small with 1 μ m. Moreover, by TEM observation, not only its shapes were spherical or rectangle with the size of $\sim 0.7 \mu$ m, but also particle edge of powder was the vary smooth and reduced remarkably a impurity such as CaO and Nd-oxide. The magnetic properties of powder Ca reduced at 1000 °C for 3 hours exhibited $_iH_c$ = 5.96 kOe, B_r = 5.52 kG. However, the final powder after washed and dried resulted in poor magnetic properties due to the high exthomeral reaction during the washing process induced a oxidation of the surface of powder and decomposition of Nd₂Fe₁₄B to Nd₂Fe₁₇B_x.

Acknowledgements

This work is supported by dual use technology program (Grant No. 01-IT-MP-07) of Ministry of Commerce, Industry, and Energy.