Mechanical-milling-induced coercivitydegradation in fine Nd-Fe-B-type HDDR Particles and its recovery

K. M. Kim[1*], H.W.Kwon[1], J. G. Lee[2], J.H.Yu[3]
[1]Department of Materials Science and Engineering, Pukyong National University, Busan, South Korea
[2]Korea Institute of Materials Science, Changwon, South Korea

INTRODUCTION

HDDR-treated Nd-Fe-B-type material has unique microstructural feature with very fine grain structure (typically around 0.3 um). Due to this fine grain structure, the Nd-Fe-B-type HDDR material exhibits high coercivity, and much interest has been attracted to utilize this HDDR material powder for high performance resin bonded micro-magnetsfor micro-motor application. For the bonded micro-magnet application, fine particles with high permanent magnetic performance are required essentially. Most common approach for the preparation of the fine particles was mechanical milling of high performance Nd-Fe-B-type sintered magnet. However, the milled particles from the sintered magnet lose radically the coercivity. This may be partly due to the coarse grain size in the sintered magnet (typically 5 – 10 um). It is expected that the coercivity variation of the milled particles from the HDDR material may be different from that of the milled particles from the sintered magnet because of its distinctively fine grain structure (typically 0.3 um). In this article, coercivity variation of the fine Nd-Fe-B-type particles prepared by mechanical milling of the HDDR-treated Nd-Fe-B-type material was investigated. The origin of radical coercivity reduction in Nd-Fe-B-type fine particles prepared by ball milling of the HDDR material and its recovery by chemical etching was discussed.

EXPERIMETAL DETAILS

HDDR-treated $Nd_{12.5}Fe_{80.6}B_{6.4}Ga_{0.3}Nb_{0.2}$material with coercivity of 13.5 kOe was used as a starting material. $Nd_{10}Pr_{2.5}Fe_{80.4}B_6Ga_1Cu_{0.1}$ sintered magnet with coercivity of 12.3 kOe was also used for comparison. The HDDR-treated material and sintered magnet were ball milled (in cyclohexane) to prepare fine particles, and the particle size was controlled by adjusting ball milling time. Some of the milled particles were chemically etched for 10 min using 1% Nital solution to recover the coercivity degraded by the mechanical milling. Hydrogen and oxygen content in the particles was determined by Hydrogen Determinator (LECO, RH-600) and oxygen analyser (Leco TC400). Lattice parameter variation and micro-strain (Williamson-Hall equation) in the $Nd_2Fe_{14}B$-type matrix phase grain in the particle caused possibly by mechanical milling was examined by X-ray diffraction (XRD) (Cu-Kα radiation). Instrumental broadening was subtracted by using pure Si in the measurement of the micro-strain.Magnetic characterization of the particles was performed by VSM. Prior to the VSM measurement, the particles were aligned in magnetic field (1 T) and fixed with wax, and then magnetized by applying a pulsing field (4.5 T).

RESULTS AND DISCUSSION

Coercivity of the HDDR-treated Nd-Fe-B-type fine particles was radically reduced by mechanical milling. This coercivity variation was correlated to the variations of residual hydrogen content (hydrogen determinator), micro-strain (XRD; Williamson-Hall method),and surface oxidation (oxygen analyser) in the fine particle. It was revealed that major contributory factor for the radical coercivity reduction in the milled fine particle was surface oxidation. The reduced coercivityin the milled fine particles was recovered remarkably by chemical etching of the oxidized surface. Unlike the fine particles prepared from sintered Nd-Fe-B-type magnet, which lose coercivity almost completely (from around 13 kOe to 0.5 kOe), the fine particles (< 80 um) prepared from HDDR-treated Nd-Fe-B-type material exhibited high coercivity over 12 kOe. In this article, origin of radical coercivity reduction in the fine HDDR-treated Nd-Fe-B-type particles and its recovery are tobe discussed.

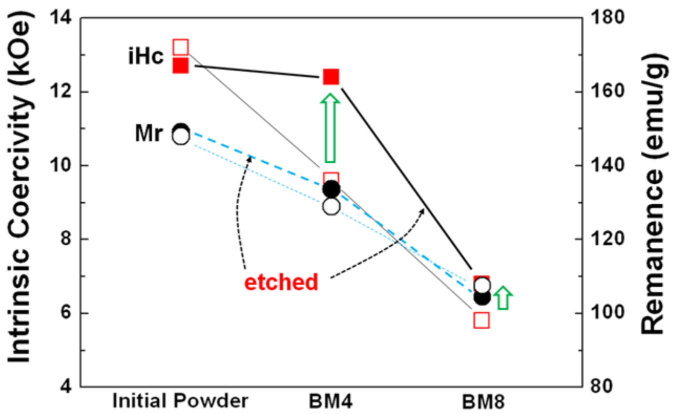

Fig.1. Variation of magnetic properties of the HDDR-treated $Nd_{12.5}Fe_{80.6}B_{6.4}Ga_{0.3}Nb_{0.2}$particles caused by mechanical milling. BM4 and BM8 denote ball milling for 4 hr or 8 hr, respectively.