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Disproportionation of $Nd_2Fe_{14}B$ by high-energy milling and its recombination

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고에너지 milling에 의한 $Nd_2Fe_{14}B$ 상의 분해와 그 재결합

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1. Introduction

Magnetically coercive powders of Nd-Fe-B are produced mainly by two commercial routes. Firstly, the Nd-Fe-B alloy is melt-spun and then the ribbon-type material is crushed to a suitable size. Secondly, HDDR process is also utilized for preparing the coercive Nd-Fe-B powder. For the magnetic powders prepared by the both methods, the coercivity depends critically on the grain size of the powder. In addition to these routes, an interesting attempt has been made to prepare the coercive Nd-Fe-B powder via high-energy ball milling. The constituent elements are ball milled with high energy to become an almost amorphous state and then annealed to crystallise $Nd_2Fe_{14}B$ phase (mechanical alloying). Meanwhile, there has also been an attempt to prepare the coercive Nd-Fe-B powder by high energy milling of pre-alloyed Nd-Fe-B lump instead of constituent elements (mechanical milling). In this study, disproportionation of the Nd2Fe14B phase due to high-energy milling and its recombination into original phase were investigated.

2. Experimental

An alloy ingot with composition $Nd_{15}Fe_{77}B_8$ was made by induction melting the constituent elements. The alloy was homogenized at 1050 °C for 3 days, and then crushed and pulverized into powder with a grain size less than 100 μm. High energy ball milling was undertaken using a shaker mill (Spex 8000 type). The alloy powder was charged into a hardened steel vial (cavity volume: 80 cm^3) together with hardened steel balls with two different diameters (8 mm and 5 mm). Mass ration of the powder to milling balls was 1:10. The charged vial was evacuated and then filled with high purity argon gas. The structure of the milled powders was examined by XRD analysis (Cu K_α radiation), and phase analysis of the material was also performed by TMA. The magnetic properties of powder were measured using VSM.

3. Results and discussion

The $Nd_2Fe_{14}B$ phase in alloy was converted into an almost amorphous phase after 3 hr milling. TMA result (Fig. 1) showed that the amorphous phase has Curie temperature of around 160 °C and a sharp magnetization reduction was observed at around 550 °C during heating. This magnetization is thought to be due to the recombination of the amorphous phase into $Nd_2Fe_{14}B$ phase during heating. The recombination into $Nd_2Fe_{14}B$ phase is clearly evidenced by the sharp magnetization increase at around 312 °C in the cooling TMA curve. Presence of α-Fe was also found in the TMA curve. Magnetic properties of the sample after TMA were measured and it was found that the high-energy milled amorphous material was converted into magnetically coercive material. The present study indicates that high energy milling process can be used as an effective means of producing high coercivity Nd-Fe-B-type powder.

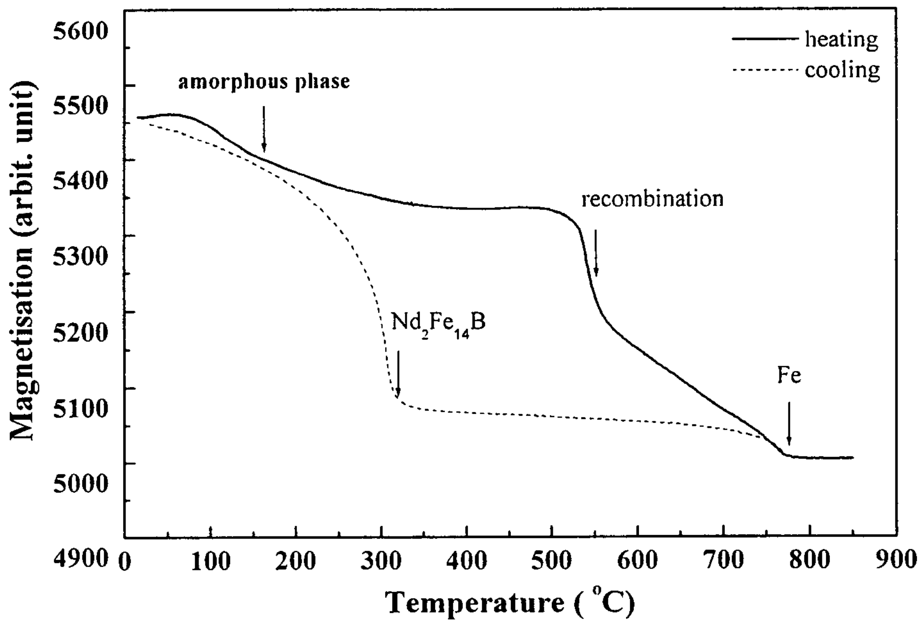

Fig. 1. TMA tracing of the Nd-Fe-B alloy high-energy ball milled for 48 hrs.