Coercivity of hot-pressed compacts of Nd-Fe-B- type HDDR-treated powder

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INTRODUCTION

The key feature of Nd-Fe-B-type HDDR (hydrogenation, disproportionation, desorption and recombination) -treated powder is the unique microstructure consisting of ultra fine $Nd_2F_{14}B$ grains, of which size is close to a single domain size (~300 nm for $Nd_2F_{14}B$). This fine grain structure can be exploited for achieving high performance, in particular high coercivity in permanent magnet. The HDDR-treated material is generally in powder form, and it would be desirable if the material can be consolidated into a high density bulk magnet keeping the fine grain structure. Our previous work revealed that the material lost the coercivity radically above 650 °C when the Nd-Fe-B-type HDDR-treated powder was compacted by hot pressing. It is important in technological point of view, therefore, to fully understand the cause of radical coercivity reduction on consolidation of the HDDR-treated powder. In the present study, the coercivity variation in the hot-pressed compact was studied emphasizing the function of residual hydrogen and consolidation of HDDR-treated powder was performed avoiding the detrimental effect of residual hydrogen in it.

EXPERIMENTAL DETAILS

HDDR-treated $Nd1_{2.5}Fe_{80.6}B_{6.4}Ga_{0.3}Nb_{0.2}$ powder (iHc =13.5 kOe, 100-150 μm) was used as a starting material for the present study. The powder was compacted by hot-pressing (1 Ton/cm^2) in vacuum (1.2 x 10^{-5} mbar at RT) at a temperature range from 500 to 800 °C with heating rate 70 °C/min. The hot-pressing was performed using WC die with different configuration. A closed-type or open die were used, and main difference in the two different configuration is that in the open die a gas from the powder sample can be evacuated more easily. Magnetic characterization of the hot-pressed compacts was undertaken by means of vibrating sample magnetometer (VSM) with a maximum field of 12 kOe. Prior to the VSM measurement, the samples were wax bonded and then magnetized in 4.5 T pulsing field. Microstructure of the compacts prepared at different dies and temperatures was observed by SEM. Differential thermal analysis (DTA) was carried out to investigate the phase change in the material during heating under different atmospheres with heating rate 7 °C/min. X-ray diffraction (XRD) (Cu-K_α radiation) was used for studying the crystallographic lattice parameter change in the $Nd_2Fe_{14}B$ grains in the compact caused by the desorption of residual hydrogen.

RESULTS AND DISCUSSION

The $Nd1_{2.5}Fe_{80.6}B_{6.4}Ga_{0.3}Nb_{0.2}$ HDDR-treated powder was compacted by hot-pressing under vacuum in the closed and open-type die at a temperature range from 500 to 800 °C with heating rate 70 °C/min. The results showed that the coercivity was reduced radically above 600 °C in the hot-pressed compacts prepared in closed-type die. No appreciable grain growth was noticed in the hot-pressed compacts observed by SEM. It can be assumed that grain coarsening is not the cause for radical coercivity loss of the compacts. Disproportionation of the $Nd_2Fe_{14}BH_x$ hydride in the HDDR-treated powder was also verified by DTA during heating in Ar and in vacuum. A noticeable exothermic event occurred at the temperature range of 620 °C - 655 °C in the course of heating in Ar atmosphere while no thermal event was found in the course of heating in vacuum,. This exothermic event is believed to be corresponding to the disproportionation of the $Nd_2Fe_{14}BH_x$ hydride in the HDDR-treated powder. In spite of identical temperature and heating rate, the coercivity reduction in the compacts hot-pressed in an open die was gradual and inconsiderable. These diverse results with identical temperature and heating rate were attributed only to the different evacuation system of hot-pressing die. This fact can be explained that residual hydrogen may be confined in a closed-type die in the course of heating for hot-pressing, hence resulting disproportionation in the HDDR-treated powder caused radical coercivity loss above 600 ℃ in the compacts. In an open die, residual hydrogen may be desorbed effectively avoiding the detrimental effect of hydrogen-related disproportionation, hence retaining the coercivity significantly in the hot-pressed compacts. In this study, coercivity in the hot-pressed compacts would be discussed in details emphasizing the function of residual hydrogen in HDDR-treated powder.

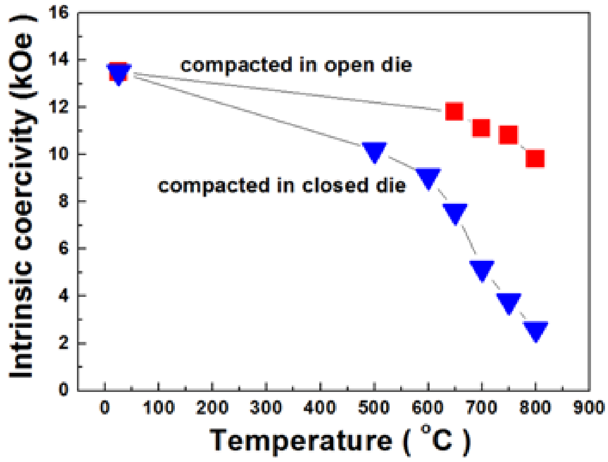

Fig.1. Coercivity variations for the hot-pressed compact of $Nd_{12.5}Fe_{80.6}B_{6.4}Ga_{0.3}Nb_{0.2}$ HDDR-treated powder prepared in closed and open-type die as a function of hot- pressing temperature.