TMA Study on Phase Evolution During Hydrogen-Assisted Disproportionation of Nd-Fe-B Alloy

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1. Introduction

The HDDR (hydrogenation, disproportionation, desorption, and recombination) process is a well-established technique of producing a highly coercive Nd-Fe-B powder with fine grain structure directly from a magnetically non-coercive ingot alloy with coarse grain structure. The disproportionation reaction is the most critical step for controlling the magnetic properties (in particular, the grain texture) of the Nd-Fe-B HDDR powder. It has been widely known that the fully disproportionated Nd-Fe-B alloy consists of mixture of NdH_x, Fe_2B and α-Fe. Recently, however, we have found that the phase constitution of fully disproportionated Nd-Fe-B alloy was rather complicated. In the present study, the phase constitution and evolution during the hydrogen-assisted disproportionation of the Nd-Fe-B alloy was investigated mainly by magnetic balance-type thermomagnetic analyser (TMA). As the disproportionated alloy consists mostly of magnetic phases, the TMA may be useful tool for precise phase analysis.

2. Experimental Work

$Nd_{12.5}Fe_{81.1-(x+y)}B_{6.4}Ga_xNb_y$ (x = 0 or 0.3, y = 0 or 0.2) alloy powder was first fully hydrogenated by heating it in hydrogen (P = 0.1 MPa) or hydrogen and argon mixed gas (PH_2 = 0.03 MPa) up to 800°C (heating rate = 7°C/min), and holding at there for given period of time (1 hr for hydrogen atmosphere, 3 hr for mixed gas). Immediately after the completion of full disproportionation, the sample was quenched to room temperature. The phase constitution in the quenched sample was analysed mainly by magnetic balance-type thermo-magnetic analyser (TMA). In order to avoid any unwanted phase change in the course of TMA heating, the sample was heated swiftly up to 800°C (swift TMA). In this swift heating mode the sample could be heated up to 800°C within 150 seconds, therefore any unwanted phase change could be suppressed practically during the TMA heating. The phase analysis by swift TMA was aided also by slow regular TMA and XRD.

3. Results and discussion

Fig. 1 shows the swift TMA result of the $Nd_{12.5}Fe_{80.6}B_{6.4}Ga_{0.3}Nb_{0.2}$ alloy fully disproportionated in hydrogen gas. As can be seen, the fully disproportionated alloy shows multiple magnetic transitions above 400°C. The magnetic transitions at 765°C and 725°C are obviously corresponding to the Tc of α-Fe and Fe_2B. In addition to these two magnetic phases, two additional magnetic phases appears to exist in the fully disproportionated $Nd_{12.5}Fe_{80.6}B_{6.4}Ga_{0.3}Nb_{0.2}$ alloy. The magnetic transition at 520°C is corresponding to the Tc of Fe_3B. Unknown magnetic phase (probably Fe-boride with other stoichiometry) with Tc of 600°C also exists. In addition to these magnetic phases, non-magnetic NdHx is also contained (by XRD). This results indicate, therefore, that the fully disproportionated $Nd_{12.5}Fe_{80.6}B_{6.4}Ga_{0.3}Nb_{0.2}$ alloy consists of NdHx, Fe_2B, Fe_3B and α-Fe, together with an extra magnetic phase with Tc of 600°C. The fully disproportionated ternary $Nd_{12.5}Fe_{81.1}B_{6.4}$ alloy also showed almost identical phase constitution, and this suggested that the phase constitution of fully disproportionated Nd-Fe-B alloy was independent upon the alloy composition. $Nd_{12.5}Fe_{81.1-(x+y)}B_{6.4}Ga_xNb_y$ (x = 0 or 0.3, y = 0 or 0.2) alloys fully disproportionated in different atmosphere of hydrogen or hydrogen and argon mixed gas showed almost identical phase constitution. In this article, the phase evolution during the hydrogen-assisted disproportionation of the Nd-Fe-B alloy is to be discussed.

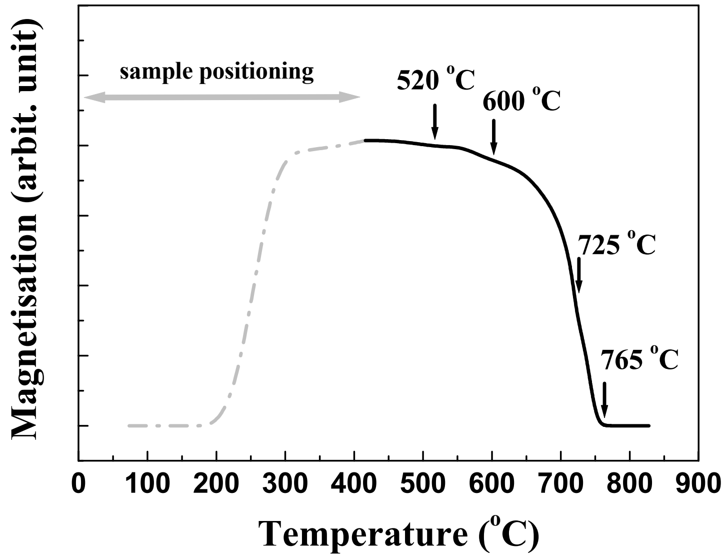

Fig. 1. Swift TMA results of the $Nd_{12.5}Fe_{80.6}B_{6.4}Ga_{0.3}Nb_{0.2}$ alloy fully disproportionated in hydrogen gas.