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Preparation of $Nd_2Fe_{14}B$ single domain particles using combination of hydrogen treatment and mechanical milling

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1. Introduction

Nd-Fe-B-type magnetic materials are widely used in the industry due to their high permanent magnetic performance. The high performance is attributed to the basic magnetic phase of $Nd_2Fe_{14}B$. This magnetic compound has outstanding intrinsic magnetic properties; high saturation magnetization, high magnetocrystalline anisotropy, and reasonably high ordering temperature. The single domain particles of this phase has been needed in a scientific community of the permanent magnetic materials. There has been intensive research effort to prepare the fine particle of the $Nd_2Fe_{14}B$ phase. However, most of them were focussed on the preparation of the particles consisting of multiple fine grains rather than the fine single grain particles. Little work has been reported on the preparation of the $Nd_2Fe_{14}B$ single domain particles. Meanwhile, the HDDR process (hydrogenation, disproportionation, desorption and recombination) using hydrogen treatment has been well established as an effective means of producing a fine grain structured Nd-Fe-B powder. The HDDR-process can easily convert a coarse grain structured Nd-Fe-B bulk material into particles with fine grain structure. In an optimally HDDR-treated Nd-Fe-B powder the size of recombined $Nd_2Fe_{14}B$ grains is comparable to the critical single domain size (~0.3 μm). Therefore, perfect single domain particles may be prepared if the fine recombined grains in the HDDR-treated Nd-Fe-B material are separated successfully. In this article, the authors intended to prepare the single domain particles of $Nd_2Fe_{14}B$ phase from the Nd-Fe-B ingot alloy using the HDDR-process and mechanical means.

2. Experimentals

$Nd_{15}Fe_{77}B_8$ starting alloy was prepared by an induction melting of the high purity component elements. The cast alloy ingot was homogenized at 1070 ℃ for 72 hrs (under Ar gas) and crushed into a coarse granules (0.5~1.0 mm). The granules were, then, hydrogenated at 350 ℃ for 60 min under 1.0 kgf/cm^2 hydrogen. Subsequently, the hydrogenated powder was disproportionated at 820 ℃ for 45 min under 1.0 kgf/cm^2 hydrogen. Desorption and recombination was carried out at 820 ℃ for 30 min under vacuum. The HDDR-treated material was roller-milled to produce single domain particles. Some of the HDDR-treated material was selected and subsequently subjected to a hydrogen decrepitation (HD) treatement in order to see the effect of HD on the disintegration of the recombined grains in HDDR-treated material into the single domain particles. Morphology of the obtained particles was observed by HRSEM. For a magnetic characterization the particles were aligned by applying 10 kOe DC field, and the aligned particles were fixed with a wax. The magnetic properties of the particles were measured using a vibrating sample magnetometer with a maximum field of 12 kOe after a pre-magnetizing with 6 T pulsing field. The likelihood of single domain particle was evaluated by the degree of alignment (DoA), which is defined by the ratio of $Mr_{(//)10}/Mr_{(\perp)10}$, where, $Mr_{(//)10}$ and $Mr_{(\perp)10}$ are the magnetization

at 10 kOe in the first quadrant demagnetization curve along the directions parallel and perpendicular to the aligning direction, respectively.

3. Results and discussion

The HDDR-treated material was briefly milled for 2 min using a mortar and pestal. Microstructural observation showed that the obtained powder had particle size of around 20 - 30 ㎛, and the grain size of each particle was around 0.3 ㎛, which is comparable to a critical single domain size of the $Nd_2Fe_{14}B$ phase. It is almost certain, therefore, that the particles in this powder consist of randomly oriented multiple grains. Multi grain structure of the particle in this powder is also supported by magnetic measurement. Being a multi grain particle, these particles can not be aligned under applied field, and as expected the demagnetization curves showing no alignment with DoA = 1.04 (Fig. 1(a)). The HDDR-treated material was subsequently milled for 12 hrs using a roller mill. The particle size of obtained powder was around 0.3 ㎛, and it is almost certain, therefore, that the particles of this powder are single domain particles. The likelihood of single domain particle of these particles is also verified by magnetic measurement. Being a single domain particle, the HDDR-treated and roller milled particles will be aligned almost perfectly. As shown in Fig. 1(b), these particles show good magnetic alignment, indicating that the recombined grains with single domain size in the HDDR-treated material have been separated successfully into the single domain particles by the subsequent mechanical milling. The findings in the present study lead us to conclude that single domain particles of the $Nd_2Fe_{14}B$ phase can be easily prepared from the Nd-Fe-B ingot alloy using the combination of HDDR-process and mechanical milling

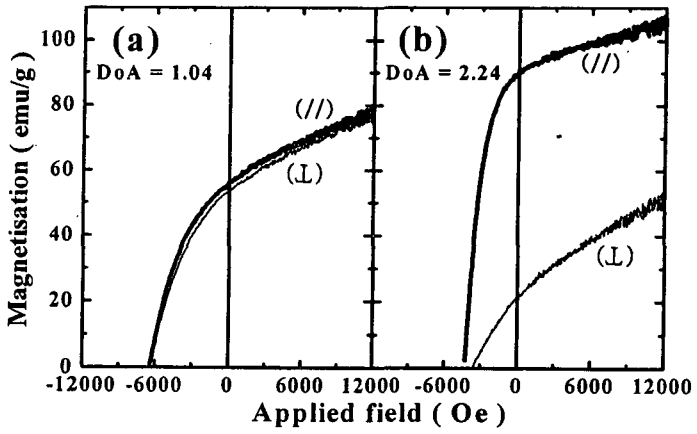

Fig.1. Demagnetization curves measured along the directions parallel and perpendicular to the aligning directions for the differently processed $Nd_{15}Fe_{77}B_8$ materials. (a) HDDR + brief milling (2 min), (b) HDDR + roller milling (12 hrs).

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