Anisotropy inducement mechanism in hydrogen disproportionation desorption recombination (HDDR) processed Nd-Fe-B powders

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Introduction

The hydrogenation-disproportionation-desorption-recombination (HDDR) process is an attractive and unique method for producing anisotropic nanocrystalline Nd-Fe-B powders. In order to develop highly textured Nd-Fe-B powders, HDDR process parameters need to be carefully chosen and controlled [1]. Although lots of investigations have been carried out to understand the mechanism of the anisotropy development in HDDR powders [1-3], some questions still remain. In this work, the microstructures of Nd-Fe-B powders that were HD processed at different hydrogen pressure (P_{H2}^{HD}) were investigated to fully clarify the mechanism of the anisotropic microstructure evolution.

Experimental

Dynamic hydrogenation disproportionation (HD) desorption recombination (DR) process was carried out on $Nd_{12.8}Fe_{80.1}B_{6.6}Ga_{0.3}Nb_{0.2}$ alloy powders at different HD hydrogen pressure, P_{H2}^{HD} = 30 kPa and 100 kPa. The microstructures of the samples in early stages of the HD process, a fully HD process, and an early DR process were investigated using SEM/FIB (Carl Zeiss 1540EsB), TEM (Titan G2 80-200), and a locally built laser-assisted three dimensional atom probe (3DAP) to characterize the memory sites responsible for the texture development.

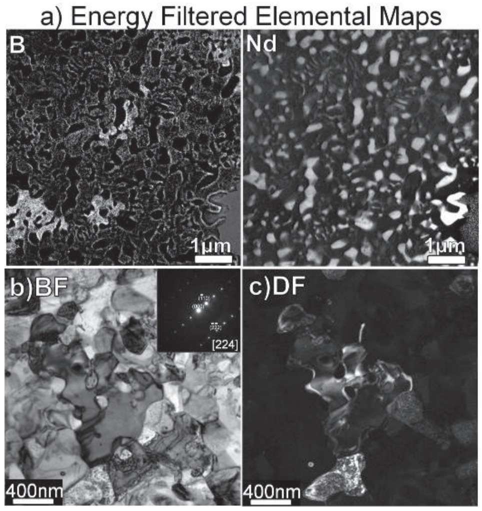

Fig. 1: (a) Energy filtered B and Nd maps of fully HD processed sample at P_{H2}=30kPa. (b) BF and (c) DF TEM images from the same sample indicating well textured Fe_2B grains. SADP obtained from a Fe_2B grain is shown in inset of (b).

Results

A high remanent magnetization of 1.43 T was obtained for the fully HDDR processed powder with P_{H2}^{HD} = 30 kPa, indicating a strong [001] crystallographic texture. However, P_{H2}^{HD} = 100 kPa led to weakly textured Nd-Fe-B powders with a remanent magnetization of 0.89 T.

TEM observations from early HD processed powder with P_{H2}^{HD} = 30 kPa showed that the Fe_2B phase has a direct crystallographic orientation relationship with the initial $Nd_2Fe_{14}B$ grains, i.e. $[420]_{Fe2B}||[211]_{Nd2Fe14B}$ and $(00\bar{2})_{Fe2B} || (\bar{1}11)_{Nd2Fe14B}$. Energy filtered (EF)-TEM and 3DAP results obtained from a fully HD processed sample showed boron not only in the Fe_2B phase but also at the NdH_2/α-Fe interfaces in both weakly and highly textured samples. High resolution STEM-HAADF image and nano-beam diffraction analysis from NdH_2/α-Fe interfaces showed that boron enrichment at these interfaces does not make a separate phase, such as iron boride. However, there is boron enrichment more in Fe grain of the NdH_2/α-Fe interface in nano-scale. Fig. 1(a) shows EF-TEM maps of B and Nd for the fully HD processed sample with P_{H2}^{HD} = 30 kPa. Bright field (BF)-TEM images obtained from the fully HD processed samples with different P_{H2}^{HD} showed that the Fe_2B regions in both weakly and highly textured samples comprise of several small Fe_2B grains. Fig. 1 (b) and (c) shows BF and dark field (DF)-TEM images obtained from fully HD processed P_{H2}^{HD}=30 kPa sample. The DF-TEM image shows that the Fe_2B sub-grains in boride region of fully HD processed P_{H2}^{HD} = 30 kPa sample is strongly textured. Fig. 2 (a) shows BF-TEM image taken from the sample fully HD processed at P_{H2}^{HD} = 100 kPa. Superimposed EF-TEM maps of Nd (red), Fe (Blue), and B (green), obtained from the same region as Fig. 2(a), is shown in Fig. 2 (b). By comparing Fig. 2 (a) and (b), NdH_2, α-Fe, and Fe_2B phases can be distinguished. Selected area diffraction patterns obtained from different Fe_2B grains in boride area are shown in Fig. 2(c), indicating that the Fe_2B grains in the boride area are not well aligned in the sample fully HD processed at P_{H2}^{HD} = 100 kPa. Orientation relationship study of P_{H2}^{HD} = 30 kPa sample at early DR processed stage showed that recombined $Nd_2Fe_{14}B$ grains have direct orientation relationship with the remaining Fe_2B phase from HD process. In addition, 3D SEM tomography obtained from 3D serial sectioning of BSE images from P_{H2}^{HD} = 30 kPa sample at very early stage of DR process showed that the recombined $Nd_2Fe_{14}B$ grains nucleate at the interface of Fe_2B/NdH_2 grains and grow through the interface of NdH_2/α-Fe grains.

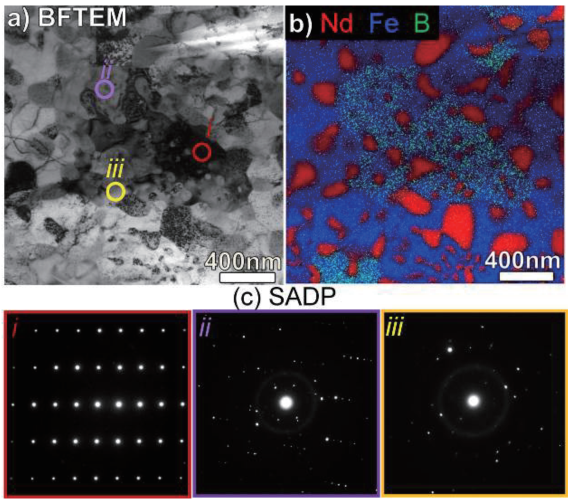

Fig. 2: (a) Bright field TEM image and (b) superimposed energy filtered Nd, Fe, and B maps of fully HD processed sample at P_{H2}=100kPa. (c) Selected area diffraction patterns obtained from different Fe_2B sub-grains in boride region.

These microstructure investigations indicate that the highly aligned Fe_2B grains act as memory sites for the development of the texture in the sample HD processed at P_{H2}^{HD} = 30 kPa, as shown schematically in Fig. 3, consistent with the previously proposed texture memory effect (TME) model. Importantly, it can now be shown that the recombined $Nd_2Fe_{14}B$ phase nucleates at the interface of Fe_2B with NdH_2 phase and grow through the interface of NdH_2/α-Fe interfaces and boron segregated at the NdH_2/α-Fe interface acts as a boron source for the growth of the recombined $Nd_2Fe_{14}B$ grains during DR process.

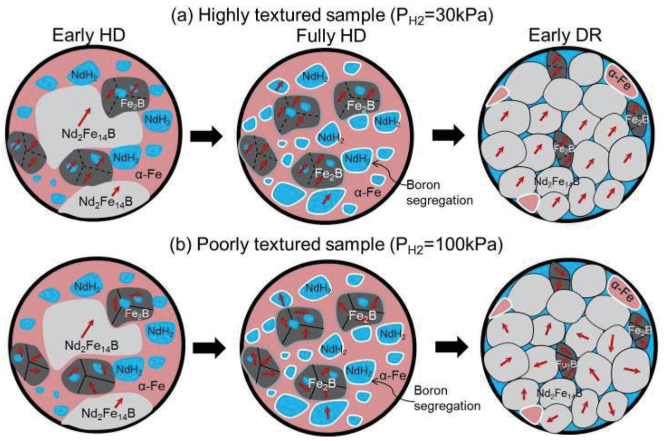

Fig. 3: Schematic illustration of microstructure evolution of HDDR processed sample with (a) P_{H2}^{HD} = 30 kPa (b) P_{H2}^{HD} = 100 kPa. This figure shows that in the highly textured powder, highly aligned Fe_2B grains act as memory sites and remembering the crystallographic orientation of initial $Nd_2Fe_{14}B$ grains and transferring the same orientation to the recombined $Nd_2Fe_{14}B$ grains.

Reference

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