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Anomalous rapid change of site preference of Ga at the beginning of recombination in the HDDR for $Nd_{12.5}Fe_{bal}Ga_{0.3}Nb_{0.2}B_{6.2}$ magnets

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Introduction

Recently, Mishima et al. developed the HDDR process with a slow reaction rate, i.e. dynamic HDDR (d-HDDR) [1]. They reported that the addition of nearly 0.3% Ga and 0.2% Nb in $Nd_{12.5}Fe_{bal}B_{6.2}$ increased $(BH)_{max}$ about 40% by using d-HDDR. In order to know the role of Ga in the HDDR process precisely, we carried out XAFS measurements of the Ga K edge for the samples at the various recombination stages: 0, 1, 1.5, 2, and 6 min after the start of dehydrogenation.

Experiment

An ingot of $Nd_{12.5}Fe_{bal}Ga_{0.3}Nb_{0.2}B_{6.2}$ alloys was prepared by arc melting under Ar gas atmosphere, subsequently homogenized in Ar gas at 1140ºC for 40h. Hydrogenation is done under 0.1MPa hydrogen at R.T. for the homogenized sample. Disproportionation is carried out under the low hydrogen pressure of 0.03MPa at 820ºC for the slow decomposition reaction. The recombination is done at 820ºC for 0, 1, 1.5, 2, and 6 min under the relatively higher hydrogen pressure of 3 kPa in order to proceed at a low reaction rate. Results of the X-ray diffraction using Cu Ka radiation show the existence of three phases of α-Fe, NdH_2, and Fe_2B for the disproportionated sample as shown Fig. 1, and the disproportionated state was preserved until about 2 min after the start of the dehydrogenation.

XAFS measurements of the Ga K edge were carried out at the temperature 20 K at BL9A by fluorescence method. Incident energy was selected by a double Si(111).

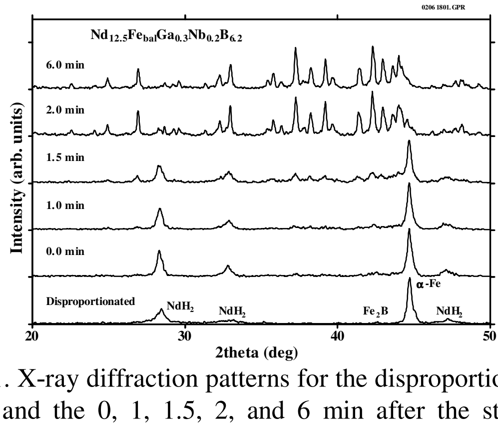

Fig. 1. X-ray diffraction patterns for the disproportionated state and the 0, 1, 1.5, 2, and 6 min after the start of dehydrogenation of $Nd_{12.5}Fe_{bal}Ga_{0.3}Nb_{0.2}B_{6.2}$.

Results and Analysis

After the usual background subtraction and normalization $\chi(k)$ was deduced using program AUTOBK. Fig. 2 shows the observed $\chi(k)$ curves (solid line) for the Ga K edge of the $Nd_{12.5}Fe_{bal}Ga_{0.3}Nb_{0.2}B_{6.2}$ for the disproportionated state and the various recombination stages. Fitting calculations to these observed XAFS curves were done using FEFFIT program. The best fitting was obtained by putting Ga at α-Fe for the disproportionated and Fe(4c) site in $Nd_2Fe_{14}B$ structure for the recombined state, respectively.

The most significant future of these XAFS results is that as soon as the recombination starts (0 min), XAFS spectrum changes abruptly from those for the disproportionated state to that for the fully recombined state (6 min). This seems very strange because it takes at least 2min. until recombined $Nd_2Fe_{14}B$ structure appears in the X-ray diffraction results (see Fig.1). This result suggests that the portion of α-Fe in which Ga includes preferentially recombined to $Nd_2Fe_{14}B$ at the early stage of recombination.

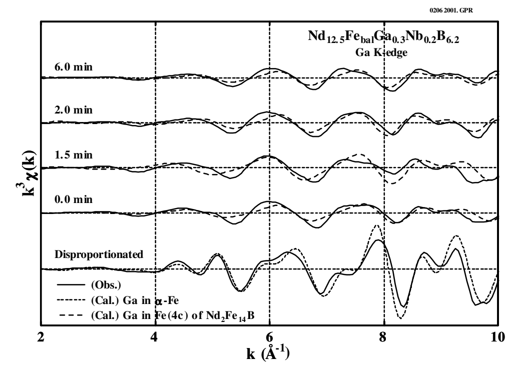

Fig. 2. XAFS spectra of the Ga K edge of $Nd_{12.5}Fe_{bal}Ga_{0.3}Nb_{0.2}B_{6.2}$ for the disproportionated state and the various recombination states (0, 1.5, 2, and 6 min after the start of the dehydrogenation). The solid lines are the observed results, and the broken lines the calculated ones for Ga in α-Fe (at the disproportionated) and in the Fe(4c) site of $Nd_2Fe_{14}B$ (at the 0, 1.5, 2, and 6 min).

References

[1] C. Mishima, M. Hamada, H. Mitarai, and Y. Honkura, proceedings of the 16th International Workshop on R. E. Magnets and Their Applications, Sendai Japan, 873 (2000).

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