

## Pulsed laser deposition of nanocomposite $\alpha$ -Fe/Nd<sub>2</sub>Fe<sub>14</sub>B magnets

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Spring magnets of the type  $\alpha$ -Fe/Nd<sub>2</sub>Fe<sub>14</sub>B consist of a soft ( $\alpha$ -Fe) magnetic phase embedded into a hard (Nd<sub>2</sub>Fe<sub>14</sub>B) magnetic phase, which results in a high remanent polarization provided that the particles are small and strongly coupled [1, 2]. This type of structure can also be obtained through a multilayered two-phase magnet, consisting of thin alternating hard and soft magnetic layers [3–9].

In this paper we propose to study the structural and magnetic properties of  $\alpha$ -Fe/Nd<sub>2</sub>Fe<sub>14</sub>B magnetic multilayers obtained by pulsed laser deposition (PLD). The  $\alpha$ -Fe and Nd<sub>2</sub>Fe<sub>14</sub>B sputtering targets were obtained from Goodfellow Corporation. The PLD process was conducted with a KrF excimer laser having a

wavelength of 248 nm and a pulse width of 8 ns. The laser delivered 450 mJ/pulse at a repetition rate of 10 Hz. For each laser pulse, a fluence at target of about 3 J/cm<sup>2</sup> was obtained. The multilayered films were deposited on (100) Si wafers, then measured and subsequently annealed before new measurements were performed.

We employed X-ray diffraction (XRD) and conversion electron Mössbauer spectroscopy (CEMS) to characterize the main features of the  $\alpha$ -Fe/Nd<sub>2</sub>Fe<sub>14</sub>B multilayer systems. XRD measurements were performed using a Rigaku D-2013 diffractometer with Cu K <sub>$\alpha$ 1</sub> radiation at  $\lambda = 1.5404$  Å. CEMS measurements were made with a constant acceleration spectrometer. The

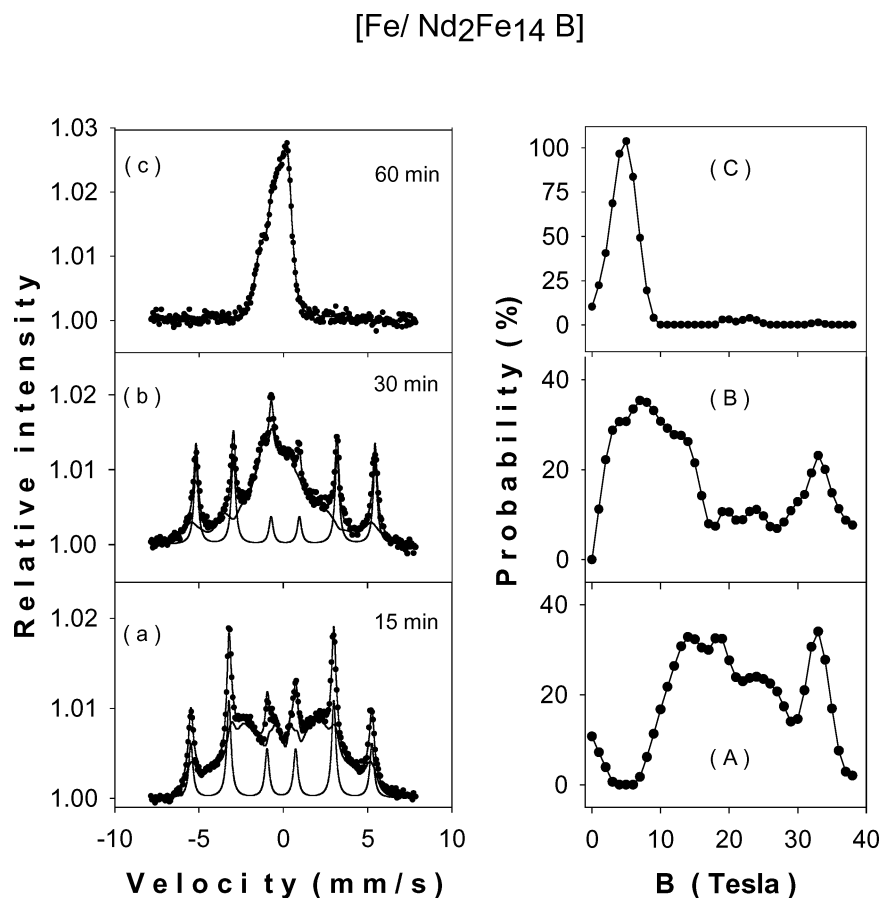


Figure 1 Conversion electron Mössbauer spectra of the  $\alpha$ -Fe/Nd<sub>2</sub>Fe<sub>14</sub>B multilayers obtained for an ablation time of (a) 15 min, (b) 30 min, and (c) 60 min for each target. In (A)–(C) the hyperfine magnetic field distributions extracted from the CEMS spectra are given.

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50 mCi gamma ray source was  $^{57}\text{Co}$  in Rh matrix, maintained at room temperature. A He-6%  $\text{CH}_4$  gas-flowed electron counter was used. All spectra were analyzed with the NORMOS-DIST program, which uses the histogram method to obtain the hyperfine magnetic field distributions.

Fig. 1a–c shows the CEMS spectra of the  $\alpha$ -Fe/ $\text{Nd}_2\text{Fe}_{14}\text{B}$  multilayers deposited for 15, 30, and 60 min for each target. In (A)–(C) the hyperfine magnetic field distributions extracted from these spectra are given. The spectra in Fig. 1a and b were analyzed by considering a six-line pattern corresponding to the  $\alpha$ -Fe soft phase and a hyperfine magnetic field distribution, representing the  $\text{Nd}_2\text{Fe}_{14}\text{B}$  hard phase. The average values of the distributions were 21.17 and 15.76 T respectively, and the distributions extended over 77.07 and 73.47% of the spectra in Fig. 1a and b. It may be observed that a decrease in the average hyperfine magnetic field and population of the disordered phase is obtained by employing longer deposition times for each target on the carousel. Moreover, when we analyze the spectrum in Fig. 1c, we observe that it consists entirely of a distribution of hyperfine magnetic fields, with an average value of 5.25 T. This finding indicates the occurrence of a superparamagnetic phase, which can be assigned to Fe atoms in Nd rich environments. These results indicate that the optimum deposition conditions correspond to 15 min ablation time for each of the two targets, which corresponds to a final film thickness of about 50 nm.

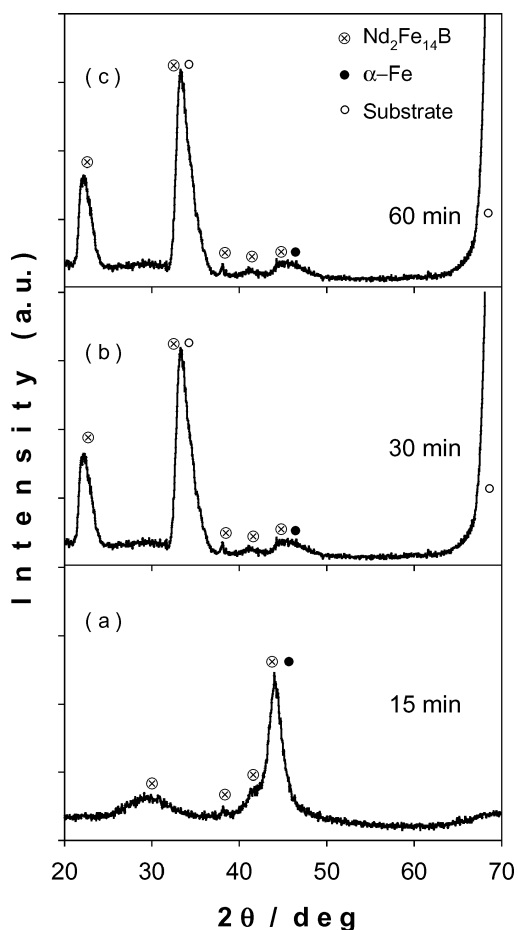


Figure 2 X-ray diffraction patterns of the  $\alpha$ -Fe/ $\text{Nd}_2\text{Fe}_{14}\text{B}$  multilayers obtained for an ablation time of: (a) 15 min, (b) 30 min, and (c) 60 min for each target.

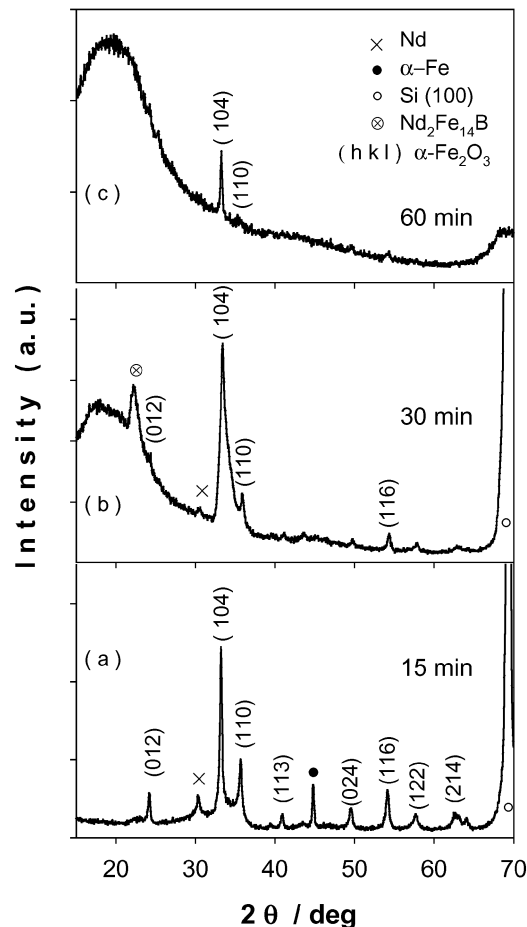


Figure 3 X-ray diffraction patterns of the  $\alpha$ -Fe/ $\text{Nd}_2\text{Fe}_{14}\text{B}$  multilayers obtained for an ablation time of: (a) 15 min, (b) 30 min and (c) 60 min for each target, followed by annealing at  $550^\circ\text{C}$  for 1 h.

Fig. 2a–c presents the XRD patterns recorded for the  $\alpha$ -Fe/ $\text{Nd}_2\text{Fe}_{14}\text{B}$  multilayers after 15, 30, and 60 min of deposition from each target. Besides the lines coming from the silicon substrate, the spectra indicate the formation of the  $\text{Nd}_2\text{Fe}_{14}\text{B}$  phase. Again, the best structure corresponds to the film ablated for 15 min per target. Fig. 3a–c displays the XRD spectra of the  $\alpha$ -Fe/ $\text{Nd}_2\text{Fe}_{14}\text{B}$  multilayers, obtained at 15, 30, and 60 min per target and subsequently exposed to thermal annealing at  $550^\circ\text{C}$  for 1 h. The spectra are consistent with the formation of  $\alpha$ -Fe and  $\text{Nd}_2\text{Fe}_{14}\text{B}$  soft and hard phases, along with lines from Nd (which supports the occurrence of the superparamagnetic fraction) and hematite phases. An amorphous component, representing the intermixing of phases, can be observed for the sample deposited for 1 h per target. In agreement with the CEMS results, the best structure is formed at 15 min ablation time for each target.

In conclusion,  $\alpha$ -Fe/ $\text{Nd}_2\text{Fe}_{14}\text{B}$  multilayer structures were obtained by pulsed laser deposition and characterized by CEMS and XRD. The optimum deposition conditions were identified and the structural and magnetic properties were examined for each sample.

#### Acknowledgment

This paper was prepared with the support of the U.S. Department of Energy, under Award No. DE-FC26-02NT41595. However, any opinions, findings,

conclusions, or recommendations expressed herein, are those of the authors and do not necessarily reflect the views of DOE.

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*Received 2 March  
and accepted 6 May 2004*