X-ray diffraction analysis and magnetic behavior of amorphous $Fe_{15}Co_{17}Ni_{58}B_{10}$ nanowires obtained by electrochemical deposition

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$Fe_{15}Co_{17}Ni_{58}B_{10}$ nanowire arrays have been deposited in alumina templates by electrochemical deposition. For comparison, the same alloy was used to deposit films on CuBe substrate. The change in boron concentration from 3% to 10% in the electrolyte promoted the amorphous phase in the films on the CuBe substrate and a partial amorphous phase in the nanowires. Thermal treatment of the deposited films and nanowire arrays at 600 °C promoted recrystallization. Magnetic hysteresis loops for 100 and 200 nm nanowire arrays are "wasp waisted." There is no evidence of a sharp reversal for the out-of-plane or in-plane hysteresis loops, just a smooth curve indicating a coherent rotation during the magnetization reversal. Annealing the 200 nm nanowire array at 600 °C for 1 h gives rise to a mixed hysteresis loop, which can be attributed to competing domain configurations. An alternative explanation for this behavior is that there might be a phase separation between Fe, Ni, and Co in the nanowire environment due to the short range ordering of the alloy. *© 2007 American Institute of Physics*. [DOI: 10.1063/1.2711700]

I. INTRODUCTION

The study of highly ordered arrays of amorphous magnetic nanowires in hexagonal alumina templates with various diameters is an interesting topic of investigation. The magnetic properties of nanowires give rise to outstanding behavior, which differs from bulk and thin films. Previous studies have been devoted to crystalline transition metal based alloys, which show a variety of shapes for the hysteresis loops.[1] This change in the shape of the loops may be obtained by varying the composition and by heat treating the alloy.[2] It has been shown that codeposition of phosphorus (P) or boron (B) with the transition metal influences the structure of the electrodeposited alloy, essentially leading them to produce an amorphous alloy.[3] A lot of work has been carried out on binary samples such as[4] Fe–Ni, Co–Ni, and Co–Fe with phosphorus as the third element, and recently work on Co–Fe–B thin films and nanowire arrays was published to understand the role of the bulk characteristics and geometric factors on the array properties.[5] The purpose of our work is to study the structure and magnetic behavior of the amorphous Fe–Co–Ni–B alloy. The investigation of the magnetic behavior of this amorphous alloy was based on the limitations imposed by its size (nanometer) on the nanowire arrays. The properties may widely vary depending on the size of the nanocrystals as well as the dimensions and magnetic properties of the amorphous matrix.[6] Analysis of the properties at the nanoscale provides information about the intrinsic magnetic characteristics. These characteristics mainly include the magnetization rotation processes determined by the strength of magnetic anisotropy at long range scale in the case of domain wall displacements and the short scale ordering/disordering.[7] This interplay between two constituent magnetic interactions in nanocrystals amidst an amorphous matrix determines the macroscopic behavior and gives rise to the variable shaped hysteresis loops.

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II. EXPERIMENTAL DETAILS

FeCoNiB alloy of thickness of 30 μm was deposited on CuBe substrate using the electrochemical deposition method. The boron concentration was varied from 3% to 10% in the alloy. The same composition of $Fe_{15}Co_{17}Ni_{58}B_{10}$ was used to deposit nanowires (diameter=20, 100, and 200 nm, thickness=60 μm) in the commercially available hexagonal alumina templates. The galvanostatic conditions used for the sample preparation are $t_{on}/t_{off}=12/380$ for 12.6 mA/cm^2 current density. Vibrating sample magnetometer was used to determine the hysteresis loops. All the samples considered for the magnetic study were prepared using the same parameters described above for the alloy deposition in alumina templates with diameters of 20, 100, and 200 nm. The 200 nm diameter nanowire array was annealed at 600 °C for 1 h in a nitrogen atmosphere.

III. RESULTS

A. Morphology and structure of electrodeposited alloys on thin film substrate

The x-ray diffraction (XRD) pattern of "electrodeposited" FeCoNiB thin film with 3% boron concentration at current density of 12.6 mA/cm^2 on the CuBe substrate is presented in Fig. 1(a). As observed in Fig. 1(a) the sample shows crystalline behavior. The diffractogram for the FeCoNiB alloy with 10% boron concentration as shown Fig. 1(b) shows a broad peak between 40° and 50°, indicating that transition metal alloys containing more than 8% boron are amorphous.[1] In order to investigate the thermal sta-

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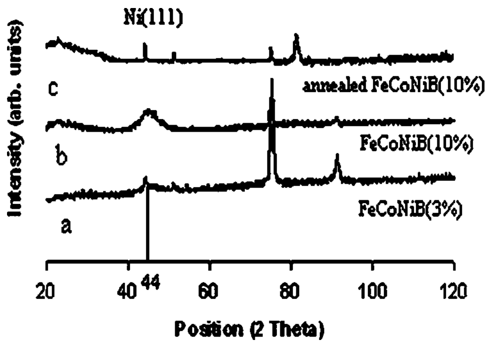

FIG. 1. X-ray diffractogram of FeCoNiB film on copper-beryllium substrate with (a) 3% boron, (b) 10% boron, and (c) 10% boron annealed at 600 °C.

bility, the electrodeposited thin film alloy on the CuBe substrate was heat treated at 600 °C in nitrogen atmosphere for a period of 1 h. The heat treated sample shows a sharp peak at 44°. The peak which disappeared at 51.8° in Fig. 1(b) reappears in Fig. 1(c). This peak corresponds to Ni (200) and shows the transition from amorphous to crystalline.

B. Morphology and structure of electrodeposited nanowires in alumina templates

The nanowires deposited in the alumina templates show a short range ordering of the alloy. The diffractograms for these nanowire arrays as shown in Fig. 2 are not exactly comparable with the thin film deposition of the alloy on the CuBe substrate. The diffractograms are different in spite of the deposition conditions and the electrolyte being the same. The sharp peaks at 38°, 77°, and 83° are the alumina peaks in Figs. 2(a) and 2(b). The diffractogram shows a very short range ordering of the alloy. The peak at 44 ° in Fig. 1(b) is broader compared to the peak in Fig. 2(a). The peak in Fig. 2(a) shows that it has nanocrystals embedded in the amorphous matrix. Using Scherrer's formula the diameter of the nanocrystals is approximately 4 nm. If this sample of $Fe_{15}Co_{17}Ni_{58}B_{10}$ is heated at 600 °C in nitrogen atmosphere for a period of 1 h, it shows high intensity peaks at 44°, 65°, and 76.5°. The diffractogram of the annealed $Fe_{15}Co_{17}Ni_{58}B_{10}$ sample shown in Fig. 2(b) is similar to the annealed $Fe_{15}Co_{17}Ni_{58}B_{10}$ thin film sample in Fig. 1(c), indicating a similar recrystallized structure.

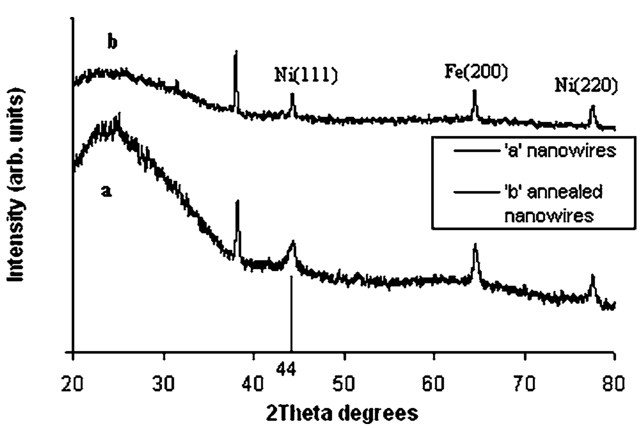

FIG. 2. X-ray diffractogram for $Fe_{15}Co_{17}Ni_{58}B_{10}$ nanowires in alumina template: (a) as cast and (b) annealed at 600 °C.

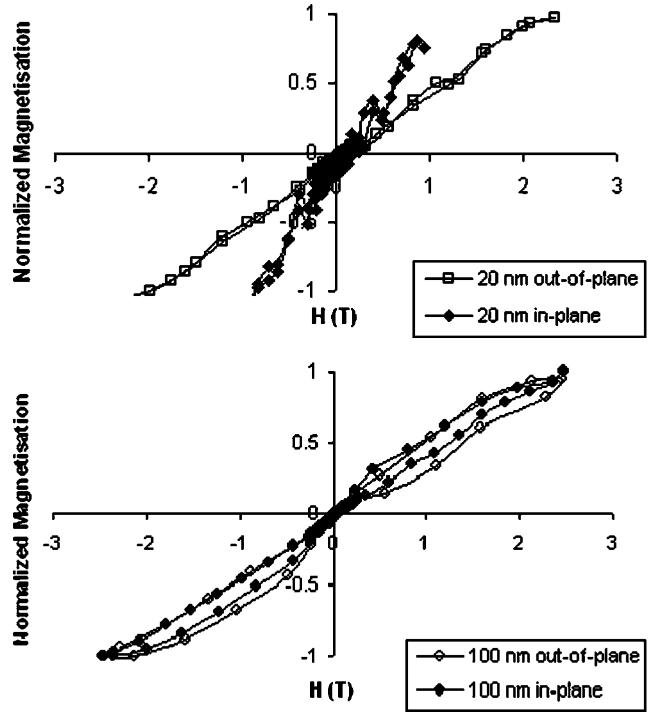

FIG. 3. M/M_{max}-H hysteresis plots for 20 and 100 nm wire arrays.

C. Magnetic hysteresis loops (MHLs)

The hysteresis loops (HLs) for the 20, 100, and 200 nm diameter nanowire arrays are constricted or "wasp waisted." This is usually the case with iron, cobalt, and nickel alloys when they are heat treated but in these cases as shown in Fig. 3; it shows this behavior without the heat treatment, which can be attributed to the amorphicity (atomic ordering) of the alloy.[2] The constricted loops could be a result of contrasting coercivities of the elements in the alloy or the interaction of grains with different sizes. The in-plane measurement for the 20 nm nanowire array indicates a positive ΔH_s $(H_s^{oop}-H_s^{ip})$, indicating an in-plane easy axes arising due to very low induction in the sample.[5] The 100 and 200 nm nanowire arrays show almost similar loops for the out-of-plane and in-plane magnetizations, indicating an intermediate switching of the easy axis from out-of-plane to in-plane direction or vice versa. This can be attributed to magnetostatic interactions amongst the nanowires. Magnetic flux closure between the nanowires might also occur, in which case a large portion of the nanowires displays a multidomain pattern such that domains in neighboring wires would be preferentially oriented as antiparallel to each other. The resulting complex magnetic ordering pattern within the array may also lead to small differences between out-of-plane and in-plane HLs.[8] MHLs for the in-plane and out-of-plane measurements show no evidence of a sharp reversal on the hysteresis loops, just a smooth curve in all the cases indicating a coherent rotation during the magnetization reversal. Annealing the 200 nm nanowire array at 600 °C for 1 h shows a complex switching behavior on the HL as shown in Fig. 4, which can be attributed to the formation of domain walls due to heating. An explanation for such loops is competing domain configurations. An alternative explanation for this behavior is that

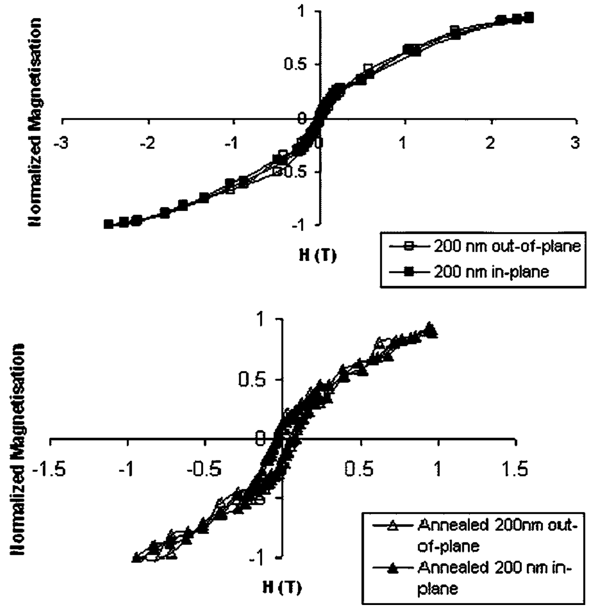

FIG. 4. M/M_{max}-H hysteresis plots for as-cast and annealed 200 nm wire arrays.

there might be a phase separation between Fe, Ni, and Co in the nanowire environment. As seen on the diffractogram in Fig. 2(a), the Ni (111) and Fe (200) phases could exhibit rather isolated behavior resulting in mixed loops.

IV. CONCLUSIONS

The preparation and the change in the structural and magnetic behaviors of FeCoNiB amorphous nanowires as a result of variation of boron concentration from 3% to 10% and annealing were studied. The x-ray diffraction analysis shows that the Fe–Co–Ni–B film consists of nanocrystals embedded in an amorphous matrix. Due to low induction, hysteresis loop for 20 nm nanowire shows transverse easy axes. Magnetic hysteresis loops for the 20, 100, and 200 nm nanowire arrays show "wasp waisted" loops, which can be expected for Fe, Ni, and Co alloy having short range order. There is an intermediate transition of easy axes from axial to transverse or vice versa for 100 and 200 nm nanowire arrays, which can be attributed to magnetostatic interactions. Annealing the 200 nm nanowire sample shows a mixed hysteresis loop behavior attributed to domain wall formations and competing domain configurations. The future work will be focused on the x-ray analysis and *B-H* loops study of the amorphous nanowire arrays in templates with different diameters, by varying the deposition parameters.

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