View metadata, citation and similar papers at core.ac.uk
brought to you by CORE
provided by Electronic Sumy State University Institutional Repository

PROCEEDINGS OF THE INTERNATIONAL CONFERENCE
NANOMATERIALS: APPLICATIONS AND PROPERTIES
Vol. **2** No 1, 01NTF09(3pp) (2013)

FeCuNbSiB Thin Films Deposited by Pulsed Laser Deposition: Structural and Magnetic Properties

M. Dobromir[1], I.L. Velicu[1], M. Neagu[1], H. Chiriac[2]

[1] *Faculty of Physics, Alexandru Ioan Cuza University, Blvd. Carol I, No. 11, RO-700506, Iasi, Romania*
[2] *National Institute of Research & Development for Technical Physics, Blvd. Mangeron, No. 47, RO-700050, Iasi, Romania*

(Received 19 May 2013; published online 30 August 2013)

Using the pulsed laser ablation technique, $Fe_{73.5}Cu_1Nb_3Si_{15.5}B_7$ amorphous thin films, with smooth and uniform surfaces, have been deposited on glass and silicon substrates. Based on the information provided by the thermomagnetic analysis, the nanocrystalline state was achieved after the thermal treatment performed at 460 °C. In nanocrystalline state, the samples present an 80 % lower coercive magnetic field and a 3.5 times higher saturation magnetization with respect to the as-deposited state.

Keywords: Amorphous and Nanocrystalline Magnetic Materials, Magnetic Properties, Thin films, Magneto-Optical Kerr Effect.

PACS number(s): 42.65.Hw, 62.23.Pq, 75.60.Ej

1. INTRODUCTION

FeCuNbSiB amorphous and nanocrystalline thin films are attractive in a wide range of engineering applications. The nanocrystalline state can be induced by adequate thermal treatment applied to the amorphous precursors. In the last years, due to the large number of industrial applications with stringent requirements and to the increasing development of technology, there has been a strong diversification, improvement and expansion of the techniques used for obtaining amorphous soft magnetic thin films [1-7]. The microstructure optimization of FeCuNbSiB soft magnetic thin films is of great importance with the view of enhancing their magnetic properties for micromagnetic device applications. Similarly with the case of amorphous ribbons and wires with the same compositional formula, in the nanocrystalline state an important improvement of the soft magnetic properties can be obtained [1-4, 6-9].

The aim of this paper is to investigate the structural, topological, and magnetic properties of $Fe_{73.5}Cu_1Nb_3Si_{15.5}B_7$ thin films obtained by pulsed laser ablation. The samples have been investigated in as-deposited state and after thermal treatments.

2. EXPERIMENTAL DETAILS

FeCuNbSiB thin films have been prepared by pulsed laser deposition technique, using as target ribbons with the nominal composition $Fe_{73.5}Cu_1Nb_3Si_{15.5}B_7$ (Vacuumschmelze GmbH). The films were deposited on glass and silicon substrates. The depositions were carried out under high vacuum (10^{-6} Torr), by means of a XeCl excimer laser (308 nm wavelength) with the pulse duration, repetition rate and pulse energy of 30 ns, 25 Hz and 55 mJ, respectively. By varying the deposition time, films with thickness between 150 nm and 300 nm have been prepared.

Techniques such as X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD), atomic force microscopy (AFM) and interferometry were used to characterize the composition, the structural properties, the morphology and the thickness of the obtained films. The XPS analyses were carried out with a PHI 5000 VersaProbe (Φ ULVAC-PHI, INC.) spectrometer (Al Kα radiation, 1486.7 eV). The XRD measurements were performed using a Bruker AXS D8 Advance diffractometer with CuKα radiation (1.5418 Å), in Bragg-Brentano geometry. The AFM analyses were carried out using a NT-MDT Solver Pro-M AFM, in tapping mode. A magnetization dependence on temperature was analyzed in the temperatures range 23°C – 850 °C. The volume magnetic behavior of the samples was investigated by a differential inductive method at 50 Hz, using an integrating fluxmeter. The surface magnetic behavior was investigated by means of magneto-optical Kerr effect, in longitudinal configuration.

The samples have been investigated in as-deposited state and after furnace annealing at temperatures between 400°C and 480°C, for 60 minutes, in high vacuum (10^{-6} Torr).

3. RESULTS AND DISCUSSION

X-ray diffraction studies confirmed the amorphicity of all as-deposited samples.

The AFM topographical scans have revealed uniform surfaces, with a root-mean-square roughness value of about 1.5 nm.

The X-ray photoelectron spectroscopy investigations demonstrated that the stoichiometry of the target has been achieved in the deposited films. Fig. 1 presents a wide-scan XPS spectrum recorded for a FeCuNbSiB thin film in as-deposited state, in the binding energies range from 0 to 1200 eV. The sputter cleaning of the surface was done under ultra high vacuum conditions on a 3 × 3 mm^2 area by using an Ar^+ ion beam (500 V, 500 nA, 1 minute). All the binding energies at various peaks were calibrated by the binding energy of C 1s (284.5 eV). From the presented spectrum, it can be clearly seen the presence of Fe, Cu, Nb, Si, B elements near the surface of the analyzed film. Surface contamination elements like carbon and oxygen can also be observed. The presence of oxygen in the XPS spectrum reflects the surface oxidation process due to the film exposure into the atmosphere.

 © 2013 Sumy State University

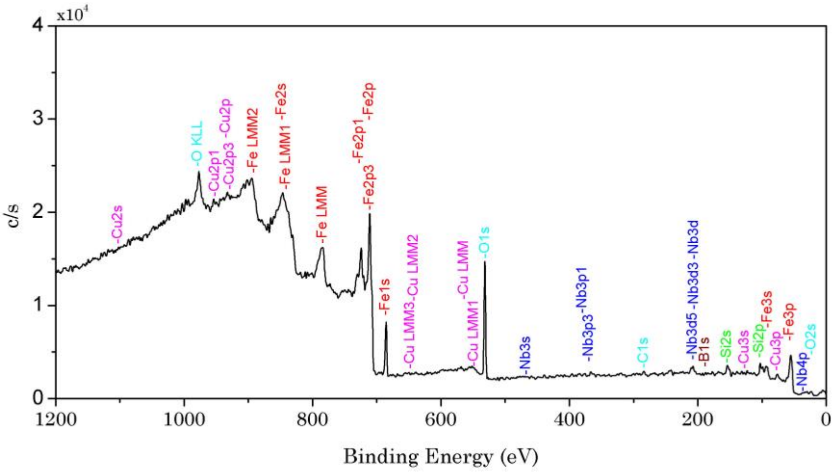

Fig. 1 – Wide-scan XPS spectrum for an as-deposited amorphous FeCuNbSiB thin film.

In order to get information on the crystallization kinetics, magnetization dependences on temperature were registered for samples deposited on silicon substrate. Fig. 2 presents a normalized thermomagnetic curve, registered for a $Fe_{73.5}Cu_1Nb_3Si_{15.5}B_7$ thin film. The critical temperatures (i.e. Curie temperature of the amorphous phase, T_C^{am}, first crystallization temperature, T_x, and Curie temperature of the crystalline phase, T_C^{cr}) were determined from the thermomagnetic curve using the intersecting tangents method [10] and also, for a better accuracy, using the differential method, d^2M/dT^2 [11]. The determination of Curie temperature of the amorphous phase by the differential method of Tauxe is also presented in Fig. 2. As it can be seen, the magnetization rapidly decreases with the temperature increment up to T_C^{am} = 363 °C, reaching beyond this (when the film is in paramagnetic state) a value close to zero. The beginning of the crystallization process (precipitation of the crystalline phase with higher Curie temperature) at about 457 °C leads to an abrupt increase in the magnetization value. The Curie temperature of the crystalline phase is about 620 °C. According to this information, the nanocrystalline state of the $Fe_{73.5}Cu_1Nb_3Si_{15.5}B_7$ thin films can be achieved through thermal treatments performed at temperatures around 460 °C.

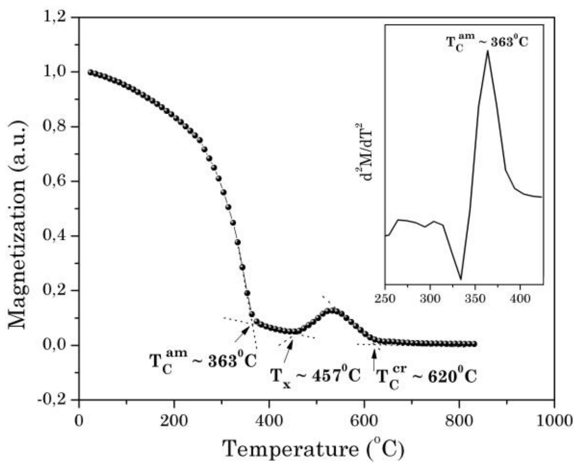

Fig. 2 – Normalized thermomagnetic curve for an as-deposited $Fe_{73.5}Cu_1Nb_3Si_{15.5}B_7$ thin film (silicon substrate).

The crystallization processes indicated by the thermomagnetic analysis are also confirmed by the XRD results. According to these, for samples thermally treated in the temperature range of the first crystallization temperature, the nanocrystalline state has been achieved.

The softest magnetic properties have been obtained after the thermal treatment performed at 460 °C. After this treatment, the film structure consists of ultra-fine bcc α-Fe(Si) grains, with average size of about 15 nm, uniformly dispersed in a residual Fe-Nb-B amorphous matrix. The average α-Fe(Si) grain size of the nanocrystalline phase was estimated using the Scherrer formula [4]. Fig. 3 presents bulk hysteresis loops for a $Fe_{73.5}Cu_1Nb_3Si_{15.5}B_7$ thin film (200 nm thickness) in as-deposited state and after the thermal treatment performed for 1 hour, at 460 °C. For samples thermally treated in the mentioned conditions, the bulk coercive magnetic field value decreases with about 80 %, while the saturation magnetization becomes 3.5 times higher with respect to the as-deposited state.

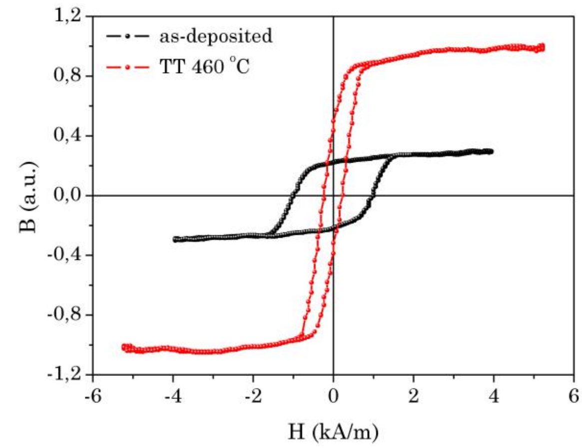

Fig. 3 – Bulk hysteresis loops for a $Fe_{73.5}Cu_1Nb_3Si_{15.5}B_7$ film in as-deposited state and after thermal treatment at 460 °C.

The bulk coercive magnetic field for the as-deposited thin films decreases with about 43 %, when the film thickness increases from 150 nm to 300 nm.

Magneto-optical Kerr analyses have revealed an almost identical surface magnetic behavior, the only difference being that the surface coercive magnetic field value is about 5 % higher than the bulk coercive magnetic field value. This difference may be due to the existing surface imperfections.

4. CONCLUSION

The pulsed laser deposition technique allows obtaining $Fe_{73.5}Cu_1Nb_3Si_{15.5}B_7$ amorphous thin films with smooth and uniform surfaces.

In order to establish the optimum annealing temperatures which lead to achieving the nanocrystalline state, the crystallization kinetics was investigated for the obtained films.

An important improvement of soft magnetic properties has been obtained after the thermal treatment performed at 460 °C.

Future studies will aim to establish new deposition parameters in order to improve the soft magnetic properties for FINEMET-type thin films obtained using pulsed laser ablation technique.

AKNOWLEDGEMENTS

This work was supported by the European Social Fund in Romania, under the responsibility of the Managing Authority for the Sectoral Operational Programme for Human Resources Development 2007-2013 under Grant POSDRU/CPP 107/DMI 1.5/S/78342.

REFERENCES

1. D. Szewieczek, T. Raszka, *Journal of Achievements in Materials and Manufacturing Engineering* **18**, 179 (2006).
2. F. Celegato, M. Coisson, P. Tiberto, F. Vinai, M. Baricco, *Phys. Status Solidi C: Current Topics in Solid State Physics* **8**, 10.1002/pssc.201000197 (2011).
3. J. Moulin, F. Mazaleyrat, A. Mendez, E. Dufour-Gergama, *J. Magn. Magn. Mater.* **322**, 1275 (2010).
4. X.D. Li, W.Z. Yuan, Z.J. Zhao, X.Z. Wang, J.Z. Ruan, X.L. Yang, *J. Magn. Magn. Mater.* **279**, 429 (2004).
5. S. Acquaviva, A.P. Caricato, E. D'Anna, M. Fernandez, A. Luches, Z. Frait, E. Majkova, M. Ozvold, S. Luby, P. Mengucci, *Thin Solid Films* **433**, 252 (2003).
6. P. Sharma, A. Gupta, *J. Magn. Magn. Mater.* **288**, 347 (2005).
7. I.-L. Velicu, M. Neagu, H. Chiriac, V. Tiron, M. Dobromir, *IEEE T. Magn.* **48**, 1336 (2012).
8. G. Herzer, *Nanocrystalline Soft Magnetic Alloys* (in Handbook of Magnetic Materials, edited by K.H.J. Buschow (Ed.), Elsevier Science BV, Amsterdam-Lausanne: 1997).
9. M.H. Phan, H.X. Peng, M.R. Wisnom, S.C. Yu, N. Chau, *Compos. Part A – Appl. S.* **37**, 191 (2006).
10. C.S. Grommé, T.L. Wright, D.L. Peck, *J. Geophys. Res.* **74**, 5277 (1969).
11. L. Tauxe, *Paleomagnetic Principles and Practice,* 1st ed. (Springer: New York: 1998).