Deposition of Nano-phase FeCo Thin Films using Plasma Focus Device

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Abstract

The dense plasma focus (DPF) device is used to deposit nano-phase FeCo thin films by ablating the high purity FeCo anode tip. The films were deposited using 5, 10, 20 and 30 DPF shots. The XRD of the film samples with 20 and 30 deposition shots show the deposition of as-grown crystalline thin films with the degree of crystallinity increasing with the number of shots. The SEM micrographs show that deposited films are smooth and crack free with nano-sized particle agglomerates. The average grain size of particle agglomerate is found vary from 30 nm for 5 shots deposition to about 76 nm for 30 shots deposition. The size of the particles which form these agglomerates, however, is much smaller than the grain size. The EDX of the samples show that the deposited films are stoichiometric.

Introduction

The dense plasma focus (DPF) is a simple device that compresses the plasma to very high densities ($\approx 10^{25} - 10^{26} \text{ m}^{-3}$) and high temperature (1 - 2 keV) and is widely known as a source of neutrons, highly energetic ions, relativistic electrons and an abundant amount of soft and hard X-rays. From application point of view it has recently been used for X-ray lithography and also for thin film deposition and surface modification [1-2]. FeCo falls under the category of magnetostrictive materials that offer the possibility of a new generation of magnetic sensors, combining the advantages of non-contact sensing with high sensitivity [3]. Nano sized Fe and FeCo materials have many other important applications such as magnetic microactuators, micro inductors with a closed ferromagnetic core and magnetic data storage [4]. In this paper, we report the deposition of nano-phase FeCo thin films using the plasma focus device.

Experimental Details

FeCo thin films have been deposited using a simple single shot, single-capacitor 3.3 kJ Mather-type DPF device. For deposition of FeCo thin films, the conventional central hollow copper anode was fitted with a solid Fe\textsubscript{50}Co\textsubscript{50} top, which is 2.0 cm long. This solid
Fe$_{50}$Co$_{50}$ top was screwed on top of the hollow copper anode. The DPF device was operated at a charging voltage of 14 kV with neon as the filling gas at a pressure of 3.0 mbar. The focusing efficiency was monitored using a voltage probe. Bertalot and Herold [5] found that the ions emitted in a plasma focus have a wide energy range in a fountain like geometry with anisotropy in their angular distribution. Most of the ions are emitted in a small solid angle along the anode axis and their flux decreases with the increasing angle. The films were deposited at an angular position, called off-center, with respect to the anode axis at the distance of 12 cm from the center of the anode tip. It was noticed that the film deposited directly along the anode axis are not very uniform because of high radial gradient in ion flux. The experimental setup is shown in Fig.1. The films were deposited using 5, 10, 20 and 30 DPF shots.

The Si (100) substrates were used for the deposition of FeCo thin films. Substrates were successively cleaned using acetone, alcohol and water respectively for durations of 5 minutes each in an ultrasonic bath. We fired 10 conditioning shots to ensure that the DPF was working at the optimum focusing. The shutter, shown in Fig.1, was used to block out any ionic deposition during the conditioning. After the 10 shots are fired and the focusing shown on the voltage probe signal is good, standard deposition will follow with the shutter removed.

The deposited films have been analyzed for their structure, surface morphology and stoichiometry using X-ray diffractometer (XRD), field emission scanning electron microscope (FSEM) and energy dispersive X-ray spectroscopy (EDX).

Results and Discussion

The XRD analysis shows that the films deposited using 5 and 10 focus shots at off-centre positions are amorphous in nature with no diffraction peaks. However, the as-
deposited films using 20 and 30 focus shots are polycrystalline with many diffraction peaks appearing in the XRD spectrum. The crystalline planes corresponding to (220) CoFe$_2$O$_4$ at 29.27$^\circ$, (311) Fe$_3$O$_4$ at 35.87$^\circ$, (109) Fe$_2$O$_3$ at 39.5$^\circ$, (400) CoFe$_2$O$_4$ at 43.16$^\circ$, (110) FeCo at 44.83$^\circ$, (316) Fe$_2$O$_3$ at 47.48$^\circ$ and (024) Fe$_2$O$_3$ at 48.59$^\circ$ were observed in the XRD spectrum of these films. The crystalline nature of the films deposited using higher number of focus shots can be attributed to greater thickness of the films as well as to more energetic treatment of the deposited films by the energetic neon ions of the following focus shots because of bigger number of focus shots used. It may be noticed that, in addition to FeCo peak, many oxide peaks have been observed. This may be due to oxygen impurity in the gas used and also may be due to the fact that the Si substrate itself may have oxide layer which could be initially ablated from the substrate surface by the energetic neon ions of
plasma focus device which then can react with Fe, Co or FeCo ionic and molecular species coming from the ablated focus anode top to form various oxide observed in diffraction spectra.

The SEM images, showing the surface morphology, of the deposited films are shown in Fig.2. The SEM micrographs show that deposited films are smooth and crack free with nano-sized particle agglomerates. It can be seen that the size of the grains increases with the increase in the number of shots. The increase in grain size with the increase in number of focus deposition shots points to the fact that the deposition with higher number of focus shots involve much higher energetic process. On higher magnification it can be seen that the grains are actually the agglomerates of many smaller size particles. The average grain size of these particle-agglomerate is found vary from 30 nm for 5 shots deposition to about 76 nm for 30 shots deposition. The size of the particles which form these agglomerates, however, is much smaller than the grain size and is difficult to resolve. The film texture is smoother for the films with smaller grain size. The EDX analysis of the deposited samples shows that they are quite stoichiometric. The EDX also confirms the presence of oxygen impurity in the films.

Conclusions

These results show the feasibility of the DPF to synthesize FeCo nanophase thin films and also demonstrate that the number of focus deposition shots plays a significant role on the morphology and crystallinity of the synthesized nanomaterials.

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References