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Sputtered FeCl/Cu multilayer thin films: effect of different thicknesses of Cu layer

ALI KARPUZ*, HAKAN KOCKAR[a], MESUT ESIYOK[a]
Physics Department, Kamil Ozdag Science Faculty, Karamanoglu Mehmetbey University, Karaman, Turkey
[a]*Physics Department, Science and Literature Faculty, Balikesir University, Balikesir, Turkey*

A series of FeCl/Cu multilayer thin films was deposited with different thicknesses of Cu layer. The multilayers were sputtered with a two dc magnetrons system. The effect of different thicknesses of Cu layer on the properties of the films was investigated. The Cu content in the films gradually increased from 0 at.% to 47 at.% as the Cu layer thickness was increased from 0 nm to 24 nm. According to crystal structure analysis, the intensities of the peaks which belong to the face centered cubic planes increased with increasing Cu content of the films. Modest and mirror-like surfaces were observed in all images obtained by a scanning electron microscope. Magnetic measurements indicated that magnetization decreased with increasing Cu contents in the multilayer thin films. The magnetization of the film without Cu was 1473 emu/cm^3 while the value of 348 emu/cm^3 was detected for the film with 24 nm Cu layer thickness.

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Keywords: FeCl/Cu, Multilayer thin films, Structural analysis, Magnetic properties

1. Introduction

Magnetic multilayer thin films have attracted growing attention due to wide range of their applications, such as sensor and storage technology [1, 2] and therefore, there is a great effort to produce such materials [3, 4]. Sputtering is a useful technique for fabrication of magnetic thin films [5-10], especially multilayer thin films [11, 12]. It has also some advantages such as quick film growth and composition control [13]. The properties of a sputtered film can be affected by many parameters, e.g. deposition rate, substrate temperature, total film thickness and the thickness of magnetic or non-magnetic layers [13].

Many studies on multilayer thin film systems have been achieved by using sputtered Cu and Fe. For instance, in the study [14], the microstructural and mechanical properties of Cu/V multilayers were investigated whereas the properties of ZnO/Cu/ZnO were reported at different substrate temperatures [15]. In addition, Ni-Co/Cu multilayers were produced to study their magnetic properties [12]. As to multilayers with layer of Fe or Fe based materials, while the magnetic and structural properties for Fe/Fe_3O_4 [16], Fe/Pt/Au [17] and $Ni_{80}Fe_{20}/SiO_2$ [18] were studied, our study [19] focused on FeCl/Cu multilayer thin films sputtered at low and high deposition rates of magnetic layer. Except for our earlier study [19], to our knowledge, it can be understood from the detailed literature study that there is no any other study focused on multilayers using FeCl layer as a magnetic constituent. Under study, FeCl/Cu multilayer thin films were deposited by using a sputtering system with two dc magnetrons, and the effect of different thicknesses of Cu layer on the properties of multilayers was studied. It was revealed that thickness of Cu layer has a substantial effect on structural and magnetic properties since the systematic change in thickness results in a gradual change in content of the multilayer thin films.

2. Experimental

Two dc magnetrons were used in the sputtering system. Commercial FeCl and Cu targets were served as a source for magnetic and non-magnetic layers, respectively. The composition of the FeCl target was also determined by using the inductively coupled plasma-atomic emission spectroscopy (ICP-AES, Perkin Elmer Optima 3100 XL). It was detected that FeCl target is composed of 70 at. % Fe and 30 at. % Cl. The magnetic FeCl source was located in the one of the magnetrons while the non-magnetic one (Cu source) was fixed to the other dc magnetron. The diameter and thickness of the source materials sputtered by two dc magnetrons was 60 mm and 0.8 mm, respectively. The films were grown on acetate substrates (5 cm × 2.5 cm) at the room temperature. The source targets and substrates were first cleaned with isopropyl alcohol and dried with dry air before all deposition. The pressure in the production chamber was reached around 5×10^{-6} mbar, and it was 2×10^{-3} mbar during deposition and deposition rate was 0.045 nm/s for all layers. The thickness of the Cu layers was changed from 0 to 24 nm while that of the FeCl layers was kept at 6 nm. In addition, the total thickness of each film was adjusted as 120 nm and the thicknesses of the layers were determined with quartz crystal microbalance thickness monitor. After deposition, the films were properly cut for characterization and then preserved in a desiccator until measurements.

The compositional analysis of the films was done using an energy dispersive X-ray spectroscopy (EDX, GENESIS APEX 4—EDAX, AMETEK). The crystal structure analysis was made with an X-ray diffraction technique (XRD, PANalytical) by using a Cu-K_α radiation. The surface morphology was observed by using a scanning electron microscope (SEM, FEI^{TM}, NOVANANOSEM430). The magnetic properties were examined with a vibrating sample magnetometer (VSM, ADE TECHNOLOGIES DMS-EV9). The hysteresis loops of the films were measured on a (6 mm diameter) circular shape of the films with different Cu layer thicknesses under 20 kOe at room temperature.

3. Results and discussion

The FeCl/Cu multilayer thin films were produced by considering different thicknesses of Cu layer. The thickness was gradually changed between 0-24 nm. The Fe, Cl and Cu contents of the films were summarized in Table 1. As seen in the table, Fe and Cl contents gradually decreased as the Cu layer thickness and hence Cu content of the films increased. The Cu contents of the films were found as 0 at. %, 13 at. %, 22 at. %, 26 at. %, 31 at. %, 40 at. %, 47 at. % for the films with 0 nm, 2 nm, 4 nm, 6 nm, 9 nm, 14 nm and 24 nm thickness of Cu layer, respectively by using EDX. The rest of the films were FeCl. It was disclosed that the Cu content increased as the layer thickness of Cu was increased. The study [20] investigated sputtered Cu/In multilayers sulphurised at different annealing temperatures and reported that Cu content can be also changed by different temperatures. Unlike the present study and [20], the atomic Cu content remained at the same value when the deposition rate of magnetic layer was increased for sputtered FeCl/Cu multilayer thin films [19]. This may be due to the same thickness of FeCl and Cu layers in all films [19].

Table 1. Contents of the FeCl/Cu multilayer thin films that have different thicknesses of Cu layer.

Cu layer Thickness (nm)	Film content (at.%)		
	Cl	Fe	Cu
0	89	11	0
2	77	10	13
4	71	7	22
6	67	7	26
9	63	6	31
14	56	4	40
24	50	3	47

In order to detect the effect of different Cu contents caused by different thicknesses of Cu layer on the crystal structure of the FeCl/Cu multilayer thin films, the XRD measurements were done and the patterns were presented in Fig. 1. The measurements of the films were carried out on their amorphous substrates. The results revealed that the peaks observed on the XRD patterns belong to the body-centered cubic structure (bcc) and face-centered cubic structure (fcc). The XRD pattern of FeCl film (0 at. % Cu) has only one main peak, bcc (110), forms at $2\theta \approx 45^o$ as shown in Fig. 1. It can be stated that the crystal phase of the FeCl film is only bcc structure. However, the films with Cu contents of 13-47 at. % have a mixture of fcc and bcc phase. In the XRD patterns of these films, two main peaks belong to fcc (111) and bcc (110) planes were observed at $2\theta \approx 43^o$ and 45^o, respectively (see Fig. 1). The intensity of the fcc (111) peak increased and that of the bcc (110) peak decreased gradually when the Cu layer thickness and hence Cu content of the films increased. In addition, the characteristic (200) peak of the fcc phase was detected at $2\theta \approx 51^o$ in the XRD pattern of the film with 47 at % Cu content. Besides, no effect from Cl atoms on the crystal structures of the multilayers was observed. It can be concluded that the crystal structure of the multilayer thin film series changed from the bcc structure to the mixture of bcc and predominantly fcc structure. In other words, while the multilayers have bcc Fe and fcc Cu, except for bcc Fe film, the bcc structure of the multilayer thin film with 47 at % Cu has been distorted by fcc structure of Cu atoms. Also, preferential textures of the multilayer thin films were determined considering the relative peak intensities as done in [21]. It was found that the preferential orientation turned from the (110) to the (111) plane when the Cu content increased from 0 at. % to 47 at. %. Since the films were deposited on the amorphous substrates, it can be assumed that the substrates used are negligible to determine the preferential orientation, unlike the study [1] which investigated the electrodeposited multilayer that has same texture with its substrate.

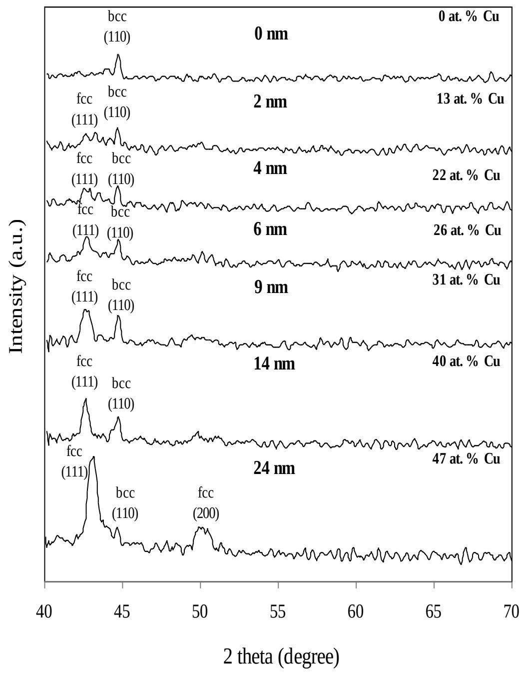

Fig. 1. XRD patterns of the FeCl/Cu multilayer thin films that have different thicknesses of Cu layer.

The SEM images of the films were presented in Fig. 2. Since all of the films have nearly the same surface morphology, the SEM images for the films with 0 at. %, 31 at. %, and 47 at. % Cu contents were illustrated in the figure. The surfaces were generally simple and bright. The films were in mirror like appearance. Besides, the SEM image with higher magnification was shown in Fig. 2d for the film with 47 at. % Cu. The surface showed in this image has some stripes and deformations probably due to the surface of the substrate. The appearance of the surfaces shows similarities with that of the surfaces of sputtered FeCl/Cu multilayer thin films [19]. The films investigated in [19] have the same thickness with the present study. The morphologies observed in this study also show considerable differences from granular morphologies indicated in study [22]. This may be due to the mostly different deposition type of materials and also substrate than the study.

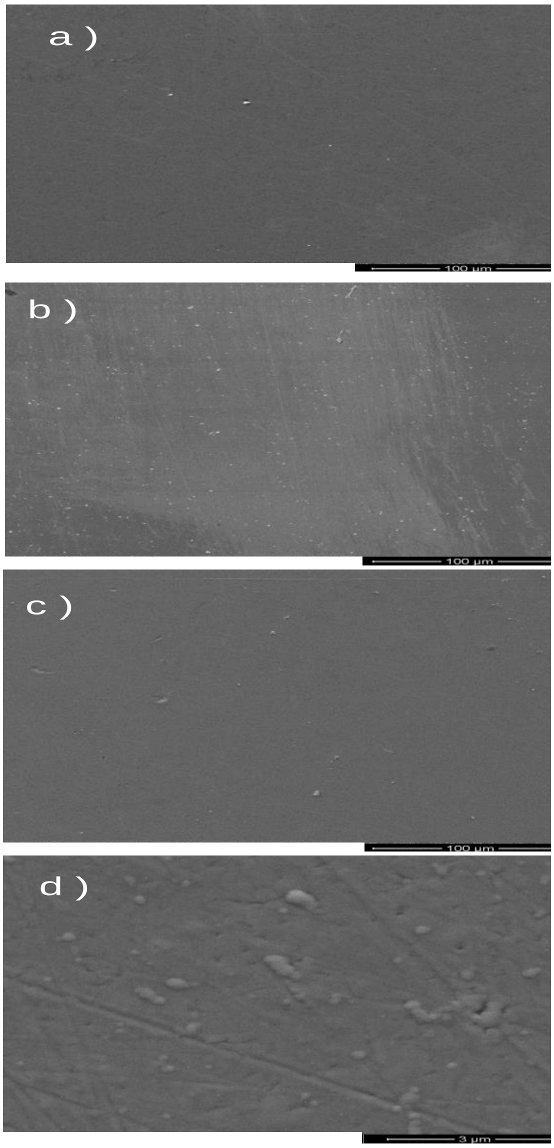

Fig. 2. SEM images of the FeCl/Cu multilayer thin films; a) 0 at. % Cu, b) 31 at. % Cu, c) 47 at. % Cu, d) 47 at. % Cu (higher magnification).

With regard to magnetic properties, the hysteresis loops were obtained from the films with 6 mm diameter in circular shape. The loops achieved by a commercial VSM considering different film contents caused by different thicknesses of Cu layer are shown in Fig. 3. The magnetizations were found as 1473 emu/cm^3, 1144 emu/cm^3, 938 emu/cm^3, 843 emu/cm^3, 672 emu/cm^3, 490 emu/cm^3, and 348 emu/cm^3 for the films with Cu layer thickness of 0 nm, 2 nm, 4 nm, 6 nm, 9 nm, 14 nm, and 24 nm, respectively, when an external magnetic field of ±20 kOe was applied. It was shown that the magnetization decreased as the Cu content in the multilayer thin films increased with increasing Cu layer thickness, see Table 1. As a result, magnetic moments per unit volume of Fe atoms decreased. In other words, since the magnetization is relevant to the number and magnitude of the aligned magnetic moments per unit volume of a sample, it can mainly change with the amount of magnetic constituent. Similarly, the saturation magnetization gradually decreased when the Co content decreased in the study [23] that investigated the Ni-Co films with different Co contents. The perpendicular hysteresis loops were also acquired. As an example, the hysteresis loop plotted by using the magnetic field perpendicular to surface of the multilayer thin film with 24 nm Cu layer thickness is presented in Fig. 3. Since the parallel (in-plane) hysteresis loop has a higher remanent magnetization and a lower coercivity than the perpendicular loop, it can be reported that the easy-axis direction of the magnetization is in the film plane due to the shape magnetic anisotropy as found in a study [24].

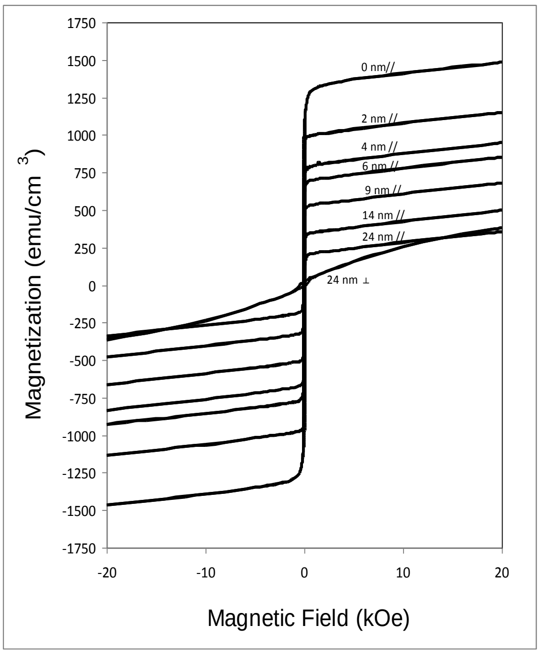

Fig. 3. Hysteresis loops of the multilayer thin films that have different thicknesses of Cu layer. (//: Parallel and ⊥: Perpendicular, Cu contents; 0 at. %, 13 at. %, 22 at. %, 26 at. %, 31 at. %, 40 at. %, 47 at. % for 0 nm, 2 nm, 4 nm, 6 nm, 9 nm, 14 nm, and 24 nm Cu layer thickness, respectively).

4. Conclusions

The FeCl/Cu multilayer thin films were grown by using two dc magnetrons sputtering system. The results revealed that the Cu content gradually increased from 0 at. % to 47 at. % as the Cu layer thickness increased from 0 nm to 24 nm. The intensities of the peaks of the fcc structure increased while that of bcc structure decreased with the increasing Cu content. The surfaces were found to be bright and simple. The magnetization value decreased as Cu contents in the multilayer thin films increased with increasing Cu layer thickness. It can be concluded that the changes in thickness of non-magnetic layer of the FeCl/Cu multilayer thin films resulted in considerable changes in the contents of the multilayer thin films and hence the structural and magnetic properties.

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*Corresponding author: alikarpuz@kmu.edu.tr