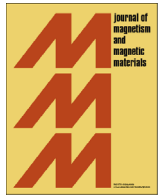




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## Characterizations of FeCl/Cu superlattices sputtered at low and high deposition rates of ferromagnetic layer

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## ABSTRACT

FeCl/Cu superlattices was grown using a dc magnetron sputtering at low (0.02 nm/s) and high (0.08 nm/s) deposition rates of ferromagnetic layer, and the properties of the superlattices were investigated. Structural characterizations by x-ray diffraction (XRD) technique showed that a mixture of the face centered cubic (111) and the body centered cubic (110) structure formed in the superlattices. In the XRD patterns, the peak intensity of (111) is higher for the superlattices grown at low deposition rate whereas the (110) peak intensity is higher at high deposition rate, and hence the preferential orientation turned from (111) to (110) when the deposition rate of the FeCl layer was increased from 0.02 nm/s to 0.08 nm/s. Compositional analysis of the superlattices by energy dispersive x-ray spectroscopy revealed that the Fe and Cl contents were 7 at.% and 66 at.%, respectively while the Cu content of the superlattices was detected to be 27 at.%. The morphology was exposed with a scanning electron microscope. The superlattice surfaces were coherent and bright. The atomic force microscopy images showed that the surfaces have almost the same roughness. The magnetic measurements revealed that the saturation magnetization and the coercivity increased with increasing deposition rates from low to high deposition rate. Also, the magnetic easy axis was found to be in the superlattice plane for all superlattices. It was seen that the variations in the magnetic properties of the superlattices might be attributed to the structural changes grown at low and high deposition rates of ferromagnetic layer.

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## 1. Introduction

In recent years, nanotechnology has been one of the valuable subjects and attracted much attention in scientific community. Also, magnetic materials with nano-scaled dimensions have shown tempting properties for comprehensive studies [1] and their applications have been reported by many researchers [2]. Therefore, the production of nano-structured materials such as superlattices, nanowires, and nanocontacts has been achieved by many scientists. Magnetic superlattices are a well known example of such materials [3]. The nanoscale magnetic superlattices can be produced by various techniques such as sputtering, electrodeposition and molecular beam epitaxy [4,5]. The sputtering technique is an advantageous method to prepare such films due to easy control

of deposition parameters and obtaining qualified product. It also has important experimental parameters which affect the properties of deposited films, such as thickness and deposition rate [6].

In this study, a series of 10[FeCl(6 nm)/Cu(6 nm)] superlattices was sputtered at low (0.02 nm/s) and high (0.08 nm/s) deposition rates of ferromagnetic layer. The superlattice properties were investigated as a function of deposition rates of ferromagnetic layers. It was observed that the changes in the magnetic properties of superlattices might have come from the structural variation of the films produced at different deposition rates of ferromagnetic layer.

## 2. Experimental

A sputtering system with a two-dc magnetron system was used to produce the magnetic superlattices. A magnetic target of FeCl composed of 70 at.% Fe and 30 at.% Cl, and a Cu target, with diameter of 6 cm, were used to fabricate the magnetic superlattices. The composition of the FeCl target was found out by inductively coupled plasma-atomic emission spectroscopy (ICP-AES, Perkin Elmer Optima

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3100 XL). The superlattices were deposited on acetate substrates with  $5\text{ cm} \times 2.5\text{ cm}$  dimensions. The targets, magnetrons and substrates were cleaned with the isopropyl alcohol before the productions. All depositions were done at room temperature and the pressure of the deposition environment was around  $10^{-3}$  mbar. The thickness of the superlattices was estimated to be 120 nm and the thicknesses of each FeCl and Cu layers were decided as 6 nm, i.e.  $10[\text{FeCl}(6\text{ nm})/\text{Cu}(6\text{ nm})]$ . The thicknesses of the layers were determined by using a quartz crystal microbalance thickness monitor. To observe the effect of different deposition rates, the low (0.02 nm/s) and the high (0.08 nm/s) deposition rates of the magnetic FeCl layers were systematically studied.

The compositional superlattice analysis was done with an energy dispersive x-ray spectroscopy (EDX, GENESIS APEX 4—EDAX, AMETEK). The crystal structure of the superlattices was found out with an x-ray diffraction technique (XRD, PANalytical) and the  $\text{Cu-K}\alpha$  radiation was used for the XRD technique. A scanning electron microscope (SEM, FEI™, NOVANOSEM430) was used to observe the surface morphologies of the superlattices. Also, the surface roughness was investigated by the atomic force microscopy (AFM, NanoMagnetics Instruments) by considering  $5\text{ }\mu\text{m} \times 5\text{ }\mu\text{m}$  scanning field to obtain detailed information about the surface of the films. The magnetic properties were investigated with a vibrating sample magnetometer (VSM, ADE TECHNOLOGIES DMS-EV9) by achieving the hysteresis loops of the superlattices between  $+20\text{ kOe}$  and  $-20\text{ kOe}$ . The loops were obtained by using the circular shaped films with 6 mm diameter as performed in [7] and at room temperature.

### 3. Results and discussion

To study the effect of deposition rates of FeCl layers on structural properties of the superlattices, the XRD patterns of the FeCl/Cu superlattices are illustrated in Fig. 1. The XRD patterns revealed that the crystal structure of the superlattices is a mixture of the face centered cubic (fcc) and the body centered cubic (bcc) structure. The main Bragg peaks arising from the fcc (111) and the bcc (110) planes were observed at  $2\theta \approx 43^\circ$  and  $45^\circ$ , respectively. The intensity of the fcc (111) peak was higher than that of the bcc (110) peak for the superlattice grown at deposition rate of 0.02 nm/s. On the other hand, the intensity of the fcc (111) peak was low and that of the bcc (110) peak was high when the deposition rate was adjusted as 0.08 nm/s. Also, the fcc (111) peak obtained at high deposition rate became larger than the fcc (111) peak of the superlattice grown at low deposition rate. Therefore, it can be decided that preferential orientation turned from (111) to

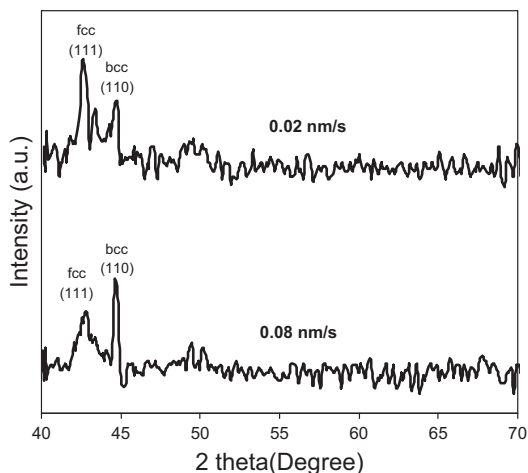


Fig. 1. XRD patterns of the FeCl/Cu superlattices grown at low (0.02 nm/s) and high (0.08 nm/s) deposition rates of FeCl layer.

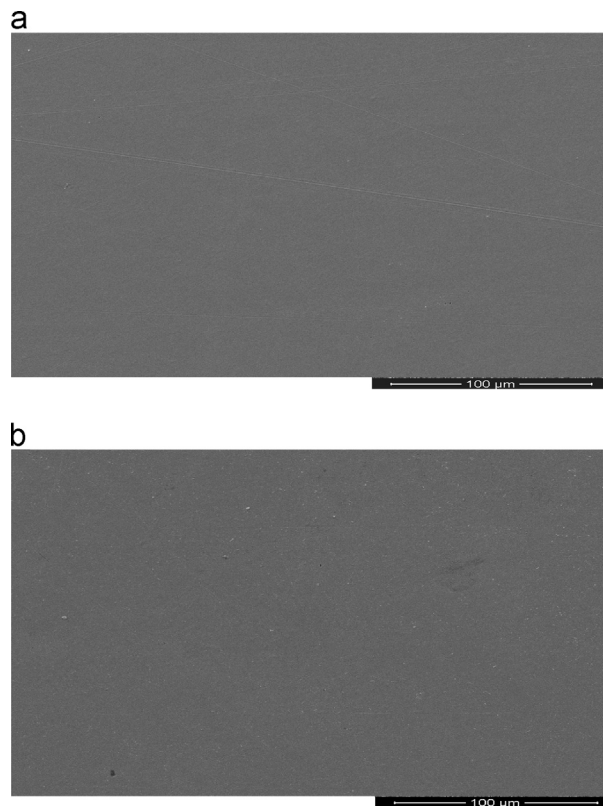


Fig. 2. SEM images of the FeCl/Cu superlattices grown at (a) low (0.02 nm/s) and (b) high (0.08 nm/s) deposition rates of FeCl layer.

(110) when the deposition rate of the FeCl layer was increased from 0.02 nm/s to 0.08 nm/s.

According to compositional analysis, the Cu content was detected as 27 at.% for the superlattices grown at low (0.02 nm/s) and high (0.08 nm/s) deposition rates of ferromagnetic layers. Atomic Fe and Cl contents were found as 7% and 66%, respectively. This result is different from that of the study [8] which investigates the effect of deposition potential and hence deposition rate on properties of electrodeposited superlattices. For surface morphology of the superlattices at low and high deposition rates, the SEM images of the superlattices were taken and are presented in Fig. 2(a) and (b), respectively. The surfaces are generally fine, i.e. coherent and bright structures were observed for the deposition rates, and any prominent grainy or dendritic regions were not observed in the SEM images. The roughness analysis of the surfaces was achieved by analyzing AFM images shown in Fig. 3. The figure shows that the surfaces have almost the same roughness irrespective of deposition rate of FeCl layer. The average roughness parameter was found to be  $\sim 29\text{ nm}$  for the films grown at low (0.02 nm/s) and high (0.08 nm/s) deposition rates of FeCl layer. A moderate bumpy surface was observed from the AFM images.

Fig. 4 shows the hysteresis loops (parallel and perpendicular) of the FeCl/Cu superlattices grown at low and high deposition rates of FeCl layer. The figure was presented between  $+18\text{ kOe}$  and  $-8\text{ kOe}$  external magnetic field. The magnetizations were found to be 755 and  $805\text{ emu/cm}^3$  for deposition rates of 0.02 nm/s and 0.08 nm/s, respectively, when an external magnetic field of 20 kOe was applied. The coercivity,  $H_c$ , values of the superlattices were 20 Oe and 25 Oe for deposition rates of 0.02 nm/s and 0.08 nm/s, respectively. In other words, the high deposition rate provoked a magnetically harder film. Also, the perpendicular hysteresis loop was obtained to find the easy-axis direction of the magnetization. The perpendicular loop of the superlattice grown at 0.08 nm/s deposition rate is also presented in Fig. 4. By comparing the parallel and perpendicular loops, it can be

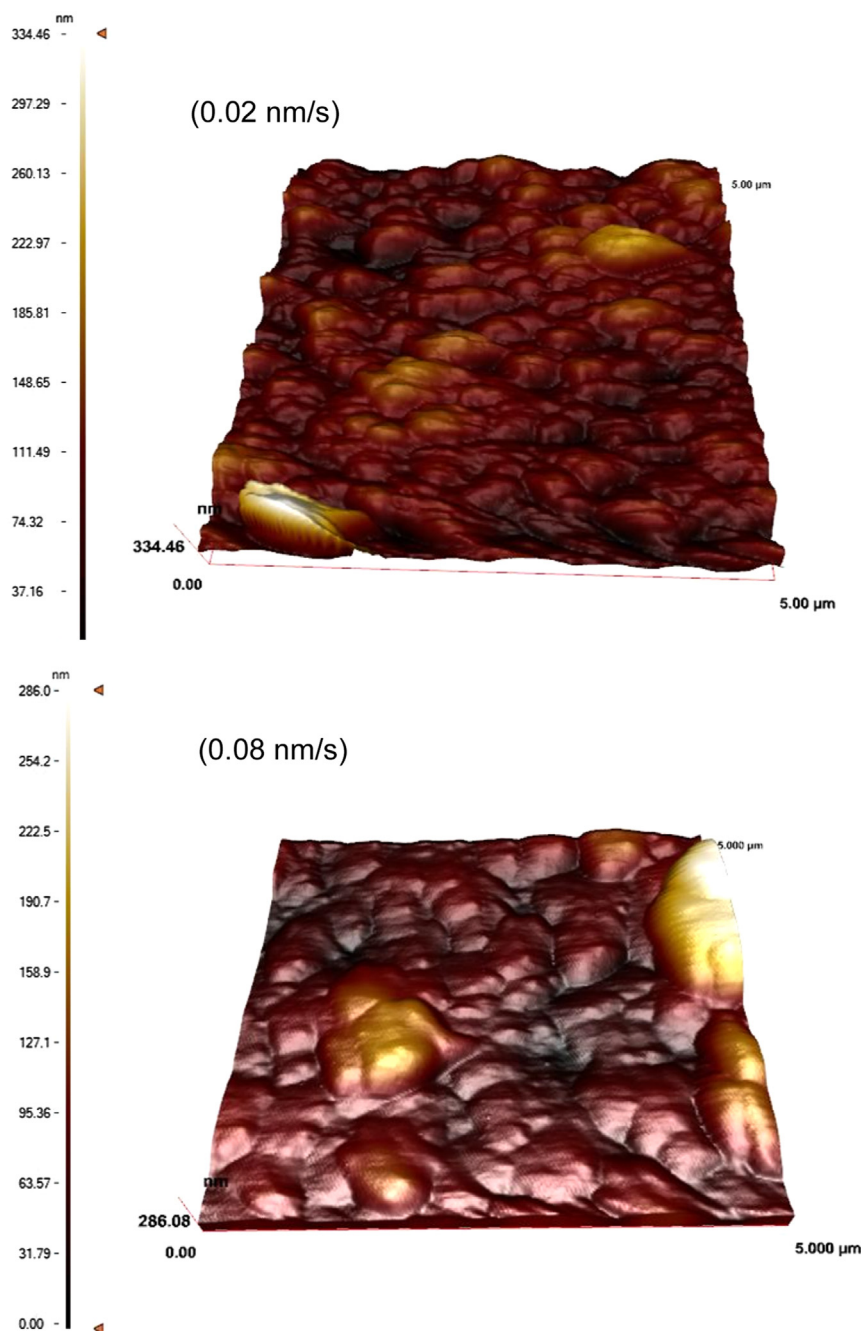


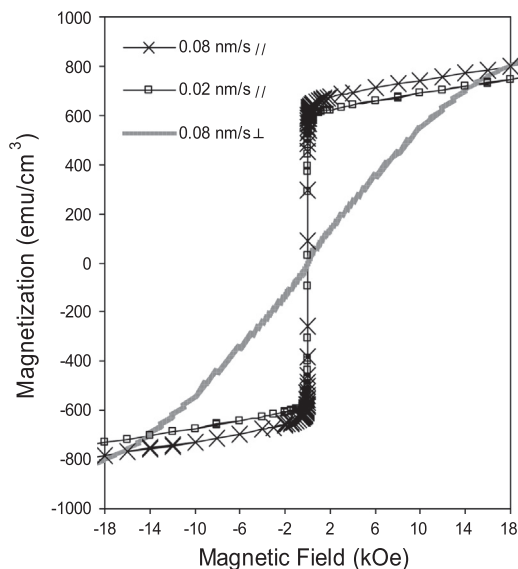
Fig. 3. AFM images of the FeCl/Cu superlattices grown at low (0.02 nm/s) and high (0.08 nm/s) deposition rates of FeCl layer.

noted that the easy-axis direction of the magnetization is in the superlattice plane, since the in-plane hysteresis loop has a higher remanent magnetization and a lower  $H_c$  than those of the perpendicular loop. This can be attributed to the shape anisotropy is in the film plane. A similar response of magnetization to magnetic field was also announced in studies [9,10] for Co–Ni–Cu ternary and Fe–Cu binary alloys, respectively. It is concluded that the differences observed in the magnetic properties of the FeCl/Cu superlattices can be referred to the structural changes caused by the deposition rates of the ferromagnetic layer.

#### 4. Conclusion

FeCl/Cu superlattices were sputtered at low and high deposition rates of FeCl layers. Two main peaks appeared at  $2\theta \approx 43^\circ$  and

$45^\circ$  in the XRD patterns and were labeled as the fcc (111) and the bcc (110), respectively. Therefore, it was concluded that the superlattices have a mixture of the fcc and the bcc structure. The intensity of the bcc (110) peak was higher than that of the fcc (111) peak for the superlattice grown at high deposition rate unlike the other superlattice deposited at low deposition rate. The preferential texture turned from (111) to (110) when the deposition rate of the ferromagnetic layer was increased from 0.02 nm/s to 0.08 nm/s. For the superlattices deposited, the Fe, Cl and Cu contents were 7 at.%, 66 at.% and 27 at.%, respectively. The surfaces of all superlattices are generally bright and coherent. Roughness analysis indicated that although the films were grown at different deposition rates, almost the same roughness was observed in the film surfaces. According to magnetic measurements, the magnetizations were detected as 755 and 805 emu/cm<sup>3</sup> for deposition rates of 0.02 nm/s, and 0.08 nm/s, respectively, at 20 kOe external



**Fig. 4.** Hysteresis loops of the FeCl/Cu superlattices grown at low (0.02 nm/s) and high (0.08 nm/s) deposition rates of FeCl layer (//: parallel and  $\perp$ : perpendicular).

magnetic field. It was also found that the  $H_c$  of the superlattices increased from 20 Oe to 25 Oe when the deposition rate of the FeCl layer was increased from 0.02 nm/s to 0.08 nm/s. Results of the magnetic measurements imply that the properties of the superlattices can be controlled with their deposition parameters for any potential sensor technology.

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