

## Magnetic properties affected by structural properties of sputtered Ni/Cu multilayer films with different thicknesses of Ni layers

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**Abstract**–Nickel-containing magnetic films have become the focus of attention due to their outstanding properties. These films are produced by many methods, including the sputtering technique. In this study, structural and magnetic properties of Ni/Cu multilayer films with different (from 92.5 nm to 17.5 nm) thicknesses of the Ni layers were investigated. The magnetron sputtering process was used to produce the Ni/Cu multilayer films. X-ray diffraction analysis showed that the films have a face-centered cubic structure with (111) plane. According to the scanning electron microscope images, while the films with the Ni layers thicknesses of 92.5 nm and 42.5 nm have some cracks and row structures on their surfaces, the films with lower thicknesses of the Ni layers have relatively more regular surfaces. As the Ni layers thickness decreased, the saturation magnetization ( $M_s$ ) decreased from 617 emu/cm<sup>3</sup> to 387 emu/cm<sup>3</sup>. Although the Ni/Cu multilayer with the Ni layer thickness of 92.5 nm had the highest atomic Ni content (76%); its coercivity ( $H_c$ ) value was also the highest with 144 Oe. There was a decrease in the  $H_c$  value and grain size with decreasing the Ni layer thickness and the change in the  $H_c$  value can be related to the film content and surface morphology. The remanent magnetization ( $M_r$ ) value changed between 492 emu/cm<sup>3</sup> and 105 emu/cm<sup>3</sup> with the reducing the Ni layers thickness. The highest  $M_r$  value and the highest  $M_r/M_s$  ratio were obtained for the Ni/Cu film with the Ni layers thickness of 42.5 nm. This Ni/Cu film has also the highest magnetization energy. The detected magnetic properties make this film desirable for permanent magnet and magnetic recording applications among the investigated Ni/Cu multilayers.

Keywords: Magnetron Sputtering, Ni/Cu Multilayers, Layer Thickness, Structural Properties, Magnetic Properties

### INTRODUCTION

Nanomaterials and their applications have been becoming more popular thanks to their excellent mechanical, electrical, and magnetic properties [1-3]. The main aim of this popular approach is to produce effective, smaller, and well-equipped microsystems by improving these properties. In the same manner, the magnetic materials used in permanent magnets and magnetic recording applications have also been extensively studied due to the increasing need for information storage and their wide application areas [4,5]. It is known that there are different nanomaterial types, including nanotubes, nanowires, nanoparticles, and nanofilms [3]. Nanofilms can also be in different forms as single layer alloy and multilayer structure [4,6-8]. A study [9] showed that material properties such as fatigue, corrosion, wear, toughness can be significantly enhanced by producing materials in a layered composite structure. Multilayer structures can be fabricated in various methods such as bonding, sputtering, electrodeposition, and evaporation [10-13]. In addition, the magnetron sputtering technique is quite common and can offer many advantages when producing multilayer structures [14-17]. For example, Schoeppner et al. [17] stressed that while a microstructural parameter such as grain size is constant, it is difficult to alter the precipitate size, but magnetron sputtering enables the alteration

of many microstructural parameters. Liu et al. [18] showed that though the pressure of the sputter chamber and the argon gas, the deposition rate, and the substrate temperature were fixed, layer thicknesses of both Cu and Ni for different multilayers could be changed. It can be considered that magnetron sputtering gives more flexibility to fabricate multilayer structures with different parameters and elements. As a magnetic element, Ni shows ferromagnetic properties and has high corrosion resistance. As for Cu, it has been extensively used in microelectronic systems and integrated circuits since Cu has relatively high thermal and electrical conductivity [19,20]. Ni and Cu show minimum lattice mismatch (2.5%) (coherent stress), and therefore tribological and magnetic features of Ni and Cu elements are broadly studied [8,21,22]. Also, Ni/Cu magnetic multilayers demonstrate enhanced magnetic properties, hardness, and strength, so they are widely used as a component in magnetic devices and microactuators [23,24]. Although there have been many articles related to Cu/Ni multilayers produced by using the magnetron sputtering [16,25-27], Ni/Cu multilayer films deposited by the magnetron sputtering process and their structural and related magnetic properties have not been sufficiently examined except for our previous studies [28,29]. Barshilia et al. [25] examined Cu/Ni multilayer coatings deposited by magnetron sputtering and characterized by nanoindentation and atomic force microscopy. Liu et al. [27], on the other hand, investigated highly textured Cu/Ni multilayers with different individual layer thicknesses grown by the magnetron sputtering on Si substrate. In our recent work [29], as well as pure Ni and Cu films with 200 nm thickness,

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Ni/Cu multilayer films with various thicknesses of the Cu layers were produced by using the magnetron sputtering. Note that production and microstructural parameters can significantly change the magnetic performance and therefore technological applications of the materials [10,30]. In addition, many physical properties of a multilayered film depend strongly on the production parameters such as layer thickness. Although Ni/Cu multilayers exhibit quality adhesion property, in order to obtain cleaner surfaces and better adhesion among layers, it is very important to choose optimal layer thickness when multilayers are fabricated [31]. Additionally, studies [1,23] emphasized that tribological properties depend highly on the layer properties of the components such as hardness, roughness, and thickness. In the presented study, magnetic films with different Ni layers thicknesses were produced by the magnetron sputtering and then the response of magnetic properties to changing microstructural properties caused by different layer thicknesses of Ni was investigated. The aim of this study was also to determine the optimal Ni layers thickness of the Ni/Cu multilayer films for effective and functional potential use of these structures in magnetic applications such as magnetic sensors, magnetic data storage and equipments using magnet technologies. In this context, some suitable applications have been determined for the materials investigated, considering their macroscopic magnetic properties.

#### EXPERIMENTAL DETAILS

Ni/Cu multilayer films were fabricated on a commercial cellulose acetate layer by using a direct current magnetron sputtering process which has two separate magnetrons at the bottom and a substrate holder placed opposite the magnetrons. To manufacture magnetic multilayer films, 99.9% purified Ni and Cu targets were used and placed on the magnetrons, separately. The target materials are also commercial and they have 2 mm thickness and 50.8 mm diameter. Thanks to the magnetron sputtering technique, multilayer structures were deposited at room temperature and low pressure (nearly  $4.5 \times 10^{-3}$  mbar). Each layer thickness of the Ni/Cu multilayers was determined by a thickness monitor until the deposited layer reached the predetermined thickness. In addition, in all samples, the deposition rate of the layers, Cu layer thickness, and the total thickness of the films were fixed at 0.05 nm/s, 7.5 nm, and 200 nm, respectively. Ni layer thickness, on the other hand, was separately set at 92.5 nm, 42.5 nm, 32.5 nm, 17.5 nm to examine the effect of the alterations in the magnetic layer thickness. Sche-

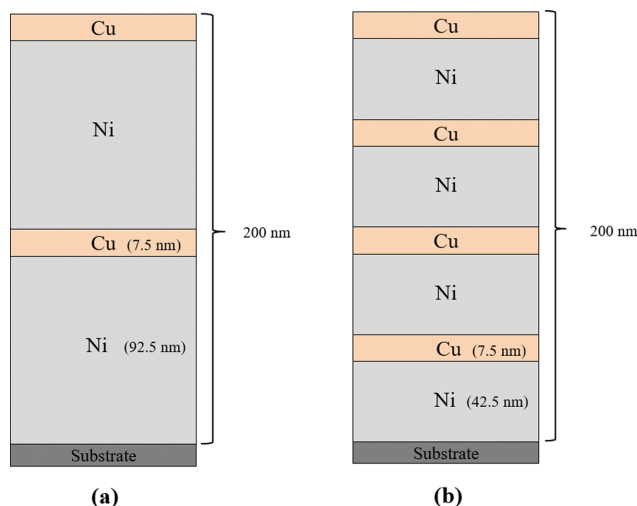


Fig. 1. Schematic drawings for two multilayer structures, (a)  $2[\text{Ni}(92.5 \text{ nm})/\text{Cu}(7.5 \text{ nm})]=200 \text{ nm}$ , (b)  $4[\text{Ni}(42.5 \text{ nm})/\text{Cu}(7.5 \text{ nm})]=200 \text{ nm}$ .

matic drawings for two multilayer structures are given in Fig. 1 and the symbolic representation,  $n[\text{Ni}(a)/\text{Cu}(b)]$ , of the films can be seen in Table 1. In this presentation, a and b denote the thickness of each individual layer, while n specifies the bilayer number. As seen in Fig. 1, while the first layer above the substrate is Ni, the top of the multilayer structures is Cu. Before the production process started, inside of the vacuum chamber, Ni and Cu targets, and acetate substrate were cleaned according to the cleaning protocol. The targets and the substrate were placed to magnetrons and to the substrate holder, respectively. The pressure inside the chamber was reduced by using pumps. Once the required pressure for producing multilayer structures was obtained, argon gas was released to the chamber, and the gas flow was fixed at 40 sccm during production. By accelerating argon ions towards the target materials, Ni or Cu atoms were plucked from the target materials and were carried to the substrate. Initially, the Ni layer was deposited on the substrate. Once the deposition of the first Ni layer was finished, the Cu layer was deposited on the initially deposited Ni layer. That process was repeated 2, 4, 5, 8 times for the films with the Ni layer thickness of 92.5 nm, 42.5 nm, 32.5 nm, 17.5 nm, respectively (see Fig. 1). When the total thickness of the deposited film reached 200 nm, the sputtering process was finished and the deposited film

Table 1. Compositional, structural, and magnetic data obtained from the analysis of the Ni/Cu multilayer films sputtered by considering different thicknesses of magnetic (Ni) layers

Nickel (Ni) thickness (nm)	Samples	Film composition (EDX)		Crystal structure (XRD)		Magnetic analysis (VSM)		
		Nickel (at.%)	Copper (at.%)	Lattice parameter (nm)	Grain size (nm)	$M_s$ ( $\text{emu}/\text{cm}^3$ )	$M_r$ ( $\text{emu}/\text{cm}^3$ )	$H_c$ (Oe)
92.5	$2[\text{Ni}(92.5 \text{ nm})/\text{Cu}(7.5 \text{ nm})]=200 \text{ nm}$	76	24	0.35167	52	617	234	144
42.5	$4[\text{Ni}(42.5 \text{ nm})/\text{Cu}(7.5 \text{ nm})]=200 \text{ nm}$	64	36	0.35345	40	558	492	129
32.5	$5[\text{Ni}(32.5 \text{ nm})/\text{Cu}(7.5 \text{ nm})]=200 \text{ nm}$	59	41	0.36113	45	550	235	114
17.5	$8[\text{Ni}(17.5 \text{ nm})/\text{Cu}(7.5 \text{ nm})]=200 \text{ nm}$	44	56	0.36118	27	387	105	111

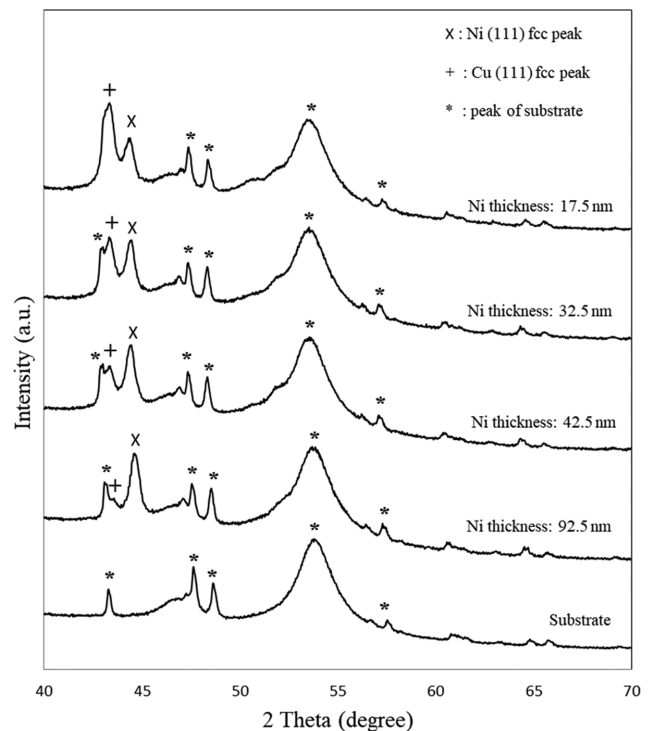
was taken from the sample holder.

The compositional analysis of the deposited films with the Ni layer thicknesses ranging from 92.5 nm to 17.5 nm was done by using the energy-dispersive X-ray spectroscopy (EDX, AMETEK). By using the X-ray diffraction technique (XRD, PANalytical), the crystal structural analysis was performed with the Cu- $K_{\alpha}$  radiation ( $\lambda=0.15406$  nm). The patterns obtained from the XRD measurement were acquired from scanning of  $2\theta$  between  $40^{\circ}$  and  $70^{\circ}$  with scanning step of  $0.026^{\circ}$ . The surface morphology of the Ni/Cu multilayers was analyzed by a scanning electron microscope (SEM, FEI, Quanta 200 FEG). Hysteresis loops of the samples were measured by using a vibrating sample magnetometer (VSM, ADE TECHNOLOGIES DMS-EV9). The magnetic field ranging from  $-20$  Oe to  $+20$  Oe was applied parallel to the film plane at room temperature in all magnetic measurements.

## RESULTS AND DISCUSSION

A cellulose acetate layer was chosen as a substrate because it shows non-magnetic behavior. Another reason why acetate substrate was preferred is that this type of substrate is also suitable for the experimental techniques used for the analysis on account of proper flexibility or low background signals. Compositional, structural, and magnetic data obtained from the analysis of the Ni/Cu multilayer films sputtered with different magnetic (Ni) layer thicknesses are shown in Table 1. According to EDX analysis, as the Ni layer thickness of the multilayer films decreased from 92.5 nm to 17.5 nm, the Ni content of the films decreased from 76% to 44%, and the Cu content of the films increased from 24% to 56%. As expected, multilayer films consist of Ni and Cu atoms and there is no other element determined in the composition of the films.

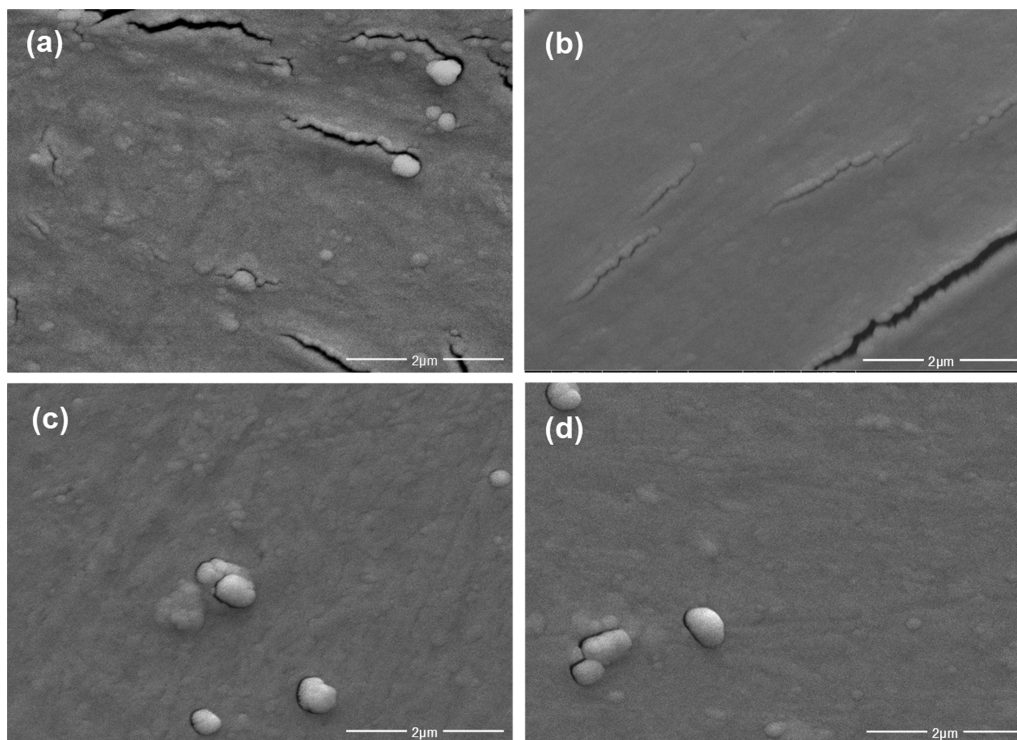
Fig. 2 shows the XRD patterns of the films and substrate. Calculated XRD data such as lattice parameters and grain sizes are given in Table 1. The acetate substrate had some peaks and they were labeled with asterisks as seen in Fig. 2. The XRD analysis reveals that multilayer thin films are in face-centered cubic (fcc) structure and their crystallinity is preferably oriented at the (111) direction. It is clear that there are some differences in the XRD patterns as the Ni layers thickness of the films changes. The intensity of the Cu (111) fcc peak appearing at around  $2\theta \approx 43^{\circ}$  increased with increasing Cu content of the films. When the Ni layers thickness and therefore Ni content of the multilayers decreased gradually, the intensity of the Ni (111) fcc peak occurring at  $2\theta \approx 44^{\circ}$  also decreased. Barshilia et al. [25] investigated Cu/Ni multilayer coatings fabricated by magnetron sputtering. Unlike the presented work, this investigation showed that both Cu (111) and Ni (111) fcc peaks can be seen at the high bilayer thickness ( $433 \text{ \AA}$ ) ( $195 \text{ \AA}$  Cu and  $238 \text{ \AA}$  Ni), while there is only one (111) fcc peak appearing at the low bilayer thicknesses. As seen in Fig. 2, the film with the thickest Ni layer thicknesses (92.5 nm) has the highest intensity of the Ni (111) fcc peak and the lowest intensity of the Cu (111) fcc peak. Conversely, the film with the thinnest Ni layer thicknesses (17.5 nm) has the highest intensity of the Cu (111) fcc peak and the lowest intensity of the Ni (111) fcc peak among the multilayers. Unlike the other multilayers, the intensities of Ni (111) and Cu (111) fcc peaks of the film with the Ni layers thickness of 32.5 nm are almost



**Fig. 2. The XRD patterns of the Ni/Cu multilayer films produced with different thicknesses of the Ni layers and the substrate used during deposition.**

equal. By using the XRD data, the lattice parameters were also calculated as 0.35167 nm, 0.35345 nm, 0.36113 nm, and 0.36118 nm for the samples with the Ni layers thickness of 92.5 nm, 42.5 nm, 32.5 nm, and 17.5 nm, respectively (see Table 1). The lattice parameters of the films with the Ni layers thicknesses of 92.5 nm and 42.5 nm are fairly close to the lattice parameter of bulk Ni ( $a_{Ni}=0.35238$  nm), while the lattice parameters for the thicknesses of 32.5 nm and 17.5 nm are fairly close to that of bulk Cu ( $a_{Cu}=0.36150$  nm). In particular, when the Ni layers thickness decreases from 42.5 nm to 32.5 nm, and therefore atomic Ni content decreases from 64% to 59%, there can be said a significant increase in the lattice parameters of the films. By using the Scherrer formula [32] the average grain sizes of the films were determined as 52 nm, 40 nm, 45 nm, and 27 nm for the Ni layers thicknesses of 92.5 nm, 42.5 nm, 32.5 nm, 17.5 nm, respectively. Kuru et al. [12] produced NiCu/Cu multilayer films by electrodeposition and announced that average grain sizes of the films were relatively constant, while ferromagnetic layer thickness (NiCu) and therefore Ni content changed. However, in the presented study, while the Ni layers thickness and therefore the Ni content decreased, average grain sizes also decreased, as seen in Table 1. When the results of the study [12] are compared with the results of the presented study, it is clear that the dependence of grain size on film composition can be different. The reason why the dependence of the calculated grain size on the film content in the presented study differs from that reported in the study [12] can be attributed to the use of Ni instead of NiCu as the ferromagnetic layer or the different thickness of the layers.

As a result, the deposited Ni/Cu films crystallized in fcc struc-



**Fig. 3.** The SEM images of the Ni/Cu films by considering different thicknesses of the Ni layers: (a) 92.5 nm, (b) 42.5 nm, (c) 32.5 nm, (d) 17.5 nm.

ture with a single plane of (111). The existence of the fcc structure despite the significant change in the film content can be attributed to the fcc structure of pure Ni and the pure Cu layers. The considerable changes were detected in the crystalline structure properties of the investigated Ni/Cu multilayers when the Ni layers thickness gradually decreased from 92.5 nm to 17.5 nm. The changes in the peak intensity and lattice parameter are also compatible with the film contents detected with the EDX analysis.

The SEM images obtained from the surfaces of the films produced to investigate the effect of different thicknesses of the Ni layers are shown in Fig. 3. Fig. 3(a) shows the surface image of the Ni/Cu film with the Ni layers thickness of 92.5 nm. When this picture is examined, it is understood that the surface is non-uniform and has some structures such as grains and irregular crevices. The length of these crevices can reach  $\sim 2 \mu\text{m}$  and also the maximum value of the diameter of the grains is up to  $0.5 \mu\text{m}$ . This surface also has some rows of structures extending diagonally. When each Ni layer thickness was 42.5 nm (see Fig. 3(b)), it was determined that the grainy structures and crevices disappeared to some extent, but the diagonally extending row structures remained on the surface of the film. McDonald et al. [26] investigated residual stresses in Cu/Ni multilayer thin films produced by magnetron sputtering with different layer thicknesses and revealed that multilayer films with 10 nm layer thicknesses displayed uniform microcracking on the surface as seen in the films with the thick (92.5 nm and 42.5 nm) Ni layers in the presented study. Also, McDonald et al. [26] explained that films with individual layer thicknesses of 25 nm and greater did not demonstrate microcracking on the surface. Another work carried out by Zhu et al. [31] displayed that

while  $\text{Bi}_2\text{Te}_3/\text{Cu}$  films had some cracks on the surface, there was no crack on the surface of the  $\text{Bi}_2\text{Te}_3/\text{Ni}/\text{Cu}$  films produced by the same technique and the conditions. Fig. 3(c) shows the surface morphology of the Ni/Cu film with the Ni layers thickness of 32.5 nm. As can be clearly seen, there is no considerable formation on the surface other than grains with different sizes. The sizes of these grains are different from each other and they can reach up to  $1 \mu\text{m}$ . It can be said that the surface of the film with the Ni layers thickness of 32.5 nm is more regular than the films with thicker Ni layers. When each Ni layer thickness is set to 17.5 nm, this relatively regular structure continues as seen in Fig. 3(d). The grains detected in the films with thinner Ni layer thicknesses (32.5 nm and 17.5 nm) were observed sparsely on the surface. Hemmous et al. [24] studied the effect of substrates, thickness, and Cu underlayer on structure, surface morphology, and electrical properties of evaporated Ni thin films. And also, this article stressed that the Ni surface with the Cu underlayer is more uniform and smoother in comparison with produced Ni/substrate films without Cu underlayer. Consequently, when the Ni layer thickness was reduced from 42.5 nm to 32.5 nm, the film surface showed significant improvement in point of qualification and it appeared in a more regular morphology and suitable shape (see Fig. 3). The gradual decrease in the thickness of the magnetic layers from 92.5 nm to 17.5 nm caused considerable alterations in the surface of the Ni/Cu films with constant Cu layers thickness (7.5 nm). The changes detected on the film surface with the decrease in Ni layers thickness can be related to the decrease in the grain size calculated by the crystal structure analysis. Namely, the significant reduction in grain size can have a supportive contribution to the increase in surface quality.

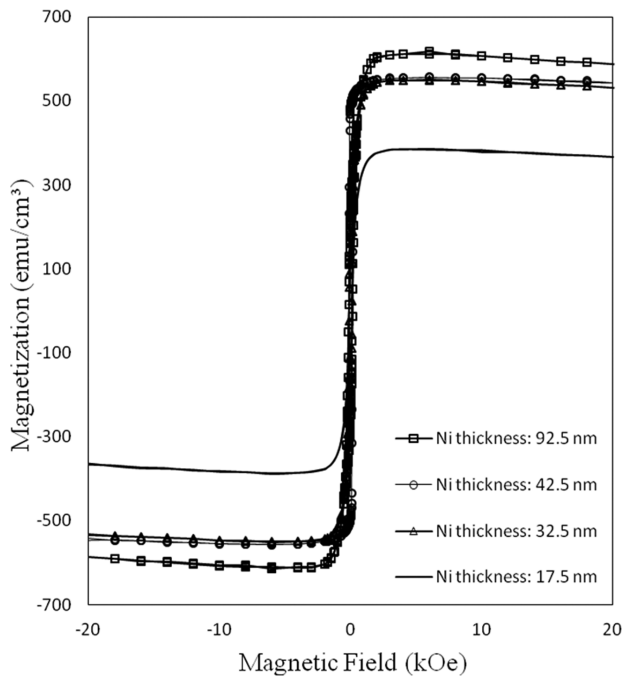


Fig. 4. Hysteresis loops of the Ni/Cu multilayer films sputtered with different thicknesses of the Ni layers.

It is clearly stated in study [33] that the grain size has an impressive role on the surface morphology. From this point of view, it can be said that different thicknesses of individual layers significantly affect the surface morphology of the Ni/Cu multilayer films.

Hysteresis loops of the Ni/Cu multilayer films with various thicknesses of the Ni layer are given in Fig. 4. The saturation magnetization ( $M_s$ ), the coercivity ( $H_c$ ), and the remanent magnetization ( $M_r$ ) values listed in Table 1 are also obtained from the hysteresis loops.  $M_s$  values were detected as 617 emu/cm<sup>3</sup>, 558 emu/cm<sup>3</sup>, 550 emu/cm<sup>3</sup>, 387 emu/cm<sup>3</sup> for the Ni layers thicknesses of 92.5 nm, 42.5 nm, 32.5 nm, and 17.5 nm, respectively. For the film with the Ni layers thickness of 92.5 nm, the  $M_s$  value was at the highest value (617 emu/cm<sup>3</sup>) because of the highest magnetic content. The value gradually decreased for the lower Ni layers thicknesses in accordance with the decreasing atomic Ni content. The same trend was also observed for the  $H_c$  values. As the thickness of the Ni layers gradually decreased from 92.5 nm to 17.5 nm, as indicated, the  $H_c$  value gradually decreased from 144 Oe to 111 Oe. The correlation of  $M_s$  and  $H_c$  depending on the Ni content of the Ni/Cu multilayers is given in Fig. 5. Generally, adding Cu into a magnetic material increases  $H_c$  value as Cu has a non-magnetic structure [30]. However, it is well-known that film surface structure also has a significant effect on the  $H_c$  value, as indicated in [10,22,34]. A study [34] stressed that various factors are associated with the increase of the coercive field such as voids and oxidation in the grain boundary, disordered structure, and crystalline defects. Besides, Hemmous et al. [24] revealed that there is an obvious correlation between grain size and coercive field. According to study [24], magnetic properties such as coercive field can be affected by the change in grain size. In the presented study, although the atomic Cu content of the film with 92.5 nm Ni layers thickness is the least

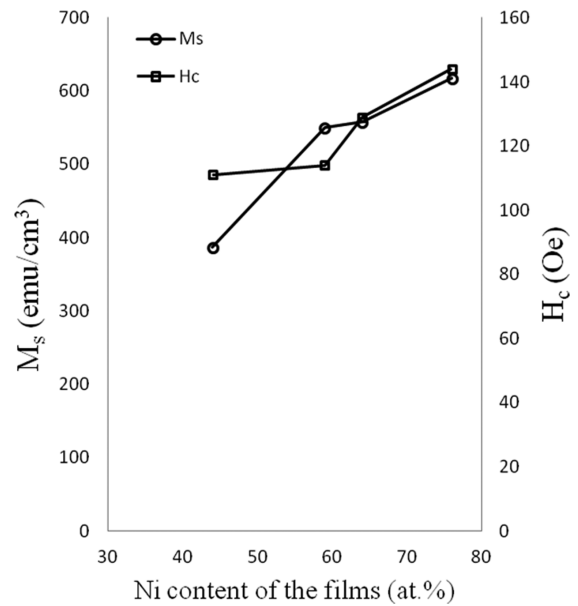


Fig. 5. The  $M_s$  and  $H_c$  values depending on the Ni content of the Ni/Cu multilayers.

(24 at%), the reason for the highest  $H_c$  value (144 Oe) can be attributed to the relatively complex surface structure of this film (see Fig. 3(a)). In other words, it can be assumed that the negative effect of stress and defects on the surface of this film on  $H_c$  outweighs the soft magnetic properties of Ni atoms. Considering the surface structure displayed in the SEM images (see Fig. 3) and the Cu content of the films (see Table 1), this situation can be generalized for all Ni/Cu multilayer films investigated. Structural, electrical, and magnetic properties of evaporated Ni/Cu and Ni/glass thin films were investigated by Nacereddine et al. [32]. The study [32] explained that while the Ni layer thickness increased, the grain size of the thin films also increased, and the increase in the grain size caused higher  $H_c$  until a critical Ni layer thickness value. A similar tendency can be seen between  $H_c$  and grain size in our presented study. Namely, while the Ni layer thickness decreased from 92.5 nm to 17.5 nm, both grain size and  $H_c$  value gradually decreased (see Table 1).  $M_r$  values were detected for all films within the scope of the magnetic analysis. According to the results,  $M_r$  values were found as 234 emu/cm<sup>3</sup>, 492 emu/cm<sup>3</sup>, 235 emu/cm<sup>3</sup>, and 105 emu/cm<sup>3</sup> for the Ni/Cu multilayers with the Ni layers thicknesses of 92.5 nm, 42.5 nm, 32.5 nm, and 17.5 nm, respectively. When the results are examined, it is seen that the film with 42.5 nm Ni layer thickness has the highest  $M_r$  ratio and it is calculated that the  $M_r/M_s$  ratio of this film is maximum (0.88) among the investigated Ni/Cu multilayers. This film has also the highest value of magnetization energy, which is mainly determined with the product of  $M_r$  and  $H_c$  values for permanent magnets [30]. Hemmous et al. [34] studied structural and magnetic properties of Ni/Cu bilayers and found that the film with the Ni layer thickness of 67 nm had the highest  $M_r/M_s$  ratio with 0.67. However, this value is lower than the calculated value (0.88) for the film with the Ni layers thickness of 42.5 nm in the presented study. To sum up, the film with the Ni layers thickness of 42.5 nm has mainly diagonally extend-

ing row structures on the surface, and also, it has the highest  $M_r/M_s$  ratio, the highest value of magnetization energy, and relatively high  $H_c$  value. This combination of magnetic properties [30] can be desirable for potential applications of permanent magnet and magnetic recording technology where the Ni/Cu multilayers are used.

### CONCLUSIONS

According to the results, as the Ni layer thickness of the Ni/Cu films decreased, the Ni content decreased while the Cu content increased. Microstructure of the films was affected by the change in film content caused by the change in the thickness of Ni layers.  $M_s$  value gradually decreased with decreasing the Ni layers thickness from 92.5 nm to 17.5 nm. Although the Cu content of the films increased with decreasing the Ni layers thickness, the  $H_c$  value decreased from 144 Oe to 111 Oe. The reason for the decrease in the  $H_c$  value can be that the effect of the change of surface on the  $H_c$  value was more dominant than the effect of the film content.  $M_r$  values, on the other hand, changed between 492 emu/cm<sup>3</sup> and 105 emu/cm<sup>3</sup>. It is also understood that it is important to determine an optimal Ni layer thickness for modifying and improving the magnetic properties of Ni/Cu multilayer films. It is disclosed that the film with the Ni layers thickness of 42.5 nm had a comparatively high  $H_c$  value, the highest  $M_r/M_s$  ratio, and the highest magnetization energy. Therefore, this film may be a better option for potential applications of magnetic recording and permanent magnets where Ni/Cu multilayers will be used. It is also inferred that as layer thickness of a multilayer film changes, different magnetic properties can be detected and these properties can be a reason for the preference for potential applications.

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