Rotation speed induced properties of quaternary FeNiCrCd thin films easy-prepared from a single magnetron sputtering

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A series of quaternary FeNiCrCd thin films were sputtered on commercial flexible acetate substrates from $Fe_{51.07}Ni_{13.42}Cr_{12.67}Cd_{22.74}$ (at.%) source under the rotation speed (0, 15, 30 and 45 rpm) of the substrates. The films were 50 nm thick and sputtered at 0.9 nm/s. As far as concerned, this work has been the first production from a single source and characterisations of the FeNiCrCd thin films by now. The atomic Fe, Ni and Cr contents in the films decreased while atomic Cd content increased with the increase of rotation speed. The crystal structure of all films was observed to have a body-centred cubic phase and the intensity of (110) and (200) peaks decreased with the decrease of Fe, Ni and Cr contents as the rotation speed increased. According to structural analysis, increasing rotation speed of the substrate caused an increase in grain size. The magnetic measurements of the films achieved, by a vibrating sample magnetometer at room temperature, displayed that the saturation magnetization, M_S values decreased from 780 to 322 emu/cm^3 and the coercivity, H_C values slightly increased from 6 to 10 Oe with increasing substrate rotation. It was observed that decrease of Fe and Ni contents, and grain size in the films influences the decrease of the M_S values and the increase of the H_C values, respectively, caused by the increase of rotation speed. The films displayed soft magnetic properties due to low H_C values. It is also seen that the magnetic easy axis were in the film plane. It can be concluded that the rotation speed of the substrate play a considerable role on the structural and corresponding magnetic properties of the sputtered quaternary FeNiCrCd thin films, and also easily controllable properties of the films can be changed by changing rotation speed for potential electric and electronic applications.

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1. Introduction

In recent years, scientists have investigated different materials due to the quite interesting properties caused in the nanostructure [1-3]. The creation of thin alloys with various combinations of metals makes possible development of new and controllable properties for technological devices such as; magnetic sensors, magnetic recording media, etc. [2-5]. On the other hand, physical techniques have also been developed for the thin alloy film production. Depending on the production parameters and application areas, the techniques have different advantages. Sputtering is one of the important technique to produce such films. With the sputtering method, obtaining thin and high-quality surfaces is possible. Another advantage of this method is that the production parameters eg. rotation speed, deposition rate, etc. [4, 5] which can easily be controlled. The rotation speed is one of the most important deposition parameters, which different speeds changes the properties of the films in the sputtering process [3-6].

The ternary alloys are becoming quite important due to their unique properties [6-9]. The important properties of FeNiCr ternary alloys have been earlier investigated [5-9]. Yu Cao [5] investigated the surface smoothness and magnetic properties as a function of thickness of the FeNiCr thin film. Addition of materials such as S and Co to ternary FeNiCr based films, FeNiCrS [10] and FeNiCrCo [11] were obtained and their investigated properties were reported. On the other hand, Cd alloys are generally preferred for battery technologies and aerospace industry as NiCd [12] and MgCd [13], respectively. By now, to our knowledge, there is no any investigation on the properties of quaternary FeNiCrCd thin films deposited on a flexible substrate by using a single source dc sputtering technique. Therefore, in this study, the influence of rotation speed (0, 15, 30 and 45 rpm) on the structural and related magnetic properties of sputtered quaternary FeNiCrCd thin films was investigated. For that, a series of quaternary FeNiCrCd alloys sputtered on commercial flexible acetate substrates by considering different rotation speeds using a single dc magnetron sputtering system. In the XRD results, the decrease of atomic contents of Fe, Ni and Cr is likely to explain the decrease of the bcc (110) and (200) peaks intensities of the present films with increasing rotation speed. For magnetic analysis, saturation magnetisation decreased with decreasing Fe and Ni contents while the coercivities slightly increased with increasing grain sizes caused by increasing rotation speed. Magnetic easy axis were found to be in the film plane.

2. Experimental

Quaternary FeNiCrCd thin films were deposited on commercial flexible acetate substrates by using single source dc sputtering system (Mantis, Q-Prep 500, U.K.). The source made from a commercial stainless steel was 0.6 mm thick and 50.8 mm in diameter. First of all, the acetate substrate was cleaned with distilled water with an ultrasonic bath before deposition. After the bath, it was dried at room temperature. The distance between substrate and source was ~125 mm in the vacuum chamber. Firstly, for the production of the films, the pressure of the vacuum chamber was 3×10^{-6} mBar before argon gas was dispatched to the vacuum chamber. A pressure value of $\sim4.20\times10^{-3}$ mBar was obtained after argon gas was dispatched and all films were produced under this pressure value. The films were sputtered at room temperature and the temperature of the substrates varied around ±2°C due to irrepressible experimental factor. The thicknesses of the films were measured by using a quartz crystal microbalance thickness monitor (Sycon Instruments, STM 100/MF, USA) which is can provide thickness information with a statistical accuracy of ±0.01 nm during the sputtering process. The thickness of all films was 50 nm. In order to investigate the effect of the different rotation speed of the substrate on the structural and relating magnetic properties of the FeCr Alloy thin films, the rotation speed of substrate was selected as 0, 15, 30 and 45 rpm at deposition rate of 0.09 nm/s since the rotation speed of substrate was limited to 50 rpm.

The atomic compositional analysis of the source was determined by using inductively coupled plasma-atomic emission spectroscopy (ICP-AES, Perkin-Elmer, Optima 7300 DV, USA. The source has the atomic contents of 51.07 % Fe, 13.42 % Ni, 12.67 % Cr and 22.74 % Cd and less than 0.2 % other elements such as Mn, see Table 1. The compositional analysis of the films was done by an energy-dispersive X-ray spectroscopy (EDX; Bruker, Quantax EDS XFlash® 6|30, USA). The films contains totally 0.2 at. % other elements of Mn. The crystal structure of films was examined by using the X-ray diffraction technique (XRD; Bruker, Advance with Davinci Design for XRD², UK). The analysis was performed with Cu-Kα radiation and by scanning the 2θ angle between from 30° to 80° with the step size of 0.005°. The XRD data was also used to calculate the grain sizes, the preferential orientations and the lattice parameters. The magnetic hysteresis curves were obtained by using a vibrating sample magnetometer (VSM; Ade Technologies DMS-EV9, USA) at room conditions between. For avoiding magnetic shape anisotropy, the films were cut in circular shape of 6 mm in diameter in order to avoid shape anisotropy. The hysteresis loops were measured at ±20 kOe with 1 Oe intervals. To determinate the magnetically easy axis, magnetic field parallel and perpendicular to the film plane were applied for each film.

Table 1. Elemental analysis, crystal structure values and magnetic properties of the FeNiCrCd thin films produced at different rotation substrate speeds

Rotation Speed (rpm)	**Elemental analysis**				**Structural parameter**	**Magnetic parameters**	
	Fe (at. %)	Ni (at. %)	Cr (at. %)	Cd (at. %)	t (nm)	M_S (emu/cm^3)	H_C (Oe)
0	26.79	10.69	12.54	49.96	25	780	6
15	24.87	9.38	11.39	54.34	32	687	6
30	24.29	9.25	11.12	55.34	45	531	11
45	22.93	9.12	10.45	57.48	51	332	10
Source	51.07	13.42	12.67	22.74			

3. Results and discussion

The atomic contents of the films by the EDX and source by ICP-AES are presented in Table 1. The source has the atomic contents of 51.07 % Fe, 13.42 % Ni, 12.67 % Cr and 22.74 % Cd and less than 0.2 % other elements such as Mn. As in the table, the EDX analysis revealed that the atomic Fe, Ni and Cr contents decreased from 26.79%, to 22.79%, from 10.69 to 9.12% and from 12.54% to 10.45, respectively while Cd increased from 49.96% to 57.48 8% with increasing rotation speed of the substrate. The reason for the differences of atomic contents between the source and the films may likely to attribute to the relatively different bond energy/melting point of metals have different contents sputtered from source material since this physical parameter is very significant for the sputtering process [14]. And, the most likely reason for the change of film content in each element may come from the variations in their cohesive forces which are likely form a different hold on to surface of the substrate at the various rotating speeds during a sputtering process.

Fig. 1 show the XRD patterns of the films produced at different rotation speeds of the substrate together with the substrate pattern. The crystalline structure have a body-centred cubic (bcc) (110) and (200) planes at $\sim43.8^0$ and $\sim64.3^0$, respectively. The bcc (110) planes may come from Fe [JCPDS-PDF-006-0696], Ni [JCPDS-PDF-004-0850] and Cr [JCPDS-PDF-01-071-4644]. And, the small peak of bcc (200) may be believed to come from Fe [JCPDS-PDF-006-0696] and Cr [JCPDS-PDF-01-071-4644]. Also, the substrate do not have any peak in XRD pattern due to polymer structure. This result consistent with the study [15-16] which investigated the FeCr superlattice. In the figure, the peak intensity of the (110) and (200) planes decreased when the rotation speed decreased from 0 rpm to 45 rpm. The decrease in the peak intensities may be attributed to the decrease in Fe, Ni and Cr contents in the films caused by the increase in the rotation speed. It is also clearly seen that the peak positions of the films slightly shifted which is compatible with Vegard's Law. The preferential orientations of the films were [100] direction since the highest peak intensities belong to bcc (110) plane. The grain sizes, t were calculated by using the full width at half maximum (FWHM) values of (110) planes with Scherer formula [17]. In Table 1, the t values increased from 25 to 51 nm with increasing rotation speed from 0 to 45 rpm, respectively. Further structural parameters of interplanar spacings, $d_{(110)}$ and $d_{(200)}$ for the (110) and (200) planes, respectively, and the lattice constant, a are also obtained. Thus, the $d_{(110)}$ and $d_{(200)}$ values were calculated by using the Bragg formula and are ~0.2067 and ~0.1455 nm, respectively. In the case of lattice parameter, a values were found from the peak position of bcc (110) plane using the cubic equation were found to be around 0.2923 nm. The calculated a values are between with those of the bulk Fe (a_{Fe}=0.2866 nm), Ni (a_{Ni}=0.3524 nm and Cr (a_{Cr}= 0.2885 nm). The contributions of Fe, Ni and Cr to XRD patterns are consistent with a value. From the results, it was shown that different rotating speeds have a considerable effect on the crystalline structures.

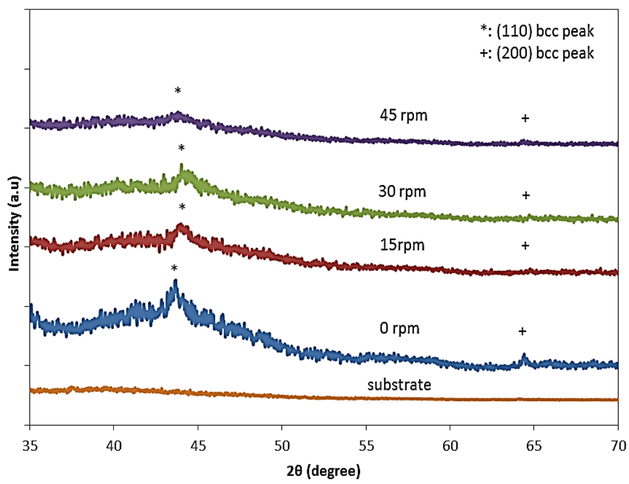

Fig. 1. XRD patterns of the FeNiCrCd thin films sputtered at a) 0, b) 15, c) 30 and d) 45 rpm rotation speed, and e) the substrate

Parallel hysteresis loops of the films sputtered at different deposition rates were plotted at ±200 Oe in Fig. 2. And, the saturation magnetisation, M_S and coercivity, H_C values of the films obtained from the hysteresis loops versus rotation speed were measured at ±20 kOe and presented in Fig. 3 and In Table 1. As in the figures and table, the M_S values decreased from 780 to 332 emu/cm^3 as decrease of atomic Fe and Ni contents caused by the increase of the rotation speed from 0 to 45 rpm. And, the H_C showed slight increase from 6 to 10 Oe as the grain size increased caused by increasing rotation speed, which displayed resulted in the soft magnetic properties since due to ≤12.5 Oe [1].

Furthermore, as an example, parallel and perpendicular loops of the films sputtered at 0 rpm is presented in Fig. 4 to see the magnetic easy axis in the film plane or not. From the figure, it is observed that the easy axis is in the film plane due to the magnetic shape anisotropy of the films. The rest of the films also showed the same result.

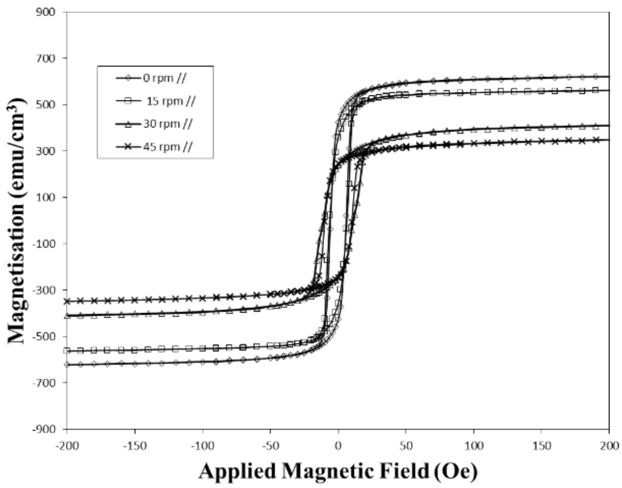

Fig. 2. Hysteresis loops of the FeNiCrCd thin films (measured parallel to the film plane) sputtered at different deposition rates

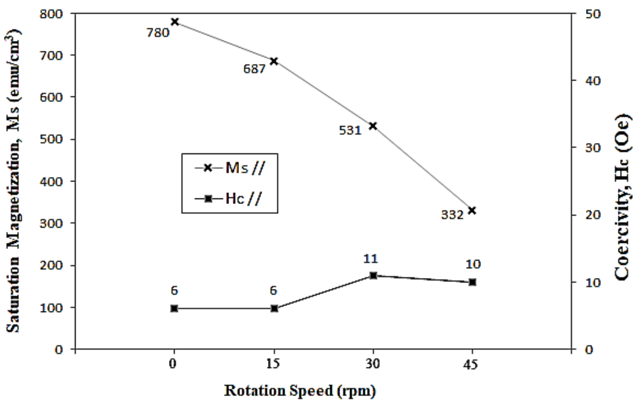

Fig. 3. Saturation Magnetisation, M_S and Coercivity Field, H_C values of the FeNiCrCd thin films (measured parallel to the film plane) sputtered at different deposition rates

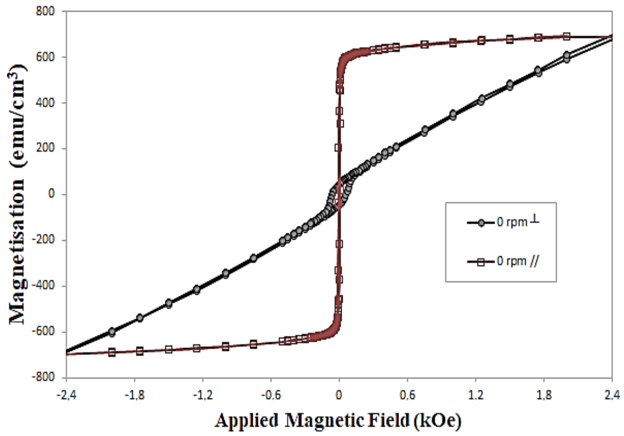

Fig. 4. Parallel (//) hysteresis loop and also perpendicular (⊥) loop of film sputtered at 0 rpm nm/s was plotted at ±15 kOe

4. Conclusions

In this study, the effect of different substrate rotation on the properties of quaternary FeNiCrCd thin films were investigated. As far as concerned, this was the first investigation of properties of these films. Increase rotation speeds resulted in a decrease in Fe, Ni and Cr content of the films with the increase of Cd contents in the films. The structural properties were also affected by the rotation speed. The intensity of the peak belongs to bcc (110) and (200) planes decreased with the decrease of Fe, Ni and Cr contents. Also, increasing rotation speed of the substrate caused an increase in grain sizes. Consequent of the elemental and structural properties, the magnetic properties of quaternary FeNiCrCd thin films were also affected by rotation speed. The saturation magnetisation decreased with decreasing Fe and Ni contents in the films caused by the increase of the rotation speed. And, the coercivity slightly increased with increasing grain sizes of the films by increasing rotation speed. It was concluded that different rotation speed of the substrate play a considerable role on the structural and thus magnetic properties of the sputtered quaternary FeNiCrCd thin films, and the properties of sputtered films are showed to be controlled by changing the rotation speed of the substrate for potential applications.

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