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Synthesis and Characterization of Electrodeposited Fe – Co alloys on Cu substrate

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Abstract : *The paper reports on synthesis and characterization of Fe-Co alloy films. These thin films were deposited on Cu-substrate by using electrodeposition technique. The morphology and crystalline characteristics of films have demonstrated dependence on deposition parameters. The electrolyte temperature has shown to have the most influence on the crystallographic structure of the film. The microstructure and crystallographic texture are studied by varying the substrate pretreatment and deposition parameters. The crystal structure and composition of the thin film samples have been investigated by employing X- ray diffraction (XRD) and X-ray fluorescence (XRF). XRD analyses have revealed the polycrystalline nature of the films, the average crystal size and the crystal orientation. The XRF analyses have revealed uniform composition of the films.*

Keywords: *electrodeposition, Fe-Co binary alloy, BCC structure, thin-films.*

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Introduction :

Electrodeposition is the process of depositing material onto a conducting surface from a solution containing ionic species. This fabrication technique is commonly used to apply thin film of material to the surface of an object to change its external properties such as to increase corrosion protection and to improve decorative quality (Suryanarayana and Koch, 2000). It is a viable, low cost process in synthesizing the nanomaterials. It can be used on metals, alloys, polymers and composites (Gurappa and Binder, 2008). It can also produce coatings on various substrate requiring higher rates (Mohanty, 2011).

Electrodeposition is also suitable for any industrial applications since it can produce deposits for restricted areas such as tiny parts in machines or any equipment (Osaka, 2000). In electrodeposition, the properties of the films can be improved along with their microstructure being controlled by optimizing the operating parameters such as deposition time, pH, bath temperature, current density and electrolytic composition (Ebrahimi and Ahmed, 2003). A diverse range of applications has resulted in the interest for nanoparticles with a wide range of magnetic properties. The study of magnetic properties for softer magnetic materials (Tabakovic et. al., 2002) has gained interest among the researchers due to potential in microelectromechanical system (MEMS) (Myung et. al., 2003).

It is extremely difficult to obtain good grown layers in these films because they are susceptible to the incorporation of stacking faults which form at the cost of very little energy. Such structural defects appear to have a strong influence on the magnetic coupling. The processing technique introduced so far for producing nanocrystalline materials have intended to minimize the problem. Among these techniques,electrodeposition has been recognized as a technologically feasible and economically superior technique for production of nano crystalline materials (Zhou, 2005). The recent investigation on the electrodeposited Fe-Co alloys have shown that their structure depend strongly on experimental parameters such as bath composition, temperature, pH and current density etc. (Baib and Hu, 2005).

In this research work, the synthesis of Fe-Co thin film samples using electrodeposition method on copper substrate was reported. The physical and structural properties of as synthesized Fe-Co deposits were investigated in relation to the different deposition times of 30, 45, 60 and 90 minutes.

Materials and Methods:

Substrate preparation and electrodeposition: The Fe-Co films were electrodeposited on a pretreated Cu substrate that was initially cleaned by acetone and distilled water. The deposition of Fe-Co films (Zhang and Ivey, 2007) were performed using a conventional sulphate bath. The electrolytic solution used in the deposition process was a mixture of $CoSO_4.7H_2O$, $Fe_3SO_4.7H_2O$ and H_3BO_3. These chemicals were properly weighed by using balance and dissolve in distilled water, mixed properly with the help of magnetic stirrer with the base plate speed of 320-360 rpm for 5-10 minutes at temperature range of 75-100°C.The Fe-Co coating was deposited on Cu-substrate while an iron rod was used as anode. The electrodes were suspended in the electrolytic bath with the help of alligator clips. The Fe-Co coatings were formed in 30, 45, 60 and 90 minutes deposition times. These thin film samples were named as FeCo-1, FeCo-2, FeCo-3 and FeCo-4. After the deposition the sample were thoroughly washed with distilled water. The samples were kept in vacuum desiccators to avoid oxidization. All the operating parameters such as temperature, amount of current and deposition time during the electrodeposition process were controlled and maintained. Each experiment was carried out with a freshly prepared solution.

The equipments used to test the characteristics of as-synthesized Fe-Co coatings were RIGAKU miniflex 600 X-Ray diffractometer (XRD) and X-Ray fluorescence (XRF)Analyzer.

Results and Discussion:

X-Ray Diffraction data: X-ray diffraction (XRD) measurements of synthesized Fe-Co coatings were carried out at room temperature (300 K) from 30 degree to 90 degree angle. Fig. 1 shows the XRD patterns of the electrodeposited Fe-Co thin film samples 1 and 2 at different deposition times 30 and 60 minutes. The XRD spectrum for

the Fe-Co phase reveals the characteristics peaks at 2θ angles of 44.84°, 65.56°, 82.85° with planes having Miller indices (110), (200) and (211), respectively. The crystal structure of Fe-Co thin film samples is identified as the BCC crystal structure (Ranjbar et. al., 2006). Observing the XRD spectrum (not shown here) for pure cobalt metal thin films deposited for 30 and 40 minutes, the maximum intensity peaks were observed at 2 angles of around and with planes having Miller indices (110) and (200), respectively. The crystal structure of pure Co metals (Chiriac, 2008) is observed to be hexagonal closed packed (HCP).

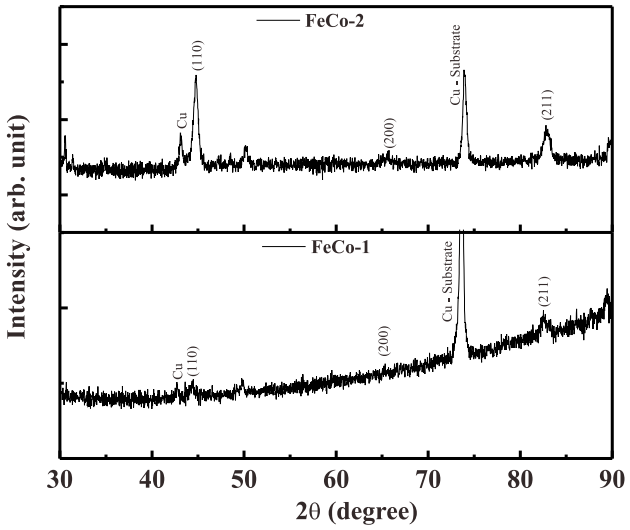

Fig. 1. XRD analysis data of Fe-Co alloy on Cu substrate

Calculations of lattice parameters: For two samples FeCo-1 and FeCo-2, calculations were accomplished for all concerned peaks, using Bragg's law.

For FeCo- 1:

(i) For peak (110) of bcc structure:

By Bragg's law

$$n\lambda = 2d\sin\theta$$

where n=1 and λ = 1.54A° and d is the interplanar spacing (Pillai, 2018).

So here $2\theta = 44.84°$ so $\theta = 22.42°$

$d = n\lambda/2\sin\theta$

d =1.54Å/2sin (22.42)

d =2.02Å

We know that in bcc

$$d = a/(\sqrt{h^2+k^2+l^2})$$

$$a = d \times (\sqrt{h^2+k^2+l^2})$$

where(hkl) are the miller indices.

$$a = 2.02 \times (\sqrt{(1)^2+(1)^2+(0)^2})$$

$a = 2.02Å \times \sqrt{2}$

$a = 2.84Å$

(ii) For peak (200) of bcc structure

By Bragg's law

$$n\lambda = 2d\sin\theta$$

where n = 1 and λ = 1.54Å and d is the interplanar spacing

So here $2\theta = 65.56°$ so $\theta = 32.78°$

$d = n\lambda / 2\sin\theta$

d = 1.54 Å/2sin (32.72)

d = 1.426 Å

We know that in bcc

$$d = \frac{a}{(\sqrt{h^2+k^2+l^2})}$$

$$a = d \times (\sqrt{h^2+k^2+l^2})$$

where (*hkl*) are the miller indices

$a = 1.426 \times \sqrt{(2)^2+(0)^2+(0)^2}$

a = 1.426 Å x 2

a = 2.85 Å

(iii) For peak (211) of bcc structure:

By Bragg's law

$$n\lambda = 2d\sin\theta$$

where n =1 and λ = 1.54Å and *d* is the interplanar spacing

So here $2\theta = 82.85°$ so $\theta = 41.425°$

$d = n\lambda / 2\sin\theta$

d = 1.54 Å/2sin (41.425)

d =1.164Å

We know that in bcc

$$d = a/(\sqrt{h^2+k^2+l^2})$$

$$a = d \times (\sqrt{h^2+k^2+l^2})$$

where (hkl) are the miller indices.

$a = 1.164 \times (\sqrt{(2)^2+(1)^2+(1)^2})$

$a = 1.164Å \times \sqrt{6}$

a = 2.84 Å

For sample Fe-Co 2:

(i) For peak (110) of bcc structure:

By Bragg's law

$$n\lambda = 2d \sin\theta$$

where n = 1 and λ = 1.54Å and d is the interplanar spacing.

So here $2\theta = 44.84^\circ$ so $\theta = 22.42^\circ$

$d = n\lambda/2\sin\theta$

d = 1.54Å/2sin (22.42)

d = 2.02Å

We know that in bcc

$$d = a/(\sqrt{h^2+k^2+l^2})$$

$$a = d \times (\sqrt{h^2+k^2+l^2})$$

where (hkl) are the miller indices

$a = 2.02Å \times \sqrt{(1)^2+(1)^2+(0)^2}$

$a = 2.02Å \times \sqrt{2}$

a = 2.84 Å

(ii) For peak (200) of bcc structure:

By Bragg's law

$$n\lambda = 2d \sin\theta$$

where n = 1 and λ = 1.54Å and d is the interplanar spacing.

So here $2\theta = 65.56^\circ$ so $\theta = 32.78^\circ$

$d = n\lambda / 2\sin\theta$

d = 1.54 Å / 2sin (32.78)

d = 1.426Å

We know that in bcc

$$d = a/(\sqrt{h^2+k^2+l^2})$$

$$a = d \times (\sqrt{h^2+k^2+l^2})$$

where (hkl) are the miller indices(Pillai et. al., 2018).

$a = 1.426 \times \sqrt{(2)^2+(0)^2+(0)^2}$

a = 1.426Å x 2

a = 2.85 Å

(iii) For peak (211) of bcc structure

By Bragg's law

$$n\lambda = 2d \sin\theta$$

where n = 1 and λ = 1.54Å and d is the interplanar spacing.

So here $2\theta = 82.85^\circ$ so $\theta = 41.425^\circ$

$d = n\lambda/2\sin\theta$

d =1.54Å/2sin (41.425°)

d = 1.164Å

We know that in bcc

$$d = a/(\sqrt{h^2+k^2+l^2})$$

$$a = d \times (\sqrt{h^2+k^2+l^2})$$

where (hkl) are the miller indices.

$a = 1.164 \times \sqrt{(2)^2+(1)^2+(1)^2}$

$a = 1.164 Å \times \sqrt{6}$

a = 2.84 Å

From these above calculation, it is clear that the lattice parameters of the samples FeCo-1 and FeCo-2 is a = b = c = 2.84Å.

Crystallite Size:

Crystallite size is the smallest –most likely single crystal in powder form. The crystallite size is commonly determined by X-ray diffraction data. Grain is either a single crystalline or polycrystalline material, and is present in bulk or thin film.

The crystallite size of the thin film samples can be easily calculated from Scherrer formula. The Scherrer formula is given by

$$\tau = \frac{0.9\lambda}{\beta\cos\theta}$$

where, τ is Average crystallite size, β is line broadening in radians, θ is Bragg angle and λ is X-ray wavelength.

$$\tau = k\lambda/\beta\cos\theta$$

(i) For the Bragg peak at $2\theta = 44.78^\circ$

Wavelength of X-ray, $\lambda = 1.54$Å

Line broadening at half of maximum intensity,

$b = 45.04^\circ - 44.51^\circ = 0.53^\circ$

$$= 0.53 \times \frac{3.14}{180} = 0.00924 \text{ radian}$$

Therefore, the crystallite size,

$$\tau = \frac{0.9 \times 1.54\text{Å}}{0.00924 \times \cos(22.39)} = 160\text{ Å}$$

(ii) For the Bragg peak at 2

Wavelength of X-ray, $\lambda = 1.54$Å

Line broadening at half of maximum intensity,

$b = 83.26^\circ - 82.56^\circ = 0.7^\circ$

$$= 0.7 \times \frac{3.14}{180} = 0.01221 \text{ radian}$$

Therefore, the crystallite size,

$$\tau = \frac{0.9 \times 1.54\text{Å}}{0.01221 \times \cos(41.4)} = 151\text{ Å}$$

Thus, the crystallite size of the thin film sample lies in the range 151Å to 160Å (15.1nm to 16nm) (Shi et. al., 2005).

Table 1. Composition of the elements in different thin film samples of electrodeposited Fe-Co alloys on Cu substrate

Fe-Co 1 (1st position)		Fe-Co 1 (2nd position)		Fe Co 1 (3rd position)		Fe Co 1 (4th position)	
Cu	73.03	Cu	73.85	Cu	70.59	Cu	70.97
Co	16.06	Co	15.45	Co	18.46	Co	18.28
Fe	10.09	Fe	10.68	Fe	10.95	Fe	10.75

Fe-Co 2 (1st position)		Fe-Co 2 (2nd position)		Fe Co 2 (3rd position)		Fe Co 2 (4th position)	
Cu	93.21	Cu	92.66	Cu	80.15	Cu	79.01
Co	3.97	Co	4.22	Co	11.42	Co	12.11
Fe	2.82	Fe	3.12	Fe	8.43	Fe	8.88

Fe-Co 3 (1st position)		Fe-Co 3 (2nd position)		Fe Co 3 (3rd position)		Fe Co 3 (4th position)	
Cu	84.52	Cu	87.06	Cu	84.80	Cu	81.92
Co	8.43	Co	7.20	Co	8.68	Co	7.99
Fe	7.05	Fe	5.74	Fe	6.52	Fe	6.14

Fe-Co 4 (1st position)		Fe-Co 4 (2nd position)		Fe Co 4 (3rd position)		Fe Co 4 (4th position)	
Cu	65.44	Cu	70.71	Cu	82.87	Cu	84.35
Co	17.06	Co	14.34	Co	7.60	Co	6.96
Fe	14.31	Fe	12.11	Fe	6.19	Fe	5.62

X-ray fluorescence Data:

X-ray fluorescence (XRF) is an analytical method to determine the chemical composition of all kinds of materials. The materials can be in solid, liquid, powder; filtered or other form. XRF can also sometimes be used to determine the thickness

and compositions of layer and coating. The method is fast accurate and non-destructive and usually requires only a minimum of sample preparation. The XRF measurements have been performed at four different positions of thin films samples. Table 1 shows the composition of the thin film samples.

From the Table 1 it is confirmed that these samples have uniform crystal composition at the different positions.

Conclusions:

Thin films of Fe-Co binary alloys have been deposited on Cu-substrate by electrodeposition technique. The crystal structure of the thin films samples shows the Bragg peaks of BCC structure. The calculated lattice parameters of these polycrystalline thin films are 2.84Å. The average grain size calculated for all Fe-Co coatings lies in the range 15.1*nm* to 16*nm*. The XRF measurements have been performed at different positions of thin films samples. The XRF data of these samples shows the uniform crystal composition in these thin film samples. It may be concluded that the process parameters such as temperature and pH has an influence on the preferred orientation of planes and the size of the nano crystals or grains of electrodeposited.

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