PERIODICO di MINERALOGIA
ISSN: 0369-8963

Volume 91, No. 4, 2022

Synthesis and Characterisation of Co-Fe-W-P Thin Films Electrodeposited Under Phosphorous Acid and Urea Electrolyte Solution

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Abstract

Cobalt, iron, and tungsten-primarily based (Co-Fe-W) thin films and their by-product thin films have vast applications in microelectronics and micro-electro-mechanical systems due to their superior smooth magnetic residences. With the blessings of simplicity, cost-effectiveness, and controllable patterning, electroplating approaches have been hired for CoFeW thin films fabrication. The electrodeposition of CoFeW and phosphorus-enabled CoFeW thin films were completed from the phosphorus solvent bath without components specifically urea. The results of bath, electrolyte attention, applied current density, and solution agitation on deposition charge has been studied. Thin films composition, morphology, crystallographic structure as well as magnetic moment, and coercivity have been characterized and as compared in opposition to the deposition conditions such as with and without components and deposition current densities. An accelerated awareness of the bath solution results in significantly quicker deposition and promotes clean thin films increase with confined cracking. As-deposited CoFeWP alloy films are observed to be ferromagnetic and nanocrystalline. In our observations, we located that electrolyte situations and carried out cutting-edge values affect the magnetism and susceptibility of thin films. Copper substrates had been used as templates for magnetic thin films. X-ray diffraction studies were conducted to research the structural traits of thin films. The crystal size and structure of the electrodeposited magnetic thin films fluctuate slightly primarily based on the composition ratio described inside the literature. As a result of assessment with thin films, their magnetic susceptibility shifted in the direction of the lower facet with increasing electrolyte concentration and current density value.

Keywords: CoFeWP thin film; Electrodeposition; Mechanical properties; Magnetic properties

https://doi.org/10.37896/pd91.4/91464

Introduction

Nanostructured coatings made of metals and composites (grain sizes lower than 100 nm) have noticeable physical and substance properties. They are utilized in various applications in light of their exceptional properties, for example, optical, electrical, attractive, synergist, natural, or mechanical [1-4]. These materials have been delivered in numerous ways and electrodeposition is perhaps the best strategies which are a lot less complex and more efficient technique. Moreover, coatings that are delivered by this method needn't bother with any vacuum gear and optional handling steps. It is deeply grounded that the twofold compounds of temporary metals present helpful elements like high strength, hardness, erosion opposition, and highly attractive properties [4-6]. Nanostructured Thin films of CoFeW and its subsidiaries (CoFeWM), (which M is separately for different metal or non-metal ions), have enormous attractive penetrability at high frequencies, high electrical resistivity, mechanical durability, and substance strength, and are somewhat reasonable. In this manner, they have been broadly utilized in the scope of electronic gadgets, for example, TVs, transformers, microwaves coordinated with nonreciprocal circuits, memory gadgets, radio recurrence loops, bar receiving wires, and read-compose sets out toward rapid computerized tape or plate recording, and broadcast communications applications [6-10]. Besides that, CoFeW are fundamentally utilized in catalysis and attractive fields.

To dispose of surpassing power utilization and adverse impacts coming about because of warming of the components, high immersion charge (Ms) and low coercivity (Hc) are expected for certain applications referenced previously. Among the attractive amalgams, Co-Fe-W has the most elevated immersion charge and might be chosen as a decent possibility for attractive recording heads. Besides, these combinations don't have a low coercive field and scientists have found various techniques for decreasing this boundary [10-13]. For this reason, the expansion of another alloying component (either metal or non-metals) and additionally diminishing the grain size can be valuable techniques to conquer this issue. It is accounted for that there are a few issues during the electrodeposition of combination coatings containing iron components. Because of the flimsiness of Fe^{2+} in fluid arrangements, ferrous particles are handily oxidized and their dissolvability diminishes quickly by expanding pH esteem. This prompts hydroxide joining in the kept films and causes film embrittlement and attractive second decrease. To make great Fe-containing composite coatings; the expansion of a decreasing specialist and the utilization of a latent environment is reasonable. The impacts of pH, shower organization, and ebb and flow thickness on the attractive properties of various compounds were likewise analysed by different analysts. It is accounted for that appropriate attractive properties as well as great mechanical ways of behaving are expected for attractive recording frameworks [14-17]. In this way, the mechanical and attractive highlights of Co-Fe-W amalgams ought to be altered by the expansion of alloying components [13]. As referenced, the electrodeposited Co-Fe-W and Co-Fe-W-P amalgams are appropriate contender to accomplish this reason.

In addition to offering fine composition control, the preparation of thin films by physical or metallurgical methods is costly. Several synthesis methods have been extensively investigated in recent years to achieve high homogeneity in magnetic thin films, including spray pyrolysis, reverse micelles, and cathodic electrophoretic deposition [12]. Yet, the electrodeposition process has some interesting applications when it comes to the synthesis of magnetic films and powders. As part of our previous work, it was used in the preparation of magnetic thin films composed of CoFeW. In comparison with other methods, the process offers the following advantages: low temperatures, low cost of starting materials and equipment, control over the microstructure, composition, and morphology of the film, as well as the ability to deposit a film on complex surfaces [18].

In the present study, a CoFeWP magnetic thin film is prepared by electrodeposition. Copper substrates were coated with magnetic thin films formed by cobalt sulphate, ferrous sulphate, sodium tungstate, phosphoric acid and urea. These salts were examined in terms of their physicochemical properties. In order to evaluate the structural, morphological and magnetic properties of the thin films various characterisation techniques were used.

Materials used

Copper substrate, Sulphuric acid, acetone, cobalt sulphate, ferrous sulphate, sodium tungstate, Phosphoric acid, urea and distilled water were used for preparation of CoFeWP thin films.

Thin film preparation

Using galvanostatic electrodeposition, the copper substrate (1.5 x 5 cm) acts as the cathode and the treated steel plate as the anode. During electrodeposition, the current was drawn from DC-controlled power supplies. In order to set up the shower arrangement, it was necessary to use synthetic compounds of a higher grade. A concentrated solution of H_2SO_4 and C_3H_6O was used to clean the substrates. Ultrasonic cleaning was followed by flushing the substrates with refined water before electrodeposition. Different current densities and electrodeposition times were used for electrodeposition. An attractive slender Co-Fe-W-P film was electrodeposited from a shower containing 0.1M cobalt sulphate, 0.1M ferrous sulphate, and 0.05 M sodium tungstate. The primary electrolyte was phosphoric acid (0.1 and 0.2 M), and urea (2.5, 5 and 7.5 g/l) was used as an electrolyte additive. An analysis of the influence of cobalt, iron, and tungsten content on pre-arranged thin films was performed at varying current thicknesses (2.5, 5, and 7.5 mA cm^{-2}) for 45 mins. When electrodeposition is taking place, a persistent pH value of 8.0 is preserved.

Characterisation techniques

The phase, structure and morphology of the magnetic films were concentrated on utilizing an X-ray diffractometer (Rich Seifert, model 3000) and scanning electron microscope (JEOL) individually. The structural parameters of the stored Co-Fe-W-P film have been determined from the XRD information utilizing the accompanying Debye Scherer equation [19-20].

$$\boldsymbol{D = \frac{k\lambda}{\beta cos\theta}}$$

$$\textbf{Strain } (\boldsymbol{\varepsilon}) = \frac{(\boldsymbol{\beta}\mathbf{cos}\boldsymbol{\theta})}{\mathbf{4}}$$

$$\textbf{Dislocation density } (\boldsymbol{\delta}) = \frac{\mathbf{1}}{\mathbf{D^2}}$$

Where, ‘D’ is average crystallite size which was taken for three main predominant peaks.

EDAX spectra were used to obtain the compositional appropriations in pre-arranged magnetic thin films. An electronic micrometer was used to determine the thickness of the thin film (Mitutoyo, Japan). A vibrating magnetometer was used to measure the magnetic properties of the thin films. Vickers hardness analyser is utilized to decide the hardness of the pre-organized thin films. Accompanied with the aid of, the grip of the thin movie became attempted via the curve and scratch procedure.

Results and Discussion

Phase and structural analysis

Figure 1 show XRD patterns of 0.1 and 0.2 M Phosphoric acid with 2.5 and 5 g/l of urea added as an additive electrolyte to the prepared films electrodeposited for 45 minutes at 7.5 mA cm^{-2} current density. The prepared magnetic thin films were observed cubic crystalline structures by XRD. Based on XRD pattern analysis, the data was perfectly matched with the standard JCPDS card number 65-7519. The XRD pattern showed FCC phase with hkl values (110), (111) and (210) approximately at 45°, 50.7° and 74.5° diffraction angles for all the thin films [21-22].

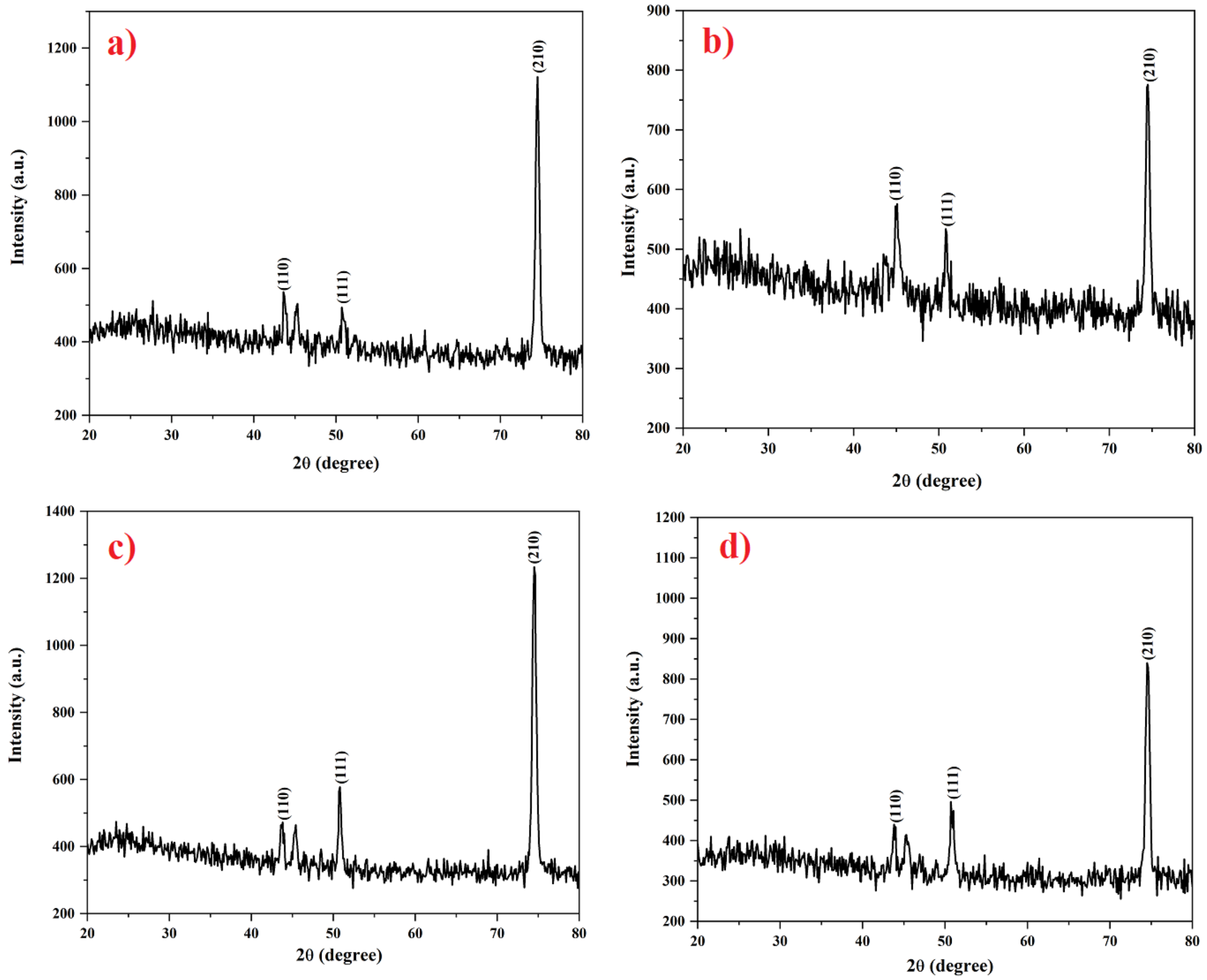

Figure 1 XRD pattern of Co-Fe-W-P films electrodeposited for 45 minutes at deposition time at 7.5 mA cm^{-2} current density with 0.1 M of H_3Po_3 (a) 2.5 g1^{-1} urea (b) 5 g1^{-1} urea and with 0.2 M of H_3Po_3 (c) 2.5 gl^{-1} urea (d) 5 g1^{-1} urea

But when urea is present, the lattice constant of those nanocrystalline films lowers, and all of the peaks move to better diffraction angles. This may be due to the Nitrogen atom in CoFeWP magnetic thin films occupying an online Fe or Co domain. The calculated crystal size of the prepared thin films is shown in table 1. The crystal size of the prepared thin films was gradually decreased with respect to increase in electrolyte and additives concentrations. The calculated crystal size was ranges from 17.014 nm to 13.388 nm. The change in rate of urea additive and current density applied plays a crucial role in the crystal size in nano scale range. When the current density increases with respect to concentration of urea, both strain value increase to 24.542 x 10^{-4} from 21.390 x 10^{-4} and 27.187 x 10^{-4} from 21.197 x 10^{-4} respectively for 0.1M and 0.2M of phosphoric acid and also the change in dislocation density also observed. The deliberated crystal size, strain value and dislocation density of magnetic thin films are given in Table 1. It is possible to use these nanostructured thin films for magnetic recording devices and online storage applications because the crystallite sizes for all electrodeposited thin films are larger than the saturation magnetization limit of Co-Fe based thin films (~3 nm) [14, 15, 19].

Morphological analysis

Figures 2 (a, c, e, and g) and 2 (b, d, f, and h) illustrate the SEM pictures and EDAX patterns of the prepared nanocrystalline thin films acquired from exceptional current densities for each electrolytes. It's far clean that the electrodeposited thin films procured from reasonable current densities have perfect and uniform surfaces while at decline current densities the lower has not many miniature breaks. The arrangement of miniature breaks is because of the inner pressure inside the coatings that the subbed bigger iotas inside the cross section or due to hydrogen development during the affidavit cycle. The breaks additionally can be designed through interfacial mechanical coupling between the slight thin films and substrate, that is extended via the film thickness or bringing down the grain size.

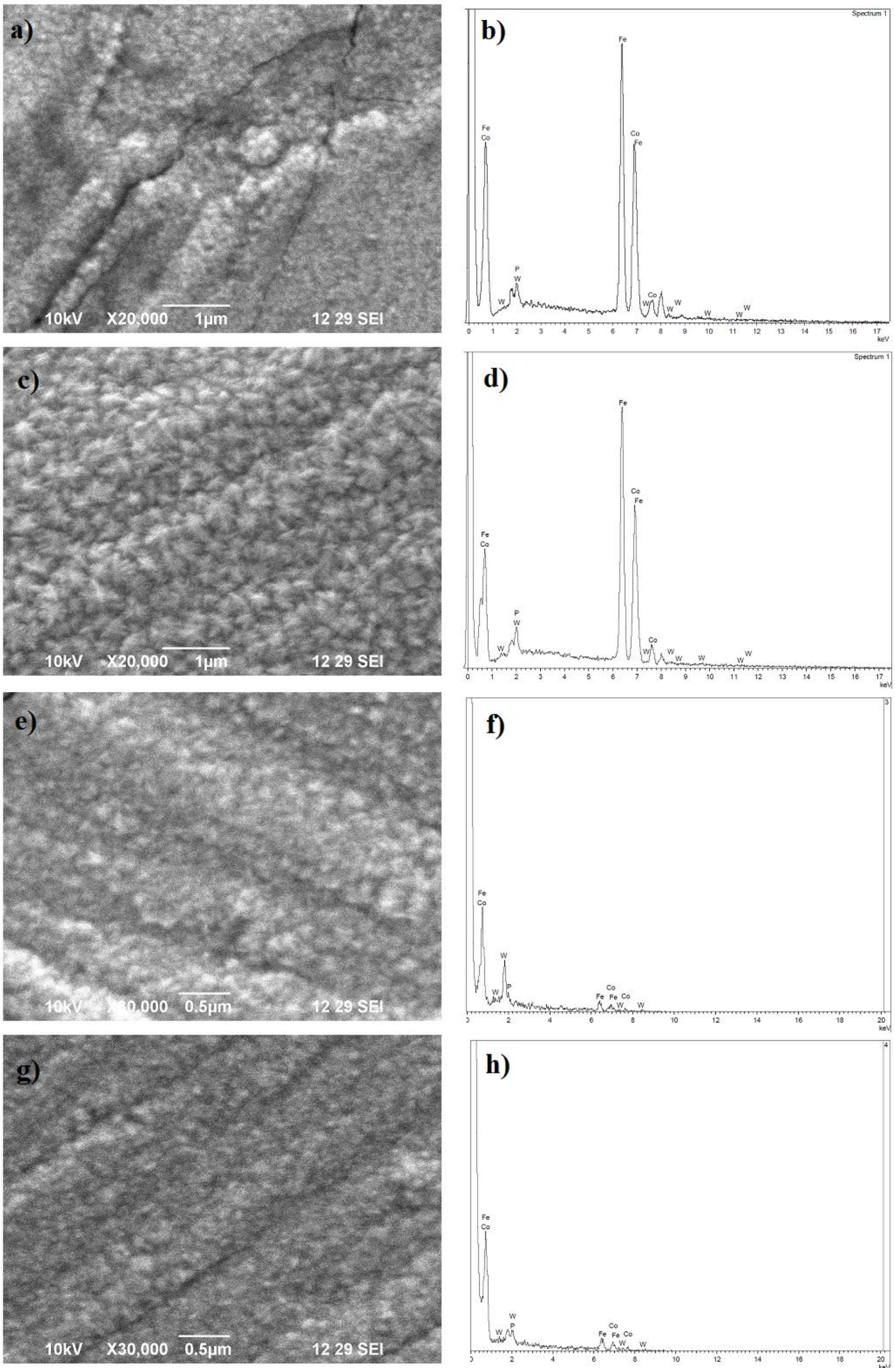

Figure 2 SEM images and EDAX pattern of Co-Fe-W-P nanocrystalline magnetic thin films by 45 min of deposition time at 7.5mA cm^{-2} current density with 0.1 M of H_3Po_3 (a and b) 2.5 g 1^{-1} urea (c and d) 5 g 1^{-1} urea and with 0.2 M of H_3Po_3 (e and f) 2.5 g 1^{-1} urea (g and h) 5 g 1^{-1} urea

Consistent with the present results, by increasing modern density crystal length and surface smoothness extended. Therefore, hydrogen evolution might be the primary issue causing micro-cracks formation in the deposits at lower modern-day densities [23].

Mechanical Properties

The Vickers hardness tester was used to identify the hardness number of nanocrystalline magnetic thin films made of nanocrystalline Co-Fe-W-P. Based on 0, 2.5, and 5 g/l of urea concentrations, 169, 177, and 189 hardness values were calculated for 0.1 M phosphorous concentration, 171, 183, and 194 for 0.2 M concentration of phosphorous concentration. Therefore, the micro hardness increases as the amount of phosphorus and urea in the electrolyte increases as thin films are less stressed and molecular strength is supported by nitrogen and phosphorus ions. Several tests were conducted to ensure that the film was adhering well. It should be noticed that under a chose incredible crystal and grain length, reverse hall-Petch influence becomes glaring. This conditioning can be because of the monster expansion in buried crystalline quantity fraction, triple intersection and diffusional creep peculiarity [24-25]. The micro hardness of the coatings got is progressively advanced from 169 to 195 HV, which can be a direct result of grain length refinement. The Vickers hardness values of the prepared thin films with respect to electrolyte with its additives and applied current densities are given in table 1.

Thickness Study

Based on a ramification of experimental conditions, table 2 summarizes the impact of different concentrations of phosphorous acid and urea on the thickness and magnetic properties of Co-Fe-W-P nanocrystalline thin films. Increasing the responsiveness of urea and phosphorous, along with growing the density of the modern-day, regularly extended the film thickness. Consistent with the published reports, increasing the thickness of the film is more advantageous their magnetic properties. The thickness of the thin film on base plate became

https://doi.org/10.37896/pd91.4/91464

calculated as 4.2 μm 0.1 M of phosphorous and 5g/l of urea and 6.6 μm of thickness was measured for 0.2 M of phosphorous and 5g/l of urea concentrations at constant 7.5 mA current density. The measured thickness values of the thin films sincerely imply the electrolyte solution plays a dominant position in thickness of the film.

Magnetic properties

The figure 3 show the VSM data of nanocrystalline thin films deposited under 0.1 and 0.2 M phosphoric acid concentrations with 2.5 and 5 g/l of urea concentrations respectively. It is evident that saturation magnetizations are inversely proportional to urea content, a nonmagnetic property. In 0.1M of phosphorus acid electrolytes with urea added, the magnetic saturation increases from 0.078 to 0.124 emu with an increase in current density from 2.5 to 7.5 mA cm^{-2}. As shown in Table 3, the coercivity increased from 151.19 to 162.51Oe when the current density was increased from 2.5 to 7.5 mA cm^{-2}. It was interesting to note that the coercivity of 165.40 Oe decreased from 158.48 Oe when the current density was increased from 2.5 to 7.5 mA cm^{-2} under 0.2 M phosphorus acid. A high magnetization value is observed in films obtained at higher current density, while the coercivity value is low. Magnetic hysteresis loop studies reveal the films have an easy magnetic direction alignment along the in-plane due to their high saturation and low coercivity. Based on surface and tiny magnetization studies, the deposition potential affects the film's roughness and the growth of magnetic domains [26-27]. Alloy coatings have enhanced saturation magnetization and reduced coercivity values, which enhance their magnetic properties. At current densities higher than 7.5 mA, the cobalt, iron, phosphorus, and tungsten contents of thin films obtained from 0.1 M H_3PO_3 electrolyte may be the cause of magnetization changes. However, coercivity is an extrinsic characteristic that is determined by many factors other than the composition of thin films. Coercivity can be influenced by grain length, microstructure, texture, inner strain, and chemical composition [28]. The coercivity of the cloth with the bcc segment appears higher than that of the fcc structure and the hcp structure. One of the most crucial factors that may affect a coercive subject is the grain length, as previously mentioned.

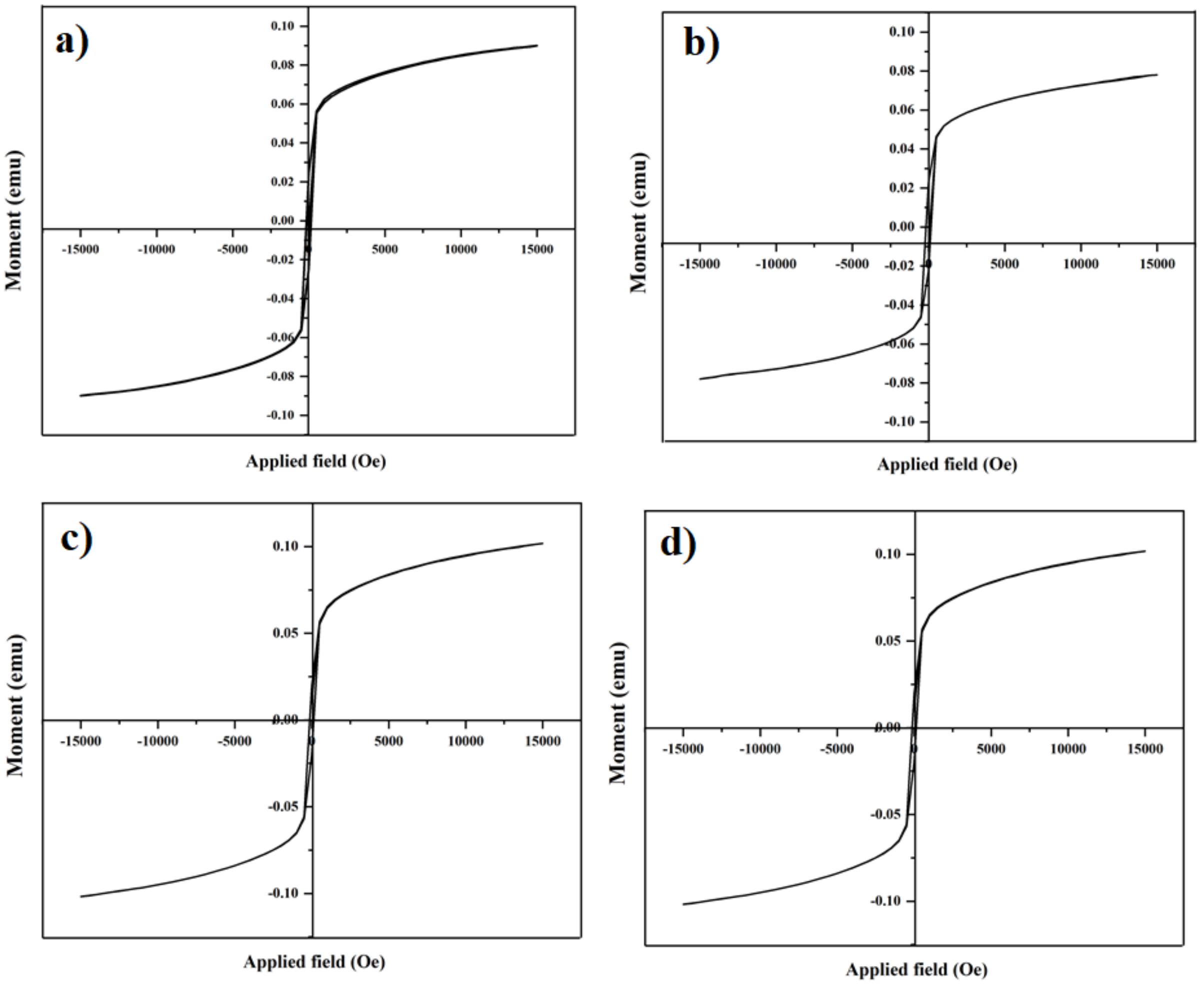

Figure 3 VSM images of Co-Fe-W-P films electrodeposited for 45 minutes at deposition time at 7.5mA cm^{-2} current density with 0.1 M of H_3Po_3 (a) 2.5 g 1^{-1} urea (b) 5 g 1^{-1} urea and with 0.2 M of H_3Po_3 (c) 2.5 g 1^{-1} urea (d) 5 g 1^{-1} urea

According to some authors, a lower Hc value is caused by a lower value of this parameter, while other researchers hold opposing views. There is a possibility of area wall pining due to grain limitations at grain sizes above magnetic exchange duration (Lex). By increasing current density, the film stress is reduced. Because low-stress formation occurs during deposition of thin films, they have lower coercivity and higher magnetization. It follows that a higher current density enhances the prepared thin films soft magnetic properties. For all thin films, the coercivity was less than 200 Oe, resulting in soft magnetic behaviour [12, 13, 22, 23].

Conclusion

In these examinations, the possessions of current density and electrodeposition synthesis on the design and attractive properties of thin films had been researched. At lower current densities, the thin films affirmed a solitary stage stable response with uniform surface morphology, even as through developing this boundary a two-segment shape with an excessive number of miniature breaks transformed into found. Through developing cutting edge thickness, nucleation charge overwhelms grain blast and as an outcome, lower grain length is expected. Be that as it may, miniature hardness of the magnetic thin films kept from low urea content added substance electrolyte changed with the guide of compound synthesis of the pre-arranged magnetic thin films even as this boundary adjusted through grain size decrease for the coatings stored from better urea content material electrolyte. The coercivity was transformed into constrained by grain size markdown and surface morphology. As a rule, the higher urea content electrolyte delivered coatings with cutting edge magnetics conduct and miniature hardness values when contrasted with decline urea content as added substance electrolyte.

Table 1 Crystalline size, strain, dislocation density, and hardness and elemental composition of Co-Fe-W-P films at 7.5 mA cm^{-2} current density for 45 mins deposition time

H_3PO_3 (M)	Urea (g/l)	Crystalline size(nm)	Strain (10^{-4})	Dislocation Density (10^{14} / m^2)	Vickers Hardness Number (VHN)	Film Composition (wt. %)			
						Co	Fe	W	P
0.1	0	17.014	21.390	34.545	169	36.66	58.08	1.64	3.62
	2.5	14.956	24.334	44.706	177	38.63	56.91	2.29	2.17
	5	14.829	24.542	45.475	189	42.32	47.42	9.17	1.08
0.2	0	17.169	21.197	33.924	171	37.31	56.80	1.33	4.56
	2.5	16.908	21.524	34.979	183	39.45	55.46	0.87	4.22
	5	13.388	27.184	55.791	195	40.20	55.14	1.16	3.51

Table 2 Effect of the thickness and magnetic properties of Co-Fe-W-P films electrodeposited for different current densities

Bath additive		Current density (mAcm^2)	Thickness of film (µm)	Magnetic saturation (emu)	Remanent polarization (emu)	Coercivity (Oe)	Squareness
Phosphorous (M)	Urea (g/l)						
0.1	0	2.5	3.7	0.078	0.020	151.19	0.256
		5	3.9	0.061	0.016	156.30	0.262
		7.5	4.2	0.101	0.019	124.18	0.1897
	2.5	2.5	4.0	0.125	0.031	148.49	0.247
		5	4.2	0.133	0.033	142.30	0.248
		7.5	4.4	0.090	0.024	138.16	0.266
	5	2.5	4.3	0.060	0.024	197.39	0.40
		5	4.4	0.078	0.023	159.18	0.2948
		7.5	4.7	0.124	0.035	162.51	0.2821
0.2	0	2.5	4.2	0.064	0.017	165.40	0.265
		5	4.5	0.078	0.020	157.18	0.256
		7.5	4.8	0.079	0.017	161.32	0.215
	2.5	2.5	5.2	0.090	0.022	143.20	0.244
		5	5.4	0.060	0.022	197.59	0.366
		7.5	5.8	0.1018	0.020	139.24	0.1964
	5	2.5	6.1	0.078	0.020	158.81	0.2564
		5	6.3	0.063	0.018	163.51	0.2857
		7.5	6.6	0.078	0.019	158.48	0.2435

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