

Influence of Phosphorus Content and Magnetic Annealing on Soft Magnetic Properties of Electrodeposited Amorphous FeMnP Alloy Films

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Abstract

In this work, we investigated the influence of phosphorus and magnetic anneal on the soft magnetic properties of electrodeposited FeMnP alloy films prepared by changing sodium hypophosphite concentrations. X-ray diffraction radiation patterns showed an amorphous structure of electrodeposited alloy films. The saturation magnetization and coercivity value decreased from 586 emu/cc to 346 emu/cc, and 52 Oe to 18 Oe, with the P content increased, respectively. The absorption resonance peak became broad as the P content increased, and the natural resonance frequency decreased from 1.8 GHz to 0.6 GHz, with the P content increasing. Magnetic annealing of samples reduced the magnetic damping, and natural resonance frequency increased by about 1.8 GHz and 3.5 GHz for the sample with lower and higher P content. The film structure with lower P content changed at 300°C, while the structure remains unchanged for the films with higher P content. Thus, the crystallization temperature could depend on the P content in the film. FeMnP alloy films could be used in high-frequency devices.

Keywords

Phosphorus, Magnetic Annealing, Soft Magnetic Property, Natural Resonance Frequency

1. Introduction

Soft magnetic thin films have been widely used for low-frequency devices such as transformers, generators, motors, and high-frequency applications. Recently,

the research focused on soft magnetic thin films operating on high frequency in order to miniaturize and integrate microelectronic devices [1]. The designer and developers of modern electronic devices seek small-size, lightweight, high-speed, and cost-effective devices. High anisotropy field, saturation magnetization, and high resistivity are prerequisites properties for those soft magnetic thin films [2] [3]. Fe-metalloid amorphous alloy demonstrated excellent soft magnetic such as high permeability, low coercivity, and lower core loss, thereby lacking grain boundary in the amorphous phase and magnetocrystalline anisotropy field [4] [5]. For instance, Fe-Si-B has been widely used in high-frequency inductors power transformers owing to the high saturation magnetization, high permeability, and low core loss. Metalloids are indispensable elements that significantly affect the properties of Fe-based amorphous alloys [6] [7].

Nevertheless, the impact of metalloids on magnetic properties is still controversial [8] [9]. The metalloid can improve some properties and degrade other properties; for example, P content could decrease coercivity, saturation magnetic flux density (Bs), and increase the initial permeability as found in FeSiCuB alloy [10]. B and Si enhance glass-forming ability, thermal stability, coercivity, and initial permeability; however, the saturation magnetic flux Bs are lower than silicon-based steel alloy [11] [12]. In order to increase the saturated magnetic flux density of Fe-based metalloid, the transition metals are added (Co, Ni) [13] in the alloy. However, further research found that the transition metals increases in the alloy could suppress the amorphous forming ability. Therefore, Bs, amorphous forming ability, and cost challenge Fe-metalloid amorphous alloys. In order to meet high-frequency properties with highly saturated magnetization, heat treatment is used to reduce internal stress and induce a uniaxial magnetic anisotropy field. Fe-based amorphous were produced by melt quenching [14], condensation, and electrodeposition techniques [15] [16]. Electrodeposition was extensively focused on cost-effectiveness, environment friendliness, and simplicity [17] [18].

Furthermore, electrodeposited alloys have different shapes and geometries. They can easily change the composition with the broad range by changing deposition parameters and having high resistivity [19]. Bassey [20] studied the corrosion properties of electrodeposited FeMnP alloy and revealed the presence of an amorphous structure. Bassey requested further studies about the magnetic properties of FeMnP alloy. The magnetic properties of FeMnP thin film with an equal ratio synthesized via metal-organic chemical vapor deposition were discussed, and the authors revealed the paramagnetic phase of FeMnP Thin-film [21]. However, the soft magnetic properties of electrodeposited FeMnP amorphous alloys have not yet been investigated. Adequate research about the magnetic properties of FeMnP amorphous alloy is still needed to clarify the specific applications of FeMnP amorphous alloy films. Being Fe-based metalloid alloy, FeMnP could have special soft magnetic properties that could have potential in use at high-frequency applications. Thus, the present work is aimed to investigate the influence of P and magnetic heat treatment on the soft

magnetic properties of FeMnP amorphous thin film through the electrodeposition method. The effect of sodium hypophosphite on static and dynamic properties was discussed. The coercivity force and saturation magnetization decreased with P increased. Resonance linewidth and natural resonance frequency decreased with phosphorous. Resonance linewidth reduced with annealing temperature increased. In contrast, natural resonance frequency increased. The uniaxial anisotropy did not induce, and the films exhibit isotropic properties.

2. Experimental Procedures

The 1.5 μm -thick FeMnP alloy films were deposited on copper sheet substrate from the sulfate-based electrolyte. $\text{MnSO}_4 \cdot \text{H}_2\text{O}$, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, $(\text{NH}_4)_2\text{SO}_4$, and NaH_2PO_2 using an electrochemical workstation (CHI860D) with three-electrode configurations: cathode (copper), saturated calomel electrode (SCE), carbon electrode (anode). A static external magnetic field with 800 Oe was applied parallel to the cathode electrode to induce uniaxial anisotropy. The bath composition and electrodeposition parameters are listed in **Table 1**. The copper sheet substrates were cleaned using acetone, ethanol, and distilled water. NaOH was used to change the pH value from 4.8 to 6. Magnetic properties have been enhanced by annealing under 300 Oe magnetic field for half an hour from 100°C - 400°C with a heating rate of 20°C/min. The base pressure was 2.3×10^{-3} Pa. The samples were cooled at room temperature by the furnace itself. The structure of FeMnP alloys was investigated by using X-ray diffraction using $\text{CuK}\alpha$ radiation (XRD). A vibrating sample magnetometer (VSM) and vector network analyzer (VNA, Agilent E8363B, USA) were used to study the magnetic properties of FeMnP alloys. A scanning electron microscopy (SEM) equipped with energy-dispersive x-ray spectroscopy (EDX) was used to determine the film composition and cross-section.

Table 1. Electrochemical deposition parameters.

Bath composition and conditions	Concentration
$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	30 g/L
$\text{MnSO}_4 \cdot \text{H}_2\text{O}$	60 g/L
NaH_2PO_2	5 - 30 g/L
$(\text{NH}_4)_2\text{SO}_4$	200 g/L
pH value	6
Deposition potential	-1.5 V vs SCE
Bath temperature	20°C
Current density	0.03 A/cm ²
Deposition time	180 s

3. Results and Discussion

3.1. Structural Analysis

Figure 1(a) displays the film deposited at 5 g/L of NaH_2PO_2 and annealed from 100°C - 400°C. The deposited film shows an amorphous structure due to the incorporation of phosphorus. Electrodeposited FeMn alloy showed an amorphous structure [22]. The incorporation of P enhances the amorphous structure [23]. The present results consent with Bassey's results [20]. After heating the sample from 100°C - 400°C no apparent change was observed in the structure. However, at 300°C - 400°C, the sample shows a crystalline α -Fe based peak with structure orientation (110). It means that from 300°C, the film has been crystallized due to the low diffusion of P [24]. The reduction of P diffusion promotes grain growth. Thus the structure turned from amorphous to nanocrystalline.

Figure 1(b) shows the film deposited at 30 g/L of NaH_2PO_2 and annealed at different temperatures. The deposited and annealing film shows a similar structure (amorphous) for all annealed temperatures. It could be concluded that the enhanced diffusion of phosphorus prevents grain growth during the annealing. Thus the grain growth was controlled by the diffusion of phosphorus in the films [24].

3.2. Compositional Analysis

Table 2 displays the EDX results of electrodeposited films from the different

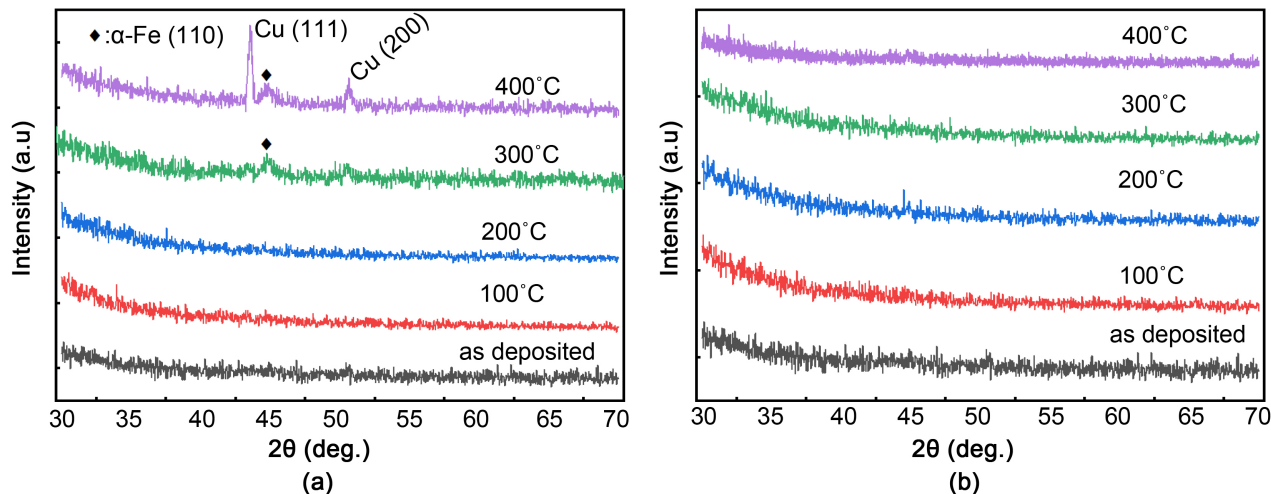


Figure 1. XRD patterns of annealed samples (a) 5 g/L, (b) 30 g/L.

Table 2. Composition of FeMnP films determined by EDX.

g/L	NaH ₂ PO ₂ Atomic. %		
	Fe	Mn	P
5	87.22	2.26	10.57
10	83.79	2.29	13.25
30	75.25	0.61	24.14

NaH_2PO_2 concentrations. The P content increased as the NaH_2PO_2 concentration increased. The Mn and Fe content decreased with the NaH_2PO_2 increases. The EDX mapping and film thickness are mentioned in the supplement file.

4. Static Magnetic Properties of Electrodeposited FeMnP Films

4.1. Effect of Sodium Hypophosphite on Static Magnetic Properties

Hysteresis loops of electrodeposited FeMnP alloy films were measured in parallel and perpendicular to the direction of applied static magnetic films during deposition. They showed that all deposited films are isotropy (see a supplementary document). We considered parallel directions to depict the influence of P on static magnetic properties of electrodeposited FeMnP alloy films.

Figure 2(a) displays the hysteresis loop of electrodeposited FeMnP films at different NaH_2PO_2 concentrations. It revealed that the saturation magnetization decreased from 586 emu/cc to 346 emu/cc with the sodium hypophosphite. Magnetization is an intrinsic property that depends on the composition and thickness of the films [25]. The distribution of non-magnetic and magnetic elements plays a key role in controlling saturation magnetization, and the high net magnetic moment is obtained for the magnetic-magnetic interaction. In contrast, non-magnetic-non-magnetic interactions produce low magnetic moments [26] [27]. This work revealed that saturation magnetization is strongly dependent on the magnetic moment. The magnetic moment is proportional to the iron content in the FeMnP alloy films. P content in the films increased with the NaH_2PO_2 increases. It reduces the iron content and increases the ratio of manganese and phosphorus in the FeMnP films. The magnetic moment decreased as the P increased due to the reduction of d-orbit electrons. Thus, the magnetic moment of Fe-based amorphous decreased with an increase in metalloid content [28]. That could be explained by atomic interactions and s-d hybridization between Fe and P elements. The incorporation of P decreases the d-orbit electrons,

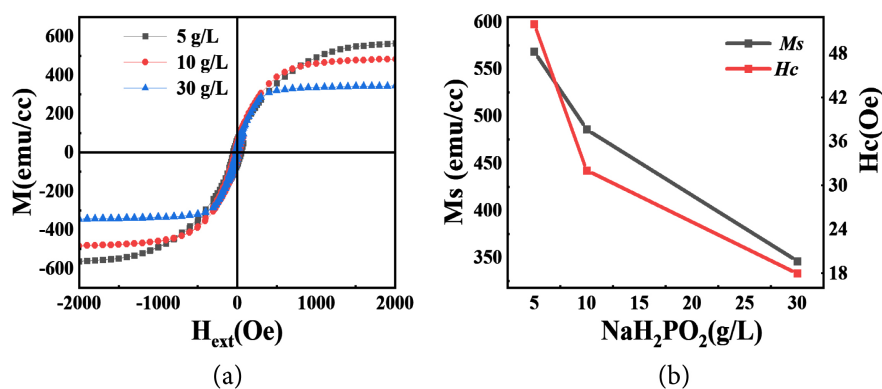


Figure 2. Hysteresis loop of electrodeposited FeMnP at different (a) sodium hypophosphite, (b) The variation of magnetization and coercivity at different concentrations of sodium hypophosphite.

leading to decreases in saturation magnetization [29] [30]. Similar results were shown in FeBSiP amorphous alloy [31].

Figure 2(b) shows linear decreases of the coercivity field from 52 Oe to 18 Oe with the NaH_2PO_2 increases. The linear decreases of H_c were also shown in the FeCOP films [32]. The coercivity change could be due to internal stress, film thickness, substrate roughness, composition, and plating pretreatment [33]. The rise of P content increases the grain refining ability (reducing grain size). Therefore the amorphous forming ability increased [23]. In the present work, the film thickness is (about 1.5 μm), substrate roughness and plating pretreatment are similar. Subsequently, the trend of coercivity is related to internal stress and film composition.

4.2. Effect of Magnetic Annealing on Static Magnetic Properties of FeMnP Films

Figure 3 shows the M-H loop of the annealed film (5 g/L) at different temperatures. The influence of magnetic annealing on hysteresis is minimal. The film is still isotropy. In magnetic annealing, the uniaxial anisotropy could be due to the directional arrangements of vacancy or impurities, the presence of a directional strain, and the directional of atomic pairs [34]. However, the results show consistency with the kinetic reorientation theory [35], which predicts the uniaxial anisotropy (K_u) is zero for mono magnetic atom films, as shown in $\text{Fe}_{75}\text{P}_{15}\text{C}_{10}$

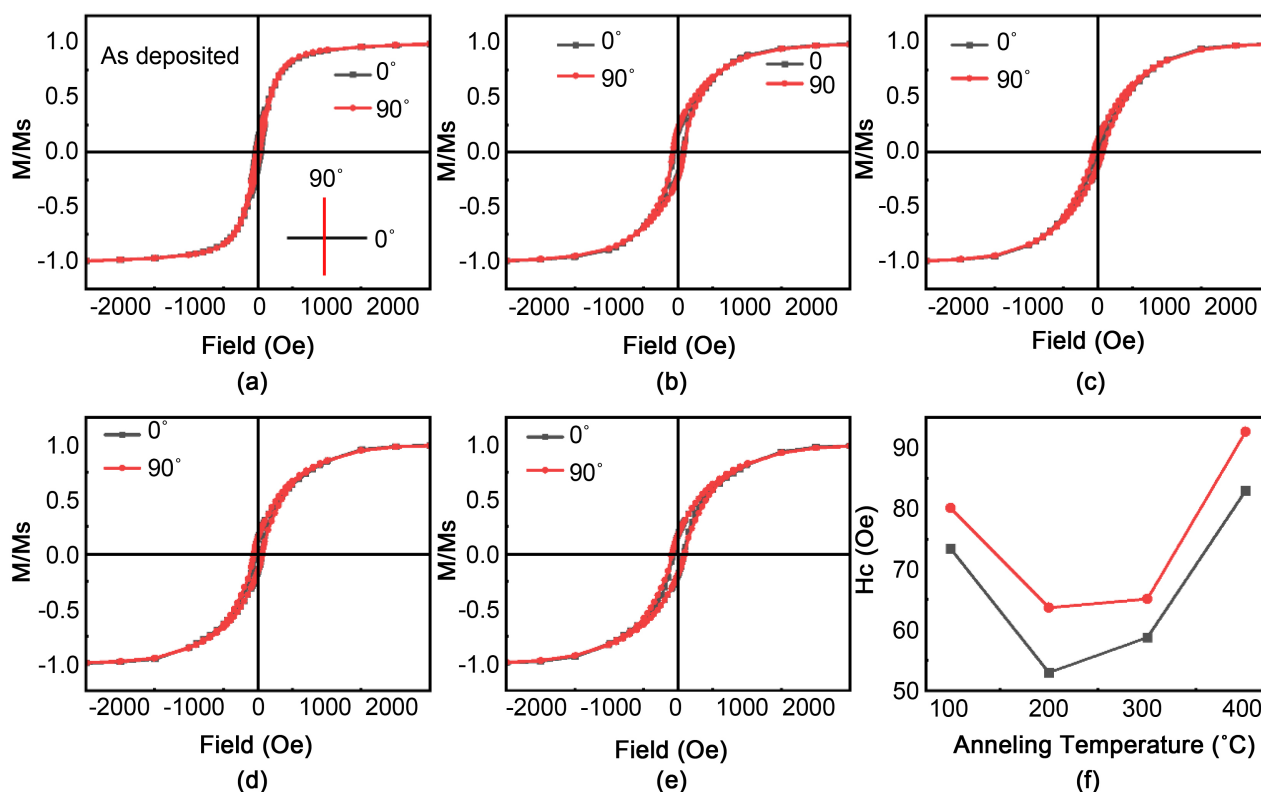


Figure 3. Parallel and perpendicular hysteresis loops of the sample (5 g/L) annealed at different temperatures and its coercivity field with temperature variation.

[36]. To induce uniaxial anisotropy by applying an external magnetic field during deposition and magnetic annealing is not suitable for inducing uniaxial anisotropy in FeMnP amorphous. However, other magnetic properties were improved. The improvements in magnetic properties are due to the relaxation of structure below the crystallization temperature and the change of internal stress [37]. The coercivity field decreased at 200°C and then increased in parallel and perpendicular directions. The change in coercivity depends on many factors such as grain size, internal stress, and crystal structure. The crystallization temperature of FeMnP amorphous alloy was related to the P composition in the films. The higher the P content, the more diffusion rate occurs and inhibits grain growth.

5. Dynamic Magnetic Properties of Electrodeposited FeMnP Films

5.1. Effect of P on Complex Permeability Spectra of FeMnP Amorphous Films

Anisotropy constant is essential to discuss the dynamics of magnetic materials; however, our samples are isotropy as the VSM results shown. In this part, we will discuss the influence of phosphorus and magnetic annealing on the natural resonance frequency and resonance linewidth of the magnetic spectra.

Figure 4 shows the magnetic spectra of electrodeposited FeMnP films from various concentrations of NaH_2PO_2 . The complex permeability ($\mu_r = \mu'_r - j\mu''_r$), where μ'_r stand for the real part and μ''_r imaginary part. The ferromagnetic resonance peak becomes broad as the NaH_2PO_2 concentration increases. The broadness of the resonance peak means that the damping factor- α increased with the P content increases in the films due to reducing the exchange interaction force between ferromagnetic metals [38]. The exchange coupling interactions are affected by doping elements and cause fluctuations such as local magnetic anisotropies, internal field, and increased damping [39].

According to the Landau-Lifshitz-Gilbert Equation (LLG)

$$\frac{dM}{dt} = -\gamma(M \times H) + \frac{\alpha}{M} M \times \frac{dM}{dt} \quad (1)$$

$$\Delta f_r = \frac{\alpha\gamma}{2\pi} \times (2H_k + 4\pi M_s) \quad (2)$$

where, Δf_r stand for full width half maximum, α damping constant. γ gyro-magnetic ratio, H_k anisotropy constant, and M_s saturation magnetization. From Equation (2), the full width at the half maximum of the resonance curve increase with damping factor- α [40]. Subsequently, spin-orbital coupling causes a spin-mixing state (the spin states of the electrons are mixtures of pure “up” and “down”, relative to the magnetic field). Shaw and colleagues calculated the spin-mixing parameter by combining FMR With XCMD, and the results obtained reveal that any small mixing parameter influences ferromagnetic resonance (FMR) spectra [41]. The extrinsic magnetic damping is often affected by

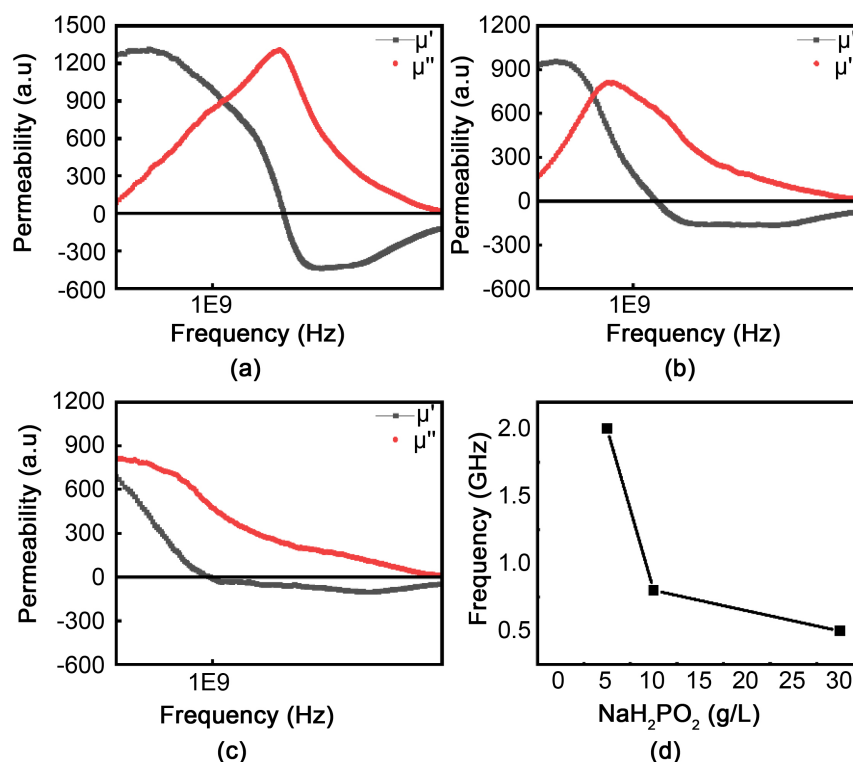


Figure 4. Magnetic spectra of electrodeposited FeMnP alloy at different concentrations of sodium hypophosphite (a) 5 g/L, (b) 10 g/L, (c) 30 g/L, and (d) variation of resonance frequency as a function of sodium hypophosphite.

doping or lattice effect, which changes the crystal structure [42]. From EDX results shown in Table 2, the films obtained at 30 g/L of NaH_2PO_2 has a high content of P. Thus, Figure 4(c) shows a high magnetic damping factor that could be related to the high composition of P in the films, the more P incorporated in the films, it could enhance the change of ferromagnetic to paramagnetic behavior [33].

Figure 4(d) shows the decreases of ferromagnetic resonance frequency with the NaH_2PO_2 concentration. It could be due to the reduction of saturation magnetization. According to the Kittel equation [43] [44]

$$f_r = \frac{\gamma}{2\pi} \sqrt{H_k (H_k + 4\pi M_s)} \quad (3)$$

where f_r , γ , M_s , and H_k stand for resonance frequency, gyromagnetic ratio, saturation magnetization, and Uniaxial anisotropy field, respectively. The resonance frequency is proportional to the saturation magnetization and magnetic anisotropy field H_k . Nevertheless, the H_k in the present work is negligible. Hence the natural resonance frequency depends on saturation magnetization.

5.2. Influence Magnetic Heat Treatment on Magnetic Spectra of FeMnP Films

5.2.1. Sample with Lower P Content

The effect of magnetic annealing on electrodeposited was carried out on the

films with lower 5 g/L and higher (30 g/L) content of P. **Figure 5** shows that the absorption peak becomes narrow after heating the sample. The film annealed at 100°C shows the magnetic spectrum with a lower resonance linewidth which means that the film has the smallest damping- α and the natural resonance frequency was around 3 GHz. The resonance frequency increased about 1.2 GHz after annealing at 100°C. The annealing temperature increased to 200°C, the resonance linewidth slightly increased, and the high natural resonance frequency value was obtained at about 3.8 GHz. At 300°C - 400°C, the magnetic damping factor- α slightly increased. The increases in damping could be related to the increase in coercivity [45]. The natural resonance frequency was reduced from 3.8 GHz to 3.5 GHz at 300°C. However, at 400°C, the natural resonance frequency increased up to 3.6 GHz. From **Figure 5(e)**, when the temperature increased from 100°C to 200°C, the natural resonance frequency increased from 3 GHz to 3.8 GHz.

Further temperature increases, the natural resonance frequency reduced up to 3.6 GHz at 400°C. The natural resonance frequency becomes two times the value of the unannealed sample at 200°C. This film with excellent soft magnetic properties was 200°C, with the lowest coercivity, damping factor, and higher resonance frequency. 200°C could be the critical crystallization temperature of amorphous FeMnP alloy film deposited at 5 g/L. The enhancement of soft magnetic properties is due to the reduced internal stress and relaxation structure [46]. Therefore, the relaxation of structure produces directional order below

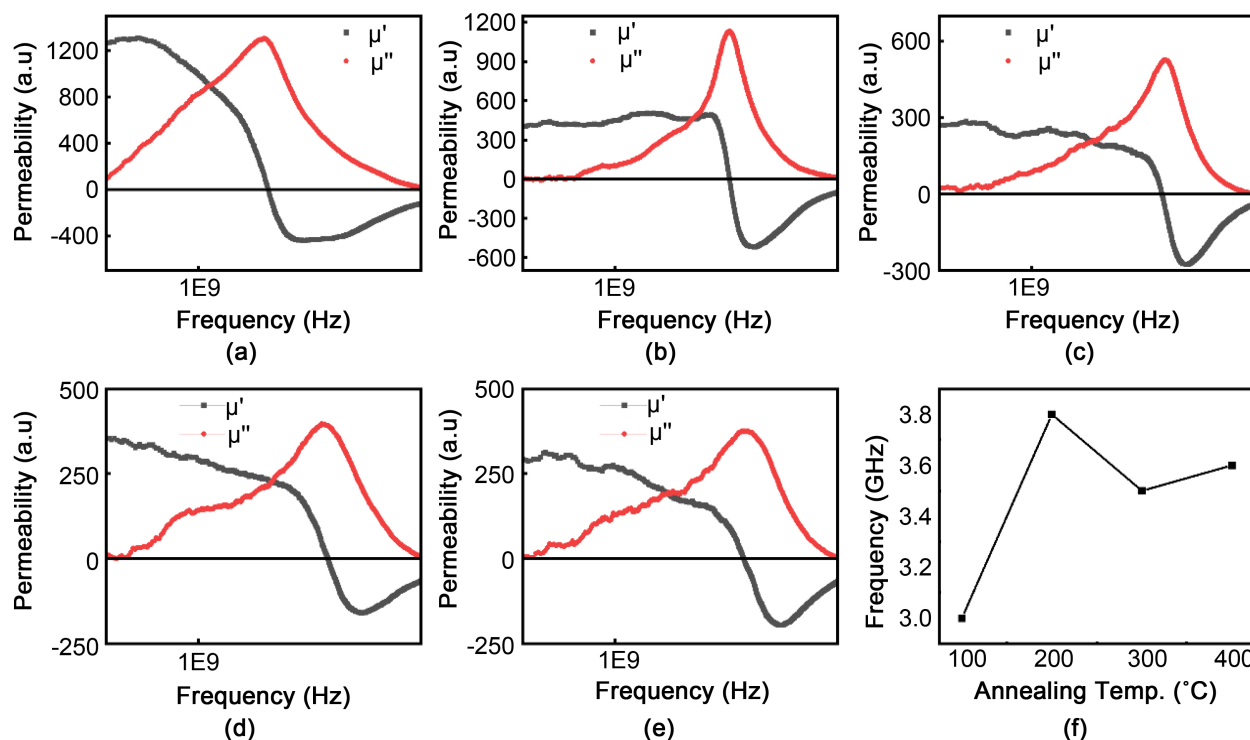


Figure 5. Magnetic spectra of annealed sample (5 g/L) at different temperature, (a) as deposited, (b) 100°C, (c) 200°C, (d) 300°C (e) 400°C, and (f) Resonance frequency as function of annealing temperature.

crystallization temperature.

5.2.2. Samples with Higher P Content

Figure 6 shows the complex permeability of FeMnP film with higher P content annealed at different temperatures. The magnetic damping factor- α decreased with the annealing temperature increases, as shown in **Figures 6(a)-(e)**. The natural resonance frequency increased with annealing temperature. The relation between magnetic damping factor- α with natural resonance frequency can be expressed using the empirical formula [36] written as

$$\Delta H = \Delta H_0 + \frac{4\pi\alpha f}{\gamma} \quad (4)$$

where ΔH is the resonance linewidth, considered full-width at half maximum (FWHM), ΔH_0 is inhomogeneous line width from extrinsic magnetic damping of the processing magnetization, α is damping parameter γ gyromagnetic ratio and f represents ferromagnetic resonance frequency. Thus the magnetic damping factor- α is inversely proportional to the resonance frequency. Thus the increase of natural resonance frequency with annealing temperature is reasonable. At 400°C, the resonance frequency became 3.7 GHz, around six times that of the unannealed sample. The change in magnetic properties during annealing could be related to the change of internal stress in the films and microstructure [47].

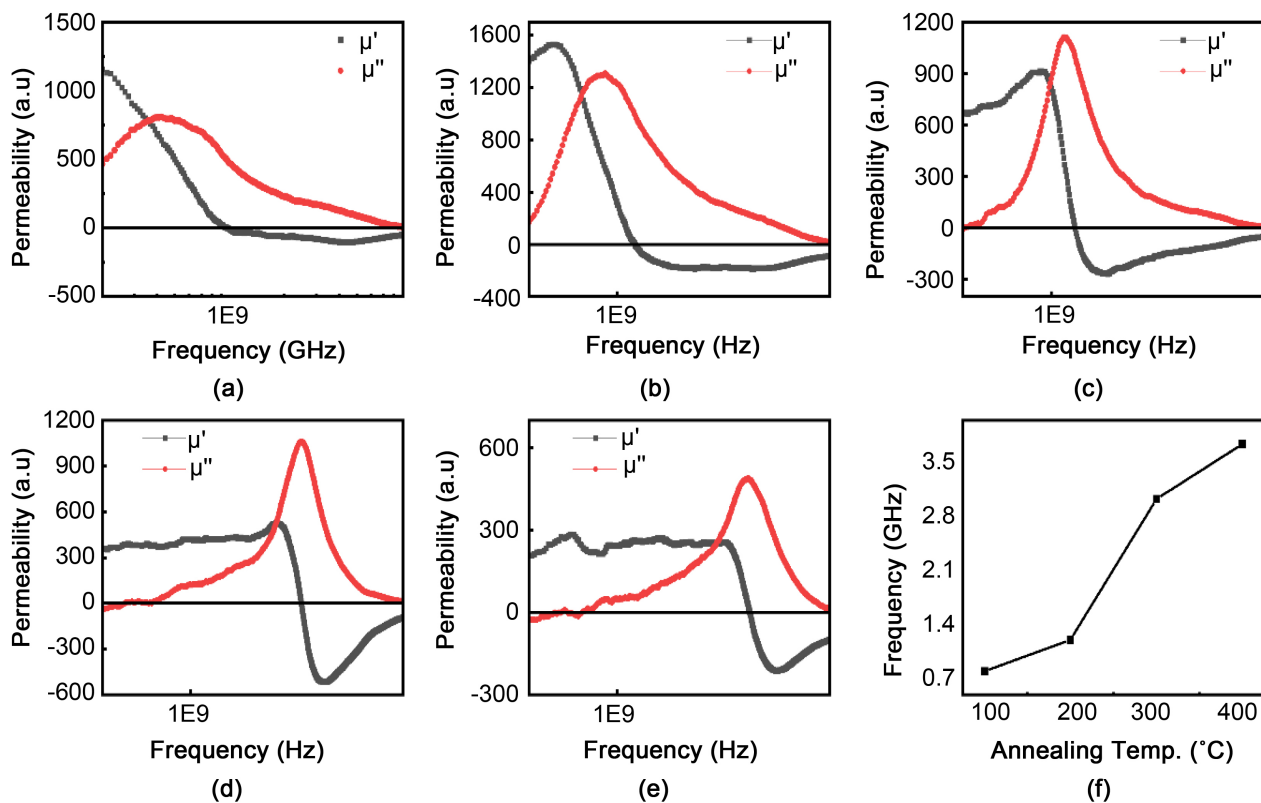


Figure 6. Magnetic spectra of annealed sample 30 g/L at a different temperature, (a) as-deposited, (b) 100°C, (c) 200°C, (d) 300°C, (e) 400°C, and (f) resonance frequency as a function of annealing temperature.

6. Conclusion

About 1.5 μm thick FeMnP amorphous thin films had successfully electrodeposited on copper sheet substrates for the first time. The influence of P and heat treatment on magnetic properties of electrodeposited was investigated. The increase of P in the films reduces the coercivity and saturated magnetization. Applying a static magnet on the electrode and magnetic annealing could not induce in-plane uniaxial anisotropy which confirms that the mono magnetic film alloys can not induce uniaxial anisotropy by applying static magnet and magnetic annealing. However, the natural resonance linewidth or magnetic damping factor decreased while resonance frequency increased with the annealing temperature. Therefore the soft magnetic properties were enhanced with annealing temperature. The crystallization temperature could depend on the composition of P in the film. The higher content of P, the higher the crystallization temperature. Future studies are required to find a suitable method to induce uniaxial magnetic anisotropy in FeMnP amorphous alloy thin films to enhance the high-frequency soft magnetic behavior of FeMnP films.

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Declaration of Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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