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Effect of Annealing and Thickness of Co_{40}Fe_{40}Yb_{20} Thin Films on Various Physical Properties on a Glass Substrate

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Abstract: The aim of this work is to investigate the effect of annealing and thickness on various physical properties in Co_{40}Fe_{40}Yb_{20} thin films. X-ray diffraction (XRD) was used to determine the amorphous structure of Co_{40}Fe_{40}Yb_{20} films. The maximum surface energy of 40 nm thin films at 300 °C is 34.54 mJ/m². The transmittance and resistivity decreased significantly as annealing temperatures and thickness increased. At all conditions, the 10 nm film had the highest hardness. The average hardness decreased as thickness increased, as predicted by the Hall–Petch effect. The highest low-frequency alternative-current magnetic susceptibility (χ_{ac}) value was discovered when the film was annealed at 200 °C with 50 nm, and the optimal resonance frequency (f_{res}) was in the low frequency range, indicating that the film has good applicability in the low frequency range. At annealed 200 °C and 50 nm, the maximum saturation magnetization (Ms) was discovered. Thermal disturbance caused the Ms to decrease when the temperature was raised to 300 °C. The optimum process conditions determined in this study are 200 °C and 50 nm, with the highest Ms, X_{ac}, strong adhesion, and low resistivity, which are suitable for magnetic applications, based on magnetic properties and surface energy.

Keywords: X-ray diffraction (XRD); adhesion; transmittance; electrical properties; nanomechanical property; low-frequency alternating current magnetic susceptibility (χ_{ac})

1. Introduction

CoFe alloys are commonly used in a variety of magnetic devices, such as sensors, actuators, read heads, and magnetic random-access memory (MRAM) [1–3]. Most non-crystal glass substrates used for perpendicular magnetic anisotropy (PMA) have a certain degree of rigidity and flatness [4,5]. The soft magnetic underlayer is very helpful for writing of perpendicular magnetic recording. The role is to help the single pole writer with magnetic field line transmission. It can be written into the material with higher magnetic stability of the recording medium. The magnetic properties of a Co-Fe (Cobalt Iron) alloy can be tuned depending on the application by varying the Co to Fe ratio [6,7]. However, the CoFe alloy does not have low coercivity (Hc), and the annealing temperature accelerates the degeneration of magnetic anisotropy faults, making it difficult to meet the magnetic...
equipment used at high temperatures. One solution for increasing the thermal stability of the CoFe alloy is to add a third element. The common characteristics of rare earth magnetic materials are high saturation magnetization (Ms), Curie temperature (Tc), and magnetic anisotropy field (Hk), particularly. In general, the addition of rare-earth (RE) elements to CoFe alloy can improve its hardness, corrosion resistance, and durability, as well as improve heat resistance and other benefits [8–10]. Due to their widespread use in numerous applications, including spintronic, fiber amplifier, photoluminescence, and laser, RE element-doped nanomaterials have become the center of interest. As a result, the primary research goal of this paper is to identify other elements that can be added to CoFe alloys to effectively improve their stability. Ytterbium (Yb) is an important RE element because of its unique optical and magnetic properties, as well as its incompletely filled 4f electronic states [11–13]. YAG (Yttrium Aluminum Garnet) nanophosphor powder doped with Yb$^{3+}$ is an excellent laser material [14]. Moreover, a free or pinned layer of Co$_{40}$Fe$_{40}$B$_{20}$ thin film is frequently utilized in magnetic tunneling junction (MTJ) structures because it has a high tunneling magnetoresistance (TMR) ratio. To test several specific features, this study predominantly formed CoFeYb films using the same Yb and B (Boron) ratio. The innovative aspects of this research include examining annealed CoFeYb thin films to determine if they would change in high temperature environments, as well as investigating the structure and magnetic properties of CoFeYb thin films as a function of thickness. The performance is more sensitive to high operating temperatures and room temperature (RT). However, a few studies on the magnetic, electrical, optical, and nanomechanical properties of CoFeYb films under as-deposited and annealed conditions have been conducted. For the convenience of readers, the abbreviations and full names of proper nouns in this article are listed in Table 1. This study also looks at the thicknesses of CoFeYb in various as-deposited and annealed conditions. Previous research has compared the magnetic and adhesive properties of CoFeYb materials to those of CoFeV and CoFeW in Table 2 [15–17].

**Table 1.** Abbreviations and full names of proper nouns.

| Abbreviation | Full Name |
|--------------|-----------|
| XRD          | X-ray diffraction |
| $\chi_{ac}$  | low-frequency alternative-current magnetic susceptibility |
| $f_{res}$    | optimal resonance frequency |
| MS           | saturation magnetization |
| MRAM         | magnetic random-access memory |
| PMA          | perpendicular magnetic anisotropy |
| Ku           | magnetic anisotropy constant |
| CoFe         | cobalt iron |
| Hc           | coercivity |
| Tc           | Curie temperature |
| Hk           | magnetic anisotropy field |
| RE           | rare-earth |
| Yb           | ytterbium |
| YAG          | yttrium aluminum Garnet |
| MTJ          | magnetic tunneling junction |
| TMR          | tunneling magnetoresistance |
| B            | boron |
| RT           | room temperature |
| DC           | direct current |
| Ar           | argon |
| GIXRD        | grazing incidence X-ray diffraction |
| $\theta$     | contact angle |
| DI           | deionized |
| MTS          | mechanical testing and simulation |
| CSM          | continuous stiffness measurement |
| AGM          | alternating gradient magnetometer |
| Hext         | external magnetic field |
Table 2. Significant properties for CoFeV, CoFeW, and CoFeYb materials.

| Materials                  | Maximum $\chi_{ac}$ (a.u.) | Surface Energy (mJ/mm$^2$) |
|----------------------------|-----------------------------|-----------------------------|
| Glass/Co$_{40}$Fe$_{40}$V$_{20}$ [15,16] | 0.02–0.04                   | 27.8–45.4                   |
| 10–100 nm at RT            |                             |                             |
| Glass/Co$_{32}$Fe$_{30}$W$_{38}$ [17] | 0.02–0.52                   | 22.3–28.4                   |
| 10–50 nm at RT and annealed conditions |                             |                             |
| Glass/Co$_{40}$Fe$_{40}$Yb$_{20}$ (Current research) | 0.04–0.35                   | 28.6–34.5                   |
| 10–50 nm at RT and annealed conditions |                             |                             |

2. Materials and Methods

CoFeYb with a thickness of 10–50 nm was sputtered onto a glass substrate at room temperature (RT) using a magnetron sputtering direct current (DC) method with a power of 50 W and the four conditions listed below: (a) as-deposited films were kept at RT, (b) annealed at 100 °C for 1 h, (c) annealed at 200 °C for 1 h, and (d) annealed at 300 °C for 1 h. A schematic of the experimental sputtering system is shown in Figure 1. Ar (Argon) operating pressure was $1.54 \times 10^{-3}$ Torr and the chamber base pressure was $1.07 \times 10^{-7}$ Torr. CoFeYb alloy with a target composition was 40% Co, 40% Fe, and 20% Yb. With a particular Ar gas, the pressure in the ex-situ annealed condition was $2.5 \times 10^{-3}$ Torr. Grazing incidence X-ray diffraction (GIXRD) patterns acquired with CuKα1 (PAN analytical X'pert PRO MRD, Malvern Panalytical Ltd., Cambridge, UK) and a low angle diffraction incidence of around two degrees were used to analyze the crystal structure of CoFeYb films. A contact angle (θ) measuring tool (CAM-110, Creating Nano Technologies, Tainan City, Taiwan) was utilized to measure the contact angle using deionized (DI) water and glycerol, which was then used to determine the surface energy [18–20]. A spectral intelligent analyzer (Collimage, Taipei, Taiwan) was used to measure the transmittance of CoFeYb. The visible light wavelength ranged from 500 to 800 nm. The electrical properties are detected by four-point probe measurement (Sadhudesign, Hsinchu City, Taiwan). Using the MTS (mechanical testing and simulation) Nano Indenter XP (MTS, Minneapolis, MN, USA) with a Berkovich tip and the continuous stiffness measurement (CSM) method, the hardness of Co$_{40}$Fe$_{40}$Yb$_{20}$ films was examined. Once the load was reduced to 10% of the maximum load, we removed the indent from the surface at the same rate. Measurement should be repeated ten times for each sample with the probe. The indentation load is multiplied by 40 stages, with each step’s indentation depth being recorded. In order to produce more precise data, six indentations from each sample were evaluated, and the standard deviations were averaged. The $\chi_{ac}$ analyzer (XacQuan, MagQu Co., Ltd., New Taipei City, Taiwan) and alternating gradient magnetometer (AGM, PMC, Westerville, OH, USA) were used to investigate the in-plane low-frequency alternate-current magnetic susceptibility ($\chi_{ac}$) and hysteresis loop of Co$_{40}$Fe$_{40}$Yb$_{20}$ thin films.
3. Results

3.1. Structure

The XRD patterns for each of the four situations are shown in Figure 2. Figure 2a–d show that the film produces no distinctive diffracted peak, indicating that it is amorphous and that the addition of Yb to CoFe alloys causes the refinement of grain size [21]. The figure also shows that the Co₄₀Fe₄₀Yb₂₀ film sputtered on the glass substrate is in an amorphous condition and that the thermal driving force is insufficient to sustain grain growth [21–24]. The Co₄₀Fe₄₀Yb₂₀ sputtering film is randomly arranged when the substrate is randomly arranged due to the amorphous nature of the glass substrate. When the film is deposited in thicknesses ranging from 10 nm to 50 nm, the film structure is not completely ordered, resulting in the substrate effect.

![Figure 1: Schematic diagram of sputtering system.](image)

![Figure 2: XRD patterns for each of the four situations.](image)
3.2. Analysis of Surface Energy and Adhesion

The contact angles measured under four conditions using DI water and glycerol are shown in Table 3. The Co\textsubscript{40}Fe\textsubscript{40}Yb\textsubscript{20} films had contact angles that were reportedly less than 30°.

Figure 2. Thin films made of Co\textsubscript{40}Fe\textsubscript{40}Yb\textsubscript{20} with X-ray diffraction patterns. (a) as-deposited, (b) post-annealing at 100 °C, (c) post-annealing at 200 °C, and (d) post-annealing at 300 °C.
3.2. Analysis of Surface Energy and Adhesion

The contact angles measured under four conditions using DI water and glycerol are shown in Table 3. The Co\textsubscript{40}Fe\textsubscript{40}Yb\textsubscript{20} films had contact angles that were reportedly less than 90°, and the drops were almost spherical, which led to good hydrophilicity and wettability. The contact angle can therefore be inferred to be decreasing as the higher annealed temperature increases. It is reasonable to infer that the large grains of annealed material exhibit significant gaps between adjacent grains when the grains are arranged in the material. As the gaps widen and the support between the crystal grains diminishes, the crystal grain size rises and a trend toward reduced contact angles develops. Water easily flows into gaps when it falls on a surface, producing small contact angles, strong adhesion, and high surface energies [25]. Surface energy and adhesion are critical because Co\textsubscript{40}Fe\textsubscript{40}Yb\textsubscript{20} film can be used as an underlayer or buffer layer. When the surface energy is high, liquid absorption is significant and the contact angle decreases. The surface energy is calculated using Young’s equation and the contact angle [18–20].

Table 3. Comparing contact angle and surface energy for Co\textsubscript{40}Fe\textsubscript{40}Yb\textsubscript{20} thin films from different fabrication processes.

| Process            | Thickness | Contact Angle with DI Water (θ) | Contact Angle with Glycerol (θ) | Surface Energy (mJ/mm\textsuperscript{2}) |
|--------------------|-----------|---------------------------------|---------------------------------|-------------------------------------------|
| As-deposited        | 10 nm     | 76.5°                           | 70.9°                           | 29.47                                     |
|                    | 20 nm     | 74.4°                           | 67.9°                           | 30.37                                     |
|                    | 30 nm     | 74.6°                           | 67.1°                           | 30.86                                     |
|                    | 40 nm     | 69.9°                           | 65.3°                           | 33.02                                     |
|                    | 50 nm     | 68.8°                           | 65.1°                           | 33.72                                     |
| Post-annealing 100 °C | 10 nm    | 81.3°                           | 71.4°                           | 28.63                                     |
|                    | 20 nm     | 84.0°                           | 72.5°                           | 29.19                                     |
|                    | 30 nm     | 79.1°                           | 71.6°                           | 29.45                                     |
|                    | 40 nm     | 74.1°                           | 72.0°                           | 29.90                                     |
|                    | 50 nm     | 79.8°                           | 69.1°                           | 30.61                                     |
| Post-annealing 200 °C | 10 nm    | 82.5°                           | 71.9°                           | 28.82                                     |
|                    | 20 nm     | 86.4°                           | 72.8°                           | 31.46                                     |
|                    | 30 nm     | 71.8°                           | 69.6°                           | 31.28                                     |
|                    | 40 nm     | 70.9°                           | 70.4°                           | 32.48                                     |
|                    | 50 nm     | 74.8°                           | 66.3°                           | 31.52                                     |
| Post-annealing 300 °C | 10 nm    | 79.7°                           | 71.8°                           | 30.17                                     |
|                    | 20 nm     | 71.1°                           | 68.3°                           | 32.45                                     |
|                    | 30 nm     | 77.5°                           | 67.7°                           | 32.35                                     |
|                    | 40 nm     | 77.1°                           | 64.7°                           | 34.54                                     |
|                    | 50 nm     | 74.8°                           | 63.7°                           | 34.23                                     |

The surface energy is depicted in Figure 3 under all conditions. The surface energy varied between 28.63 and 34.54 mJ/mm\textsuperscript{2}. The adhesion was strongest when the films had a higher surface energy. According to the calculations, the highest surface energy of 40 nm at 300 °C was 34.54 mJ/mm\textsuperscript{2}.
3.3. Examination of Optical Properties

Figure 4 depicts the optical transmittance (%) spectra at visible wavelengths ranging from 500 nm to 800 nm. As shown in Figure 4a, as the thickness increased from 10 to 50 nm, the transmittance at RT decreased from 76.21% to 26.50%. Figure 4b shows that at a temperature of 100 °C, the transmittance decreased from 77.98% to 25.83%. Figure 4c shows that the transmittance decreased from 84.75% to 27.45% at the 200 °C annealing temperature, while Figure 4d shows that the transmittance decreased from 80.12% to 25.50% at the 300 °C annealing temperature. Only a minor change in transmittance was observed as the annealing temperature increased, and the trend was not immediately apparent. These findings imply that thinner Co$_{40}$Fe$_{40}$Yb$_{20}$ films have a higher transmission rate because thicker films obstruct the incident signal [26–28]. The increased grain size with thickness and annealing treatment can be attributed to the improved crystallinity. Crystallinity increases due to the increased ability of add atoms to move to stable sites in the lattice. Thicker films are more homogeneous, resulting in fewer defects and localized states and an increase in the optical band gap [29].
Figure 4. Transmittance of Co$_{40}$Fe$_{40}$Yb$_{20}$ films. (a) RT, (b) following annealing at 100 °C, (c) following annealing at 200 °C, and (d) following annealing at 300 °C.
3.4. Electrical Analysis

The resistivity and sheet resistance are depicted in Figure 5a,b under all conditions. The resistivity at 10 nm was not tested because there was insufficient thickness to be examined. The sheet resistance and resistivity of Co$_{40}$Fe$_{40}$Yb$_{20}$ films varied with thickness and annealing treatment. The conduction mechanism of CoFeYb film is thought to be related to the carrier present in the structure. The electrical properties of CoFeYb films are strongly influenced by their as-deposited and annealed environments. However, higher annealed temperatures and thicker thicknesses appear to result in better crystalline structure and lower carrier hindrance in the films, which is the dominant factor of conductivity and leads to lower resistivity. The results show that the sheet resistance and resistivity of CoFeYb films prepared using annealing treatments are lower than those obtained using unheated substrates [30]. In total, the resistivity varied between 0.02 and 113.47 Ω·cm, and the sheet resistance varied between $4.5 \times 10^3$ and $5.7 \times 10^7$ Ω. The resistivity and sheet resistance of as-deposited and thinner films are higher than those of annealed and thicker films because they have lower electron mobility through the film and more defects and impurities, which increase the presence of electron scattering [31,32].

![Figure 5](image-url)
3.5. Hardness Analysis

Figure 6 demonstrates that the films' hardness ranged from 7.24 GPa to 7.70 GPa under all circumstances. The hardness shows a decreasing trend as thickness increases. As-deposited films have a harder surface than annealed ones. As the penetration depth increased, the hardness of the thin film with varying glass substrate thicknesses decreased; this could be a characteristic of the substrate when the penetration depth is sufficient [33,34]. The Pharr–Oliver method is commonly used to calculate hardness from loading and unloading curves, which demonstrate the combined hardness of the glass substrate and CoFeYb films [35]. Because of the thinness of the CoFeYb film, it is reasonable to assume that the substrate effect in nano-indentation measurement exists [36,37]. Furthermore, as the thickness increases, the hardness decreases, which is consistent with the Hall–Petch effect [38]. Because the harness value of annealing 300 °C thin film at a thickness of 30 nm is exhibiting abnormal behavior, it can be reasonably concluded that higher annealing treatment induces oxidation or impurity, resulting in dislocations that are difficult to move and increasing hardness [39].

![Average hardness of the Co$_{40}$Fe$_{40}$Yb$_{20}$ thin films.](image)

**Figure 6.** Average hardness of the Co$_{40}$Fe$_{40}$Yb$_{20}$ thin films.

3.6. Magnetic Analysis

Figure 7a–d depict the low-frequency alternative-current magnetic susceptibility ($\chi_{ac}$) under four conditions. The $\chi_{ac}$ decreased as the frequency increased in the 50–25,000 Hz range. As the film thickness increased from 10 to 50 nm, the corresponding $\chi_{ac}$ value increased. At high frequency, the $\chi_{ac}$ values of all films dropped dramatically. The maximum $\chi_{ac}$ value for as-deposited 50 nm was 0.214. The maximum $\chi_{ac}$ value for the 50 nm film was 0.176 after post-annealing at 100 °C, 0.358 after post-annealing at 200 °C, and 0.280 after post-annealing at 300 °C.

Figure 8 depicts the corresponding maximum $\chi_{ac}$ values under all conditions. Because of the thickness effect, the maximum $\chi_{ac}$ increased [40]. The annealing treatment produced the highest $\chi_{ac}$ value of 0.358 at post-annealing 200 °C of the 50 nm film. Furthermore, due to thermal disturbance, the ac of post-annealing at 300 °C was lower than that at 200 °C. The maximum $\chi_{ac}$ for the optimal resonance frequency ($f_{res}$) under four different conditions is shown in Table 4. At the optimal resonance frequency, the maximum $\chi_{ac}$ exhibited the strongest spin sensitivity [41]. The $f_{res}$ value was less than 100 Hz at each thickness. The optimal resonance frequency was determined to be less than 500 Hz, making it appropriate for use in low-frequency sensors, transformers, and magnetic components.
Figure 7. Cont.
Figure 7. The relationship between the low-frequency alternate-current magnetic susceptibility ($\chi_{ac}$) and frequency. (a) as-deposited, (b) post-annealing at 100 °C, (c) post-annealing at 200 °C, (d) post-annealing at 300 °C.

Figure 8. Maximum alternate-current magnetic susceptibility for Co$_{40}$Fe$_{40}$Yb$_{20}$ thin films.

Table 4. The optimal resonance frequency for various thicknesses of films.

| Thickness (nm) | As-Deposited Optimal Resonance Frequency (Hz) | Post-Annealing at 100 °C of Optimal Resonance Frequency (Hz) | Post-Annealing at 200 °C of Optimal Resonance Frequency (Hz) | Post-Annealing at 300 °C of Optimal Resonance Frequency (Hz) |
|---------------|-----------------------------------------------|---------------------------------------------------------------|---------------------------------------------------------------|---------------------------------------------------------------|
| 10            | 100                                           | 50                                                            | 50                                                            | 50                                                            |
| 20            | 50                                            | 100                                                           | 50                                                            | 50                                                            |
| 30            | 50                                            | 50                                                            | 50                                                            | 50                                                            |
| 40            | 50                                            | 50                                                            | 50                                                            | 50                                                            |
| 50            | 50                                            | 50                                                            | 50                                                            | 50                                                            |

The maximum $\chi_{ac}$ value of the film is 50 nm. As a result, the magnetic properties of a 50 nm film were investigated at various annealing temperatures. Figure 9a depicts the magnetic hysteresis loops of the 50 nm under four different conditions. With an external magnetic field ($H_{ext}$) of 10 kOe in the plane, the saturated magnetic spin state was visible. The expanded figure shows low Hc, implying that the Co$_{40}$Fe$_{40}$Yb$_{20}$ films have soft magnetization. The saturation magnetization (Ms) of the Co$_{40}$Fe$_{40}$Yb$_{20}$ thin films is shown...
in Figure 9b under all conditions. The maximum value of $M_s$ is comparable to the $\chi_{ac}$ result after post-annealing at 200 °C. Due to thermal disturbance, the $M_s$ and $\chi_{ac}$ values of 50 nm films annealed at 300 °C were less than those at 200 °C. $M_s$ decreased significantly after post-annealing at 100 °C, owing primarily to the temperature compensation effect [42].

![Image of magnetic hysteresis loop](image)

**Figure 9.** (a) In-plane magnetic hysteresis loop of Co$_{40}$Fe$_{40}$Yb$_{20}$ thin films at 50 nm. (b) Saturation magnetization ($M_s$) of Co$_{40}$Fe$_{40}$Yb$_{20}$ at 50 nm.

In short, the underneath glass substrate has an effect on significant properties, which is worth discussing. The glass substrate itself is an amorphous structure, so even after heat treatment, the film is still an amorphous structure, resulting in no magnetocrystalline anisotropy and reduced magnetic property, surface energy decreases, transmittance increases, and grain refinement leads to an increase in hardness [43].

### 3.7. Challenges and Prospects

A new soft magnetic material is Co$_{40}$Fe$_{40}$Yb$_{20}$ thin film. In the future, the question and challenge will be whether it can be used as a free or pinned layer in the MTJ structure. The key question is whether spin polarization can generate high TMR when applied to magnetic fields, as well as have high PMA and improve magnetic recording density.
4. Conclusions

Because of the addition of Yb and insufficient thermal driving force for grain growth, XRD results reveal that the structure is amorphous. A decrease in transmittance indicated that the thickness and interfacial effects were responsible for the transfer of photon signals through the material. The resistivity and sheet resistance significantly decreased as the thickness increased. As-deposited films have a harder surface than annealed ones. Hardness decreased as thickness increased, which is consistent with the Hall–Petch effect. At a post-annealing temperature of 200 °C, which is consistent with $\chi_{ac}$, the greatest Ms for a 50 nm was observed. The Ms and $\chi_{ac}$ values of the 50 nm film when annealed at 300 °C were lower than those at 200 °C, due to thermal disturbance. As a result, it was discovered that a 50 nm thick film and a 200 °C annealing temperature produced the highest ac and Ms and the lowest resistivity. The films were suitable for use in magnetic storage devices at this temperature because the Ms and $\chi_{ac}$ values were at their highest.

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