Annealing Effect on the Structural, Magnetic, Electrical, Optic Property, Nanomechanical, and Adhesive Characteristics of Co$_{60}$Fe$_{20}$Yb$_{20}$ Thin Films on Glass Substrate

Wen-Jen Liu $^{1}$, Yung-Huang Chang $^{2}$, Yuan-Tsung Chen $^{3,*}$, Po-Chun Chiu $^{3}$, Yu-Zhi Wang $^{3}$, Shih-Hung Lin $^{4}$ and Po-Wei Chi $^{5}$

Abstract: In this study, X-ray diffraction (XRD) analysis showed the amorphous nature of the Co$_{60}$Fe$_{20}$Yb$_{20}$ films deposited at room temperature (RT), 100 $^\circ$C, and 200 $^\circ$C. The body-centered cubic (BCC) CoFe ($110$) characteristic peak was visible at 44.7$^\circ$ after annealing films of 40 nm and 50 nm at 300 $^\circ$C. The highest alternating current magnetic susceptibility ($\chi_{ac}$) value was 0.21 at 50 Hz in a 50 nm, and the lowest resistivity value was 1.02 ($\times 10^{-2}$ $\Omega$ cm) in a 50 nm. In terms of nano-indication measurement, the highest value of hardness was 9.29 GPa at 300 $^\circ$C in a 50 nm. When the thickness increased from 10 nm to 50 nm, the hardness and Young’s modulus of the Co$_{60}$Fe$_{20}$Yb$_{20}$ film also showed a saturation trend. The Co$_{60}$Fe$_{20}$Yb$_{20}$ film had the maximum surface energy at 50 nm after 300 $^\circ$C annealing. The transmittance of Co$_{60}$Fe$_{20}$Yb$_{20}$ films decreased when the thickness was increased because the thickness effect suppresses the photon signal. Due to high $\chi_{ac}$, low electrical performance, strong nano-mechanical properties, and high adhesion, it was discovered in this work that 50 nm with annealing at 300 $^\circ$C was the ideal condition for the magnetic and adhesive capabilities of Co$_{60}$Fe$_{20}$Yb$_{20}$ film. More importantly, replacing the CoFeB seed or buffer layer with a thin CoFeYb film improved the thermal stability, making CoFeYb films attractive for practical magnetic tunnel junction (MTJ) applications. Furthermore, the specific properties of Co$_{60}$Fe$_{20}$Yb$_{20}$ films were compared to those of Co$_{60}$Fe$_{20}$Y$_{20}$ films, demonstrating that the specific properties of these two materials may be compared.

Keywords: annealed Co$_{60}$Fe$_{20}$Yb$_{20}$ thin films; X-ray diffraction (XRD); low-frequency alternating current magnetic susceptibility ($\chi_{ac}$); optimal resonance frequency ($f_{res}$); surface energy; adhesion; magnetic tunnel junction (MTJ); electrical properties; nanomechanical properties; transmittance
alloy [6–8]. Co_{60}Fe_{20}Yb_{20} was a new substance in the realm of magnetic materials in this study. CoFeYb film can be used to replace the CoFeB alloy layer in the MTJ structure. By adding Yb, the crystallinity is improved, and its heat resistance is increased. Although dynamic random access memory (DRAM) and flash memory still maintain the dominant position in the current market in the next few years, in the era of rapid technological development, driven by the demand for artificial intelligence (AI), Internet of Things (IoT), fifth generation wireless systems (5G), and emerging memory magnetoresistance random access memory (MRAM) will develop rapidly in the next few years, and gradually become a mainstream product. MRAM uses magnetic force to store data [9–15]. When the computer is turned off, the magnetic force of its memory chip still exists, so it still retains the data in the memory. The rare earth element Yb has physicochemical properties comparable to Ca. Both Yb and Ca, for example, are divalent ions with identical atomic sizes and elastic moduli. They are miscible not only in liquids, but also in crystals and solids. Corrosion resistance is higher in Yb-containing Mg-based amorphous alloys than in Ca-containing magnesium-based amorphous alloys [16–18]. Because the addition of Yb not only improves the plasticity of the alloy and it also improves its thermal stability, the classic amorphous alloy Mg-Zn-Ca is replaced by rare earth Yb [19,20]. Co_{60}Fe_{20}B_{20} thin film is commonly used in MTJ structure as a free or pinned layer, and can achieve a high tunneling magnetoresistance (TMR) ratio. This study primarily used the same Yb/B ratio to form CoFeYb films in order to investigate some specific properties. The novelty of this research is to investigate the structure and magnetic properties of CoFeYb thin films as a function of thickness, as well as to investigate annealed CoFeYb thin films to see if they will change in high temperature environments. However, following a series of sample testing and analysis, it was discovered that increasing the annealing temperature did not cause any damage to the sample. The structure, magnetic properties, electrical properties, mechanical properties, contact angle adhesion efficiency, and optical characteristics of Co_{60}Fe_{20}Yb_{20} thin films with varying thicknesses and heat treatments were all measured in this work. In previous research, the as-deposited and post-annealing Co_{60}Fe_{20}Y_{20} films were compared with Co_{60}Fe_{20}Yb_{20} films for their magnetic, optic, and adhesive properties, as mentioned in Table 1 [21].

### Table 1. Significant properties for Co_{60}Fe_{20}Y_{20} and Co_{60}Fe_{20}Yb_{20} materials.

| Type of Film | Maximum $\chi_{ac}$ (a.u.) | Maximum Surface Energy (mJ/mm$^2$) | Transmittance (%) |
|--------------|-----------------------------|----------------------------------|-------------------|
| Glass/Co_{60}Fe_{20}Y_{20} [21] | 0.23 | 31.84 | 58 |
| Glass/Co_{60}Fe_{20}Yb_{20} 10–50 nm at as-deposited and annealed conditions (Current research) | 0.21 | 31.3 | 54 |

### 2. Materials and Methods

CoFeYb thin films with thicknesses ranging from 10 to 50 nm were created on glass substrates using direct-current (DC) magnetron sputtering. The films were created in four different ways: (a) at room temperature (RT), (b) annealed for an hour at 100 °C, (c) annealed for an hour at 200 °C, and (d) annealed for an hour at 300 °C. The operating pressure for Ar was 3 × 10$^{-3}$ Torr, base pressure was 3 × 10$^{-7}$ Torr, and ex-situ annealed pressure was 2.5 × 10$^{-3}$ Torr in particular Ar gas. The target composition of CoFeYb alloy is 60% Co, 20% Fe, and 20% Yb. Additionally, grazing incidence X-ray diffraction (GIXRD) patterns produced with CuKα1 (PAN analytical X’pert PRO MRD, Malvern Panalytical Ltd., Cambridge, UK) and a low angle diffraction incidence at a two-degree angle were used to analyze the structure of the CoFeYb films. A low-frequency alternate-current magnetic susceptibility ($\chi_{ac}$) instrument (XacQuan, MagQu Co., Ltd., New Taipei, Taiwan) with measured frequency ranges of 10 to 25,000 Hz was used to study Co_{60}Fe_{20}Yb_{20} thin films. The resistivity and sheet resistance (Rs) values of Co_{60}Fe_{20}Yb_{20} films were measured using a
standard four-point approach for electrical properties. The hardness and Young’s modulus were measured using the MTS Nano Indenter XP with a Berkovich tip (MTS, Minneapolis, State of Minnesota, USA) and continuous stiffness measurement (CSM) methodology. The loading was then decreased to 10% of the maximum load before the indenter was gradually removed from the surface. The indenter took measurements for each sample at 10 different points. The depth of the indentation was measured at each of the 40 steps that increased the indentation load. Six indentations in each sample were examined, and the standard deviation was averaged to generate more trustworthy results. The contact angle was determined using deionized (DI) water and glycerol (CAM-110, Creating Nano Technologies, Tainan, Taiwan). When the sample was removed, the contact angle was measured. Finally, the surface energy was calculated using the contact angle [22–24]. The optical characteristics were assessed using a Spectro Smart Analyzer (Collimage, Taipei, Taiwan) with visible light with a wavelength range of 500–800 nm.

3. Results

3.1. X-ray Diffraction

XRD patterns of as-deposited and annealed CoFeYb thin films with thicknesses varying from 10 to 50 nm are shown in Figure 1a–d. Figure 1a exhibits as-deposited thin film patterns, whereas those generated after annealing at 100 °C, 200 °C, and 300 °C are illustrated in Figure 1b–d. Figure 1a–c indicate that CoFeYb films deposited at RT and post-annealed at 100 °C and 200 °C are amorphous status. However, Figure 1d shows that when CoFeYb thin films are 40 nm and 50 nm at 300 °C, the characteristic peak CoFe (110) appears at 44.7°, which increases with the thickness. Its characteristic peak intensity has a tendency to increase. From the above results, it can be speculated that the CoFeYb thin film needs to be annealed at a temperature of at least 300 °C and a thickness of 40 nm or more before crystallization occurs.

3.2. Magnetic Property

Figure 2a–d show the $\chi_{\text{ac}}$ for four distinct preparation circumstances, with thicknesses ranging from 10 to 50 nm at RT and annealed temperatures of 100 °C, 200 °C, and 300 °C, respectively. In the low-frequency range of 50–100 Hz, the value of $\chi_{\text{ac}}$ decreases with frequency. The outcomes also demonstrate that the corresponding $\chi_{\text{ac}}$ value rises when thickness is between 10 nm and 50 nm. These findings show that all CoFeYb samples exhibit magneto–nanocrystalline anisotropy and the thickness effect. A greater annealing temperature resulted in a higher $\chi_{\text{ac}}$ value than a lower annealing temperature. In general, stronger crystallization and grain development were generated by the higher annealing temperature and thicker thickness. Magneto–nanocrystalline anisotropy caused the $\chi_{\text{ac}}$ value of CoFeYb to rise [25,26].

Figure 3 displays the matching maximum $\chi_{\text{ac}}$ for different CoFeYb thicknesses under four preparation conditions. The highest $\chi_{\text{ac}}$ of annealed CoFeYb thin film was at 300 °C, and at that temperature, the thickness was 50 nm, which is more than it was at other investigational settings. These findings clearly demonstrated the thickness effect of $\chi_{\text{ac}}$ in CoFeYb films. The thickness effect causes a rise in $\chi_{\text{ac}}$ as thickness increases. The maximum $\chi_{\text{ac}}$ of annealed CoFeYb thin films was larger than that at RT. Table 2 shows the maximum $\chi_{\text{ac}}$ for the ideal resonance frequency ($f_{\text{res}}$) under four different situations. The greatest $\chi_{\text{ac}}$ had the highest spin sensitivity at the $f_{\text{res}}$ [27,28]. At different thicknesses, $f_{\text{res}}$ value was less than 100 Hz. The $f_{\text{res}}$ was determined to be less than 500 Hz, making it suitable for usage in transformers, magnetic components, and low-frequency sensors.

3.3. Electrical Properties

The resistivity and sheet resistance (Rs) with various thicknesses and temperature conditions are shown in Figure 4. According to the findings of this investigation, resistivity and sheet resistance (Rs) both reduced with increasing thickness and annealed temperatures. The sheet resistance decreased as the annealing temperature increased in terms of temperature.
It is hypothesized that grain aggregation in the film increases with increasing annealing temperature. In the case of larger grains, the conductivity will also be easier [29,30].

Figure 1. Thin films of CoFeYb with XRD patterns. (a) RT, (b) following annealing at 100 °C, (c) following annealing at 200 °C, and (d) following annealing at 300 °C.

3.4. Nano-Indentation

The hardness and Young’s modulus of the CoFeYb thin films are shown to increase with thickness in Figure 5a,b. The Pharr–Oliver method is widely used to determine the hardness of a nano-indentation based on the loading and unloading curve, which exposes the combined hardness of the glass substrate and the CoFeYb thin film [31]. Because CoFeYb thin film is so thin, it is reasonable to expect a substrate effect in the nano-indentation measurement. According to Figure 5, the hardness and Young’s modulus of the as-deposited CoFeYb thin films increased from 8.35 GPa to 8.94 GPa and 89.3 GPa to 100 GPa, respectively. The hardness and Young’s modulus of the annealed 100 °C CoFeYb thin films increased from 8.52 GPa to 8.84 GPa and 90.2 GPa to 98.2 GPa, respectively. The hardness and Young’s modulus of the annealed 200 °C CoFeYb thin films increased from 8.68 GPa to 9.08 GPa and 94.1 GPa to 100.1 GPa, respectively. The hardness and Young’s modulus of the annealed 300 °C CoFeYb thin films increased from 8.63 GPa to 9.29 GPa and 94.8 GPa to 104.2 GPa, respectively. The findings of the experiment show that although temperature has little impact on hardness and Young’s modulus, thickness has a major impact. As thickness increased, both hardness and Young’s modulus significantly increased [32,33].
Figure 2. The relationship between the low-frequency alternate-current magnetic susceptibility ($\chi_{ac}$) and frequency, from 50 to 25,000 Hz. (a) RT, (b) following annealing at 100 °C, (c) following annealing at 200 °C, and (d) following annealing at 300 °C.

Figure 3. Maximum $\chi_{ac}$ for the CoFeYb thin films.

| Thickness | RT  | Annealing at 100 °C | Annealing at 200 °C | Annealing at 300 °C |
|-----------|-----|---------------------|---------------------|---------------------|
| 10 nm     | 50  | 100                 | 50                  | 50                  |
| 20 nm     | 50  | 50                  | 50                  | 50                  |
| 30 nm     | 50  | 50                  | 50                  | 50                  |
| 40 nm     | 50  | 50                  | 50                  | 50                  |
| 50 nm     | 50  | 50                  | 50                  | 50                  |

3.3. Electrical Properties

The resistivity and sheet resistance ($R_s$) with various thicknesses and temperature conditions are shown in Figure 4. According to the findings of this investigation, resistivity and sheet resistance ($R_s$) both reduced with increasing thickness and annealed temperatures. The sheet resistance decreased as the annealing temperature increased. It is hypothesized that grain aggregation in the film increases with increasing annealing temperature. In the case of larger grains, the conductivity will also be easier [29,30].
wettability. As a result, it is possible to conclude that the contact angle decreases as the annealed temperature rises. This is mostly due to the heat treatment increasing the size of the grains in the film. Higher grain size leads to a decrease in the support force between the grains. Surface energy and adhesion are important factors for CoFeYb film since it can be used as a seed or buffer layer. Liquid absorption is strong and the contact angle is minimal when the surface energy is large. Surface energy is calculated using the contact angle and Young's equation [22–24].

### Table 2. The optimal resonant frequency for various thicknesses.

| Thickness | RT | Annealing at 100 °C | Annealing at 200 °C | Annealing at 300 °C |
|-----------|----|---------------------|---------------------|---------------------|
| 10 nm     | 50 | 100                 | 50                  | 50                  |
| 20 nm     | 50 | 50                  | 50                  | 50                  |
| 30 nm     | 50 | 50                  | 50                  | 50                  |
| 40 nm     | 50 | 50                  | 50                  | 50                  |
| 50 nm     | 50 | 50                  | 50                  | 50                  |

Figure 4. (a) The resistivity of CoFeYb thin films, (b) the sheet resistance of CoFeYb films.

Figure 5. Nano-indentation of CoFeYb thin films. (a) Hardness and (b) Young’s modulus.

3.5. Surface Energy and Adhesion Analysis

Figure 6a–d depict the contact angles (θ) of Co60Fe20Yb20 thin films under four different conditions while utilizing DI water and glycerol. In particular, Co60Fe20Yb20 thin films were found to have contact angles that were consistently less than 90 degrees and drops that were almost spherical, showing that the films exhibited good hydrophilicity and wettability. As a result, it is possible to conclude that the contact angle decreases as the annealed temperature rises. This is mostly due to the heat treatment increasing the size of the grains in the film. Higher grain size leads to a decrease in the support force between the
grains. Surface energy and adhesion are important factors for CoFeYb film since it can be used as a seed or buffer layer. Liquid absorption is strong and the contact angle is minimal when the surface energy is large. Surface energy is calculated using the contact angle and Young’s equation [22–24].

Figure 6. Contact angles of four conditions: (A) RT, (B) following annealing at 100 °C, (C) following annealing at 200 °C, and (D) following annealing at 300 °C. DI water: (a) 10 nm, (b) 20 nm, (c) 30 nm, (d) 40 nm, and (e) 50 nm. Glycerol: (f) 10 nm, (g) 20 nm, (h) 30 nm, (i) 40 nm, and (j) 50 nm.

Figure 7 shows the surface energy of CoFeYb films under all conditions, including the thickness increase from 10 nm to 50 nm at RT after annealing at 100 °C, 200 °C, and 300 °C. The surface energy ranged from 22.3 to 31.3 mJ/mm², with post-annealed films having a higher surface energy than the as-deposited films. In this experiment, the surface energy of the 50 nm film after 300 °C annealing was the highest. Because of the crystallization effect, the annealing temperature rises, increasing surface energy and adhesion. The capacity of the surface to absorb liquid increased as surface free energy increased. The contact angle would also be reduced as a sizeable portion of the liquid would be absorbed [34]. Weak adhesion and low surface energy are associated with this [35]. When the films’ surface energies were higher, the adhesion was strongest. These results imply that it is easier to combine with the layer of the MTJ structure.

3.6. Analysis of Optical Property

Figure 8a–d show the transmittance (%) for CoFeYb thin films at visible wavelengths ranging from 500 nm to 800 nm (d). Figure 8a indicates that when the thickness increased
from 10 to 50 nm at RT, the transmittance (%) decreased from 48% to 14%. Figure 8b shows that following 100 °C annealing, the transmittance (%) decreased from 54% to 14% as the thickness changed from 10 to 50 nm. Figure 8c indicates that as the thickness increased from 10 to 50 nm, the transmittance (%) decreased from 52% to 11% following annealing at 200 °C. Figure 8d demonstrates that following annealing at 300 °C, the transmittance (%) decreased from 54% to 14% as thickness varied from 10 to 50 nm. The findings demonstrate that the thickness and interface effects attenuate the photon signal, decreasing transmittance and having an impact on the overall film optic transmittance properties [36,37].

Figure 7. Surface energy of CoFeYb thin films.

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Figure 8. Transmittance of CoFeYb thin films. (a) RT, (b) following annealing at 100 °C, (c) following annealing at 200 °C, and (d) following annealing at 300 °C.
4. Conclusions

This study focused on the structure, magnetic properties, electrical properties, nanomechanical characteristics, adhesion efficiency, and optical properties of Co$_{60}$Fe$_{20}$Yb$_{20}$ thin films. XRD analysis revealed that the Co$_{60}$Fe$_{20}$Yb$_{20}$ films deposited at RT, 100 $^\circ$C, and 200 $^\circ$C were amorphous. However, 40 nm and 50 nm CoFeYb thin films at 300 $^\circ$C exhibited the characteristic peak CoFe (110) at 44.7 $^\circ$, which increased with the thickness. The magnetic characteristics exhibited a thickness effect: as the thickness increased, the induced saturation magnetization of $\chi_{ac}$ increased. The electrical properties revealed that as thickness and annealed temperatures increased, resistivity and sheet resistance (Rs) decreased. The nano-mechanical characteristics rose in hardness and Young’s modulus as the thickness of the CoFeYb film increased, and there was a substrate effect in the nano-indentation test. The surface energy of the annealed CoFeYb thin films was greater than that of the as-deposited film. In terms of optical qualities, as thickness increased, transmittance fell, while the thickness effect and the interface effect suppressed the photon signal, causing the transmittance to decrease. As a result of this research, the film is appropriate for usage as a free layer of MTJ and can be used in MRAM and recording heads.

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