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Research Article

Magnetic Properties, Adhesion, and Nanomechanical Property of Co₄₀Fe₄₀W₂₀ Films on Si (100) Substrate

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In this study, a $Co_{40}Fe_{40}W_{20}$ alloy was sputtered onto Si (100) with thicknesses ($t_{\rm f}$) ranging from 18 to 90 nm, and the corresponding structure, magnetic properties, adhesive characteristics, and nanomechanical properties were investigated. X-ray diffraction (XRD) patterns of the $Co_{40}Fe_{40}W_{20}$ films demonstrated a significant crystalline body-centered cubic (BCC) CoFe (110) structure when the thickness was 42 nm, and an amorphous status was shown when the thickness was 18 nm, 30 nm, 60 nm, and 90 nm. The saturation magnetization (M_s) showed a saturated trend as t_f was increased. Moreover, the coercivity (H_c) showed a minimum 1.65 Oe with 30 nm. H_c was smaller than 4.5 Oe owing to the small grain size distribution and amorphous structure, indicating that the $\mathrm{Co_{40}Fe_{40}W_{20}}$ film had soft magnetism. The low-frequency alternating current magnetic susceptibility ($\chi_{\rm ac}$) decreased as the frequency was increased. The $\chi_{\rm ac}$ revealed a thickness effect when greater thicknesses had a large $\chi_{\rm ac}$. The maximum $\chi_{\rm ac}$ and optimal resonance frequency ($f_{\rm res}$) of Co₄₀Fe₄₀W₂₀ were investigated. The maximum $\chi_{\rm ac}$ indicated the spin sensitivity and was maximized at the optimal resonance frequency. The 90 mm thickness had the highest χ_{ac} 0.18 value at an $f_{\rm res}$ of 50 Hz. The contact angles of the ${\rm Co}_{40}{\rm Fe}_{40}{\rm W}_{20}$ films are less than 90°, which indicated that the film had a good wetting effect and hydrophilicity. The surface energy was correlated with the adhesion and displayed a concave-down trend. CoFeW films can be used as a seed or buffer layer; therefore, the surface energy and adhesion are very important. The highest surface energy was 30.12 mJ/mm² at 42 nm and demonstrated high adhesion. High surface energy has corresponding strong adhesive performance. The increased surface roughness can induce domain wall pinning effect and high surface energy, causing a high coercivity and strong adhesion. The increase of hardness and Young's modulus could be reasonably inferred from the thinner CoFeW films. The hardness and Young's modulus of CoFeW films are also displayed to saturated tendency when increasing thickness.

1. Introduction

In recent years, the direction of information storage is gradually developing to the field of magnetic recording, in which CoFe is one of the most studied alloys. In 2002, George et al. added 2% vanadium (V) to CoFe alloy and improved its brittleness to promote low-temperature application [1]. The CoFe layer can be a pinned layer or a free layer in the spin-valued magnetic tunneling junction (MTJ), which can be applied to the magnetoresistance random access memory

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(MRAM) and recording head applications [2-7]. Adding B and V elements to CoFe data has advantages in my previous work, including mechanical strength and magnetic properties [8]. At present, tungsten (W) is added to CoFe data to study its mechanical strength and magnetic properties. W is a hard metal with high melting temperature. In recent years, researchers have increased attention to adding a novel third element into the original CoFe matrix in magnetic fields [9-14]. However, few studies have focused on adding W to CoFe. In 2012, Pai et al. investigated the thickness phase transition of rare earth transition metal W [15]. In this reference, W is used as a seed layer to study the effect of MTJ spin Hall angle. In 2016, Ghaferi et al. studied the composition ratio of tungsten in CoFeW alloy with citrate borate solution, and the morphology, microstructure, and magnetic properties of these films at different pH values were analyzed [16]. The results show that the CoFeW alloy has low coercivity (H_c) due to the grain refinement. Sun et al. also provided an important discussion on CoFeW ternaries based on oxygen evolution reactions [17]. Compared with other materials, CoFe alloy has high saturation magnetization (M_s) [18–21]. However, adding W as the third element can improve the mechanical properties of CoFe alloy [22]. In addition, a CoFeW layer can be combined with the magnetic process and be compatible with other layers in bilayered or multilayered systems. The adhesion of CoFeW film is an important characteristic of its compatibility with other films. For these reasons, it is of great significance to study the magnetic properties, adhesion, and nanomechanical properties of CoFeW films by sputtering deposition technology.

2. Experimental Details

CoFeW was deposited onto a Si (100) substrate at room temperature (RT) in thicknesses (t_f) ranging from 18 to 90 nm using the direct current (DC) method at a power of 50 W. Moreover, the power density was 1.65 W/cm², and the deposition rate was 1.2 nm/min. The geometry of the deposition system is square, and the target-substrate distance is approximately 15 cm. The target composition of the CoFeW alloy was 40 at% Co, 40 at% Fe, and 20 at% W. The typical base chamber pressure was 2.6×10^{-7} Torr, and the Ar working chamber pressure was 3×10^{-3} Torr. The structure of Co₄₀Fe₄₀W₂₀ thin films was detected by X-ray diffraction (XRD) of $CuK_{\alpha 1}$ (Philips X'pert). In addition, the in-plane low-frequency alternate-current magnetic susceptibility (χ_{ac}) of $\mathrm{Co_{40}Fe_{40}W_{20}}$ was studied using a χ_{ac} analyzer (XacQuan). Firstly, the χ_{ac} analyzer with external magnetic field is used to calibrate the reference standard sample. Then, insert the tested sample into the $\chi_{\rm ac}$ analyzer. The driving frequency is between 10 and 25000 Hz. The χ_{ac} was determined by magnetization measurement. All tested samples have the same shape and size to eliminate demagnetization factors. The χ_{ac} valve is an arbitrary unit (a.u.), because the alternating current result corresponds to the reference standard sample and is a comparative value. The χ_{ac} analyzer measured the relationship between susceptibility and frequency. The optimal resonance frequency ($f_{\rm res})$ is measured by the $\chi_{\rm ac}$ analyzer, which repre-

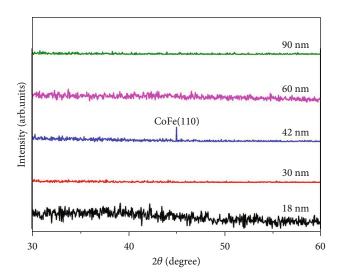


FIGURE 1: XRD results for Co₄₀Fe₄₀W₂₀ films.

sents the frequency of the maximum $\chi_{\rm ac}$. The in-plane hysteresis loop of the Co40Fe40W20 films was obtained by an alternating gradient magnetometer (AGM). Finally, using deionized (DI) water and glycerol as experimental liquids, the surface energy of Co₄₀Fe₄₀W₂₀ thin films was calculated by measuring the contact angle [23-25]. It is defined as the surface excess-free energy of a specific crystal surface area [26]. To investigate the relation of coercivity (H_c) and surface energies, the surface roughness of CoFeW films was examined by atomic force microscopy (AFM). Hardness and Young's modulus of the CoFeW thin films were measured using an MTS Nano Indenter XP with a Berkovich indenter. An indentation load of 1 mN was used to limit the depth of penetration of the indenter to less than 10% of the film thickness. The indentation load was increased in 40 steps, and the penetration depth was measured at each step. Six indentations were investigated in each sample and averaged with a standard deviation for more accurate results.

3. Results and Discussion

3.1. Structure Property. Figure 1 shows the X-ray diffraction (XRD) of the $Co_{40}Fe_{40}W_{20}$ film. Because the intensity of the Si (100) peak on the single crystal substrate is stronger than that of the CoFeW crystallization, the XRD is presented at diffracted angle (2 θ) between 30 and 60 degrees. An especially high crystalline body-centered cubic (BCC) CoFe (110) peak occurred at 42 nm because it can be reasonably concluded that it had a continuous mode of film growth and induced strong crystallization. The CoFeW films showed an amorphous status when the thicknesses at 18 nm, 30 nm, 60 nm, and 90 nm. It can also be reasonably concluded that the Co₄₀Fe₄₀W₂₀ thickness will have discontinuous growth model and random arrangement of atoms, leading to amorphous state [27]. The 42 nm of the CoFeW thin film has a strong crystallization, while the other thicker and thinner films are amorphous. It can be reasonable that this sputtering

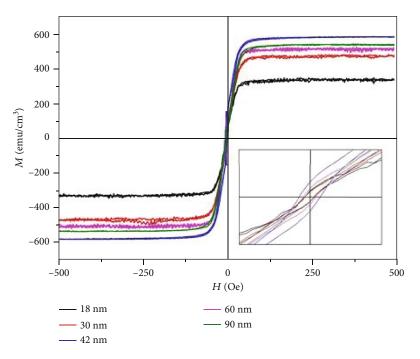


Figure 2: In-plane magnetic hysteresis loop of Co₄₀Fe₄₀W₂₀ thin films.

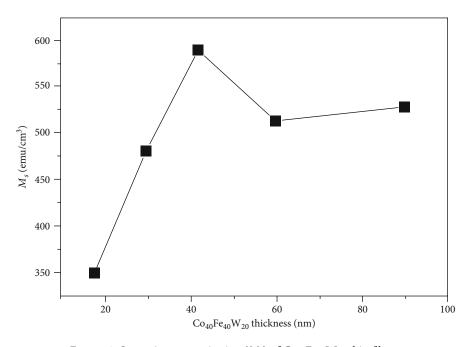


Figure 3: Saturation magnetization $(M_{\rm s})$ of ${\rm Co_{40}Fe_{40}W_{20}}$ thin films.

condition of 42 nm may be slightly different from other films and induces this result [28].

3.2. Magnetic Properties. Figure 2 depicts the hysteresis loop of the $\mathrm{Co_{40}Fe_{40}W_{20}}$ film using AGM measurement. The inplane magnetic field (H_{ext}) of 500 Oe is enough to detect the saturated magnetic spin state. The results also show that the $\mathrm{Co_{40}Fe_{40}W_{20}}$ film has low coercivity (H_{c}) and soft magnetic properties.

The saturation magnetization $(M_{\rm s})$ of the ${\rm Co_{40}Fe_{40}W_{20}}$ film is shown in Figure 3. The results show that there is a significant relationship between $M_{\rm s}$ and thickness. When the film thickness increases from 18 nm to 90 nm, $M_{\rm s}$ shows a saturated trend, which shows the thickness effect of $M_{\rm s}$ in the ${\rm Co_{40}Fe_{40}W_{20}}$ film. The maximum $M_{\rm s}$ at 42 nm is about 590 emu/cm³ owing to strong magneto-crystalline anisotropy, which indicates that it has high spin coupling strength and can induce large $M_{\rm s}$.

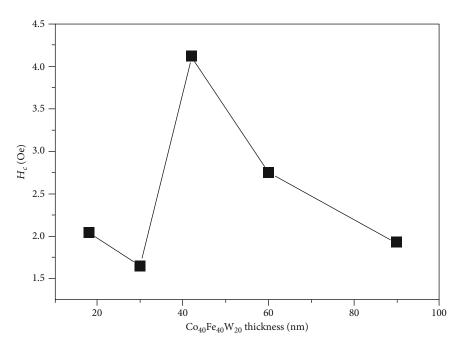


Figure 4: Coercivity (H_c) of $Co_{40}Fe_{40}W_{20}$ thin films.

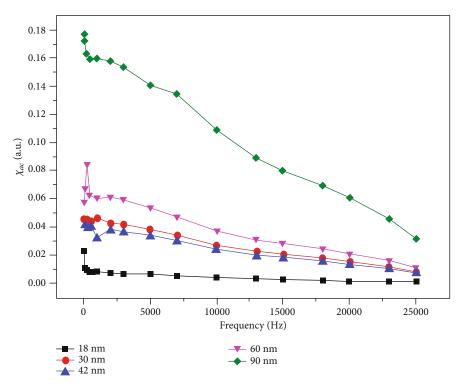


Figure 5: The low-frequency alternate-current magnetic susceptibility (χ_{ac}) as a function of the frequency from 10 to 25,000 Hz.

The corresponding $H_{\rm c}$ is shown in Figure 4. When $t_{\rm f}$ is in the range from 18 to 30 nm, $H_{\rm c}$ decreases from 2.04 Oe to 1.65 Oe. When $t_{\rm f}$ is in the range from 30 to 42 nm, $H_{\rm c}$ increases from 1.65 Oe to 4.12 Oe. Finally, $H_{\rm c}$ decreased to 1.93 Oe at 90 nm. The maximum $H_{\rm c}$ was 4.12 Oe at 42 nm

due to the pinning effect of the domain wall, which was not easy to move, resulting in the increase in coercivity [29, 30]. The amorphous structure of the $\mathrm{Co_{40}Fe_{40}W_{20}}$ films indicated a smaller H_{c} due to the small grain size distribution [31, 32]. A strong crystalline peak occurred at 42 nm in XRD result.

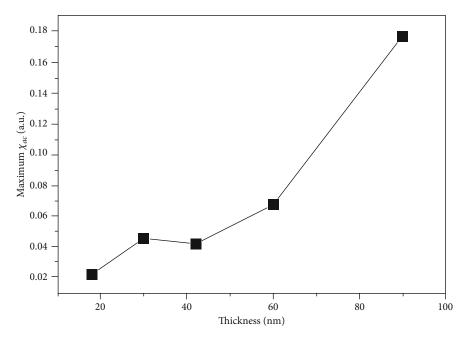


FIGURE 6: Maximum alternate-current magnetic susceptibility for the CoFeW films.

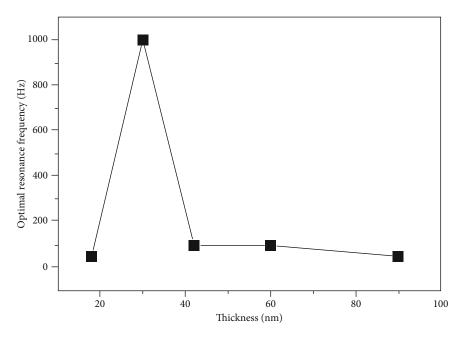


FIGURE 7: Optimal resonance frequency for films of various thicknesses.

According to Scherrer's formula (1), the average grain size (*D*) can be estimated from the full width measured at the half-height width (FWHM, *B*) of the 42 nm diffraction peak. Scherrer's formula is [33]

$$D = \frac{k\lambda}{B\cos\theta}.$$
 (1)

In the formula, k (0.89) is the Scherrer's constant, λ is the X-ray wavelength of the $\text{CuK}_{\alpha 1}$ line, B is the

FWHM of the 42 nm diffraction peak, and θ is the half angle of the diffraction peak. This formula shows that D is proportional to 1/B, so smaller B corresponds to larger grains. The 42 nm of $\mathrm{Co_{40}Fe_{40}W_{20}}$ thin film has a smaller B and can induce larger grains to be deposited. Furthermore, the large grain size distribution has higher H_{c} [34, 35].

The $H_{\rm c}$ value is lower than 4.5 Oe, which indicates that the ${\rm Co_{40}Fe_{40}W_{20}}$ film has soft magnetic properties and is suitable for MRAM and magnetic head applications.

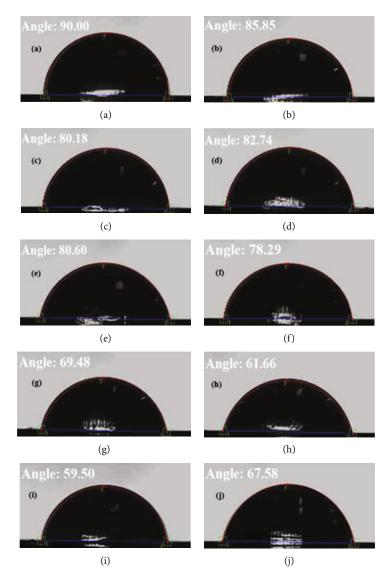


FIGURE 8: Contact angles of the $Co_{40}Fe_{40}W_{20}$ thin films with DI water: (a) 18 nm, (b) 30 nm, (c) 42 nm, (d) 60 nm, and (e) 90 nm. Contact angles of the $Co_{40}Fe_{40}W_{20}$ thin films with glycerol: (f) 18 nm, (g) 30 nm, (h) 42 nm, (i) 60 nm, and (j) 90 nm.

Figure 5 shows the $\chi_{\rm ac}$ results of the CoFeW film. The results show that the $\chi_{\rm ac}$ value increases with the increase of $t_{\rm f}$ in the range of 18 nm to 90 nm. With the increase of measurement frequency, $\chi_{\rm ac}$ decreases sharply.

The maximum $\chi_{\rm ac}$ corresponding to different thickness of CoFeW is shown in Figure 6. Due to the thickness effect, when $t_{\rm f}$ increases from 18 nm to 90 nm, the maximum $\chi_{\rm ac}$ increases from 0.02 to 0.18. The results show that the spin sensitivity is the strongest at the optimal resonance frequency ($f_{\rm res}$). The maximum $\chi_{\rm ac}$ at the optimal resonance frequency has the following physical significance. At low frequency, the alternating-current dipole moment is caused by the volume dipole moment oscillation in each domain. The external alternating-current magnetic field plays a driving role. The recovery of the magnetic interaction occurred between domains. A resonance frequency acted as the driving force acting on the system. Therefore, the peak frequency of the

low frequency susceptibility corresponds to the resonance frequency of the pole moment oscillation. In frequency, χ_{ac} peak represents spin exchange coupling interaction and domain dipole moment [8, 36].

The optimal resonance frequency ($f_{\rm res}$) of CoFeW film is shown in Figure 7. At this frequency, the maximum $\chi_{\rm ac}$ is measured with the strongest spin sensitivity [36, 37]. The $f_{\rm res}$ shows a downward concave trend with a maximum critical frequency of 1000 Hz. The $f_{\rm res}$ values of all CoFeW thicknesses are less than 1000 Hz, indicating that the film is advantageous to the application of sensors, transformers, and low-frequency magnetic recording media.

3.3. Analysis of Surface Energy and Adhesion. Figure 8 illustrates the contact angle of CoFeW films using deionized (DI) water and glycerin. The results show that the water droplets on the CoFeW film are nearly spherical and the

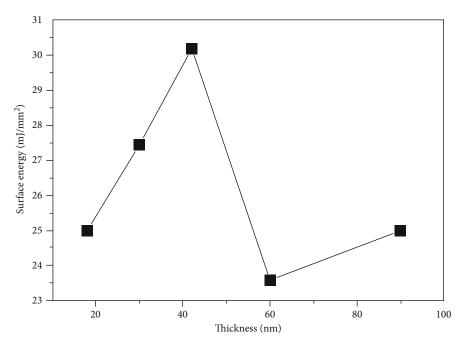


Figure 9: Surface energy of Co₄₀Fe₄₀W₂₀ thin films.

contact angle is less than 90° , indicating that the film has good wettability and hydrophilicity. The CoFeW film can be used as a seed layer or a buffer layer, and its surface energy and adhesion are very important. When the surface energy is high, the liquid absorption is large and the liquid absorption area is large, resulting in the decrease of the contact angle [8, 38].

Figure 9 shows the calculated surface energy using the contact angle [23–25]. Due to the larger pinning effect of domain walls, the surface energy results show a downward concave trend, and the maximum critical value at 42 nm is $30.12 \, \text{mJ/mm}^2$ [28, 29]. Low surface energy corresponds to weak adhesion [39]. Therefore, the adhesion of 42 nm film is higher than that of other $\text{Co}_{40}\text{Fe}_{40}\text{W}_{20}$ films.

3.4. Analysis of Surface Roughness. To confirm the relation of coercivity and surface energies, the surface roughness of CoFeW films was detected by AFM, which is shown in Figures 10(a)-10(e). When the thickness is increased from 18 nm to 90 nm, the root mean square values (R_q) of surface roughness are 0.41 nm, 0.35 nm, 0.63 nm, 0.59 nm, and 0.47 nm, respectively. The increased surface roughness can lead to the pinning effect of the domain wall, which is not easy to move, resulting in the increase in coercivity [40, 41]. Moreover, the rough surface roughness can be induced high surface energy, causing a high adhesion [42, 43]. This result is consistent with Figures 4 and 9.

3.5. Analysis of Nanoindentation. Figures 11(a) and 11(b) show that the hardness and Young's modulus increase with the increase of the thickness of the CoFeW film. In general, nanoindentation hardness is determined from the loading unloading curve by the Pharr-Oliver method [44], which indicates the mixed hardness of silicon substrate and CoFeW

film. Because the thicknesses of CoFeW films are too thin, it can be reasonably concluded that the substrate effect must exist in nanoindentation measurement. The corresponding hardness and Young's modulus values of the substrate are 4.12 GPa and 133.3 GPa in the nanoindentation measurement. To reduce the silicon substrate effect, the experimental results carried out for this work average value and standard deviation must be presented in the error bar in Figures 11(a) and 11(b). When the thickness increased from 18 nm to 90 nm, the hardness and Young's modulus of CoFeW films increased from 4.02 GPa to 10.94 GPa and 132.1 GPa to 186.3 GPa, respectively. The hardness and Young's modulus of CoFeW films are also displayed to saturated tendency when the thickness is from 18 nm to 90 nm. According to the result, the Young's modulus of adding the W effect to CoFe films in thicker films is larger than that of the CoFe film. The influence of thinner CoFeW films on the substrate is more significant, which is consistent with the phenomenon [45].

4. Conclusions

XRD patterns of the $\mathrm{Co_{40}Fe_{40}W_{20}}$ films demonstrated a significant crystalline body-centered cubic (BCC) CoFe (110) structure when the thickness was 42 nm. The other films showed an amorphous status. The highest χ_{ac} value was 0.18 at 90 nm at an f_{res} value of 50 Hz. The f_{res} value was less than 1000 Hz at all thicknesses, demonstrating that the CoFeW films were suitable for magnetic component applications in low-frequency environments. The H_{c} indicated that the critical thickness was 42 nm due to a greater pinning site effect, which induced high H_{c} and strong adhesion. The H_{c} value was smaller than 4.5 Oe, owing to small a grain size distribution and amorphous structure and indicating that

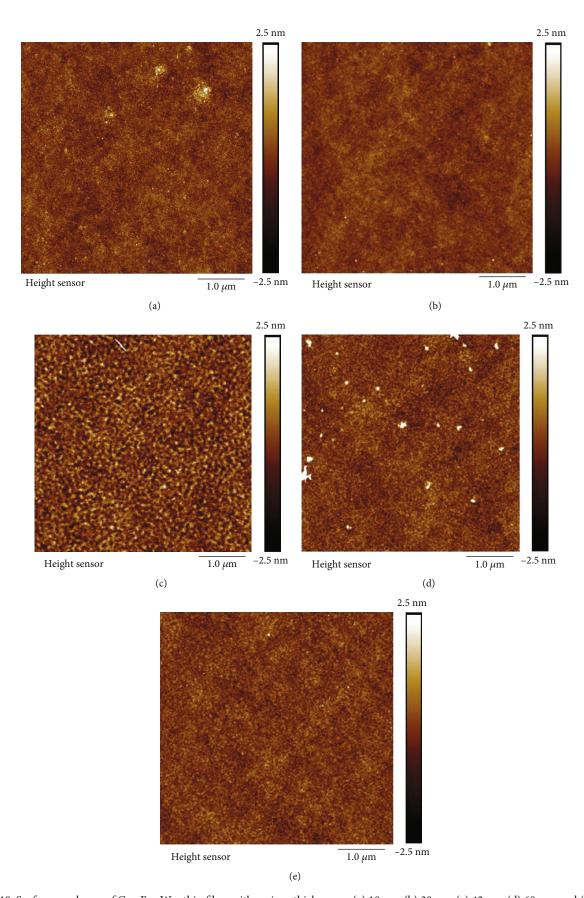


Figure 10: Surface roughness of $Co_{40}Fe_{40}W_{20}$ thin films with various thicknesses: (a) 18 nm, (b) 30 nm, (c) 42 nm, (d) 60 nm, and (e) 90 nm.

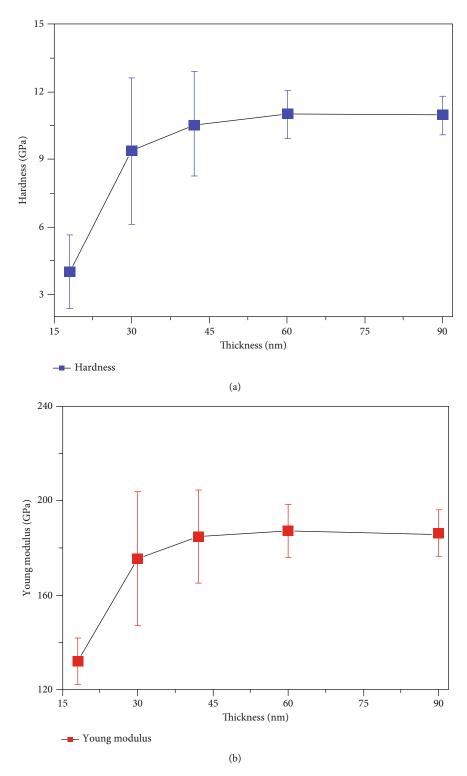


Figure 11: The analysis of nanoindentation of $Co_{40}Fe_{40}W_{20}$ thin films: (a) hardness and (b) Young's modulus.

the $\mathrm{Co_{40}Fe_{40}W_{20}}$ films had soft magnetism. Furthermore, the contact angles of the $\mathrm{Co_{40}Fe_{40}W_{20}}$ films were smaller than 90°, indicating that the films were hydrophilic. The surface energy had a downward concave feature, and the critical thickness was 42 nm. The high surface energy corresponded

to strong adhesion. The increased surface roughness can induce domain wall pinning effect and high surface energy, causing a high coercivity and strong adhesion. Furthermore, the hardness and Young's modulus of CoFeW films are also displayed to saturated tendency when increasing thickness.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Disclosure

The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

Conflicts of Interest

The authors declare that there is no conflict of interests regarding the publication of this paper.

Authors' Contributions

W.-J.L., S.-L.O., and Y.-T.C. are responsible for the conceptualization. Y.-T.C., Y.-C.L., C.-Y.C., and C.-C.C performed the methodology. Y.-H.C., Y.-C.L., and C.-Y.C. are involved in the validation and formal analysis. Y.-T.C. and W.-J.L. did the the investigation. T.-H.W. acquired the resources. Y.-T.C. is responsible for the original draft preparation. Y.-T.C. and W.-J.L. reviewed and edited the manuscript. Y.-T.C. and S.-L.O. supervised the study. Y.-T.C. and T.-H.W. are assigned to the project administration. W.-J.L. and Y.-H.C are involved in the funding acquisition. All authors have read and agreed to the published version of the manuscript.

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