

Article

Co₄₀Fe₄₀Y₂₀ Nanofilms' Structural, Magnetic, Electrical, and Nanomechanical Characteristics as a Function of Annealing Temperature and Thickness

Wen-Jen Liu ¹, Yung-Huang Chang ², Chia-Chin Chiang ³ , Yuan-Tsung Chen ^{4,*} , Yu-Chi Liu ⁴, Sin-Liang Ou ⁵, Sin-Yan Li ⁴ and Po-Wei Chi ⁶

¹ Department of Materials Science and Engineering, I-Shou University, Kaohsiung 840, Taiwan

² Bachelor Program in Interdisciplinary Studies, National Yunlin University of Science and Technology, Douliou 64002, Taiwan

³ Department of Mechanical Engineering, National Kaohsiung University of Science and Technology, 415 Chien Kung Road, Kaohsiung 807, Taiwan

⁴ Graduate School of Materials Science, National Yunlin University of Science and Technology, Douliou 64002, Taiwan

⁵ Bachelor Program for Design and Materials for Medical Equipment and Devices, Da-Yeh University, Changhua 51591, Taiwan

⁶ Institute of Physics, Academia Sinica, Taipei 11529, Taiwan

* Correspondence: ytchen@yuntech.edu.tw; Tel.: +886-5-534-2601

Abstract: To investigate the correlations between different thicknesses and heat treatments, this study used a sputtering method to create CoFeY films. The results of X-ray diffraction (XRD) revealed the appearance of oxide peaks at $2\theta = 47.7^\circ$, 54.5° , and 56.3° in agreement with YFeO₃ (212), Co₂O₃ (422), and Co₂O₃ (511), respectively. The findings also demonstrated a relationship between the low-frequency alternating-current magnetic susceptibility (χ_{ac}) values and the thickness of the CoFeY thin films. At a thickness of 50 nm and an annealing temperature of 300 °C, the ideal value of χ_{ac} was 0.159. The presence of Y and the thickness impact were both evident in the χ_{ac} value, which improved spin-exchange coupling as well as grain refining. With increasing thickness, the resistance decreased. At 300 °C and 40 nm in thickness, this film has a maximum surface energy of 31.2 mJ/mm². The hardness of the 50-nm films reached a maximum of 16.67 GPa when annealed at 100 °C. Due to the high χ_{ac} , strong adhesion, good nanomechanical properties, and low resistivity, the optimal conditions were determined to be 50 nm with annealing at 300 °C.

Keywords: low-frequency alternating-current magnetic susceptibility (χ_{ac}); optimal resonance frequency (f_{res}); electrical properties; nanomechanical properties



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1. Introduction

CoFe alloys have excellent mechanical properties, high Curie temperatures (T_c), and low angular stiffness [1–4]. They are used in magnetic read heads, magnetoresistance random access memory (MRAM), and other applications [5,6]. In the region of the magnetic read head, high saturation magnetization strength (M_s), low coercivity (H_c), and magnetic anisotropy are necessary [7]. CoFe alloys have the problem of losing their magnetic anisotropy at high temperatures. However, when applied to high-temperature devices, adding a third element can solve this problem [8]. The rare-earth element yttrium (Y) was historically used in the red emitters of cathode ray tubes. The addition of Y to alloys can lessen the difficulties of processing while also enhancing their mechanical qualities, resistance to corrosion, thermal stability, and resistance to high-temperature oxidation [9–13]. When pure Y is exposed to air, it spontaneously forms yttrium oxide (Y₂O₃) with high thermal stability, and excellent mechanical, optical, and electrical properties [14]. Y₂O₃ films are often used in thermal barrier coatings and can be used as light antireflection

coatings [15–17]. Compounds are formed by combining rare-earth elements and transition metals such as iron and cobalt, and it is worthwhile to explore the performance of the resulting compounds. CoFeY alloys are frequently utilized in the magnetic-recording-head sector for write poles, yokes, and shields [18–20]. In more recent nanofilm literatures, the effects of thickness and annealing temperature on characteristics have been investigated [21,22]. The use of nanofilms is a fascinating topic with many potential applications. Particularly, exotic magneto-electronic properties of nanostructured magnetic thin films were discovered. Due to their ability to produce large positive magnetoresistance (MR), these materials are appealing for spintronic and magnetic applications. Therefore, in this study, a CoFeY film was formed on an Si substrate using a sputtering device, which was then heat-treated in a vacuum annealing furnace to investigate the influence of annealing temperature and thickness on structural, magnetic, electrical, and nanomechanical properties. In this study, numerous causes were further studied, and the structural, magnetic, adhesive, and mechanical properties were examined in relation to thickness and annealing temperature.

2. Materials and Methods

CoFeY was deposited onto substrates made of Si(100) which ranged in thickness from 10 nm to 50 nm using a direct current (DC) magnetron sputtering process under following four conditions: (a) maintaining the as-deposited films at room temperature (RT), (b) annealing for an hour at a processing temperature (T_A) of 100 °C, (c) annealing for an hour at a processing temperature (T_A) of 200 °C, and (d) annealing for an hour at a processing temperature (T_A) of 300 °C. While the Ar operating pressure was 3×10^{-3} Torr, the chamber pressure was 3×10^{-7} Torr. A total of 2.5×10^{-3} Torr of pressure was applied during the use of Ar gas for the annealing process. The requisite alloy composition of the CoFeY was chosen to be 40%, 40%, and 20% for Co, Fe, and Y, respectively. In order to determine the precise thickness, high-resolution cross-sectional field emission scanning electron microscopy (SEM, Hitachi SU 8200, Tokyo, Japan) was employed to investigate the calibration thickness of the matching sputtering period. Grazing incidence X-ray diffraction (GIXRD) patterns using CuK α 1 (PAN analytical X'pert PRO MRD, Almelo, The Netherlands) and a low-angle diffraction incidence of approximately two degrees were used to determine the structure. The in-plane low-frequency alternate-current magnetic susceptibility (χ_{ac}) was measured using an χ_{ac} analyzer (XacQuan, MagQu Co., Ltd., New Taipei, Taiwan) with driving frequencies ranging from 10 to 25,000 Hz. The four-point probe technique (Sadhudesign, Hsinchu, Taiwan) was used to measure electrical characteristics. Utilizing glycerol and deionized (DI) water, the contact angles (CAM-110, Creating Nano Technologies, Tainan, Taiwan) were measured. Through calculations, the surface energy from the contact angle was determined [23–25]. An MTS (MTS, Minneapolis, MN, USA) Nano Indenter XP with a Berkovich indenter was used to test hardness and Young's modulus. The indenter's depth of penetration was kept to less than 10% of the film thickness using an indentation load of 1 mN. At each of the 40 steps in increasing the indentation load, the penetration depth was recorded. Each sample contained six indentations that were examined, and the results were averaged with a standard deviation for more precise analysis.

3. Results

3.1. X-ray Diffraction Structure

Figure 1a–d depicts the XRD patterns of Co₄₀Fe₄₀Y₂₀ nanofilms with thicknesses ranging from 10 nm to 50 nm, both as-deposited and after annealing at 100, 200, and 300 °C. No characteristic peaks for CoFeY were found, but all oxides were found. For all CoFeY samples, oxide peaks emerged at $2\theta = 47.7^\circ$, 54.5° , and 56.3° , corresponding to YFeO₃ (212), Co₂O₃ (422), and Co₂O₃ (511), respectively. However, even though the chamber was evacuated to 10^{-7} Torr, oxygen may still have been present. Native oxides on the Si (100) substrate and oxygen impurities on the sputtering target both contributed to the

establishment of the oxidation peak [26]. Another possible reason for the formation of oxides is the affinity and negative electric degree relationship between the elements [27]. The intensity of all oxide peaks dropped as the thickness of CoFeY increased. The thin layers of the CoFeY were believed to have an oxide thickness that was essentially the same. The strength of each oxide peak dropped as the CoFeY thickness rose. Through a reduction in interference, the waning oxide peak might improve the magnetic and electrical properties.

3.2. SEM Image

Images of the samples taken with a high-resolution cross-sectional field emission scanning electron microscope (SEM) after they had been deposited and annealed at 350 °C are shown in Figure 2a,b. According to the SEM findings, the annealed CoFeY film is more dense than the film in its as-deposited condition. After annealing, the CoFeY thin film thickness somewhat decreased.

3.3. Magnetic Analysis

Low-frequency alternating-current magnetic susceptibility (χ_{ac}) graphs are depicted in Figure 3a with function logarithmic frequencies at 10–50 nm. Figure 3b–d display the annealed χ_{ac} which were annealed for one hour at 100, 200 and 300 °C in comparison to a logarithmic frequency range for each thickness of the CoFeY thin film. CoFeY thin films ranged in thickness from 10 to 50 nm, and under each of the four conditions, the amplitude of the χ_{ac} signal decreased during the course of the high-frequency period.

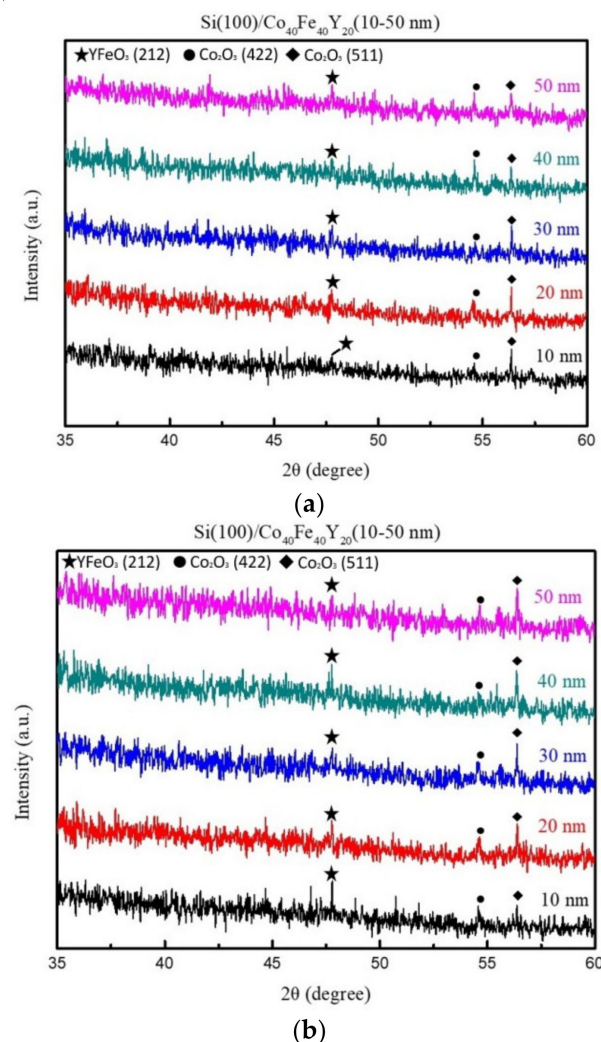


Figure 1. Cont.

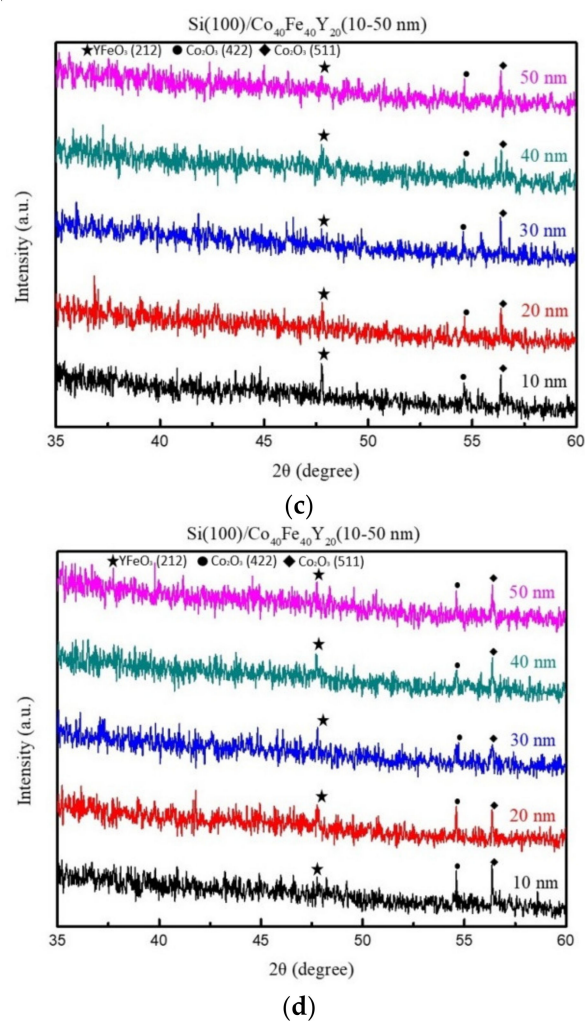
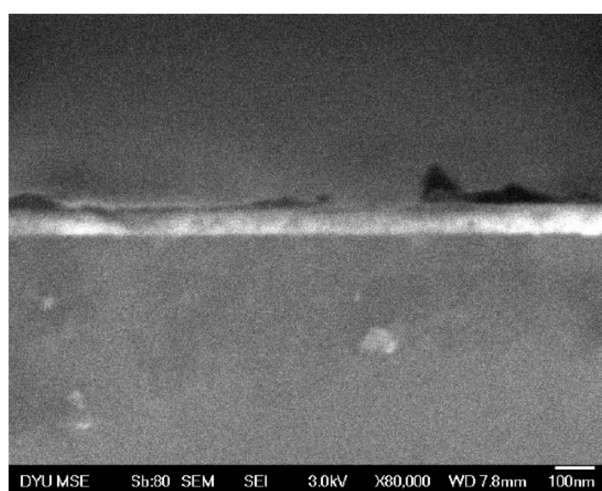
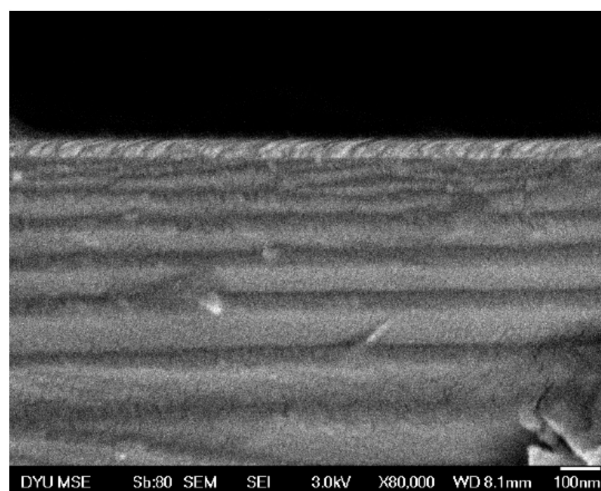


Figure 1. Patterns produced using XRD. (a) RT, (b) after 100 °C annealing, (c) after 200 °C annealing, and (d) after 300 °C annealing.



(a)



(b)

Figure 2. Cross-sectional SEM images of CoFeY at 40 nm. (a) RT and (b) annealed at 350 °C.

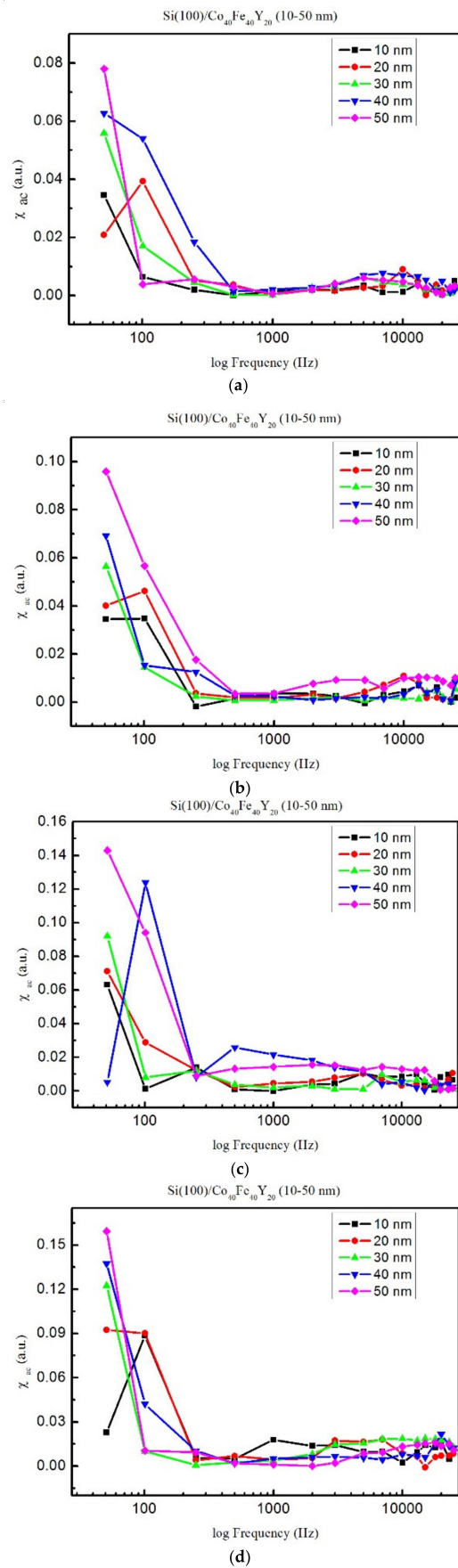


Figure 3. The relationship between the χ_{ac} and frequency. (a) RT, (b) after 100 °C annealing, (c) after 200 °C annealing, and (d) after 300 °C annealing.

Figure 4 and Table 1 show the maximum χ_{ac} values for the four different temperatures and thicknesses of the CoFeY films, respectively. The results showed that as thickness increased, all χ_{ac} values increased. The maximum value of χ_{ac} with a thickness of 50 nm was 0.078, 0.095, 0.142, and 0.159 when χ_{ac} was deposited in its as-deposited state and annealed at 100, 200 and 300 °C. Due to the thickness effect and Y inclusion, the maximum value of χ_{ac} increased with the thickness of the CoFeY film, which improved the coherence of the spin exchange and grain refining [28]. After annealing, the maximum χ_{ac} value was greater than the value of the deposited samples. Table 2 shows that the CoFeY films' ideal resonance frequencies (f_{res}) were in the low-frequency region of 50–100 Hz, indicating that χ_{ac} had the greatest spin sensitivity at this frequency. The conclusion that the film is acceptable for employment as a low-frequency magnetic recording medium may be drawn from the fact that the f_{res} was less than 100 Hz.

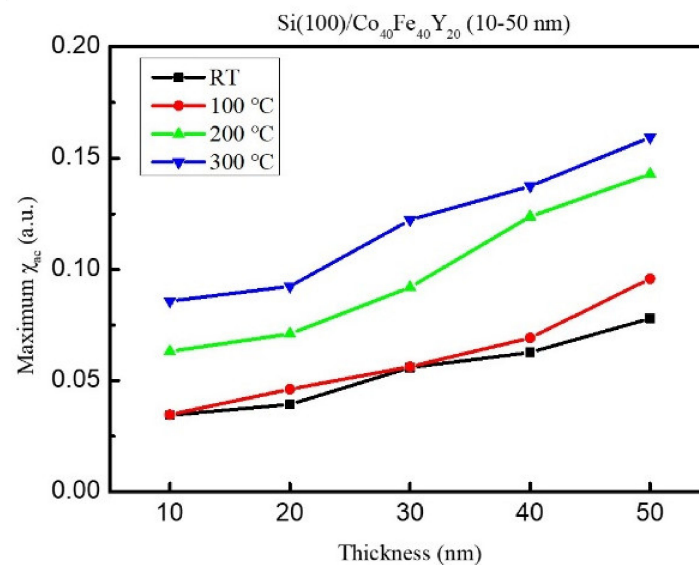


Figure 4. Maximum χ_{ac} of CoFeY films under four conditions.

Table 1. Maximum χ_{ac} value under four conditions.

Film Thickness	RT Maximum (a.u.)	100 °C Maximum (a.u.)	200 °C Maximum (a.u.)	300 °C Maximum (a.u.)
10 nm	0.0345	0.0348	0.0631	0.0858
20 nm	0.0394	0.0462	0.0711	0.0924
30 nm	0.0559	0.0564	0.092	0.1223
40 nm	0.0627	0.0693	0.1238	0.1374
50 nm	0.0780	0.0959	0.1429	0.1594

Table 2. Resonance frequency that works best for films of different thicknesses.

Film Thickness	RT Frequency(Hz)	100 Frequency(Hz)	200 °C Frequency(Hz)	300 °C Frequency(Hz)
10 nm	50	100	50	100
20 nm	100	100	50	50
30 nm	50	50	50	50
40 nm	50	50	100	50
50 nm	50	50	50	50

3.4. Electrical Properties

The resistivity of the CoFeY films in the four conditions is depicted in Figure 5a at various thicknesses. The sheet resistance of CoFeY films at various temperatures and thicknesses is depicted in Figure 5b. Accordingly, thinner films showed lower resistivities and sheet resistance amplitudes. In direct proportion to the rise in film thickness, the resistivity and sheet resistance decreased. The electronic conductivity was enhanced by increasing the thickness of the grain size and reducing the obstacles impeding electron migration [29,30].

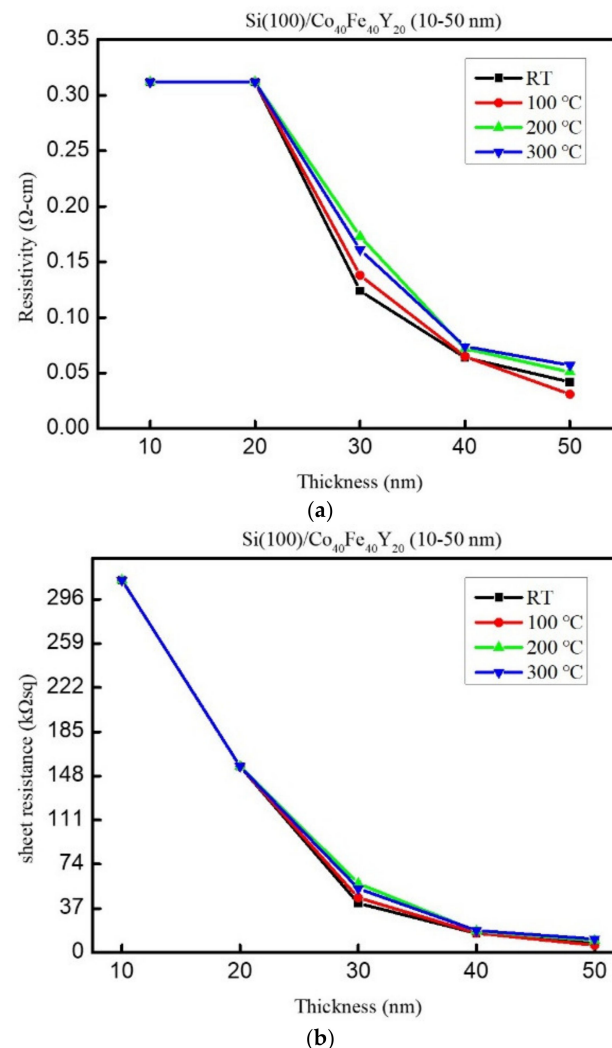


Figure 5. (a) The resistivity and (b) the sheet resistance of CoFeY films.

3.5. Surface Energy and Adhesion Evaluation

Figure 6a–d show the results of the contact-angle measurements using DI water and glycerol (d). The droplets were seen to be virtually spherical in all circumstances and the contact angle was less than 90° . The film's hydrophilicity and wettability were both good, according to this finding. The data suggest that the contact angle reduced as the annealing temperature rose, which is a logical inference. Similar to C60 and ZnO materials, it displays this behavior [31–33]. This discovery was mostly caused by the increased crystallization that occurred during the annealing process. Surface energy and adhesion are important factors to take into account when using CoFeY films as seed or buffer layers. Higher liquid absorption and smaller contact angles are produced by higher surface energy. The hydrophilicity of the layer on the Si (100) substrate was demonstrated by the adhesion effectiveness and contact angle, both of which were smaller than 90° .



Figure 6. Contact angles of the CoFeY following four situations: (a) RT, (b) after annealing at 100 °C, (c) after annealing at 200 °C, and (d) after annealing at 300 °C.

Figure 7 displays the surface energy of the CoFeY films for each situation. The film that had been annealed had more surface energy than the film that had just been deposited. A layer that was 40 nm thick and annealed at 300 °C had the highest surface energy at 31.1 mJ/mm². Higher surface energy results in stronger film adhesion. According to the aforementioned results, all samples had contact angles that were lower than 90°, indicating that they were all hydrophilic films with good wetting capabilities. In addition, there was evidence of a strong correlation between the surface energy and the contact angle. The area and volume of liquid absorbed increased and the contact angles decreased proportionally when the surface free energy was high [34].

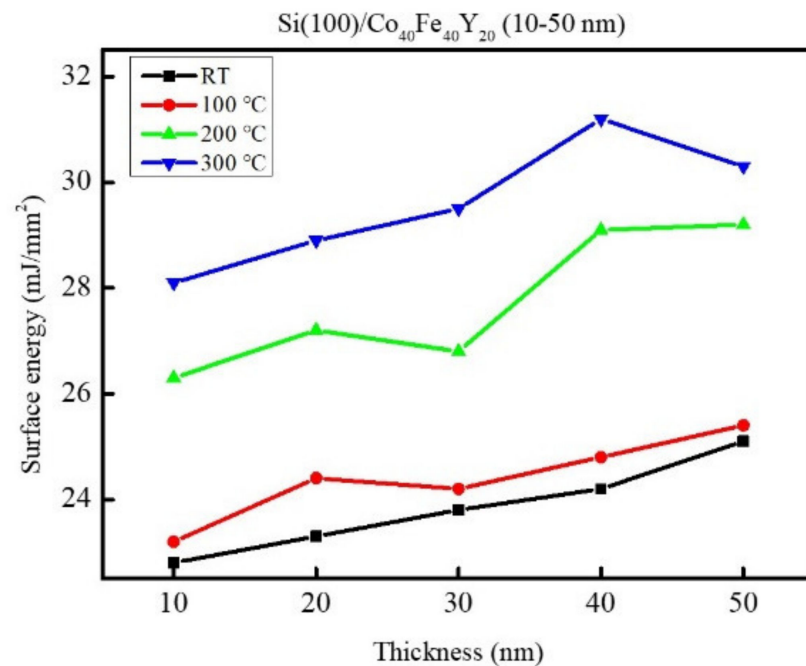


Figure 7. Surface energy of CoFeY films under four conditions.

3.6. Hardness and Young's Modulus

The hardness is displayed in four different situations in Figure 8a. Initial-coating hardness values of 13.5 and 16.55 GPa, respectively, were found for films that were 10 and 50 nm. At 100 °C, the hardness of the 10-nm and 50-nm-thick films was 13.98 and 16.67 GPa. For the 10-nm and 50-nm thick films, the hardness was 13.38 GPa and 16.35 GPa at 200 °C and 14.00 GPa and 16.27 GPa at 300 °C, respectively. An increasing trend in hardness was observed with the increasing film thickness, and the maximum value was observed at 100 °C for the film with a thickness of 50 nm. Figure 8b depicts a graph of the Young's modulus of the CoFeY films, which exhibits an increasing trend with increasing thickness that progressively leveled out, with a maximum value of 235.9 GPa for the 50 nm thick as-deposited film. The experimental results revealed that the hardness and elastic modulus of the Si (100) substrate did not vary considerably with temperature; nevertheless, when the film thickness grew, the hardness and elastic modulus of the Si (100) substrate increased dramatically [35,36].

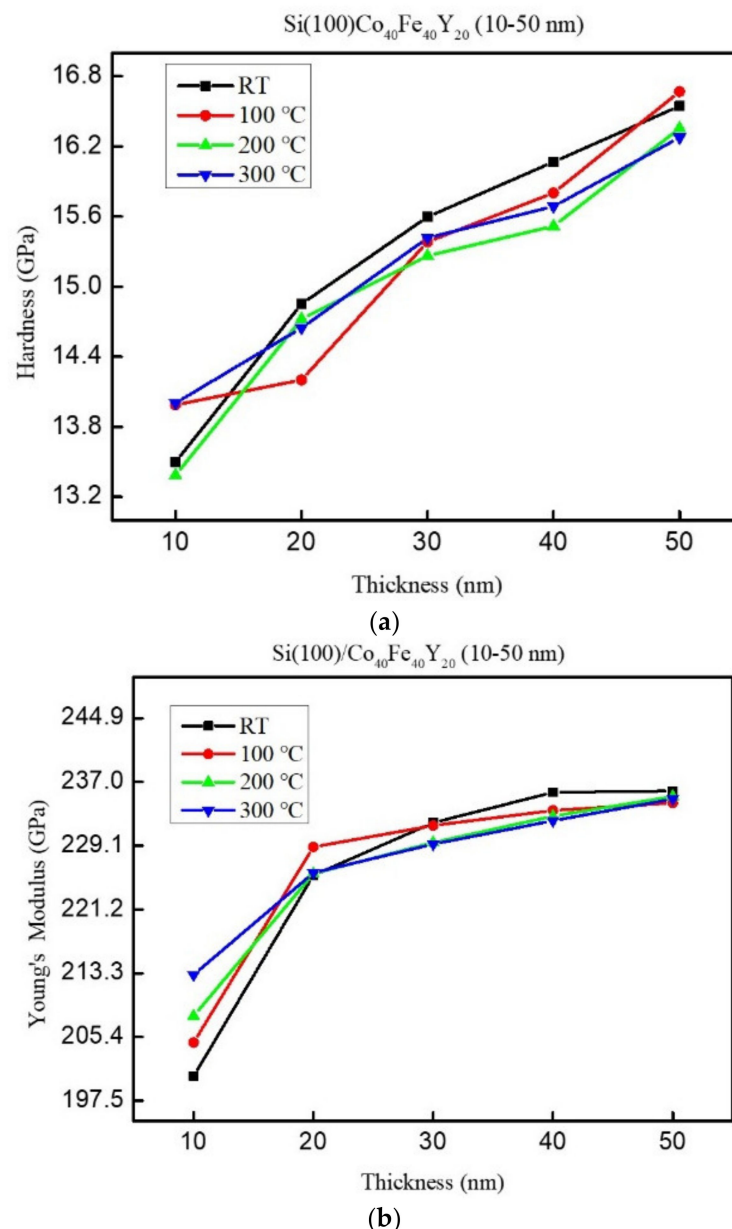


Figure 8. (a) Hardness and (b) Young's modulus of CoFeY films.

4. Conclusions

The typical CoFeY alloy peaks were not immediately visible using XRD, and the majority of them appeared to be amorphous. Oxides may be produced by improper sample storage or by the oxidation of the original alloy target. The affinity and negative electric degree relationship between the elements is another potential explanation. The χ_{ac} value was decreased at the high-frequency period, as evidenced by the results of the ac susceptibility. The highest χ_{ac} value, 0.159, was observed in a CoFeY film that was 50 nm thick and heated to 300 °C. The maximum value of χ_{ac} increases as the thickness of the CoFeY film increases due to the thickness effect and the addition of Y, which improved the coherence of the spin exchange and grain refinement. The improved exchange coupling, which led to a rise in the χ_{ac} value, was the main factor in the $\text{Co}_{40}\text{Fe}_{40}\text{Y}_{20}$ films' increase in χ_{ac} value after annealing. The optimal resonance frequency range for getting the maximum χ_{ac} value was between 50 and 100 Hz. Furthermore, the electronic conductivity was improved by increasing the grain-size thickness and removing obstacles to electron migration. Hydrophilicity was apparent because none of the films had contact angles greater than 90°. With an increase in

annealing temperature, the contact angle decreases. As annealing temperatures rose, the surface energy of the $\text{Co}_{40}\text{Fe}_{40}\text{Y}_{20}$ films rose as well. After 300 °C annealing, the greatest surface energy for a 40 nm thick film was 31.1 mJ/mm². The results of the nanomechanical analysis demonstrated that the temperature had little effect on the elastic modulus and hardness. The maximum hardness was determined to be 16.67 GPa at a thickness of 50 nm at 100 °C, and both the hardness and Young's modulus showed an upward trend with increasing thickness. Due to the high χ_{ac} value, strong adhesion, favorable nanomechanical properties, and low resistivity, it was discovered that the optimal conditions were at 50 nm with annealing at 300 °C.

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