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The influences of annealing on surface morphology and microstructure of NdFeB thin film

Z Z Guo^{1*}, J W Cao¹, D M Yan¹, F F Liu¹, C Zhang¹, J B Guo¹ and Y D Fu²

¹No. 52 Institute of China North Industry Group, Yantai 264003, China

²College of Materials Science and Chemical Engineering, Harbin Engineering University, Harbin 150001, China

*Email: guozaizai@163.com

Abstract. NdFeB thin films are produced by DC magnetron sputtering. The influences of annealing on surface morphology and microstructure of NdFeB thin films are studied in this paper. The result shows that the formation of Nd₂Fe₁₄B is prevented by depositing film directly on the substrates. Nd₂Fe₁₄B phase exists in crystalline films with Mo as a buffer layer and protective layer. Mo is a good material for being a buffer layer and protective layer. The (110) oriented texture of Mo is formed on (100) mirror polished Si after annealing. The deposited films are amorphous. SEM results show that the grain size is fine and uniform after annealing. Atomic Force Microscope investigation reveals that the film is smoother and denser after annealing. It is found that the films are still amorphous with annealing temperature below 400 °C, and the films begin to crystallize after annealing at 500 °C; the films annealed at 600 °C are fully crystallized.

1. Introduction

Demand for permanent magnet thin films with high magnetic energy densities is recently growing with a technological trend toward miniature devices. Various methods such as magnetron sputtering, molecular beam epitaxy and pulsed laser deposition have been employed to synthesize NdFeB thin film [1-4]. The suitable sputtering conditions for making aligned films are likely to depend on the sputtering system, including the substrate material, target composition, substrate temperature, sputtering gas atmosphere and deposition rate [5-8]. Recently, Nd₂Fe₁₄B films with much better c-axis alignment may be obtained using the DC sputtering method [9,10].

In this paper, NdFeB films were made by the DC sputtering method. The influences of annealing on surface morphology and microstructure of NdFeB thin films were studied.

2. Experimental details

2.1. Experimental material

A target with a nominal composition of Nd₂Fe₁₄B and a commercial Mo target of 99.9% purity were used. Si(100) and glasses were used as the substrates.

2.2. Preparation of thin films

The growth of films was carried out in a high-vacuum chamber equipped with multiple sputtering guns. The base pressure of the chamber was better at 4.0×10^{-4} Pa and the Ar gas pressure kept at 0.7 Pa during sputtering. The sputtering power was 100 W and the target-substrate distance was 6 cm.



2.3. Post annealing method

In this experiment, as-deposited NdFeB film sample prepared by DC sputtering was annealed in vacuum at $400^{\circ}\text{C} \leq T_{\text{ann}} \leq 800^{\circ}\text{C}$ to favour the crystallization process of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase. The samples were annealed in a 1.0×10^{-3} Pa vacuum to protect neodymium from oxidization.

2.4. Analysis of surface morphology and phase structure of NdFeB thin films

The phase structures were determined by X-ray diffraction (XRD) measurements with Cu $K\alpha$ radiation. The microstructures were studied by scanning electron microscopy (SEM). The surface topography was analysed by Atomic Force Microscopy (AFM).

3. Result and discussion

3.1. Determination of the composition of films

Figure 1 shows the X-ray diffraction pattern of the sample directly deposited on glass and then annealed at 700°C for 20 minutes. α -Fe peak is very strong in the pattern, and no other $\text{Nd}_2\text{Fe}_{14}\text{B}$ diffraction peak is clearly observed. It implies $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase directly deposited on glass after annealing is difficult to form.

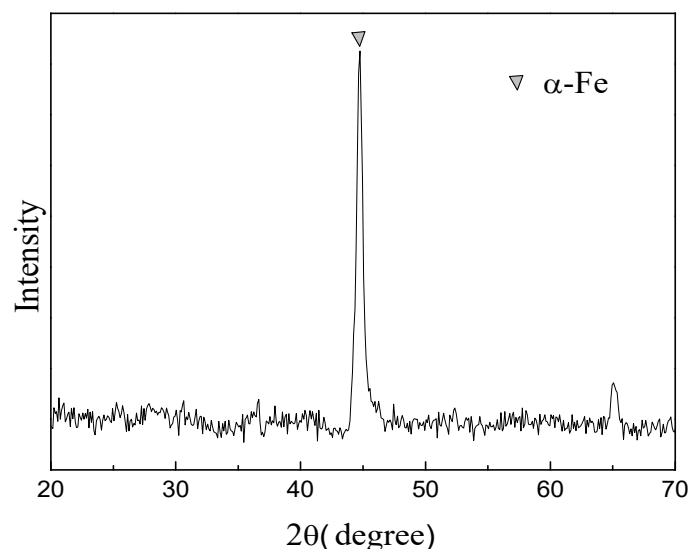


Figure 1. X-ray diffraction patterns of the sample deposited on glass and then annealed at 700°C for 20 minutes.

Figure 2 shows the X-ray diffraction patterns of Mo(50nm)/ NdFeB(800nm)/ Mo(50nm) thin film. From the shape of this peak, it seemingly consists of both Mo(110) phase and $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase. When Mo is used as buffer and cover layer, $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase is easy to form, as seen in figure 2.

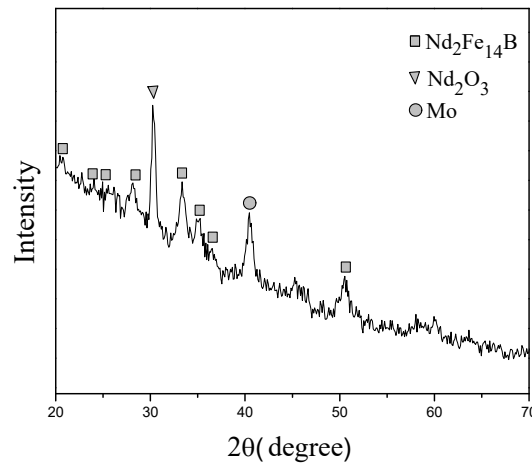


Figure 2. X-ray diffraction patterns of Mo(50nm)/NdFeB(800nm)/ Mo(50nm) thin film.

3.2. Analysis of morphology of thin films

Figure 3 shows SEM images of NdFeB thin film annealed at different temperatures for 20 minutes. When the annealing temperature is 550°C, the grains do not appear and the film exhibits a smooth and dense surface, as seen in figure 3. When the annealing temperature is 600°C, the grain size is small. With increasing annealing temperature, the grain size increases generally. When the annealing temperature is 800°C, the film shows a rather inhomogeneous morphology and some island-like bigger grains embed in the surface of the film.

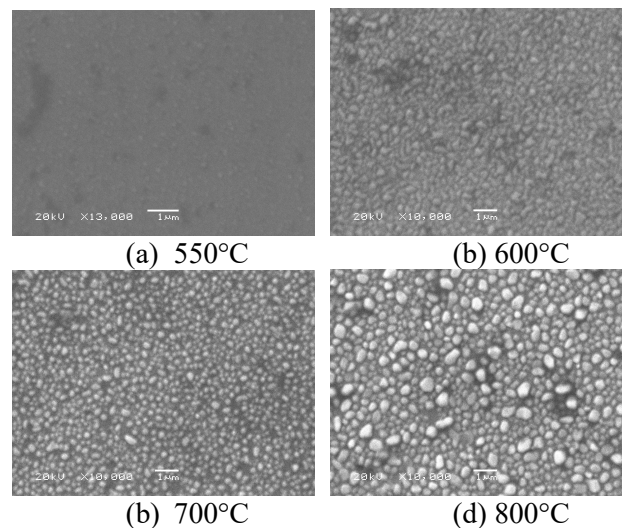


Figure 3. SEM images of NdFeB thin film annealed at different temperatures.

Figure 4 shows the SEM images of NdFeB thin film annealed at 650°C for different times. When the annealing time is 30 seconds, the grain size is small but not homogeneous. When the annealing time is 10 minutes, the grain size is small and homogeneous. With increasing annealing time, the grain size increases slowly and the distribution is more uniform.

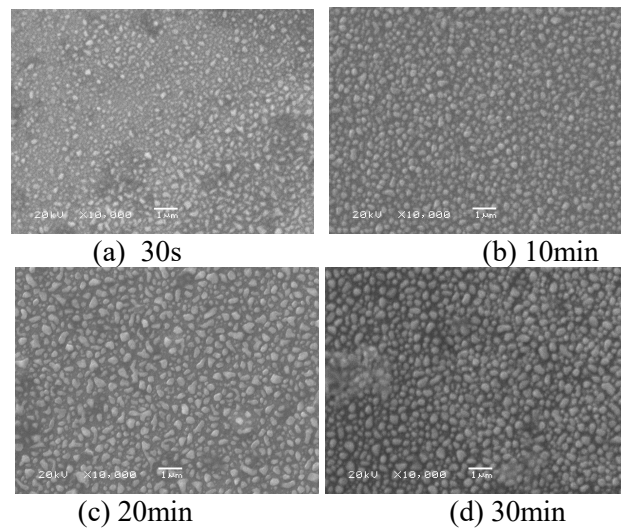


Figure 4. SEM images of NdFeB thin film annealed at 650°C for different time.

Figure 5 shows the effects of annealing to NdFeB thin film three-dimensional AFM images. The annealing temperature is 600°C for 20 minutes in figure 5(b). The NdFeB thin film after annealing exhibits smoother and denser than as-deposited film as seen in figure 5(a). The grain size is smaller and the films become columnar structure.

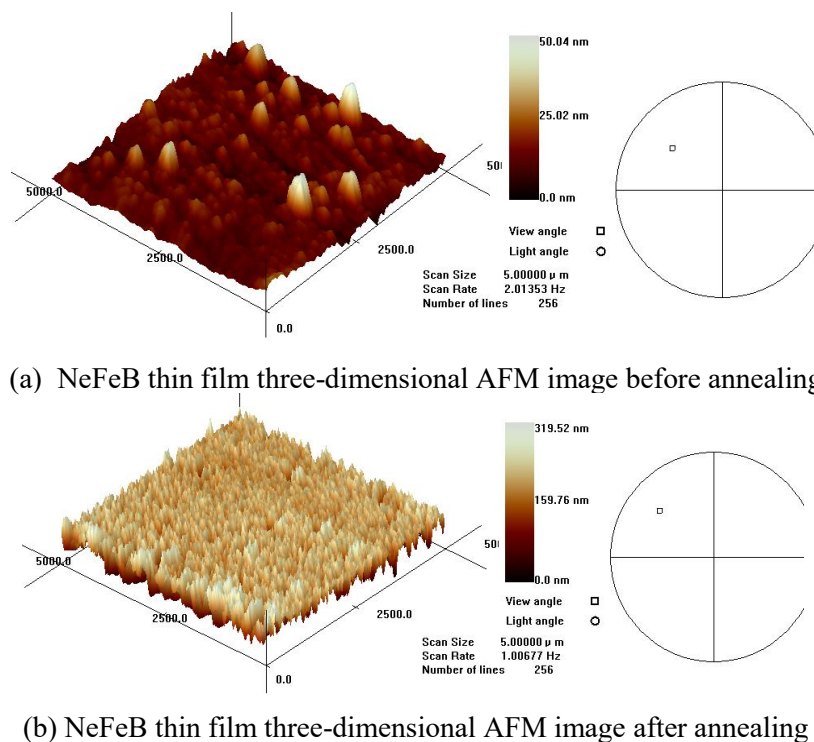


Figure 5. The effects of annealing to NdFeB thin film three-dimensional AFM images.

3.3. Analysis of the phase and crystallization of NdFeB thin films

Figure 6 shows the X-ray diffraction patterns of Mo(50nm)/NdFeB(800nm)/Mo(50nm) thin film annealed at different temperatures. When the annealing temperature is 400°C, the film shows a single and weak Mo peak, as seen in figure 6. It reveals that the NdFeB thin films after annealing at 400°C are amorphous and the Nd₂Fe₁₄B phase is not found in this film. Similar results were reported by Serrona et al. [11]. They reported that when the annealing temperature was low, the Nd₂Fe₁₄B phase

did not form in the XRD patterns [11]. When annealing temperature is 500°C, the diffraction peaks begin to appear which reveals that the films begin to crystallize. When the annealing temperature is 600°C, a large amount of $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase exists in the films and the intensity of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ peak is much stronger. It is found that the films annealed at 600°C are fully crystallized.

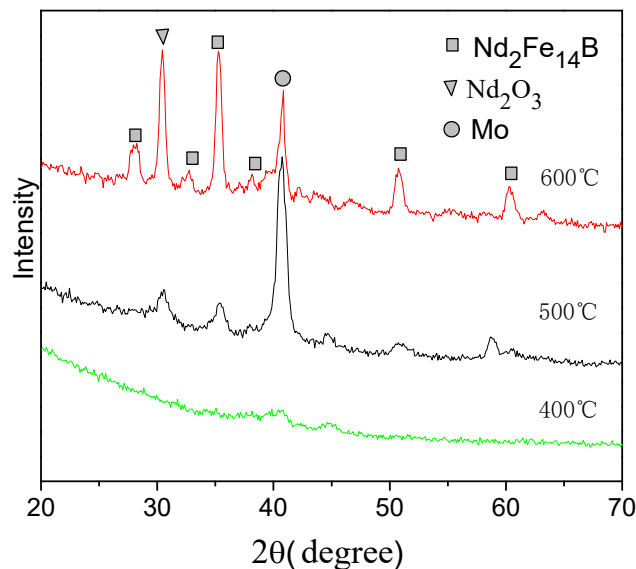


Figure 6. X-ray diffraction patterns of Mo(50nm)/NdFeB(800nm)/Mo (50nm) thin film annealed at different temperatures.

4. Conclusions

We have prepared NdFeB thin films with DC magnetic sputtering. It has been found that the surface morphology and microstructure of NdFeB thin films are strongly dependent on annealing. As-deposited NdFeB thin films are amorphous. The planar view SEM images of them exhibit a smooth surface, and the grains cannot be distinguished from each other. The NdFeB thin films after annealing at 650°C exhibit more uniform grains. The grain size is smaller. AFM investigation reveals that the film is smoother and denser after annealing. It is found that the films are still amorphous with annealing temperature below 400°C, and the films begin to crystallize after annealing at 500°C, the films annealed at 600°C are fully crystallized, which is due to the existence of $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase.

5. References

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