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Barium hexaferrite thin films prepared by the sol-gel method

Kookmin Univ. Sung Yong An*, Sang Won Lee, Byoung Ki Min, In-Bo Shim, and Chul Sung Kim

Chungju National Univ. Seung Wha Lee

Ⅰ. Introduction

The sol-gel technique has emerged in recent years as a versatile method for synthesizing different inorganic materials. Apart from the advantage of low temperature processing a sol-gel route makes it possible to obtain nano-particle materials [1]. Though the sol-gel process has made an impact on materials technology, the high cost of alkoxides appears to be a hindrance to its large scale use in preparing conventional materials. In this work, thin films with barium hexaferrite (BaM) layers on thermally oxidized silicon wafers were fabricated by the sol-gel method. We have a avoided using alkoxides thereby ensuring that the preparation cost is not high.

Ⅱ. Experimental procedures

Barium nitrate [$Ba(NO_3)_2$] and iron nitrate [$Fe(NO_3)_3 \cdot 9H_2O$] were used as starting materials, and were combined so that the composition ratio Ba/Fe=1/10. These were dissolved in methanol and distilled water. The solution was refluxed at 80 ℃ for 24 h. The resulting precursor solutions were made at 0.2 M. Films were spin-coated onto thermally oxidized silicon substrates (10 × 10 mm), dried and then heated in air at various temperatures. The crystal structures were measured by x-ray diffraction using CuKα. Thermal analysis such as thermogravimetry analysis (TGA) and differential thermal analysis (DTA) were performed on the dried powder obtained from the $BaFe_{12}O_{19}$ precursor solution. The FTIR studies were done using an FTIR spectrophotometer on films coated on KBr single-crystal substrates. Magnetic properties were measured using a vibrating sample magnetometer at a maximum applied field of 10 kOe.

Ⅲ. Results and discussion

The thermal decomposition characteristic of gel system with temperature was studied by DTA and TGA, as shown in Fig. 1. In this study, the $BaFe_{12}O_{19}$ (BaM) was pre-dried at 120 ℃ for 12 hours and the thermolysis behavior analyzed using a heating rate of 5 ℃/min. TGA analysis indicated a major weight loss was completed at 260 ~ 600 ℃. It can be seen that the gel exhibited approximately 68 % weight loss until 600 ℃. While the rapid decrease of the weight was due to evaporation of the solution and thermal decomposition of organic components, the gradual decrease in the weight is thought to be caused by the thermal decomposition of organic group bonded to an oxide network. DTA analysis indicated a series of exothermic peaks. The peaks at temperature beteen 150 ℃ and 280 ℃ are associated with the organic weight losses. However, the final peak which appears at about 328 ℃. No additional peak was observed above 328 ℃, indicating that complete thermal decomposition as well as crystallization of $BaFe_{12}O_{19}$ occurs simultaneously. Figure 2 shows a hysteresis loop of the sample annealed at 650 ℃ for 2 h. The perpendicular coercivity $H_{C\perp}$ and in-plan one $H_{C//}$ were 4766 Oe and 4480 Oe, respectively, at room temperature under an applied field of 10 kOe. This shows that the film has isotropic properties [2]. The perpendicular coercivity squaeness $S^* = 1 - \frac{M_r / H_c}{dM / dH}$, a measure of the slop of the loop at H_c, are 0.65. The orientation coefficients $S=M_r/M_s$ in both the parallel and perpendicular directions are analyzed as a function of annealing temperature. Above 650 ℃ insignificant differences are observed and the corresponding values lie around 0.5, revealing that thin films are magnetically isotropic.

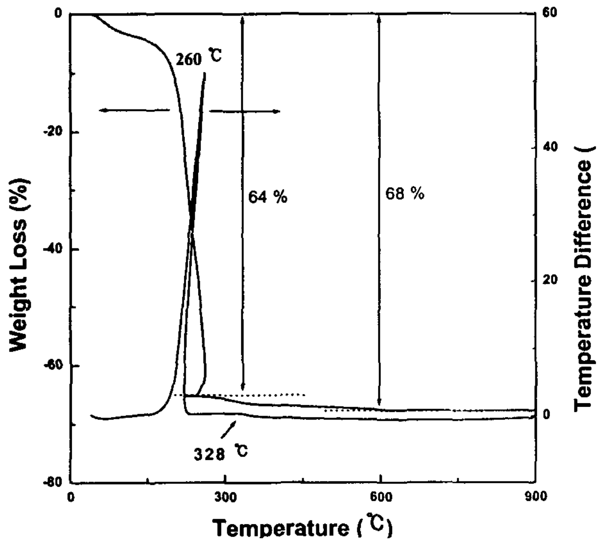

Fig. 1. TGA/DTA curves of $BaFe_{12}O_{19}$ gel powder

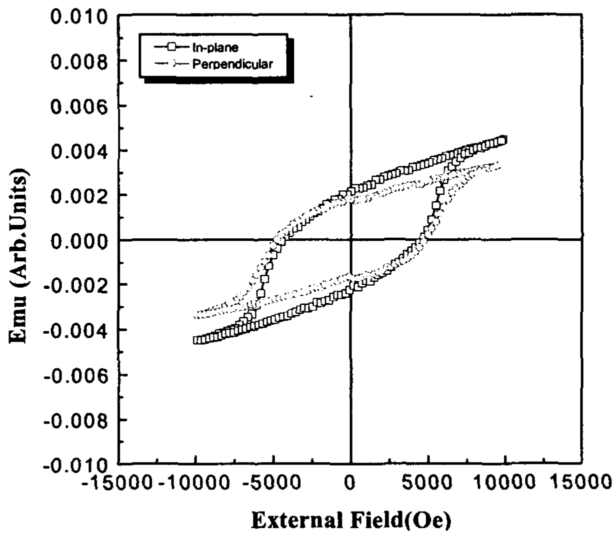

Fig. 2. In-plane and perpendicular hysteresis loop of the thin film annealed at 650 ℃ for 2h.

References

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