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Effect of ethylene glycol on the orientation and magnetic properties of barium ferrite thin films derived by chemical solution deposition

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ABSTRACT

M-type barium ferrite (BaM) film is a promising candidate for magnetic recording media and the next-generation high-frequency microwave material. BaM films were fabricated using a chemical solution spin-coating deposition method on (001) plane sapphire. The effect of ethylene glycol (EG) on precursor solution and annealing temperature on microstructure and magnetic properties were investigated. X-ray diffraction showed that strong out-of-plane (001) texture in BaM films can be achieved with adequate EG addition. Films fabricated without EG addition or annealed at lower temperatures showed partial hematite secondary phase. The epitaxial formation of hematite secondary phase on sapphire substrate was confirmed by the X-ray diffraction φ-scan technique. Magnetic measurements showed out-of-plane anisotropy in BaM films. The crystallographic texture and magnetic anisotropy of the films are related to the film microstructure.

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1. Introduction

M-type barium ferrite ($BaFe_{12}O_{19}$, BaM) thin films are promising candidates for perpendicular recording media owing to its large magnetic anisotropy [1]. BaM films have received widespread attention in recent years owing to its high uniaxial anisotropy, high saturation magnetization and low conduction loss, which enable operation under frequencies up to 60 GHz [2,3]. Also, due to the large uniaxial crystalline anisotropy in BaM, self-biased operation can be achieved in textured barium ferrite films that have (0001) axis aligned to a certain preferred orientation [2,4–6]. Much effort has been made to fabricate highly textured films with out-of-plane or in-plane orientations by methods such as RF magnetron sputtering [7], pulse laser deposition [8–10] and liquid phase epitaxy [11,12]. Although such methods are effective, expensive equipment limits large-scale operation. Cheaper solution deposition techniques have also been demonstrated to be able to fabricate textured barium ferrite films [13–17]. In solution deposition processes, the composition of precursor solution plays a critical role in the microstructure and properties of deposited films. In previous efforts, the chemical solution depositions of BaM films often utilize solutions containing ethylene glycol (EG). EG is a drying control agent widely utilized in the deposition of textured $(K,Na)NbO_3$ films. However, due to the low volatility of EG, large amounts of EG in precursor solutions can lead to large thickness inhomogeneity due to dewetting, which is common in plain nitrate spin-coating and dip-coating processes [18]. In this work, the effect of EG content in precursor solution on BaM films was investigated. Results showed that EG plays an important role in the microstructure and texture of BaM films.

2. Experimental details

$Ba(NO_3)_2$ and $Fe(NO_3)_3 \cdot 9H_2O$ (99%, Alfa Aesar) with a molar ratio of 1:6 were dissolved in deionized water. The low Fe/Ba ratio was chosen to suppress the formation of Fe_2O_3 secondary phase and improve magnetic properties [19]. Various amounts of EG were added into the resulting solution. 1 g polyvinyl pyrrolidone (PVP) was then added to the obtained solution to adjust the viscosity of the solution. The volume of the resulting solution was then adjusted to 25 ml with deionized water. The final molar concentration of Ba^{2+} was 0.032 mol/L. *R* values in this paper are defined to be the volume of EG addition divided by the total volume of final solution at room temperature. The precursor solution was spin coated onto a (001) sapphire substrate at 4000 rpm for 30 s. The deposited films were then dried at 100 °C for 5 min and subsequently annealed in a rapid thermal processor at 500 °C in air atmosphere for 5 min in order to decompose organic additive and precursor nitrates to prevent the dissolution of formerly deposited layers in succeeding deposition steps. The relatively high pyrolysis temperature was chosen to decompose the PVP additive [20]. The deposition–pyrolysis procedure was repeated to reach a thickness of 150 nm. The resulting films were then annealed in a conventional furnace at temperatures ranging from 950 °C to 1035 °C for 30 min.

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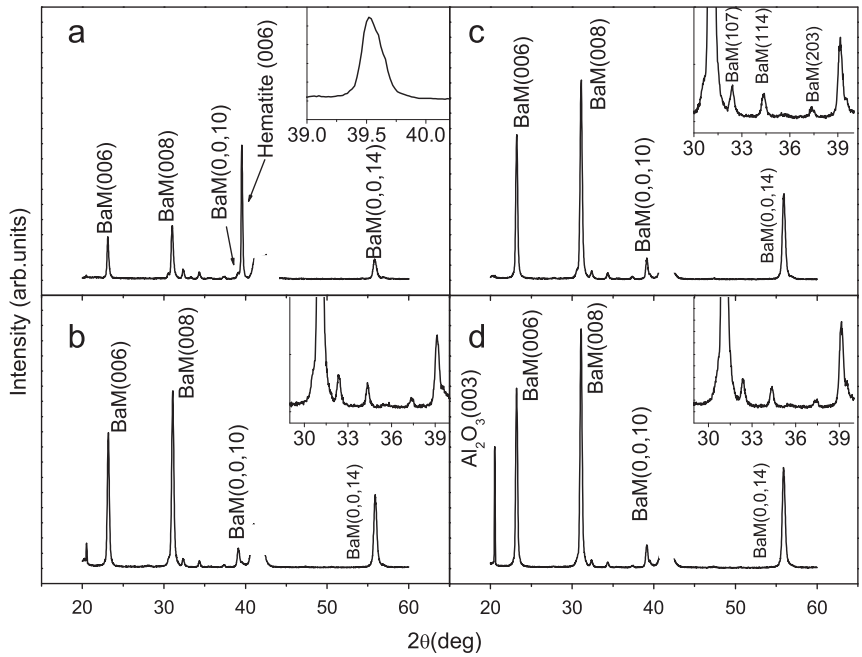

Fig. 1. XRD patterns for barium ferrite films prepared from precursor solutions with different volume ratios of ethylene glycol to solution: (a) $R=0.00$, (b) $R=0.08$, (c) $R=0.16$, and (d) $R=0.24$. The scales are consistent from (a) to (d) for the ease of comparison, and all films were annealed at 1000 °C. Inset in (a) shows the hematite (006) peak and the BaM (0,0,10 peak). Inset in (b)–(d) shows minor diffraction peaks of the corresponding spectrums.

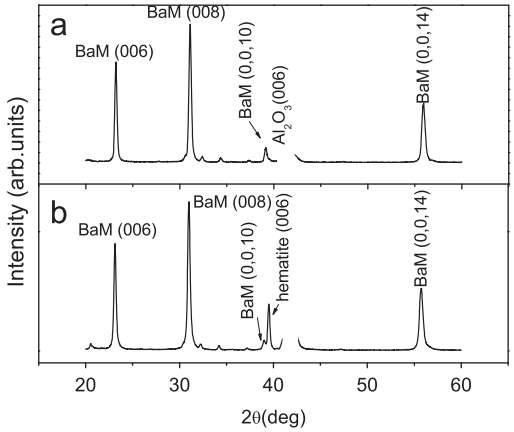

Fig. 2. XRD patterns for barium ferrite films fabricated from precursor solution with $R=0.16$ annealed at different temperatures: (a) 1000 °C and (b) 950 °C.

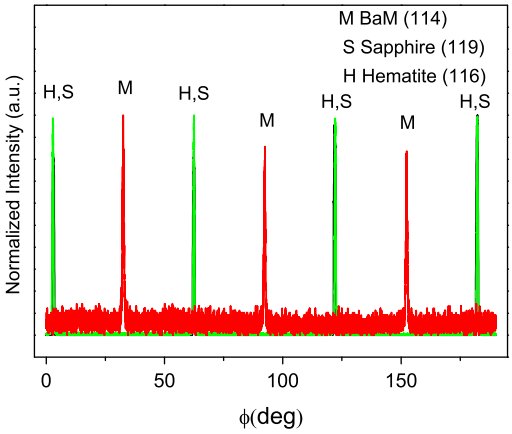

Fig. 3. φ-Scan spectrum of sample fabricated without glycol.

X-ray diffraction (XRD) system with Cu-K_α radiation (40 kV, 200 mA, H2500, Rigaku, Tokyo, Japan) was used to analyze the phase formation and orientation of the BaM films. The microstructure of the films was observed using a scanning electron microscope (SEM, JSM 7001, Tokyo, Japan). A vibrational sample magnetometer (VSM, PPMS-7, Quantum Design) was used to measure the room temperature magnetic properties of annealed films.

3. Results and discussion

X-ray diffraction patterns of films fabricated with various volumes of EG in precursor solutions annealed at 1000 °C are shown in Fig. 1. Al_2O_3 (006) peaks were excluded from the scan. It can be seen that while film fabricated with solutions containing 2–6 ml of EG (i.e. $R=0.08$–0.24) exhibited similar (001) peaks perpendicular to surface texture, film fabricated with solution containing no EG yielded abnormally high (0,0,10) peak intensity, which is in fact an additional peak adjacent to BaM (0,0,10) peak, as is shown in the inset in Fig. 1(a). Also, as is shown in Fig. 2, film derived from EG containing solution annealed at lower temperature also showed a similar shoulder peak. There are two explanations proposed for the additional peak. One is BaM (109) peak as was proposed by Zhang et al. [14], confirmed by magnetic properties. The other is Fe_2O_3 (hematite) (006) peak, which has an epitaxial relationship that is experimentally observed in the previous research using the MBE technique [21] and sputtered films [22] on sapphire (001) substrates. Unfortunately, the two peaks in question (BaM (109) and hematite (006)) are located too close together and thus cannot be distinguished by $\theta-2\theta$ scan alone. In order to clear the confusion caused by overlapping diffraction peaks, additional φ-scan was carried out in order to distinguish between the two phases.

Fig. 3 shows the φ-scan in the range of 0–190° of the film fabricated with no EG addition. BaM (114), hematite (116) and sapphire (119) peaks were measured to analyze the

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