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Preparation of nano-sized Bi-YIG particles for micro optics applications

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Abstract

Nano-size Bi-YIG (Bi substituted Yttrium Iron Garnet: Bi_{1.8}Y_{1.2}Fe₅O₁₂) particles and their coated film were prepared. The particles were made by coprecipitation and heat treatment processes. The magnetic ink and coated films were prepared with milling and printing techniques. We optimized the ink preparation and coating conditions. The particles in the films were observed by a transmission electron microscope and an atomic force microscope. The size of the coprecipitated particles was about 10–20 nm. The milled particles size was the same as the coprecipitated particle. The magnetic and magneto-optical properties of the particles and coated films were investigated. The maximum value of the figure of merit was 1.5° which was comparable to the value of a sputtered film. The magneto-optical ink and coated film are new materials for magneto-optical devices. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: Bi-YIG; Magneto-optical; Nano-size particle; Dispersed material; Coating film

1. Introduction

Magnetic garnet is an attractive magneto-optical material for micro magneto-optical devices. The garnet film has been prepared with deposition and heat treatment processes. These processes require expensive substrates, e.g. GGG (Gadolinium Gallium Garnet). We have developed preparation processes of nano-size Bi-YIG (Bi substituted Yttrium Iron Garnet) fine particles and coated films [1,2]. For preparation of the coated

films, we have used milling and coating techniques. In this preparation techniques, the main processes consist of coprecipitation of Bi-YIG particles, heat treatment for crystallization, milling for disaggregation of the sintered particles and coating of the fluid on substrates. In order to offer the film for micro optics applications, it is important to clear the relation between the milling time and magneto-optical properties.

In this paper, we observe the shape of the particles throughout the preparation process and the nano-size Bi-YIG fine particles in the coated films which are made with various milling times. The relations between the sizes of the particles and magneto-optical properties of coated films are investigated.

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2. Experimental

2.1. Preparation of Bi-YIG fine particles

Fig. 1 shows the preparation processes of the Bi-YIG particles and coated film. The Bi-YIG particles were prepared by coprecipitation and heat treatment processes [3]. Aqueous solutions of nitrates of Bi, Y and Fe were mixed where the ratio of the cations corresponded to the composition of Bi_{1.8}Y_{1.2}Fe₅O₁₂. Then the solution was mixed with a NH₄OH solution with stirring at room temperature. The obtained slurry was washed, filtered and dried at 100°C for 2 h. Then the coprecipitate was heated in air for 4 h. The temperatures of the heat treatments were from 550 to 700°C. The crystal phases of the particles were examined by X-ray diffraction (XRD) analysis. The particles were observed with a transmission electron microscope (TEM).

2.2. Preparation of the Bi-YIG coating films

The particles were mixed with an epoxy binder dissolved by a cyclohexanone and milled with a planetary micro milling machine for 0-100 h (Fig. 1), the mixtures were then coated by a spin coater on Corning 7059 glass. The volume content of the magnetic particles in the coated films was about 0.2. The particles in the films were observed with an atomic force microscope (AFM). The saturation magnetization (M_S) and

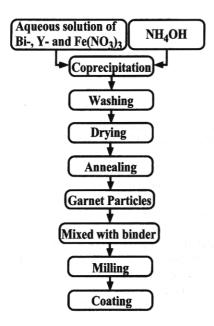


Fig. 1. Preparation process of Bi-YIG particles by coprecipitation and heat treatment.

coercive force ($H_{\rm C}$) of the films were measured with a vibrating sample magnetometer (VSM). The Faraday rotation ($\theta_{\rm F}$) of the coating films was measured by the polarization modulation method. The absorption coefficient (α) was measured with a spectrophotometer.

3. Results and discussions

3.1. The Bi-YIG particles

Fig. 2 shows the TEM image of the coprecipitated particles. The size of the coprecipitated particles is about 10–20 nm.

The Bi-YIG particles were synthesized through the reaction Eq. (1).

 $5\text{Fe}(OH)_3 + 1.8\text{Bi}(OH)_3$

$$+1.2Y(OH)_3 \rightarrow Bi_{1.8}Y_{1.2}Fe_5O_{12} + 12H_2O.$$
 (1)

This reaction was proceeded by heat treatment. Fig. 3 shows the TEM images of the heat-treated particles. It is clear that the size of the particles increases with increasing heat treatment temperature (T_a) . Fig. 4 shows the XRD patterns of the heat treated particles. For $T_a \ge 650^{\circ}\text{C}$, most of the diffraction peaks are identified as garnet structure, indicating the formation of Bi-YIG particles with micro meter size (see Fig. 2).

3.2. Nano-size Bi-YIG particles dispersed films

Using optimized milling conditions, the aggregated particles were milled to nano-size [2]. Fig. 5 shows the AFM images of the nano-size Bi-YIG particles in the coating films as a function of milling time. The typical

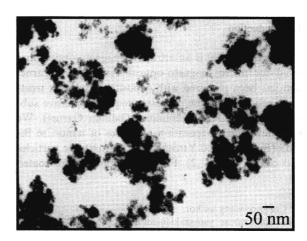
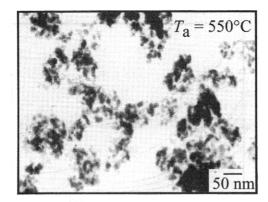
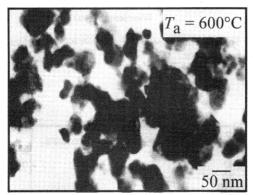
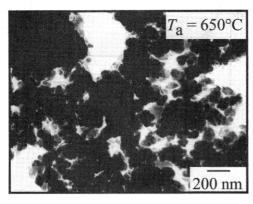


Fig. 2. TEM image of the as prepared coprecipitated particles.







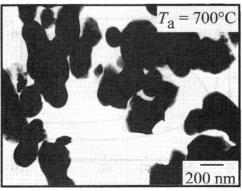


Fig. 3. TEM images of Bi-YIG particles for various heat treatment temperature $T_{\rm a}$.

size of the particle is about 10-20 nm when milled more than 30 h, which is almost the same size of the coprecipitated particles.

Fig. 6 shows transmitted light spectra of the films. The spectra are divided into two groups by milling time. The A group contains the films made with 1–20 h milling. The B group contains the films made with 30–100 h milling time. The A group films show large absorption over the entire visible wavelength region. This increase in the absorption is partly due to the scattering of light by Bi-YIG particles in the coated films. The B group films which are constituted with fine Bi-YIG particles show moderate absorption. From AFM images, the sizes of these particles are found to be smaller than the wavelength of visible light.

3.3. Magnetic and magneto-optical properties of the coating films

Fig. 7 shows the $M_{\rm S}$ and $H_{\rm C}$ of the coated films. The $M_{\rm S}$ dropped with milling time. After 30 h milling, the $M_{\rm S}$ and $H_{\rm C}$ of the films became very small and constant.

Fig. 8 shows Faraday rotation spectra and absorption spectra of the coating films. The film, which consists of the particles milled for 30 h, exhibit the highest Faraday rotation. The absorption coefficient of the films decreased with increasing milling time. Fig. 9 shows the relation between the figure of merit (θ_F/α) and the milling time. The θ_F/α of the 30 h milling film is about 1.5° at 520 nm. This value is comparable to the sputtered Bi-AlGdIG film [4].

Fig. 10 shows Faraday rotation spectra of the coating films with various milling times. The films

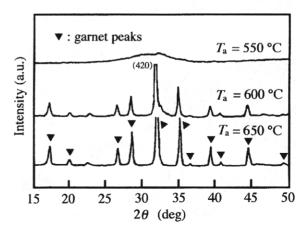
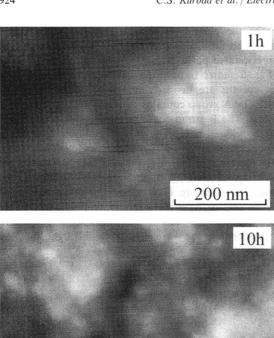
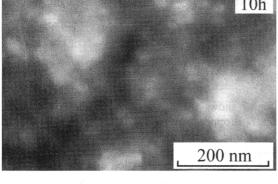
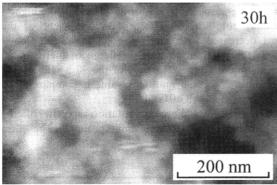


Fig. 4. XRD patterns of the Bi-YIG particles for various heat treatment temperature T_a .







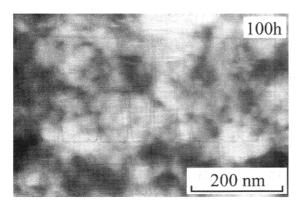


Fig. 5. AFM images of the Bi-YIG particles in the coated films for various milling time.

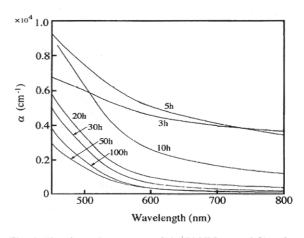


Fig. 6. The absorption spectra of the Bi-YIG coated films for various milling time.

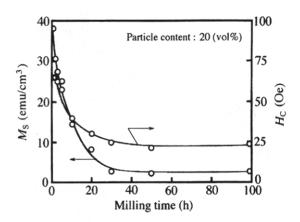


Fig. 7. The $M_{\rm S}$ and $H_{\rm C}$ of the Bi-YIG coated films as a function of milling time.

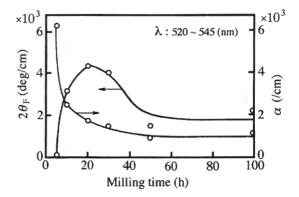


Fig. 8. Faraday rotation spectra θ_{F} and absorption spectra α of the Bi-YIG coated films as a function of milling time.

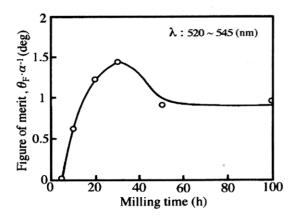


Fig. 9. Figure of merit of the Bi-YIG coated films as a func-

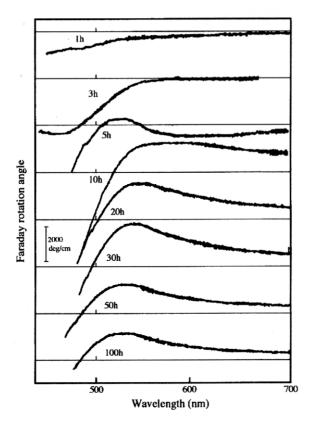


Fig. 10. Faraday rotation spectra of the Bi-YIG coated films for various milling time.

of A group do not show peculiar Faraday rotation spectra which is different from that of Bi-YIG. The profile of these spectra changes with milling time. The films of B group show typical Faraday rotation spectra of Bi-YIG. The Faraday rotation spectra are modified by the size of Bi-YIG particles in the coating films.

4. Conclusions

We prepared Bi-YIG particles by coprecipitation and heat treatment processes. The coated films were made with milling and printing techniques. The synthesis condition of the particles and milling process were optimized. The particles were investigated with TEM and AFM. The coating films were made with nano-size Bi-YIG particles.

The magnetic and magneto-optical properties were measured. The properties of the coated film differed from those of the sputtered film and the bulk materials. The figure of merit of the film was about 1.5° at 520 nm. This value is comparable to that of the sputtered film.

The dispersed magneto-optical material is suitable for micro optics devices with printing technique.

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