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# Preparation and Faraday rotation of Bi-YIG/PMMA nanocomposite

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## ABSTRACT

Bismuth-substituted yttrium iron garnet (Bi-YIG) nanoparticles (NPs) were prepared by coprecipitation and subsequent heating treatment. Thermal gravity-differential thermal analysis was performed to investigate the thermal behavior of the Bi-YIG precursors and to decide the best annealing temperature. Phase formation of garnet NPs was investigated by X-ray powder diffraction. The size of Bi-YIG NPs was investigated by transmission electron microscopy, and the magnetic properties of Bi-YIG NPs were measured using a vibrating sample magnetometer. The results show that the temperature needed for the transformation of Bi-YIG from the amorphous phase to the garnet phase decreases with increasing Bi content, and Bi-YIG NPs with sizes of 28–78 nm are obtained after heating treatment at 650–1000 °C. The saturation magnetization of Bi-YIG NPs increases as the Bi content increases. Moreover, the Faraday rotation of polymethyl methacrylate (PMMA) slices doped with Bi-YIG NPs was investigated. The results indicate that the angle of Faraday rotation increases with increasing Bi content in PMMA composites, and the maximum value of the figure of merit is 1.46°, which is comparable to the value of a sputtered film. The Bi-YIG NPs-doped PMMA slices are new promising materials for magneto-optical devices.

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# 1. Introduction

Ferromagnetic garnets were discovered in 1956 and had attracted much interest due to their high resistivity and application in microwave devices [1]. Yttrium iron garnet (YIG) is a very interesting material because of its novel potential application in magneto-optical switches, modulators, optical circulators, laser isolators, magnetic field and electric current sensors based on the Faraday effect [2]. In addition, from an industrial viewpoint, YIG is applicable to the media for high-density magnetic or magnetooptical information storage.

Many researchers pay much attention to the conventional ceramic method [3,4] in order to improve the homogeneity and density of garnet particles. New synthesis methods have been developed to obtain homogeneous nanoparticles (NPs), including coprecipitation [5,6], mist pyrolysis [7], sol-gel [8], hydrothermal [9] and mechanochemical processing [10]. It has been demonstrated that the coprecipitation processing offers

considerable advantages, such as better mixing of the starting materials and excellent chemical homogeneity of the final product. Furthermore, the molecular level mixing and the tendency of partially hydrolyzed species to form extended networks facilitate the structure evolution, thereby lowering the crystallization temperature of garnet NPs.

It has been found that bismuth-substituted YIG (Bi-YIG) exhibits stronger Faraday effect than that of YIG [11,12]. This makes the Bi-YIG NPs much more promising in magneto-optical devices [13,14]. The development of new devices based on high magneto-optical effect of Bi-YIG NPs has intensified the study of these materials. Lee et al. [15] investigated the magneto-optical properties of organic films doped with Bi<sub>1.8</sub>Y<sub>1.2</sub>Fe<sub>5</sub>O<sub>12</sub> NPs and found that the figure of merit  $(\theta_{\rm F}/\alpha)$  of the thin films ranged from 0.5 to 3.2, with the wavelength range from 410 to 520 nm. Hasanpour et al. [16] studied the Faraday rotation of the composite films filled with BiY<sub>2</sub>Fe<sub>5</sub>O<sub>12</sub> NPs, and the results showed that the Faraday rotation angle increased as the content of NPs increased and the maximum values shift to shorter wavelength. Since the magneto-optical effect of materials strongly depends on the crystal structure, particle size, morphology and magnetic properties, it is necessary to systematically investigate the influence of synthesis conditions on the properties of the nanocomposites. In this investigation, we report the influence of

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annealing temperature on the garnet phase formation and the Bi content on the magnetic properties of Bi-YIG NPs. The Faraday rotation of polymethyl methacrylate (PMMA) slices doped with  $Bi_xY_{3-x}Fe_5O_{12}$  (x = 0, 1 and 1.8) NPs is also measured.

## 2. Experimental

### 2.1. Materials

Ferric nitrate ( $Fe(NO_3)_3 \cdot 9H_2O$ ), yttrium nitrate ( $Y(NO_3)_3 \cdot 6H_2O$ ) and bismuth nitrate ( $Bi(NO_3)_3 \cdot 5H_2O$ ), nitric acid, ethanol and other materials were all of analytical grade, purchased from commercial market and used without further purification. 25% ammonia aqueous solution was used as precipitator. Methyl methacrylate (MMA) of chemical grade (at a purity of 99.9%) was distilled under reduced pressure prior to use. Azo-bis-isobutyronitrile (AIBN) was used as an initiator, which was recrystallized in ethanol.

# 2.2. Preparation

#### 2.2.1. Preparation of Bi-YIG NPs

Polycrystalline  $Bi_xY_{3-x}Fe_5O_{12}$  (x = 0, 1 and 1.8) NPs were prepared by coprecipitation method using Fe(NO<sub>3</sub>)<sub>3</sub> · 9H<sub>2</sub>O,  $Y(NO_3)_3 \cdot 6H_2O$  and  $Bi(NO_3)_3 \cdot 5H_2O$  as starting materials and 25% ammonia aqueous solution as a precipitator. Aqueous solution of nitrates of Bi, Y and Fe were mixed in which the ratio of the cations corresponded to the compositions of Bi<sub>x</sub>Y<sub>3-x</sub>Fe<sub>5</sub>O<sub>12</sub> with different x values (x = 0, 1 or 1.8). The obtained metallic ion solution was added into the diluted ammonia (pH = 11) dropwise using a tundish under stirring at room temperature, and some more ammonia aqueous solution was added during the reaction so that the pH of the suspension could be kept around 11. The suspension was still fiercely stirred for 1 h after the addition of metallic ion solution. The obtained slurry was washed using deionized water for 5 times and absolute ethanol for 3 times, respectively, and dried at 80 °C for 10 h. Then, the precipitates were calcined at different temperatures for 2 h. The crystal phase of the particles was analyzed by X-ray diffraction.

#### 2.2.2. Preparation of Bi-YIG NP-doped PMMA slices

The  $Bi_xY_{3-x}Fe_5O_{12}$  (x = 0, 1 and 1.8)-doped PMMA slices were prepared by in situ bulk polymerization according to the procedure of the published article [17]. In a typical run, some Bi<sub>x</sub>Y<sub>3-x</sub>Fe<sub>5</sub>O<sub>12</sub> NPs (0.05 wt%) were dispersed into MMA monomer, and the obtained suspension was mixed in a high-energy ball mill operating at 40 rpm for 4 h. Some AIBN (0.1 wt% of monomer) as initiator was added. The prepolymerization was performed at 80+2 °C under mechanical stirring for about 20 min until the conversion of MMA was about 12-15%. Then, the suspension was poured into a stainless steel mold, which was coated with a thin film of Garry Mould Release Agent (non-paintable). The aperture at the top of the mold was wrapped with a plastic foil. Thereafter, the mold containing the prepolymer and Bi<sub>x</sub>Y<sub>3-x</sub>Fe<sub>5</sub>O<sub>12</sub> NPs was kept at 40 °C for 24 h, and the solidification took place. Then, the temperature was adjusted to 100°C and kept for 1 h to increase the molecular weight of PMMA. Finally, PMMA slices were obtained after disassembling the molds.

#### 2.3. Characterization

The crystalline phases were identified by X-ray powder diffraction (XRD) measurement with a D/Max-III C, using Cu-K $\alpha$  radiation. The particle size was obtained from transmission electron microscopy (TEM) using an H-600-II transmission

electron microscope (Hitachi, Japan) and by analyzing the broadened major peak of XRD spectra using Scherrer's equation. The thermal behavior of the obtained precursor was measured by thermal gravity-differential thermal analysis (TG-DTA) (TA Instruments, SDT-2960). The magnetic property of NPs was measured using a BHV-55 vibrating sample magnetometer (VSM). The apparatus (Shanghai Fudan Tianxin Sci. & Edu. Instruments Co., Ltd.) used to measure the Faraday rotation consists of two light polarizers, one on each side of the sample, an electromagnet producing a magnetic field of 5000 Oe, a semiconductor laser as a light source and a silicon photoelectric generator as a power meter.

#### 3. Results and discussion

#### 3.1. Thermal behavior of precursors

As shown in Fig. 1, there are characteristic peaks on the TG-DTA curves of the samples, and the peaks are different from their location and values. The TG-DTA curve of the  $Y_3Fe_5O_{12}$  precursor (Fig. 1(a)) shows an overall weight loss of about 32%. We can also conclude that 75% of weight loss occurs before 300 °C, which corresponds to the region of the exothermic peak around 284.08 °C in DTA curve. Such an exothermic peak can be associated with the removal of adsorbed organics and moisture. The rest weight loss, which corresponds to two exothermic peaks (around 782.84 and 836.76 °C) in the DTA curve, takes place in small steps. The exothermic peak at 782.84 °C indicates the crystallization of yttrium iron perovskite (YFeO<sub>3</sub>, YIP) and YIG, and the last exothermic peak at 836.76 °C is due to the conversion of YIP to YIG, which is also proved by the result of XRD in Fig. 2(a).

For the Bi-YIG samples, the TG-DTA curves of the precursors of  $Bi_xY_{3-x}Fe_5O_{12}$  (x = 1 or 1.8) are shown in Figs. 1(b) and (c). We can see that the overall weight loss of the samples is about 22% and 17% corresponding to x = 1 and 1.8, respectively. There are two characteristic peaks in both Figs. 1(b) and (c). The exothermic peak around 284.08 or 324.31 °C corresponding to Figs. 1(b) or (c) is caused by the burning of the residual organics and the removal of the physical adsorbed water, which results in the large weight loss in the TG curves. The exothermic peak at 731.17 or 662.38 °C indicates the crystallization of  $BiY_2Fe_5O_{12}$  or  $Bi_{1.8}Y_{1.2}Fe_5O_{12}$ , respectively, which is consistent with the results of XRD discussed in Section 3.2.

The above results show that the minimum phase formation temperature for YIG is related to the Bi content in  $Bi_xY_{3-x}Fe_5O_{12}$ . Previous studies have shown that Bi-substituted YIG can be prepared at much lower temperature than that of pure YIG due to the lower melting temperature of  $Bi_2O_3$  [12,18,19]. Except that, in the present investigation, the annealing temperature of Bi-YIG is also lower than the phase formation temperature of Bi-YIG prepared by conventional ceramic method [3,4], which may offer many advantages in commercialization of the coprecipitation processing.

#### 3.2. Structure characterization of Bi-YIG NPs

The XRD pattern of Bi-YIG NPs is shown in Fig. 2. The evolution of crystalline phase of  $Bi_xY_{3-x}Fe_5O_{12}$  (x = 0, 1 and 1.8) is investigated as a function of annealing temperature. There is no characteristic peak corresponding to the garnet phase before 750 °C for the pure YIG NPs, and the characteristic peaks corresponding to YIP (JCPDS Card No.39-1489) appear at a higher temperature of 800 °C, as shown in Fig. 2(a). As the annealing temperature increased to 850 °C, the presented diffraction peaks are well corresponded to the cubic YIG phase (JCPDS Card

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