

## Microwave-assisted synthesis and characterization of Bi-substituted yttrium garnet nanoparticles

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### ABSTRACT

Bi-substituted yttrium iron garnet (Bi-YIG,  $\text{Bi}_{1.8}\text{Y}_{1.2}\text{Fe}_5\text{O}_{12}$ ) nanoparticles were prepared by microwave-assisted co-precipitation as well as conventional co-precipitation using ammonia aqueous solution as precipitant. The nanoparticles were characterized by thermal gravity-differential thermal analysis, X-ray powder diffraction, transmission electron microscopy, dynamic light scattering and vibrating sample magnetometer, respectively. The Faraday rotation of Bi-YIG modified PMMA slices was also investigated. Results demonstrate that the Bi-YIG nanoparticles prepared by microwave-assisted co-precipitation show smaller particle size and higher Faraday rotation than those prepared by conventional co-precipitation.

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### 1. Introduction

Yttrium iron garnet (YIG,  $\text{Y}_3\text{Fe}_5\text{O}_{12}$ ) and substituted ones have been extensively studied for decades for their magneto-optical applications, and have been widely used in electronic devices, such as circulators, isolators and phase shifters for microwave and magneto-optical devices [1–4]. Bismuth-substituted yttrium iron garnet (Bi-YIG) has received much attention, since it has been found that Bi-YIG has large Faraday rotation in the visible and infrared wavelength region and the doping of bismuth can enhance the Faraday effect of the YIG [5,6].

The synthesis of monodispersed, nanosized particles is of key importance since the properties of nanoparticles strongly depend on their dimensions. A variety of methods have been used to prepare YIG nanoparticles, including ball milling [7,8], co-precipitation [9–12], microemulsion [13], hydrolysis of metal alkoxides and amorphous citrate gel [14–16]. Each method has its own distinctive advantages. Among them, ball milling of  $\text{Y}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$  powders is usually used to prepare YIG, however, this method needs a high sintering temperature. In microemulsion

processing, one can prepare particles with controllable size by varying the molar ratio of water to surfactant, however, large amount of solvent is used to synthesize small amount of material. The process is thus not efficient and rather difficult to be scaled up [17]. The sol-gel method can be used to synthesize high-purity ceramic materials with excellent homogeneity, but the method is time-consuming. The co-precipitation is a facile and convenient way to synthesize iron oxides and is easy to be scaled up. The size, shape and composition of the particles strongly depend on the type of salts used.

Since 1975, Williams [18] reported that some chemical reactions could be accelerated by the irradiation of microwave; the application of microwave in liquid reactions has been rapidly growing. Comparing with conventional process, one can find that microwave irradiation synthesis is generally quite faster, simpler and more energy efficient. Recently, microwave has been introduced in the preparation of nanoparticles [19–21]. Under microwave irradiation, reactants can be uniformly heated in short time, and precipitates are generated simultaneously and uniformly dispersed throughout the solution, which results in obtaining homogeneous nanoparticles.

Herein, we combined co-precipitation method with microwave technique (thereafter named microwave co-precipitation) and synthesized uniform Bi-YIG particles. Moreover, a contrastive study has been conducted to compare the properties of YIG powders prepared by microwave-assisted and conventional

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co-precipitation. The transparency and Faraday rotation of Bi–YIG modified PMMA slices were also investigated.

## 2. Experimental

### 2.1. Materials

Ferric nitrate ( $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ), yttrium nitrate ( $\text{Y}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ ) and bismuth nitrate ( $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ ), nitric acid, polyethylene glycol 20000 (PEG-20000) and ethanol were all analytical grade. Methyl methacrylate (MMA) and azobisisobutyronitrile (AIBN) were all chemical grade. All materials mentioned above were employed directly except that the AIBN was recrystallized with ethanol and MMA monomer was purified by distillation under reduced pressure. Ammonia aqueous solution (25 wt%) was used as precipitant and deionized water was used throughout the experiments.

### 2.2. Preparation of Bi–YIG ( $\text{Bi}_{1.8}\text{Y}_{1.2}\text{Fe}_5\text{O}_{12}$ ) particles

The microwave co-precipitation process was similar to the conventional co-precipitation [12]. In a typical procedure, a mixture of bismuth nitrate, iron nitrate and yttrium nitrate was used as starting solution where these nitrates were mixed according to the ratio of Bi, Y and Fe equal to the composition of  $\text{Bi}_{1.8}\text{Y}_{1.2}\text{Fe}_5\text{O}_{12}$ . Afterwards, a certain amount of surfactant (PEG-20000) was added to avoid aggregation. Such solution was added into the ammonia aqueous solution (pH = 11) dropwise with vigorous stirring under the irradiation of microwave, and some more ammonia aqueous solution was added to keep the pH constant during the co-precipitation. The obtained slurry was washed with deionized water and ethanol for several times then dried in an oven under vacuum. Finally, Bi–YIG particles were obtained after being calcined at 700 °C for 1 h. As a contrast, a sample prepared by conventional co-precipitation. The sample prepared by microwave-assisted or conventional co-precipitation was named sample M1 or C2, respectively.

### 2.3. Preparation of modified PMMA slices

Bi–YIG modified PMMA slice was prepared by bulk polymerization. Bi–YIG particles were mixed with MMA monomer, the result mixture was then milled with a planetary milling machine operating at the speed of 40 rpm for 3 h. PMMA slices named M-P1 and C-P2 which was modified by sample M1 and C2, respectively, were prepared after polymerization and solidification. The content of the particles is 0.2 wt% and the thickness of the slice is about 2.5 mm.

### 2.4. Characterization

Thermal behavior of Bi–YIG precursors was measured by thermogravimetry (TG) (Perkin-Elmer TGA7) and differential thermo-analysis (DTA) (TA Instruments, SDT-2960). The structure of Bi–YIG particles was identified by X-ray powder diffraction (XRD) measurements with a D/Max-III C, using Cu–K $\alpha$  radiation. The diameter of Bi–YIG particles was determined from transmission electron microscopy (TEM) images using H-600-II transmission electron microscope (Hitachi, Japan) and from analysis of the broadened major peak of XRD spectra. The size distribution of Bi–YIG particles was obtained using Malvern HPPS5001 laser particle-size analyzer. Magnetic properties of Bi–YIG particles were measured using a BHV-55 vibrating sample magnetometer (VSM). Ultraviolet-visible (UV-vis)

absorption spectrums of the Bi–YIG modified PMMA slices were achieved by HITACHI U-2810 spectrophotometer and Faraday rotation of the modified slices were measured by a Faraday rotation meter.

## 3. Results and discussion

### 3.1. Thermal analysis

The combined TG and DTA curves of Bi–YIG precursors (sample M1, prepared by microwave co-precipitation) are shown in Fig. 1. The TG curve shows an overall weight loss of 17.15%. The loss of mass is gradual, about 90% of the overall weight loss occurs in the temperature range 11–274.21 °C and an endotherm peak is found at 44.69 °C in the DTA corresponding to this weight-loss step. This event is the result of the dehydration process wherein significant amount of water is released from the gel matrix. The further 7.17% weight loss occurs in the temperature range 274.21–502.40 °C, corresponding to a broad exothermic peak in DTA. This event is due to the decomposition reaction of the hydroxide. A constant mass is obtained at temperatures as low as

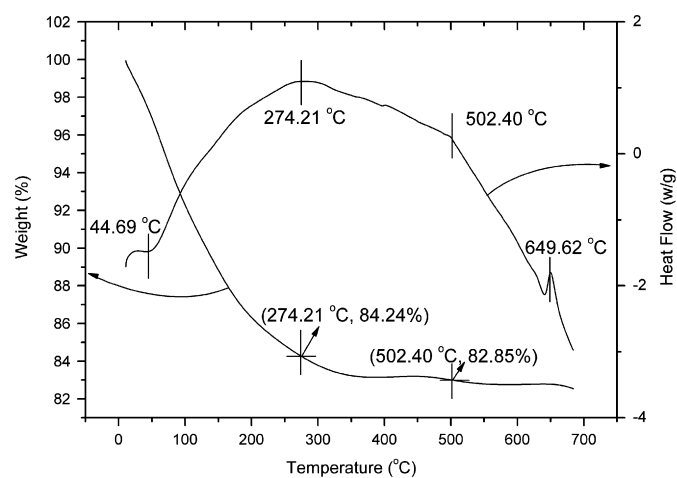


Fig. 1. Combined TG and DTA curves of Bi–YIG precursors (sample M1 prepared by microwave co-precipitation)

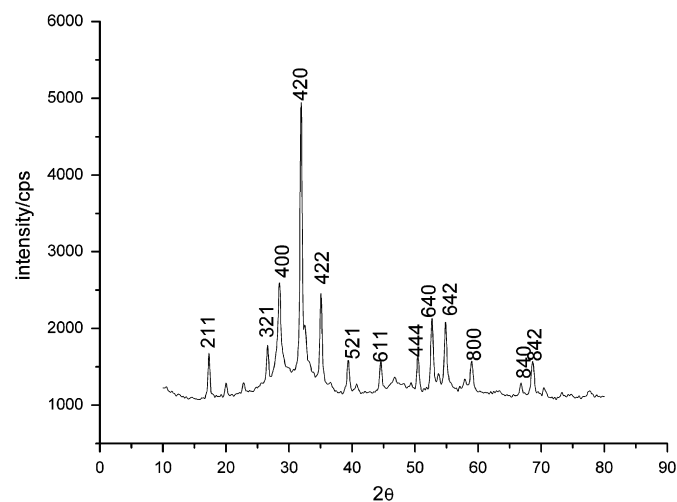


Fig. 2. XRD patterns of sample M1 annealed at 700 °C.

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