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Synthesis and magnetic properties of $Y_{3-x}Dy_xFe_5O_{12}$ nanoparticles

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Abstract

 Dy^{3+} -substituted garnet nanoparticles $Y_{3-x}Dy_xFe_5O_{12}$ (x = 0, 0.2, 0.4, 0.6, 0.8 and 1.0) were fabricated by a sol-gel method and their crystalline structures and magnetic properties were investigated by using X-ray diffraction (XRD), infrared spectroscopy and vibrating sample magnetometer (VSM). The XRD patterns of $Y_{3-x}Dy_xFe_5O_{12}$ have only peaks of the garnet structure and the sizes of particles range from 37 to 63 nm. Results of VSM show that the saturation magnetization of $Y_{3-x}Dy_xFe_5O_{12}$ ($0 < x \le 1$) particles is not only obviously less than pure Yttrium iron garnet, but also decreases with increasing the Dy concentration (x) in a linear manner. Meanwhile, it is observed that with the enhancement of the surface spin effects, the saturation magnetization rises as the particle size is increased. \mathbb{C} 2006 Elsevier B.V. All rights reserved.

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1. Introduction

Yttrium iron garnet (YIG) is a ferromagnetic material which is extensively used in optical communication and magneto-optical devices [1,2]. Ferromagnetic garnets are assigned to cubic structure (space group Ia3d), every cell contains eight $R_3^{3+}Fe_5^{3+}O_{12}$ molecules, and the ion distribution structure can be represented by writing the garnet formula as $\{R_3\}[Fe_2](Fe_3)O_{12}$; $\{\}$, [] and () are represented for 24c (dodecahedral), 16a (octahedral) and 24d (tetrahedral), respectively. YIG is the most representative and well-known compound among the rare earth iron garnets [3], and the various magnetizations can be achieved by substitution in the YIG [4]. Thongmee and Winotai [5] studied the magnetic properties of dilution of the Fe³⁺ by Al³⁺ in the substituted $Y_3Fe_{5-x}Al_xO_{12}$. They discovered that the saturation magnetization and coercivity both decreased in samples containing Al³⁺. Furthermore, because of the unique magneto-optical property of YIG, researchers have done a lot of work and succeeded in substituting Bi^{3+} and Ce^{3+} for Y^{3+} , and Co^{3+} and Co^{2+} for Fe^{3+} in YIG so as to obtain materials with different magneto-optical properties.

When YIG is prepared by ceramic methods, YFeO₃ and Fe₂O₃ are produced as intermediates and these phases remain as impurities unless heated to high temperatures [6,7], so the sol-gel method has attracted attention due to the lower synthesis temperature and finer and more homogeneous particles produced. A systematic study of the sol-gel technique for the synthesis of $Y_3Fe_5O_{12}$ is presented by Vaqueiro [3,8], and the results show that the citric is a suitable chelating agent to obtain the fine particles, which exhibit a rounded surface morphology and are without faceted borders. Meanwhile, YIG could be formed by a single-step process, where an amorphous precursor powder is formed first and then transformed to YIG during calcination, without any intermediate phase formation. The experimental results [9,10], obtained using the sol-gel method, indicate that the saturation

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magnetizations of Ce:YIG and La:YIG materials are different from pure YIG. Furthermore, Leleckaite and Kareiva use the sol–gel method to prepare rare earth-doped yttrium aluminum garnets YAG:Ln (Ln = Ce, Nd, Ho and Er),which are important solid-state laser materials widely used in luminescence systems, window materials for a variety of lamps and fiberoptic telecommunication systems [11].

In this article, we substituted the magnetic rare earth ion Dy^{3+} for some non-magnetic ions Y^{3+} in YIG, and prepared $Y_{3-x}Dy_xFe_5O_{12}$ (x = 0, 0.2, 0.4, 0.6, 0.8 and 1.0) nanoparticles using a sol-gel method. At the same time, the crystalline size and magnetic properties were observed and studied in detail.

2. Experiment

A stoichiometric mixture of Fe(NO₃)₃, Dy(NO₃)₃, Y(NO₃)₃ (Dy₂O₃ and Y₂O₃ were dissolved in HNO₃) was dissolved in an aqueous solution of citric acid, and then NH₃·H₂O was added to adjust pH = 2. The resulting solution was heated at 353 K in order to obtain the gel; to obtain the Y_{3-x}Dy_xFe₅O₁₂ samples, the gels were dried initially at 383 K for 36 h and further heat treated in air between 1023 and 1373 K for 3 h.

The structure and the crystallite sizes were tested by X-ray diffractometer (SHIMADZU Co., Tokyo, Japan) in the 2θ range $25-60^{\circ}$ using CuK α radiation ($\lambda = 0.15405$ nm), and the crystallite sizes of the samples are estimated from the line width of the (422) XRD peaks. The infrared (IR) spectra were recorded as KBr pellets on Perkin–Elmer Spectrum One FT/IR spectrometers.

Magnetic measurements were carried out at room temperature using a vibrating sample magnetometer (VSM; Digital Measurement System JDM-13) with a maximum magnetic field of 10 000 Oe.

3. Results and discussion

3.1. Structure characterization

3.1.1. X-ray diffraction

Fig. 1 shows XRD of samples with Dy concentration (*x*) from x = 0 to 1 treated at 1073 K for 3 h. Through Fig. 2, the evolution of the crystalline phase of Y₂DyFe₅O₁₂ was investigated as a function of heating temperature, and the treating temperatures are from 1023 to 1373 K. From these XRD patterns ,one can observe that all samples have only a single phase of garnet structure and crystallization occurs around 1023 K, which is lower than the 1573 K used in the ceramic method [6]. This lower crystallization temperature could be related to the good homogeneity of the gel prepared at pH = 2. At the same time, one can observe that the crystallization of samples is more complete as the annealing temperature is increased.

According to the Scherrer's relationship, we calculated the crystalline sizes for samples with the same Dy concen-



Fig. 1. XRD of samples with Dy concentration (x) from x = 0 to 1, treated at 1073 K for 3 h.



Fig. 2. XRD patterns of $Y_2 Dy Fe_5 O_{12},$ samples heated from 1023 to 1373 K.

tration (x) but treated at different temperature. The obtained sizes increase as the heating temperature is increased from 1073 to 1373 K (Fig. 3). Furthermore, we can also observe that for similar annealing, the crystallite sizes of $Y_{3-x}Dy_xFe_5O_{12}$ ($0 \le x \le 1$) are similar to each other and the variation has no apparent regularity (Fig. 4). The phenomenon is probably due to the similar ionic

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