Fabrication and characterization of 
Ce: YIG thin film

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Abstract: Yttrium iron garnet (YIG) is widely used for microwave ferrite devices, especially for optical isolators. In this paper, ferrite ceramic target using cerium (Ce)-substituted YIG was made using ferric oxide (Fe$_2$O$_3$), cerium oxide (CeO$_2$) and yttrium oxide (Y$_2$O$_3$) powders as raw material. Ce; YIG thin films were fabricated by radio frequency (RF) magnetron sputtering. The experimental result showed that the polycrystalline YIG was obtained after sintering at 1350 °C for 6 h, and the polycrystalline Ce; YIG thin films were achieved annealing up to 700 °C in the air.

Key words: optical isolator; Ce; YIG thin film; crystallization

1 Introduction

An optical isolator is a necessary component in a laser based optical system. By blocking optical feedback in the laser, an isolator is indispensable for eliminating one of the main noise sources. The nonreciprocity caused by magneto-optical (MO) effects is the basis of an optical isolator.

Recently, there are types of isolator structure based on optical waveguides$^{[1-3]}$. Most research toward integrated optical waveguide isolators has been focusing on using MO ferromagnetic iron garnet materials in order to induce optical nonreciprocity in the component, because of their unique combination of low optical loss at telecom wavelengths and a considerably strong MO effect, and the source of the nonreciprocity. Yttrium iron garnet (YIG) is a typical ferrimagnetic material. It exhibits a large MO effect and low propagation loss, and is extensively used in optical communication and MO devices$^{[4-5]}$. It has been discovered that MO effect of YIG has greatly enhanced if doping with cerium (Ce). Compared with YIG and bismuth (Bi)-substituted YIG, they show a much lager Faraday rotation angle$^{[6-9]}$. Because the sintering temperature of Ce; YIG is about 100 °C lower than that of pure YIG which is above 1450 °C$^{[10]}$, it is possible to get smaller grains and lower coercivity ($H_c$)$^{[11-12]}$.

In this paper, we utilized two processes to prepare the MO thin films of Ce; YIG. Oxide process for solid phase synthesis was used to prepare the ceramic target and Ce; YIG thin films were grown on silicon substrate by radio frequency (RF) magnetron sputtering method. The grown amorphous Ce; YIG thin films were annealed at different temperatures.

2 Experiment

2.1 Preparation of the ceramic target

Oxide powders of ferric oxide (Fe$_2$O$_3$), cerium oxide (CeO$_2$) and yttrium oxide (Y$_2$O$_3$) with the purity of 99.99% were used as raw materials. Firstly, the Fe$_2$O$_3$, CeO$_2$ and Y$_2$O$_3$ powders were evenly mixed with the molar ratio of 2.5:1:1 after weighed by an analytical balance. Secondly, the wet powders were dried after ball-milling in the tank for 8 h, and then the powders are continuously ground, poured into 5% polyvinyl alcohol (PVA) solution, and sieved through a 60 mesh screen. To prepare a wafer with 3 inches (1 inch=25.4 mm) diameter, the suppressing molding is at 40 MPa and the holding time is 10 min. Finally, the target is sintered at 1350 °C. To avoid target cracking, the temperature is increased from room temperature up to 200 °C at the speed of 100 °C/h, and the heat preservation time is 2 h. And then the temperature increased up to 1350 °C at the speed of
3 °C/h and insulated for 6 h. Finally, the temperature is cooled down to room temperature again.

The target is tested using a scanning electron microscope (SEM), an X-ray diffraction (XRD) instrument and a vibrating sample magnetometer (VSM).

2.2 Fabrication of Ce: YIG thin film

Ce: YIG thin films were made by RF magnetron sputtering. The process parameters were shown in Table 1. In order to avoid the target and equipment to be damaged at high temperature, there is a 5 min interval for each 20-min sputtering. The film thickness is about 120 nm after sputtering for 80 min. The films were annealed at 600 °C and 700 °C in the air with the heating rate of 150 °C/h.

Table 1 Sputtering process parameters of Ce: YIG

<table>
<thead>
<tr>
<th>Target</th>
<th>Substrate</th>
<th>Substrate temperature</th>
<th>Sputtering atmosphere</th>
<th>Sputtering pressure</th>
<th>Sputtering power</th>
<th>Base vacuum</th>
</tr>
</thead>
<tbody>
<tr>
<td>3 inches diameter</td>
<td>Si (111)</td>
<td>Room temperature</td>
<td>Ar</td>
<td>1.33 Pa</td>
<td>150 W</td>
<td>&lt;1.0×10⁻⁷ Pa</td>
</tr>
</tbody>
</table>

3 Results and discussion

3.1 Characterization of the ceramic target

A SEM is used to measure the Ce: YIG ceramic target. The image of the target is shown in Fig.1. It can be seen that the sintered ceramic target is uniform and meticulous microstructures while a small amount of pores are evenly distributed. The evolution of the crystalline phase of Ce₃Y₂Fe₅O₁₂ was investigated and proved to be related to the heating temperatures as shown in Fig.2. The synthesis of solid phase sintering YIG generally has the following two reactions.

\[
\begin{align*}
\text{Fe}_2\text{O}_3 + \text{Y}_2\text{O}_3 &\rightarrow 2\text{YFeO}_3 \\
\text{Fe}_2\text{O}_3 + 3\text{YFeO}_3 &\rightarrow \text{Y}_3\text{Fe}_5\text{O}_{12}
\end{align*}
\]

Fig.1 SEM image of Ce: YIG ceramic target

Powders calcined below 1 200 °C have mixed phases of garnet, Fe₂O₃, and YFeO₃ structures. After sintered at 1 350 °C, it became a polycrystalline YIG. During the sintering process, CeO₂ always exists because of radius difference between Ce and Y ions.

The saturation magnetization of ceramic target was measured by a VSM. The measurement result is shown in Fig.3. It can be seen that Ce: YIG is classified ferrimagnetic material and the magnetic susceptibility is significant positive (χ > 0). Saturation magnetization of Ce: YIG is about 17.8 emu/g, and coercivity (Hc) is about 50 G. When Ce: YIG reaches the saturation magnetization, the applied magnetic field is very small. It means that Ce: YIG is suitable for making waveguide devices.

Fig.2 XRD results at different temperatures

Fig.3 M-H hysteresis loop of the ceramic target
3.2 Crystallization of Ce: YIG thin films

The Ce: YIG thin films grown on silicon substrates by RF sputtering have smooth uniform surface, but there is a large number of oxygen vacancies and crystal defects. These influence the performance of the films. Film crystallization consists of two processes, nucleation and growth. At the nucleation stage, certain activation energy is necessary and the crystallization temperature is very low. There is no sufficient activation that can make thin films crystallization. Fig.4 shows the films deposited on the silicon substrate were amorphous when annealed below 600 °C. However, grains were widely distributed in the film surface when the temperature increases up to 700 °C. The sizes of particles were relatively uniform. Fig.5 shows the surface morphology. Therefore, the ideal crystallization temperature is 700 °C.

![Fig.4 SEM images of the film annealed at different temperatures](image)

![Fig.5 Surface morphology of Ce: YIG film annealed at 700 °C](image)

4 Conclusions

In this paper, precursor powders of Ce; YIG were synthesized using solid reaction method, and polycrystalline Ce: YIG ceramic target was prepared at sintering temperature of 1350 °C. The Ce: YIG garnet was relatively easy to achieve magnetic saturation, and had a good prospect of application in integrated optical isolators. Amorphous Ce: YIG thin films with good uniformity were obtained using RF sputtering method, and crystallization films were prepared after annealed at 700 °C in the air.

References

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