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# Transparent Tb<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub> ceramics as Mid-IR isolator

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#### 1. Introduction

Re<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub> (Re: Lanthanide rare earth elements) is a promising isolator material in the 1.3–1.5  $\mu$ m band, and in particular, Bi doped single crystals are widely applied to optical communication [1,2]. However, Re<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub> (without adding Bi) can be synthesized only by FZ (Floating Zone) method because it passes through peritectic reaction [3], and there are problems in size limitation and high production cost, which made it difficult to use in industrial applications. In the case of Bi-doped Re<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub>, additional technical problem is the difficulty of solid solubility of Bi in the host material. Therefore, it can be manufactured only by the LPE (Liquid Phase Epitaxial) method [2], and a thick film of approximately 1 mm can be grown on single crystal wafer mainly composed of GGG (Gd<sub>3</sub>Ga<sub>5</sub>O<sub>12</sub>) composition with crystal lattice constant-matching control. Also, the growth rate is very slow, leaving problems on productivity and production cost.

In the case of the isolator application,  $\text{Re}_3\text{Fe}_5\text{O}_{12}$  single crystals with  $\langle 111 \rangle$  orientation are generally used because of low free energy of crystal growth and the magnetic anisotropy issue ( $\langle 111 \rangle$  is the easy axis of magnetization) [4]. Therefore, only single crystalline materials have been used for isolators, and studies on polycrystalline ceramic materials have not been performed. Imaeda et al. succeeded in the development of  $\langle 111 \rangle$  YIG single crystal material with ceramic fabrication technology by applying abnormal

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#### ABSTRACT

Fabrication of transparent TIG (Tb<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub>) ceramics promising for  $1.3-1.5 \,\mu$ m optical isolator was successful for the first time by a solid-state reaction between Tb<sub>4</sub>O<sub>7</sub> and Fe<sub>2</sub>O<sub>3</sub>. The obtained TIG ceramics was free from grain boundary phases and inclusions but some residual pores were observed in the depth direction. Its in-line transmittance reached ca. 50% in infrared region of  $1.1-2.0 \,\mu$ m. Faraday rotation angle of the TIG ceramics was 350deg./cm for 1300 nm, which is about 1.5 times larger than that of the YIG (Y<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub>) single crystal. Removal of those residual pores can significantly improve optical properties.

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grain growth behavior without melting the materials, and they obtained magnetic properties which are similar to the single crystal grown by melt and solidification process. However, the optical quality (especially the insertion loss) was remarkably low-grade [5]. Regarding iron garnet ceramics, powder synthesis [6] and some reports on synthesis of sintered bodies [7,8] can be found in the literatures. However, due to the above mentioned theoretical problems and also the requirement of advanced synthesis techniques, there were no reports on polycrystalline Re<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub>, as well as polycrystalline transparent TIG ceramics.

In this report, we succeeded for the first time in the synthesis of transparent TIG ceramics which has the most excellent magnetic properties and temperature characteristics among Re<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub> materials. We have confirmed that there was no problem with optical characteristics and Faraday rotation function of the TIG ceramics and its optical quality can be further improved for practical application by optimizing the fabrication conditions.

#### 2. Experimental procedures

For synthesis of TIG ceramics,  $Tb_4O_7$  (agglome-rated particle size:  $3 \mu m$ ) and  $Fe_2O_3$  (particle size:  $1 \mu m$ ) with high purity of 99.99 wt% were used. Both of the raw materials were weighed to have a stoichiometric composition, a small amount of dispersant and binders were added, and then ball milled with steel balls in ethanol for 10 h. Here, the amount of wear of the steel ball during milling was measured, and an extra amount of  $Tb_4O_7$  was added in advance to compensate the trace amount of  $Fe_2O_3$  increased during ball milling. The ethanol solvent was removed by drying the







obtained slurry at 90 °C. Powder compacts with sizes of  $\phi 12 \times 5$  mm,  $\phi 42 \times 6$  mm and  $10 \times 100 \times 30$  mm were prepared by a uniaxial press, followed by CIP (Cold Isostatic Press) at 98 MPa, and then calcined at 900 °C for 3 h to remove the residual binders. After that, they were pre-sintered in oxygen gas at 1320 °C for 3 h and then treated with HIP (Hot Isostatic Press) furnace at 1300 °C for 1.5 h under a pressure of 176 MPa using 10%O<sub>2</sub>-Ar gas medium. After adjusting the thickness of the obtained ceramic samples, both surfaces were mirror polished and their magneto-optical properties were measured.

#### 3. Results and discussion

TIG ceramic sample having a thickness of  $100 \mu m$  (both surfaces mirror-polished) was observed by a transmission near-infrared microscope with a wavelength of 900 nm light under non-polarized (open nicol) and polarized (cross nicol) conditions.

Results are shown in Fig. 1(a) and (b). Birefringences were not detected inside the material. Non-cubic phase, grain boundary phase and optical stress were not observed in the materials. In the transmission image, residual pores of approximately 1  $\mu$ m are observed in the depth direction of the material, which are the only scattering sources in transmission optical microscope observation. Fig. 1(c) shows a reflection microscope photograph of the same sample after thermal etching at 1300 °C. The material was composed of uniform grains of approximately 5–10  $\mu$ m, and scattering sources such as pores and inhomogeneous phases cannot be detected in the surface observation. In addition, fracture surface of the same sample observed by SEM (Scanning Electron Microscopy) is shown in Fig. 1(d). It was a highly dense microstructure with pore free condition as the surface. The fracture surface of the material was almost transgranular fracture.

Fig. 2 shows the observation at the triple point of the material by HR-TEM (High Resolution Transmission Electron Microscopy). The

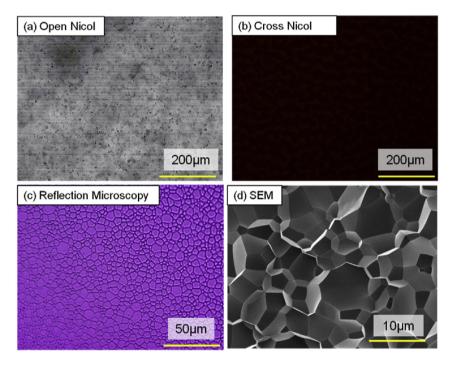
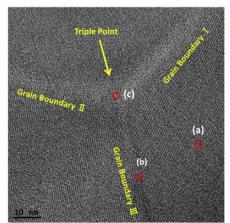


Fig. 1. (a) Open and (b) cross nicol images by polarizing transmission microscopy, (c) reflection microscopy image after polished and thermal etching, and (d) fracture surface of TIG ceramics by SEM.



	Fe / at%	Tb /at%
Point (a)	62.46	37.54
Point (b)	62.56	37.44
Point (c)	62.36	37.65

Fig. 2. High -resolution TEM image around triple point of produced TIG ceramics and EDS analysis of inner grain, grain boundary and triple point of TIG ceramics.

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