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Fabrication of Thin YIG Ferrite Platelet Using SPS method

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Abstract

Preparation method of YIG (yttrium iron garnet) ferrite platelet compacts for small height isolators has been proposed. The YIG ferrite platelets with thicknesses from 0.2 mm to 1.0 mm were sintered by Spark-Plasma-Sintering (SPS) method. The as-sintered compacts had a relatively high coercivity of about 55 Oe and a too large half value width of a ferromagnetic resonance (⊿H, which relates to insertion loss of isolators) of approximately 650 Oe. To improve the magnetic properties, the compacts were post-annealed in air using an infrared ray gold image furnace in which rapid heating and cooling were possible. Both coercivity and ⊿H of the samples drastically decreased, and magnetization increased by post-annealing at 1100 degrees C and above. The samples annealed at 1350 degrees C showed lowest coercivity of 6 Oe and smallest ⊿H of 71 Oe. The post-annealed YIG ferrite platelet compacts with acceptable magnetic properties for isolators were obtained even when the thickness of the compacts was reduced to 0.2 mm.

Key words: YIG ferrite, SPS, sintering, post-annealing, isolator, ⊿H

1. Introduction

Isolators and circulators play an important role to control a transmission direction of electromagnetic wave in mobile phones. Although height of most of all the electronic component devices used in mobile phones are decreasing toward 1mm, the height of the isolators still remains at 1.6 mm in the lowest case. Therefore, the development of isolator and circulators with a small height is an urgent and most important subject. We have succeeded in design of the circulators with a height of 1mm [1]. In this circulator, a 0.2 mm thick YIG (yttrium iron garnet) ferrite platelet compact is assumed to be used as the medium in which electromagnetic wave propagates. The thickness is less than a half of that of the YIG ferrite used in current isolator products.

Conventional sintering method using an electric furnace is employed to prepare the YIG ferrite compacts for current isolator products. In order to prepare 0.2 mm thick YIG ferrite compacts, additional process such as slicing and polishing are necesasry after sintering. As a novel preparation method of very thin YIG ferrite platelet compacts at an acceptable production cost, we are focusing on Spark-Plasma-Sintering (SPS) method [2]. In this method, starting powder materials are heated directly in a mold composed of electrically conductive die and punches, through which a large pulsed direct current flows. High density compacts are obtainable because the starting powder materials suffers one axis pressurization from the punches [3]. It is expected that thin compacts may be easily produced without slicing process if the quantity of starting material is decreased.

The purpose of this study is to develop the fabrication method using SPS and investigates optimal process condition of thin YIG ferrite compact for isolators.

2. Experiment

Recently SPS technology is much interested as a new sintering method to prepare ceramics materials [4-6]. Starting powder materials suffer pressure in uniaxial direction and are heated up by Joule heat and the effect of spark plasma generated in the gap between powder materials in case of electrically conducting starting materials. Specific features of SPS include lower sintering temperature and processing time much shorter than conventional other sintering method.

Figure 1 shows a schematic illustration of an SPS apparatus (SPS-1050, Sumitomo Coal Mining Co.) used in this experiment. Starting material (ferrite powder) was put in a electrically conductive graphite

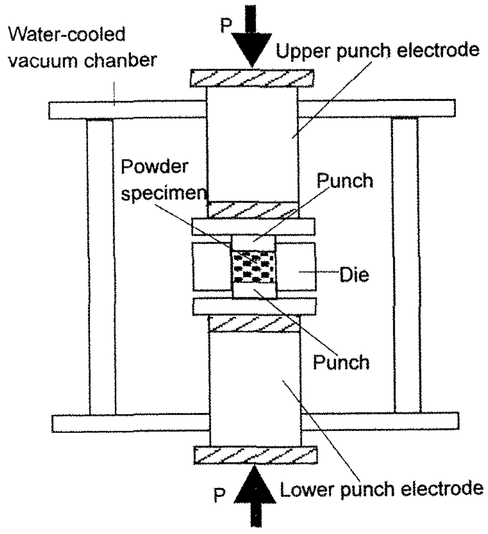


Fig.1 Schematic illustration of SPS apparatus.

die, and was pressed from both up and down side by a pair of graphite punches. Carbon films were inserted between ferrite powder and graphite die/punches as a separator. A DC on and off current was applied to the punches.

YIG ferrite platelet compacts were sintered by SPS method in a vacuum atmosphere under pressurization of 0.3 tf/cm^2. The temperature was rose to 800 ℃ at a rate of 100 ℃/min, and was held for 5 minutes, and then cooled down to 600 ℃ at a rate of 20 ℃/min and then naturally cooled to room temperature. The reason why the sintering temperature was set at 800 ℃ was that the YIG ferrite samples melted at a higher temperature. The samples sintered by SPS method had a coercivity of over 55 Oe and ⊿H value of 650 Oe. These magnetic properties were insufficient to be used for the isolators [7].

Therefore, the post-annealing was tried to improve the magnetic properties. The samples prepared using SPS method was annealed with an infrared ray gold image furnace (Model MR-39S/H, ULVAC) with maximum operating temperature of 1800 ℃. As a comparison, the samples was also annealed with a conventional electric furnace with maximum operating temperature of 1400 ℃. In use of the electric furnace, temperature was risen to the setting temperature (annealing temperature) with a rising rate of 10 ℃/min, and after the temperature was held for 2 hours, then naturally cooled down to room temperature. In use of the infrared ray gold image furnace, rapid heating and cooling is possible. To investigate the feasibility of short time annealing, temperature rising rate was set at 200 ℃/min, and after temperature was held at annealing temperature for only 10 minutes, it was cooled to 100 ℃ at 100 ℃/min. After that, samples are naturally cooled to room temperature. Roughly, the total anneal processing time in the infrared ray gold image furnace use is 1/8 of that in the electric furnace use.

3. Results and Discussion

The post-annealing temperature dependence of saturation magnetization and coercivity of YIG ferrite sintered compacts are shown in figure 2 and 3, respectively. Post-annealing temperature was varied in the range below 1500 ℃, because the YIG ferrite samples melted at temperatures over 1500 ℃. The as-sintered sample has a saturation magnetization of 13.50 emu/g and a coercivity of 67 Oe.

Although there is a slight difference in the annealing temperature dependence of saturation magnetization and coercivity between the electric furnace and the infrared ray gold image furnace use, saturation magnetization increased and coercivity decreased with rising annealing temperature. For the reference, broken lines in figure 2 and 3 shows the typical values for the YIG bulk ferrite compacts sintered by conventional sintering method using an electric furnace for long sintering time over ten hours.

The sample post-annealed at 1350 ℃ showed the saturation magnetization of 14.44 emu/g which was almost same as that of the YIG bulk ferrite sintered in conventional method using electric furnace. Coercivity

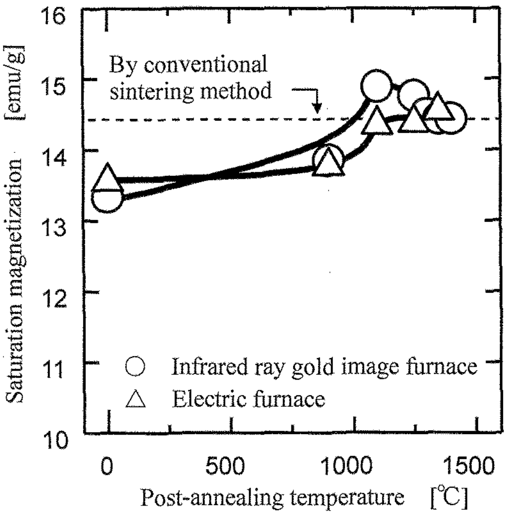


Fig.2 Post-annealing temperature dependence of saturation magnetization.

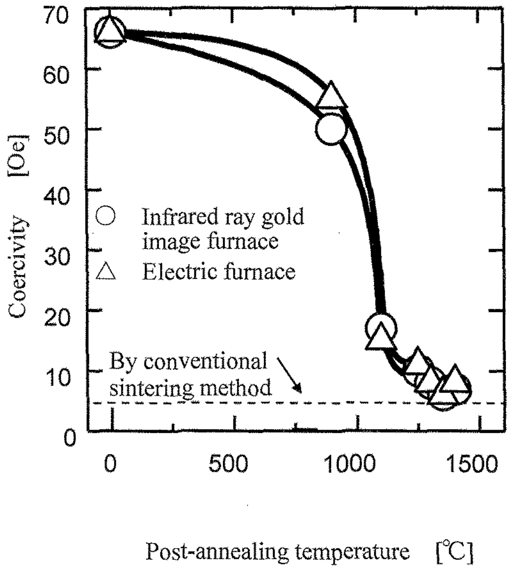
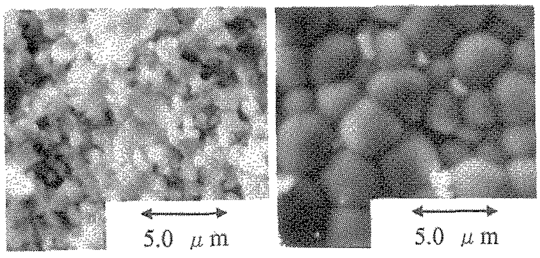


Fig.3 Post-annealing temperature dependence of coercivity.



(a) As-sintered (b) Post-annealed

Fig.4 SEM cross-sectional photographs of YIG ferrite compact: (a) before and (b) after post-annealing.

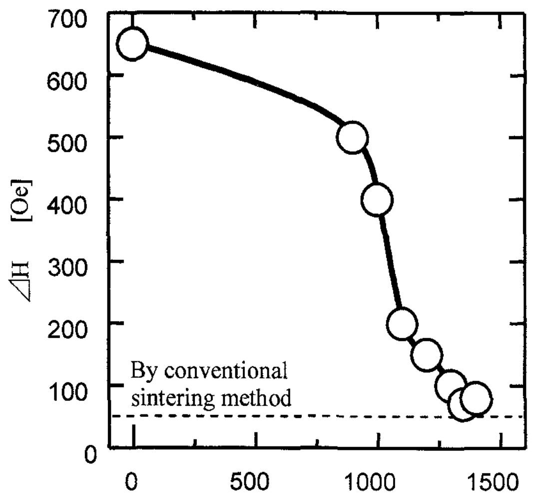


Fig.5 Annealing temperature dependence of ⊿H.

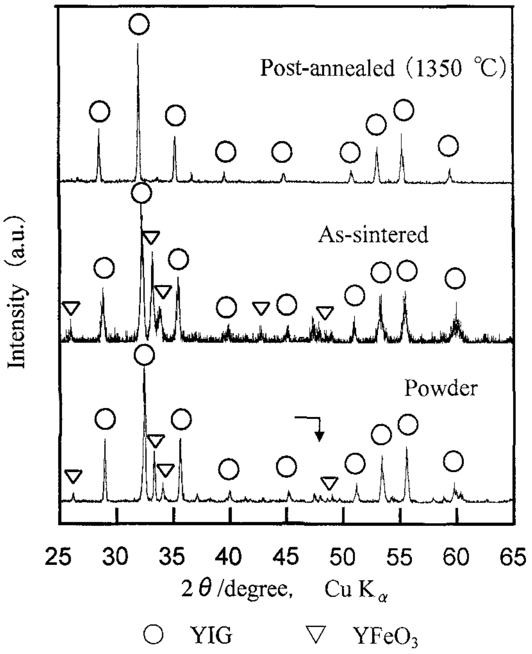


Fig.6 XRD diagrams for post annealed sample (top), as-sintered (middle), and powder of starting method (bottom).

of the sample post-annealed at 1350℃ reaches to 6 Oe.

Fig.4 shows SEM cross-sectional images of (a) as-sintered and (b) 1350℃ post-annealed YIG ferrite compacts. The grains grew by post-annealing, and average grain size changed from 0.8 μm (as-sintered) to 3.0 μm (after post-annealing). The increase of saturation magnetization by post-annealing was due to diminishment of pores. Grow of grains caused magnetic wall motion [8], and resulted in the achievement of low coercivity.

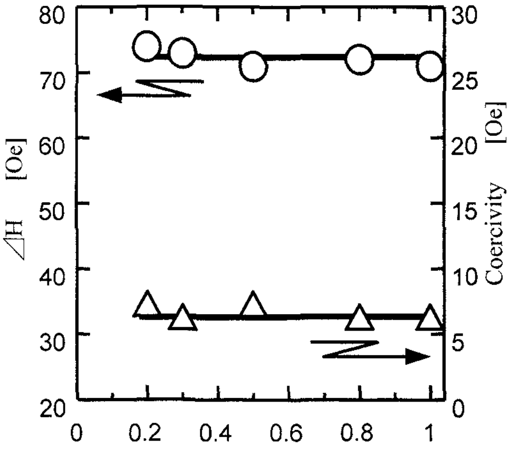


Fig.7 Thickness dependence of magnetic properties of YIG ferrite sintered compact.

From these data, almost identical annealing effects on magnetic properties were confirmed in use of the two different furnaces. From the viewpoint of mass production, there is a merit in saving a post-anneal processing time in annealing with infrared ray gold image furnace use.

Figure 5 shows Post-annealing temperature dependence of ⊿H of the YIG ferrite sintered compact post-annealed with the infrared ray gold image furnace. ⊿H defined as a half-value width of ferromagnetic resonance curves was determined from the frequency characteristics measured one of S parameters, S_{11} which represents reflection characteristics with a network analyzer. The ⊿H value greatly decreased when the YIG ferrite compacts were post-annealed at over 1100 ℃. The sample post-annealed at 1350 ℃ showed lowest ⊿H of 71 Oe which was almost same as that sintered by conventional sintering method with electric furnace only and used current isolator/circulator application [7].

⊿H of ferrite samples is increased from that of a single crystal by some reasons: anisotropy magnetic field, existence of nonmagnetic portion such as pore or magnetic non-uniformity, and demagnetizing field caused by surface roughness and shape of the sample [8-11]. Primal reason of drastic decrease in ⊿H was diminishment of pore as shown in SEM image (Fig. 4) and of heterogeneous phase by post-annealing. To confirm the latter, crystallographic properties were studied.

Figure 6 shows XRD diagrams for three YIG ferrite specimens: starting materials (powder), as-sinterd compact, and compact post-annealed at 1350 ℃ using the infrared ray gold image furnace. Not only YIG phase but also $YFeO_3$ phase was observed in starting powder materials. The $YFeO_3$ phase were introduced to improve grindability of the starting materials. The $YFeO_3$ phase has still remained after SPS treatment, because the maximum sintering temperature was limited up to 800 ℃ preventing from sample melting. In the samples post-annealed at 1350 ℃, the $YFeO_3$ phase has completely vanished, and the diffraction peaks from YIG phase increased remarkably. Thus, drastic decrease in ⊿H by post-annealing is partly due to diminishment of heterogeneous phase.

Figure 7 shows the relationship between magnetic roperties of YIG ferrite compact post-annealed at 1350 ℃ and their thickness. Substantial change in ⊿H and coercivity was not found in the compact thickness range from 0.2 mm to 1.0 mm.

4. Conclusion

Preparation method of thin YIG ferrite platelet compacts has been proposed. The proposed preparation process is a combination of Spark-Plasma-Sintering at 800 ℃ and subsequent post-annealing at 1350 ℃ with an infrared ray gold image furnace. Preparation of 0.2 mm thick YIG ferrite compact with small ⊿H of about 70 Oe became possible. This work will thrust the low height isolators and circulators we have designed into practical production.

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