THE SIZE ESTIMATION OF THE Bi-YIG NANO-PARTICLES DISPERSED IN A PLASTIC BINDER

Teruyoshi Hirano
Toppan Printing, Co.,Ltd., TRI, Takanodai-minami, Sugito, Saitama, 345-8508 Japan

Tatsuru Namikawa
Tokyo Inst. Tech., Nagatsuta, Midori-ku, Yokohama, 226-8502 Japan

Abstract - **Nano-size $Bi_{1.8}Y_{1.2}Fe_5O_{12}$ particles were prepared with a coprecipitation and heat-treatment method. The coating films of the particles were prepared with coating techniques. The crystalline sizes of the $Bi_{1.8}Y_{1.2}Fe_5O_{12}$ particles were calculated using the Scherrer equation with the X-ray diffraction data. The calculated size of the 0 h milling particle is about 400 nm that is almost the same as the measured size with a transmission electron microscope. The calculated particle size of 100 h milling is about 50 nm that is almost the same as the measured size with an atomic force microscope. The size of the prepared $Bi_{1.8}Y_{1.2}Fe_5O_{12}$ particles is changed with the milling process. The crystalline size calculation using the Scherrer equation with the X-ray diffraction data is a method of a size estimation of nano-size $Bi_{1.8}Y_{1.2}Fe_5O_{12}$ fine particles.**

I. INTRODUCTION

We have been studying the preparation process and applications of Bi-YIG fine particles and their coating films for several years [1][2]. We established the preparation condition to make the nano-particle dispersed films by using coating and milling techniques. The particle size of the nano-Bi-YIG particles is an important aspect for transparent magneto optical coating films.

In nano-size regions, the size measurement has some difficulties. The sizes measured with some microscopic technologies were not average sizes of the particles. In this paper, we estimated the Bi-YIG particle sizes in the coating films in the Scherrer equation with X-ray diffraction data. We discussed the calculated crystalline sizes of the particles compared to the measured sizes with some microscopic technologies.

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T. Hirano, +81-480-33-9048, fax +81-480-33-9022, thirano @ tri . toppan . co . jp ; T. Namikawa, +81-45-924-5429, fax +81-45-924-5433, namikawa @ iem . titech . ac . jp

II. EXPERIMENTAL

A. Preparation of the Bi-YIG particles and coating films

The $Bi_{1.8}Y_{1.2}Fe_5O_{12}$ particles were prepared by coprecipitation and annealing processes [3]. Figure 1 shows the preparation process of the particles. Aqueous solutions of nitrates of Bi, Y and Fe were mixed where the ratio of the cations corresponded to the composition of $Bi_{1.8}Y_{1.2}Fe_5O_{12}$. The solution was mixed with a NH_4OH solution with stirring at room temperature. After the coprecipitation reaction, the *p*H of the solution was 12 ~ 13. The obtained slurry was washed, filtered and dried at 100°C for 10 h. Then the coprecipitate was heated in air at 700°C for 4 h. The crystal phases of the particles were examined by X-ray diffraction analysis. The particles were observed with transmission electron microscope (TEM).

The particles were mixed with an epoxy binder (Epo-tek 396 ; Epoxytechnology Co.) solved by a cyclohexanone and milled with a planetary milling machine (Pulverisette 7 ; Fritsch Co.) from 1 to 100 h, then were coated by a spin coater on Corning 7059 glass substrate. The thickness of the films was about 2 μm. It was controlled by the viscosity of the ink.

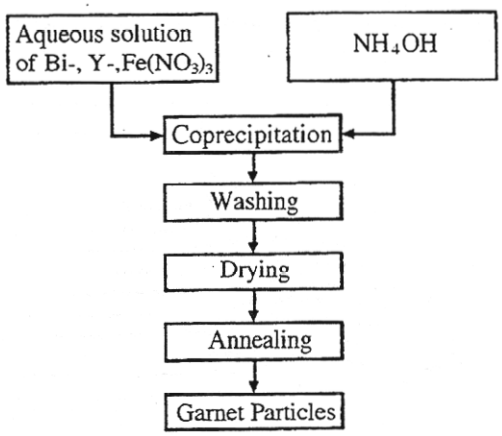

Fig. 1 The process of the particle preparataion.

The films were dried at 80°C for 1 h in an oven. The volume content of the particles in the coating films was about 0.2.

The particles in the films were observed with an atomic force microscope (AFM).

B. Estimation of the particle size

Using the X-ray diffraction data, the particle size was able to be measured in the Scherrer equation.

$$d = \frac{0.9\ \lambda}{B \cos\ \theta_B} \qquad (1)$$

In this equation, d : crystalline particle size (nm), λ : X-ray wavelength (Å), θ_B : diffraction angle (°), B : full width at half maximum (F.W.H.M.) of the diffraction peak at θ_B (rad).

The X-ray diffraction (XRD) was measured by the Rad-B diffract meter system (Rigaku Co.). The full widths at half maximum (F.W.H.M.) of the diffraction peaks were calculated with the RINT system (Rigaku Co.). The F.W.H.M. of the main diffraction peaks (420) of the Bi-YIG particles were used for size calculation. The θ_B of the (420) was about 16°, λ was 1.54 Å (CuKα).

III. RESULTS

A. Size calculation of the particles

Figure 2 shows the XRD patterns of the Bi-YIG coating films with various milling times. The (420) peaks of the particles were disappeared with the milling time.

Figure 3 shows the F.W.H.M. of the (420) peaks of the Bi-YIG particles in the coating films with various milling times. The F.W.H.M. increased with the milling time. This result indicates that the size of the Bi-YIG particles was decreased through the milling process.

Figure 4 shows the calculated particle size with the milling time. The size of the Bi-YIG particles was decreased by the milling process. The size of the 100 h milling particles was about 50 nm that was much smaller than the visible-lights wavelengths.

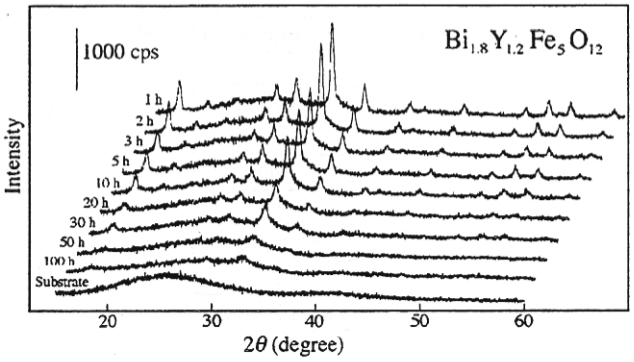

Fig.2 The XRD patterns of the Bi-YIG particles in the coating films.

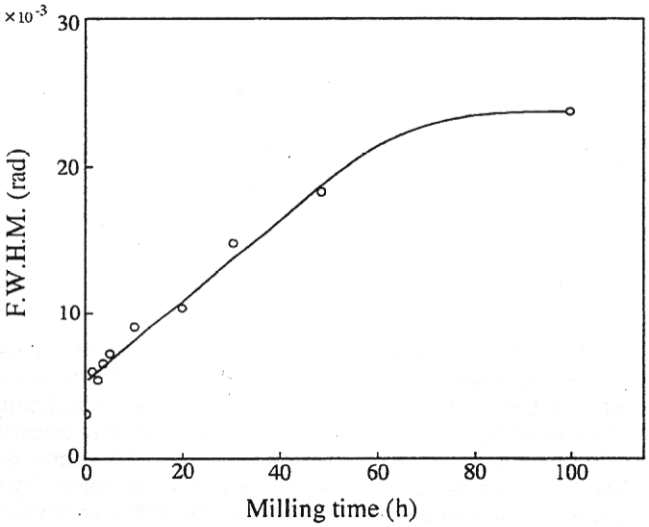

Fig.3 The Full Width at Half Maximum (F.W.H.M.) of the XRD peak (420) of the Bi-YIG particles.

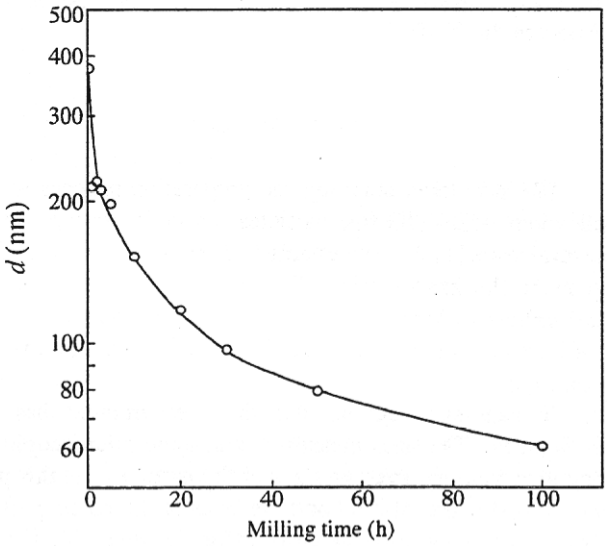

Fig.4 The calculated crystalline particle size of the Bi-YIG particles.

B. Estimating the calculated particle size

The calculated size d in the Sherrer equation (1) means crystalline size of the particle. Because the equation (1) is on X-Ray diffraction measurements. The d is not the same as the size that was measured with a TEM, an AFM and other measurements technologies. We estimated the particle sizes that were measured with the equation (1), a TEM and an AFM.

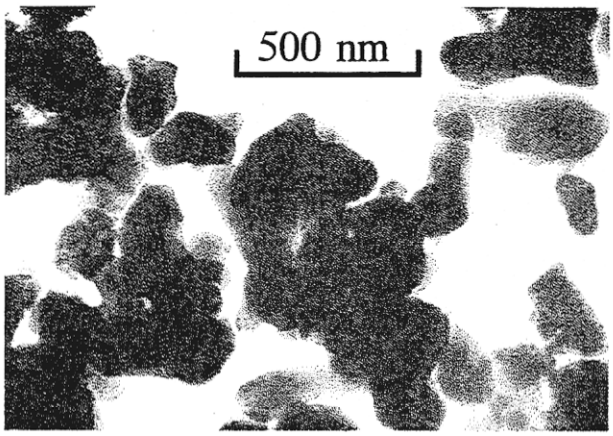

Fig. 5 The TEM image of the Bi-YIG particle. The size is about 300 nm.

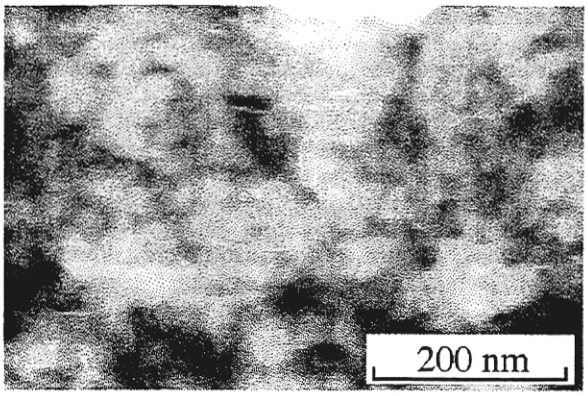

Fig. 6 The AFM image of the Bi-YIG particle in the 100 h milled film. The size is about 30 nm.

Figure 5 shows TEM image of the prepared Bi-YIG particles heat treated throughout 700°C, 4h. The *d* is about 300 nm. In the Figure 3, the *d* of 0 h milling particle is about 400 nm that is the almost same as the size measured with the TEM image.

Figure 6 shows the AFM image of the Bi-YIG particle in the 100 h milled film. The size of the particle is about 30 nm. In the figure 4, the *d* of 100 h milling particle is about 50 nm that is almost the same as the size measured with the AFM image.

IV. CONCLUSIONS

Nano-size Bi-YIG particles were synthesized. Thin films were prepared by coating technique with 0 to 100 h milling inks. The sizes of the particles in the films were calculated using the Scherrer equation with X-ray diffraction data.

The calculated size of the 0 h milling particle is about 400 nm that is almost the same as the measured size with a transmission electron microscope. The calculated particle size of 100 h milling is about 50 nm that is almost the same as the measured size with an atomic force microscope.

The size of the prepared $Bi_{1.8}Y_{1.2}Fe_5O_{12}$ particles is changed with the milling process. The crystalline size calculation using the Scherrer equation with the X-ray diffraction data is a method of a size estimation of nano-size $Bi_{1.8}Y_{1.2}Fe_5O_{12}$ fine particles.

REFERENCES

[1] T. Hirano, T. Namikawa and Y. Yamazaki, "Magneto-optical properties of Bi-YIG particles dispersed in a plastic binder", *Denki Kagaku*, vol. **64**, pp. 307-310, April 1996.

[2] Y. Yamazaki, T. Namikawa, T. Hirano and K. Yoshida, "Magnetic and magneto-optical properties of nano-size Bi-YIG particles dispersed in a plastic binder", *JOURNAL DE PHYSIQUE IV*, C1-543-544, 1997.

[3] Teruyoshi Hirano, Tatsuru Namikawa and Yohtaro Yamazaki, "Bi-YIG Magneto-Optical Coated Films for Visual Applications", *IEEE Trans. Magn.*, Vol.**31**, pp.3280-3282, No.6 1995.