

Thin Film Preparation by PLD Method Using Several Metal Powder Targets

複数種の粉体ターゲットを用いたPLD法による薄膜の作製

Hiroharu Kawasaki, Tamiko Ohshima, Yoshihito Yagyū, Takeshi Ihara, Yuki Tanaka and Yoshiaki Suda

川崎仁晴, 大島多美子, 柳生義人, 猪原武士, 田中雪, 須田義昭

*Department of Electrical and Electronic Engineering, National Institute of Technology, Sasebo College
1-1 Okishin, Sasebo, Nagasaki 857-1193, Japan*

佐世保工業高等専門学校 〒857-1164 長崎県佐世保市沖新町1-1

Bismuth(Bi), Iron(Fe) and their mixture thin films were prepared by a pulsed laser deposition method using a metal powder target. X-ray photoelectron spectroscopy, X-ray diffraction measurements of the films prepared using the powder target indicated nearly the same properties as those of films prepared using bulk target. Bi and Fe mixture powder target were also used to prepared $\text{Bi}_3\text{Fe}_5\text{O}_{12}$ thin film, which was used for magnet optics thin film. Experimental results suggest that prepared film were Bi rich film which may be due to low melting temperature of Bi(271 °C) compared with that of Fe(1539 °C)

1. Introduction

Pulsed laser deposition (PLD) is a widely used technique for the deposition of thin films. The PLD method uses a high power pulsed laser beam that is focused inside a vacuum chamber such that it strikes the target material[1-4]. In the PLD method, high density bulk targets are generally used. Therefore, when preparing a thin film with certain element ingredients using this method, it is necessary to form new targets by other methods, such as spark plasma sintering. However, these target making systems require considerable time, and are usually expensive. Therefore, the PLD method may become more attractive if powder material targets are used. Kajima et al prepared ferromagnetic nanocomposite oxide films were prepared by sputtering deposition using a $\text{Bi}_2\text{O}_3\text{-Fe}_2\text{O}_3\text{-PbTiO}_3$ ternary system and powder targets. They suggested that films prepared using powder targets worked well as thin film capacitors[5]. We also prepared several kinds of functional thin films using powder targets by sputtering deposition method[6-10]. However, film properties, such as crystallinity, composition ratio, hardness were not high enough to use for hard coating. If the thin film were prepared by PLD method, high crystallinity and hardness film may be prepared.

In this study, Bi, Fe thin films were prepared by a PLD method using a metal powder target, and the film properties were examined. In addition, the properties of the processing plasma were studied. Emission spectra of the sputtering plasma were measured with a monochromator. On the basis of the results, the mechanisms of thin film deposition

using powder targets were explored.

2. Experimental

The deposition chamber was fabricated of stainless steel with a diameter of 400 mm and a length of 370 mm. In this experiment, Bi and Fe mixture powder targets were used for $\text{Bi}_3\text{Fe}_5\text{O}_{12}$ thin film preparation. Diameter of the Bi powder (99.9 %) was 63-106 μm and that of the Fe powder (99.9 %) was 75 μm , respectively. The target holders were covered with the Bi, Fe and their mixed powders such that the powder was level on the target. A pulsed Nd:YAG laser (Spectra-Physics Quanta-Ray PRO-230-10; wavelength 532 nm, pulse duration 1-2 ns, maximum output energy 650 mJ) was used to irradiate the powder targets. The radiated area on the targets was maintained at 0.32 cm^2 . The laser fluences used were 0.36, 0.53 and 0.62 J/cm^2 . Si(100) and SiO_2 (TEMPAX float) substrates were located 2.2 cm from the targets. Prior to loading into the deposition chamber, the substrates were cleaned using an ultrasonic agitator with repeated bathing in ethanol, followed by rinsing in high-purity deionized water. The substrates were maintained at room temperature. The crystalline structure and crystallographic orientation of the thin films were characterized by XRD (Rigaku, RINT2100V) using $\text{CuK}\alpha$ radiation. The composition of the films was measured by XPS (JEOL JPS9010). The surface morphology of the films was observed using an AFM (JEOL JSPM4210). Film thickness was measured using α -step (Kosaka Laboratory Surfcoorder ET4000A).

3. Results and Discussion

Fig. 1 shows the dependence of the deposition rate on the Bi and Fe powder mixture. In this experiment, laser wavelength was 532 nm, and laser fluence was 1.0 J/cm^2 . As the results, deposition rate was increased with increasing Bi mixture. This may be due to the difference of melting and boiling temperature of Bi and Fe. Melting and boiling points of Bi are 544 K and 1837 K, and those of Fe are 1811 K and 3134 K, respectively. Both values for Fe are much higher than that of Bi. In general, pulsed laser deposition, which used $>400 \text{ nm}$ in wave length, target surface is evaporated by the thermal energy of laser, and then they deposited on the substrate surface as thin films. Therefore, it is considered that the energy which needs to evaporate Bi is lower than that for Fe. That may be due to high deposition rate of high Bi mixture powder target.

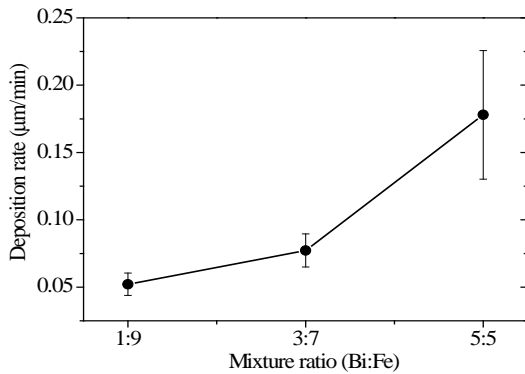


Fig.1. Dependence of the deposition rate on the Bi and Fe powder mixture

Figure 2 shows the dependence of the XRD pattern of the film prepared by PLD method on Bi and Fe powder target mixture. In this experiment, powder target mixture was Bi:Fe = 1:9 wt% ~ 5:5 wt%, laser wavelength was 532 nm, laser fluence was 1.0 J/cm^2 and O_2 gas was fed into deposition chamber and the gas pressure was 10 Pa. XRD patterns of Fe, Bi and their mixture powder were also shown in the Fig. 6. As the results, XRD patterns of the prepared films using mixture powder shows peaks of $2\theta=22^\circ, 27^\circ, 38^\circ, 40^\circ, 44^\circ, 46^\circ, 49^\circ, 56^\circ$. The peaks are almost same peaks of Bi powder, and the patterns are independent of the Bi:Fe powder mixtures. The result may be due to This may be due to the difference of melting and boiling temperature of Bi and Fe. XRD peak intensity was increased with increasing Bi powder mixture.

5. Conclusions

Bi and Fe thin films were prepared by a pulsed laser deposition method using their powder

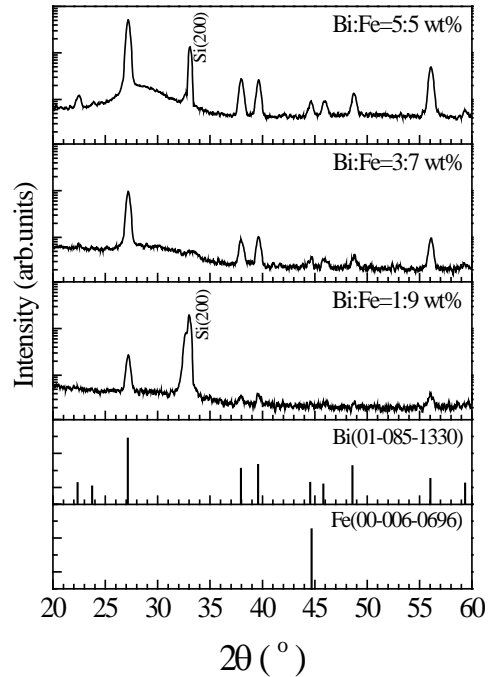


Fig.2. XRD patterns of the films prepared using Ti powder target as parameters of Ar: O_2 gas mixture.

target. X-ray photoelectron spectroscopy, X-ray diffraction measurements of the films prepared using the powder target indicated nearly the same properties as those of films prepared using a Ti bulk target. Bi and Fe mixture powder target were also used to prepare $\text{Bi}_3\text{Fe}_5\text{O}_{12}$ thin film. Experimental results suggest that prepared film were Bi rich film which may be due to low melting temperature of Bi (271°C) compared with that of Fe (1539°C)

References

- [1] J.-I. Song: Appl. Phys. Lett. **90** (2007) 022106.
- [2] N. Itagaki, T. Iwasaki, H. Kumomi, T. Den, K. Nomura, T. Kamiya, and H. Hosono: Phys. Status. Solidi A **205** (2008) 1915.
- [3] H. Fujiyama: Surf. Coat. Technol. **131** (2000) 278.
- [4] E. Morisaki and H. Fujiyama: Surf. Coatings Technol. **98** (1998) 834.
- [5] A. Kajima, T. Arita, Y. Tsuji, M. Inoue, and T. Fujii: J. Magn. Soc. Jpn. **30** (2006) 174, (2006) 062103.
- [6] D. Dzubrou, A. M. Grishin, H. Kawasaki, Y. Suda, and V. Pankov: J. Phys.: Conf. Ser. **100** (2008) 082035.
- [7] H. Kawasaki, K. Shibahara, T. Ohshima, Y. Yagyu, and Y. Suda: Jpn. J. Appl. Phys. **49** (2010) 08JF01.
- [8] H. Kawasaki, T. Ohshima, Y. Yagyu, and Y. Suda: Trans. Mater. Res. Soc. Jpn. **36** (2011) 495.
- [9] H. Kawasaki, T. Shigematsu, K. Imasaka, T. Ohshima, Y. Yagyu, and Y. Suda: Trans. Mater. Res. Soc. Jpn. **36** (2011) 479.
- [10] T. Ohshima, Y. Murakami, H. Kawasaki, Y. Suda, and Y. Yagyu: Jpn. J. Appl. Phys. **50** (2011) 08JD09.