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Efficient Approach to Measure Crystallization Temperature in Amorphous Thin Film by Infrared Reflectivity

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This paper shows a new effective approach to measure crystallization temperature of soft magnetic underlayer (SUL) for next generation of heat assisted perpendicular recording media. This approach uses temperature dependent reflectivity, which shows a clear jump when samples are crystallized. To achieve this measurement, an optical system is set up using hot plate and infrared laser. Reflectivity of SUL $(Co_{70}Fe_{30})_{92}Ta_3Zr_5$ shows a clear jump at its amorphous-crystalline transition temperature. Experiment results show this effect is clear in infrared region, and is weak for visible light.

Keywords : Amorphous thin film, Soft magnetic thin film, Optical property, Perpendicular recording media

1. Introduction

In perpendicular recording media, the SUL is a critical component for magnetic flux concentration [1]. In situ heating are usually used during deposition process of heat assisted magnetic recording media; as a result, magnetic properties of soft underlayer must be thermally stable. Therefore, the crystallization temperature of soft magnetic thin film is of great technological interest. The crystallization temperature was reported to be measured by successive annealing and X-ray diffraction measurement [2], and a rather long time is needed for this measurement. On the other hand, in phase-change optical memory, when its structure changes from amorphous state to crystallization state, its optical properties such as reflectivity has a clear changing [3]. In this paper, this effect is also found in metal, whose amorphous-crystalline transition will induce a clear shifting of infrared reflectivity. This process need much short time, and will be a new approach to achieve crystallization temperature in metal.

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2. Experiments and Results

In this experiment, sample with structure of CrTi(2 nm)/SUL(100 nm)/Si_3N_4(10 nm) is deposited on glass by DC sputtering, where the SUL is $(Co_{70}Fe_{30})_{92}Ta_3Zr_5$. No substrate heating is applied during films deposition, and its as-deposition state should be in amorphous state.

According to our earlier experiments, noise at high temperature is an big problem for temperature-dependent reflectivity measurement. Therefore, an optical system, as shown in Fig. 1, is set up for this measurement.

Fig. 1(a) is the experimental configuration, where samples are annealed on hot plate, and simultaneously reflectivity of weak infrared laser (1550 nm) was detected by high resolution detector. The laser intensity is modulated by the function generator with frequency of 320 Hz for higher signal/noise ratio. Its laser diode has temperature controller, so laser intensity wouldn't be affected by environment temperature or temperature of hot plate. Sample temperature is controlled by hot plate. Due to the temperature heating speed is rather low 500°C/hour, temperature of sample and of hot plate should be same, and we monitor the temperature by a contact thermal sensor. The distance between laser diode and the hot plate is about 0.5 m. Surface vibration of hot plate is the main reason of noise, especially the large noise at high temperature. In this

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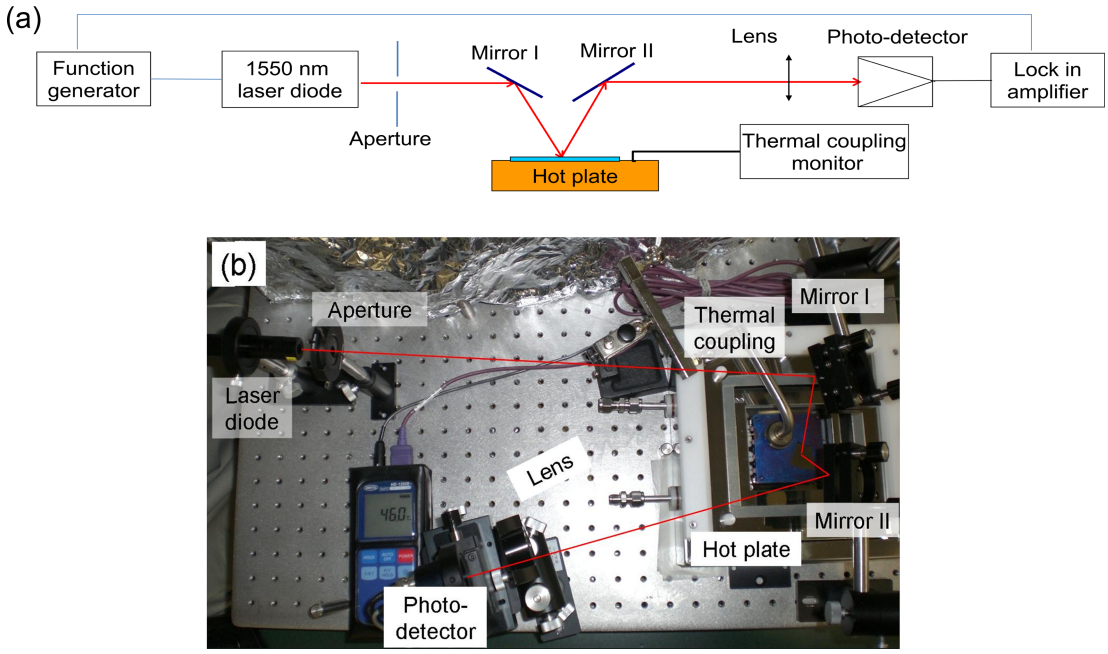

Fig. 1. (Color online) (a) Experimental configuration employed for temperature dependent infrared reflectivity measurement, where infrared wavelength is 1550 nm. (b) Photograph of the optical system.

work, hot plate with metal stage is used, and it is quite stable for low and high temperature. Aperture and lens are used to further reduce the noise. Fig. 1(b) is the photo of this system. The system has large signal/noise ratio, and its noise is lower than 0.2% from room temperature to 500°C.

The reflectivity of $(Co_{70}Fe_{30})_{92}Ta_3Zr_5$ as a function of temperature is shown in Fig. 2(a). In this figure, red dot line shows its reflectivity from its as-deposition state to temperature increasing to 500°C, and blue triangle and black diamond shows its reflectivity when sample was cooled after first time heating and then heated again.

As the figure shown, during the first heating process, when temperature was lower than 380°C, its reflectivity was almost constant; when temperature was increasing from 380°C to 400°C, its reflectivity was increasing at the same time. This up-shifting of reflectivity is about 1.5%, which is much larger than noise level 0.2%. After this shifting, reflectivity doesn't show any shifting with further heating, or cooling, or heating again. This effect is also clearly shown in *dR*/*dT* result (Fig. 2(b)), where *dR*/*dT* is around zero except for one peak at 380°C to 400°C during first heating. This suggests this up-shifting is due to an irreversible structure changing. For our metal sample, this structure changing should be amorphous-crystallization phase transition.

To confirm the phase transition, repeating annealing and X-ray diffraction were used to check its crystallization

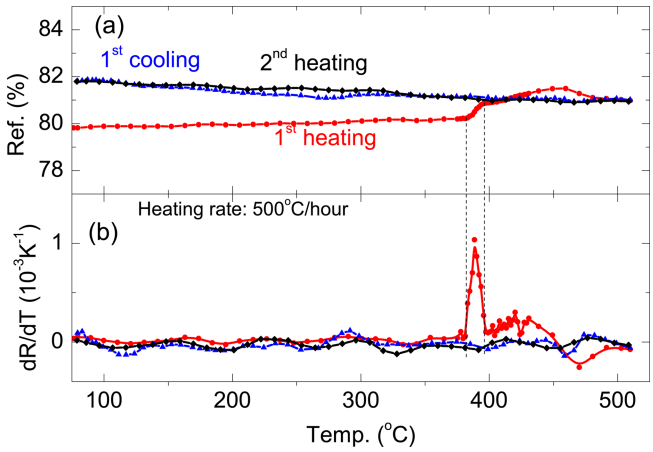

Fig. 2. (Color online) Temperature dependent infrared (1550 nm) reflectivity (a) and its derivative *dR*/*dT* (b) of $(Co_{70}Fe_{30})_{92}Ta_3Zr_5$ film. In both of (a) and (b), red circle is sample's reflectivity during first heating process, blue triangle is during first cooling process, and the black diamond is during second heating process. Temperature heating and cooling speeds were 500°C/hour.

structure. Samples were annealed and cooled by the hot plate with maximum temperature of 350°C, 450°C, 500°C. Fig. 3 shows the X-ray diffraction of those samples. Sample was in amorphous structure when sample was in as-deposition state and annealed at 350°C. However, when sample was annealed at higher temperature (450°C and 500°C), its structure was crystallized during heating.

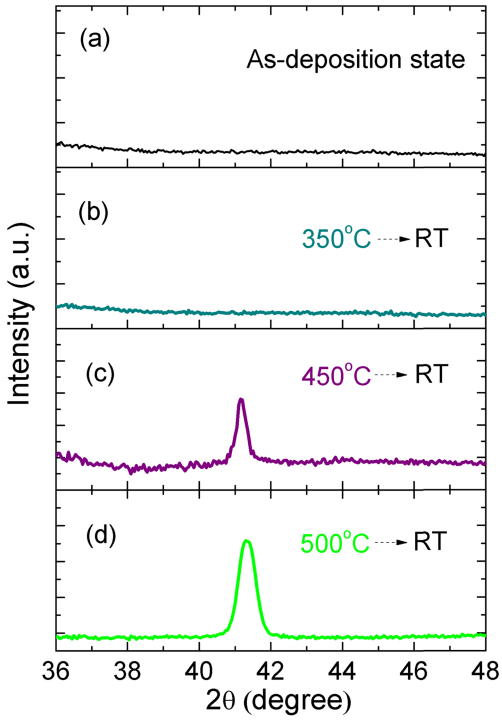

Fig. 3. (Color online) X-ray diffraction results of CrTi(2 nm)/$(Co_{70}Fe_{30})_{92}Ta_3Zr_5$ (100 nm)/Si_3N_4(10 nm) after annealing at different temperature: (a) as-deposition sates; (b) annealed at 350°C, (c) annealed at 450°C; (d) annealed at 500°C. $(Co_{70}Fe_{30})_{92}Ta_3Zr_5$ film is crystallized between 350°C and 450°C.

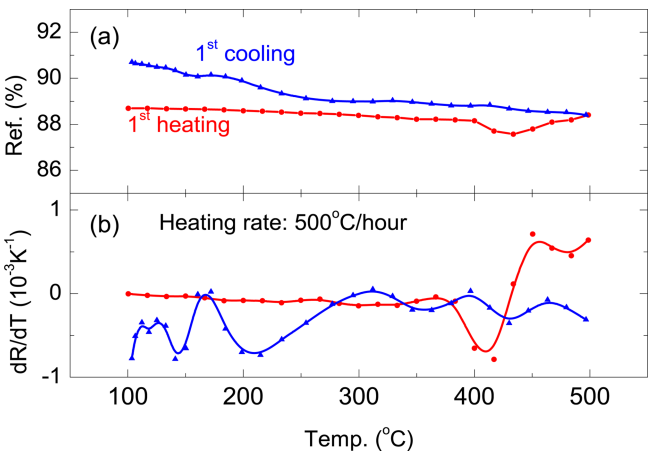

Fig. 4. (Color online) Temperature dependent reflectivity visible laser (405 nm) (a) and its derivative *dR/dT* (b) of $(Co_{70}Fe_{30})_{92}Ta_3Zr_5$ film. In both of (a) and (b), red circle is during first heating process, and blue triangle is during cooling process.

Therefore, its crystallization temperature is between 350 °C and 450°C, which is consistent with infrared reflectivity result.

Normally, optical constants highly depend on wavelength. A similar experiment was done to check temperature-dependent reflectivity at shorter wavelength. 405 nm laser was employed in same optical system shown in Fig. 1. Its reflectivity at different temperature is shown in Fig. 4(a). At sample's crystallization temperature, there is a tiny decreasing in this curve, and it is hard to indentify its crystallization temperature from this figure. Therefore, only reflectivity at longer wavelength (infrared) is effective to identify crystallization temperature.

3. Discussion

In this work, infrared reflectivity is used to check amorphous-crystallization temperature of metal. However, in phase-changing optical memory material, visible light is used to show the different proper between amorphous and crystallization structure [4]. We attribute this difference into intraband/interband absorption, which is related to low/high energy photons.

Normally, phase-changing optical memory has semiconductor atoms. In Ovshisky's theory [3], when its structure changes from amorphous state to crystallization state, the atom bonding will change, and a high density of localized states is formed. Interband absorption depends highly on density of state, so its optical property changes for high energy photons (visible light).

However, for our sample, metal is totally in different situation, whose atoms are ion and free electrons, and atom bonding shows little differences for amorphous state and crystallization. Experiments and calculation results give such evidences in some metal alloys [5, 6]. Therefore, density of state changes little, and reflectivity of 405 nm shows tiny changing at phase transition point. In this case, photon with low energy, which is related intraband transition, shows a much clearer changing for this phase transition.

4. Conclusion

In summary, an optical system is set up to measure temperature dependent reflectivity with sensitivity higher than 0.2% during room temperature to 500°C. Reflectivity of SUL $(Co_{70}Fe_{30})_{92}Ta_3Zr_5$ is measured with 1550 nm laser. Its reflectivity has a big up-shift of 1.5% when it is crystallized. The result shows this approach can be employed as a rapid and efficient method for crystallization temperature measurement, especially for SUL in next generation of heat assisted perpendicular recording media. Crystallization temperature can be achieved within one time heating during a short time. Moreover, this effect is found to be effective for infrared, but not for visible light.

Intraband transition may be the reason for this difference.

Acknowledgement

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