3C/2008G569

In-situ measurement of X-ray reflectivity of amorphous alloy thin films

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Introduction

Amorphous alloys possess various attractive properties for structural materials, such as high strength, soft magneticity and corrosion resistance. Because of those excellent properties, amorphous alloy thin films are promising for micro/nano-electro mechanical systems (MEMS/NEMS). For application of amorphous alloy thin films to MEMS/NEMS devices, it is important to measure crystallization temperature, T_x, of the films, because various properties change drastically at T_x. Measurement of resistivity upon heating is the conventional method to measure T_x when the thickness of amorphous alloy thin films is less than 1μm. However, changes in thickness and surface roughness are much more important for MEMS/NEMS application than that in resistivity. Thus we performed in-situ measurement of energy dispersive X-ray reflectivity of amorphous alloy thin films upon heating by using strong white X-ray.

Experimental procedures

In-situ measurement of energy dispersive X-ray reflectivity of a Pd-based amorphous alloy thin film on heating in a nitrogen gas flow was performed by using white X-ray and a pure Ge type solid state detector (SSD) at BL-3C. The grazing incidence angle and reflection angle were fixed at 0.2° and 0.4°, respectively. The cross sections of the incident and reflected X-ray are 50 × 120 μm^2 and 25 × 50 μm^2, respectively. X-ray reflectivity was measured for 8 s every 10 s. The film was crystallized during the in-situ X-ray reflectivity measurement, because diffraction peaks from crystalline phases are found in an X-ray diffraction pattern recorded after the reflectivity measurement.

Results and discussion

Fig. 2 shows the profiles of in-situ X-ray reflectivity measured for the film. Al attenuator whose thickness is 10 mm is used in front of the SSD, thus the intensity of the reflected X-ray is weak at lower photon energy. The thin film specimen was heated up at a heating rate of 10 – 20 K/min. The profile of oscillations of the reflectivity changes at about 25 keV, corresponding to Pd L-edge absorption. The oscillations are clearly observed even though the photon energy is high at lower temperature than about 140 °C. The thickness of the thin film is obtained by measuring the energy distance of the peak of the oscillations in Fig. 1 [1, 2]. Fig. 2 shows the thickness of the film as a function of temperature. The thickness was about 130 nm when the temperature was lower than 140 °C, while it was about 127 nm for higher temperature than 140 °C. It seems that this change in the film thickness is discrete. The decrease in the film thickness upon heating means increase in the density of the film. Therefore, it is considered that the discrete change in the film thickness was caused by crystallization and that the crystallization temperature is about 140 °C.

References

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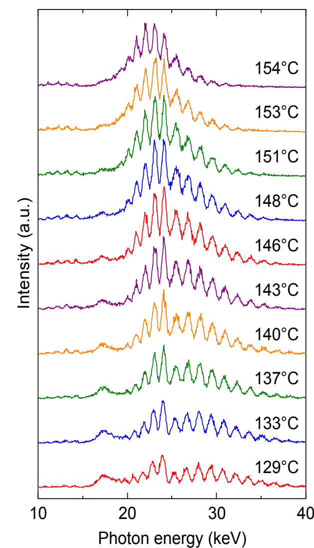

Fig. 1 Energy dispersive X-ray reflectivity of the Pd-based amorphous alloy thin film upon heating.

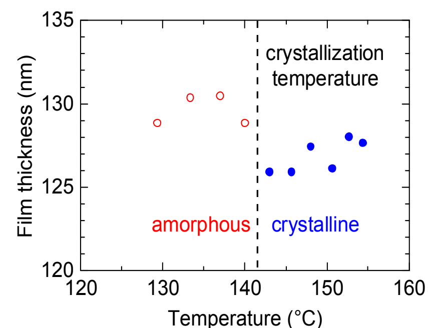

Fig. 2 The film thickness as a function of temperature.