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Stress Measurement of Thin Films Using Synchrotron Radiation

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Introduction

Stress measurements of TiN thin films have been carried out by the authors using conventional laboratory X-rays. Almost all crystals in TiN films had a {111} axis close to the films surface normal. In this case, it is usually used the two-exposure method as to stress measurement [1]. However, the measurable diffraction planes are limited to several planes because of constant wavelength by characteristic X-rays. When X-rays from synchrotron radiations are used, optional monochromatic X-rays are obtained by the double-crystal monochromator. Thus, highly precision measurements will be expected due to a high brightness beam with small divergency. In adittion, users can select favorable wavelength.

In this study, residual stresses in TiN thin films were measured by X-rays from synchrotron radiation. Results were compared with those of laboratory X-rays.

Experimental

TiN thin films were deposited with Physical Vapor Deposition (PVD) on steel substrates. A substrate had a dimension of a length of 12mm, a width of 12mm and a thickness of 5mm. Five samples deposited thin films were prepared under same PVD conditions. After deposition, one of those specimens was as-deposited, others were annealed in a furnace at temperatures of 573K, 798K, 843K and 893K respectively.

Residual stresses were measured at BL-3A in Photon Factory (PF). Measurement conditions are listed in Table 1. The wavelength was adjusted to the value corresponding to the Bragg angle 154 deg [2]. The tilt angle ψ was limited, because the Ω-diffractometer method was only applied due to limited optical system. Therefore, it is impossible to apply the two-exposure method. The stress was calculated using an unstressed lattice spacing d_0 instead of this method. A strain perpendicular to diffraction plane, ε^L_{33} were measured for each diffraction plane. And the stress was obtained from $\varepsilon^L_{33} - \sin^2\psi$ diagram.

Results and Discussion

Figure 1 shows the relationship between residual stress in thin film and annealing temperatures. As annealing temperature increased, the value of compressive residual stress remained nearly constant up to 573K, and decreased at temperature higher than 573K. The change in full-width at half maximum (FWHM) obtained from X-ray profiles corresponded to the change in residual stress by annealing. The third kind stresses, which are related to the FWHM, are defined as the deviation from the average of stresses within smaller regions than a grain. Behavior of the third kind stress can be qualitatively evaluated by change in FWHM. A decrease of FWHM results from a decrease of the third kind residual stress in the film due to annealing. The relaxation of compressive residual stress in films may be related to the relaxation of the third kind of residual stress in grains.

In an experiment at laboratory, stresses that are shown in Fig.1 were measured using Co-Kα radiation with the two-exposure method. Residual stresses were decreased by annealing. This tendency was relatively similar to that of synchrotron radiation source. However, absolute values of residual stress did not correspond to those of synchrotron radiation. Since the wavelength of Co-Kα radiation is shorter than that by synchrotron radiation, the penetration depth of X-rays to the film is larger. If the stress gradient existed in thin films, the values of stress should have a difference between both results.

References

[1] T.Hanabusa et al., J. Soc. Mat. Sci., Japan, 42-472, 90 (1993).
[2] S.Takago et al., J.Soc. Mat. Sci., Japan, 49-7, 729 (2000).

Table 1: X-ray stress measurement conditions.

Diffraction plane	TiN222	TiN420
Wavelength, nm	0.2386	0.1848

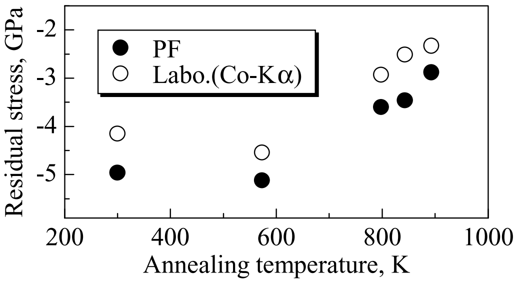

Fig. 1: Relation between residual stress and annealing temperature.

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