Influence of interfacial neighborhood on residual stress due to deposition of TiN thin films made by PVD

Influence of Interfacial Neighborhood on Residual Stress Due to Deposition of TiN Thin Films Made by PVD

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Abstract. In depositing the TiN thin films to the substrate by Physical Vapor Deposition (PVD), it influences the substrate interface. Change of the residual stress and the full-width at half maximum (FWHM) in each process of the TiN deposition of thin film was measured by the X-ray stress measurement. As a result of the X-ray stress measurement, there are no changes in the residual stress and the FWHM. It is thought that there is a difference in the penetration depth to the substrate of X-rays and Ti ion.

Introduction

Ceramic hard films such as TiN,TiCN and TiC made by PVD is used from excellent in heat resistance, wear and abrasion resistance, and the protection against corrosion, etc. as a surface reforming material such as cutting tools for the machine. The mechanism of the occurring residual stress generation is considered to generate the residual stress by the temperatures fluctuate of the deposition temperature and the room temperature because coefficient of thermal expansion of the thin films material and the substrate material is different at the deposition of thin film[1]. The compressive residual stress value reaches -5GPa , and it is difficult to explain as a mechanism of the residual stress generation by the difference of coefficient of thermal expansion of the thin films material and the substrate material. Because it is thought that the stretch residual stress equal with the compressive stress of the thin films is generated by the internal stress balance in the substrate, and the influence on the substrate by the deposition of thin film appears more strongly in interfacial neighborhood if a very large compressive residual stress has been generated in the thin films. It aimed to examine the residual stress generation and the change in the process to the deposition of

thin film, and to clarify the mechanism. Then, the film before and behind the deposition of thin film and the change in the residual stress of the substrate were measured in this research, and the influence that the thin films material exerted on the residual stress of the substrate was examined.

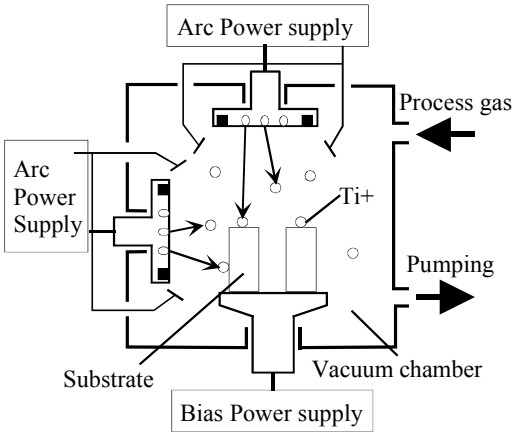


Fig.1 The block diagram of the AIP device.

Table 1 The specimen making procedure.

Buff	Ion Bombard	TiN Deposition
	Arc current 70~100A Bias voltage 1000V 3min	Arc current 70~100A Bias voltage 80V 35min
Annealing 793K 5h		

Experiment

Specimen. SS400 was used as a circuit material. The substrate material was processed to the shape of $12\times12\times5mm^3$. Afterwards, the TiN thin films is deposited to the surface of the substrate material. The TiN clad is film production with Arc Ion Plating (AIP) which is a kind of PVD device (Fujita Giken Co. Ltd. Ishikawa, Japan). Fig.1 shows the block diagram of the AIP device. The process of the deposition of thin film is three processes, and, first, does the buffing in the first process. Next, the cleaning that is called an ion bombardment that throws the Ti ion at the substrate to remove impurities on the surface of the substrate in the second process is done for three minutes under the condition of the arc current 70-100A and the electrode bias 1000V. The specimen that gave processing up to each process was made respectively with the TiN deposition of thin film as 35 minutes under the condition of the arc current 70-100A and the electrode bias 80V in the third process at the end. The specimen made three kinds as a substrate that processed it up to each process, and measured the residual stress of the substrate. Moreover, the process of the stress relief heat treatment (maintenance of 793K and 5 hours) is added after the buffing in the first process and the thin films is deposited. Three kinds of substrates that processed it up to each process as well as the above-mentioned were made as a specimen, and the residual stress of the substrate was measured. Moreover, the film thickness prepared two kinds (about 1.5μm and about 3μm). Both the thin films and the substrate residual stresses were measured about the specimen that changed the film thickness. The specimen making procedure is shown in Table 1.

Pole figure measurement. Before doing the stress measurement of the thin films, the pole figure was made and the crystal orientation was investigated. Because it was reported that these thin films have preferred orientation[2]. The pole figure was made by using the Schulz method in {111} {100}

{110} of TiN. The outline of the Schultz method is shown in Fig.2. The pole figure measurement used RINT2500 made of science electric Ltd.

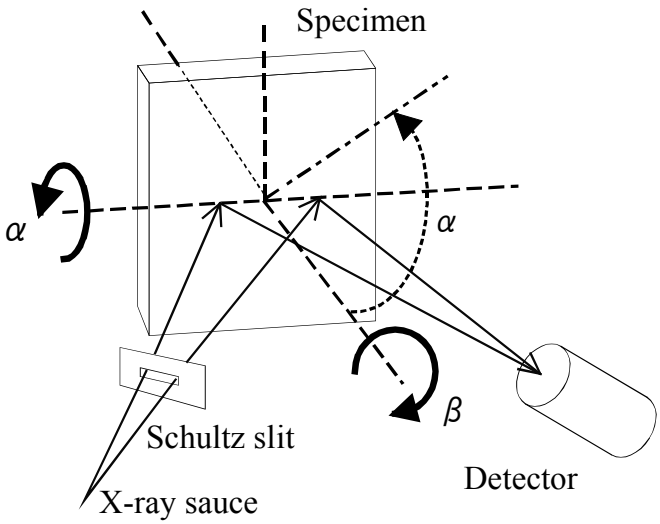


Fig. 2 The outline of the Schultz method

X-ray stress measurement. The residual stress measurement did the stress measurement of the thin films and the substrate with X-ray stress measurement device of the collimated beam type. The direction of the measurement was assumed to be a parallel direction of the specimen. The measurement condition is shown in Table 2.

Table 2 X-ray stress measurement conditions

(a) For films.

Characteristic radiation	Co-Kα
Tube voltage [kV]	40
Tube current [mA]	200
Kβ filter	Fe
Diffraction plane	TiN420
Measured angle ϕ [deg]	39,75
Scan method	Ψ-diffractometer

(b) For substrates.

Characteristic radiation	Cr-Kα
Tube voltage [kV]	30
Tube current [mA]	10
Kβ filter	V
Diffraction plane	α-Fe211
Number of ψ angles	7points
Scan method	Ω- diffractometer

The stress measurement of the substrate used MSF-3M made of RIGAKU Co. Ltd. X rays passed the TiN thin films and the residual stress of the substrate was measured. The stress measurement of the TiN thin films used RINT2500 made of RIGAKU Co. Ltd. When the TiN thin films has the aggregate structure for the {111} priority distribution for the direction of the normal of the sample side, the $\sin^2\psi$ method to which an isotropic homogeneous material is required is not applicable. In that case, residual stress σ_x is calculated by using the diffraction angle θ measured by two different ψ angles of the same diffraction plane as X-ray stress measuring method that considers the fiber aggregate structure[3].

$$\frac{\varepsilon_{33}^{L}}{\sigma_{\chi}} = S_k = \frac{2}{3}S_0{}^{C} + 2S_{12}{}^{C} + \frac{1}{2}S_{44}{}^{C}\sin^2\psi_k (k = 1,2)$$

$$S_0{}^{C} \equiv S_{11}{}^{C} - S_{12}{}^{C} - \frac{1}{2}S_{44}{}^{C} \quad (1)$$

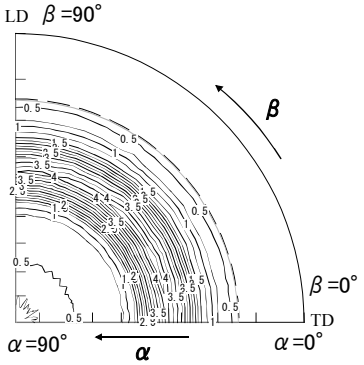
Eq. 1 is a relational expression strain and stress of X-ray. Moreover, $S_{ij}{}^{C}$ was elastic compliance of the TiN singlecrystal, and $S_{11}{}^{C}$=2.17, $S_{12}{}^{C}$=-0.38, and $S_{44}{}^{C}$=5.95 [1/TPa] were used.

$$\sigma_{\chi} = -\frac{\sin\theta_2 - \sin\theta_1}{S_2\sin\theta_2 - S_1\sin\theta_1} \quad (2)$$

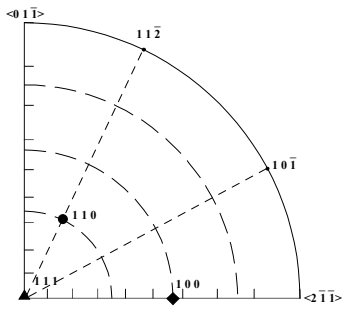
Even if the diffraction angle in the distortionless is an unknown, the residual stress can be requested by Eq. 1 and Eq. 2 by measuring diffraction angle θ_1 and θ_2 in ψ_1 and ψ_2 of the same diffraction plane.

Results

Pole figure measurement of TiN thin films. The pole figure of {100} of the specimen to deposite the TiN thin film to Fig. 3(a) is shown. The fiber orientation of 360° symmetry was confirmed in β angle for the crystal distribution. Moreover, it is understood to strengthen in α angle for the {111} distribution compared with the direction of the normal on the sample side compared with the {111} stereographic projection chart shown in Fig. 3(b)



(a) {100} pole figure of the TiN as-deposited.



(b) {111}stereographic projection of single crystal.

Fig. 3 Comparison between measured pole figure and ideal stereographic projection.

Residual stress measurement. There was no change in the residual stress value of the thin films regardless of the presence of the stress relief heat treatment to the substrate, and the compressive residual stress value was -5GPa. X-ray diffraction profile in each process in substrate α-Fe{211} is shown in Fig 4. The change in the FWHM in each process is shown in Fig 5. The FWHM was changeless in each process, and the plastic deformation to the surface of the substrate by the ion bombardment and the deposition of thin film was not able to be confirmed to the surface of the substrate.

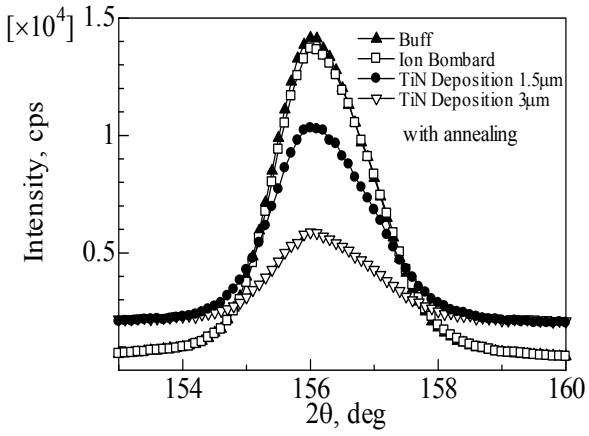


Fig. 4 X-ray diffraction obtained near interface of substrates.

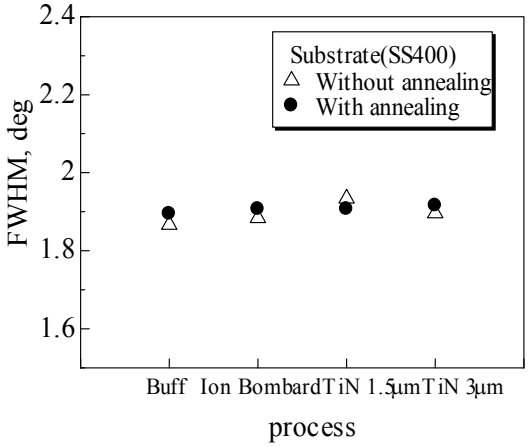


Fig. 5 The change in FWHM in each process.

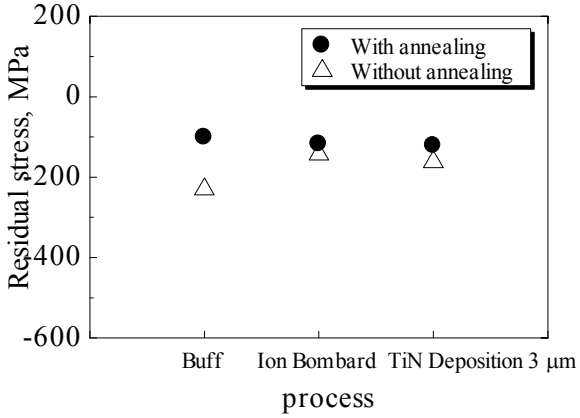


Fig. 6 The residual stress of the substrate of each process.

The residual stress of the substrate of each process is shown in Fig. 6. It was confirmed for the compressive residual stress of the substrate to process the ion bombardment from figure, and to show the tendency to settle to a certain definite value. Afterwards, the residual stress of the substrate almost showed the definite value after the deposition of thin film of the TiN, and the influence of an interfacial substrate on the stress by the deposition of thin film was not able to be confirmed. It is thought that it is a cause that the penetration depth to the substrate is different in X-rays and the ion. The interaction of the Ti ion that is the charged particle with the circuit material (Fe atom) is larger than that of the electromagnetic wave (X-rays). The penetration depth is 10μm or less when assuming that X-ray energy used by the residual stress measurement is 5.4KeV. It is thought that the penetration depth is a level of nm because the energy of the Ti ion under the acceleration voltage of the electrode bias 1000V at the ion bombardment is about 1KeV. Therefore, it is thought that the change doesn't appear in the residual stress value and the FWHM because the influence on the substrate by the deposition of thin film is below the range of detection of X-ray stress measurement because it is very small.

Conclusions

(1) TiN thin films had the preferred orientation of {111}, and the residual stress value was a compressive residual stress of about -5GPa regardless of the presence of the annealing to the substrate. (2) As a result of the X-ray stress measurement, the residual stress change and the plastic deformation of the substrate due to the ion bombardment and the deposition of thin film were not able to be confirmed. (3) It is thought that the stress change of the substrate interface cannot be measured by the X-ray stress measurement because the penetration depth of the Ti ion to the substrate by the deposition of thin film is very shallow when penetration depths to the substrate are compared with X-rays and Ti ion.

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