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Residual stress in TiN thin films studied by the grazing incidence X-ray diffraction method

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INTRODUCTION

The deposition of thin films on substrates is usually inherently associated with the development of residual stress. In general, Physical Vapor Deposition (PVD) processes result in compressive stress. Factors such as the relative dimensions of film and substrate, generally lead to a tendency of the stresses to be higher in modulus in the former. The level of film residual stresses depends on processing parameters [1], [2], [3] such as substrate temperature, substrate bias, operating pressure, coating thickness and deposition rate. Therefore, one could think that the variation of the parameters during deposition could produce residual stress gradients in the films. Thus, the mean of this work was to study the residual stress, through the film thickness, generated by either increasing or decreasing the substrate bias voltage during deposition of TiN films. The residual stress values were determined with the grazing x-ray diffraction method, and different angles were used in order to analyze stress gradients.

EXPERIMENT

Six TiN films were deposited on ferritic steel substrates (AISI D2) using Triode Magnetron Sputtering, in an atmosphere of N_2 (4 sccm) and Ar (20 sccm) at a temperature of 300°C. The films differ from each other by the bias voltage applied to the substrate. Three films (C1, C2 and C3) were deposited with constant bias voltage of -40, -100 and -150 V respectively. The film C0 was deposited without any bias voltage. To the other films the bias was varied during deposition. For film V1, the substrate bias voltage was increased from -20 to -200 V in different steps with interval times of 45 min for step. For film V2, the bias was decreased from -200 to -20 V in different steps of 45 min. Details of the deposition of films V1 and V2 are shown in Table I. The total film thickness of samples V1 and V2 is 2.1 μm, it was measured with a scanning electronic microscopy (SEM).

The residual stress was calculated by x-ray diffraction, the grazing x-ray diffraction method was used in order to limit the penetration depth of the beam. In this method the incident beam is kept constant at a low and constant incidence angle (α), the position of the detector varies along the goniometer circle registering data at several (hkl) reflections. Considering a biaxial stress state in the film, for TiN, which is a cubic material, the following equation can be used to calculate the residual stress [4], [5]:

TABLE I: Deposition conditions for films V1 and V2

Sample	Layer	Time (min)	Bias (V)
V1	1	45	-20
	2	45	-40
	3	45	-100
	4	45	-150
	5	45	-200
V2	1	45	-200
	2	45	-150
	3	45	-100
	4	45	-40
	5	45	-20

$$a = a_0(f(\psi)+1), \tag{1}$$

with:

$$f(\psi) = \frac{1}{2}S_2^{hkl}\sin^2(\psi) + 2S_1^{hkl}, \tag{2}$$

where a is the measured lattice parameter for each reflection (hkl) that can be calculated from the Bragg law, a_0 is the stress free lattice parameter, and σ is the stress value. For a 2θ position (θ is the Bragg angle), the angle ψ is formed by the normal to the sample surface and the normal to the diffraction plane, and it can be calculated from the relashionship: $\psi = \theta - \alpha$. For samples V1 and V2, in order to obtain information of residual stress at several penetration depths, the incident beam was fixed at different angles: 1.5°, 2.5°, 3.5°, 4.5°, resulting in average penetration depths of 0.3, 0.5, 0.7 and 0.9 μm [6]. The energy of the x-rays was 8.04 keV. To reducing the divergence, slits were employed in the primary beam (0.5 mm) and in the diffracted beam (1.0 mm) as well as graphite analyzer. The diffraction planes scanned for these angles were: $\langle 111\rangle$, $\langle 200\rangle$, $\langle 220\rangle$, $\langle 311\rangle$, $\langle 222\rangle$, $\langle 331\rangle$ and $\langle 420\rangle$. But the plane $\langle 200\rangle$ was not used in calculations because the diffraction peak is very close to a peak of the substrate, in which case the error increases when calculating a. For the films deposited with constant bias voltage, the residual stress was measured only at incidence angle of 1.5° with the diffraction conditions described previously. It must be noted that at a given angle of incidence, the residual stress value is an averaged measurement of the entire volume of the layer which contributes to scattering.

TABLE II: Residual stress in thin films deposited with constant bias voltage

Sample	Bias (V)	Residual Stress (GPa)
C0	0	2.1 ± 0.51
C1	-40	-5.6 ± 0.7
C2	-100	-7.9 ± 1.1
C3	-150	-12.2 ± 0.7

RESULTS AND DISCUSSION

The results of residual stress, for films deposited with constant bias voltage, are shown in Table II. As expected, the compressive residual stress level increased as the bias voltage increased. This behavior is well-known in thin films deposited by sputtering and have been previously reported in several works [7], [8], [9], [10], it is due to increment of the energy of ions arriving at the surface of the substrate.

In Figure 1, the results of residual stress of samples V1 and V2 are shown as a function of the angle of incidence. In film V2, deposited with decreasing bias voltage, can be observed the influence of the bias voltage on generation of residual stress gradients. Smaller compressive residual stress level was observed near the surface, what was expected because this region of the film was deposited at low value of bias. The compressive stress level increases with increasing of the penetration depth, therefore, with increasing of the bias. For film V1, in which the bias voltage was increased during deposition, a reduction of the residual stress level was expected at higher angles of incidence. However, not differences were found in residual stress level with increasing of the penetration depth. Greater dispersion of data was observed in film V1, this could be caused by the difference of preferred orientation between the layers, that produce differences in the relative intensity of the peaks, hence, the contribution of each peak in the value of the parameter *a* depends not only on the penetration depth as well as on the preferred orientation.

CONCLUSION

The variation of the substrate bias during deposition run produced gradients in compressive residual stress when bias was decreased during deposition run, lower compressive residual stress were found at the surface, and the average compressive stress increased with depth. In film deposited with increasing bias voltage, the measures did not show gradients of compressive residual stress, it can be due to the effect of the differences of preferred orientation between the layers of the film.

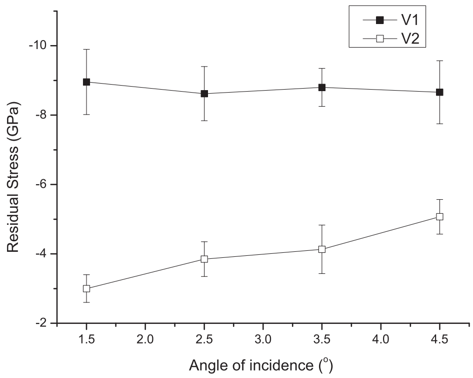

FIG. 1: Residual stress gradients in TiN deposited with increasing and decreasing voltage bias during deposition

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