Evaluation Of The Damage In The Stainless Steel Coatings By Residual Stress Measurement

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Thin solids films of 304L stainless steel are prepared by the ion beam sputtering technique. The obtained films present a debonding phenomenon and a high compressive stress state. The critical stress value causing the debonding phenomenon is determined using atomic force microscopy (AFM). By ion beam assisted deposition (IBAD) process during the films elaboration, we observe a stress relaxation. This effect is measured using the X-ray diffraction method $\sin^2\Psi$

Keywords: Stainless steel films; Residual stresses; X-ray diffraction; Atomic force microscopy

I - INTRODUCTION:

Sputter-deposited films are largely studied. One particular characteristic of these layers is the development of high residual compressive stresses during the deposition process [1,2].

Due to the presence of such large residual compressive stresses, the films debond from the substrates, and present interesting topographical patterns [3].

Concerning the sputter-deposited 304 stainless steel (SS) films, their structural, mechanical properties and adhesion behaviour, have been already studied using X- ray diffraction, transmission electron microscopy (TEM), scanning electron microscopy (SEM) and X-ray diffraction $\sin^2\psi$ method [3,4,5]. In the other hand, the wear and corrosion behaviours of 304 SS films were examined. In comparison with a bulk 304 L SS, an improvement of the wear and corrosion resistance is observed [6,7].

This work is devoted to complete the study concerning the 304 L SS coating layers produced by ion beam sputtering technique.

The first part of this paper is concerned with the buckling phenomenon observed in 304 L SS films. The study is performed using the SEM and atomic force microscopy (AFM) techniques. The observed wrinkles are examined, and critical stress producing debonding phenomenon is obtained using calculations based on the phenomenological model developed by J. W.Hutchinson [8].

The second part is devoted to study the ion beam assisted deposition (IBAD) effect on the structural and mechanical properties of 304 L SS prepared using the ion beam sputtering technique. During the deposition process, the ion beam sputtering technique can be associated to assistance by ions of rare gas during a film growth. This can allow modifying the stress state in the film, often responsible to the debonding phenomenon. In our study, tree kinds of ion irradiation with 25, 50 and 75µA in focalised currents respectively were performed. We apply X-ray diffraction (XRD) technique to determine the residual stresses ? and stress- free lattice parameter $a_{0.}$

II - EXPERIMENTAL PROCEDURE

1 - Films preparation

Thin 304 L SS films were deposited at room temperature using an Ar ion beam sputtering technique. The sputtering system was already described in details in reference [9]; let us recall here, the deposition conditions and parameters:

- The pressure witch start at 2 x 10^{-6} Torr, was maintained around 5 x 10^{-4} Torr during the deposition.
- The deposition rate was equal to 0.05 nm s-1.
- The films thicknesses were successively in range of 60 to 600 nm. The thickness is chosen as a function of the technical analysis method used and/or the studied effect.
- The deposition rate and films thicknesses were controlled by a quartz oscillator.
- The used substrates were successively, silicon wafers and polycarbonate in parallelepiped form.
- The sample-holder temperature, was limited (less than 350K) during the deposition process.

2 - Buckling phenomenon observations

The spontaneous debonded regions of the films were observed using a Jeol scanning electron microscope, and a specific Atomic Force Microscope coupled to a compression machine has been used to follow in situ the emergence of buckling and debonded regions of the films under axial stress. This apparatus has been described in reference [10]. The wrinkles dimensions were measured using a DEKTAK profilometer.

3 - Stress determination

X-ray experiments for residual stresses were performed on a beam line D22 of the synchrotron radiation LURE at Orsay using an original diffractometer developed in LMP (Laboratoire de Métallurgie Physique) de Poitiers, France [11] and dedicated to nanocrystalline material studies, with a wavelength of 0.2137 nm and ? configuration [12]. Stress determination was performed using the well-known $\sin^2\psi$ method. This method is based on the measurement of the shift of diffraction peak position recorded for different Euler angles [13].

In this study, we have followed the evolution of the (211) peak position of the bcc structure as function of the angle ψ Experimental data have been analysed when considering an in-plane isotropic stress state ($?_{11}$ = $?_{22}$ = $?_{11}$ and $?_{33}$= 0) and using the rational definition of strains [14].

$$\ln\left(\frac{1}{\sin\theta_{hkl}}\right) = \frac{1}{2} S_2 \sigma \sin^2\Psi + 2 S_1 \sigma + \ln\left(\frac{1}{\sin\theta_0}\right) \quad (1)$$

where, $?_{hkl}$ stand for diffraction angle of (hkl) plane, $?_0$ is the stress-free diffraction angle and between the surface normal and diffracting plane normal. The X-ray elastic constants (XECs) values used are the one of ?-Fe based materiel calculated by the self-consistent method of Kroner-Eshelby [15]: 1/2 S_2 = 5,71x10^{-3} GPa^{-1} et S_1 = -1,12x10^{-3} . For cubic materials, this method always gives results, which are in good agreement with experimental values [16]. When plotting Ln (1/sin?) as a function of $\sin^2\psi$ one get a straight line (eq.1), its slope is related to the in-plane stress ?, and it's origin ordinate to the stress-free lattice parameter a_0 [17,18].

III - EXPERIMENTAL RESULTS AND DISCUSSION

a- The buckling phenomenon study

1 - AFM observations under applied stress

As mentioned in previous work [19], using an AFM apparatus, coupled to a compression machine, a buckling regions were observed in the 304 SS films deposited on polycarbonate substrate. The film studied here, has a thickness of 60 nm, and no spontaneous wrinkle is observed before the compression test. This can be observed in figure 1, where the initial state of the film is presented.

A scheme representing the compressive test is given in the figure 2.

After uniaxial compressive stress, the film presents a debonding phenomenon. The obtained final state of the film after a whole cycle of compression is presented in the figure 3. The buckles obtained here propagate in sinusoidal form. This propagation growth in the perpendicular direction to the uniaxial applied stress. The apparition of this phenomenon happened when the applied stress equal a critical value of 0.33 GPa.

2 - Spontaneous debonding phenomenon observations

When the film thickness exceed a value about 140 nm, a spontaneous buckling phenomenon is observed. Different and various forms and shapes of debonded regions are observed.

In figure 4, we observe, the first state of the spontaneous debonding, the shape of the blisters presents various forms.

The stress effects can be observed in the figure 5. Under the internal stresses, the blister shape evolution can be followed. The debonding phenomenon indicates the compressive nature of the stress. The obtained blister is circular and stays in this form. There is no propagation of the debonded part of the film, but the blister submitted to the internal stress growth, and the final result of this evolution is the apparition of cracking into the blister (two holes into the observed blister presented in figure 5). This may indicate that the internal compressive stress has many directions.

The other forms of spontaneous buckling phenomenon may observed. In the figure 6, the debonded region presents a sinusoidal buckling. The apparition of the same form of buckling in one direction, indicate a possible existence of uniaxial compressive internal stress.

As mentioned in previous works [3,19], this phenomenon is related to the internal stresses depending on the film thickness.

Let us now determine the critical stress value witch gave rise to the spontaneous debonding phenomenon. Using the J.W. Hutchinson model [8], the critical stress value can be obtained from the relationship: σ_c = 1,22 [E / (1 - ν)](h / R $)^2$ where:

E : is the Young modulus of the film, ν: is the Poisson's ratio of the film,

h : is the film thickness, R : is the radius of the deflexion of the film as presented in the figure 7.
The parameter's values are successively: E = 145 GPa [20] and ν= 0,3
If we consider the radius average value, witch equal 12.5 nm, using the relationship given above, we obtain the critical value σ_c= 0.31 GPa. This value can be compared well with the value obtained under applied stress (0.33 GPa). This is the stress witch allows the initiation of the delamination in the film. However to obtain internal residual stress σ in the film, the X-ray diffraction technique is until now, the better used method. The results may be found in the following part.

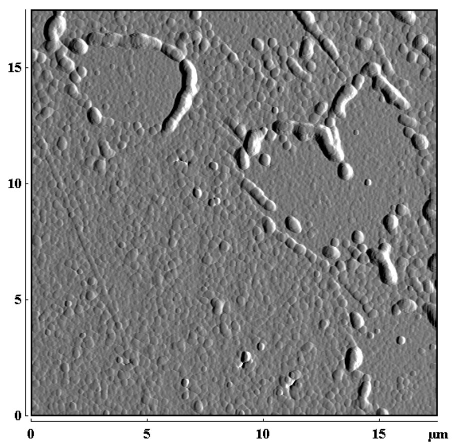

Figure 1: The initial state of the film with out any wrinkles

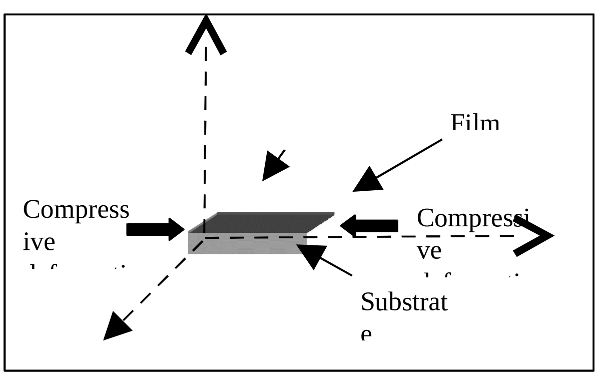

Figure 2**:** Scheme representing the compressive test

Figure 3: Final state of the film showing the buckling phenomenon in one direction

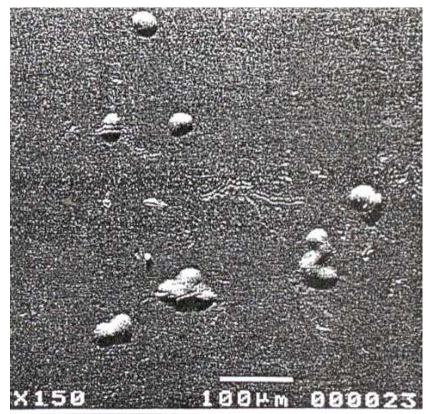

Figure 4**:** Spontaneous debonded regions in different forms

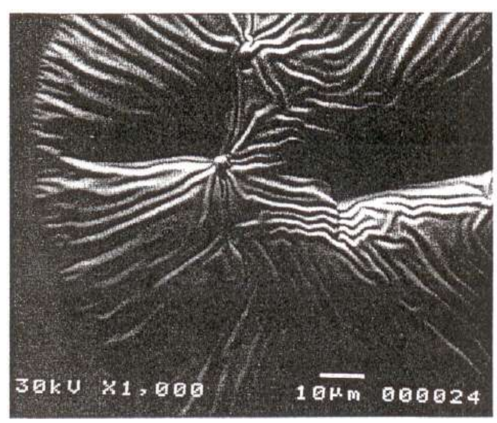

Figure 5: The evolution of the blister: apparition of cracking in the blister

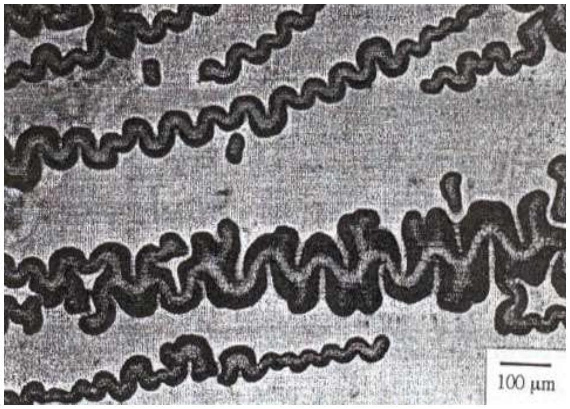

Figure 6: Spontaneous debondiong showing a propagation in sinusoidal forms

b – The stress study by the X-ray diffraction

The experimental values deduced from X-ray measurements are reported in table 1. For the as prepared films the obtained value (-3.20 GPa), shows the high compressive state of the film. The ion-assisted process strongly influences the stress magnitude. Indeed, it induced a great stresses compressive relaxation.

The origin of such a high magnitude of stress in thin films deposited on substrate can be explained by two models based on volume expansion due to energetic particle bombardment during deposition [21].

The lattice expansion effect responsible for compressive stress is clearly shown in table 1. The stress-free lattice parameters (a_0) found are greater than the ferrite bulk stainless steel given in the literature [22]. On the other hand, the stress free-lattice parameters measured on assisted films are lower than those of as prepared film ($a_0 = 0.2888$ nm). This implies an elimination defect in films crystalline domains with assistance. The same phenomenon was observed by Goudeau et al [23] in 304 L SS films deposited on a silicon substrate by ion beam sputtering technique and assisted during deposition with an Ar+ energy of 160 keV.

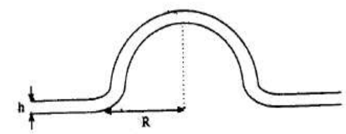

Figure 7: Schematic view of circular blister

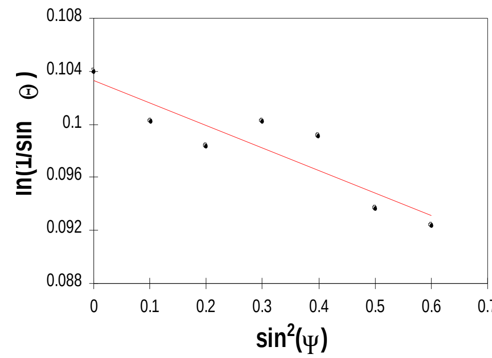

Figure 8: Evolution of the Ln $1/\sin\theta$ as a function of $\sin^2\psi$ (The stress value is determined from the sloop of the straight line)

IV. CONCLUSION:

First, in this work, we have presented the debonding phenomenon, which happened in the 304 L SS films elaborated by ion beam sputtering technique. The resulting buckling exhibit blisters and sinusoidal forms. This is attributed to the existence of a high compressive internal stresses in the films. Taking advantage to its high resolution, we have used the AFM to analyse the first stage of the debonding phenomenon, and the obtained results were compared quite to the Hutchinson's model calculations used in the case of the spontaneous debonding phenomenon.

We have also, remark that the stress direction can be uniaxial or multiaxial given arise to different forms of buckling. The origin of this behaviour will be explored in the further study.

In the second, we have shown that the residual stresses (responsible of the buckling patterns) are considerably reduced when using IBAD processes. This is allows to consider the possibility to obtain a stress-free state of the 304 L SS films (films with out debonding), by using an IBAD processes.

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Tables:

Samples	Film Thickness (nm)	Focalised current (µm)	Assistance rate (%)	? ± ? ? (GPa)	a ± ? a (nm)
As-prepared	235	00	0.00	- 3.20 ± 0.29	0.2888 ± 0.0003
A25	235	25	0.18	-0.70 ± 0.07	0.2880 ± 0.0001
A50	280	50	0.37	0.66 ± 0.11	0.2882 ± 0.0001
A75	300	75	0.55	-2.75 ± 0.18	0.2883 ± 0.0002

Table 1: Evolution of residual stress and lattice free parameter as a function of assistance rate