Internal friction and its thermal evolution on 304 L stainless steel films

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Internal friction has been measured between 300 and 760K on 304 L stainless steel (SS) using a vibrating reed device. The 0.6 µm thick samples were deposited with ion beam sputtering technique on (100) silicon substrate. It has been shown that the damping level is considerably reduced by annealing between 500 and 760K. The calculated activation energy and the reaction order, using the Johnson-Mehl-Avrami (J-M-H) kinetic enable us to assume that the observed mechanism is closely related to microstructural rearrangements located at grain boundaries.

Keywords: 304 L Stainless Steel, Ion Beam Sputtering, Internal Friction.

I. INTRODUCTION

Internal friction as method of detection of microscopic motion on metals has been widely used. In the past few years, the investigations on thin metallic films have proved to bring new insight on these transport mechanisms [1,2]. More recently, internal friction has been determined on ultrathin copper films deposited on silicon substrate[3].The authors had used the membrane technique [4] that enables to determine concurrently the internal stress and internal damping on layer as thin as 1.8 nm.

The aim of the present work is the analysis of the internal friction modification during thermal treatments measured on 304 L stainless steel (SS) with 0.6 µm of thickness. The films are deposited on (100) silicon substrata using the ion beam sputtering technique. The measurements of internal friction thermal evolution have been performed using a vibrating reed device.

II. EXPERIMENTAL PROCEDURE

Thin film of bcc 304L (SS) were deposited using a sputtering system equipped with ion-beam source operating in a vacuum chamber. The starting pressure was 5 10^{-5} Pa and during deposition the pressure was maintained at about 5 10^{-3} Pa. The apparatus it self as well as the conditions of deposition are already described in previous works [5,6]. An ion current of 70 mA for beam diameter of 7.5 cm permitted a deposition rate of about 0.05 nm s^{-1} with an Ar^{+} ion energy of 1.2 KeV. The film is deposited on monocrystalline silicon (100) covered with native oxide. It prevents any epitaxial relationship between the substratum and the film. The film with a surface of 5 x 17 mm^2 and total thickness (as verified with a Decktak IIa [7]) 0.6 µm remained attached to the substratum.

Internal friction measurements were performed using a vibrating reed device specially adapted to thin metallic films. The experimental set up has been described elsewhere [8]. Shortly, the sample is vertically fixed between two copper plates, in which is encrusted a heating system. This system enables coverage of a broad temperature range, 300-760K. Heating and cooling rates are generally 2 Kmn^{-1}. The free end of the sample is electrostatically exited at its resonant vibration and the detection is ensured by an electrode placed in front of the free end of the sample. The variation of the distance between the strip and the electrode induces a misfit of the electronic line and enables the measurement of the amplitude vibrations. The damping was determined from the free decays of vibration amplitude. All measurements were performed at 2 10^{-6} Torr pressure. The measured internal friction Q^{-1} is then a combination of that of the film $Q^{-1}{}_f$ and the substratum one $Q^{-1}{}_s$, according to the following formula [1,9].

$$Q^{-1} = Q^{-1}{}_s + 3\frac{e_f}{e_s}\frac{E_f}{E_s}Q^{-1}{}_f \qquad (1)$$

where:

E_f, E_s are respectively the Young modulus of the film and the substrate.
e_f, e_s, are respectively the thickness of the film and the substrate.

To avoid clamping damping, the substrata have been specially shaped as shown in figure1. Strips of silicon single crystal (600µm) were thinned to approximately 90 µm by chemical etching. A 5 mm large boss is preserved to its initial thickness and is used to clamp the sample.

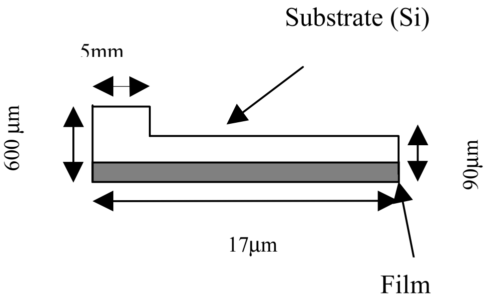

FIG.1: Scheme of the specially shaped sample

III. RESULTS AND DISCUSSION

The results of internal friction measured on 304 L (SS) film adherent to 90 µm silicon substratum are presented in figure 2. The annealing temperature has been successively fixed to 400, 500, 600, 700 and 760K. The continuous line reports the damping measured on the uncoated silicon strip. At low temperature (T< 400K), we can not observe any great difference in internal friction between coated system and substrate.

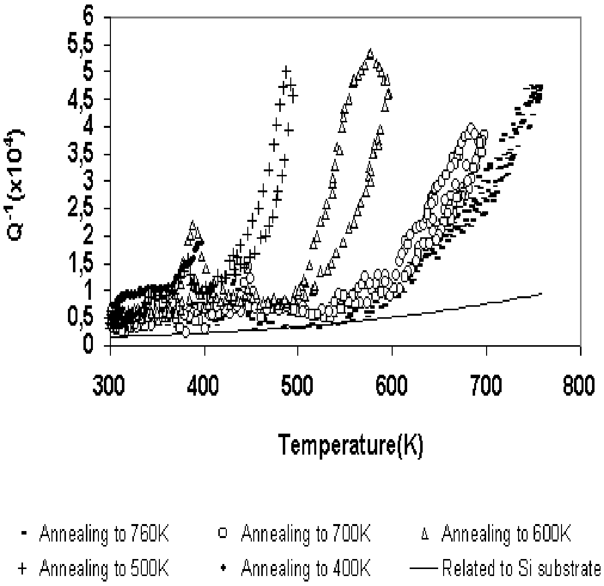

FIG.2 : Internal friction versus temperature measured on 304 L (SS) films during annealing cycles between 300 and 760K.

A maximum of damping appears firstly at 380-460K-temperature range. This peak appears to be stable in temperature since it occurs during the 500, 600, 700, 760K annealing cycles respectively. This implies that the phenomenon relaxation starts at this temperature range.

A second peak appears at the end of 500K cycle annealing and shifted to higher temperature at following cycles annealing (600,700 and 760K). The major change is observed between 450 and 760K temperature. Internal friction values measured at 480K for 500 and 600K cycles annealing decreases from 5.09 10^{-4} to 0.81 10^{-4} between the two successive annealing. This may certainly be attributed to interatomic rearrangements in the metallic layer. The same phenomenon was observed recently by Pelosin [10] in Nickel films deposited on silicon substrate by magnetron-sputtering. The determination of activation enthalpy by using the change in slope method [10], shows that the diffusional mechanism is located at grain boundaries. Indeed, in some cases this mechanism is preponderant at low temperature $T < ½ T_f$ (T_f is the melting temperature of the material) [15]**.** Previous works on 304 L (SS) films [11,19] have shown that a phase transformation α (bcc) $\rightarrow \gamma$ (fcc) happened at range of temperature between 823 and 930K. Using the Johnson Mehl Avrami (J-M-A) kinetic model [12], Boubeker et al. [11] have calculated the activation energy and the reaction order of the α to γ phase transformation. The activation energy found was included in the 251 - 418 kJ mol^{-1} range determined from references [13,14,16,17,18]. This value may be attributed to the intergranular diffusion since the reaction order *n* determined in reference [11], was ascribed to an interface-controlled dominated by boundary nucleation. Moreover, X-ray diffraction and mechanical spectroscopy experiments were carried out recently [20] ; the obtained results allow us to confirm this characterisation.

IV. CONCLUSION

The internal friction measurements have revealed structural evolution of 304 L (SS) films between 500 and 760K. The correlation between internal friction measurements and activation energy determined using the J-M-A kinetic shows that the underlying process is located at grain boundaries. It reveals a topological short range ordering.

[1] B.S Berry and W. C Pritchet, Thin Solid films **33**,19 (1976).
[2] H.G. Bohn and C.M. Su, in *Internal Friction and ultrasonic attenuation in solids*, edit by L.B. Magalas (Trans Tech, Zurich, 1993) P274.
[3] C.M. Su and M.Wutting, App. Phys.lett.**63**, 3437 (1993).
[4] B.S. Misubayashi, Y. Yoshihara and S. Okuda, Phys. Status Solidi A **129**, 475 (1992).
[5] J.P. Eymery, G. Laplanche, M. Cahoreau and M.F. Denanot, Thin Solid films **217** (1992) 1.
[6] M. Jaulin , G. Laplanche, J. Delafond and S. Pimbert-Michaux, Surf. Coat. Technol., **37** (1989) 225.
[7] A. Serrari, Thèse de troisième cycle, Université Hassan II-Mohammedia (1997).
[8] V. Pelosin and J. Hillairet, NanoStructured Mater. **4**, 229 (1992).

[9] Damask and Dienes, in *Defects in Metals* (Gorland and Breach Sciences, New York, 1963),P.145.
[10] V. Pelosin, Thèse d'Université de Grenoble (1993).
[11] B. Boubeker, J.P. Eymery, P. Auric, M. Rahmoune, Nuclear Instruments and Methods in Physics Research B **101** (1995) 267-274.
[12] W.A. Jonhson , R.F. Mehel, Trans. AIME **135** (1939) 416.
[13] T. Thorvaldsson and A. Salwen, Scripta Metall. **18** (1984) 739.
[14] CF Jenkins and G.V Smith, Trans. AIME **245** (1969) 2149.
[15] Dieter Landolt, *Traité des matériaux 12 (corrosion et chimie de surfaces des métaux)*, première édition (1993) presses polytechniques et universitaires romandes CH-1015 Lausanne.
[16] V. Linnenbom, T. Tetenbaum and C. Cheek, J. Appl. Phys. **26** (1955) 932.
[17] V. Ganesan, V. Seetharaman and V.S. Raghunathan, J. Nucl. Mater. **118** (1983) 313.
[18] H.S. Daruvala and K.R. Bude, Mater. Sci.Eng. **41** (1979) 293.
[19] B. Boubeker, Thèse d'état, Université Hassan II-Mohammedia (1994).
[20] M. Idiri, Ph. Goudeau, V. Pelosin, B. Boubeker, A. Serrari, In preparation