Effect of Xe^+ ion bombardment induced patterns in stainless steel on plasma nitriding processes.

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Abstract

The texturing of surfaces is an important method applied in various technological areas of research. Much of the current interest on surface modifications stems from the possibility of obtaining special optical, magnetic, tribological, and mechanical properties in a variety of materials. The texturization of steel surface by ionic bombardment (atomic attrition) is an interesting route to modify plasma nitriding process. The observed changes in plasma nitriding are attributed to several causes such as increasing surface nitrogen retention, stress, and creation of lattice defects, altogether improving nitrogen diffusion.[1] In this paper we report the effect of Xe^+ ion bombardment on the surface of the steel (SS 316L and AISI 4140). The bombardment effect on plasma pulsed nitriding process is also reported.

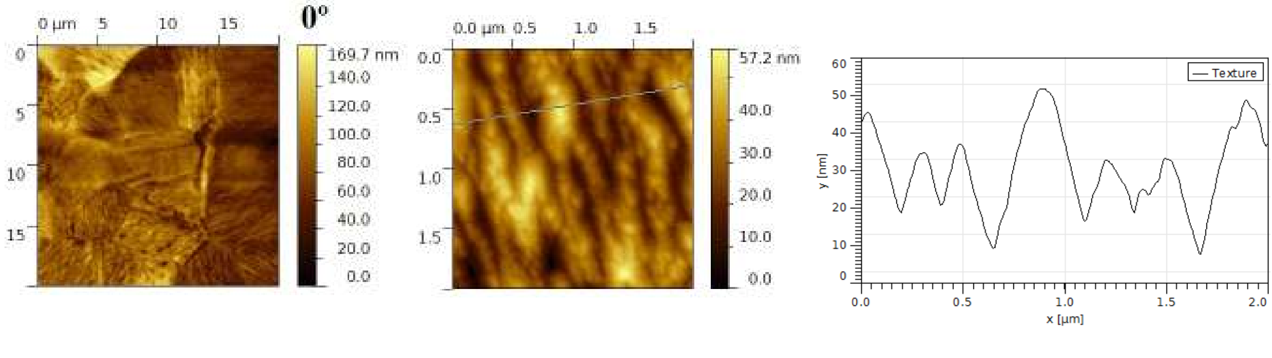
Experimental

Rectangular samples of 20x10 mm and 2 mm thick, from the same source of SS 316L (C: <0.08, Si: <0.5 P: 0.05, S: 0.03, Mn: 1.6, Mo: 2.1, Ni: 12.0, Cr: 17.0, Fe: balanced in wt. %) were study. The samples were mirror polished using standard metallurgical techniques. The Xe^+ ion bombardment was performed at room temperature, during 30 minutes using a Kaufman source 3 cm diameter beam. Details of the apparatus are described elsewhere.[2] The ion beam energy and current density were fixed at 1 keV and 0.37mA/cm^2, respectively. According with TRIM ion bombarding simulations, the stopping distance of the ions is between ~11 to 18 Å.[1,3] The background chamber pressure is ~10^{-7} mbar, and the working pressure during Xe^+ bombardment (1.40±0,02)x10^{-3} mbar. Five impinging ion bombardment angles (θ = 0º, 15º, 30º, 45º and 60º) were selected for the study. Samples of AISI 4140 steel (C: 0.4, Si:0.25, P:0.04, S:0.04, Mn: 0,85, Mo: 0.20, Cr: 1, Fe: balanced in w%) were also studied (20x10 mm, 3 mm thick, mirror polished). These samples were partially covered with a silicon wafer during the pre-bombardment treatment, i.e., the screened part of the sample was not bombarded and the other part suffered the texturization. Afterwards, these samples were 20 hs nitrided in a commercial pulsed plasma vacuum furnace (Plasma-LIITS, Campinas São Paulo, Brazil) at 380ºC, ~1.2 Torr chamber pressure, gaseous mixture of nitrogen and hydrogen (20% = $[N_2]/[H_2+N_2]$). Therefore, the two regions (i.e., the pre- and not bombarded parts of the sample) few millimeters apart were analyzed to study the effect on nitriding produced by the initial state of the surface.

Results and discussion

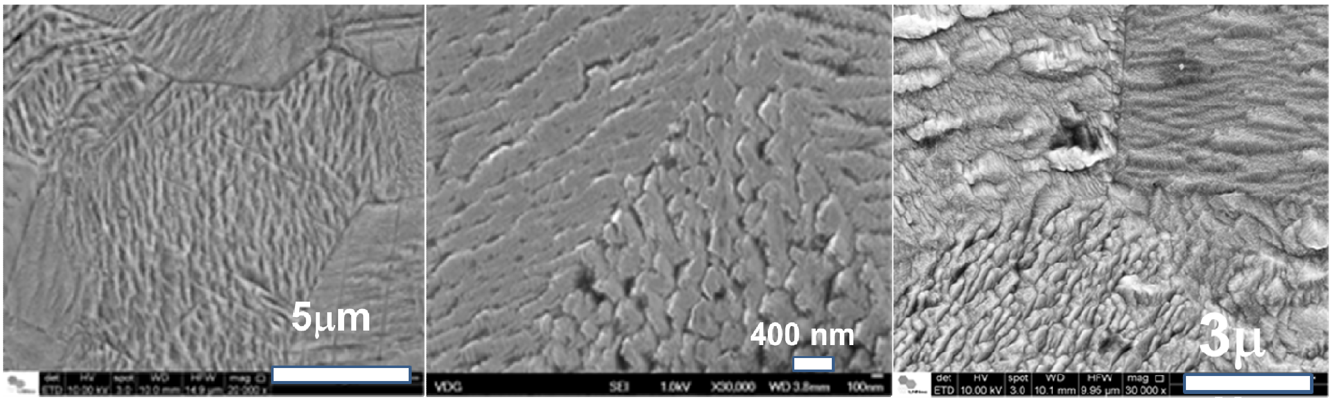
II.1. Bombarding effect on the material surface

As reported for other authors, the analysis of images obtained by scanning electron microscopy (SEM-FEG) and atomic force microscopy (AFM) show that the noble gases ion bombardment evidenced the crystalline grains and stimulated the formation of quite regular patterns. In metals, these patterns depend on the crystalline orientation of the grains rather that the direction of the ion beams (Figures 1, 2). This behavior is explained by the roughening instability model due to Ehrlich-Schwoebel diffusion barriers in metal.[4] Roughly speaking, the regular pattern stems from essentially two mechanisms inducing surface instability. The first one is related to the surface curvature dependence of the ion sputtering and the second one is due to the presence of an energy barrier to diffusing adatoms to descend step edges.



(a) (b) (c)

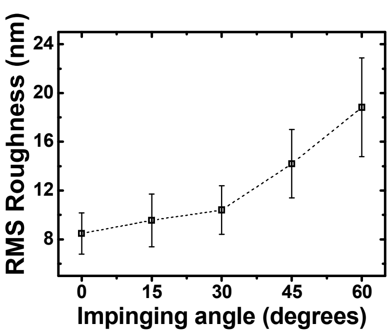
Figure 1: AFM pictures from SS316 of the perpendicularly bombarded sample (1 keV). (a) an overview of several grains with different patterns orientations; (b) grain pattern detail; (c) profile along the line indicated in (b).



(a) (b) (c)

Figure 2: SEM-FEG images from SS316 a different ion beam impinging angles (1 keV)**:** (a) perpendicularly; (b) 30º; (c) 60º.

Figure 2 shows the micrographs corresponding to bombarded samples at different magnifications and incident bombarding angles. For limitation of space only a selected group of pictures are displayed. The varieties of patterns and grains borders evidenced by the ionic bombarding are vividly illustrated in the pictures. Figure 3 shows the RMS roughness vs. impinging ion beam angle measured along several grains of the studied samples. The increasing slope of the curve on the incident ion beam impinging angles is related with the sputtering yield (Figure 3). In order to obtain information about



the residual stress induced by the bombardment treatment, grazing X-ray diffraction (GIXD) were performed at $\theta = 0.5º$, 1º and 5º incident angles corresponding to 0.5, 1 and 4.5 µm depth penetration of the radiation, respectively. As shown in Figure 4 left, the austenitic stainless steel SS316L studied samples show a γ(220) preferred orientation. The shifting of the γ(111) reflection on depth in the pristine sample stems from polishing procedure and is indicative of compressive stress. Also, the α (110) reflection is present only at the surface and disappears after the bombardment

Figure 3: RMS roughness obtained in the studied samples.

(Figure 4, middle). The origin of this subtle thin phase is uncertain but probably comes from the polishing procedure. The ion bombarding modify both the positions and width of the γ (111) reflection peak, indicating stress and disorder, respectively. These effects are a consequence of the energy transferred by the projectile (knock-on) and the occupancy of small spaces by the massive Xe atoms (misfitting).[1,5,6,7] Figure 4, right shows the peak position shifts and width corresponding to the (111) reflection as a function of the sample depth. As noted, the peak position is changing a little bit. On the other hand, the width of the lines is wider suggesting an increasing disorder up to ~ 5 μm depth.

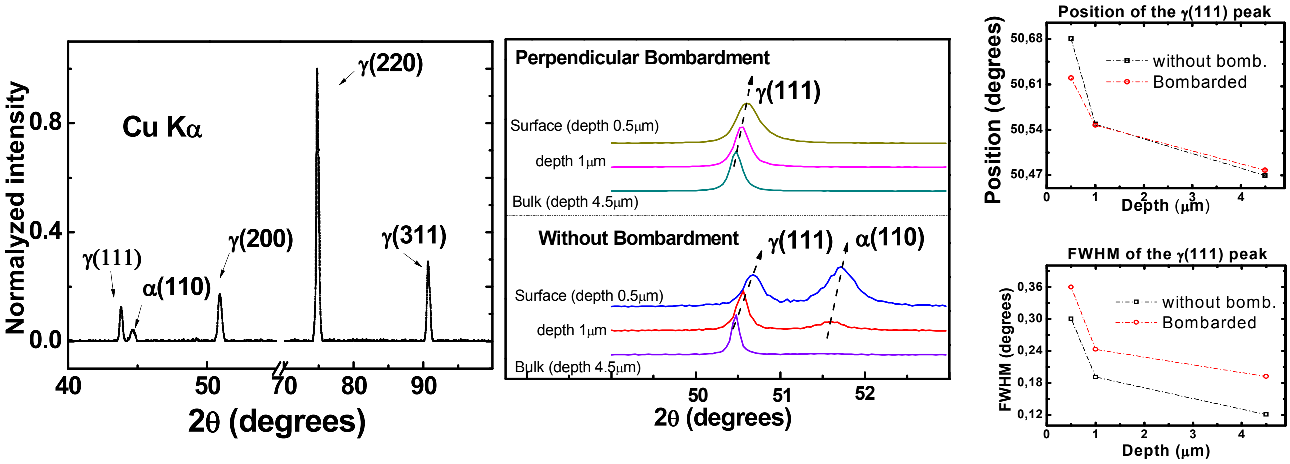


Figure 4: Left: X-ray diffraction of the SS316L sample. Middle: pristine sample and bombarded sample at three x-ray angles. Right: Peak and width evolution as a function of depth.

II.2. Effect of bombarding on pulsed plasma nitriding process

In this section we shall discuss the influence of the texture and related stress generated in the material by the ion bombardment on the plasma nitriding process. As explained in section II, the samples were prepared by partially screening the material surface during bombardment pretreatment in such a way that only a portion of the sample is texturized, as it is schematically indicated in Figure 5, top. After nitriding, a cross-section sample was sliced, mirror polished, attacked with Nital solution (5%) was studied, and its morphology scrutiny by electron microscopy (SEM-FEG, Figure 5, bottom).

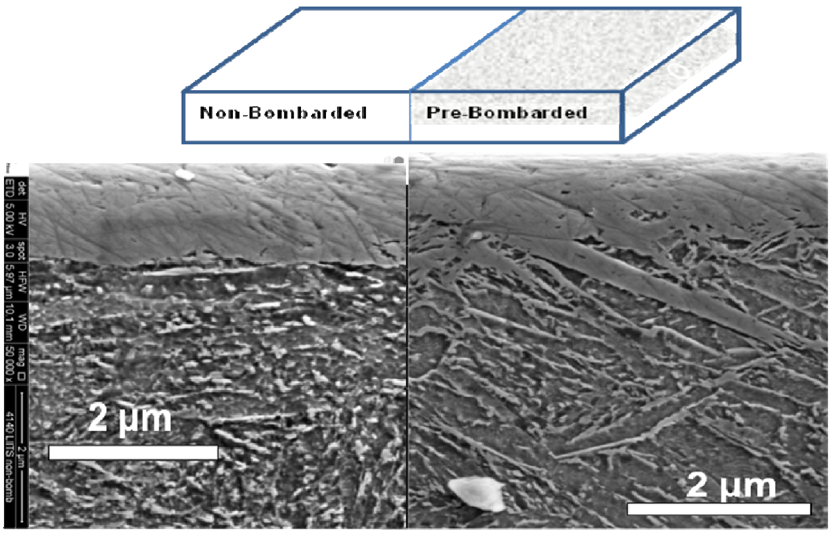


Figure 5. Top: in order to guarantee exactly equal starting condition, the sample left side was screened during the Xe^+ bombarding preparation, as schematically indicated in the sketch before nitriding. Bottom: SEM-FEG images of nitrided

sample after chemical etching (Nital Solution). Bottom left (right): SEM cross section micrograph of the non (pre) bombarded and posterior nitrided sample.

As observed in the micrographs, the microstructure nitride region is well different in the two regions of the studied sample. The diffusion region in the non-pre-bombarded sample shows fragmented and discontinues small nitride particles precipitates. On the other hand, the pre-bombarded region of the sample shows longer shaped needles extending few microns. This effect suggests that the bombardment is creating channels where nitrogen can easily diffuse for relative long distance, i.e., an effective larger nitrogen diffusion coefficient allows nitrogen moving faster into the material. Also, the increasing area due to the texturization is probably increasing nitrogen retention on the surface. The thicker white layer observed in the no pre-bombarded region of the sample is probably due to same reason, i.e., a smaller diffusion coefficient in this part of the sample result in a piling up of nitrogen at the surface of the material surface and consequently, a thicker white layer is formed. Nevertheless, more work is necessary in order to confirm this hypothesis.

II.3. Conclusions

The effect of the Xe^+ ion beam bombardment incident angle on the surface texture and strain of the surface of SS316L steel is reported. The generation of characteristic patterns depends on the crystalline orientation of the constitutive grains of the steel. The heavy ion bombardment induces effects at distance orders of magnitude larger than the stopping region of the projectiles. Plasma nitriding experiments show that the pre-bombarded samples present a peculiar microstructure forming long nitrides needles probably due to the presence of diffusion channels. On the contrary, the non-pre-bombarded samples show small, fragmented precipitates suggesting a smaller effective diffusion coefficient.

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