Low Temperature Plasma Diffusion Treatment of AISI316L Stainless Steel for Improved Hardness.

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Abstract

Samples of AISI316L stainless steel were plasma nitrided in a N_2 and mixture of $90N_2$-10Ar for treatment time of 4,5,10 and 15 houres by using dc glow plasma discharges. The treatment temperature was selected in 450–600 ºC intervals, in steps of 500C. X-ray diffraction and Vickers microhardness were used as analytical techniques. The XRD results of the plasma-nitrided samples showed that the modified intensities of the α and γ Fe phases along with those of the γ(CrN), γ(Fe_3N), and Fe_2O phases. The XRD patterns indicated that with treatment time of plasma nitriding increases, the Fe_3N and CrN fraction increases proportionally. The Microhardness measurements showed significant increase in the hardness from 220HV for the untreated samples up to 508HV for nitride samples at temperature of 600C in the nitrided layer.

Keywords: Plasma nitriding; Stainless steel; Glow discharge; Microhardness

1-Introduction

Glow discharges are used in various applications such as deposition of thin films, etching and modification of surfaces in semiconductor industry and materials technology [1-3]. The glow discharge plasma is created by applying a potential difference between two electrodes of certain distance apart in a partial vacuum. The potential difference is either constant in time (d.c. mode) or it can vary as a function of time (r.f. mode). Glow discharge plasma nitriding uses an abnormal glow discharge produced in a nitrogen gas or in a mixture of nitrogen and hydrogen and argon . Abnormal glow are discharges where the cathode of the discharge is fully covered by the glow. The substrate, which is also the cathode of the discharge, is typically biased negative between 400 and 800 V, and draws a current of 1–5 mA/cm^2. The operating pressure is usually between 1 and 10 mbar. Stainless steel hardened by nitriding is potentially such a useful material, and plasma nitriding gives such promise of a commercially practicable method of producing it, that much effort worldwide is now being devoted to realising this potential . When steel surfaces at elevated temperature are exposed to nitrogen plasma, the nitrogen atoms dissolve in the metal and form nitrides of the alloying elements. This is the plasma equivalent of a thermochemical diffusion process called nitriding. The layer of surface nitrides form a hard shield against wear, corrosion etc.[3-6].

2-Experimental

The samples that were used in this experiment was prepared from the cylindrical rod of 316L – stainless steel with diameter of 10mm. The samples were cut into a disk-shape with a height of 5mm. The specimens' stainless steel 316L were machined such as grinding and mirror polishing respectively using Struers-RotoPol-21 system from Denmark. The samples polished with a DP-suspension for (15, 9, 6, 3,1µm) and using the DP-cloth of the same company. All samples were cleaned in acetone by an ultrasonic cleaning machine for 30 min. prior to plasma nitriding.

In-home made system was used for this work, which has been manufactured and based on the following principal components: It is a low pressure gas discharge unite that consists of an evacuated chamber, the cathode faced the anode, which provide electric field for the gas discharge. Figure (1) shows photograph of the main experimental set-up used in this work (the plasma chamber, the electrical electrodes and the associated DC-power supply of 4 kV). The clean samples were kept on the sample holder and placed on the cathode plate for glow-discharge plasma nitriding. The plasma treatment of stainless steel (316L) samples were done under a N_2 (1-3mbar) and ratio of ($90N_2$: 10Ar) by pressure. The nitriding holding times were varied from 4-15 hour. The applied bias voltage was (400-500 V) with direct current density of (1-2 mA/cm^2). Table (1) summarized the nitriding conditions where the nitriding temperature in this study was adjusted to 450-600 C.

The treatment by plasma of only argon was used as a standard procedure to clean the surface of stainless-steel specimens. This process was performed only to ensure the exposure of the active surface of the samples for glow plasma, nitriding and be removing the native oxide and contaminated layer over the surface. The nitriding gas (N_2) was introduced in the evacuated chamber and the flow rate was adjusted until the pressure was stabilized at the desired pressure. The high voltage dc power supply was turned on and the cathode voltage and the discharge current were adjusted. The discharge current increased and adjusted until the sample temperature established of the nitriding temperature.

Fig.1: A photograph of the main experimental set-up.

Table (1): Working parameters of plasma nitriding for stainless steel (316L) specimen

specimen	Chamber gas	Pressure (mbar)	Supply voltage (Volt)	Discharge voltage (Volt)	Discharge current (mA)	Treatment time(h)	Specimen temperature (c)
	Ar	0.075	1340	1300	6	0.5	90
St.st(1)	N_2	1.3	1300	490	60	4	480
St. st (2)	N_2	1.3	1300	490	60-62	5	500-510
St.st (3)	N_2	1.3	1300	490	60-62	10	500-510
St. st (4)	N_2	1.3	1300	490	60-65	15	550-560
St. st (5)	N_2	3	1300	396	90-100	15	600-625
St. st (6)	$90N_2$-10Ar	3	1300	398	98-100	15	600

3- Results and Discussion

In the present work, diffraction studies are carried out using X- Ray shemadzu XRD – Diffractrometer (operated at 40 kVan accelerating potential and 30mA with filtered CuKα radiation

0.15418 A^{o} wavelength) was performed to identify the crystalline phases present in the surface layers. Untreated samples shows four γ-Fe (anstenite) peaks and one α-Fe. The presence of an α peak in untreated sample is due to the stress – induced transformation of γ to α during surface finishing, which is in good agreement with an earlier observation [3,7,8,9,10]. The observed γ peaks correspond to the (111) and (200) reflection planes, and the α peak corresponds to the (110) plane, as shown in figure (2). This figure shows the x-ray diffraction patterns of stainless steel samples after plasma nitriding. The plasma nitriding produces the well-resolved nitrided phases of CrN (200), Fe_3N (101) and FeO_2 (111). The γ phase has a fcc structure, where as the α phase has a bcc structure. The fcc structured γ phase resists nitride formation due to high packing fraction compared with the α phase. The compound layer of our work (nitrided specimens) has a gold like appearance (figure 3b) and consists of Fe_3N (200) nitrides. The specimens after nitriding during a time between 10 and 15h ,gas N_2 under a total pressure of 3mbar and voltage and discharge current were adjusted to maintain the cathode temperature at 500-550°C exhibit a golden greenish-red compound layer (figure 3c) composed of FeO_2, CrN and Fe_3N. Furthermore, the increasing of the discharge current (treatment time and temperature of nitriding) to 58 mA at 3mbar show a bluish compound layer consisting of FeO_2, CrN and Fe_3N and FeN_xO_y. Moreover it should be noted that specimens treated at high temperature shows compound layer that is highly adherent to the substrate. The Microhardness of plasma nitrided stainless steel-316L at (500-600) C as function of process time is shown in table2. For nitriding at higher temperature above 500C, the chromium nitrides precipitated in the nitride layer and cuaused austenite phase transform into ferrite phase or iron nitrides.This results shows the microhardness values measured on loading of 0.29N and timing of 20 second. The samples which nitrided for 5-15 hours exhibit higher microhardness values than untreated samples (table 2). This probably due to higher concentration of nitrogen and by the formation of the δ-Fe phase in the compound layer of samples surface [7,8,9,10].

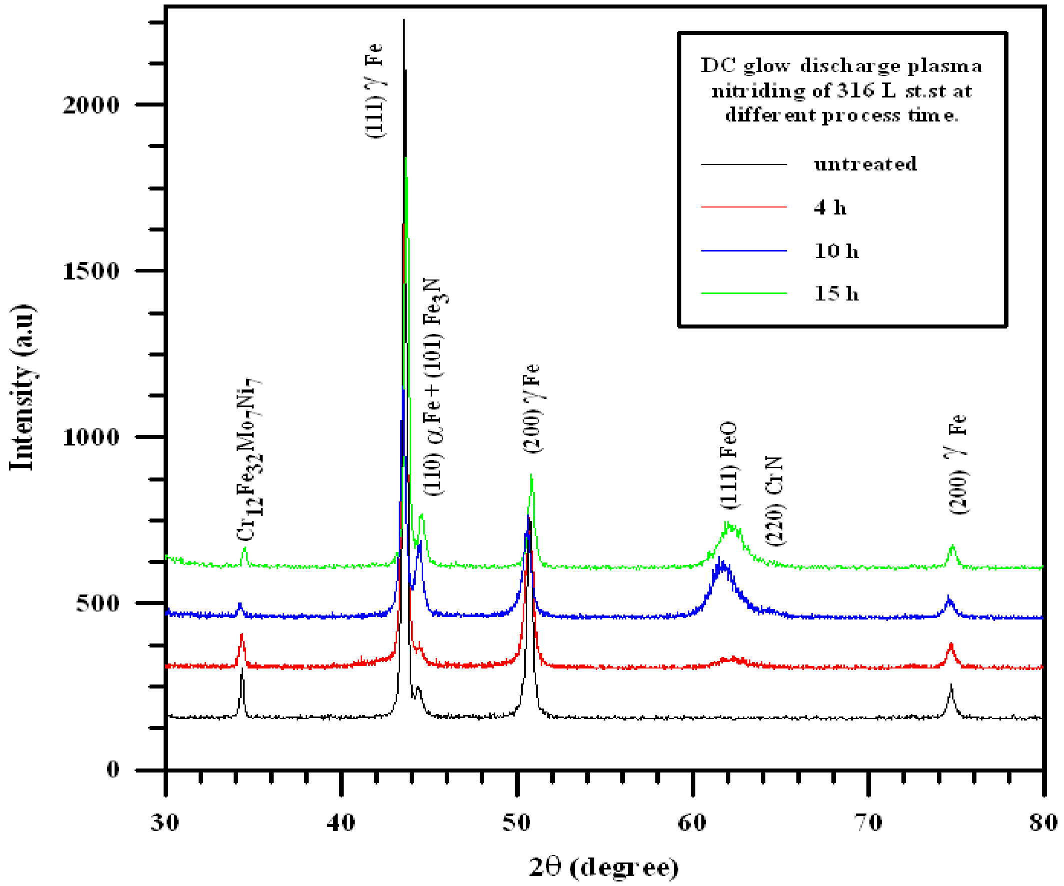

Fig.2: XRD diffraction pattern of specimen of untreated (a) and plasma nitrided at N_2, working pressure of 3 mbar, T=500C, for 10h (b) and 15h (c)

Table (2): Micro hardness of untreated and nitrided stainless steel (316L) specimens

Specimen	Plasma	Temperature treatment C°	Process time	Vickers hardness (VH)	Color
Untreated st.st.				218-220	Silver
st.st. 1	N_2	480	4	307-311	Golden
st.st. 2	N_2	500-510	5	307-330	Golden
st.st. 3	N_2	500-510	10	482-494	Golden greenish-red
st.st. 4	N_2	550-560	15	276-289	Bluish
st.st. 5	N_2	600-625	15	300-499	Bluish
st.st. 6	$90N_2$-10Ar	600	15	498-508	Golden-bluish

a b c

Fig.3: Micrographs showing typical surfaces colors of (a) untreated, plasma nitrided samples at 500°C for (b) 10h and (c) 15 h respectively.

3. Conclusion

From the results, it can be concluded that plasma nitriding of stainless steel (316L) improved the surface properties of the material. Surface hardness reaches a maximum of about 508 HV after 15 hours of plasma-nitriding at 600C. Plasma nitriding this material under the described experimental conditions resulted in a diffusion layer only without the white nitride layer. Thus, the mechanism of plasma-nitriding in these conditions was a diffusion-controlled process.

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