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Wear of plasma nitrided and nitrocarburized AISI 316L austenitic stainless steel

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ABSTRACT

Purpose: the purpose of the work is to compare the wear resistance, in dry and lubricated conditions, of AISI 316L austenitic stainless steel samples that were plasma nitrided or nitrocarburized at 450°C for 5 and 10 h, respectively.

Design/methodology/approach: Hardness and wear resistance of austenitic stainless steel can be increased substantially, without losing corrosion resistance, by plasma nitriding or nitrocarburizing surface treatments. In this work, AISI 316L austenitic stainless steel was plasma nitrided and nitrocarburized at 450°C, for 5 and 10 h respectively.

Findings: The obtained layers were characterized by optical microscopy, X-ray diffraction, microhardness and micro-wear tests in dry and lubricated conditions. Optical microscopy and X-ray diffraction analysis demonstrated that the nitrided layer is homogeneous and primarily composed of nitrogen rich expanded austenite with a thickness of about 15 μm. Nitrocarburized samples exhibited an external layer of chromium and iron compounds and a sub-layer of expanded austenite with a total thickness of 45 μm. Microhardness profiles showed that the hardness near to the surface was close to 1100 HV for nitriding and 1300 HV for nitrocarburizing. Plasma nitrided and nitrocarburized layers exhibited substantial wear reduction in dry and lubricated test conditions. The use of a lubricant oil reduces wear by a factor of approximately 200 compared to the dry test results.

Research limitations/implications: The plasma nitrided layer yielded the best wear performance in both dry and lubricated conditions.

Originality/value: Plasma nitriding resulted in the best wear performance when compared with nitrocarburizing in dry and lubricated sliding which is probably due to reduced layer fragility.

Keywords: Plasma; Nitriding; Nitrocarburizing; Wear; AISI 316; Stainless steel

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1. Introduction

The use of coatings and surface engineered components is increasing and is driven by the need for improved hardness, corrosion and wear resistance [1]. Austenitic stainless steels are the most important family of stainless steels with respect to both the number and types of applications. In fact, the omnipresence of stainless steels in our daily life makes impossible to enumerate their applications. They exhibit excellent corrosion resistance but their surface hardness is low and their wear resistance is insufficient for many applications. Several works have shown that plasma nitriding and nitrocarburizing at relatively low temperatures produced a hard surface layer which resulted in increased wear resistance without reducing corrosion resistance [2-7].

Nitriding and nitrocarburizing are processes where carbon and/or nitrogen are introduced into the surface of the steel at a specific temperature. Plasma or ion nitriding and nitrocarburizing are methods of surface hardening utilizing glow discharge technology to introduce nascent (elemental) carbon and/or nitrogen into the surface with subsequent diffusion into the material. The process is conducted in a vacuum under high voltage and the ions in the plasma that is formed are accelerated for impingement on the surface of the workpiece. This ion bombardment process heats the workpiece and cleans the surface, providing active nitrogen under the influence of the glow discharge to form the nitrided or nitrocarburized layer [8, 9].

Plasma nitriding and nitrocarburizing can produce layers composed of a phase called expanded austenite, "S-phase or γ_N". This very interesting phase is supersaturated with respect to nitrogen and is characterized by X-ray diffraction patterns with wider peaks dislocated to the left (higher d values) relative to the diffraction patterns of a standard austenitic matrix of austenitic stainless steels. This is due to a lattice constant expansion caused by the nitrogen insertion [10-12]. This layer of saturated interstitial solid solution possesses a good combination of mechanical, tribological, and corrosion properties.

This work compares the wear resistance, in dry and lubricated conditions, of AISI 316L austenitic stainless steel samples that were plasma nitrided or nitrocarburized at 450°C for 5 and 10 h, respectively.

2. Materials and methods

Samples of AISI 316L austenitic stainless steel were initially prepared according to conventional metallographic techniques by sanding and polishing. The chemical composition of the AISI 316L steel is given in Table 1. The samples were then cleaned in acetone and placed into the plasma chamber. Initially, a sputtering process with argon at a temperature of 400°C for 1 h was performed to remove the chromium oxide surface layer. After the sputtering process, the nitriding and nitrocarburizing treatment is initiated.

Plasma nitriding and nitrocarburizing were conducted in a vacuum chamber at 450°C with a pressure of 4 mBar in direct current mode. Nitriding was performed with a gas mixture of 80% H_2 and 20% N_2 for 5 h, and nitrocarburizing for 10 h in atmosphere comprising of 80% H_2, 18% N_2 and 2% CH_4. The plasma treated samples were analyzed by optical microscopy, X-ray diffraction analysis, measurements of microhardness and wear testing in dry and lubricated conditions.

Measurements of Vickers microhardness were performed using a Buehler digital equipment with load 25 gf and load time application of 10 s. The optical microscopy analysis were performed on a Zeiss microscope using the interference contrast technique after etching the samples with nitromuriatic acid.

X-ray diffraction patterns were obtained on the surface of the samples using a Rigaku Gergerflex equipment with scanning angles ranging from 10 to 80°. The analyses were performed using copper Kα radiation and continuous scanning with a speed of 2 °/min.

The micro-wear tests were conducted using a fixed ball machine without the use of abrasive. The diameter of the sphere was 25.4 mm and rotational speed of 500 rpm was used. For the dry sliding condition the applied load was 245 g (2.45 N) and for the tests with lubrication, the load was 1325 g (13.25 N). Lubricated wear testing was conducted using an SAE 20W-50 lubricant oil dripped between the sample and the sphere every 30 s Consecutive wear scars were produced with test times of 5, 10, 15 and 20 min to obtain the volume loss curve. The volume (V) removed of each wear crater and its depth (h) were calculated according to the following equations:

$$V \approx \frac{\pi \cdot d^4}{64 \cdot R}, \text{ for } d << R \tag{1}$$

$$h \approx \sqrt{\frac{V}{\pi \cdot R}}, \text{ for } h << R \tag{2}$$

in which *d* is the crater diameter (mm) and *R* is ball radius (mm).

3. Results and discussion

Microstructures of plasma nitrided and nitrocarburized AISI 316L stainless steel treated at 450°C are shown in Figure 1. The nitriding treatment performed for 5h (Fig. 1a) resulted in the formation of a homogeneous and continuous layer containing the S-phase. Nitrocarburizing for 10 h (Fig. 1b) indicates the presence of chromium and iron compounds occurring in the most external layer indicating that S-phase nucleates first.

Table 1.
Chemical composition of AISI 316L austenitic stainless steel (wt%)

C	Cr	Ni	Mo	Mn	Si	Fe
0.028	17.06	10.48	2.44	1.49	0.53	bal.

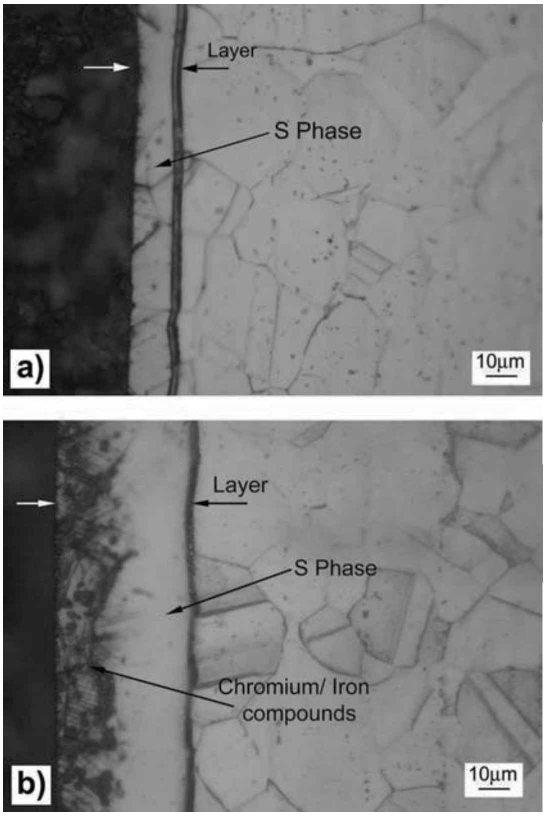

Fig. 1. Optical microscopy of: (a) nitrided and (b) nitrocarburized, AISI 316L

The resulting layer thickness was measured directly from the optical micrographs and was about 15 µm for nitriding and 45 µm for nitrocarburizing.

Figure 2 shows the X-ray diffraction patterns of the untreated, nitrided and nitrocarburized AISI 316L steel samples. The untreated AISI 316 steel contains only diffraction peaks related to the gamma (γ) iron phase which is characteristic of austenitic stainless steels. Nitriding at 450°C yielded the appearance of some diffraction peaks shifted to the left which is a characteristic of a nitrogen expanded austenite (S-phase). At this temperature, the layer is thin and the diffraction peaks of the substrate also appear.

The production of expanded austenite is characteristic of low plasma nitriding. At temperatures higher than 450°C chromium nitrides precipitation is favored. For nitrocarburizing with an increase in treatment time (from 5 to 10 h), the diffraction peaks of the substrate disappear due to the increase of the layer thickness. X-ray diffraction indicates that chromium nitrides (CrN and Cr_2N) and carbides (Cr_3C_2) are present together with an iron nitride (Fe_4N). Moreover, the expanded austenite is also detected. This confirms that the dark layer formed on the white expanded austenite after nitrocarburizing is a mixture of nitrides and carbides.

Microhardnesses profiles of the plasma treated test specimens are shown in Figure 3. Microhardness increased up to 450% for nitrided and 550% for nitrocarburized test specimens, in relation to the un-treated material. Nitrocarburizing produced a harder surface layer than nitriding.

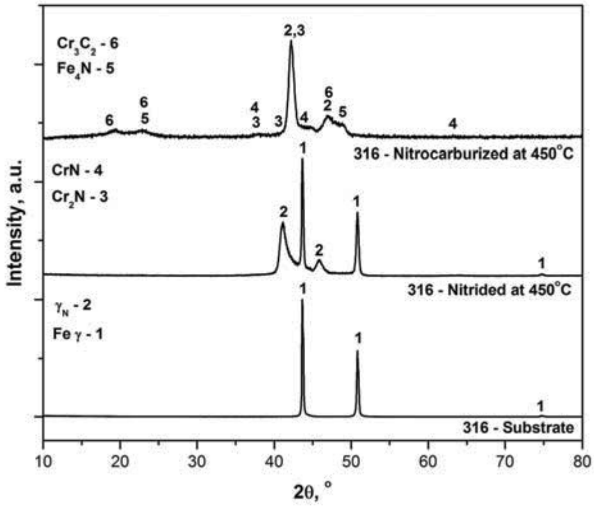

Fig. 2. X-ray diffraction patterns of untreated, nitrided and nitrocarburized AISI 316L steel

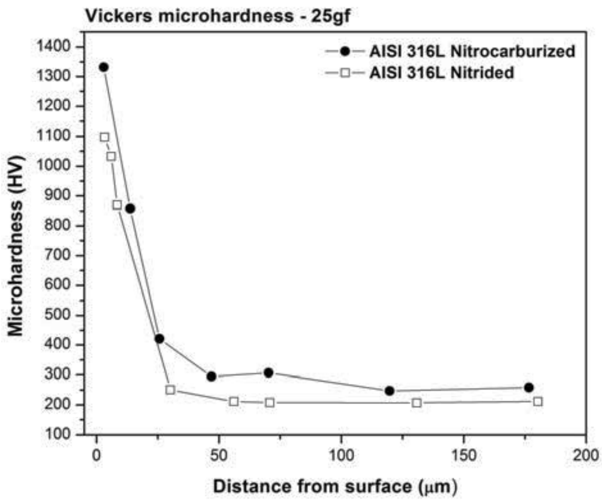

Fig. 3. Microhardness profiles obtained for plasma nitrided and nitrocarburized AISI 316 stainless steel

For both plasma treatments the hardness reaches the substrate in a value close to 250 HV, which is the characteristic of a austenitic stainless steel.

Table 2 provides values for the wear scar diameter after wear testing in dry and lubricated conditions for the AISI 316 steel untreated, nitrided and nitrocarburized. For all the testing conditions, whether dry or lubricated, the scar diameter increases gradually with time. The measured diameter is always greater for the dry wear test than the lubricated which shows the effectiveness of the oil for wear reduction.

The nitrided test specimens yielded the best wear performances with the lowest scar diameters in both dry and lubricated sliding conditions. According to the microhardness profiles shown in Fig. 3, nitrocarburizing produces a higher hardness than nitriding which can lead to superior layer fragility. Fernandes, *et. Al.* observed the same tendency after plasma nitriding of an austenitic stainless steel and found that the harder the nitrided layer, the higher the wear volume [13].

Table 3 shows the wear scar depth (h) calculated using Eq. 2 which is based on the volumetric loss after a traveled distance of 798 m (or 20 min of sliding) and the thickness (e) of the plasma treated layers. None of the wear tests resulted in perforation of the nitrided or nitrocarburized layer. Therefore, the wear results represent the intrinsic properties of the produced layers.

The wear scar diameters values were converted to volume using Eq. 1 and plotted. Figure 4 shows the volumetric wear curves of the substrate, the nitrided and nitrocarburized AISI 316L steel in dry (Fig. 3a) and lubricated (Fig. 3b) test conditions.

The layers were very effective in increasing wear resistance of AISI 316L stainless steel in the two test conditions employed. At the beginning of the dry test, the wear of the layers was about 3 times less than that of the substrate and at the end of test, this value was 7 times lower. This indicates that the nitrided and nitrocarburized materials stabilize the wear process relative to the AISI 316L steel substrate.

With respect to the sliding wear using oil as a lubricant, the nitrided layer exhibited wear performance slightly superior to the nitrocarburized AISI 316L steel test samples. However, both layers produced exhibited a decrease in wear of about 4 times less than the substrate.

Table 2.
Wear scar diameter measurements for plasma treated and untreated 316L in dry and lubricated condition

	Untreated		Nitrided		Nitrocarburized	
	Scar diameter, mm		Scar diameter, mm		Scar diameter, mm	
Time, min	Dry	Lubricated	Dry	Lubricated	Dry	Lubricated
5	1.74±0.03	0.35±0.02	0.405±0.02	0.276±0.005	1.153±0.005	0.271±0.002
10	1.93±0.06	0.444±0.009±	0.755±0.05	0.298±0.009	1.36±0.02	0.30±0.01
15	2.18±0.04	0.48±0.02	0.787±0.03	0.317±0.004	1.48±0.04	0.33±0.02
20	2.29±0.05	0.55±0.01	0.811±0.03	0.367±0.007	1.53±0.03	0.403±0.09

Table 3.
Scar depth (h) after 20min of wear testing and nitrided or nitrocarburized layer thickness (e)

	e (μm)	Wear condition	h (μm) – 20min
AISI 316L	---	Dry	52±2
		Lubricated	2.9±0.2
AISI 316L - Nitrided	14.5±0.8	Dry	6.48±0.06
		Lubricated	1.33±0.06
AISI 316L - Nitrocarburized	44±1	Dry	23.1±0.9
		Lubricated	1.60±0.07

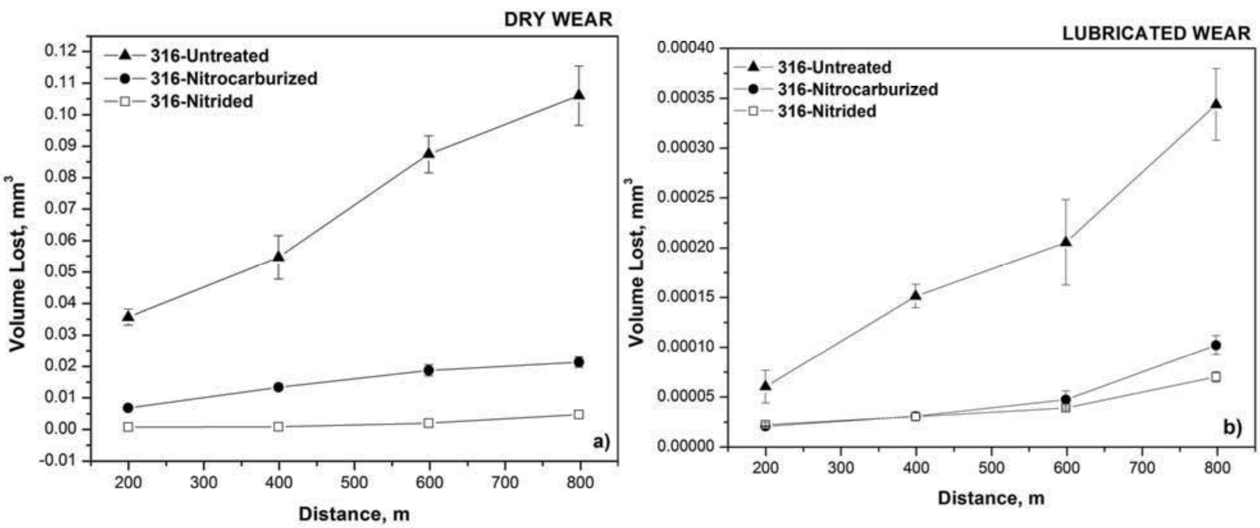

Fig. 4. Volumetric loss curves against distance for: (a) dry wear and (b) lubricated wear, of the plasma nitrided and nitrocarburized AISI 316 stainless steel

4. Conclusions

In plasma nitriding and nitrocarburizing of austenitic stainless steel a compound layer was produced, which, depending on the process, can result in hardness improvements of 450 to 550% respectively relative to the non-treated steel. Nitrocarburizing at 450°C for 10h produces a layer thicker than that produced by nitriding at the same temperature for 5 h.

According to the X-ray diffraction patterns, the nitrided layer is mainly composed of expanded austenite and nitrocarburizing resulted in the formation of a two-phase layer constituted of chromium/ iron nitrides and chromium carbide at the top and expanded austenite beneath.

Plasma nitrided and nitrocarburized layers presented substantial wear reduction in both dry and lubricated conditions. With the use of a lubricant during the wear tests, the volumetric loss is reduced by a factor of 200 relative to dry sliding.

Plasma nitriding resulted in the best wear performance when compared with nitrocarburizing in dry and lubricated sliding which is probably due to reduced layer fragility.

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