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Effect of Temperature and Pressure on Wear Properties of Ion Nitrided AISI 316 and 409 Stainless Steels*

ABSTRACT: Stainless steels are widely used in chemical and other industries due to their corrosion resistance. However, because of their low hardness and wear properties, their applications are limited. Many attempts have been made to increase the surface hardness of these materials by using plasma techniques. Plasma nitriding is distinguished by its effectiveness, and for presenting a relatively low cost and being a clean process, producing hard surface layers on stainless steels. Aiming to verify the influence of the temperature and pressure on the modified resultant layers, samples of AISI 316 and 409 stainless steels were plasma nitrided in two different temperatures (450 and 500°C) and pressures of 400, 500, and 600 Pa for 5 h. After the nitriding treatment, the layers were analyzed by means of optical microscopy and wear tests. Wear tests were conducted in a fixed-ball micro-wear machine without lubrication. After the plasma nitriding treatment on AISI 316 and 409 samples, homogeneous and continuous layers were produced and their thicknesses increased as the temperature increased and as the pressure decreased. The nitriding treatment on the AISI 316 steel resulted on the formation of expanded austenite layers at 450°C and chromium nitrides (CrN and Cr_2N) phases at 500°C. The nitriding treatment on AISI 409 yielded the formation of similar layers for both treatment temperatures; these layers constituted mainly chromium (Cr_2N) and iron (Fe_2N, Fe_3N, and Fe_4N) nitrides. After the nitriding treatment, the AISI 316 steel sample presented higher wear resistance for lower temperature and pressure values. The increase on layer fragility, for higher temperature and pressure values, can be responsible for this inverse tendency. The wear resistance of the nitrided AISI 409 sample followed a logic tendency: the harder the layer, the better the performance, i.e., the performance was improved with the increase in both the temperature and pressure.

KEYWORDS: stainless steel, plasma nitriding, wear

Introduction

Stainless steels are an important class of ferrous alloys. In fact, the omnipresence of stainless steels in our daily life makes it impossible to enumerate their applications [1,2]. Their corrosion resistance is excellent, whereas their hardness and wear resistance are relatively poor, limiting some of their industrial applications. Therefore, many attempts have been made to increase the surface hardness and wear of this class of materials.

Low temperature plasma nitriding treatment of stainless steels provides an attractive process to improve their wear resistance without affecting their corrosion properties [1–4]. Lower temperature

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TABLE 1—*Chemical composition (weight percentage) of the employed steels.*

	C	Cr	Ni	Mo	Mn	Si	Fe
AISI 316L	0.028	17.06	10.48	2.44	1.49	0.53	Bal.
AISI 409	0.030	11.25	0.06	0.11	0.74	0.49	Bal.

means lower cost, less distortion, and less surface roughening [5]. During this treatment, a very hard (greater than 1000 HV) and corrosion resistant metastable phase, expanded austenite "γ_N," is formed at the surface of these steels [6–8]. This very promising coating can only be achieved if the treatment temperature is lower than 500°C [3]. Above 500°C, chromium atoms are removed from the austenitic structure resulting in the precipitation of chromium nitrides (CrN and/or Cr_2N), compromising the corrosion properties [9].

The expanded austenite is a metastable phase with a supersaturation of nitrogen, which remains in solid solution [9–11]. This thin precipitate-free layer of saturated interstitial solid solution possesses a good combination of mechanical, tribological, and corrosion properties [11]. The produced layers on ferritic steels are mainly constituted by iron and chromium nitrides [12]. The nature of the plasma nitrided layer on austenitic stainless steel, which is considerably different from that of the usual ferritic steels, is not completely understood yet [13].

The purpose of the present work is to verify the influence of the treatment temperature and pressure on the morphology and wear properties of plasma nitrided AISI 316 and 409 stainless steels.

Materials and Methods

The stainless steels investigated in this work are commercially available materials, and its chemical compositions are given in Table 1. The samples employed in this investigation were solubilized at 1050°C for 30 min followed by water quenching.

Samples of AISI 316L and AISI 409 were prepared according to conventional metallographic techniques and then were plasma nitrided. The samples were cleaned by argon sputtering (under working pressure and a temperature 50°C lesser than the nitriding temperature, for 30 min) inside the plasma chamber. The plasma treatment was done using the direct current method with the following conditions: a gas mixture of 80 vol. % H_2 and 20 vol. % N_2, pressure values of 400, 500, and 600 Pa, temperature values of 450 and 500°C, and time period of 5 h. Ion nitrided samples were analyzed by optical microscopy, measurements of microhardness, X-ray diffraction, and wear testing.

Microstructural analysis was carried out by means of a Zeiss Axiotech optical microscope using the interference contrast technique after etching the samples with nitromuriatic acid. Brinell indentations were carried out on the surface of the nitrided samples to evaluate the indentation crack network aspect. The indentation was performed with a load of 15.625 kg using a 2.5 mm steel sphere, applying the load for 10 s. Micro-wear tests were performed in a fixed-ball machine type without the use of abrasive using a sphere of 25.4 mm in diameter. The rotation speed and load were 500 rpm and 2.45 N, respectively. Consecutive wear scars were produced with test times of 5,10,15, and 20 min to obtain the volume loss curve. The removed volume (V) of each wear crater and its depth (h) were calculated according to the following equations [14,15]

$$v \approx \frac{\pi \times d^4}{64 \times R}, \text{ for } d \ll R \quad (1)$$

$$h \approx \sqrt{\frac{V}{\pi \times R}}, \text{ for } h \ll R \quad (2)$$

where:

d= scar diameter and

R= sphere radius.

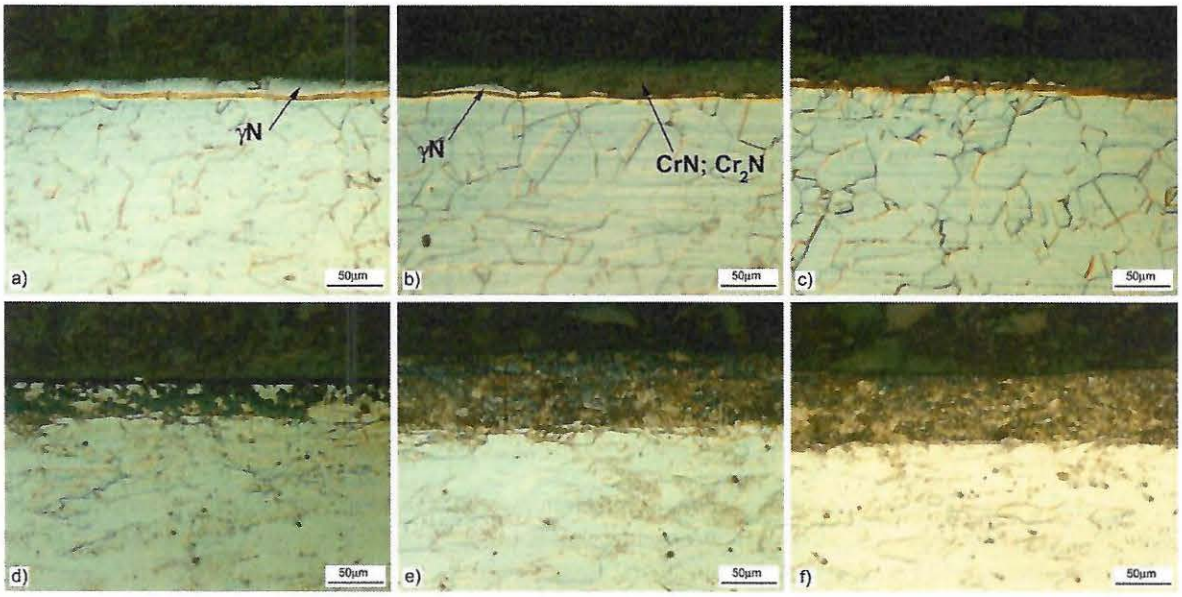


FIG. 1—*Optical cross-section of nitrided samples: (a) AISI 316-450°C-400 Pa; (b) AISI 316-500°C-400 Pa; (c) AISI 316-500°C-600 Pa; (d) AISI 409-450°C-400 Pa; (e) AISI 409-500°C-400 Pa; and (f) 409-500°C-600 Pa.*

Results and Discussion

Figure 1 presents the optical micrographs of the cross-section of plasma nitrided AISI 316 and 409 samples at 450 and 500°C for 5 h of treatment and pressures of 400 and 600 Pa.

Figure 1(*a*) shows a cross-section of an AISI 316 sample nitrided at 450°C and 400 Pa, in which the formed layer is constituted by expanded austenite and possibly the beginning of chromium nitrides precipitation, which are characteristic of low temperature plasma nitriding (<500°C) of austenitic stainless steels. Increasing the temperature to 500°C (Fig. 1(*b*)), under the same pressure, the white expanded layer is replaced almost entirely by a dark chromium nitride layer that grew on top of the γ_N layer. The AISI 409 ferritic stainless steel presented the same layer constituent (mainly iron and chromium nitrides) for both 450°C (Fig. 1(*d*)) and 500°C (Fig. 1(*e*)) nitriding temperature. It was observed for both austenitic and ferritic steels that increasing nitriding temperature results in thicker layers.

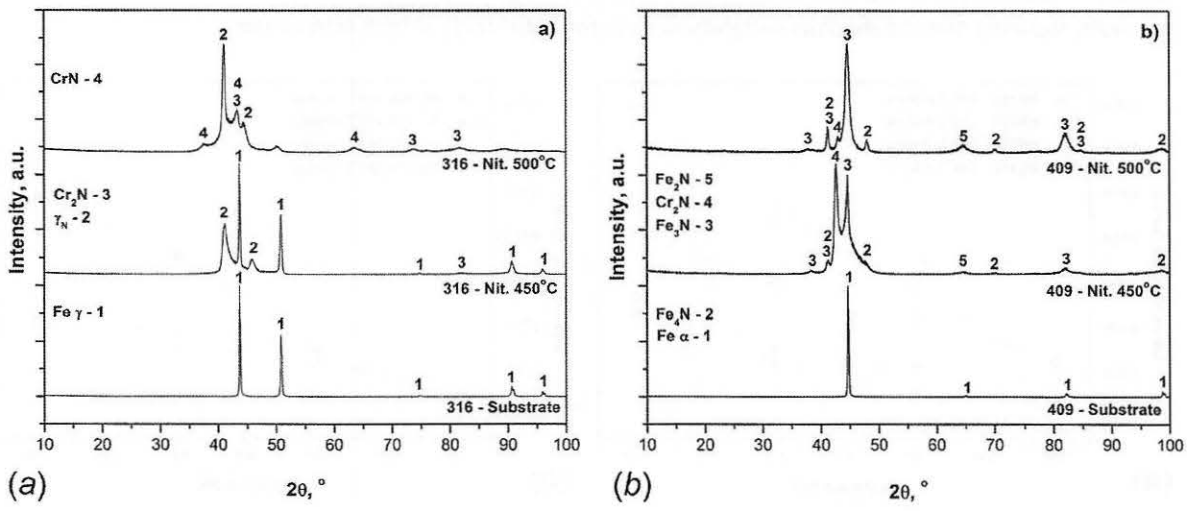


FIG. 2—*X-ray diffraction patterns of the non-nitrided and plasma nitrided samples of: (a) AISI 316; (b) AISI 409, treated with 400 Pa.*

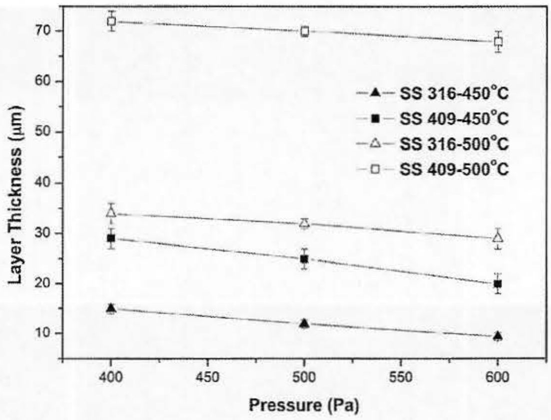


FIG. 3—*Layer thickness versus nitriding pressure of plasma nitrided AISI 316 and 409.*

Nitriding the AISI 316 and 409 steels under different pressures (400 and 600 Pa) at the same temperature (500°C) resulted on a slightly decrease on layer thickness after increasing the pressure (Fig. 1(*b*) and 1(*c*) for austenitic and Fig. 1(*e*) and 1(*f*) for ferritic stainless steel).

The X-ray diffraction patterns of the AISI 316 and 409 substrates and the plasma nitrided samples at different temperatures (450 and 500°C) at 400 Pa are presented in Fig. 2. The untreated AISI 316 and 409 steels contain diffraction peaks related to the gamma (γ) iron phase and alpha (α) iron phase, which correspond to austenitic and ferritic stainless steels, respectively.

Nitriding the AISI 316 at 450°C (Fig. 2(*a*)) yielded the appearance of some diffraction peaks shifted to the left, which correspond to a nitrogen expanded austenite (γ_N) phase. Also a peak related to Cr_2N appears, which indicates the beginning of the precipitation of chromium nitrides. At this temperature (450°C), the layer is thin and the diffraction peaks of the substrate (γ iron) also appear. Further increase in the nitriding temperature (500°C) maintain the expanded austenite diffraction peaks, and more CrN and Cr_2N peaks are detected, indicating clearly the precipitation of these nitrides. These findings confirm that the dark layer formed onto the white expanded austenite is a mixture of chromium nitrides.

The X-ray diffraction patterns of the plasma nitrided AISI 409 (Fig. 2(*b*)) shows, for both employed temperatures (450 and 500°C), a mixture of chromium (Cr_2N) and iron (Fe_2N, Fe_3N, and Fe_4N) nitrides. No expanded phase but rather a compound layer and/or precipitates were formed, which are in agreement with the literature [11].

The thicknesses of the layers, obtained by direct measurements using optical micrographs, versus nitriding pressure for AISI 316 and 409 samples are plotted in Fig. 3, which clearly shows that the thicknesses of the layers are superior for the AISI 409 samples compared to the AISI 316 samples for both temperatures. However, an inverse effect was observed when plotting layer thickness versus nitriding pressure, showing that the thickness slightly decays for both steels at both temperatures.

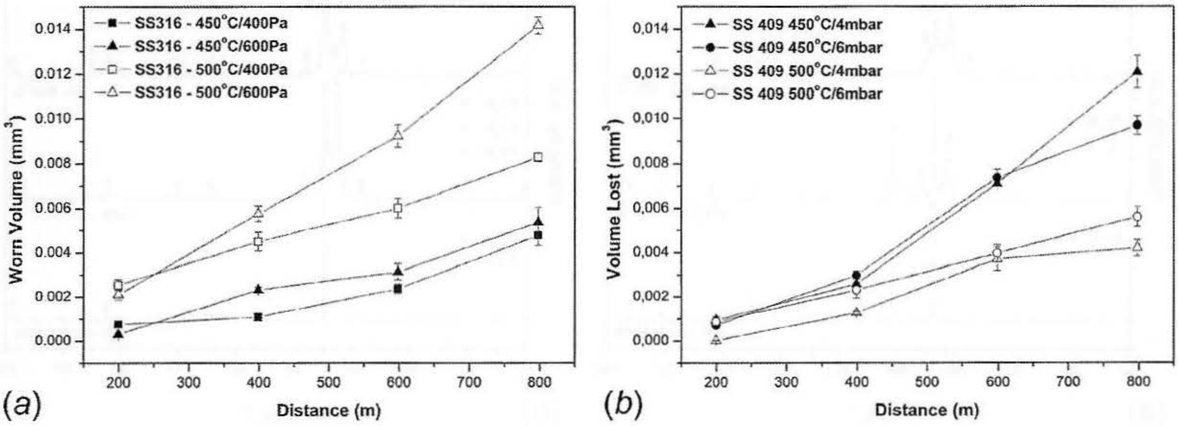


FIG. 4—*Worn volume of plasma nitrided samples of: (a) AISI 316; (b) AISI 409, as a function of sliding distance.*

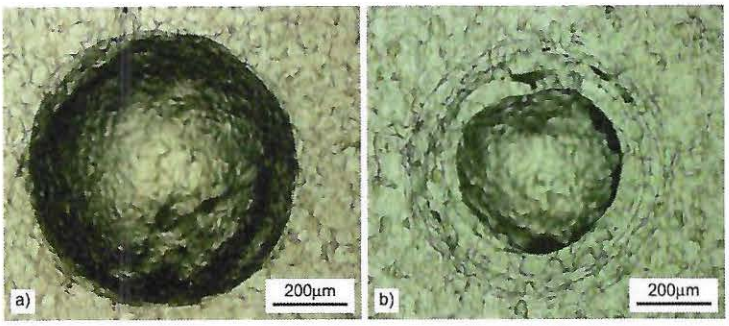


FIG. 5—*Brinell indentations on plasma nitrided samples of: (a) AISI 316 at 450°C; (b) AISI 316 at 500°C, 600 Pa.*

Wear Characterization

Figure 4 presents the wear volume loss curves after tribological characterization in dry sliding for the AISI 316 and 409 samples nitrided at 450 and 500°C under 400 and 600 Pa.

The worn volumes were calculated based on Eq 1 using the crater diameters. The volumetric wear curves of the nitrided AISI 316 sample (Fig. 4(*a*)) show that nitriding at 450°C presented a lower worn volume than at 500°C. As the pressure was increased (from 400 to 600 Pa), the resistance against wear decreased. This inverse tendency is elucidated after observing Fig. 5 and Table 2, which show two photomicrographs of a Brinell indentation on the nitrided AISI 316 sample and Brinell hardness values, respectively.

Brinell indentations on the AISI 316 samples nitrided at 450°C (Fig. 5(*a*)) and 500°C (Fig. 5(*b*)) under 600 Pa, displayed in Fig. 5, show that a crack network is produced around the indentation, for the nitriding temperature of 500°C, which is an evidence of higher fragility than that indentation performed on the sample nitrided at 450°C.

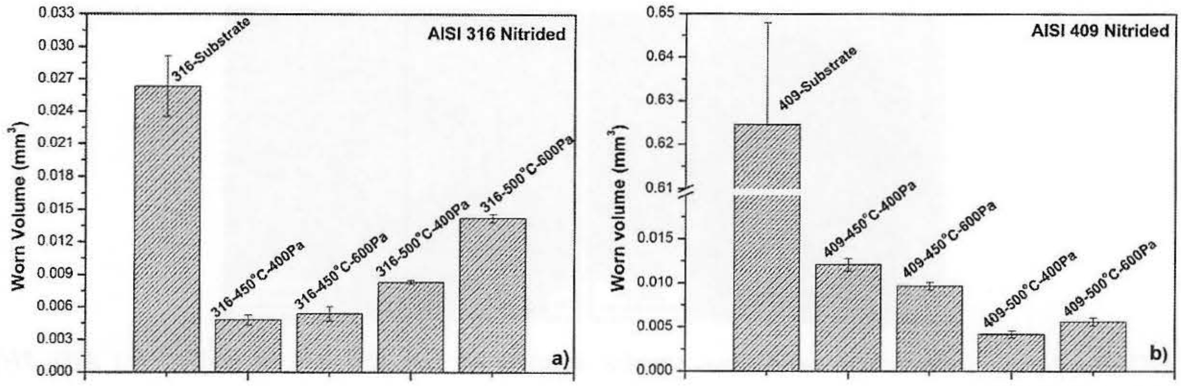
Increasing the nitriding temperature from 450 to 500°C caused an increment on hardness (Table 2) and also an increase on the layer fragility (Fig. 5(*a*) and 5(*b*)). During the sphere sliding on wear testing, a fragile layer will generate a greater amount of wear debris, which in turn causes acceleration in the wear process. The plasma nitriding of the AISI 316 sample at 500°C resulted in different layer constituents when compared to the treatment done at 450°C (Figs. 1(*a*), 1(*b*), and 2(*a*)) with distinct properties, which yielded different tribological behaviors.

Wear curves of the ferritic stainless steel (Fig. 4(*b*)) showed that with the increase of both nitriding temperature and pressure, the wear resistance improved. Thus, the volume lost decreased as the temperature and pressure raised and also as hardness increased (Table 2). Since the layers produced at 450 and 500°C are mainly composed by the same compounds (Figs. 1(*d*), 1(*e*), and 2(*b*)), their wear behavior follows a natural and logic tendency. A harder layer results in a better wear performance.

Table 2 also lists the layer thicknesses (e) and the wear scar depths (h) after 20 min (800 m) of wear testing obtained for each nitriding condition for 400 and 600 Pa and for the untreated AISI 316 and 409 samples, for comparison purposes. The h values were obtained based on Eq 2.

TABLE 2—*Scar depth (h) after 20 min of wear testing, nitriding layer thickness (e), and Brinell hardness (HB).*

Sample	h (μm)—20 min	*e* (μm)	HB
AISI 316	26 ± 1	...	147 ± 3
AISI 316 450°C/400 Pa	10.9 ± 0.5	14.9 ± 0.9	180 ± 11
AISI 316 450°C/600 Pa	11.6 ± 0.7	9.4 ± 0.8	188 ± 8
AISI 316 500°C/400 Pa	14.4 ± 0.2	34 ± 2	313 ± 14
AISI 316 500°C/600 Pa	18.9 ± 0.3	29 ± 2	444 ± 8
AISI 409	125 ± 2	...	188 ± 9
AISI 409 450°C/400 Pa	17.4 ± 0.5	29 ± 2	242 ± 11
AISI 409 450°C/600 Pa	15.6 ± 0.3	20 ± 2	246 ± 20
AISI 409 500°C/400 Pa	10.2 ± 0.4	72 ± 2	291 ± 3
AISI 409 500°C/600 Pa	11.9 ± 0.5	68 ± 2	353 ± 23



(*a*) (*b*)

FIG. 6—*Worn volumes after 800 m of wear testing of the (a) AISI 316; (b) AISI 409 plasma nitrided samples, and the substrates.*

Comparing the layer thicknesses (*e*) and the scar depths (*h*) after 800 m of wear testing for all nitrided samples, it can be noted that all tests did not perforate the layer, except for the AISI 316 sample plasma nitriding at 450°C under 600 Pa. Table 2 also shows that the Brinell hardness (HB) increases as temperature and pressure were raised, for both, austenitic and ferritic steels.

Figure 4 shows the worn volumes, which occurred at the end of the wear tests carried out on nitrided layers and the substrates, for comparison purposes. Worn volume of the AISI 409 steel sample (Fig. 6(*b*)) is around 20 times greater than that observed for the AISI 316 one (Fig. 6(*a*)).

The worn volumes after 800 m of sliding are plotted in bars and show that, for the nitrided steel samples, the maximum volume lost value was about 0.015 mm^3, which is 2 times lesser than the observed for the AISI 316 substrate and 40 times lesser than the observed for the AISI 409 substrate. This confirms that the plasma nitriding treatment is effective regarding the wear resistance.

Conclusions

Plasma nitriding treatment on the austenitic (AISI 316) and ferritic (AISI 409) stainless steels produced continuous and homogeneous layers. The nitrided AISI 316 sample presented expanded austenite layers, for the treatment temperature of 450°C, and layers having chromium nitrides (CrN and Cr_2N), for the treatment temperature of 500°C. Nitriding the AISI 409 samples yielded similar layers for both treatment temperatures, mainly chromium (Cr_2N) and iron (Fe_2N, Fe_3N, and Fe_4N) nitrides. The layer thickness increased as the temperature was raised. An inverse effect was observed for the layer thickness in respect to the pressure. The surface hardness of the nitrided steels increased as the treatment temperature and pressure were raised.

The AISI 316 substrate steel showed a better wear performance than the AISI 409 one. After plasma nitriding, the AISI 316 steel yielded higher wear resistance for lower treatment temperature and pressure. The increase in layer fragility, for higher temperatures and pressures, observed for the nitrided AISI 316 samples, was responsible for this inverse tendency.

The wear resistance of the nitrided AISI 409 sample followed a logic tendency: the harder the layer, the better the performance. In other words, the performance was improved with the increase of both temperature and pressure.

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