



Duplex treatment of 304 AISI stainless steel using rf plasma nitriding and carbonitriding

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ABSTRACT

Surface of 304 AISI austenitic stainless steel has been modified using duplex treatment technique of nitriding and carbonitriding. A thick modified nitrided layer, of approximately 20 μm , has been achieved when rf inductively coupled plasma was adjusted at 450 W for processing time of only 10 min. After performing the nitrided layer, the nitrided samples were carbonitrided using the same technique at different acetylene partial pressure ratios ranges from 10% to 70%, the balance was pure nitrogen. Different amount of nitrogen and carbon species are diffused underneath the surface through the nitrided layer during carbonitriding process and are found to be gas composition dependent. The treated samples were characterized by glow discharge optical spectroscopy, X-ray diffractometry, scanning electron microscopy and Vickers microhardness tester. The microstructure of the duplex treated layer indicates the formation of γ -Fe₄N, Fe₃C, CrN and nitrogen-expanded austenite (γ_N). The thickness of the duplex treated layer increases with increasing the acetylene partial pressure ratio. The surface microhardness of duplex treated samples has been found to be gas composition dependent and increased by 1.29 fold in comparison to the nitrided sample.

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1. Introduction

Stainless steels are introduced into various today's industrial applications such as food, storing, chemical, vacuum modeling tools and medical industries for their excellent corrosion resistance. However, they exhibit relatively low wear resistance and relatively low microhardness, which impede some of their specific applications. Thermo-chemical treatments of nitriding, carburizing and nitrocarburizing have been used for a long time as single treatment techniques, essentially to improve their superficial mechanical properties [1–7]. Different plasma techniques have been used for nitriding stainless steels such as ion implantation [8,9], low-energy high-current nitriding [10,11], plasma immersion ion implantation [12–13] and rf plasma nitriding [14–16]. Alternatively, rf plasma carbonitriding has been used recently for surface modification of 304 stainless steel using gas mixture of acetylene and nitrogen [2–3,5,17].

Duplex treatment technique has been recently considered as a newly developed surface treatment method to improve the surface properties of stainless steels and other ceramic alloys [18–28]. It is a surface modification method combined two sequence techniques; a hard coating is performed on the surface treated substrate. It has

been found that the substrate should have a sufficient hardness and flow strength to support the coating without plastic deformation when subjected to a high-intensity loading. Duplex treatment of calorizing followed by plasma nitriding [18], plasma nitriding followed by PVD coating [19], preheating followed by PVD coating [20] and plasma electrolytic nitrocarburising coated with diamond-like carbon [21] are mentioned here as examples. Duplex treatment process has been controlled by varying the conditions of specific treatment techniques. An adhered coating of TiN, CrN or carbon deposition which was individually achieved on different nitrided substrates using various duplex treatment processes revealed superficial tribological properties, greater load-bearing capacity and higher corrosion resistance compared to the only nitrided substrates and to the direct coating on un-nitrided substrates [22–24].

It has been found that the carbonitrided layers of austenitic stainless steel exhibit better corrosion resistance in comparison to these treated by pure nitriding or carburizing. This improvement has been reverred to the existence of a solid solution phase γ_N [3], which is hard phase and it has a good corrosion resistance with respect to γ -austenite [4]. Blawert and coworkers were found that the chemical compound phases (nitrides and carbides) in the presences of the solid solution phases (nitrogen-expanded austenite and carbon-expanded austenite) are accountable about the significant improvement of surface microhardness [29].

To improve mechanical properties of 304 austenitic stainless steels, duplex surface treatment using inductively coupled rf plasma

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nitriding and carbonitriding has been employed. A carbonitrided layer was formed on the nitrided AISI 304 stainless steel substrate using different gas composition of nitrogen and acetylene with respect to gas pressure ratio. The duplex treated substrates were examined using various analytical techniques of glow discharge optical spectroscopy (GDOS), X-ray diffraction (XRD), scanning electron microscopy (SEM), and Vickers microhardness tester.

2. Experimental

A 1 mm thick of 304 austenitic stainless steels was cut into specimens with dimensions 20 mm × 10 mm × 1 mm, which were cleaned in acetone before the treatment. It has a chemical composition of 0.5 wt.% Si, 1.2 wt.% Mn, 8.5 wt.% Ni, 19.1 wt.% Cr, 0.075 wt.% C and 69.95 wt.% Fe. Duplex surface treatment process was carried out using an inductively coupled rf plasma nitriding and carbonitriding. Rf plasma system consists of a cylindrical quartz tube of 500 mm length and 41.5 mm diameter. The cylindrical quartz tube was evacuated to a typical base pressure of 7×10^{-3} mbar by a rotary pump. In the pretreatment process of rf plasma nitriding, pure nitrogen gas was introduced in the reactor tube to increase the base pressure from 7×10^{-3} mbar to about 8×10^{-2} mbar. The discharge was generated by a three-turn copper induction coil energized from a 13.56 MHz rf power supply through a tunable matching network. The distance between the substrate surface and the rf coil was fixed at 29 mm. The water cooling rate of the substrate was adjusted to be 1500 cm³/min. The substrates were treated at a fixed input plasma power of 450 W for a processing time of 10 min. The substrate temperature is measured during the rf plasma process by a Chromel-Alumel thermocouple which is attached to the surface of the sample. It was approximately stable during the treatment process at about 450 °C. At the end of the pretreatment process, the nitrided sample was leaved in the evacuated reactor tube until it cool down to the room temperature. Duplex treatment process was employed without initial sputtering or heat treatment prior to nitriding or carbonitriding to remove the native oxide layer from the surface of the untreated substrates. The nitrided samples were carbonitrided using different acetylene partial pressure ratios from 10% to 70%. The total gas pressure was adjusted to be 8×10^{-2} mbar. The distance between the substrate surface and the rf coil was decreased to 24 mm. The water cooling rate of the substrate was reduced to 600 cm³/min. The nitrided substrates were carbonitrided at the same plasma power (450 W) and plasma processing time (10 min). The treatment temperature of carbonitriding was varied from 520 °C to 590 °C depending on gas composition. Finally, the duplex treated samples were leaved in the evacuated reactor tube until they cool down to the room temperature.

Different characterization techniques were used to analyze the duplex-treated samples and the results were compared with those obtained for the nitrided one. A diffractometer with Cr radiation was employed for structure analysis. A glow discharge optical spectroscopy (GDOS) was utilized to measure the elemental concentration depth profiles. Scanning electron microscopy (SEM) was employed to study the microstructure of the duplex treated substrates. Vickers microhardness measurements were taken on the untreated and treated substrates using a load of 100 g at room temperature.

To measure the thickness of the treated layers, the treated samples were cut into small work pieces using ISOMET™ low speed saw. These small parts were cold mounted as cross-sections and grinded using abrasive grit with different grades started by 40 and ended by 400 meshes and polished to mirror like using micro polish of alumina suspensions 0.3 and 0.1 micron. After that, the cross-sectioned samples were etched for 30 s to reveal the surface microstructure under optical microscopy. The etching solution was prepared using 50 ml acetic acid and 50 ml nitric acid added to 50 ml distilled water. Finally, the layer thickness was measured by microhardness tester and visibly confirmed by the optical images.

3. Results and discussion

3.1. Treatment temperature

The austenitic stainless steel substrates are heated up only by the electric field of the inductively coupled rf plasma coil in both processes of nitriding and carbonitriding. At the initial stage of the treatment process, the substrate temperature is increased very fast and stabilized after approximately 2 min of plasma processing time. The substrate temperature has been found to be 450 °C for pure nitriding where the distance between the substrate and rf coil was adjusted at 2.9 cm. The carbonitriding process is carried out at shorter distance of 2.4 cm and lower cooling rate of 600 cm³/min. Therefore, it is expected that the substrate has higher temperature at nearer distance from the rf coil and lower water cooling rate for the same gas composition. Fig. 1 shows that the effect of gas compositions on the substrate temperature where the other plasma parameters were fixed. It has been observed that the substrate temperature is gradually increased from 520 °C to 590 °C, depending on the acetylene gas pressure ratios. The interplay between the temperature and the gas composition might be caused by the effect of hydrogen species created from the dissociation of acetylene under plasma conditions. The mass difference between hydrogen, carbon and nitrogen plasma species and the ionization potential of atoms can play important role in the resulting plasma temperature (electron and ion temperature) which has a significant influence on the temperature of the substrate. In this case, the ionization potential of hydrogen is 6.4% lower than of that nitrogen and the light hydrogen ions are easily accelerate by the same rf plasma field compared to heavy ions of nitrogen. These ions itself contribute to the plasma heating due to secondary electrons generation by elastic collisions with the plasma species.

3.2. GDOS analysis

Fig. 2 shows the elemental concentration depth profiles of the nitrided sample and duplex treated samples at different acetylene gas pressure ratios of 10% and 30%. Nitrogen and the basic elements of the bulk substrate (Fe, Cr, and Ni) are detected in the investigated depth of the nitrided layer. Furthermore, little concentration of carbon is also detected along the same investigated depth. Detection of carbon is attributed to the hydrocarbon contamination which could be found in the reactor tube during nitriding process [30]. One can observe from the figure that the value of nitrogen concentration (≈ 16 at.%) is very high compared to that of carbon (≈ 0.8 at.%). The diffusion mechanism of nitrogen and/or carbon into austenitic stainless steel has been

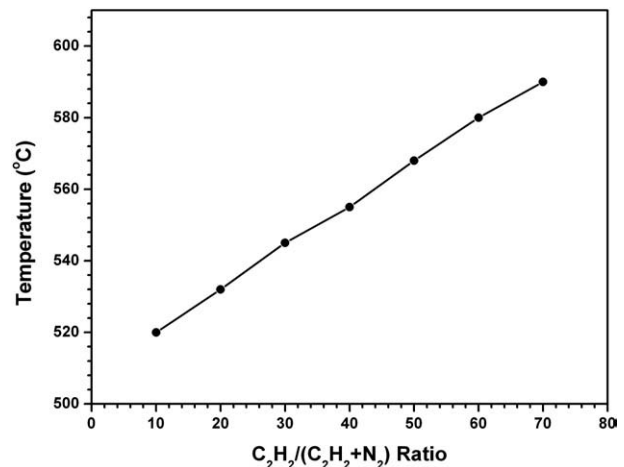


Fig. 1. Treatment temperature variation as a function of the acetylene partial pressure ratio ranges from 10% to 70%.

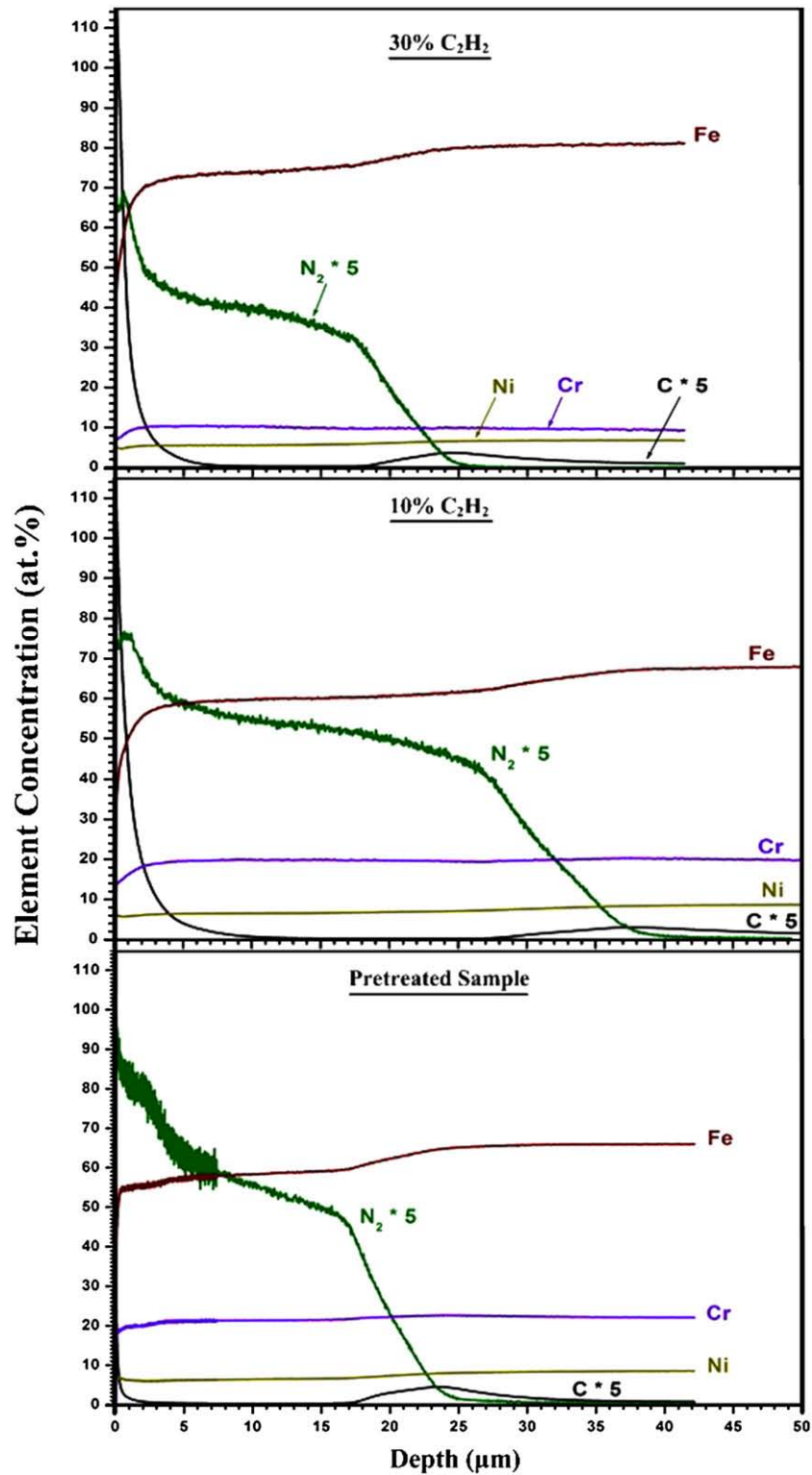


Fig. 2. Elemental concentration depth profiles of the nitrated sample and duplex treated samples at different acetylene partial pressure ratios of 10% and 30%.

discussed elsewhere [3,31–32]. After duplex treatment, carbon diffuses to a few micrometers into the surface of the nitrated substrate. On the contrary, no further diffusion of carbon is observed near the end of the nitrogen saturation region. The thick and dense nitrated layer impedes further nitrogen diffusion into austenitic stainless steel according to nitrogen/carbon diffusion mechanism [3,31–32]. Nearly the same distribution of nitrogen has been observed after duplex treatment process at different C_2H_2/N_2 gas composition.

It has been quantified that the concentration of nitrogen has a maximum value of approximately 12 at.% along the saturation region of the nitrated layer. Moreover, the thickness of the duplex treated layer gradually increases with increasing the acetylene pressure ratio and reaches a maximum value of 34.3 μm at 70%. This value equals 1.72 times of the nitrated layer thickness treated in the absence of acetylene. The thickness increment is ascribed to the further diffusion of carbon and nitrogen species into the surface of the nitrated layer

towards the bulk substrate. Furthermore, the high substrate temperature of carbonitriding causes a partial decomposition of some nitrated phases (as we will see in XRD analysis). The excess of free nitrogen atoms, after decomposition, can be diffused beneath the nitrated layer towards the bulk substrate. Temperature gradient and nitrogen/carbon concentration gradient enhance nitrogen diffusion acceleration process beneath the nitrated layer [30].

3.3. Microstructure analysis

The XRD patterns of untreated sample, nitrated sample, and duplex treated samples at different acetylene gas pressure ratios (10%, 30%, 50%, and 70%) are shown in Fig. 3. The obvious microstructure of the untreated sample is related to austenitic stainless steel (γ -phase). An overlapping between the peaks of γ_N , γ_C and γ -austenite phases is expected whereas the lattice expansion has a small value. However, the unchanged intensity of this phase in all duplex treated samples and the thick modified layer exclude the probability that the phase might be γ -austenite. Therefore, nitrogen expanded austenitic stainless steel (γ_N) with low lattice expansion up to 2.6% is the most feasible. It has been found that the lattice expansion of γ_N -phase is processing temperature dependent; a lattice expansion of 13% and 6.7% were detected at temperatures of 400 °C and 465 °C, respectively [33,34]. The low expansion of γ_N , in this work, is attributed to low nitrogen content inserted into austenitic stainless steel at relatively high processing temperature. Moreover, most of nitrogen content is dissipated in growing other nitrated phases, γ -Fe₄N and CrN, which are also found in the spectra. It is evident that the residual oxygen partial pressure is not sufficient to form oxynitride or metallic oxide phases during nitriding at treatment temperature of 450 °C. In previous work, the oxynitride phase of NO_x is formed at higher oxygen partial pressure, reducing the metallic nitride phases [35]. Furthermore, the existence of active hydrogen species, could remove the residual oxide layer chemically from the nitrated surface during carbonitriding.

The XRD pattern of duplex treated layers shows additional iron carbide phase of Fe₃C. Its intensity seems to be has a maximum value at 30% acetylene partial pressure ratio. Moreover, the intensity of the γ -Fe₄N phase decreases dramatically with increasing the acetylene partial pressure ratio and completely disappears at 70%. This is attributed to the gradual increase of the carbonitrided thickness layer. Moreover, a deposition of amorphous film on the nitrated layer, at high carbon content, decreases the intensity of peaks yield from nitrated phases. Furthermore, at high carbon content, the relatively high treatment temperature partially leads to phase decomposition into γ -Fe. The γ -Fe₄N phase starts to decompose into γ -Fe at 540 °C [36–37]. It is well known that both of CrC and CrN have the same crystal structure of face centered cubic and nearly similar lattice constant [3]. However, it is difficult to detect CrC phase where the Cr atoms are chemically bonded with the nitrogen atoms to form CrN during pretreated process of nitriding.

3.4. Surface morphology

The surface morphology of the nitrated and duplex treated samples at different acetylene gas pressure ratios of 10%, 30%, and 50% is displayed in Fig. 4. The surface of the nitrated and carbonitrided samples are characterized by non-uniform large grains with thin boundaries. Fine precipitations are observed on the carbonitrided surface which was treated at 30% acetylene partial pressure ratio. These fine precipitations can be ascribed to amorphous carbonitride film which is difficult to detect by X-ray diffraction. It has been observed that the grains are randomly packed, forming a porous microstructure. Furthermore, many cavities are observed in the surface of the duplex treated samples, depending on the gas composition. Moreover, a slipping between closest layers on the

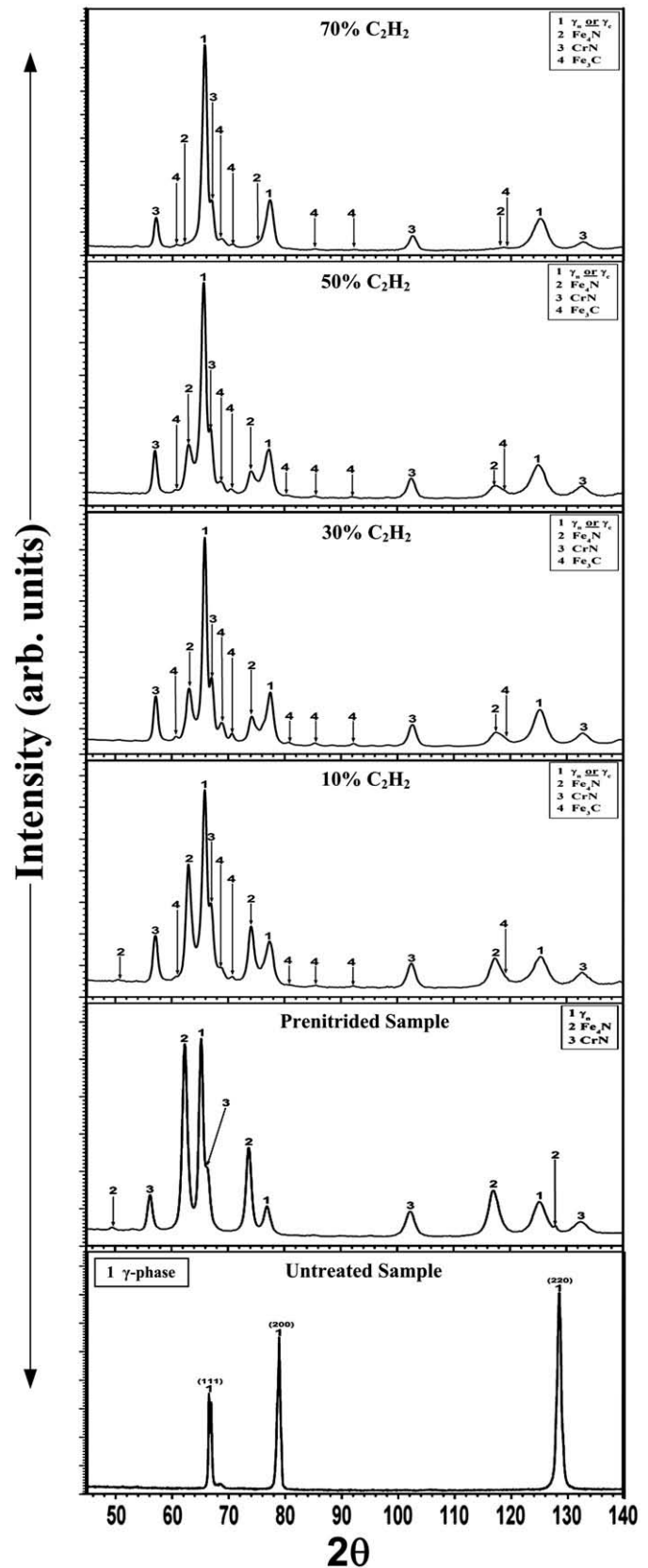


Fig. 3. X-ray diffraction patterns of untreated sample, nitrated sample and duplex treated samples at different acetylene partial pressure ratios (10%, 30%, 50%, and 70%).

surface of the duplex treated samples is appeared in the surface morphological micrographs. This slipping can be ascribed to the high nitrogen/carbon concentration in some regions compared to others,

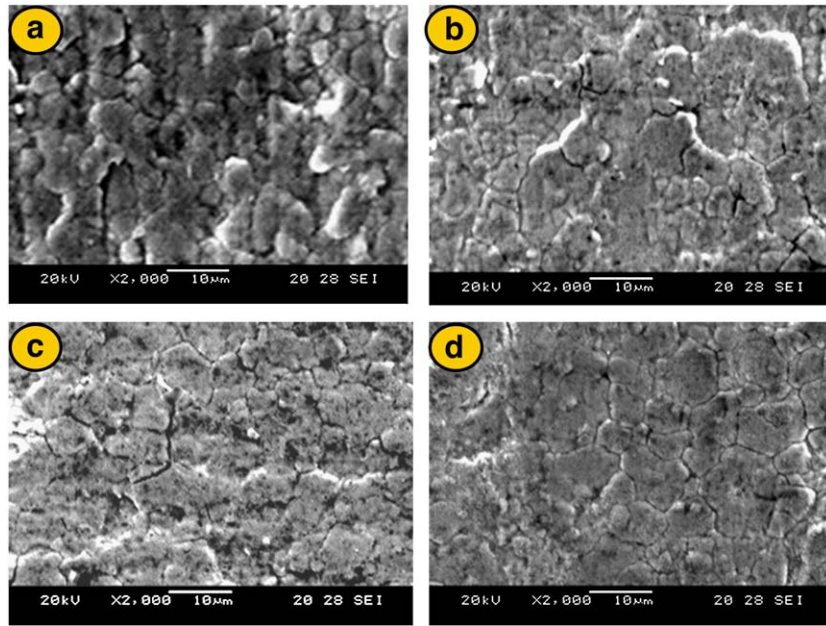


Fig. 4. SEM micrograph of (a) the nitrided sample and duplex treated samples at different acetylene partial pressure ratios: (b) 10%, (c) 30%, and (d) 50%.

leading to an expansion in these regions. The expanded regions affect on the adjacent regions, creating a compressive stresses which leads to a plastic deformation in the surface [38].

3.5. Cross-section morphological analysis

SEM micrographs of the cross-section of nitrided sample and duplex treated samples carbonitrided at different acetylene partial pressure ratios of 10%, 30%, and 60% are shown in Fig. 5. The nitrided sample shows a modified layer with a thickness of approximately 20 μm. It appears uniform with a sharp edge and the diffusion zone has a little change as shown in Fig. 5(a). The relatively thick modified layer has been formed without removing the native surface oxide layer, demonstrating that nitriding and carbonitriding of stainless steel at these treatment rf plasma conditions is achievable [3]. Figueroa and co-authors have found that a small potential barrier,

which creates as a result of small native oxide layer, would not forbidden nitrogen diffuses in the bulk and a relatively high thickness of modified layer occurs [35]. In contradiction to these results, other research workers have showed that a sputter removal of the surface oxide layer is required for successful nitriding of stainless steel by implantation at low energy (1 keV) and at lower temperature of 300 °C [11].

The duplex treated layer consists of two sublayers; carbonitrided layer is created on the nitrided one. However, the carbonitrided sample treated at acetylene partial pressure ratio of 60% has only a deposited layer of approximately 2 μm as shown in Fig. 4(d). This revealed that the duplex treatment mechanism of completely surface modification is convenient to apply until acetylene partial pressure ratio reaches 60%. At this point of high carbon content, the surface treatment mechanism is accompanied by the deposition mechanism which produces amorphous carbonitride film (2 μm) deposited on the

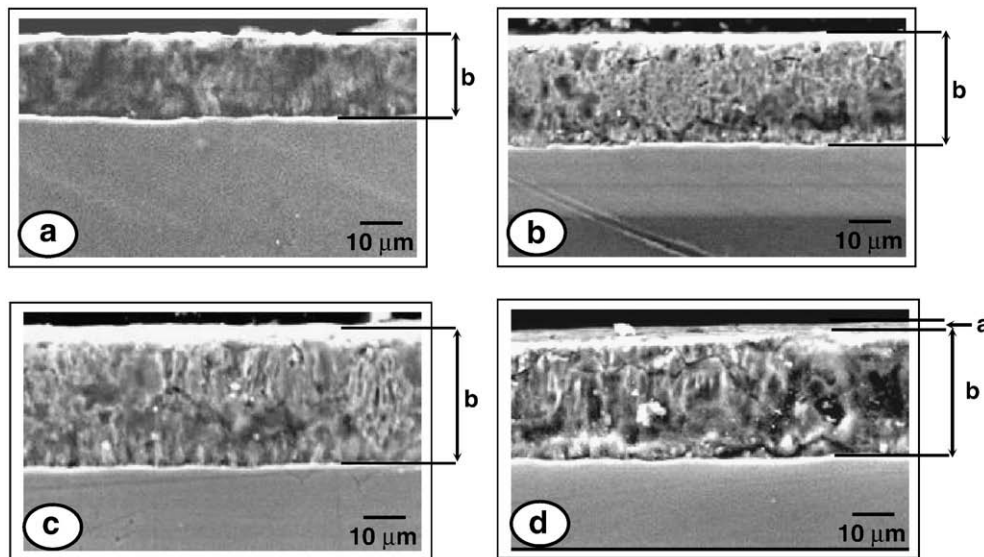


Fig. 5. SEM of cross-section for (a) nitrided sample and duplex treated samples at different acetylene partial pressure ratios: (b) 10%, (c) 30% and (d) 60%. a and b symbols refer to the carbonitrided and nitrided layer, respectively.

carbonitrided layer (30.9 μm). It has been found that the thickness of the treated layer increases gradually with increasing the acetylene partial pressure ratio, which in turn increases the treatment temperature as shown in Fig. 6.

3.6. Microhardness

Fig. 7 shows the surface microhardness value, measured at an applied load of 100 g, of duplex treated samples prepared at different acetylene partial pressure ratios. The surface microhardness value of the nitrided sample was measured to be 1051 HV0.1, which equals 4.8 times the associated value of the untreated substrate (220 HV0.1). The surface hardness improvement of the nitrided layer in comparison with the untreated substrate is attributed to the formation of hard phases of $\gamma\text{-Fe}_4\text{N}$ and nitrogen expanded austenitic stainless steel (γ_{N}). It has been found that the surface hardness of all duplex treated samples is improved with respect of the hardness of the nitrided one. The formation of carbide phases (carbon expanded austenitic stainless steel (γ_{C}) and Fe_3C) could be accountable about the variations in the surface microhardness after carbonitriding for different gas pressure ratios.

The surface hardness of the duplex treated samples significantly increases with increasing the acetylene partial pressure ratio up to 20%. After that it decreases gradually with increasing the acetylene partial pressure ratio up to 70%. It has a maximum value of 1356 ± 12 HV0.1 at acetylene partial pressure ratio of 20% which equals approximately 6.16 and 1.29 times relative to the associated values of the untreated substrate and the nitrided sample, respectively. The present duplex treatment technique achieves high surface hardness compared to other duplex treatment techniques [24,39].

The higher surface microhardness achieved for duplex treated layer at acetylene partial pressure ratio of 20% has been ascribed to the combine of hard nitride phases of $\gamma\text{-Fe}_4\text{N}$, γ_{N} and CrN with carbides phases of Fe_3C and γ_{C} . These phases are added further internal stress to the surface of the treated layer. Furthermore, the expected precipitation of small amount of amorphous phase in the polycrystalline matrix could be another contributing factor. However, with increasing the acetylene partial pressure ratio to higher values, some of the nitride phases gradually decompose at relatively high temperature, releasing the internal stress. It was found previously that, with increasing carbon content, the portion of the $\gamma\text{-Fe}_4\text{N}$ -phase and γ_{N} -phase are decreased and the portion of γ_{C} -phase is increased with the formation of amorphous layer [40]. It's known that the γ_{N} -phase is harder than the γ_{C} -phase [41]. Therefore, the sample treated at high

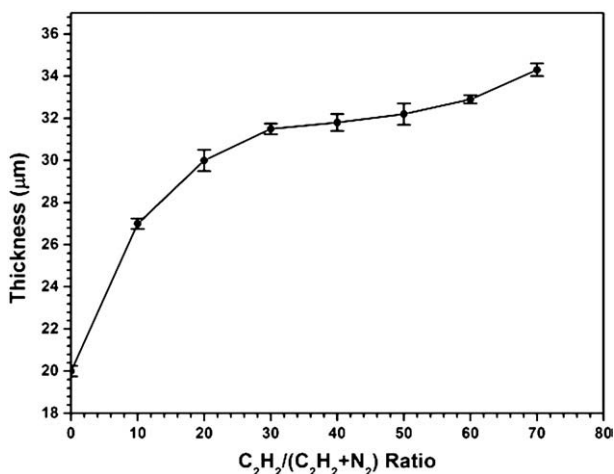


Fig. 6. Treated layer thickness measured by Vickers microhardness tester versus acetylene partial pressure ratio; the thickness of the treated layer at 0% acetylene partial pressure ratio refers to the nitrided sample.

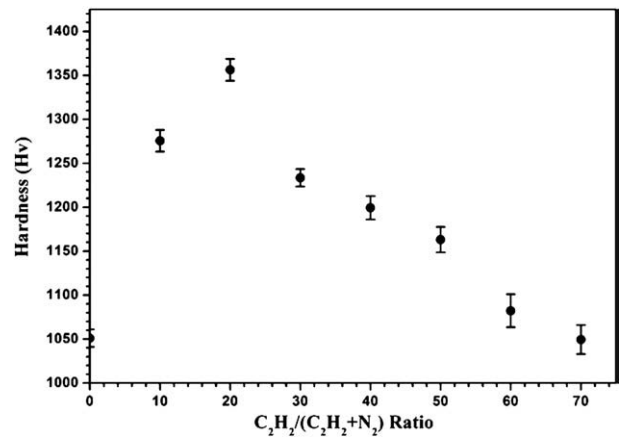


Fig. 7. Vickers surface microhardness of the duplex treated samples versus acetylene partial pressure ratios.

carbon content exhibit lower microhardness values compared to that treated at lower carbon content which is agreed well with another published data [3].

4. Conclusion

The results we obtained show that the surface modification of carbonitriding using rf plasma inductively coupled technique is succeeded to improve the surface hardness of the nitrided austenitic stainless steel. The maximum value of the surface microhardness obtained on the duplex treated sample is 1356 ± 12 HV0.1, which equals 1.29 times compared to that of the nitrided sample. During carbonitriding process, more carbon and nitrogen species diffuses into the surface of the nitrided substrate. Moreover, the relative increase of the substrate temperature of carbonitriding enhances the diffusion process of the reactive species. At high carbon content, a deposited layer of approximately 2 μm is formed on the surface of the duplex treated sample. The thickness of the duplex treated layer is gas composition dependent; and it has a maximum value of approximately 34.3 μm at 70% acetylene partial pressure ratio. The carbonitriding process has some affects on the microstructure of the treated samples at different gas compositions.

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