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Influence of Surface Nano/Ultrafine Structure Achieved by Deep Rolling Process on Plasma Nitriding And Tribological Properties of the AISI 316L Stainless Steel

A. Jafari Tadi*, S. R. Hosseini and M. Naderi Semiromi

* *abdolrezajafari68@gmail.com*

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Department of Materials Engineering, Maleke-Ashtar University of Technology, Isfahan. Iran.

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Abstract: *Influence of formation of surface nano/ultrafine structure using deep rolling on plasma nitriding and tribological properties of the AISI 316L stainless steel was investigated. Initially, the deep rolling process was carried out on the bar-shaped specimensat 15 cycles with 0.2 mm/s longitudinal rate and 22.4 rpm bar rotation.Then, plasma nitriding treatment was applied on the as-received and deep rolled kind sat 450 °C and H2-25%Vol. N2 gas mixture for 5h. Surface micro-hardness and un-lubricated pin-on-ring sliding wear tests were carried out on the as-received, deep rolled, plasma nitrided and deep rolled-plasma nitrided kinds. Results revealed that deep rolled-plasma nitrided kind is shown the highest wear resistance than the others, due to the further increased surface hardness achieved via the combined process.*

Keywords: *Austenitic Stainless Steel, Deep Rolling, Plasma Nitriding, Wear Resistance.*

1. INTRODUCTION

Austenitic stainless steels (ASSs) have an excellent corrosion resistance and good formability [1, 2]; but low hardness and weak wear resistance, which has been led to the usage decrease at tribological applications. Several techniques such as grain refinement and alteration of chemical composition have so far been represented to solve this problem. Grain refinement is a well-known method to increase of hardness and wear resistance of metals such as ASSs. Grain refinement can be applied in surface regions via severe plastic deformation [3] particular surface mechanical treatments (SMTs), such as shot peening [4], surface mechanical attritioning treatment (SMAT) [5, 6], ultrasonic shot peening [7, 8], laser shock peening [9, 10], surface burnishing [11, 12] and deep rolling [13, 14]. These treatments can induce severe plastic deformation in surface layers, leading to the work hardening, structural alterations and induction of residual stresses in deformed zones. Formation of nano/ultrafine grains, mechanical twinnings and strain induced martensite are some structural changes observed in the surface mechanical treated ASSs. Hence, hardness and ductility of surface layers as well as wear resistance may be increased by SMTs. More over the grain refinement, chemical composition alteration is another effective way to improve mechanical properties of ASSs. Change and modification of chemical composition in surface layers can be resulted in increasing of wear resistance. This purpose has been achieved by very techniques, such as carburizing [15, 16], boronizing [17, 18] and nitriding [19, 20]. In these methods, the change in near-surface chemical composition is caused to the induction of residual stresses and formation of hard phases. Nitriding is a well-known diffusional treatment that has so far been used to increase the surface hardness and wear resistance of ASSs. By applying the nitriding on an ASS, an expanded austenitic phase "γN" and nitrided compounds are formed on the surface [21], resulting in increased surface hardness and wear resistance. However, ASSs are recognized as materials that are hardly nitrided due to low nitrogen diffusion into the austenite. In addition to the improve of surface hardness and wear resistance, SMTs also change the surface conditions; a suitable way to enhance the nitrogen diffusion into austenitic matrix by increased crystalline defects that raise diffusion rate of elements such as nitrogen, meaning a thicker and harder nitrided layer formed on the

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ASS surface.

The experimental studies that have so far been reported by investigators about effects of SMTs and nitriding on wear resistance of ASSs are contradicting. Sun. [22] showed that surface nanostructure formed on the surface mechanical attrition treated AISI 304 stainless steel can improve its wear resistance under the un-lubricated and lubricated sliding conditions. Due to the high surface roughness of the treated specimen, wear resistance was found better in lubricated conditions than the un-lubricated one. Wang et al. [23] and Ma et al. [24] exhibited that fine particle bombardment and sandblasting treatments can increase wear resistance of ASS under the un-lubricated sliding conditions. Hashemi et al. [25] studied the wear resistance of shot peened and gas nitrided 316L stainless steel and found that shot peening and gas nitriding can improve the wear resistance of the steel. Also, wear resistance of shot peened–gas nitrided 316L stainless steel was detected further than the individually shot peened or nitrided kinds. Lim et al [26] studied enhancement the abrasion resistance of duplex stainless steel by laser shock peening. It is shown that abrasion resistance of laser shock peened duplex stainless steel is better than the untreated one. Lin et al. [27] studied the wear resistance of the plasma nitrided AISI 321 stainless steel and found that wear resistance of treated sample is better than the untreated one. In addition, it was seen that surface mechanical attrition treatment combined with plasma nitriding induces an increased wear resistance in the 321 stainless steel.

The aim of the present work is the evaluation of the effects of deep rolling on plasma nitriding and tribological properties of the AISI316L austenitic stainless steel. Initially, deep rolling process was applied on the bar-shaped 316L stainless steel specimens. Then, plasma nitriding was accomplished on the deep rolled and as-received parts. In addition, un-lubricated sliding wear tests were conducted on the as-received, deep rolling, plasma nitriding and deep rolled-plasma nitrided processed specimens.

2. EXPERIMENTAL

2. 1. Material and Experimental Procedure

Experiments were carried out on the bar-shaped specimens of the AISI 316L austenitic stainless steel with 30 mm diameter, 210±5 $HV_{0.025}$ average hardness and chemical composition (wt. %): 0.03 C, 17.5 Cr, 10.5 Ni, 2.15 Mo, 1.81 Mn, 0.5 Si, 0.034 S, 0.046 P and balance Fe. The as-received steel (UT) was a hot-rolled material with full-austenitic microstructure and an average grain size of 25 μm as shown in Fig. 1.

Cyclical deep cold rolling process named DR was carried out using a ball-point rolling device equipped by WC ball at 15 cycles on the bar-

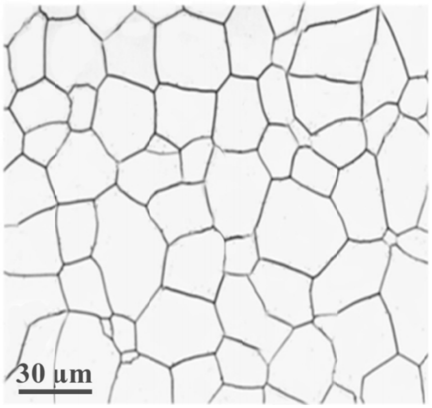

Fig. 1. An optical micrograph showing the microstructure of the as-received AISI 316L stainless steel (UT).

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Table 1. Processing conditions and nominal properties of the deep rolling processed parts (DR)

Parameter	Device longitudinal rate (mm/s)	Bar rotation (rpm)	Device feed, each cycle (μm)	No. passes	Surf. Roughness (μm)	Grain size, to 40 μm depth (nm)
value	0.2	22.4	-	15	0.01	200

shaped specimens that led to the 110 μm diameter reduction of bar, totally. Multiple cycles were selected to create a severe plastic deformation on the material surface and therefore induction an appropriate grain refinement in the near-surface regions. Some processing conditions and nominal properties of the deep rolling processed parts (DR) are listed in Table 1. According to this table, the microstructure of the deep rolled material into 40 μm depth evaluated by FESEM has been consisted from ultrafine grains with 200 nm average size. Therefore, performed process has induced a relative-appropriate grain refinement in the near-surface regions, which affect the subsequent treatments such as nitriding, agreeing with similar works [25, 27].

Prior to the nitriding, the UT and DR specimens were cleaned by acetone solution for 15 minutes and then mounted on the machine sample holder for ion sputtering by H_2-50 % Vol. N_2 gas mixture at 110 °C and 0.4 Torr pressure for 60 min and afterwards heating up to 450 °C. Plasma nitriding was performed by plasma nitriding equipment manufactured by Iranian Company of Plasma fanavar Amin (http://plasmafanavar.com) on the as-received (UT) and deep rolling processed (DR) kinds at 450 °C and H_2-25% Vol. N_2 gas mixture with 5 Torr pressure for 5 h, which resulted in production of plasma nitrided (PN) and deep rolled-plasma nitrided (DPN) kinds. According to the studies, the plasma nitriding at 450 °C for 5 h can produce a relatively-thick nitrided layer with good hardness and saved corrosion resistance [5]. Hence, the treatment conditions are appropriate selections and the AISI 316L stainless steel with good wear resistance and sufficient corrosion properties may be achieved.

2. 2. Characterization of the Surface

A PME3 optical (OM), ZEISS S416 field emission scanning electron (FESEM) and Philips XL30 scanning electron (SEM) microscopes were employed to observe the microstructure of the UT, DR, PN and DPN processed kinds. The cross-section of the samples were wet ground by SiC grinding papers and then mechanically polished in order to achieve a mirror-liked surface and finally etched by "10 HCL+10 HNO_3+10 acetic acid+2 drop glycerol" etchant. Phasic characterisation was done using X-ray diffractometer (MPD-X′PERT) conducted using Cu Kα radiation with 1.542515 A° wave length at scanning range from 30 to 100°, 0.05° step size and scan step time of 1 sec. Un-lubricated wear tests were carried out using a pin-on-ring tribometer equipped with cylindrical-shaped WC pin (Φ 5 mm diameter×150 mm length) at 35 % relative humidity, ambient temperature, 52 rpm work-piece rotation and 60 N contact load for 1000 m sliding distance. Profile, morphology and hardness of worn track were respectively determined using SJ210 stylus profilometer, SEM and micro-indenter at 25 gf load.

3. RESULTS AND DISCUSSION

3. 1. Microstructure and Hardness

X-ray patterns of the as-received (UT), deep rolled (DR), plasma nitrided (PN) and deep rolled-plasma nitrided (DPN) kinds are shown in Fig. 2. As shown, the deep rolling has been resulted in ά-martensitic phasic transformation (measured about 8 % Vol. by feritoscope) in the austenitic matrix; 92 % Vol. retained austenite. Therefore, near-surface regions at the deep rolling processed status is shown a martensitic-austenitic mixture with increased density of crystalline defects consisting of ultrafine (200 nm size) austenitic and martensitic grains, agreeing with the similar previous works [3, 5, 10, 13, 14]. According to the later works, a composite of

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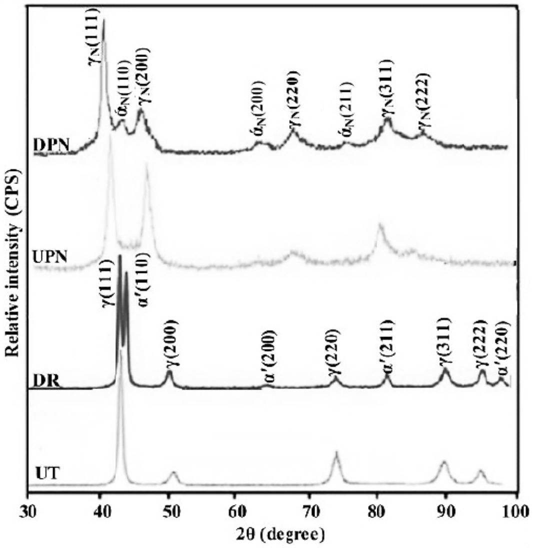

Fig. 2. X-ray diffraction patternsat the different conditions; UT=as-received, DR=deep rolled, UPN=plasma nitrided and DPN=deep rolled-plasma nitrided.

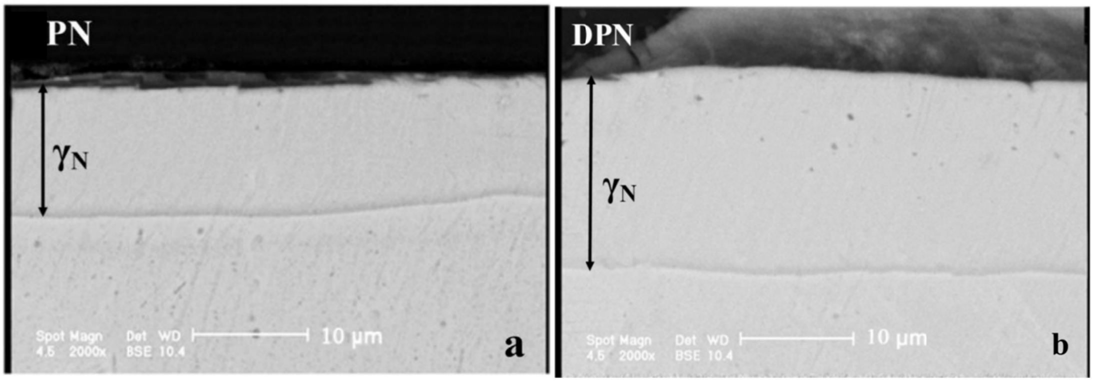

Fig. 3. SEM micrograph from the top-surface microstructure related to the UN and DPN kinds.

equal-sized austenitic and martensitic grains with nano/ultrafine scale is observed in a surface mechanical treated structure of such austenitic stainless steels. Both the nitriding processes, PN and DPN, have transformed the austenite and martensite phases to the expanded austenite (γN) phase with relatively-high peaks and the expanded martensite (άN) with low-height of peaks that expansion is achieved via N enrichment [25, 27].

Cross-section micrographs of the plasma nitriding processed kinds have been shown in Fig. 3. Nitriding is a diffusional process and crystalline defects such as twinning boundaries and grain boundaries are suitable paths for rapid diffusion of nitrogen that led to the thicker and harder nitrided layer [5, 25, 27]. As shown, relatively uniform nitrided layers with 11 and 18 μm thicknesses have respectively been formed on the PN and DPN surfaces, which means that increased density of diffusional rapid channels such as grain boundary, twinning boundary and dislocations at the deep rolling processed conditions has been led to the further diffusion depth of nitrogen and thus thicker nitrided layer in the DPN part.

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Table 2. Surface micro-hardness (HV$_{0.025}$) at the different processing conditions.

Process	UN	DR	PN	DPN
Surface micro-hardness	210±5	450±7	650±8	1150±10

Surface micro-hardness at the different processing conditions is listed in Table 2. As shown, surface hardness has respectively been increased from 210 to 450, 650 and 1150 HV$_{0.025}$ after DR, PN and DPN processes. Moreover, hardness enhancement using the plasma nitriding (PN) is further than that of the deep rolling (DR). Therefore, conducting of the deep rolling prior to the nitriding (combined status) is led to the form of the thicker nitrided layer with more hardness on the AISI 316L stainless steel, agreed to the several previous works [5, 10, 25, 27] that mechanical surface treatments such as shot peening and mechanical attrition combined with plasma nitriding treatment induce similar results in such austenitic stainless steels.

3. 2. Wear Properties

Mass loss plots related to the UT, DR, PN and DPN kinds worn by 60 N load are given in Fig. 4. According to the figure, UT has the highest mass loss, while DPN lowest value. This is due to the higher surface hardness in the DPN that leads to its further wear resistance compared to UT with lowest hardness and thus weak resistance against wear, agreeing to Refs [25, 27]. According to later works, increase of surface hardness leads to the wear resistance enhancement. Therefore, it can be told, performing of deep rolling combined with nitriding improves significantly wear resistance of the AISI 316L stainless steel compared with plasma nitriding and deep rolling processes conducted individually with lower enhancement of wear resistance, due to the lower increase of surface hardness. The relatively-equal mass loss values of the PN and DPN kinds reveal that the nitriding has the major role in the tribological behavior.

Friction curves of the selected specimens recorded during the applied wear tests are shown in Figs. 5 & 6. Moreover, average value of static friction coefficient and its oscillation at the

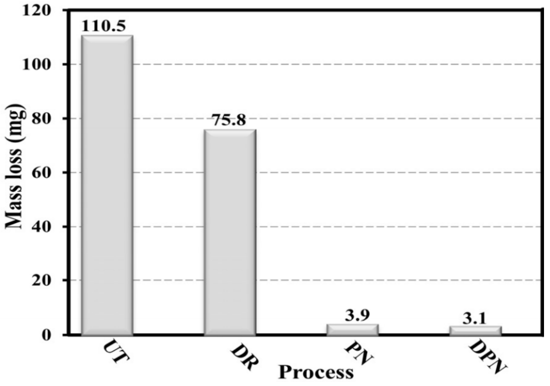

Fig. 4. Mass loss of the UT, DR, PN and DPN processed kinds after the wear testing atthe mentioned conditions.

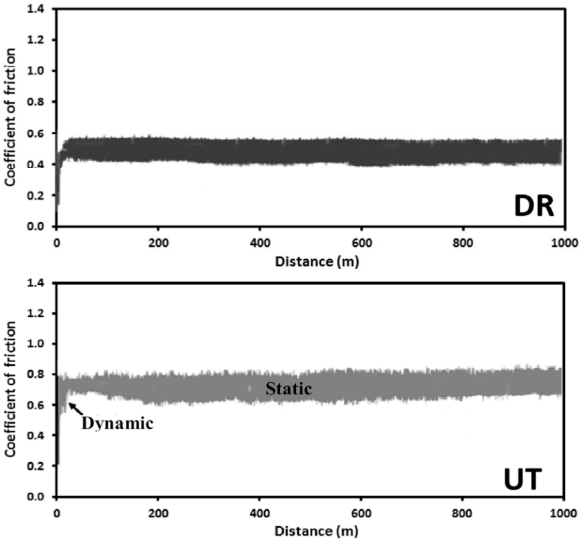

Fig. 5. Friction curves of the UT and DR processed specimens after wear testing.

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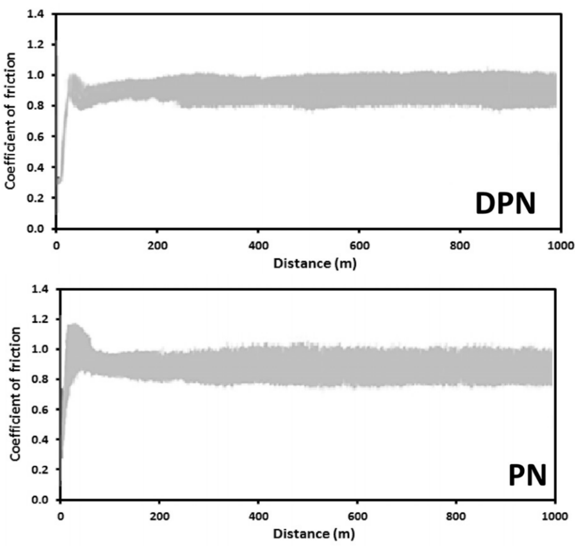

Fig. 6. Friction curves of the PN and DPN processed specimens after wear testing.

applied conditions are listed in Table 3. It is observed that lowest coefficient of friction is related to the deep rolled (DR) kind than that of the others. While, the PN and DPN kinds have the highest friction coefficient fixed at an equal value; 0.9 value. As described in the Archard relationship, surface roughness has the furthest influence on the friction coefficient [28]. Therefore, lower friction coefficient of the DR is due to its lower roughness (0.01 μm), a prevalent property in deep rolled work-pieces [11, 12]. While, increased friction coefficient in the nitrided kinds, PN and DPN, is due to the further surface roughness measured about 0.1 μm for both samples raised by plasma nitriding, agreeing to several previous works [29] that has exhibited this treatment increases surface roughness as well as friction coefficient of materials. As shown in Table 3, oscillation of friction coefficient for the nitrided samples is highest, probably because of high adhesion between the nitrided surface and slider pin, confirming that maximum and minimum values of friction coefficient at each sample are respectively related to wee stopping and easy sliding of pin on nitrided surface, named as "stick-slip" phenomenon [29].

Typical wear track profiles related to the worn surfaces have been illustrated in Figs. 7 & 8. It is shown that surface with higher hardness has a wear track with lower width and depth. Wear tracks produced on the UT and DR surfaces have a similar feature excepting material pile up at two edges of wear track on the UT one, which indicates the plastic deformation during wear testing. Predominant feature of the worn nitrided surfaces is low-depth wear track contained deep depressions produced due to the high adhesion of the nitrided surfaces and thus local adhesion between the surface and pin slider that is resulted in material improve and deep depressions.

Morphology of the worn track at the various conditions determined by SEM is shown in Figs. 9 & 10. Plastic deformation, adhesion and abrasion are predominant wear mechanisms in the selected samples. Plastic deformation and abrasion have the predominant role in the UN and DR kinds, while abrasion and adhesion are predominant in both the nitrided ones. Moreover, surface micro-hardness measured before and after wear testing are listed in Table 4. As shown, surface hardness of the UN and DR kinds has been raised after wear testing, higher value in the UN than the other one. This is due to work-hardening effect as a result of surface plastic deformation during sliding of pin on the sample

Table 3. Average volume and oscillation of static friction coefficient for various samples.

Sample	**UN**	**DR**	**PN**	**DPN**
Average of static friction coefficient	0.75	0.5	0.4	0.3
Oscillation of static friction coefficient	0.125	0.15	0.45	0.55

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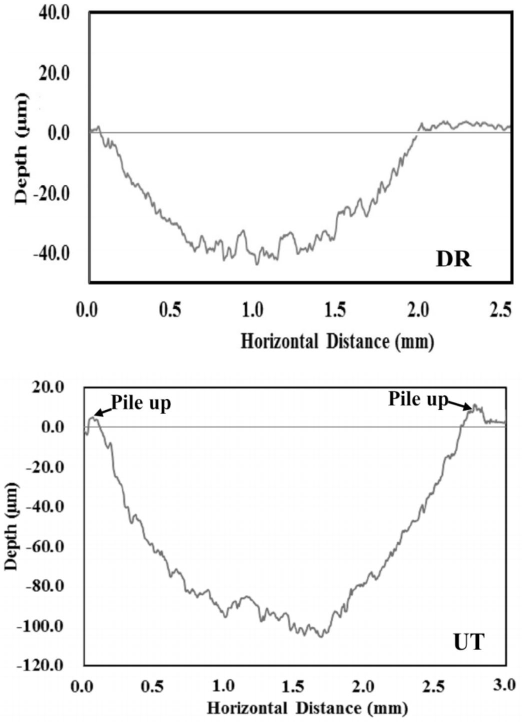

Fig. 7. Typical wear track profiles of the UT and DR processed specimens after the wear testing.

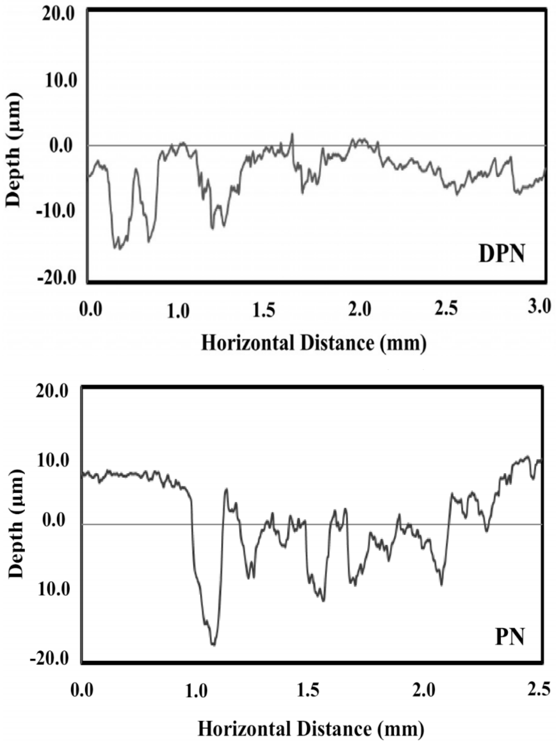

Fig. 8. Typical wear track profiles of the PN and DPN processed specimens after the wear testing.

Table 4. Surface micro-hardness ($HV_{0.025}$) before and after wear testing.

	UN	DR	PN	DPN
Before wear	210	450	650	1150
After wear	460	420	590	820

surface, which led to the increased surface hardness. It is noticeable that for the deep rolled sample with higher internal stored energy, grains growth and softening may be occurred after high sliding distances, resulting in lower surface hardness than the UN. On the contrary, surface hardness of the worn nitrided samples is lower than the un-worn ones. It should be told, nitrided zone with lower nitrogen has a less hardness [29, 30]. Therefore, since top-surface layer with higher nitrogen is eliminated during wear test, a material with lower nitrogen and thus less hardness remains.

4. CONCLUSIONS

1. The performed deep rolling produced a surface ultrafine-grained layer with increased hardness from 210 to 450 $HV_{0.025}$.
2. Moreover, the plasma nitriding at the used conditions generated a hard nitrided layer with 440 $HV_{0.025}$ hardness increase on the top-surface layer.
3. Deep rolling process combined with plasma nitriding created a thicker nitrided layer with more hardness due to the increased

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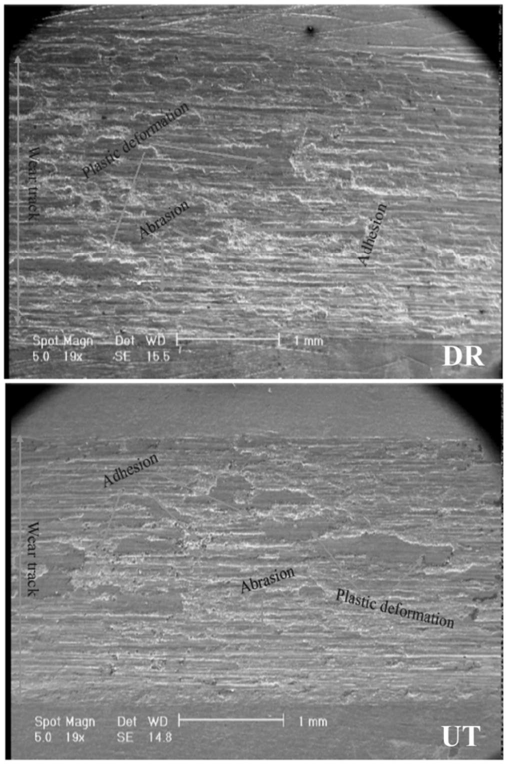

Fig. 9. Morphology of wear track related to the UT and DR samples after the wear testing.

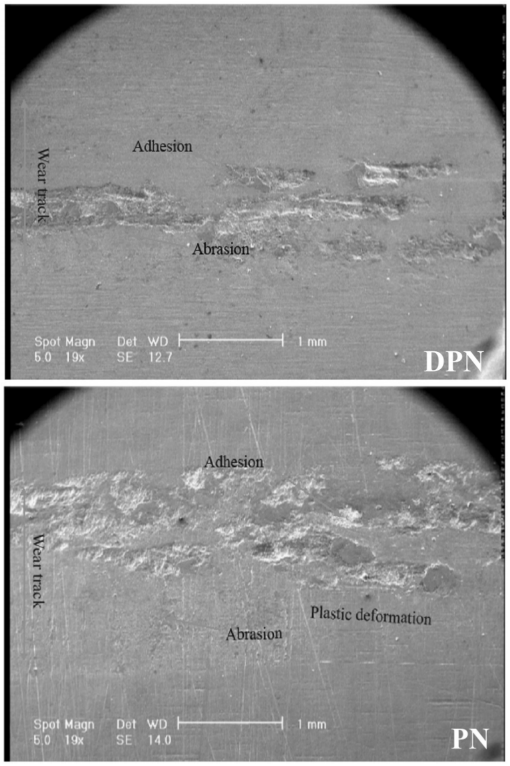

Fig. 10. Morphology of wear track related to the PN and DPN samples after the wear testing.

diffusional rapid channels.

4. Un-lubricated wear resistance was improved by both the processes particular the plasma nitriding.
5. Wear resistance at the combined status was found better than the individually processed conditions
6. During un-lubricated sliding wear tests, the wear mechanisms consisting of plastic deformation, adhesion and abrasion were caused to the material failure at the different processing conditions.

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