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Plasma immersion ion implantation on 15-5PH stainless steel: influence on fatigue strength and wear resistance

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Abstract. Surface improvement in steels is of great interest for applications in industry. The aim of this investigation is to study the effect of nitrogen ion implantation on the axial fatigue strength and wear resistance of 15-5 PH stainless steel. It is well known that electroplated coatings, which are used to improve abrasive wear and corrosion properties, affects negatively the fatigue strength. It is also important to consider requirements to reduce the use of coated materials with electroplated chromium and cadmium, that produce waste, which is harmful to health and environment. The HVOF (High velocity oxygen fuel) process provides hardness, wear strength and higher fatigue resistance in comparison to electroplated chromium. Plasma immersion ion implantation has been used to enhance the hardness, wear, fatigue and corrosion properties of metals and alloys. In the present research the fatigue life increased twice for 15-5 PH three hours PIII treated in comparison to base material. From the abrasive wear tests a lower pin mass reduction was observed, associated to the superficial treatments. The improvement of fatigue and mechanical performance is attributed to a combination of nitrides phase structure and compressive residual stresses during the PIII treatment.

1. Introduction

Surface treatments in mechanical components are considered necessary to improve wear and corrosion properties of applied materials. It is well known that wear and corrosion processes on aircraft structures are controlled by electroplated chromium and cadmium, hard anodizing and, more recently, HVOF thermal spray coatings [1-3]. The decrease in fatigue strength of coated parts is associated to microcracks density, high tensile residual stresses and to the presence of high density of porous and oxide inclusions into the coating, that commonly forms during the process [4-5]. Compressive residual stresses induced in the surface layers of the material after controlled shot peening processes, delay nucleation and propagation of fatigue cracks, increasing materials performance [6-7]. Rotating bending fatigue experimental results indicate that electroplated aluminum and IVD aluminum coatings may replace cadmium electroplating on AISI 4340 steel [8].

The PIII process avoids common problems like microcracks and poor adhesion coating/substrate. The plasma immersion ion implantation and deposition (PIII D) technique was responsible for the good adhesion strength between the nitrogen doped diamond-like carbon (N-DLC) coating and NiTi substrate. The corrosion resistance of NiTi alloys was also improved [9]. The effect of temperature on the surface mechanical properties of H13 steel implanted with nitrogen ions using plasma immersion ion implantation was to improve hardness and wear resistance by increasing the PIII treatment



temperature. An opposite behavior was observed in the presence of carbon (C) ion [10]. Results were attributed to nitride formation and lattice expansion. A relatively constant concentration of nitrogen with depth was observed in experiments using AISI 304 stainless steel plates subjected to nitrogen ion implantations by plasma immersion [11]. Experiments performed with AISI 304 stainless steel treated by plasma immersion ion implantation showed that high nitrogen concentration and/or thicker layers improved hardness and wear resistance, whereas simultaneously reduced the surface fatigue strength [12].

Improvement in the mechanical behavior and in the rolling contact fatigue strength of nitrogen PIII treated AISI 52100 bearing steel was attributed to a combination of nitrides phase structure and compressive residual stresses during the process [13].

In the present research, fatigue and wear resistance of 15-5 PH stainless steel nitrogen PIII treated for one, two and three hours was evaluated Axial S-N curves were obtained in fatigue tests. In order to study the effect of residual stresses on the fatigue strength, the stress field was measured by an X-ray tensometry after the treatments. Scanning electron microscopy was used to investigate the fatigue crack initiation points. Wear resistance was evaluated using pin-on-disk apparatus.

2. Experimental procedures

2.1. Material and mechanical properties

The 15-5 PH martensitic stainless steel, which combines high mechanical strength and good corrosion resistance, has the chemical composition according to table 1.

Table 1, Chemical composition (wt%). 15-5 PH stainless steel.

C	Mn	P	S	Si	Cr	Ni	Mo	Nb	Cu
0.07	1.0	0.03	0.015	1.0	14.0 – 15.5	3.5 – 5.5	0.5	0.45	2.5 – 4.5
max	max	max	max	max			max	max	

The desired mechanical properties were obtained by a precipitation hardening treatment at 552°C for four hours. Ultimate tensile strength: 1125 MPa and Hardness: 39.5 HRc.

2.2. Plasma immersion ion implantation

A vacuum chamber composes the PIII system with a specimen holder, plasma source and a high voltage pulse modulator. The specimen to be treated is placed in the chamber in which a plasma containing the ions to be implanted is generated. To start the cleaning step with argon gas, the chamber was pumped to a base pressure of 2.0×10^{-2} mbar. The cleaning procedure was concluded in 15 minutes. The nitrogen implantation process was performed by applying negative high voltage pulses of 10.0kV, pulse length 50 μ s and bombardment frequency 1.5KHz. The average working temperature was 396°C and treatment times were chosen as one, two and three hours. The experimental setup is represented in figure 1.



Figure 1. PIII system experimental setup.

The PIII treated specimen surfaces were analyzed by atomic force microscopy. Nanohardness tests were performed with a nanointenter XP System, with 400mN maximum load. Distribution of indentations occurred in a matrix 5x5, separated by a distance equal to 50 μ m. To investigate the fatigue crack initiation in the specimen groups and possible correlation with treatment times, scanning electron microscopy was used. Vickers microhardness measurements were performed according to ASTM E384.

2.3. Axial fatigue tests

Fatigue tests were conducted to complete fracture or 10^6 load cycles, using a sinusoidal load of 10Hz frequency and load ratio $R=-1.0$ at room temperature. The test specimen used for the axial fatigue tests, in figure 2, was prepared according to standard ASTM E466.

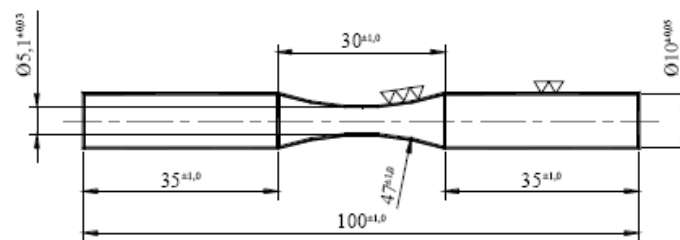


Figure 2. Axial fatigue test specimen.

Four groups of axial fatigue specimens were prepared to obtain S-N curves:

- 12 specimens/15-5 PH stainless steel;
- 12 specimens/15-5 PH stainless steel/ 1 hour PIII treated;
- 12 specimens/15-5 PH stainless steel/ 2 hours PIII treated;
- 12 specimens/15-5 PH stainless steel/ 3 hours PIII treated.

2.4 Residual stress measurement

The X-ray diffraction method was used to determine the residual stress field induced by the PIII treatments. The accuracy of the stress measurement was $\Delta\sigma=\pm 10$ MPa.

2.5 Abrasive wear test

Dry sliding wear tests were performed on 15-5PH (one, two and three hours PIII treatment)/aluminum-bronze 630 tribological pair, using a pin-on-disk equipment according to ASTM G99 standard. Tribological characterization was conducted with a normal force of 5N and a speed of 0.5m/s. Parameters were collected to measure mass loss of the metal pin and disk using an analytical balance. At every 400m route, specimens were cleaned in ultrasonic bath and weighted. The total distance for each tribological pair was 2000m.

3. Results and discussion

Residual stresses for base metal and 15-5PH stainless steel PIII treated for one, two and three hours are represented in table 2.

Table 2. 15-5 PH stainless steel. Residual stresses.

Specimen	Depth (mm)		
	0.00	0.01	0.1
Material	Stress (MPa)		
15-5 PH	+36	+110	+110
15-5 PH PIII/1H	-115	+30	+43
15-5 PH PIII/2H	-230	+12	+78
15-5 PH PIII/3H	-270	-60	+25

Tensile residual stresses were obtained for 15-5PH stainless steel at surface and 0.01mm/0.1mm depth, respectively 36MPa, 110MPa and 110MPa. Increase in compressive residual stresses at specimen surface occurred associated to increase in the PIII treatment times, respectively -115MPa, -230MPa and -270MPa for one, two and three hours treatments.

For one and two hours PIII treatment, tensile residual stresses are observed for 0.01mm and 0.1mm from surface, but still lower than residual stresses for base metal at the same depth. In the case three hours PIII treatment, -60MPa and 25MPa residual stresses are related to 0.01mm and 0.1mm, respectively.

The axial S-N curves for 15-5PH stainless steel base metal and PIII treated for one, two and three hours, are indicated in figure 3.

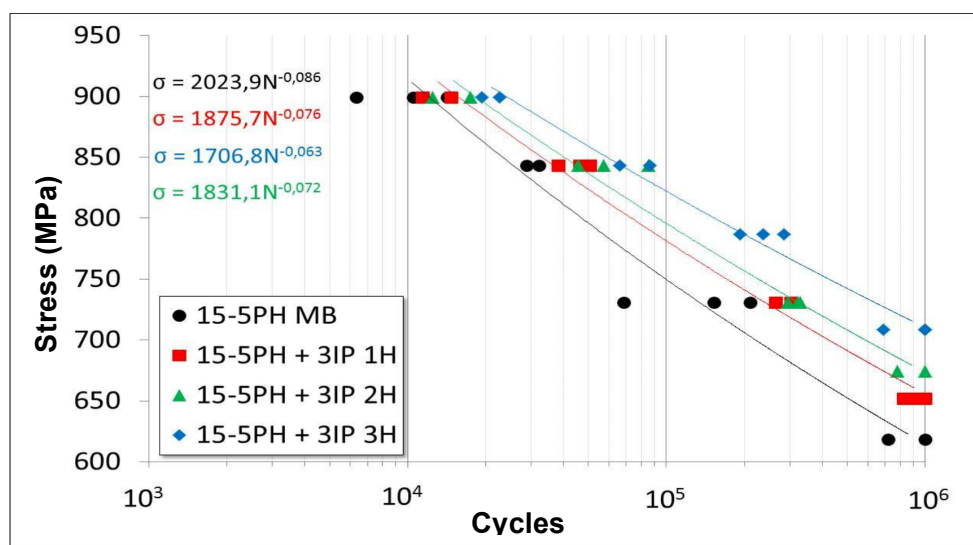


Figure 3. Axial S-N curves. Base metal and 15-5PH stainless steel PIII treated for one, two and three hours.

In table 3, the average number of cycles to failure for 15-5PH stainless steel and base metal PIII treated for one, two and three hours are N, N1, N2 and N3, respectively.

Table 3. Number of cycles to failure. 15-5PH stainless steel.

Cycles Stress (MPa)	15-5PH N	15-5PH/1H N1	15-5PH/2H N2	15-5PH/3H N3
900	10358	13046	14960	21029
843	30409	45021	62773	76029
787	-	-	-	237735
730	144017	283071	315348	-
710	-	-	-	688274
675	-	-	783129	-
650	-	857727	-	-
620	718405	-	-	-

According to figure 3 and table 3, the PIII treatment increased the low and high cycle axial fatigue resistance of 15-5PH stainless steel. Table 3 indicates, for maximum applied stress 900MPa, 10358, 13046, 14960 and 21029 average cycles to failure for 15-5PH stainless steel and base material PIII treated for one, two and three hours, respectively. An improvement in fatigue life twice for PIII/3H was observed in comparison to 15-5PH stainless steel. With respect to the high cycle fatigue regime, table 3 shows fatigue strength for number of cycles to failure near to 10^6 , equal to 620 MPa, 650 MPa, 675 MPa and 710 MPa for base material and 15-5PH PIII treated, respectively, for one, two and three hours. As the improvement in fatigue strength is correlated to the compressive residual stresses, which may avoid fatigue crack nucleation or even retard propagation, an increase in fatigue life is expected with higher compressive residual stresses from PIII treatments. According to table 2, higher compressive residual stresses at surface and 0.01mm depth were obtained for 15-5PH stainless steel PIII treated for three hours. Table 3 shows higher number of cycles to failure for this treatment condition. Improvement in the rolling contact fatigue of AISI 52100 bearing steel occurred by nitrogen plasma immersion ion implantation technique [13]. The maximum microhardness and the effective elastic modulus value also increased.

In table 4, increase in the fatigue strength related to the PIII treatments is represented by the ratios N1/N, N2/N and N3/N.

Table 4. Ratios Ni/N.

Ni/N Stress (MPa)	N1/N	N2/N1	N2/N	N3/N1	N3/N	N3/N2
900	1.26	1.15	1.44	1.61	2.03	1.4
843	1.48	1.39	2.06	1.69	2.5	1.21
730	1.96	1.11	2.19	-	-	-

From table 4, for maximum applied stresses 900MPa, 843MPa and 730MPa an increase in the ratio Ni/N is observed with increase in the PIII treatment times one, two and three hours. As an example, for 900MPa, values for Ni/N are 1.26, 1.44 and 2.03, respectively for 15-5PH PIII treated for one, two and three hours respectively. An improvement in Ni/N is observed with the increase in PIII treatment times. The same behavior is indicated in table 4 for 843MPa and 730MPa. The influence of maximum applied stress on Ni/N for each PIII treatment temperature studied is also represented in table 4. In case one hour PIII treatment, values for N1/N are 1.26, 1.48 and 1.96 for 900MPa, 843MPa and 730MPa, respectively. The same tendency occurs for N2/N and N3/N. The conclusion is that improvement in Ni/N is also associated to the decrease in maximum applied stress, from low to high cycle fatigue.

SEM photomicrographs for 15-5 PH stainless steel and base metal PIII treated for 1, 2 and 3 hours are shown in figures 4a, 4b, 4c and 4d, respectively.

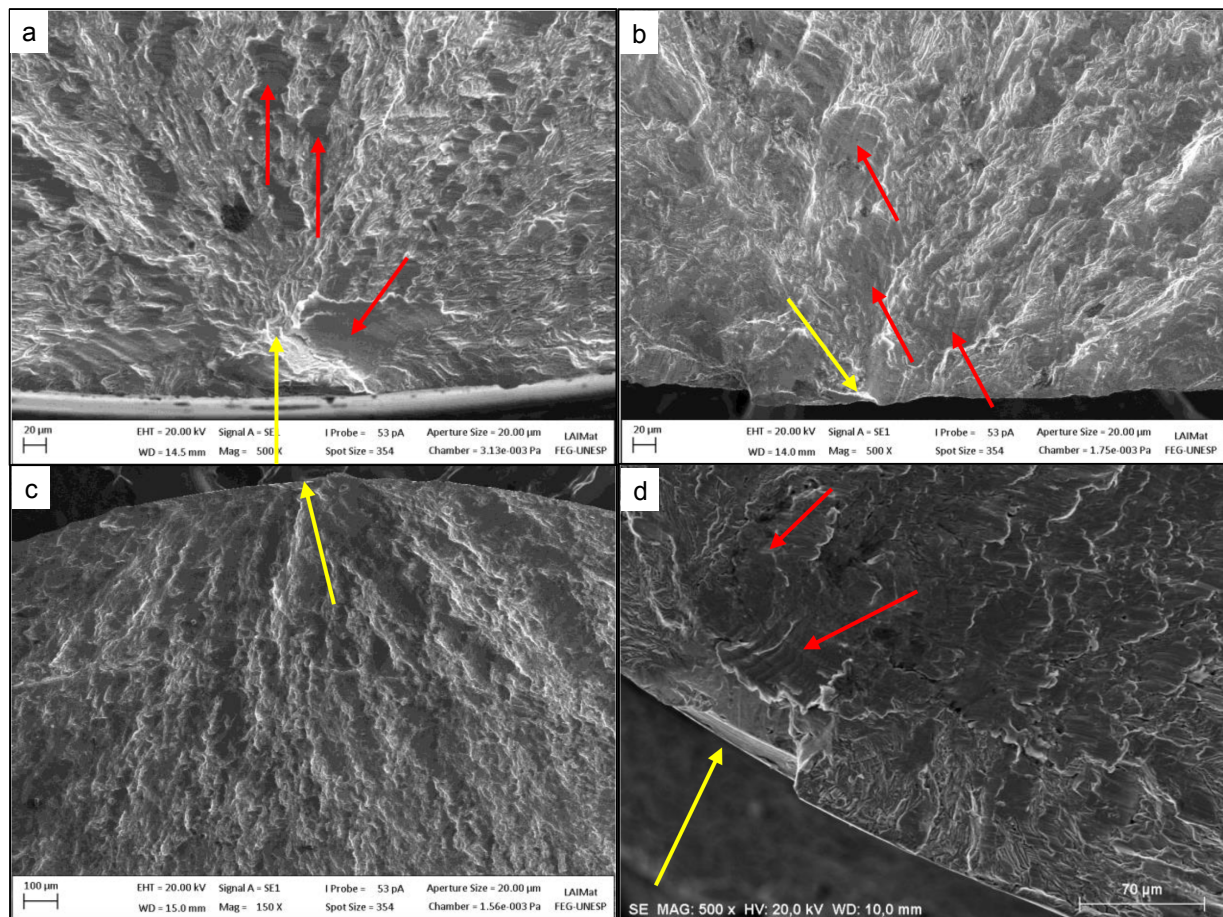


Figure 4. Fatigue fracture surface. a) Base metal, b) PIII/1H, c) PIII/2H, d) PIII/3H.

Figure 4 indicates fatigue crack initiation at surface, for all conditions studied. Results from nanohardness test are indicated in figure 5.

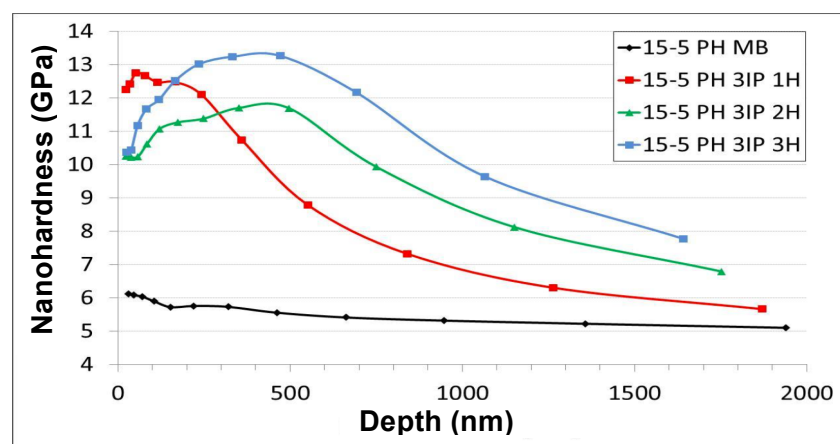


Figure 5. Nanohardness against depth. 15-5PH stainless steel 1, 2 and 3 hours PIII treatments.

An increase in hardness close to the surface is attributed to the specimens preparation. For 500nm depth, considering the base material nanohardness equal to 5GPa, experimental data in figure 4 shows for PIII treatments, 9GPa, 12GPa and 13GPa for one, two and three hours, respectively.

It can be seen in figure 6, that the PIII treatment applied on the 15-5PH disk decreased the pin mass loss in comparison to the base material. Moreover, the highest reduction was obtained with one hour treatment. It can be inferred that the decrease in mass loss is related to two changes caused by the disk PIII treatments: increase in roughness and hardness both in nanometric scale, according to figures 5 and 7. Improved tribological properties were observed for N-PIII into SS304 samples, as a consequence of a surface layer composed of oxygen and nitrogen [12]. Experimental results also indicate improvement in hardness and wear resistance of H13 steel due to the PIII treatment [10].

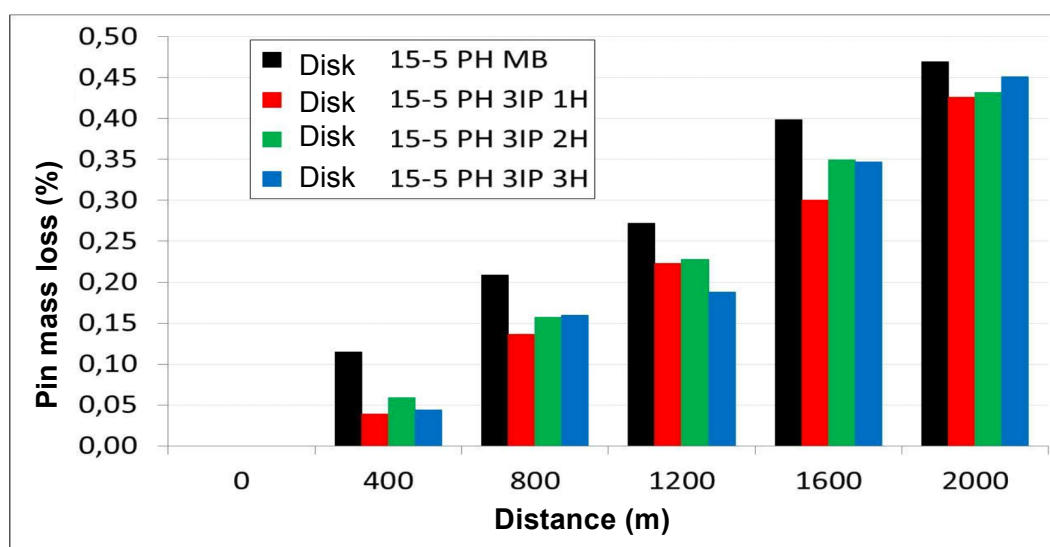


Figure 6. Pin mass loss.

Figure 7 shows atomic force microscopy images of the 15-5PH stainless steel before and after the implantation process.

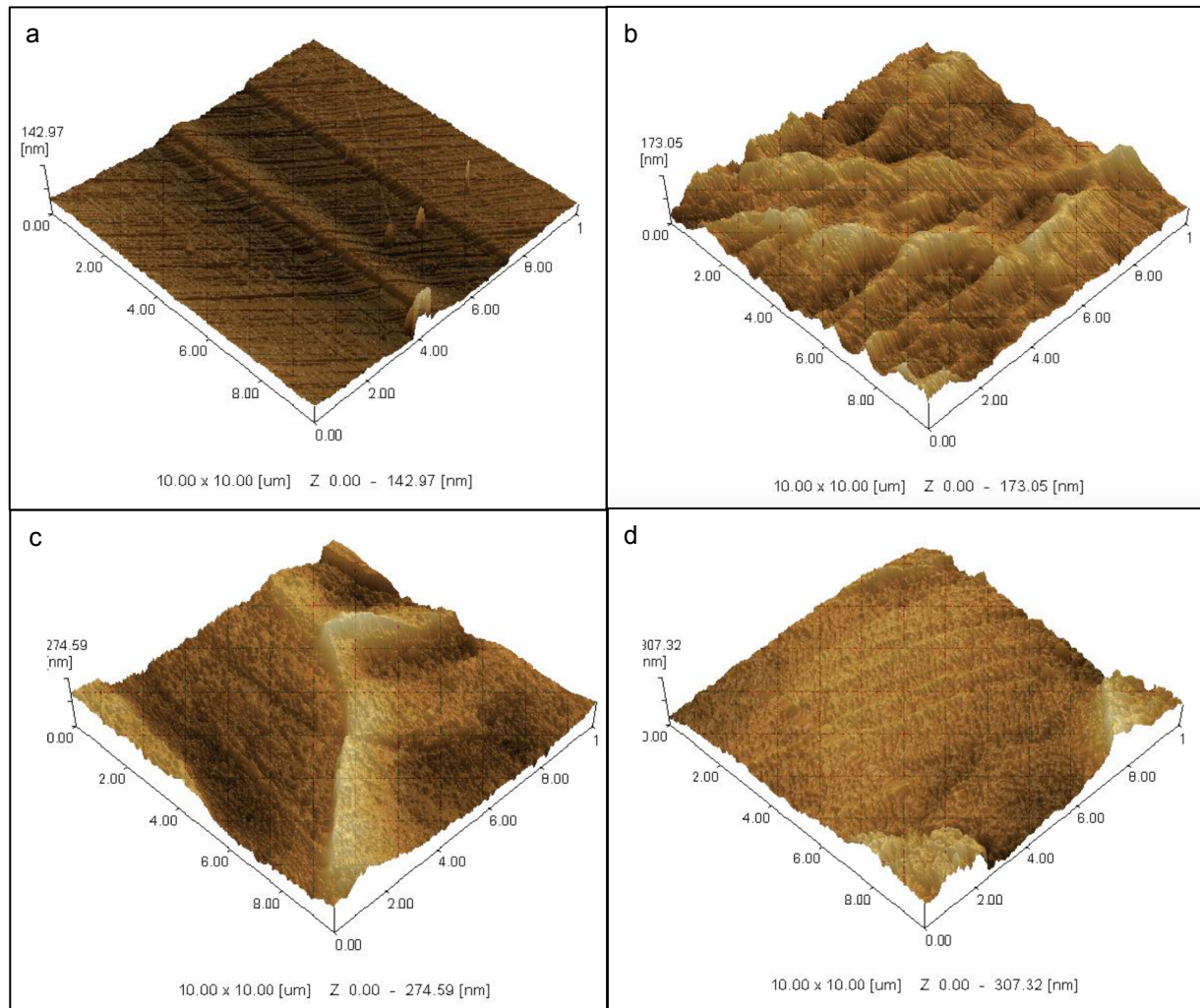


Figure 7. Atomic force microscopy. a) 15-5PH stainless steel, b) PIII/1H, c) PIII/2H, d) PIII/3H.

A characteristic feature observed in the untreated 15-5PH stainless steel is the presence of two regions on the sample surface, maybe originated by the polishing. Possible interference in the measured roughness, 142.9nm, occurred. Surface roughness for PIII treatments were 173nm, 274.6nm and 307.3nm for 1,2 and 3 hours, respectively.

As already mentioned, the increase in surface roughness and material nanohardness associated to the PIII treatments, played an important role on the pair pin-disk abrasive wear results obtained.

4. Conclusions

1. Increase on the 15-5 PH stainless steel axial fatigue strength occurred with PIII treatments for 1, 2 and 3 hours. Higher number of cycles to failure is directly related to the increase in treatment time.
2. Tensile residual stresses are present at base material surface and 0.01/0.1mm depth. Increase in compressive residual stresses at surface was observed for PIII treatments, -115MPa; -230MPa and -270MPa, respectively for 1, 2 and 3 hours.
3. The plasma immersion ion implantation increased, significantly the 15-5 PH stainless steel nanohardness at 500nm depth. Higher nanohardness resulted from longer treatment times.

4. The PIII treatment applied on the 15-5PH disk decreased the pin mass loss in comparison to the base material. Increase in roughness and hardness in nanometric scale, are associated to this behavior.

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