Improvement Corrosion Behaviour of Lean Duplex Stainless Steel 2101 alloy in Ringer solution by Plasma Nitriding for Biomedical applications

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Abstract. "Plasma nitriding" is a surface hardening process that involves diffusion of the atoms of nitrogen onto the surface of metal under different plasma nitriding conditions. The new alloys used in the field of Biomedical applications are Lean Duplex Stainless Steel. The alloys of Lean DSS are corrosion resistant, lightweight, and have good mechanical properties such as fatigue strength, but in aggressive environments, they lack "wear resistance". In a vacuum chamber of air (3 mbar), 400 V, and 30 mA, a lean duplex stainless steel (2101) alloy rod was plasma nitrided. The procedure of plasma nitriding was carried out at various times. The effect of plasma nitriding at different times (5, 10, 15, 20, 25) hrs on the chemical structure of LDX 2101 DSS alloy and the form of phases was investigated using OM, FESEM with EDS, XRD, and antibacterial test, tafel potential polarizaton and cyclic polarization for Orthopedic application. The results show that layers and phases S, Fe3N, and Fe2-3N were formed on the alloy's surface, which would improve mechanical properties and corrosion resistance in Ringer solution at 37 °C.

1. Introduction
Duplex stainless steel categories have a multiphase microstructure, usually ferritic-austenitic and contain about equal fractions of them. The addition of nitrogen to modern duplex stainless steels improves their properties. DSS has "good toughness, high strength, and good stress corrosion" properties, as well as the lower nickel content caused a lower alloy cost [1]. The addition of nitrogen to modern duplex stainless steels improves their properties. Attributed to the existence of the austenitic phase, duplex stainless steels have better tensile, yield strengths, and weldability than ferritic or austenitic stainless steels, they also have higher hardness, are tougher than austenitic grades, and more ductile than ferritic stainless steel, and have strong corrosion resistance. In fact, duplex stainless steel grades show well resistance to uniform and intergranular corrosion than austenitic grades. Duplex stainless steels have good corrosion resistance due to the presence of the austenitic phase [2]. Modern duplex stainless steels have evolved into groups: Lean grades, Regular grades, Super and Specialized “hyper-duplex” grades. The development of lean duplex with much less accepting elements than the standard duplex grade is currently the primary aim. The target is to replace 316 and even 304 grades because 316L stainless steel has a poor resistance to pitting and crevice corrosion in the human body, which causes for more than 90% of implant failure. Industeel offers lean duplex grades as a cost-effective alternative to "304L and 316L" in a variety of applications [3 - 6].
2. Experimental Work

2.1 Materials and Methods

For rod form, LDX 2101 DSS alloy (provided by Sweden OutoKumpu factory) was cut with a wire cut machine (knuth smart DEM manufactured in Germany) to a diameter of 15 mm and a thickness of 5 mm. Table 1 shows the chemical structure of the LDX 2101 DSS alloy.

Table 1. Chemical composition of LDX 2101 DSS alloy.

| LDX 2101 DSS Alloy | EN 1.4162 | ASTM S32101 | C 0.03 | Cr 21.5 | Ni 1.5 | Mo 0.3 | N 0.22 | Others 5Mn Cu | Fe Bal |
|--------------------|-----------|--------------|--------|--------|-------|--------|--------|-------------|--------|

2.2 Plasma Nitriding Modification

The technique of plasma nitriding was done on polished finished LDX 2101 DSS sample for various nitriding times (5, 10, 15, 20 and 25 hrs) in sputtering plasma nitriding system. Low-pressure gas discharge unit containing of a vacuum chamber with two parallel electrodes. The plasma chamber includes a Target (cathode) and disk of stainless steel (anode). The anode faced the cathode (which provides an electric field for the gas to be discharged) as shown in Fig.1. The top of cathode is shielded by cathode space assembly which included by ceramics insulator and stainless steel holder. The bottom shaft of the cathode electrode is shielded by insulator disk (ceramic). The target electrode (effective area of cathode dark space) is 7.5 cm while the diameter of the top electrode is 14.5 cm and the gap distance between them can be differed from 4 to 6 cm, in the present work a 4 cm distance is used. After evacuating the process chamber to high vacuum (≈ 1 × 10⁻⁵ mbar) with a high vacuum system containing rotary and turbo molecular vacuum pumps, N₂ gas was fed to confirm the complete removal of heavy gases such as hydrocarbons. A 4 kV DC voltage–power supply is used to confirm the necessary voltage for plasma nitriding and the discharge voltage for plasma nitriding was 400 V. The plasma nitriding process was accomplished in 3 mbar pressure of nitrogen. The clean samples are placed on the anode in the center of the dark shield as shown in Fig 1. The cathode is connected to the DC power supply, while the grounded chamber served as anode. After the process was finished, switch off voltage dc power supply and aeration the chamber in nitrogen gas to purpose reduce the pressure inter the vacuum chamber and exit the samples.

Figure 1. The plasma nitriding unit.

2.3 Metallographic Characterization of Plasma Nitriding Samples

Optical microscopy (OM) was used to detect the ferrite and austenite phases. Surface morphology observed by using SEM, "LEITS-type", "FESEM", ("ZEISS-type microscopes") at different times, as well as determination of affected depth, thickness of the diffusion zone and the average grain size.
associated to the plasma nitriding process. Nikon’s, ("EclipseME600, Digital Camera DXM 1200F microscopy"), made in China, was used to OM examination, with a magnification of (X500-X1000), and identification of phases formed during plasma nitriding performed by X-ray diffraction (XRD) ("The Shimadzu Lab X XRD- 6000, Japan"), the test was performed by using a copper target (Kα radiation = 0.15418 nm) with a nickel filter. The scanning speed of the diffractometer was adjusted to 5 deg per minute with the range of the diffraction angle of 20° was 20°–80°.

2.4 Electrochemical Corrosion Tests
Two types of corrosion tests were performed in this study. The first test was potentiodynamic polarization, which was used to determine corrosion parameters, and the second test was cyclic potentiodynamic polarization, which was used to determine the pitting resistance of the LDX 2101 DSS alloy. At various times, all samples of the substrate (15 x 5) mm diameter and thickness were prepared and coated by a plasma nitriding technique. In a Ringer solution at 37°C, the corrosion behaviour of all samples was investigated, and the test was done at 0.4 mV/s from an initial potential of 200 mV above the open circuit potential and continued up to 200 mV below the open circuit potential. CE: counter electrode (platinum), RE: reference electrode (saturated calomel electrode), and WE: working electrode make the potentiodynamic polarization cell.

2.5 Antibacterial test
Biomaterials surfaces is susceptible to microbial colonization and bacterial attack; thus it was necessary to study the antibacterial activity of plasma nitrided samples then the application of this alloy in the orthopaedic applications. The antibacterial activity and antibacterial kinetics of Escherichia coli, a gram positive bacteria, were studied using the inhibition zone method. The bacteria were then spread around a petri dish containing “Mueller Hinton agar,” and 0.5 mL of suspension was added to the dish, which was then incubated for 24 hours at 37 °C.

2.6 Adhesion Test.
The adhesion was measured with Rockwell-C adhesion test; it uses tester of a standard Rockwell hardness fit by Rockwell (Type-C) diamond cone by an applied load (150 kg). Rockwell hardness test was carried out on untreated LDX 2101 DSS and nitrided samples.

3. Results and discussion

3.1 Characterization of Primary Materials (LDX 2101 DSS)
LDX 2101 duplex stainless steel before surface modifications has microstructure biphasic, with darker ferritic and brighter austenitic phase grains in approximately area fraction of 50.6 % ferrite (α) and 49.4 % austenite (γ) without secondary phases (Fig.2), that calculated by using Image J-1.46r program, which is in agreement with reference [7].

Figure 2. Initial microstructure of LDX 2101 duplex stainless steel, with ferrite (dark) and austenite (light) phases.
3.2 Characterization of Nitriding Samples

3.2.1 Microstructure and Morphology Observations

A. Chemical Composition Distribution of Phases

The concentrations of major alloying elements in phases were identified by EDS. The results of EDS analysis of the microstructures for LDX 2101 alloy show, before and after plasma nitriding, the element concentrations in each alloy were significantly affected by nitriding times, which had an important influence on the corrosion resistance of each alloy. The elemental composition of the nitriding layer deposited on the LDX 2101 DSS alloys for different time parameters are shown in Figure 3.

![Figure 3. EDS for nitrided samples at condition: (R) untreated (a) 5, (b) 10, (c) 15, (d) 20, (e) 25 hrs.]

The main elements of Fe, Cr, O, N, and Ni are found in the nitriding layers, as can be observed in figure 3. These elements confirm the successful formation of a nitrided surface on the LDX 2101 DSS alloy. Moreover, there is no other Peaks different from nitride and this assure the coating purity. Table.2 illustrates these elements percent for each sample.
### Table 2. Elements Percent of nitriding layer on LDX 2101 DSS.

| No. | Condition | Fe%  | Cr%  | O%   | N%   | Ni%  |
|-----|-----------|------|------|------|------|------|
| A   | 5 hrs     | 66.6 | 20.83| 8.15 | 2.89 | 1.50 |
| B   | 10 hrs    | 65.72| 20.16| 9.40 | 2.87 | 1.85 |
| C   | 15 hrs    | 53.96| 16.35| 23.71| 3.60 | 2.38 |
| D   | 20 hrs    | 52.92| 17.30| 21.53| 4.59 | 3.66 |
| E   | 25 hrs    | 66.35| 22.76| 5.60 | 2.54 | 2.75 |

### B. Phase Identification of LDX 2101 DSS Alloy

The prescience phases were identified using XRD research. Figure 4 displays the sample's diffraction patterns before nitriding, revealing that the peaks only refer to phases of austenite and ferrite. Figure 4 also shows an XRD diffractograph of samples using standard JCPDS-ICDD (Joint Committee on Powder Diffraction Standards—International Center for Diffraction Data) standards. The XRD results, (Fig.4), revealing crystalline α and γ phases in the reference (untreated) LDX sample, these phases are identified with their crystalline planes according to the JCPDS card numbers where the diffraction pattern of the ferrite phase match with the (JCPDS Card No. 06-0694) peaks and the austenite phase match with the (JCPDS Card No. 33-0397). In all of the surfaces after nitriding, the N (expanded austenite - S-phase) and nitride (-Fe4N and -Fe2-3N) phases were identified. Similarly, as the treatment time increased, the α phase of plasma nitrided DSS converted into γN, Ɛ-Fe2-3N, and Ɛ + CrN (see Fig.4 – d and e), the γN phase being the main phase on the surfaces in accordance in the literature [8 - 11].

![Figure 4. XRD patterns of nitriding LDX (2101) samples at: (R) un treated (a) 5, (b) 10, (c) 15, (d) 20, (e) 25 hrs.](image)

### C. Field Emission Scanning Electron Microscopy

Fig.5 (a,b,c,d,e) shows the SEM micrographs of nitriding layer coatings at different times. It can be observed that the microstructure was densely packed and contains a homogeneous particles distribution within them in the coating and this results in the formation of homogeneous nitriding layers. The expanded austenite was the main phase of the nitrided layer. Each pre-existing ferrite in extended austenite was decomposed and E-nitride precipitated when the nitriding time was raised to 16 hrs. A significant amount of E-nitride and CrN precipitates were detected in the extended austenite's pre-existing ferritic area when the time of treatment was increased to various hours. In addition, several nitrides formed in the pre-austenite region [9].
Figure 5. SEM micrographs of nitrided samples of LDX 2101 DSS alloy at: (R) untreated (a) 5 (b) 10 (c) 15 (d) 20 (e) 25 hrs.

At 5-hour deposition time (Fig.5 a) as seen the distribution of particles is better than un coated morphology with porous structure. While Fig.5 (b,c) shows that a crack-free, uniform nitriding layer with a smooth, dense (closely packed microstructures), high homogeneity surface with less porous structure were obtained with deposition times of 10 and 15 hrs. On the other hand, after (20, 25) hrs as shown in Fig.5 (d,e), it can be observed that there are coarse particles with non-uniform distribution and high percent of porosity with some uncovered areas. These observations are due to the coalescence of excessive amount of Ɛ-nitride and CrN precipitates [9]. The cross-sectional topography of nitriding coatings at (5, 10, 15, 20, 25) hrs is illustrated in Fig.6 (a,b,c,d,e). The coated layer has a standard thin film structure, as can be seen, of (97.32, 176.28, 248.70, 200.06, 113.67) nm in thickness with homogeneity, higher compact, and comprehensive adhesion between the substrate and the nitrides layer. As well as Fig.6 shows mapping of elements that present in nitrided samples. Its thickness usually increases as the nitriding time is increased. When nitriding time is over 15 hrs, the thickness decreases slowly. Obviously, as the nitriding time is increased, the compound layer's growth rate slows down. The reason for this is that as the nitriding time is extended, the number of nitrides formed on the surface of LDX 2101 increases, resulting in increased nitrogen diffusion resistance and decreased nitride formation ability [10]. In PN, excessively long treatments lead to thinner coatings because of the plasma formation and continuous sputtering on the metal surface [11]. Some cracks were observed on the surface layer of the treated samples when the nitriding time is between 20 and 25 hrs, and the long nitriding time lead to appear more cracks. Surface layer-substrate adhesion strength worsens, and long nitriding time cause the more obvious decay. This may be due to the fact that as the nitriding time increases, the brittle phase of CrN (as seen in Fig.4) increases and coarsens, causing the surface layer's toughness to decrease. Furthermore, the structural variations between the phases in the surface layer increase residual stress [10].
3.2.2 Antibacterial Study

The effect of antibacterial of nitriding LDX (2101) Duplex Stainless Steel alloys was examined against E. coli culture as shown in (Fig.7). The formation of clear region around the disc refer to the bacterial inhibition zone. It can be observed that the both cases exhibited good antibacterial effect towards E.coli after 24 h of incubation period [12] and then a strong antibacterial effect of the nitride particles is obtained. Therefore, the presence of N2 nano particles were effective in preventing of bacterial adhesion on the surface and in turn, preventing bacteria growth that would improve the antibacterial activity. Bacterial test shown the nitrided layer treated the excellent anti-bacterial properties [13].

Figure 6. illustrates Cross-sectional SEM micrographs and EDS elemental mapping of N, O, Cr, Fe and Ni of nitried samples at (a) 5, (b) 10, (c) 15, (d) 20, (e) 25 hrs.
3.2.3 Adhesion Test

Optical microscope (OM) photographs of Rockwell C indentation on nitrided samples are seen in Fig. 8. In the nitrided samples, long and deep cracks can be seen near the indentation point. In addition, there is no delamination around the Rockwell C indentation effect. The results demonstrate that these samples’ adhesion strength quality is related to HF1 and HF2 but also radial cracking, and these defects are classified as HF2–HF3 adhesion strength quality. The adhesion of all nitrided samples is acceptable, while the substrate has poor adhesion, as shown in (Fig. 8 R) [14–16].

3.3 Corrosion Behavior of LDX 2101 Alloy

Investigation of corrosion behavior of base substrate and nitrided samples is obtained by potentiodynamic and cyclic polarization tests. At first, the sample is immersed in "Ringer’s solution" for (30 min) and after this time the "OCP" (open circuit potential) has been reported, where potential of a steady state is attained. The corrosion behavior has been studied for nitriding time.
3.3.1 Potentiodynamic Polarization

Fig. 9 shows the curves of potentiodynamic polarization for untreated LDX 2101 DSS alloy and nitrided samples in the "Ringer's solution" at 37 °C for different treatment times. Tafel extrapolation is used to extract the Icorr (corrosion current densities) and Ecorr (corrosion potentials) from the curves of potentiodynamic (Table 3). In addition, the corrosion rates (CR) were also included in this table.

![Potentiodynamic polarization curves of untreated and nitrided samples of LDX 2101 at different deposition time (5, 10, 15, 20 and 25 hrs).](image)

It can be seen from the results obtained in table 3, the untreated alloy has high corrosion current density (Icorr = 2.5 μA/cm²) and thus corrosion resistance because of the occurrence of metal ions dissolution on the surface of the uncoated substrate. After nitriding, it can be observed that the corrosion current density of some samples decreases, this mean that the nitriding provide a protective layer on the alloy surface that would reduce the corrosion rate. When nitriding at 10 h, a lowest corrosion current (Icorr = 0.118 μA/cm²) is obtained and this result has the highest corrosion resistance between the other one. This is attributed to dense structure of this coating layer. While, the nitriding at 15, 20 and 25 hours, showed some micro cracks in the structure and the presence of CrN in structure (see Fig.5 – c, d and e), this lead to decrease the corrosion resistance.

| Sample no. | Condition | Icorr (μA/cm²) | Ecorr (mV) | CR (mpy) |
|------------|-----------|----------------|------------|----------|
| R          | untreated | 2.75           | -34.2      | 1.329    |
| 1          | 5 hrs     | 1.56           | -37.1      | 0.754    |
| 2          | 10 hrs    | 0.118          | 13.2       | 0.057    |
| 3          | 15 hrs    | 1.87           | -84.8      | 0.903    |
| 4          | 20 hrs    | 2.66           | 15.3       | 1.285    |
| 5          | 25 hrs    | 5.64           | -145       | 2.725    |

In addition, coating at extension time lead to produce a coarse particles and inhomogeneity in the nitride layer that result in rough and poor deposit quality. On the other hand, nitriding at 25 h has the lowest corrosion resistance than the other as a result of the low layer thickness compared with the other samples. The nitriding with lower time (5 h) results in incomplete deposition that produce a
nonhomogeneous layer. Corrosion resistance of LDX 2101 DSS is higher than the common biomedical austenitic SS (316L) when they are immersed in the physiological solutions as Ringer’s, as mention in literature review, which means less toxicity and consequently the implant life is expected to be higher. Finally, as the nitriding time increases, the diffusion layer increases corrosion resistance, but after 10 hrs of nitriding, the uptrend remains stable [17]. The nitrided layer of samples at (5, 10 hrs) revealed good corrosion resistance because the coating layer was compact, lacking cracks or pits. However, when the nitriding time was above 15 hrs, dark etched patches and some cracks were seen (as shown in Fig.5- c, d and f), showing a low corrosion resistance and poor surface layer quality [7]. With limited or no passivation, the current density for the relatively long time nitrided (15, 20, and 25 hrs) increased rapidly. The passive Icorr (current density) of their samples is at least one order of magnitude greater than that of nitried samples. Clearly, the high time nitrided samples had lower corrosion resistance than the untreated samples (due to CrN formation and Cr depletion in the surrounding areas), whereas the low time treated samples had higher corrosion resistance [7].

Polarization resistance (Rp) calculations were used to measure the protecting ability of the nitriding layer since the recorded Rp values are inversely proportional to the corrosion current (higher polarization resistance means lower corrosion current). The rate of corrosion was relative to the Rp.

### Table 4. Polarization resistance of un nitrided and nitrided LDX 2101 DSS samples.

| Sample no. | Condition | Icorr (μA/cm²) | Eα (mV) | Bc (mV) | Rp (kΩ.cm²) |
|------------|-----------|----------------|---------|---------|-------------|
| R          | Un treated| 2.75           | 80      | -52.6   | 5.017       |
| 1          | 5 hrs     | 1.56           | 42.7    | -124.9  | 8.86        |
| 2          | 10 hrs    | 0.118          | 69      | -50.1   | 106.94      |
| 3          | 15 hrs    | 1.87           | 82.4    | -86.9   | 9.83        |
| 4          | 20 hrs    | 2.66           | 92.7    | -101.5  | 7.91        |
| 5          | 25 hrs    | 5.64           | 125     | -78.3   | 3.71        |

According to Table 4, un treatment LDX 2101 DSS has low polarization resistance (Rp = 5.017 kΩ.cm²). Nitriding led to increase in polarization resistance to (Rp = 106.94 kΩ.cm²) at 10 h which has lowest corrosion current density led to lowest corrosion rate.

### 3.3.2 Cyclic Polarization

In order to study the samples’s resistance to the localized corrosion, the cyclic polarization of nitrided samples at different times is carried out in the “Ringer’s solution” at 37 °C. Fig.10 illustrates the cyclic polarization curves for bare and nitriding samples at different times. The reading usually begins at the open circuit potential and progresses upwards before breakdown is reached. After a certain amount of localized corrosion has been formed and the passive film has been damaged, the scan path is reversed. Breakdown potential (Eb) or pitting potential (Ep) is the potential at which a stable pit forms. Consequently, Ep imply the potential over which the pits initiated and developed. The material has a high resistance to pitting corrosion if the Ep is high [18]. During the reverse scan, a negative hysteresis can be seen on the curve, as seen schematically in Fig.10. The difference (ΔE) between Ep and Eprot will give an indication to the corrosion resistance of the sample, where the smaller ΔE value mean the highest corrosion resistance. It is cleared from Fig.10 that the nitrided sample at 25 hrs had the lowest pitting or break down potential and thus low resistance to the pitting corrosion, this is due to the increasing of coarse particles. However, the nitrided sample at 10 hrs has highest pitting and protection potential with a close hysteresis loop where this polarization behavior tends to be resistance to the localized corrosion. In addition, the lowest value of ΔE as shown in table.5 is obtained and consequently this sample would have the more resistance to pitting corrosion. This means that the pit initiation and propagation have been effectively hindered by this sample. Therefore, based on the results from the potentiodynamic and cyclic polarization curves, it is supposed that the nitrided layer
at 10 hrs had a strong ability to act as a barrier to prevent the localized corrosion attack of many aggressive ions appeared in Ringer's solution during the corrosion test. Moreover, the nitriding layer have the ability to exhibit efficacious chemical barriers against metal ions release from the metallic implants. Thus, the osseointegration properties could be improved and the composite coating on LDX could be used as a biofilm for orthopedic implant applications.

**Figure 10.** Cyclic polarization curves for untreated and the nitriding samples at different deposition time in "Ringer's solution" at 37 °C.

**Table 5** Values of pitting potential for the substrate and the nitrided samples at different times from cyclic polarization.

| Sample no. | Condition | Ep (mV) |
|------------|-----------|---------|
| R          | untreated | 694     |
| 1          | 5 hrs     | 787     |
| 2          | 10 hrs    | 801     |
| 3          | 15 hrs    | 710     |
| 4          | 20 hrs    | 685     |
| 5          | 25 hrs    | 670     |

4. Conclusions

Following are some conclusions that can be extracted from the experimental results:

1. Plasma nitriding of LDX 2101 DSS results in a strengthening of the surface layer increasing in thickness from (97.32 to 248.70, then decreasing to 113.67) based on the nitriding times.

2. Plasma nitriding affects the initial duplex stainless steel phases of ferrite and austenite differently. The initial austenite grains in the surface of the LDX 2101 DSS form the S-phase (expanded austenite) as plasma nitrided at low temperatures, whereas "Fe,N" was precipitated from nitrogen saturated ferrite grains.

3. FE-SEM and EDS micrographs demonstrate that the nitrided layer thickness formed on the surface of LDX 2101 increases as plasma nitriding times increase, then decreases at 20 and 25 hours. This coating is also anti-bacterial and has good adhesion to the substrate, so it can be used in biomedical applications.
4. XRD studies showed the formation of nitrides (Fe3N, Fe2-3N), whereas increasing of nitriding times at 20 and 25 hrs leads to the formation of CrN.
5. The curves of polarization show that the plasma-nitried LDX 2101 alloy increased corrosion resistance in "Ringer solution" as compared to the untreated alloy for various nitriding times. These improvements are demonstrated by the low passive current density obtained after 10 hours of nitriding.
6. Plasma nitriding at 10 hrs has been reported as the best plasma nitriding conditions for LDX 2101 lean duplex stainless steels for biomedical applications.

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