Investigation of the Surface Properties of Nitride Coating Created Using Conventional and Active Screen Plasma Nitriding on Al

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Abstract

Active screen plasma nitriding (ASPN) is an emerging surface engineering technology that offers many advantages over the conventional plasma nitriding (CPN). In this research aimed to investigate the nitride coating FeN or AlN created on Al1050 by using in conventional plasma nitriding and active screen plasma nitriding. At first the rectangular cube-shaped samples with dimensions, 20 × 20 × 5 mm^3 prepared. Then under atmosphere %20 H_2-%80 N_2, at 500 °C, 80% duty cycle and the frequency of 10 KHz for 2, 5, 10 and 15 hours were plasma nitrided. The coatings were characterized using GIXRD, FESEM and AFM. The dominate phase in the ASPN of compound layer was ε:$Fe_{2\text{-}3}N$ and the dominate phase in the CPN was AlN, Also with increasing processing time, the layer thickness was increased. According to FESEM results the sample surface was formed of Nano sized iron nitride particle. The results showed that the morphology of the surface of samples coated with the method CPN are formed the nitride particles with irregular shapes. While the Surface of samples coated with the method of ASPN, to form hexagonal nitride particles covered with uniform distribution.

Keywords: CPN, ASPN, Aluminum, nitride coating.

Introduction

The automotive and aerospace industries increasingly employ aluminum and aluminum alloys in manufacturing. Coast savings results from reduced fuel consumption due to the lower weight of Al compared to most other metals. Aluminum and Al alloys compared to most other metals. Aluminum and Al alloys have high specific strength, good machinability, and formability. However, they lack good wear resistance. Wear resistance can be improved by the application of a surface hardening process. Some surface hardening processes change the composition of the surface layers and can lead to the formation of a surface compound with optimum wear resistance [1].

Plasma nitriding is one of the structural improvement processes of steel surface that improve wear and corrosion resistance, fatigue and breaking of industrial pieces and it results in better performance and increase durability. That most of the time apply for increasing and improvement of hardness surfaces and effective penetration depth in steel pieces [2, 3]. There is several kind of nitriding like pulse plasma nitriding, plasma nitriding with low pressure and duplex treatment and etc. Initial hardness of surface develops with this operation. Pulse plasma nitriding has below advantages in contrast with conventional DC plasma nitriding: 1- Arc suppression during treatment, 2- Overcoming on complex geometry, 3- Deeper plasma penetration to narrow holes and grooves, and 4- Prepare of controlling of surface roughness. The major mechanism of penetration treatment is reaction between plasma and metal surface [4, 5]. Currently conventional plasma nitriding has the more use in steel nitriding. Never and else's that There are many advantages of conventional plasma nitriding than other traditional nitriding methods but there are many problems like: keeping of uniformity of temperature in inlet case of piece – restriction of conducting treatment on pieces with complex shapes – creating of non-uniform layer in piece surface – the risk of arc damage and Hallow cathode phenomena during conducting process [6].

Recently, active screen plasma nitriding (ASPN) has been developed to overcome the limitation of conventional plasma nitriding such as arc damages, edging and hollow cathode effect. In this method, the components are enclosed in a metal screen or cage and instead of the components (which are placed in a floating potential) the high active voltage is applied to the screen. In conventional plasma nitriding, the coating material strongly depends on the substrate material. While in ASPN, a large variety of nitride coatings can be deposited using different type of metallic screens. ASPN technique has been widely used for deposition of iron nitride coating on a steel substrate [7].

According to literature, there is no report on deposition of the iron nitride on Al alloys to improve their functional properties. In this research the pure Aluminum sample was coated by conventional plasma nitriding and active screen plasma nitriding technique to investigate the effect of increasing time on the properties of surfaces coating in both method nitriding. Moreover, plasma nitriding compared to other technique used for producing iron nitride needs less sophisticated and cheaper equipment which facilitate its industrial applications. Hence, in this research we aim at the deposition of method using an iron cage. Finally, produced iron nitride coatings were compared to the ALN one which was deposited via conventional plasma nitriding.

Experimental Procedure

In this research, Al 1050 with the purity of 99.5 was used as substrate material. Chemical composition of the alloy shown in Table 1.

Table 1. Chemical composition of used aluminium alloy (wt %)

Al	Fe	Ni	Pb	Cu	Mg	Mn	Si	Na	Other
99.531	0.322	0.016	0.006	0.005	0.004	0.003	0.087	0.002	0.03

At first the rectangular cube-shaped samples with dimensions, 20 × 20 × 5 mm^3 prepared. Samples were ground mirror polished with SiC paper from 100 to 3000 grit and 0.5mm alumina powder. Plasma nitriding, the samples placed on an insulator porous chamotte in an iron screen with 0.8 mm thickness where they were plasma nitride in a pulse DC plasma reactor. The parameters related to Screen are listed in Table 2 and shown in Figure 1.

Table 2. Parameters related to screen

Material	Thickness	height	Hole diameter	Screen diameter
St37	0.8mm	100mm	10mm	100mm

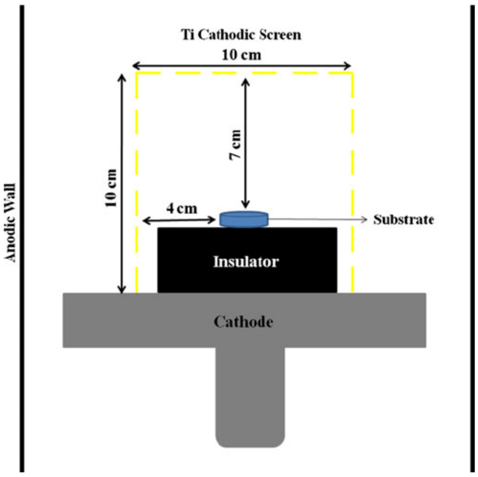

Figure 1. Schematic diagram of active screen Set-Up [7]

Then Samples were plasma nitrided under an atmosphere of %80H_2–%20N_2 with the frequencies of 10 kHz and in the duty cycles of % 80 at the temperature 500°C for 2, 5, 10 and 15 hours. The phase composition of the coating was studied by Grazing Incidence X-Ray diffraction (GIXRD) analysis using a PANalytical, X'Pert Pro MPD with a Cu $K\alpha_1$: $K\alpha_2$ radiation (λ=1.5418 A˚). The surface morphology and cross sectional microstructures of the coatings were studied using a Field Emission Scanning Electron Microscopy (FESEM) equipped with energy dispersive X-ray spectroscopy (EDS). Surface topography and Nano Hardness were studied using AFM (Ara Research Nanotechnology, Iran) and depth-sensing indentation (Triboscope system, Histron Inc, USA) Technique, respectively. For each sample, five measurements were performed at different point on surface and the average roughness value (Ra) has been reported.

Results and Discussions

The Grazing Incidence X-ray Diffraction pattern of samples treated at the temperature 500 ˚C after 2, 5, 10 ,and 15 hour of plasma nitriding shown in Figure 2. It can be seen that the phases are formed of f.c.c.-AlN, ε: $Fe2_{-3}N$ and f.c.c.-Al in the coating. It is possible that some of the aluminium belonging to the coating, and substrate is another part. Then the coating are formed several composition, that It can be say compound layer. Afterward, by increasing the coating time from 2h to 15h, the intensity of the ε: $Fe2_{-3}N$ and f.c.c.-AlN peak increase, respectively.

Therefore, increasing the coating of treatment time caused thickening the surface of coating. Increasing in coating thickness with increasing time is one of the unique properties of active screen plasma nitriding technique. It can be seen that the sample treated at temperature of 500 ºC after 2 hour of plasma nitriding is not formed of f.c.c.-AlN in coating. It is well known that during the ASPN-ing of steels, the iron particles with the phase composition of FeN will be deposited on the surface of the steel [8]. Therefore, due to being thermodynamically unstable, the FeN will lose a fraction of its nitrogen atoms and obtain the chemical composition of Fe_XN (X=2, 3, 4). Released nitrogen will diffuse into the steel substrate forming the case. Steels have the capability of solving atomic nitrogen and forming solid solution [9]. However, some of the released nitrogen atoms will react with the Al and it is formed f.c.c.-AlN. Regarding the Al substrate, thermodynamic data clearly support the formation AlN particles. The standard free energy of nitride formation of iron and aluminium were calculated at the different temperature and the results are presented. It is seen that at the temperature range off 100-600 ˚C, the standard free energy of formation of AlN is much lower compared to that either Fe_2N or Fe_4N. Hence, it could be considered that the tendency of the AlN is much higher than that of iron nitride phase. In other word, there is a strong driving force for nitrogen atoms to leave the iron nitride and form AlN [10]. Therefore, the thermodynamic evidence shows that there is grate possibility for the formation AlN along with $Fe_{2\text{-}3}N$. It may also be possible Al not combines with nitrogen then the coating consists of a mixture ε: $Fe_{2\text{-}3}N$, AlN and Al uncompounded. Previously, several experimental results have reported that the AlN layer has two different crystal structures such as hexagonal close packed structure (h.c.p) of wurtzite –type and face cantered cubic structure (f.c.c) of NaCl-type. Expected that f.c.c.-AlN had higher hardness and higher strength than h.c.p.-AlN film due to the higher volume density of f.c.c.-AlN [11].

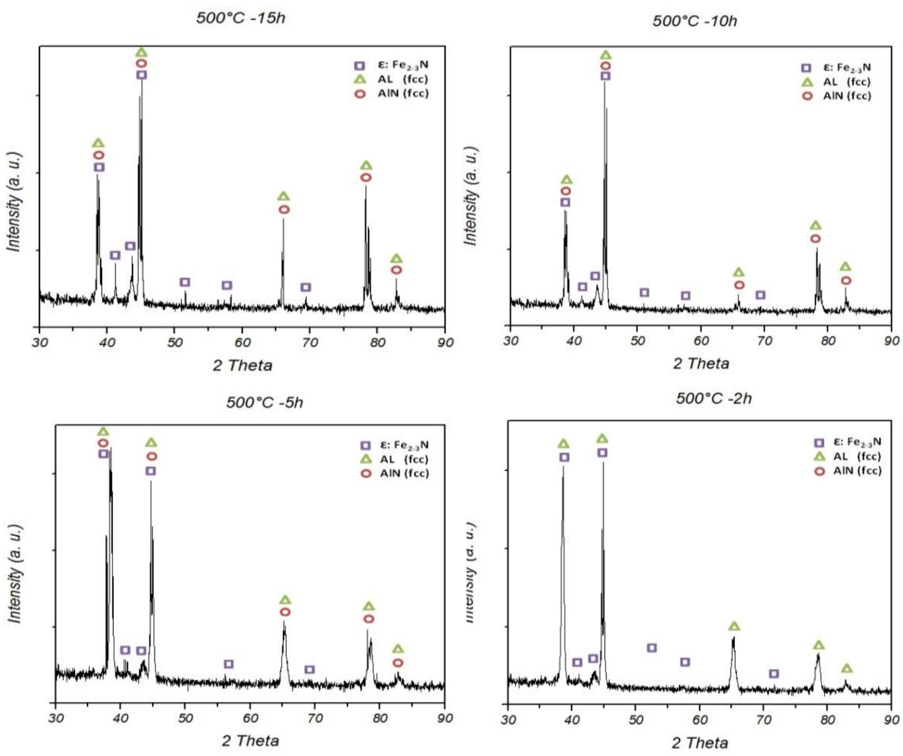

Figure 2. GIXRD patterns of active screen plasma nitrided samples for 2, 5, 10, and 15 h.

FESEM image from the surface of coated samples by ASPN for 2, 5, 10, 15 hours, are shown in Figure 3. As it is seen coating layer consists of very small particles of iron nitride and Aluminium nitride. Such a particulate structure is one of the unique properties of coating deposited using active screen plasma nitriding [12]. It can be seen that Hexagonal particles is formed on the surface of coating. Also, it is observed that with increasing processing time, particles size increases. This particle coarsening phenomenon is an evidence of particle growth that occurs with increasing time. It is obvious that increase of treatment time causes more formation of FeN at plasma, increase spattering iron cage and consequently more deposition on the surface. Then the particles size is larger on the surface of coating by increasing treatment time [13].

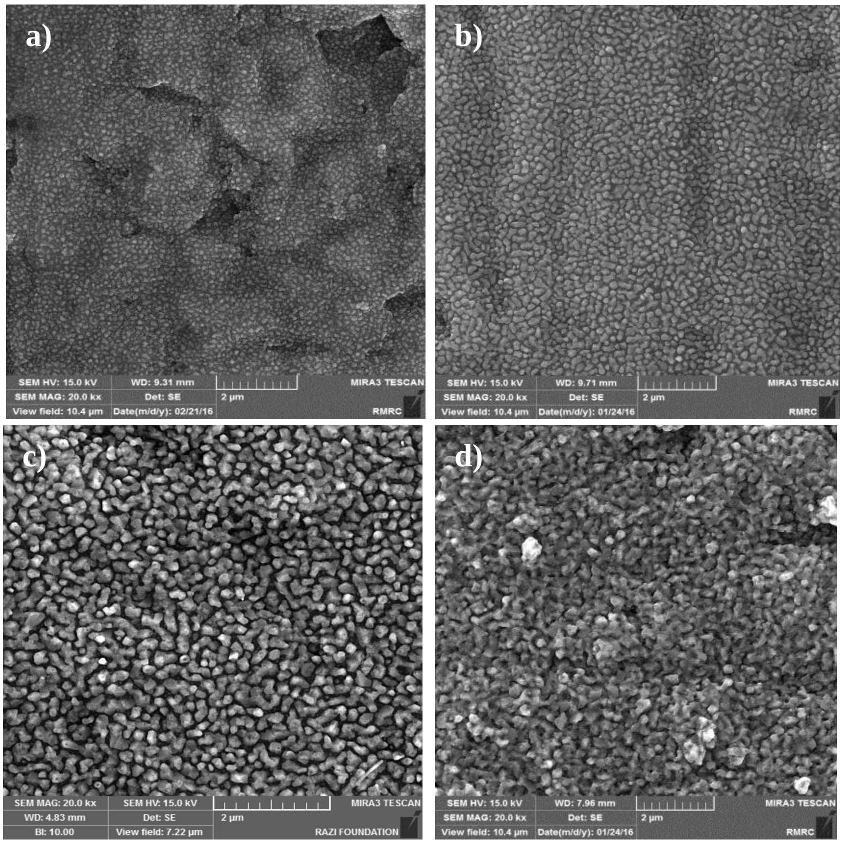

Figure 3. FESEM image of coated samples surface by ASPN at 500 ºC for a) 2, b) 5, c) 10, and d) 15 h.

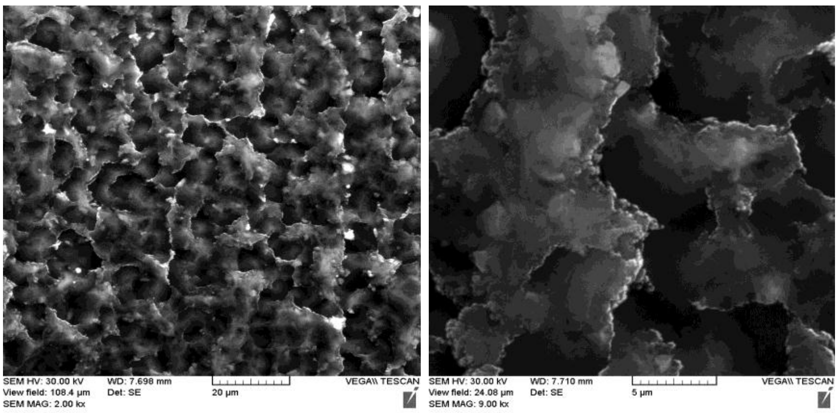

Figure 4. FESEM image of coated samples surface by CPN at 500 ºC for 10h.

FESEM image from the surface of coated samples by CPN at the 500 ºC temperature for 10 hour are shown in Figure 4. It can be seen that AlN nodules have produced from the surface of the Al. The dominate phase in the CPN was AlN, Also with increasing processing time, the layer thickness was increased. FESEM image showed that the morphology of the surface of samples coated with the method CPN are formed the nitride particles with irregular shapes.

Topology features of the deposited coatings were exanimated using AFM method as shown Figure 5. As can be seen in, a highly dense iron nitride coating with no defect, such as pores or cracks in either the coating itself or the interface region with the substrate, has been deposited on the Al. such features could be considered as strong evidence of the high adhesion and cohesion strength of the produced. It is clearly seen that at ASPN method roughness of surface the lower than CPN method and uncoated Al.

a) **b)** **c)**

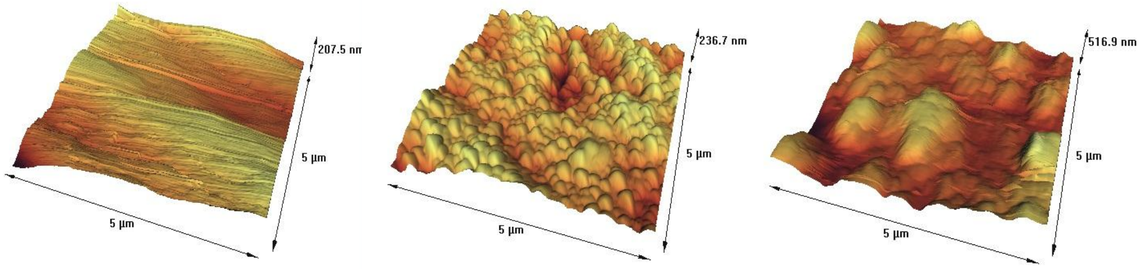

Figure 5. AFM image of coated samples surface by a) uncoated, b) ASPN, c) ASPN, at 500 ºC for 10h.

Table 3 shows the value of Nano Hardness, Friction Confident and Roughness of the Samples. As can be seen, the Nano hardness of the aluminium has improved drastically by applying $Fe_{2-3}N$ coating. Moreover, by increasing the nitriding time, the hardness value of the coating increases. Though longer coating time will lead of the formation of the larger constituent particles, the incremental trend of the coating hardness could be attributed of the formation of AlN layer. AS mentioned previously, at long coating periods the nitrogen atoms will diffuse towards the Al substrate and from the AlN layer beneath the iron nitride coating.

Consequently, the presence of the hard AlN coating will increase the surface hardness of the top iron nitride coating. In order to compare the mechanical properties of the conventionally plasma nitrided aluminium (AlN coating) with those of coated Al via ASPN method Al via ASPN method ($Fe_{2-3}N$ + AlN) coating, the same Al sample was nitrided in similar conditions. The mechanical properties of both coatings were assessed and the results are also tabulated in Table 3. It seen had been that the AlN coating created by CPN method has the higher hardness compared to the sample nitrided by ASPN method.

Table 3. Nano Hardness, friction confident and Roughness for produced coating.

Sample	Nano hardness (Gpa)	Roughness, Ra (nm)	Friction coefficient
Uncoated Al	**0.65**	**30.56**	**0.85**
CPN	**8.4**	**61.25**	**0.7**
ASPN	**7.9**	**25.38**	**0.3**

Conclusion

In this research, aluminum samples were plasma nitrided using iron active screen. It was found that the coatings are formed from $Fe_{2\text{-}3}N$, AlN, and Al. Moreover, it can be seen that enhancement of the mechanical characteristics of coating with increasing time treatment. Therefore, the thermodynamic evidence shows that there is grate possibility for the formation AlN along with $Fe_{2\text{-}3}N$ formed on the surface of plasma nitrided aluminum sample has particular and f.c.c. structure.

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