Study the Effect of Diffusion Heat Treatments for Nickel Electroplating on the Surface Hardness of 7075 Aluminum Alloy

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ABSTRACT: In this research a surface hardening process by Ni coating and subsequent diffusion heat treatments was studied at 7075 Aluminum alloy. Nickel coatings with different thickness were obtained by change the coating time and current density. Heat treatments at 450 °C, 500 °C, and 550 °C for times (6, 12, 24) hours were performed in order to obtain surface hardening required of the aluminum alloy by diffusion of nickel into the substrate. The effect of temperature and diffusion time on surface hardness of Al 7075 alloy was studied. Surface hardness about 800 [HV] were achieved after heat treatment at 500 °C for 24 hour by diffusion of Ni in the substrate and cause of formation of Al_3Ni_2 intermetallic phase, as x-ray diffraction tests of samples showed. While is about 670 [HV] after heat treatment at 550 °C for 24 hour because of diffusion of aluminum toward the surface of sample, as x-ray diffraction tests of samples showed.

1. INTRODUCTION

Aluminum and its alloys are attractive for many applications in chemical, automobile and aerospace industries because of their excellent properties as height strength-to- weight ratio, high electrical and thermal conductivities and good formability, however their hardness is poor in comparison of steel and continuous efforts are made in the research into new possibilities for making use of the advantage of the aluminum in application that were reserved up to now for harder materials.

The solution mainly adopted is to produce a thick hardened layer on the substrate by three major methods:

- Thermochemical Diffusion treatments methods (case hardening) as Nitriding [1] and Carburising [2]
- Oxidation of the surface layer of aluminum alloys by Hard Anodizing or Plasma Electrolytic Oxidation (PEO) [3]
- Nickel deposition with other element as tungsten, phosphorus, and boron. Researchers have completed deposition in different ways are: Nickel Electroplating [4], Nickel Electroless plating [5,6,7], Physical vapor deposition (PVD) and Chemical vapor deposition (CVD) [8], all this methods followed by diffusion heat treatment.

We have known that researchers have studied the surface hardening of 7075 aluminum alloy by various methods mentioned, except by nickel electroplating. Moreover, there is a few of researchers have studied the effect of nickel electroplating on the surface hardness of different aluminum alloys. Where A.Ul-Hamid et al studied the effect of the heat treatment of the 2014 aluminum alloy that plated electrically with nickel on the surface mechanical properties as the surface hardness, micro scratch and friction coefficient [4], without study the effect of different temperatures and times of heat treatments on these mechanical properties.

So in this work, we studied the effect of temperatures and times of heat treatments on the diffusion of nickel deposited by electroplating of 7075 aluminum alloy, and thus on the surface hardness of the 7075 aluminum alloy.

2. EXPERIMENTAL

Rectangular specimens with a surface area of 6 [cm²] with a thickness of 5 [mm] were obtained from 7075 Al alloy, The chemical composition of 7075 aluminum alloy is reported in Table 1 [9], A “mirror-like” finish was finally achieved by grounding the samples with successive SiC papers grit 400 to 1200 and subsequently polished using 0.25 μ m diamond paste.

Table 1. Chemical composition of 7075 aluminum alloy

Element	Al	Ti	Cr	Si	Fe	Mn	Mg	Zn	Cu
wt %	89.5	0.05	0.18	0.08	0.17	0.009	2.3	5.8	1.8

The surface of the specimens was degreased with alcohol and air dried, then treated in alkaline treating bath, The chemical compositions and operation conditions of this bath is reported in Table 2. Then the specimens were rinsed with a deionized water and then the surfaces were activated by chemical etching in a 6 % HF (hydrofluoric acid) aqueous solution for 5 seconds, after chemical etching, the specimens were rinsed with a deionized water and immersed in the solution for the electrolytic Ni deposition.

Table 2. Chemical compositions and operation conditions of alkaline treating bath

Compound	Formula	Concentration [gr/L]	Anode	stainless steel
Sodium Hydroxide	NaOH	71	Cathode	Aluminum samples
Sodium Phosphate	NaH ₂ PO ₄	30	Applied voltage	2 [V]
Sodium Hydrogen Carbonate	NaHCO ₃	30	Treatment time	20 – 30 [Sec]

In nickel coating cell the Al 7075 specimens were connected with cathode while the anode is nickel, the chemical compositions and operation conditions of nickel deposition bath is reported in Table 3.

Table 3. Chemical compositions and operation conditions of nickel deposition bath

Compound	Formula	Concentration [gr/L]	Anode	Nickel
Nickel Sulfate	NiSO ₄ (H ₂ O) ₆	300	Cathode	Aluminum samples
Nickel Chloride	NiCl ₂ •6H ₂ O	50	PH	2-3
Boric Acid	H ₃ BO ₃	50	Bath temperature	60 °C

The Figure 1 shows the principle of nickel electroplating and interactions taking place on both the anode and the cathode [10]

Diffusion heat treatments of the coated specimens were carried out in a furnace at 450°C, 500° C and 550°C for different times (6, 12, 24) hours.

Figure 1. Principle of nickel electroplating

3. RESULTS AND DISCUSSION

3.1. Adjust Coating Parameters

During the course of this study, several trial runs of electrodeposition of nickel on 7075 aluminum alloy were undertaken using different conditions. It was observed that factors such as bath temperature, rate of stirring, pH level of the electrolyte, level of applied current and potential. All influence the nature and adherence of the nickel coating formed on the Al 7075 specimen surface. Higher than optimal temperature and stirring rate resulted in non-adherent and flaky deposits while lower temperature and stirring led to non-uniform deposition, lower temperatures and higher stirring rates led to black flaky non-adhering deposits. Increased pH levels of the bath gave rise to cracked, flaky and blackened deposits.

Optimal coating results were obtained when the bath solution was maintained at a temperature of 60 °C, Stirring was kept at approximately 80 rpm, a pH level of 3.6 was maintained by adding HCL to the cell after each deposition. The best results were obtained by using a constant deposition current density of 0.2 A/cm² when current intensity was 1.2 [Am] and coating time was 10 [min], and then coating thickness was 40 [μm]. The Table 4 and Figure 2 show coating thickness change with change the current density and coating time

Table 4. Coating Thickness values for different Current Density and Coating Time

Current Density [Am/Cm ²]	Coating Time [min]		
	5	10	15
0.03	4	7	11
0.08	9	17	26
0.16	16	32	51
0.2	21	42	63

Figure 2. Coating Thickness Changes by Change Current Density and Coating Time

3.2. Diffusion Heat treatments and microstructural characterization

The coated samples were cut and metallographically mounted in cross-section, ground with 400, 600, 800, 1000 and 1200 grit size SiC paper and then polished using 0.25 μm diamond paste. A FEI Quanta 200 scanning electron microscope (SEM) was used to examine the samples. Scanning electron microscope analysis of the electrolytic coating showed that the layer, constituted by metallic nickel, was uniform, adherent and thick about 40 μm Figure 3.

The diffusion heat treatments, performed on the first three coated samples at 450° C for 6, 12, 24 [hours], and any significant nickel-aluminum diffusion not appeared, as it is shown in the Figure 4.

Figure 3. Nickel coated samples before heat treatment

The diffusion heat treatments of second three coated samples performed at 500° C for 6, 12, 24 [hours], and any significant nickel-aluminum diffusion not appeared when the treatment times were 6 and 12 [hours], as it is shown in the Figure 5 (a) (b), while a unique, uniform and compact diffusion zone appeared when the treatment time was for 24 [hours], as it is shown in the Figure 5 (C).

Figure 4. Coated samples at 450° C for 6 (a), 12 (b), 24 (C) [hours]

Figure 5. Coated samples at 500° C for 6 (a), 12 (b), 24 (C) [hours]

Figure 6. Coated samples at 550° C for 6 (a), 12 (b), 24 (C) [hours]

Finally the diffusion heat treatments of last group of three coated samples performed at 550° C for 6, 12, 24 [hours], and any significant nickel-aluminum diffusion not appeared when the treatment times was 6 [hours], while a diffusion zone clearly appeared when the treatment time were for 12 and 24 [hours], as it is shown in the Figure 6.

3.3. Surface Hardness

For measuring the surface hardness of coated and treated samples of 7075 aluminum alloy, nickel coating layer was removed by manual grounding, until we get the aluminum surface and then measure its hardness. Rockwell hardness tester was used, and results are measured on the superficial N-scale with a cone indenter of angle 120° at 15 [kgf] load for high values, while we use B-scale with a ball indenter of diameter 1/16" at 100 [kgf] load for low values. Finally, all values are converted to Vickers hardness by the conversion tables listed in ASTM – E140, all measurements are performed according to ASTM E-18. The Table 5 and Figure 7 show the surface hardness values of 7075 aluminum alloy for different temperatures and times of diffusion heat treatments.

Table 5. Surface hardness values of 7075 Aluminum alloy for different diffusion heat treatments.

		6 [hours]	12 [hours]	24 [hours]
Untreated	179	-	-	-
450 °C	-	178.8	185	192
500 °C	-	190	380	800
550 °C	-	196	460	670

Figure 7. Surface hardness of 7075 aluminum alloy for different temperatures and times of diffusion heat treatments.

3.4. X-Ray Diffraction tests

In order to explain the rise in surface hardness of 7075 aluminum alloy, the X-ray diffraction (XRD) was employed to investigate phase composition of the samples; analysis was carried out using the Philips XRD tester model PW 1830. The XRD pattern of 7075 aluminum alloy sample without nickel coating was given in Figure 8. The XRD pattern of nickel coated sample was given in Figure 9. The result showed several peaks that indicate for nickel. The peak for Al cannot be found after coating, which indicates the quality of nickel coating.

Figure 8. XRD pattern of 7075 Aluminum alloy sample without Nickel Coating

Figure 9. XRD pattern of Nickel Coated sample

The XRD pattern of coated sample and treated under 500°C for 24 hour was shown in figure 10. The results show new peaks that indicate for the presence of Al_3Ni_2 intermetallic phases on the surface of the sample. This intermetallic phases is the main responsible of the surface hardening of this sample.

The XRD pattern of coated sample and treated under 550°C for 24 hour was shown in Figure 11. The results show peaks that indicate for the presence of Al_3Ni_2 intermetallic phases, and new peaks that indicate for the presence of aluminum on the surface of the sample, which indicates to aluminum migrate from sample substrate towards the surface during heat treatment. This migrate of aluminum led to a decrease of the surface hardness of this sample.

Figure 10. XRD pattern of coated sample and treated under 500°C for 24 hour

Figure 11. XRD pattern of coated sample and treated under 550°C for 24 hour

4. CONCLUSION

- Heat treatments of Nickel coating on 7075 Aluminum alloy for different times and temperatures allowed the diffusion of Nickel in 7075 Aluminum alloy substrate with the formation of a hard diffusion layer.
- The better results have been obtained after 24 hours of heat treatment at 500°C which led to more uniform zone of diffusion.
- Surface hardness about 800 HV was achieved by diffusion of Nickel and formation of Al_3Ni_2 intermetallic phases, as XRD analysis was shown.
- Surface hardness decreased to 670 HV after 24 hours of heat treatment at 550°C because of Aluminum migrate from sample substrate towards the surface, as XRD analysis was shown.

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