MICROSTRUCTURE CHARACTERIZATION OF (Ni-P-AL2O3) ELECTROLESS COATING OF LOW ALLOY STEEL (AISI 4140)

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ABSTRACT
In this paper, investigating micro hardness, surface roughness measurements of electroless composite coatings (Ni-P-AL2O3) deposited in electroless immersion on low alloy steel. All specimens (20 mm diameter × 10 mm height) were used as the substrates for low alloy steel (AISI 4041). were grinded and polished as ASTM by emery paper (tungsten oxide paper) no (400–2500), then samples were washed by distilled water and ethanol and dried by using an electrical dryer. Polishing was conducted by diamond paste, specimens were immersed in acetone for (60 min). Before coating, specimens were immersed in solution including materials (30 g/L NaOH, 60 g/L NaCO3 and 60 g/L NaPbO4) for two-minute period at( 75°C) temperature moving the electrolyte using magnetic stirring by supply power (3 volts) to remove oil and any dust from metal surface. After that, the specimens were washed with distilled water, and Placed in sulfuric acid solution (20%) for (10 sec) .after that it directed immersion in coating solution . The electro less (Ni-P-AL2O3) coatings were prepared by using nickel chloride as a source of nickel in alkaline baths, using (alpha alumina). The formulas were characterized by means of measurements of micro hardness, surface roughness. Our the results revealed that the heat-treated sample (Ni-P-AL2O3) showed higher hardness (2061HV). Increasing in the concentration was noted of (AL2O3) induced as-coated(Ni-P) conversion structure from amorphous to crystalline. The composite coating (Ni-P-AL2O3) is better compared to the Ni-P coating, and they get even better with heat treatment increased. The addition of (AL2O3) makes the coating denser and the grain size to decrease according to the formulae being heat treated.

KEYWORDS: Electroless Ni-P-AL2O3 Coating, Micro Hardness ,Surface Roughness

1. INTRODUCTION
Electro less coating of Ni-P alloy technique is a well-known commercial treatment and has applications in many fields because of very good properties such as high resistance to corrosion, high wear resistance, high hardness and excellent ductility[1]. Electro less coating is a form of coating metal with chemical instead of electrical means[2]. It is advantageous because it makes metal coating via a controlled autocatalytic or self-continuous reduction process. Chemical reaction reduction with controlled chemistry coating and electroless has grown as metal finishing, surface engineering, and etc. growth of the regions[3]. Electroless coatings have mechanical and physicochemical properties, and are increasingly being used[4]. The benefits of this technique are the regularity of the thickness of the coating layer even on parts with complex and small shapes, which is one of the key difficulties in electroplating due to a low ionic strength for electroplating, because the coating layers are usually devoid of porosity, which prevents the entry into force of ions and reduces the corrosion cycle. For these technical features, as the microstructure of the coating layers, crystalline changes and the chemical compounds are deposited by heat treatment to improve resistance to corrosive environments [5-6]. Improved mechanical properties, wear resistance and composite coatings are of great attention. Large Micro Durability[7]. Composite coatings (Ni-P-AL2O3) are widely used in the military industry[8]. The formation of a metastable phase of equilibrium and precipitation of( Ni3P) compound is primarily responsible for improving the abrasive wear behavior of heat treatment coatings[9-10]. A change in mechanism of deformation (Orowan mechanism) determined by the coarsening of Ni3P Precipitates at higher thermal treatments[11-12]. This study aims to explain the effects of electroless behavior. (Ni-P-AL2O3) coating with specific heat treatment coating concentrations on some mechanical properties (micro hardness, roughness) and low alloy steel microstructure(AISI 4140).

2. EXPERIMENTAL

2.1. PREPARATION OF SUBSTRATE

The substrate the launch tube of the Kalashnikov (MK). Specimens (20 mm diameter × 10 mm height) were used as the substrates. The chemical analysis for this alloy and details composition (weight %) is shown in Table 1.

Table 1: Chemical Compositions The shotgun tube (MK) for (AISI 4140) low alloy steel actual

| Element | W%  | Fe   | Mo  | Cr  | Ni  | Co  | Mn  | Cu  |
|---------|-----|------|-----|-----|-----|-----|-----|-----|
|         |     | 95,43| 0,234| 1,629| 0,352| 0,113| 0,995| 0,18|

All Specimens (20 mm diameter × 10 mm height) were used as the substrates for low alloy steel (AISI 4041). were grinded and polished as ASTM by emery paper (tungsten oxide paper) no (400–2500), then samples were washed by distilled water and ethanol and dried by using an electrical dryer. Polishing was conducted by diamond paste, specimens were immersed in acetone for (60 min). Before coating, specimens were immersed in solution including materials (30 g/L NaOH, 60 g/L
NaCO3 and 60 g/L NaPbo4) for two-minute period at (75°C) temperature moving the electrolyte using magnetic stirring by supply power (3 volts) to remove oil and any dust from metal surface. After that, the specimens were washed with distilled water, and Placed in sulfuric acid solution (20%) for (10 sec ).after that it directed immersion in coating solution.

2.2. ELECTROLESS BATH PREPARATION

After completion of preparing surfaces for coating, electroless bath were prepared for the process according to concentrations shown in table (2 and 3). The coating Ni-P was deposited on low alloy steel (AISI 4140) with electro less plating process. Value of PH, the plating bath was varied between (3.5 and 14). The electroless coating of (Ni-P-AL2O3) was performed at( 85 -90°C) for( 60 min)[13-14]. Through coating, solution of bath are agitated by a magnetic stir to avoid localized overheating and reduce the fluctuation of ionic concentration. Figure( 1) shows the experimental setup for the electroless composite deposition. After the completion of the process of coating in Figure (1), specimens were placed inside a vacuum oven to for (60 minutes), at a temperature of (200,300,400)°C.

### Table 2: Operating conditions for electro less bath I.

| Bath composition                  | Quantity     |
|----------------------------------|--------------|
| Nickel Sulfate (NiSO4.6H2O)       | 15g/l        |
| Sodium Hypophosphite (NaH2PO2.H2O) | 15 g/l      |
| Sodium acetate (Na(CH3COO).3H2O ) | 10 g/l      |
| **Operating conditions**          |              |
| pH                               | 4-6          |
| Temperature                      | 85-90°C      |
| Time of immersion                | 1 hour       |

### Table 3: Operating Conditions for Electro less Bath with alumina addition II.

| Bath composition                  | Quantity     |
|----------------------------------|--------------|
| Nickel Sulfate (NiSO4.6H2O)       | 15g/l        |
| Sodium Hypophosphite (NaH2PO2.H2O) | 15 g/l      |
### Operating conditions

|                | Value |
|----------------|-------|
| Sodium acetate (Na(CH3COO).3H2O) | 10 g/l |
| Alfa Alumina (Al2O3) | 3 g/l |
| **pH**          | 11.3  |
| **Temperature** | 85-90 °C |
| **Time of immersion** | 1 hour |

#### 2.4. HEAT TREATMENT

In this work, annealing process is conducted in furnace-coated specimens, which were soaked at a temperature (200, 300, 400°C) for a period of one hour. Then, specimens were furnace cooled at room temperature. In Figure (2)

#### 2.4.1. HEAT TREATMENT AT (200 °C)

Annealing process was performed in a vacuum oven. Coated samples were immersed at (200 °C) temperature, where every 1 minute the oven temperature rose to (6° C) and the room temperature (25° C) was required (30 minutes) to reach a temperature (200° C) After leaving the oven for one hour, samples were leaved in oven to cool until its reach room temperature.

#### 2.4.2. HEAT TREATMENT AT (300 °C)
Annealing process was performed in a vacuum oven. Coated samples were immersed at (300 °C) temperature, where every (1) minutes the oven temperature rose to (6° C) and the room temperature (25° C) was required (45 minutes) to reach a temperature (300° C) After leaving the oven for one hour, samples were leaved in oven to cool until its reach room temperature.

2.4.3. HEAT TREATMENT AT (400 °C)

In this work, the annealing process was performed in a vacuum oven. Coated samples were immersed at (400 °C) temperature, where every (1) minutes the oven temperature rose to (6° C) and the room temperature (25° C) was required (60 minutes) to reach a temperature (400° C) After leaving the oven for one hour, samples were leaved in the oven to cool until its reach room temperature.

![Figure2: Heat treatment](image)

2.5 CHARACTERIZATION

Micro hardness of the coating layers were measured by using (TH-717 Vickers hardness tester), a load of (25 g) was applied for (15 sec). Three readings were recorded for each specimen coated and one at the substrate. The value of measurement for surface roughness was completed for the coated samples before and after heat treatment using a common parameter Ra in μm, surface roughness Test (HER210. Model) was used to calculate Ra with an accuracy of 0.05 μm and the average were calculated based on the measurements.

3. RESULTS AND DISCUSSIONS

3.1. Phases Analysis of Electro less Plating (Ni-P) &(Ni-P-AL2O3)

X-ray-diffraction analysis has been used to identify all phases present in the specimens, as well as crystalline planes and their orientation for the phases are also analyzed. Figure (3) shows the X-ray-diffraction pattern for Ni-P plating bath as
plated. It is clear that the presence of NiP and Ni3P in the coating layer after heat treated. It is clear that the presence of Ni3P in the coating layer which consider the major phase, so it more scattering of X-ray through this phase. In most of electroless plating by Ni-P, the presence of the intermetallic compound especially the (Ni3P) is necessary to improve most of mechanical properties such as hardness, corrosion resistance and wear resistance.

![XRD Pattern of (Ni –P-AL₂O₃) After Heat Treated](image)

**Figure 3: XRD Pattern of (Ni –P-AL₂O₃) After Heat Treated**

3.2. MICROSTRUCTURE RESULT SCANNING ELECTRONIC MICROSCOPE (SEM)

Figure (4) expresses the cross-sectional surface coating (Ni-P-AL₂O₃) and thickness of coating (8.87 μm). It was treated at (400°C) for one hour

![SEM Of Ni-P –AL₂O₃ Coating of Surface of Cross Section of Heat Treatment](image)

**Figure 4: SEM Of Ni-P –AL₂O₃ Coating of Surface of Cross Section of Heat Treatment**

The SEM study that was performed on the agglomerates reveals that they are composed of the particles. Figures (4 and figure 5) expresses the morphology of
the (AL2O3) embedded in Ni-P matrix and the AL2O3 precipitates. They are of irregular shapes. They act as reinforcement particles in Ni-P coatings. In addition, AL2O3 appears in the coatings, which contain Ni-P particles. The coatings composite expresses a structure of spherical nodule by good uniformity and coverage of dense.

![Figure 5: SEM of Ni-P-AL2O3 at higher Magnification](image)

![Figure 6: SEM of Ni-P-AL2O3 at higher Magnification](image)

3.3. EDS CHARACTERIZATION.

Figure (7) shows the EDS of (Ni-P-AL2O3) composite electroless coatings at the required parameters of the process that indicates that the coating contains (72.84% Ni, 9.07% P, 7.46% AL and 10.63 O%) (wt%), which gives exactly the initial amount of 9.07% P when the percentage of P is lower. 9% of the structure of the coating is expected to be amorphous. This is exactly what happened. The phosphorus
content indicates that the coating is a type of coating with high phosphorus. This result is supported by the SEM metallography.

**Figure 7:** EDS Spectrum of Electro less Ni –P-AL2O3 Coating.

### 3.4 MICRO HARDNESS.

Table (4) shows the surface of micro hardness samples of heat treatment. As noted, segregation happened at the composite, which led to a decrease in the composite micro hardness of coated layer. However, the hardness of the heat-treated coatings of Ni-P was significantly increased. It was increased from (350.7 HV) to (2061 HV) times with respect to the corresponding value of the uncoated steel. However, the coating itself is an effective technique to achieve this goal, whether using Ni-P only or incorporating with AL2O3 particle. However, the reasons behind such an increase in hardness appear to be different before and after heat treatment. P. Gadhari et al. (2014)[9], pointed out that the micro hardness of annealed composite coatings depends on three factors; level of incorporation of particles, annealing temperature and uniform distribution with less agglomeration of particles. For instance, the enhancement of hardness of Ni-P coatings when the deposition process variables remain; the nature of the coating layer only features and defines the extent of protection that can be provided by the coating.

| Sample | Value (HV) |
|--------|------------|
| Substrate | 350.7 |
| (Ni-P) a plated | 750.2 |
| (Ni-P-AL2O3) a plated | 1155.6 |
| (Ni-P) a annealed at (200C) | 802 |
| (Ni-P) a annealed at (300C) | 1199 |
| (Ni-P) a annealed at (400C) | 1522 |
| (Ni-P-AL2O3) a annealed at (200C) | 1189 |
| (Ni-P-AL2O3) a annealed at(300C) | 1723 |
| (Ni-P-AL2O3) a annealed at (400C) | 2061 |

**Table 4:** Micro hardness Results of as-Plated and Heat Treatment of Samples Surface.
3.5. SURFACE ROUGHNESS.

Roughness of the substrate data was illustrated in Table (5), as-plated and annealed. As mentioned above, increase in AL₂O₃ concentration causes a significant rise in the roughness. The roughness of sample Ni-P was low in comparison with other specimens because large concentration of AL₂O₃ which may produce agglomeration.

| Sample                  | Ra(μ) |
|-------------------------|-------|
| Substrate               | 0.042 |
| (Ni-P) a plated         | 0.278 |
| (Ni-P-AL₂O₃) a plated   | 2.94  |
| (Ni-P) a annealed at (200C) | 0.279 |
| (Ni-P) a annealed at (300C) | 0.362 |
| (Ni-P) a annealed at (400C) | 0.801 |
| (Ni-P-AL₂O₃) a annealed at (200C) | 5.46  |
| (Ni-P-AL₂O₃) a annealed at (300C) | 6.32  |
| (Ni-P-AL₂O₃) a annealed at (400C) | 8.87  |

Table 5: Result of Surface Roughness Samples

4. CONCLUSIONS

· Coating low alloy steel (AISI 4140) with Ni-P increases the micro hardness from (350.7 HV) to (2061 HV) after heat treatment.

· Incorporation of both (AL₂O₃) particles in (Ni-P) coating layers increased micro hardness (Ni-P-AL₂O₃) to (750.2 HV) and (2061 HV) after heat treatment.

· No flaws were observed in the coating layers after heat treatment.

· An increase in surface roughness is observed when painting in (Ni-P) and increases during heat treatment.

· An increase in the surface roughness is observed when painting in (Ni-P) and increases during heat treatment more than in pal (Ni-P-AL₂O₃) much more.

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