



Microstructure Study and Morphology of Ni / Nano Al₂O₃ Composite Coating Synthesized by Electroless Plating

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ABSTRACT

The electroless deposition method was used to prepare Ni-based nanocomposite coatings, this approach represents an alternate way of having coatings on the different substrates. Most of the previous literatures on this subject and work deals with a study the effect of the process conditions and, Bath composition on the microstructure at the macroscale of Ni-Al₂O₃ composite coatings. Though, effect on the step structure of the composition of the bath and microstructure are little, the present work aims to study the effect of hard ceramic Al₂O₃ nanoparticles at different concentrations (0.0, 0.5, 1.0, 2, 4) g/L, on the phase structure, microstructure and morphology of Al₂O₃ nanocomposite coating, in order to enhance the mechanical, physical and chemical properties of nanocomposite coating. In this paper, An X-Ray diffraction method, spectroscopy (EDS), energy dispersive, and scanning electron microscope (SEM) were studied in the phase structure, chemical composition and morphological nanocomposition coatings. In the present paper, is evident from EDS study that the composite coating consists of Ni and nanoparticles of Al₂O₃. The micrograph study of the EDS showed that A flat and smooth surface is present in the deposited nanocomposite coating. Uniform distribution of nanoparticles of alumina within Ni-Matrix. And the XRD study showed the crystalline structure of the Ni- Al₂O₃ nanocomposite coating.

1. Introduction

Due to aluminium and its alloys characteristics like low density, light weight, and good corrosion resistance, therefore used widely in the aerospace and automotive technology [1]. Aluminium has a corrosion resistance to a surface layer of thin protective aluminium oxide, and it is considered to be one reactive metal. Thus, in aqueous solutions and air is stable. Aluminium oxide is usually safe, but localized corrosion may result because of pores and other defects caused by alloys [2]. One of the most key part is a component surface, and characteristic that are directly responsible for the materials performance since on the surface of a material, most failures such as

corrosion and fatigue may be happen. [3]. Because effective factors in industrial parts, degradation such as corrosion and abrasion may be occurred, therefore numerous attempts had been made to get methods to reduce these effective factors [4]. There are different methods for the production of protective layers of the aluminium components, such as physical vapour deposition, micro arc oxidation (MAO), plasma spray, flame spray, and chemical vapour deposition methods have been recently developed [5-10]. Coating's methods enhance the tribological and mechanical characteristics of the base materials. Electroless deposition nanocomposite coating technique is one of most common techniques used in scientific fields, as well as in industrial fields. Its ability to produce

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hard and wear proof surfaces with resistance to corrosion, nanocomposite electro-less coatings have recently been used in many applications in the chemical, automotive, aerospace and mechanical industries [11]. Completely new materials are successfully used, specifically for coatings to obtain optimal property with reduced material usage, low technical effort and low cost of process. Wurtz in 1844[12] was first noted the electroless deposition of metallic nickel with the involvement of hypophosphite as a chemical accident in an aqueous solution. Electroless plating is an autocatalytic procedure that improves substrates by dipping it in an electroless solution, also referred to an electroless bath, which has a reduction agent source, metal ions, complexants, weathering agents, supplements and stabilizers, etc. Electroless coatings are commonly divided into four categories: Coatings of alloy, poly-alloy, nickel pure, black nickel, composite coatings, and electroless nanocomposite coatings [13]. Electroless Ni-P coating is a new candidate technology for using in a wide range of engineering industries due to its many advantages, such as thickness uniformity, good wear resistance, lubricity, good ductility, corrosion resistance, excellent solder ability, and electrical properties [14]. Metal matrix composites (MMCs) synthesized by adding fine inert particles in an electroless bath metal matrix, electroless nickel composite coatings are manufactured. Composite coating materials have been extensively used in engineering applications. The best way of depositing metals and particles in a coating is known as electroless composite deposition. As a metal matrix, many metals are used, such as copper, nickel, and chromium. Hard oxides such as (ZrO_2 , Al_2O_3 , SiO_2), carbides (WC, SiC), diamonds or solid lubricants (MoS_2 , graphite, or PTFE) can be co-deposition particles. Efficient deposition of composite coatings has been reported in the last few decades, with particles having a standard diameter ranging from 0.1 to 100 μm . The related systems and properties have been studied by many scholars [15-20]. There is now a movement towards using nanosized particles to enhance the mechanical properties wear and corrosion resistance as an additional component

of composite coatings [21-25]. Another technique for obtaining coatings on the different substrates is electroless nickel matrix nanocomposite coatings. Thus, most of the topic and work literature deals with the effects of bath compositions, and process conditions on the macroscale and microstructure of Ni- Al_2O_3 composite coatings. Though, little is the influence of a bath composition (alumina content) on the phase structure and microstructure, so, the present study seeks the effect of hard ceramic Al_2O_3 nanoparticles and their concentration on the microstructure, phase structure, and electroless Ni-matrix nanocomposite coating morphology. Ni- Al_2O_3 coatings are highly abrasive, heat resistant and quite anti-corrosion properties. They can be attractive and alternative particularly to chromium coatings [26-27].

2. Experimental Work

Effective coating deposition is dependent on preparing the substrate, so careful and proper preparation of the surface of the substrate is necessary. In the current analysis, 20 mm x 20 mm x 2 mm Aluminum alloy specimens (4045) are used as a substrate for nanocomposite Ni- Al_2O_3 coatings. The sample preparation is properly based on the shaping, sorting and frying methods. The specimen is subjected to the grade 800 emery paper surface grinding method. The washed mechanically from international affairs and corrosion materials. After that, the aluminum substrate is washed with distilled water, acetone and heater-dried. Rinse with distilled water and clean with acetone to remove any surface layer shaped as rust. a chemical cleaning therapy is sequentially provided to the surface of substrate with 100 percent nitric acid for a short period. The substrate surface is enabled for a limited time by dipping into a zinc coat. This step is important for the deposition to begin as soon as possible when it is placed on the substrate inside the electroless bath. The activated substrate is next immersed at 85oC in the electroless bath and the coating takes place over a period of three hours. After several tests, the composition of bath and the conditions of operating for electroless Al_2O_3 reinforced Ni-nanocomposite coating are

selected and correct the parameters ranges are accordingly chosen. The most key parameters for coating deposition are varied; others have been held constant. Table 1 displays the range of composition and operating conditions of the electroless bath used for depositing electroless Ni- Al₂O₃ nanocomposite coatings small amounts of Surfactant Sodium Dodecyl Sulphate (SDS) is used to achieve a more potent distribution and to stop agglomeration, of the electro-free nickel poly alloy bath, of the second

phase of aluminum particles. The mortar and pestle are mixed thoroughly with a suitable amount of alumina powder, approximately 250 ml of electroless nickel solution. The magnetic punch is used to ensure the particles in the solution are evenly suspended. For a first time, a ni-layer is deposited to avoid the porosity of the coating. The solution with Al₂O₃ particles is then added in the same bath for the next two hours, to achieve Ni- Al₂O₃ coding position.

Table 1: Composition and operating conditions of the electroless bath

Composition of bath		Operation Condition
Nickel Sulphate source of Nickel metal	10, 10, 35, 35, 20 g/L	pH 5.5-6
Sodium Hypophosphite Auxiliary and stimulating materials in the painting process	5, 10, 15, 15, 10 g/L	Temperature 85±3°C
Al₂O₃ Nanoparticle Nanoparticles that are added to the coating material	0, 0.5, 1.0, 2.0, 4 g/L	Bath Volume 250 ml
Tri Sodium Citrate A stimulant and a stimulant for the chemical reactions that occur in the salt bath	5, 15, 15, 15, 10 g/L	Deposition Time 3 Hours
Sodium Dodecyl Sulphate (SDS) 0.025 A surfactant substance		
Sodium Acetate encourages the stability of the reactions and the salt bath during the coating process	5g/L	
Lead Acetate	2 mg/L	

NiSO₄ provides the Ni ions in the solution during the chemical reaction, while NaH₂PO₂ reduces the Ni ions to their zero valence states from their positive valence states. Since the reaction is very quick and intense between NiSO₄ and NaH₂PO₂, it is inevitable that the bath will decompose immediately. Complexity factors are therefore necessary to delay the response in a viable form (tri-sodium acetate). Complexion factors with Ni ions from metastable complexes, releasing them slowly for the reaction. But there is still a high possibility of solution breakdown even after the addition of complicating agents. In order for the solution to remain stable for the duration of the coating, a stabilizer (lead acetate) is therefore necessary. The Al₂O₃ particulate surfactant (SDS) is used to increase weight and surface charge. The thickness of the coating is

between 28-33 micron in the current sample. The samples are cleaned with distilled water once the coating is finished. Several factors influencing nanocomposite coating characteristics, such as Concentration of nickel source, decreasing concentration of agents, solution pH, bath temperature, concentration of stabiliser and wetting agent, second phase substratum particulate concentration, etc. The literature review shows, however, that the three variables are viz. Nickel source concentration, sodium solution concentration and second-phase particulate concentration (Al₂O₃ nanoparticles), are usually used in the first place. Nickel source (Nickel Sulphate Solution) in the researchers' nanocomposite coating control systems. For compositional, microstructural, and phase structure studies,

energy-dispersive X-ray spectroscopy (EDS), scanning electron microscope (SEM), and X-ray diffraction analyzers are used.

3. Result and discussion

3.1. Microstructural aspects and composition study

3.1.1. Compositional analysis results

The EDS test is used to determine the nanocomposite coatings' weight and presence of nickel and aluminum oxide. The EDS analysis of a nano- Al_2O_3 reinforced Ni-matrix composite coated sample with various Al_2O_3

nanoparticle concentrations is shown in Figure 1. Where the cross-section and the weight percentage of nickel is found to be (1.79 percent) is shown in Figure 1, and that is (66.65 percent) for aluminum and that is (11.52) for oxygen. Table 2 offers a comparative weight percentage study of the nanocoating composition for 0.0, 0.5, 1.0, 2.0, and 4.0 (g/L), respectively, in the plating bath. The compositional study shows that the existence of alumina nanoparticles in the coating increases as the concentration of Al_2O_3 nanoparticles in the electroless bath increases, resulting in mechanical properties being increased.

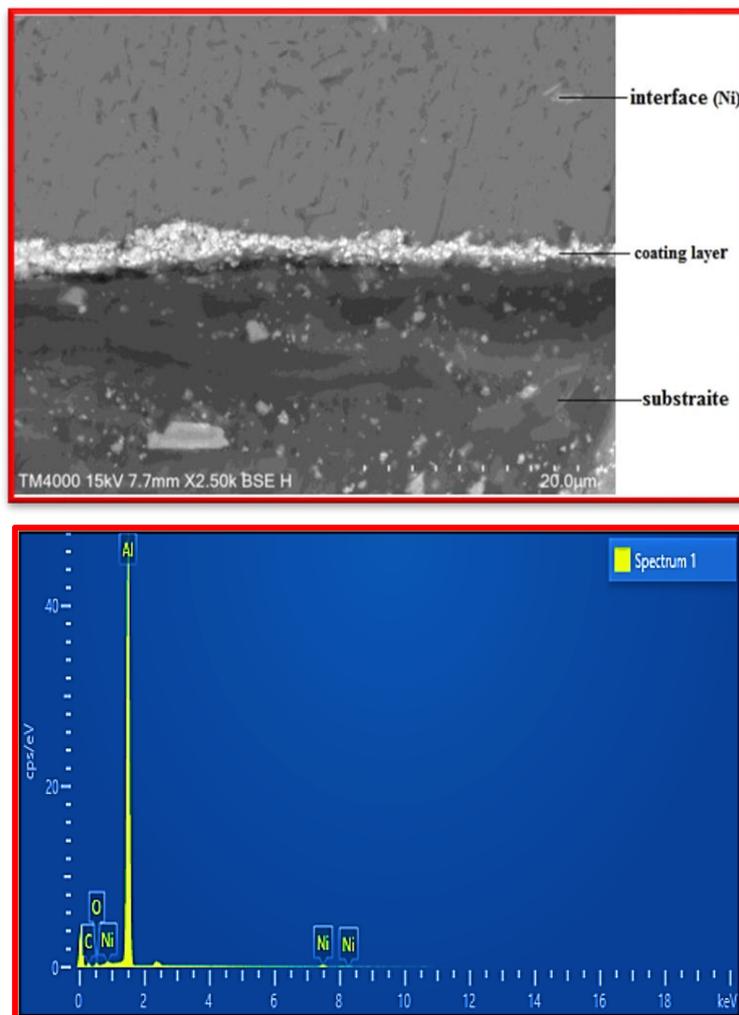


Figure 1. Ni - Al_2O_3 nanocomposite coating with alumina 4 g/L concentration, EDS & cross-sectional SEM image

Table 2: EDS results of Ni- Al_2O_3 nanocomposite coatings

Concentration of Al_2O_3 nanoparticles	0 g/L	0.5 g/L	1g/L	2 g/L	4 g/L
% of Al	8.64	26.78	34.63	61.32	66.65
% of O	0.0	11.52	6.39	6.11	4.30
% of Ni	29.14	23.44	23.41	2.79	1.76

3.1.2. Scanning electron microscope analysis results

The SEM surface images of nanocoating as deposited condition is shown in the Figure 2.

Nanoparticles of the second phase in a non-matrix are uniformly disturbing. The addition of the surfactant to the electroless bath results in the uniform distribution of nanoparticles.

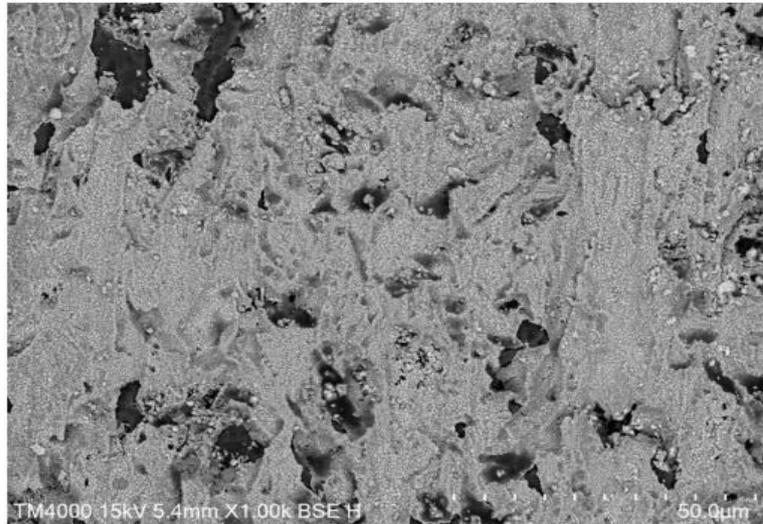


Figure 2: SEM images of nanocomposite coating Ni-Al₂O₃ at Al₂O₃ 4 g/L nanoparticle concentration

3.1.3. X-Ray diffraction results

The XRD test is applied to know the major coating phases in an as-deposited condition. Figure 3 displays XRD plots for the nanocomposite coating of Ni- Al₂O₃ at a

concentration of 4 g/L Al₂O₃ nanoparticles. XRD research also reveals that the nanocomposite coating structure is crystalline in nature. It can be seen from the XRD map the larger single big Al₂O₃ peak with Ni with diffraction angle equal to 44.648° is observed.

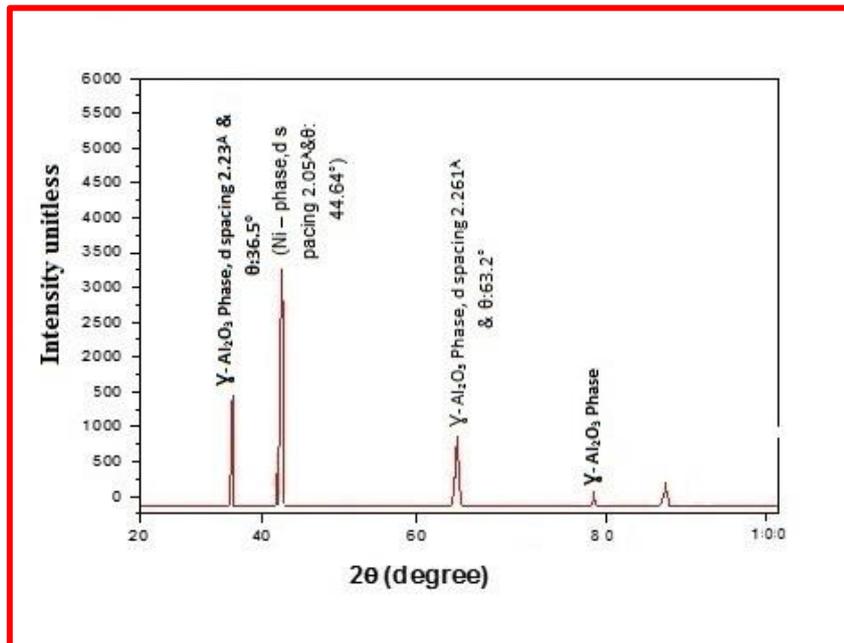


Figure 3. XRD chart of Ni- Al₂O₃ nanocomposite coating

4. Conclusions

The present work may be drawn these conclusions:

- Al₂O₃ reinforcement Ni-matrix nanocomposite coating produces successfully by using electroless deposition technique.
- It is evident from EDS study that the composite coating consists of Ni and Al₂O₃ nanoparticles.
- The SEM micrograph showed that A flat and smooth surface is present in the deposited nanocomposite coating. Uniform distribution of nanoparticles of alumina within Ni-Matrix.
- The results of the XRD study showed the crystalline the structure of the Ni- Al₂O₃ nanocomposite coating is crystalline.

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