Uluslararası İleri Doğa Bilimleri ve Mühendislik Araştırmaları Dergisi
Sayı 7, S. 227-233, 11, 2023
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Araştırma Makalesi

IJANSER
https://alls-academy.com/index.php/ijanser
ISSN: 2980-0811

International Journal of Advanced Natural Sciences and Engineering Researches
Volume 7, pp. 227-233, 11, 2023
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Research Article

Effect of current density on the structure and tribological properties of WS_2 reinforced Ni-P metal matrix composite coating produced by electrodeposition

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(Received: 04 December 2023, Accepted: 12 December 2023)

(2nd International Conference on Frontiers in Academic Research ICFAR 2023, December 4-5, 2023)

ATIF/REFERENCE: Uluğ, A., Karataş, Ş., Duru, E. & Uysal, M. (2023). Effect of current density on the structure and tribological properties of WS_2 reinforced Ni-P metal matrix composite coating produced by electrodeposition. *International Journal of Advanced Natural Sciences and Engineering Researches*, 7(11), 227-233.

Abstract **–** 2D (two-dimensional) materials have been the focus of recent material science studies. These materials are generally lamellated. Due to these physiological properties, they act as solid lubricants as anti-wear in parts operating under a certain load. In this study, WS_2 particles of 1 micron size were added to the Ni-P bath and Ni-P-WS_2 composite coatings were produced at different current densities at the same concentration. The aim of this study is to obtain Ni-P metal matrix composites reinforced with WS_2 particles on steel surfaces with high hardness and wear resistance for parts working under intense load in automobiles and aerospace vehicles and for use in anti-wear applications. In the study, WS_2 particles were added to the bath at a concentration of 0.1 g/L and 3 coatings were produced at current densities of 13 A/dm^2, 20 A/dm^2, 26 A/dm^2 respectively. The effects of different current densities applied during the deposition process on particle distribution, hardness and wear resistance were investigated. During the electroplating process, a suitable stirring speed and a certain temperature were kept constant for the deposition of WS2 particles on the Ni-P matrix. The produced films were characterised by scanning electron microscopy (SEM) and X-ray diffraction (XRD) analysis.

Keywords – Electrodeposition Coating, 2D Materials, Ni-P-WS_2 Coatings, Tribology, Wear Resistance, Current Density Effect.

I. INTRODUCTION

In the coating industry, electrodeposition and electroless coatings are improving result to protect and improve the surfaces of various objects. These advanced coating techniques stand out by offering advantages such as durability, corrosion resistance, wear resistance and enhanced functionality. Electroplating and electroless coatings have found a wide range of applications in different industries such as automotive, aerospace, electronics and manufacturing [1]. These coatings can be customised to suit specific requirements such as hardness, lubricity and adhesion, offering a range of advantages, particularly corrosion resistance, wear resistance, improved electrical conductivity and aesthetic properties. This makes them versatile solutions in a wide range of applications [2].

Electroless coatings, also known as autocatalytic coatings, are a type of coating that does not require an external electric current for the deposition of metal onto the substrate [3]. This type of coating works through an autocatalytic chemical reaction between the metal ions and the reducing agent in solution to carry out the deposition process. Electroless coatings have a number of advantages over conventional electroplating methods, including the ability to produce uniform coatings on complex geometries and excellent corrosion resistance [4]. Ni-P coatings are known for their excellent wear resistance properties, making them suitable for applications where durability is critical. These coatings provide an effective protective barrier against wear and corrosion, making them a preferred choice, especially in industries such as oil and gas pipelines [21]. The wear resistance of Ni-P coatings can be enhanced by methods such as alloying with iron, further improving their performance in high-stress environments.

Electrodeposition coatings provide excellent control over coating thickness, composition and surface morphology, making them suitable for a wide range of applications. One of the most important advantages of these coatings is that they provide high corrosion resistance [7]. The coating is continuous and compact, making the preferred orientation possible in this method [8]. Furthermore, this method can produce coatings on a wide variety of substrates and allows the fabrication of structural features with sizes ranging from nanometres to micrometres [9] Electrochemical deposition provides a better interfacial bonding between the coating material and the substrate, along with a relatively low cost [10]. While e-coating is widely used for corrosion protection in the metal industry, it is preferred for vehicle bodies, wheels and other parts in the automotive industry [12]. Furthermore, this technology plays an important role in the electronics industry for printed circuit boards and other electronic components [13].

In recent years, the development of composite coatings has been an active area of research [16]. Traditional uses include composite electrodeposition, Ni-SiC or Co-WC for wear resistance, Ni-PTFE, Ni-MoS_2 or Ni-WS_2 for combined self-lubrication and wear resistance [14]. Electro-deposition is often used to apply copper, nickel, tin and zinc coatings [15]. Electroplating coatings are becoming increasingly popular in various industries due to their versatility and cost effectiveness. These coatings involve the addition of nanoparticles or other materials to the coating in order to improve its properties such as wear resistance or corrosion resistance[17], [18].

Tungsten disulphide (WS_2) is a two-dimensional layered material with particularly good lubrication properties [21], [22]. With the addition of WS_2 in nickel-based flow coatings, "cactus-like" structures are often observed, these structures have nodular rough surfaces [23], [24]. This special "cactus-like" nodular structure consists mainly of alloy particles deposited on the granules and WS_2 granules partially embedded in the alloy substrate. In recent years, tungsten disulphide has been used in composite coatings to improve resistance to friction, especially by using it as a solid lubricant [23], [24], [25], [26]. Roy et al. studied the Ni-WS_2 nano-composite film produced by pulse electroplating [25]. This composite film exhibits excellent solid lubrication properties with 1/5 of the coefficient of friction of steel substrates. Liu et al. fabricated electroplated Ni-Co/WS_2 composite coating [23]. Compared with the Ni-Co alloy coating, the Ni-Co/WS_2 composite coating shows that the coefficient of friction is reduced by 3/2 and the corrosion rate is reduced by about half. Furthermore, Tudela's team produced three different coatings by electroplating: Ni plating, Ni/h-BN composite coatings and Ni/WS_2 composite coatings [26]. Both h-BN and WS_2 coatings have lubricating properties. However, for the study of corrosion resistant nickel-based composite coatings, there are few reports on the preparation of composite deposits using WS_2 particles as dispersed phase material.

Ni-P-WS_2 coatings offer higher corrosion resistance than Ni-P coatings. This innovative combination opens new possibilities, especially in high-stress environments where corrosion and wear resistance are critical. The incorporation of tungsten disulphide (WS_2) nanoparticles into the electroplating process improves the tribological properties of the coating. WS_2 is a solid lubricant that reduces friction and improves wear resistance. The addition of WS_2 nanoparticles to the Ni-P matrix combines the corrosion resistance and wear properties of Ni-P with the lubricating properties of WS_2 and forms a composite coating that improves wear resistance [27]. The wear resistance of Ni-P-WS_2 coatings has been investigated in several research papers and their superior performance compared to other coatings has been emphasised. For example, electroless Ni-P-SiC-WS_2 composite coatings have been shown to exhibit excellent wear resistance and anti-friction properties [28].

In the study, you will examine the effect of Ni-P-WS_2 coatings on wear behaviour based on experimental results. Such studies are important for understanding the performance of coating materials and optimising them for use in specific industrial applications. Furthermore, determining in which conditions and applications Ni-P-WS_2 coatings are particularly effective can help you evaluate their potential for industrial use.

II. EXPERIMENTAL PROCEDURE

Ni-P-WS_2 composite coatings were produced by the flow-through method. A concentration of 0.1 g/L WS_2 was used in all coatings produced. Pulse current was used for flow coating. A total of 3 coatings with current densities of 13 A/dm^2, 20 A/dm^2 and 26 A/dm^2 were produced respectively. The solution containing WS_2 particles was mechanically stirred for 1 hour before the current coating and then ultrasonically dispersed for 2 hours to prevent agglomeration during the coating. The substrate (1040 steel) was mechanically polished using 240-400-800-1200 grit sandpaper. In the final process before current coating, the substrate steel was kept in HCl (hydrochloric) acid for 45 seconds to remove contaminations.

The plating electrolyte used in the fabrication process was a nickel sulphate bath. Table 1. shown in the composition and range of the experimental process parameters. The WS_2 particle size used in the experiment was 1 micron. A 30 mm × 40 mm nickel plate was used as the anode and a 20 mm × 30 mm Steel substrate (AISI 1040) was used as the cathode. The bath was heated to 65°C, stabilised at pH 4 and stirred with a magnetic stirrer at a stirring speed of 250 rpm. The amount of surfactant (SDS) was adjusted to 200 mg/L.

Table 1. Bath compositions and electrodeposition conditions for Ni-P-WS_2 composite coating.

Nickel sulfate ($Ni_2SO_4·6H_2O$) (g/L)	180
Nickel chloride ($NiCl_2·6H_2O$) (g/L)	10
Phosphoric acid (H_3PO_4) (g/L)	20
Sodyumdodecyl sulfate (SDS) (g/L)	0.2
Phosphorous acid (H_3PO_3) (ml/L)	10
pH	4
Temperature (°C)	65
Current density (A/dm^2)	13, 20, 27
Stirring Speed (rpm)	250
Plating time (h)	0.75
Duty cycle	% 60

Following the electrodeposition process, the surface microstructures of the deposits were examined utilizing a scanning electron microscope (SEM, model JEOL – JSM 6060 LV). For surface and cross-sectional microstructure observations, the SEM was employed. XRD analysis was conducted using a Rigaku D/MAX/2200/PC X-ray diffractometer, scanning at a speed of 1°/min within the 2θ range of 10° to 100°. The hardness of the coatings was measured with a Vicker's microhardness indenter (Leica VMHT) under a load of 50 g.

To assess the reciprocating tribological behaviors, the coatings were tested by sliding against an Alumina ball (Ø 6mm) on a Tribometer (CSM Instruments) configured in a ball-on-disk arrangement following DIN 50 324 and ASTM G 99-95a standards. The wear tests were conducted under a constant applied load of 1.0 N and a sliding speed of 50 mm/s. The morphology of the wear marks was examined using SEM, and energy-dispersive X-ray spectroscopy (EDS) was employed to study the morphologies of the wear tracks.

III. RESULTS AND DISCUSSIONS

Surface SEM images of WS_2 reinforced Ni-P coatings produced at different current densities are given in Figure 1. The surface image of Ni-P-WS_2 composite coatings produced at current densities of 13 A/dm^2 in Figure 1a, 20 A/dm^2 in Figure 1b and 27 A/dm^2 in Figure 1 is given. As the current density increased, the size of the nodules decreased and the amount of particles entering the matrix increased. Since the WS_2 particles settled in the matrix create new nucleation areas, the shape and size of the nodules have also changed. In addition, in the coating with a current density of 27 A / dm^2, an agglomeration occurred due to the high amount of WS_2 particles.

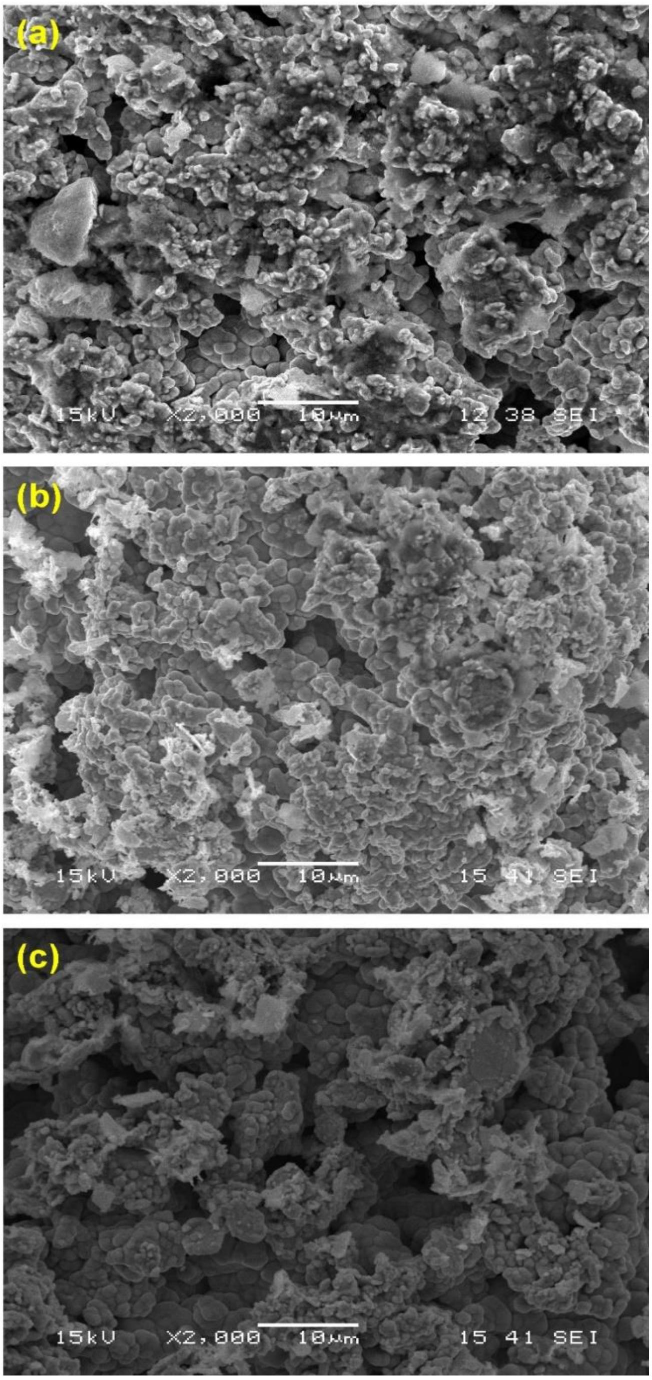

Fig.1. SEM pictures of (a) 13 A/dm^2 (b) 20 A/dm^2 (c) 27 A/dm^2

XRD analysis results of WS_2 doped Ni-P composite coatings with different current densities are given in Figure 2. XRD analyses showed that the structure of the coating is semi-crystalline-semi-amorphous and only peaks belonging to nickel and WS_2 materials were detected. The peak belonging to the (111) plane of nickel and located at $2\Theta=44°$ was seen in all three coatings, but a change in intensity occurred. The main reason for the change in the intensity of the peak belonging to the nickel (111) plane is the different amount of WS_2 particles entering the matrix.

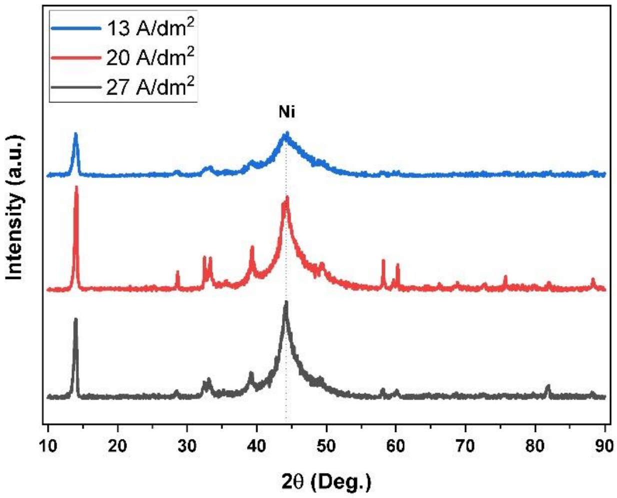

Fig. 2. XRD Patterns

The measured average hardness values of Ni-P-WS_2 composite coatings are given in Figure 3. When the current density was increased from 13 A/dm^2 to 20 A/dm^2, there was an increase in the hardness of the coating. When the current density was increased to 27 A/dm^2, a decrease in the hardness of the coating occurred. When the second phase particles added to the matrix cause dispersion hardening, hardness increase occurs. However, in the coating with a current density of 27 A / dm^2, porosities are formed in the matrix as a result of the agglomeration of WS_2 powders and the density decreases. This leads to a decrease in hardness.

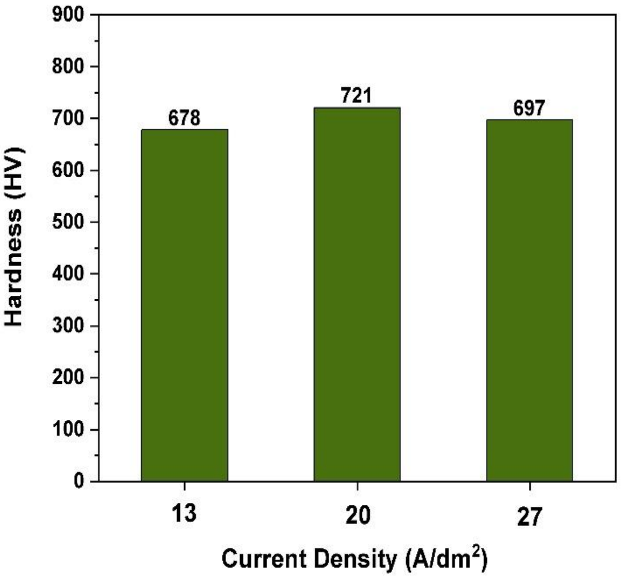

Fig. 3. Hardness of (a) 13 A/dm^2 (b) 13 A/dm^2 (c) 27 A/dm^2

The results of the wear tests performed at room temperature and dry environment are given in Figure 4. The average friction coefficient values of the composite coatings were measured as 2.98, 2.52 and 2.86 μ, respectively. The wear rates of the composite coatings were calculated as 4.97, 4.02 and 4.62 *10-7 mm^3/Nm, respectively. The lowest wear rate and coefficient of friction were obtained for the coating with a current density of 20 A/dm^2. In the coating with a current density of 27 A / dm^2, the wear rate value and coefficient of friction value increased due to the agglomeration of reinforcement powders.

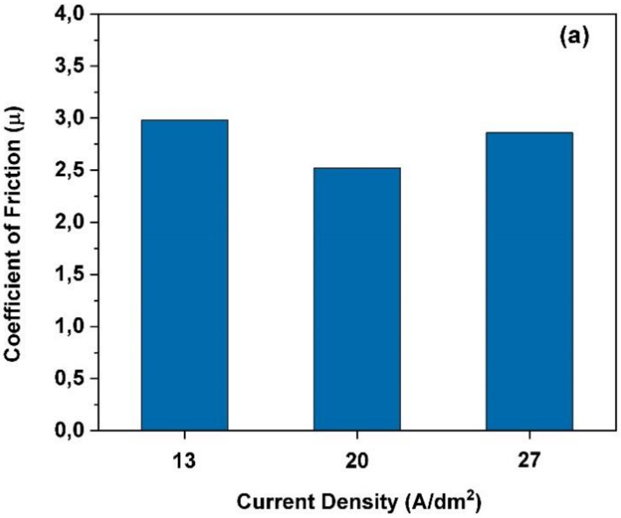

Fig. 4. Coefficient of Friction of 13,20,27 A/dm^2 Samples

The surfaces of the composite coatings after wear were imaged in SEM. The images at current densities of 13, 20 and 27 A/dm^2 are given in Figure 5, respectively. On the surface of the coating with a current density of 13 A/dm^2 (Figure 5a), large and continuous cracks as well as traces of plastic deformation were observed.

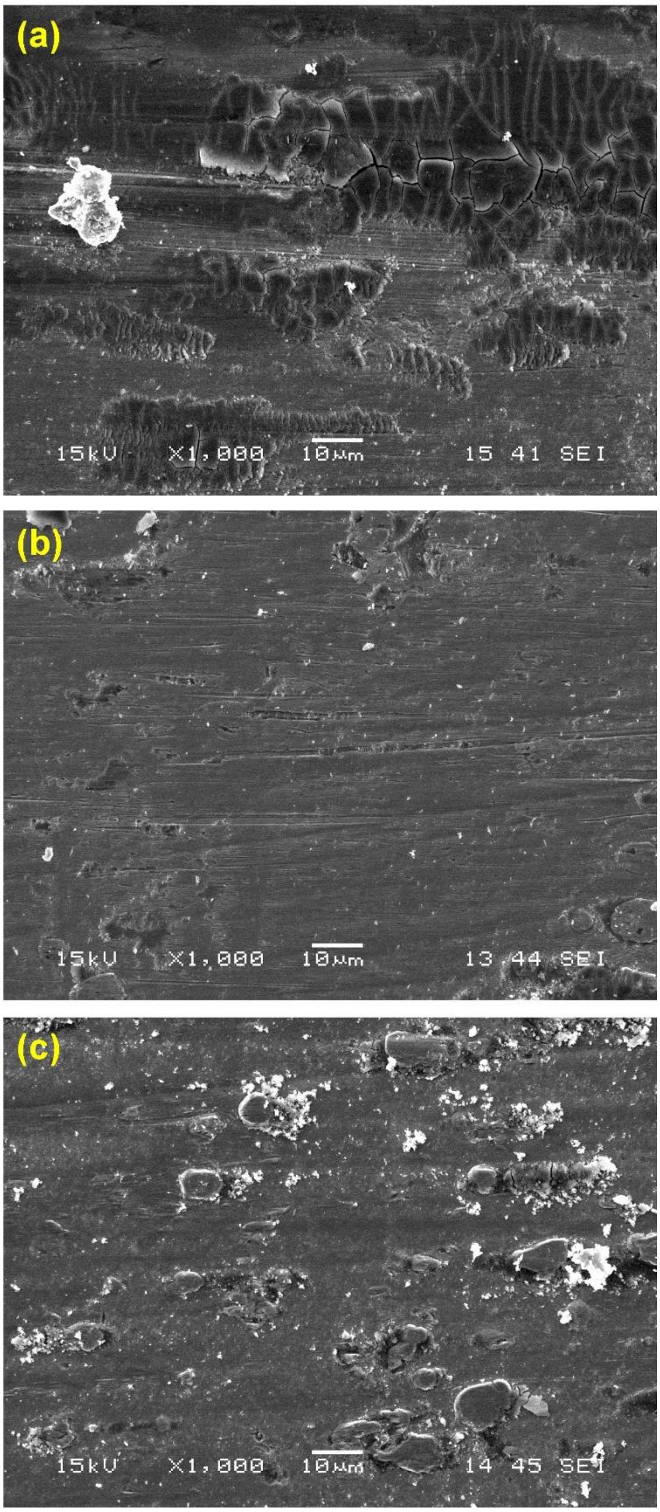

Fig. 5. SEM images of the wear track comparison of wear patterns for (a) 13 A/dm^2 (b) 13 A/dm^2 (c) 27 A/dm^2

IV. CONCLUSION

Ni-P matrix composite coatings were successfully fabricated at different current densities. Differences in the morphology of the coating were observed with WS_2 reinforcement. When the current density was increased from 13 to 20 A/dm^2, the hardness of the coating increased. However, when the current density was further increased, a decrease in the hardness value was observed. The best wear performance and wear resistance were observed in the coating with a current density of 20 A/dm^2.

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