J. Electrochem. Sci. Eng. **15(4)** (2025) 2619; http://dx.doi.org/10.5599/jese.2619

Open Access : : ISSN 1847-9286
www.jESE-online.org

Original scientific paper

Pulse electrodeposition of nickel-nitride composite coating and its oxidation under heating treatment

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Received: December 29, 2024; Accepted: May 3, 2025; Published: June 3, 2025

Abstract

Nickel-based composite coatings exhibit increased mechanical and oxidation properties due to utilization of reinforced particles as co-deposits within the nickel matrix. Meanwhile, pulse current electrodeposited composite coatings have higher mechanical properties compared to direct current electrodeposited coatings, since they provide a smaller electric force by which nickel ions are captured by particles and inhibit the grain growth of nickel. However, particles near the cathode surface cannot be reloaded in time, leading to lower adsorption of nickel ions on the particle surfaces. In this study, Ni-AlN composite coatings were developed using pulse current electrodeposition at 0.4 to 0.8 mA mm^{-2}. The surface modification of the composite coating was performed by using microwave heating at a temperature of 700 °C for 2 hours. The results showed that Ni and AlN reveal cubic crystal structures of the composite coating. The highest hardness coating was obtained by the sample deposited at 8 mA mm^{-2}. After microwave heating, the surface morphology is composed of microspheres and irregular particles. XRD analysis results suggested that the high temperature oxidation of Ni-AlN composite coating causes the formation of nickel and aluminium oxide. Post-heated coating samples showed a higher hardness than pre-heated. However, for samples deposited at the highest pulse current density, the hardness is lower due to the formation of a weak particle-matrix interface.

Keywords

Nickel-based composite; pulse current density; microwave heating; coating microstructure; coating hardness

Introduction

The pulse current electrodeposition has offered an advantage compared to the direct current electrodeposition in enhancing mechanical and oxidation properties of nickel-based composite coating [1-4]. The technique is better in controlling the adsorption of particles that influence the crystallite size and particle concentration. Pulse current electrodeposition was applied to produce Ni composite coating and it was found that the best wear resistance relates to the structure and particle content influenced by pulse current [5]. The pulse current enhances the nucleation process and inhibits lateral growth of the matrix crystal, providing finer morphology and strength of the grain boundaries [4]. Repeating pulse deposition allowed the reinforced particles sedimented outside the deposition area to redisperse and incorporate into the coating area [2].

The key to the improvement of nickel composite coating properties is the addition of the reinforced particles into the nickel matrix as the second phase of the composite system. Typical particles used as the second phase of nickel-based coatings are nitride particles such as TiN, AlN and Si_3N_4 that improve their hardness, oxidation, and wear resistance. Other particle types include oxides and carbides that are frequently used due to their consistent physical and chemical characteristic [1]. The second phase particles are used as reinforced elements that are embedded and dispersed in the metal matrix [6]. Smaller size of nitride particles induces more homogeneous and compact coating, leading to an increase in coating hardness [7]. Another type of reinforced particles of carbide (SiC) effectively protects the matrix through the formation of a mechanical mixing layer [8]. The oxide particles, such as CeO_2, improve the coating properties by forming a crystallization nucleation core of alloy matrix of the composite [9].

The incorporation of nitride as well as oxide, carbide or ceramic into nickel-based composite coating forms various combinations of metallic, covalent and ionic bonds. In Ni-superalloy fabrication for high-temperature strength applications, the bonding among complementary phases is required by forming a wavy structure at the nanoscale interface [10]. The wavy interface is provided by the random orientation of the particles. High hardness is achieved due to the incorporation of ultrafine grains, intermetallic particles, and the amorphous phase as reinforcement for the composite coatings [11]. In this study, the amorphous phase exhibits higher hardness and strength of the coating compared to the crystalline phase.

The anti-corrosion performance of pulsed composite coatings is due to the incorporation of particles occupying the crevices, gaps and pores or holes in the matrix [4]. The uniformly distributed carbide and nitride particles contribute to the improvement of corrosion resistance by filling the cracks and gaps. The pulse frequency and duty cycles enhance the particle migration and eventually improve the particle amount in the composite [3]. High temperature oxidation is challenging for nickel-based composite coating application, since the oxygen molecule can easily diffuse into the coating. High temperature oxidation rate depends on the reaction and diffusion process. However, the improved performance is reached by controlling the initial oxidation process on the surface and the mass transfer process within the material [12].

Surface modification of coating materials is a challenge for future manufacturing industries in improving their performance, especially in high-temperature applications [13]. The problem with the composite coatings at elevated temperatures is particle erosion. This problem can be diminished by modifying the coating microstructure during the deposition process and post process. An alternative option for enhancing the mechanical characteristics of composite coatings is the implementation of post-heat treatment [14]. The excellent wear and corrosion resistance are achieved by the formation of the oxide layer and the intermetallic compound on the coating surface.

The microwave heating has already been found to improve the coating wear resistance due to the intermetallic formation and metallurgical bonding [15].

In this study, Ni-AlN composite coating was processed using pulse electrodeposition. AlN particles were added as a reinforcement and further fused by using a microwave heating process at high temperature. The high temperature heating process has two objectives: first, to investigate the high temperature oxidation process and second, to modify the microstructure of composite coatings. The investigation is done to provide support for discovering a new nickel-based alloy composite coatings with high strength at high-temperature applications.

Experimental

Materials

In this study, the substrate material used is tungsten carbide (WC) plate (Widia 78-2 YG6) with a dimension size (W×D×) of 4×4×38 mm. Electrolyte bath for electrodeposition consisted of 100 g l^{-1} $NiSO_4 \cdot H_2O$, 40 g l^{-1} $NiCl_2 \cdot 6H_2O$, 6 g l^{-1}AlN, 30 g l^{-1} H_3BO_3 and 0.6 g l^{-1} SDS (sodium dodecyl sulphate). All powders were mixed with 5 ml of aquadest and magnetically stirred (NescoLab, MS-H280-Pro, Taiwan) for 3 hours. The electrochemical cell consisted of a Pt wire as a counter electrode and tungsten carbide bar as a substrate or working electrode, respectively.

Deposition process

Before the deposition process, the substrate was polished by using 400, 800, 1000 and 1500 mesh sandpapers and then rinsed in water. After that, the substrate was cleaned by using an ultrasonic cleaner and dried on a hot plate. The electrodeposition process of Ni-AlN composite coating was performed by varying pulse current densities of 0.4, 0.6 and 0.8 mA mm^{-2} for 30 minutes. Pulsed current was supplied by a stable circuit with IC 555 that generates a square or rectangular output signal with a duty cycle that can be adjusted from 0 to 100 % and connected to a DC power supply (Sanfix 30 V/5A). The times on and off were set at 1 and 10 s, respectively. The current passing through the electrolyte was controlled by a low duty cycle (longer off-time) to provide more time for ions to be adsorbed at the surface. During deposition, the bath was stirred by a magnetic stirrer and kept at a temperature of 40 °C. The experiment set-up is presented in Figure 1.

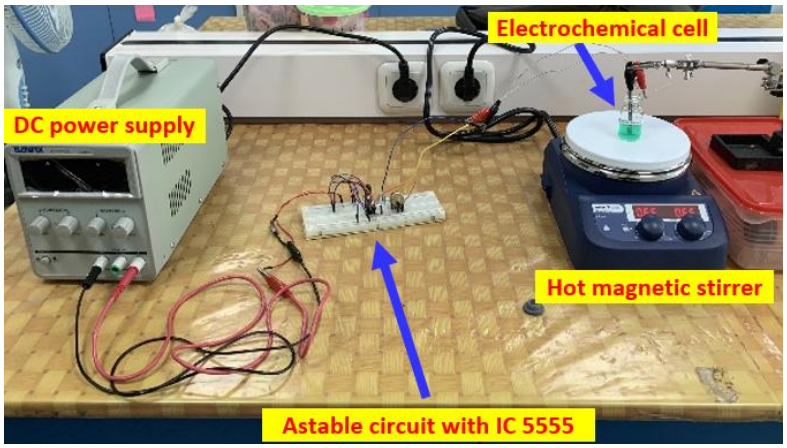

Figure 1. *Apparatus set up for pulse electrodeposition*

Heat treatment

The samples were heated by a microwave oven at a temperature of 700 °C for 2 hours under atmospheric conditions. Before heating, the samples were cleaned by an ultrasonic cleaner in an

acetone solution for 20 minutes and dried on a hot plate. The samples were covered by aluminium foil during the heating process to eliminate spark fire and were positioned properly on the refractory brick. The microwave oven (Samsung ME731k) was set up at a power of 450 W.

Characterization

The surface morphology, surface roughness and chemical composition of samples were characterized by a scanning electron microscopy (SEM) (JEOL-JSM 6510LA, Japan), coupled with an energy dispersive spectroscopy (EDS). The crystal structure of the samples was characterized by X-ray diffraction (Panalytical Empyrean Philips, UK). The grain size of Ni-AlN composite coatings was calculated based on Scherrer's Equation (1):

$$d = k\lambda / B \cos \theta \quad (1)$$

where d is the grain size of Ni-AlN composite coatings, k = 0.89, λ is the wavelength of X ray, B is the breadth of the diffraction peaks at half height (FWHM) and θ is the Bragg diffraction angle. The samples were tested by Vickers hardness with a 1000-gram load (Mitutoyo microhardness tester HV-100), Japan, used to obtain the indentation measurement. Three different indentation locations on the surface samples were performed to obtain the average coating hardness.

Results

Surface morphology and composition of coatings

SEM micrographs of samples electrodeposited with different pulse current densities are shown in Figure 2. A fine morphology with small grain size is seen on samples deposited at low pulse current (Figure 2a). However, as the pulse current increased, the grain grew and bulged on the surface (Figure 2b and 2c).

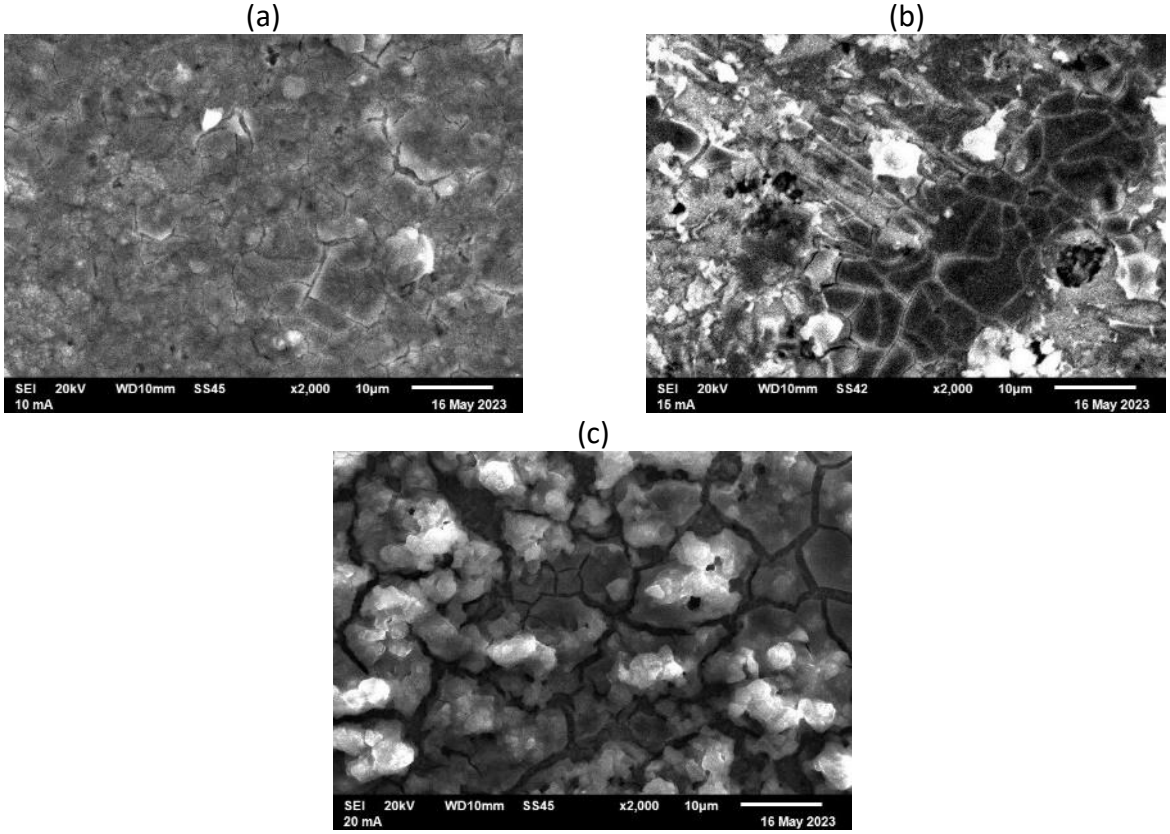

Figure 2. *SEM micrographs of Ni-AlN composite coatings electrodeposited with different pulse current densities: (a) 0.4, (b) 0.6 and (c) 0.8 mA mm^{-2}*

EDS element analysis showed that Ni content tends to decrease, while Al content tends to increase as the pulse current increases (Figure 3). The bulge grains of surface morphology are caused by the increase of Al content, which increases AlN phase and inhibits the nickel crystal growth further. It was already reported that nickel nucleation rates are directly related to the pulse current density, *i.e*. the nucleation rate increases as the pulse current density increases [5]. Higher pulse current density generates a higher electric field, increasing the probability of the Ni ion being trapped by the nitride particle. In this study, the content of AlN that traps Ni ions increases as the pulse current density increases and continues to grow, producing the bulge grains on the surface.

The composite coating obtained by pulse current electrodeposition shows a smooth surface and smaller grains than that obtained by direct current electrodeposition [4]. The surface is also compact due to higher current density and stronger cathode polarization of pulse current than of direct current electrodeposition, providing a faster nucleation rate [16].

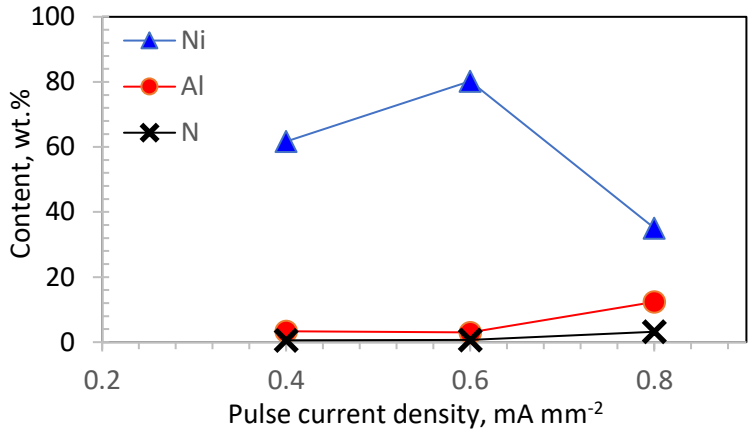

Figure 3. *Mass percentages of elements in Ni-AlN composite after electrodeposition at various pulse current densities*

The surface morphologies of Ni-AlN composite coatings after heating for 2 h at 700 °C are shown in Figure 4. The rough surfaces are shown by samples deposited at 0.4 and 0.6 mA mm^{-2}, while a smooth surface is shown by a sample deposited at 0.8 mA mm^{-2}, where some pores could also be observed (Figure 4a, 4b and 4c). It has already been reported that the microwave heating process has a positive effect in eliminating microcracks, pores, and voids due to the melting of unmelted particles [15]. It was also reported that the formation of pores or voids is due to the diffusion coating in nickel-based alloys [17]. The outward and inward diffusion of the element during high temperature oxidation may not be balanced, providing the vacancies and void formation as occurred on this sample (see Figure 4). The coating sample deposited at 0.8 mA mm^{-2} showed no boundary separation due to blending grains induced by microwave heating. The microwave heating causes the formation of the molten zone that decreases the surface tension of the grains and accelerates the intermixing of elements [18]. The pores and voids on the coating surface are reduced because the pores have been oxidised on exposure to high temperatures during microwave heating [15]. However, unmelted/ /partially melted particles are observed on the coating surface, indicating the heterogeneous structure formation.

Further analysis on the coating composition of metal oxide after microwave heating is shown in Table 1. It shows that Ni and Al elements are still detected even after the microwave heating, indicating their role in material protection ensured by nickel and aluminium oxide formation at temperature oxidation [19,20]. This result proves that Ni and Al are important elements in high-temperature oxidation resistance by capturing the oxygen molecules that diffuse into the coating.

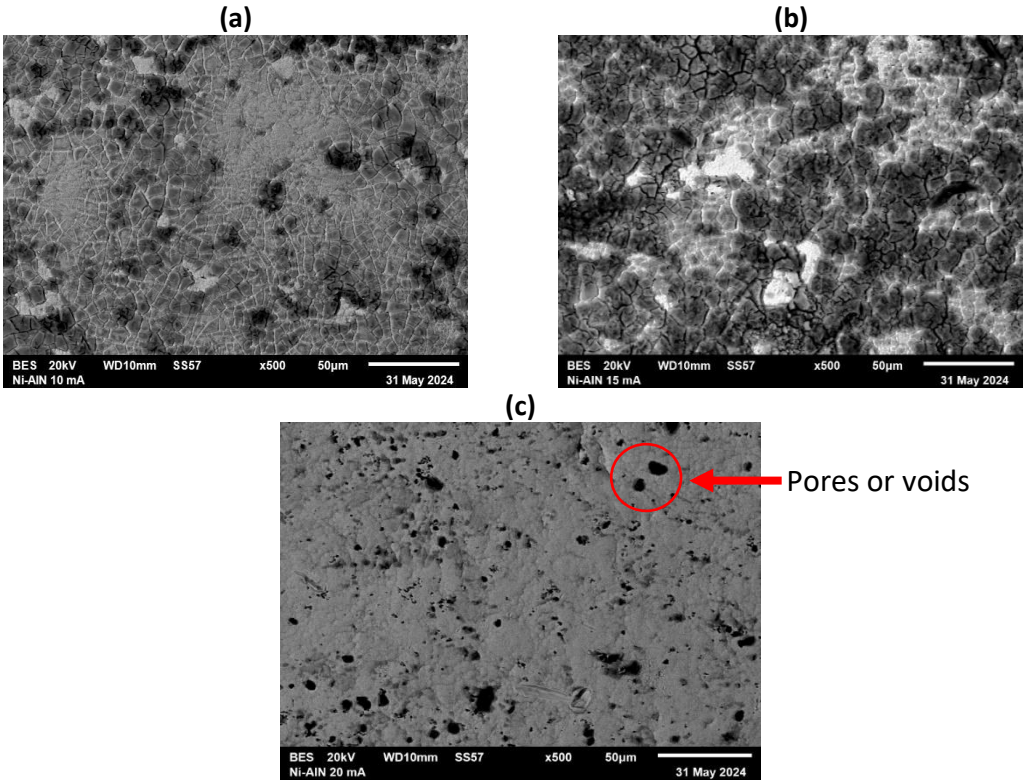

Figure 4. *Surface morphology of Ni-AlN composite coatings after microwave heating at 700 °C for 2 hours for samples electrodeposited at pulse current densities: (a) 0.4, (b) 0.6 and (c) 0.8 mA mm^{-2}*

Table 1. *Content of metal oxides of Ni-AlN composite coatings after microwave heating of samples electrodeposited at different pulse current densities*

Pulse current density, mA mm^{-2}	Content, wt.%	
	NiO	Al_2O_3
0.4	75.61	0.85
0.6	61.30	22.41
0.8	99.13	0.87

EDS analysis on oxide compound composition was done by the ZAF Method Standardless Quantitative Analysis and presented in Table 1. It shows that the content of nickel oxide is generally higher than that of aluminium oxide. It was reported that the formation of aluminium oxide at high temperatures might be caused by the reaction of aluminium with nickel oxide, which leads to the reduction of nickel content [21]. It is known that the free energy of nickel is higher than aluminium, thus, at high temperatures, the nickel reaction with oxygen is faster than that of aluminium. When the nickel oxide content reduces, the aluminium oxide content is increased.

Crystal structure

The effect of pulse current density on the phases and constituents of Ni-AlN composite coatings is shown in Figure 5. Two diffraction peaks of Ni (111) and Ni (002) are of high intensity in coating samples deposited at pulse current densities of 0.6 mA mm^{-2} and 0.8 mA mm^{-2}. The results show that grains of the nickel phase grow as the pulse current density increases, as indicated by the increase in intensity peaks of Ni (111) and Ni (002). According to calculations based on XRD data and Equation (1), the average grain size for Ni crystal is about 31.03 nm and is relatively constant even the pulse current density is increased (Figure 6). Meanwhile, the size of AlN crystals tends to increase with increasing pulse current density, showing an average size of 48.58 nm. The results are consistent with those from the SEM results.

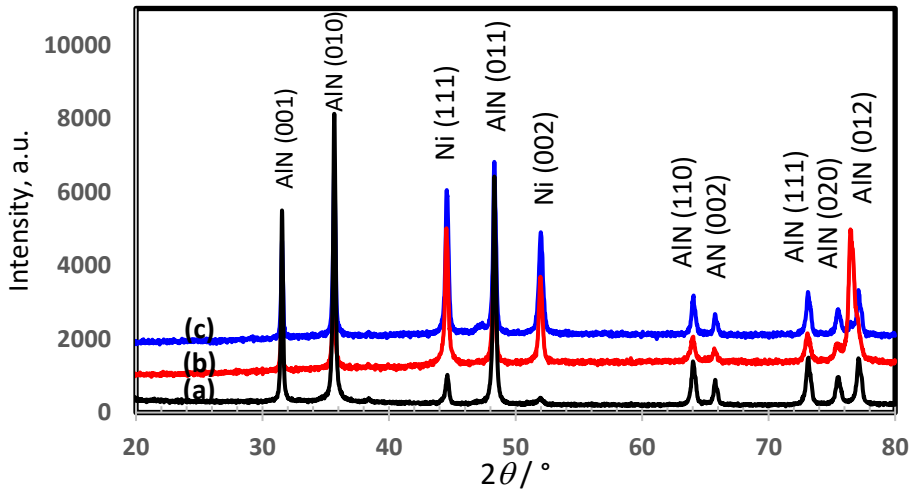

Figure 5. *XRD patterns of Ni-AlN composite coatings electrodeposited at pulse current densities of (a) 0.4, (b) 0.6 and (c) 0.8 mA mm^{-2}*

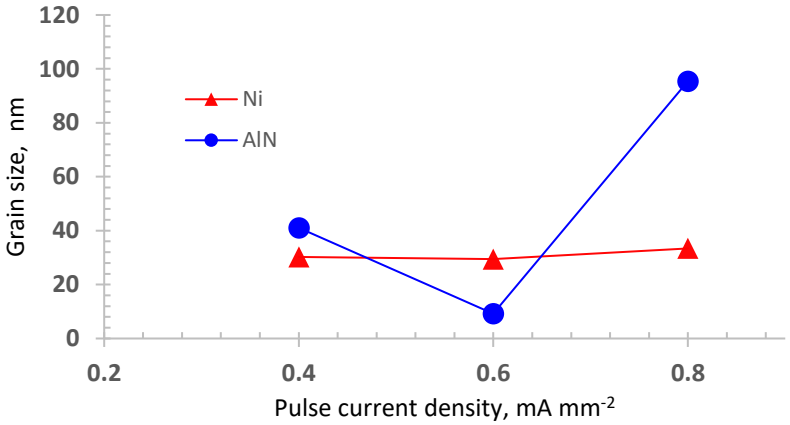

Figure 6. *Average grain size of crystals in Ni-AlN composite coatings before microwave heating of samples electrodeposited at various pulse current densities*

As was already said above, post-deposition treatment of the samples was carried out by microwave heating at a temperature of 700 °C for 2 hours. The objectives were to treat samples by high-temperature oxidation, as well as to modify the coating microstructure. XRD results showed that Ni and Al elements of Ni-AlN composite coatings remain after the oxidation process, indicating the formation of nickel oxide and aluminium oxide (Figure 7).

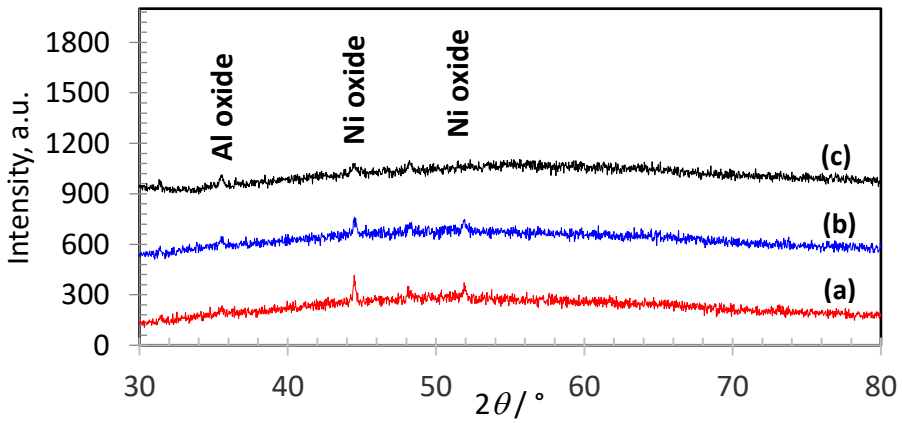

Figure 7. *XRD spectra of Ni-AlN composite coatings after microwave heating at temperature of 700 °C for 2 hours. Pulse current densities: (a) 0.4, (b) 0.6, and (c) 0.8 mA mm^{-2}*

The result is in line with the previous result [22]. It was also reported that at high temperatures of 600 to 1000 °C, the formation of nickel oxide (NiO) and aluminium oxide (Al_2O_3) occurs and prevents diffusion of oxygen and metal ions and therefore inhibits oxidation [19,20].

Hardness

The results of hardness testing for Ni-AlN composite coatings before and after the microwave heating process are presented in Figure 8. An example of Vickers measurement is presented in Figure 9. Ni-AlN coated sample has the highest hardness value when the pulse current density is increased up to 0.8 mA mm^{-2}. After the heating process on the samples, however, the hardness of this coating was decreased, whereas the hardness of coatings deposited at a pulse current density of 0.4 to 0.6 mA mm^{-2} are increased. Before the heating process, the increase in coating hardness as the pulse current density increases due to the increase in AlN content. Based on the morphology analysis result, the nitride grains (such as AlN) bulge out of the metal matrix, inhibiting the load transmission from the matrix and leading to the improvement in mechanical properties [23]. During the heating process, the hardness of the coating can decrease. It is explained that the interface bond between particles and matrix could produce a weak particles-matrix interface and cause a decrease in hardness [6]. When the particle content increases, the surface energy between the substrate and particles increases, leading to a decrease in hardness. In this study, the sample that is deposited at a pulse current density of 0.8 mA mm^{-2} has lower hardness compared to other coating samples, due to its higher AlN content. At the heating process, the melting phase in the particle-matrix interface, which contains higher AlN particles, produces a weaker particle-matrix interface.

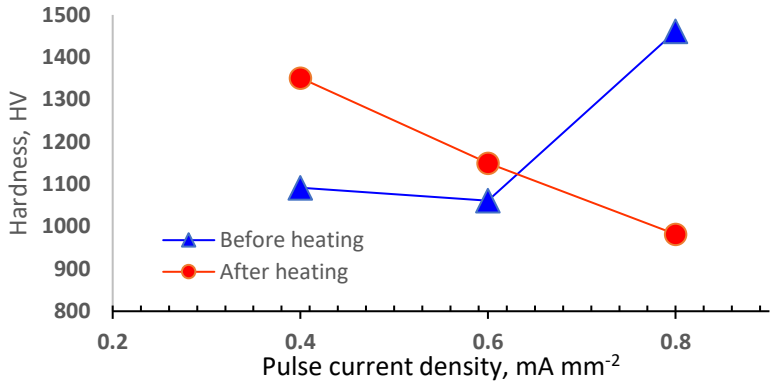

Figure 8. *Hardness of Ni-AlN composite coatings electrodeposited at various pulse current densities, before and after microwave heating process*

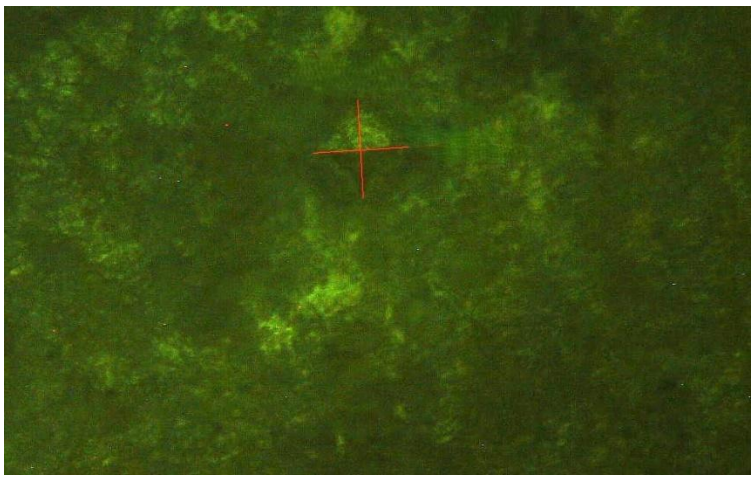

Figure 9. *Vickers hardness of Ni-AlN composite coating before oxidation process for the sample electrodeposited at pulse current density of 0.4 mA* mm^{-2}

Discussion

The application of pulse electrodeposition for developing nickel composite coating provides higher hardness than direct current electrodeposition. The key factor of improvement controls the particle nucleation that is embedded as a reinforced second phase or precipitate within the nickel matrix [24]. The deposition method is also a key factor since the microstructure of the composite coating can be controlled by process parameters. Pulse electrodeposition is one of the methods for development of nickel composite coatings since it is simple and allows for high-rate production. The parameter of electrodeposition current is an important factor since, based on Gugliemi's model, it controls the motion and adsorption of nickel and particle ions to the surface that is forced by the electric field [6,25] (Figure 10). It is reported that in the pulse electrodeposition, the current passing through the electrolyte can be controlled, where at a longer off-time (low duty cycle more time is provided for ions to arrive at the double layer before being adsorbed by the surface. Meanwhile, in the case of direct current, the continuous current passes through the electrolyte, causing less time for ions to adsorb at the surface [4].

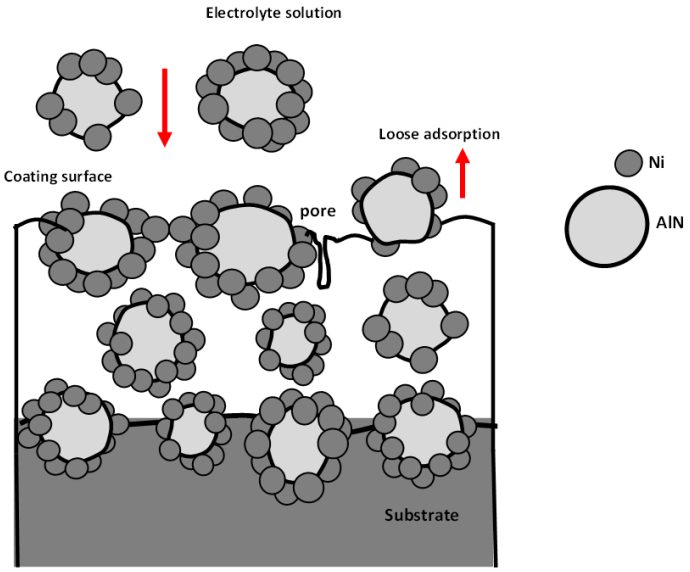

Figure 10. *Schematic diagram of the electrodeposition process of Ni-AlN composite coating based on Gugliemi's model [6]*

The absorbed nickel and particles on the substrate enhance as the pulse current density is increased. As the absorbed particles increase, the particle nucleation also increases, resulting in finer coating grain and eventually increasing the coating hardness. It is known that the particles act as a growth inhibitor of matrix grains [6]. In this study, the nitride particles (AlN) act as a growth inhibitor of the nickel matrix. As the pulse current is further increased, the nitride particle grain grows, resulting in a bulge morphology and inhibition of the grain growth of the nickel matrix. AlN particles tend to form conglomerates uniformly distributed on the surface, and some of them protrude above the surface [26]. The structure analysis result shows that the AlN particles are successfully embedded in the matrix composite. It is shown that the intensities of Ni (111) and Ni (220) increase as the pulse current is increased. Based on the grain size calculation, the nickel grain size increases as the pulse current density increases. However, the increase in nitride particle grains is much larger than that of nickel at high pulse current density. Thus, it seems that the pulse current density has little effect on the grain growth of the nickel grain size due to the high increase of nitride particle grain growth, which inhibits the nickel grain growth.

In this study, the highest hardness is achieved for the sample deposited at the highest pulse current density. It is reported that the nucleation rate increases as the pulse current is increased, resulting in grain refinement. The nucleation rate of nickel is directly related to the pulse current density and increases as the pulse current density is increased [5,27]. Based on Hall-Pecth relation, the high hardness is contributed by grain refinement. Furthermore, the even distribution of nitride particles in the nickel matrix creates a denser microstructure with reduced flaws like empty spaces and displacements contributing to the coating hardness [28]. In this study, as the pulse current is increased, the nickel grains are constantly small compared to the increased nitride grains, resulting in a bulge surface morphology and leading to an increase in hardness due to denser microstructure (Figure 2c). From the chemical composition analysis, the nickel content decreases while nitride content increases, indicating that nitride particles suppress the nickel grain growth as the pulse current increases. The use of pulse current also has an advantage in releasing hydrogen gas during the deposition process [29]. As the pulse current is increased, more hydrogen gas is released, resulting in an increase in hardness.

The microwave heating process improves the mechanical properties [15,30]. When the microwave heating process is applied, the surface morphology becomes finer due to the melting process of occupied particles at the grain boundaries of the matrix composite, which blends the grains and eventually improves the coating hardness. However, in this study, the hardness of nickel composite coating after the microwave heating process is somewhat higher than before heating, except for the sample deposited at high pulse current density. Besides partially melted particle surface, there are unmelted surface particles, which could result in a weak particle-matrix interface and cause lower hardness [15]. In this study, the microwave heating process generates the formation of nickel and aluminium oxide, which also contributes to the coating hardness through partially melted oxide surface at grain boundaries, resulting in blended grains. However, metal oxide is an effective microwave absorber that generates nonuniform heating and reduces the dissipation power and eventually produces a poor product [30,31]. It seems that for samples deposited at high pulse current density and high metal oxide content, most particles are unmelted. These unmelted particles provide a weak interaction at the matrix-particles interface and cause low hardness.

Conclusions

Ni-AlN composite coatings were produced by the electrodeposition process using various pulse current densities. The post-treatment of the coating samples was carried out by the microwave heating process. The surface morphology, chemical composition, crystal structure and hardness of the coatings were examined before and after the heating process. It was observed that AlN particles were incorporated into Ni composite coating, providing a bulge surface morphology as the pulse current density is increased. This is due to the increase in AlN content and grain growth. After the microwave heating process, the surface morphology of the composite coating becomes smoother, especially for the coating sample deposited at high pulse current density, due to interface bond formation between particles and matrix. After the microwave heating process, the nickel and aluminium oxides are formed, indicating their important role in high-temperature oxidation protection. Generally, the coating hardness is higher after microwave heating, except for the coating sample deposited at the highest pulse current density, due to higher particle content that produces a weak particle-matrix interface.

Acknowledgements*: The study is financially supported by DRTPM with contract No. 8/UN39.14/PG.02.00.PL/PFR/VI/2024 and BLU UNJ No with contract No. 8/KI/LPPM/III/2024.*

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