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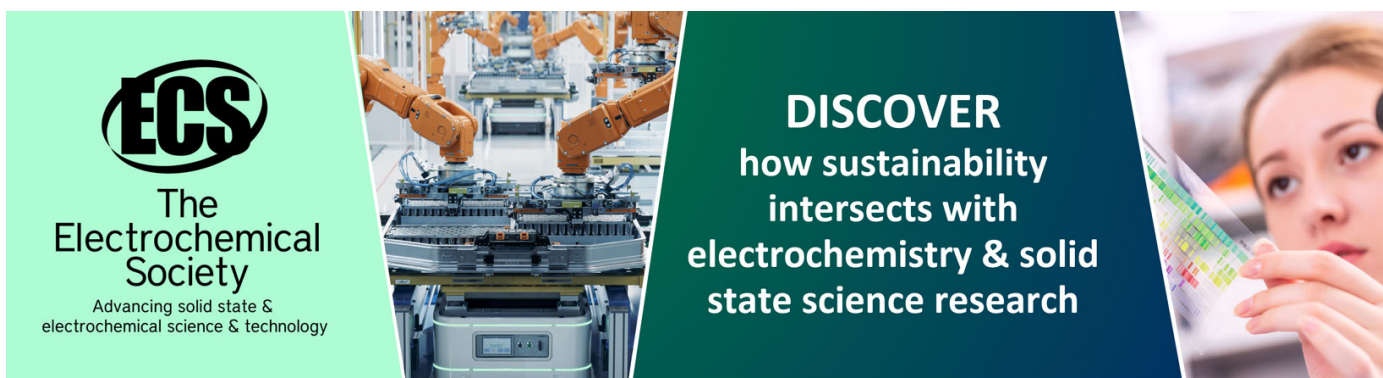
Structure and mechanical properties of electrodeposited Ni-AlN/Si₃N₄ composite coating

To cite this article: Esmar Budi *et al* 2019 *J. Phys.: Conf. Ser.* **1317** 012050

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Structure and mechanical properties of electrodeposited Ni-AlN/Si₃N₄ composite coating

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Abstract. Ni-AlN/Si₃N₄ composite coatings were successfully deposited on tungsten carbide by electrodeposition process. The influence of Si₃N₄ powder concentration on the microstructure, crystal structure and mechanical properties was investigated by using Scanning Electron Microscopy, Energy Dispersive Spectroscopy, X-Ray Diffraction and Vickers hardness tester, respectively. The results revealed that variation of Si₃N₄ concentration influenced the structure and mechanical properties of Ni-AlN/Si₃N₄ composite coatings. The fine surface morphology of composite was due to the inclusion of AlN and Si₃N₄ particles while it is rougher as the Si₃N₄ concentration was increased due to agglomeration effect and Ni crystal growth. The increase of microhardness is related to the particles dispersion-strengthening.

1. Introduction

The incorporation of hard particles such as AlN particles as a reinforcement in the metal matrix that successfully has improved the mechanical properties of composite [1-3]. It was reported that theoretically and experimentally, alloying AlN with transition metal such Ti in form TiAlN coating may improve its thermal stability and hardness for hard coating application due to AlN phase transformation [4,5]. However, the thermal stability of coating hardness decreases as the temperature increase up to 1000 °C. The further improvement of TiN and TiAlN coating properties, especially its mechanical property, has been done by introducing silicon nitride Si₃N₄ into the coating which the thermal stability of coating hardness reached until above 1000°C. [6].

Recently, the development of hard coating by using electrodeposition has been done [7]. The increase of coating microhardness was attributed to the incorporation of Si₃N₄ particles in metal matrix and led to the grain refinement of coating. It could fill crevices and micron holes thus enhance the effect of physical barrier in the composite coating [8]. Therefore, in this study, Si₃N₄ particle was selected as strengthening phase for the modification of Ni-AlN composite coating. The aim of this study is to investigate the effect of Si₃N₄ particles concentration variation on Ni-AlN/Si₃N₄ composite coating properties in term of structure and mechanical properties.



2. Experiment

The experiment of this study was based on our previously studies in development of nickel-nitride composite coating [3,10,15]. Ni-AlN/Si₃N₄ composite coating was prepared by electrodeposition in electrolyte containing 0.38 M Ni₂SO₄.6H₂O, 0.17 M NiCl₂.6H₂O, 0.49 M H₃BO₃, 6 gr/lit Sodium Dodecyl Sulfate (SDS), 4 gr/lit AlN and various concentration of Si₃N₄ from 20, 30 and 40 gr/lit. The coatings were deposited on Tungsten Carbide (WC) bar of 4 mm x 4 mm x 3.8 mm dimension as substrate and previously was washed by detergent and cleaned by using ultrasonic cleaner. Subsequently, the substrate was dried on hot plate and cleaned again by using ultrasonic cleaner in 96% alcohol solution. The electrodeposition process was carried out by potentiostat system with fixed current of 4 mA and voltage of 12 volt. The electrodeposition cell consists of Pt and AgCl wires were used as counter and reference electrodes, respectively. The surface morphology and composition of composite coatings were characterized by using Scanning Electron Microscopy (SEM) JEOL-JED 2300 and Energy Dispersive Spectroscopy (EDS), respectively. Meanwhile, the crystal structure and hardness were characterized by using X-Ray Diffractometer (XRD) Shimadzu 7000 (Cu K α , λ =1.54 Å, 40 kV, 30 mA) and Micro Vickers hardness tester Leco LM 800AT, respectively.

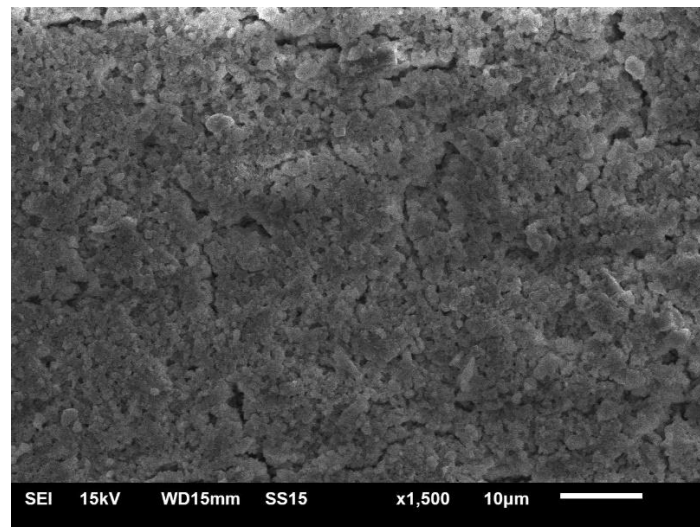
3. Results and discussion

3.1. Surface Morphology

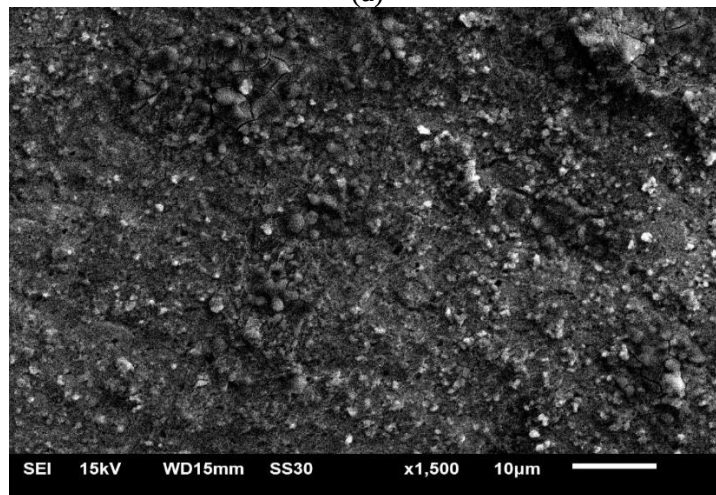
Figure 1 displays the surface morphology of Ni-AlN/Si₃N₄ composite coating on Tungsten Carbide (WC) bar with different Si₃N₄ concentration. It is clear that all the coatings are uniform coverage the substrate. However, there are some holes and crack on the surface coating and it might be detachment of incompletely adhered AlN cluster [7]. At low Si₃N₄ concentration (20 gr/lit), the surface morphology is relatively smooth, and it becomes rough as the Si₃N₄ particles concentration is increased due to agglomeration formation onto the surface. The fine surface morphology was mainly attributed by the grain refinement due to the inclusion of AlN and Si₃N₄ particles and act as nucleation sites that restrict the crystal grain growth [2,8,9]. In this study, AlN and Si₃N₄ particles are incorporated into Ni matrix lead the refinement of the coating surface morphology. The rougher coating surface at higher Si₃N₄ concentration is due to the agglomeration effect and grain growth [10]. It was reported that AlN particles tend to form conglomerate uniformly distributed onto the coating surface and some of them protrude above the surface of the nickel [11].

3.2. Composition

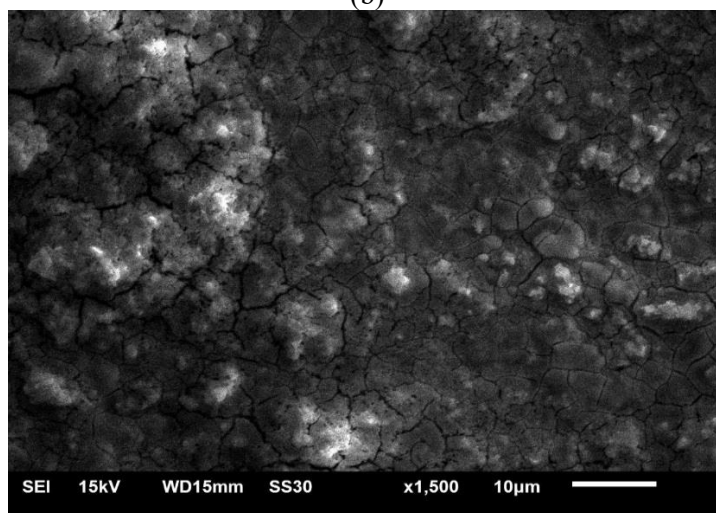
Figure 2 showed the composition of Ni-AlN/Si₃N₄ composite coating at different Si₃N₄ particles concentration that were analysed by using EDS. It showed that all main elements of the composite coating were detected such as Ni, Al and Si. It indicated that the AlN and Si₃N₄ particles were successfully deposited and dispersed homogeneously into Ni matrix. In fact, the incorporated particles to metal matrix depend on the electrodeposition condition. However, the decrease of the incorporated Si and Al particles content into composite with increasing Si₃N₄ concentration might be due to collision factor [7] and this phenomena led to the increase of Ni content and Ni crystal growth in the composite coating. A large particles content indicated large particles embedded in the composite coating [13] and by contrast, a low particle content indicated low particles embedded in the coating.



(a)

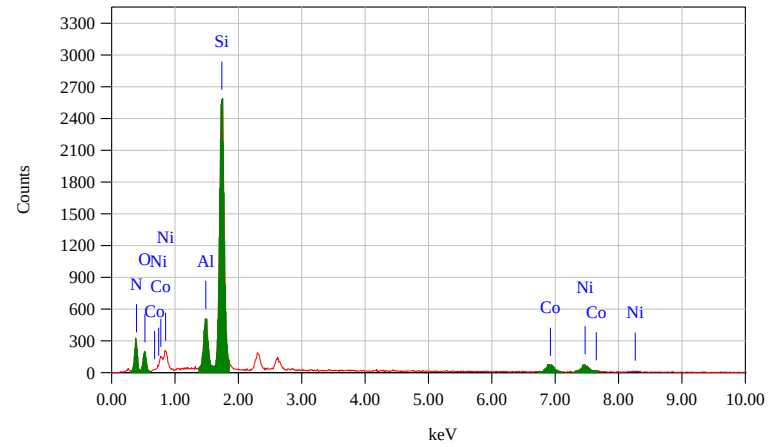


(b)

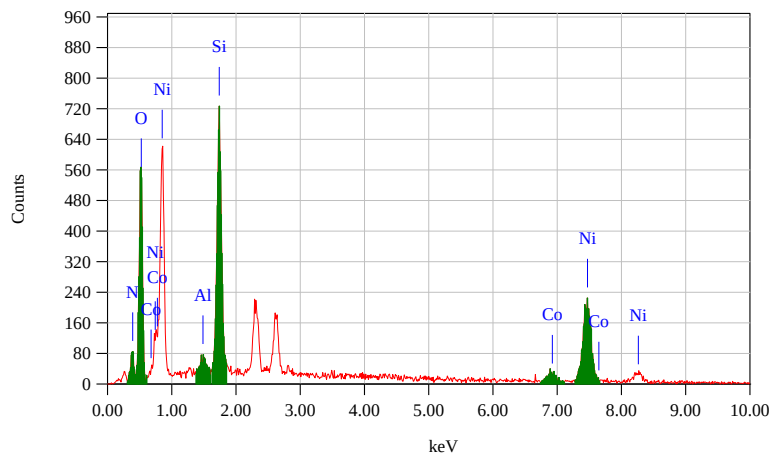


(c)

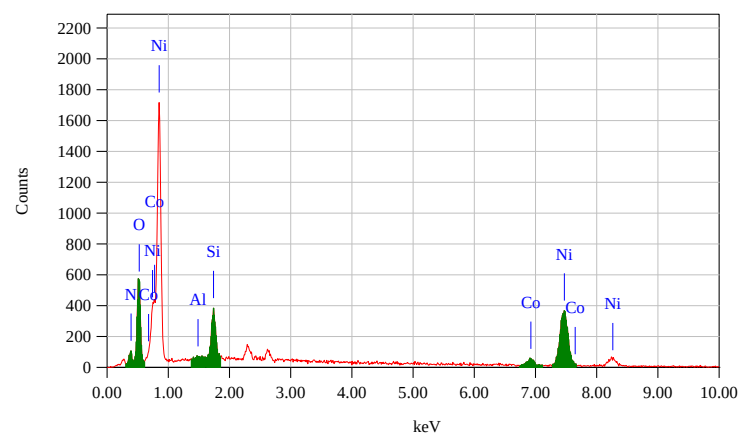
Figure 1. SEM images of Ni-AlN/Si₃N₄ composite coatings at different Si₃N₄ particles concentration.



(a)



(b)



(c)

Figure 2. EDS analysis on the composition of Ni-AlN/Si₃N₄ composite coatings at different Si₃N₄ particles concentration of (a) 20 gr/lit (b) 30 gr/lit (c) 40 gr/lit

3.3. Crystal Structure

Figure 3 displays the pattern of XRD spectrum of Ni-AlN/Si₃N₄ composite coatings at different Si₃N₄ particles concentration. All the peaks were identified by comparing the d-spacing value obtained by XRD pattern with AlN (010), AlN (111), AlN (012) and AlN (222) peaks appear at around $2\theta = 31.54^\circ$; 35.7° ; 48.32° and 76.6° , respectively. Ni (111), Ni (002) and Ni (022) peaks are observed at $2\theta = 44.5^\circ$; 52° and 77.1° , respectively. Meanwhile, Si₃N₄ (140), Si₃N₄ (012) and Si₃N₄ (141) are observed at $2\theta = 64.5^\circ$; 65.5° and 73.2° , respectively. Low intensities of Si₃N₄ phase indicated its amorphous nature [14].

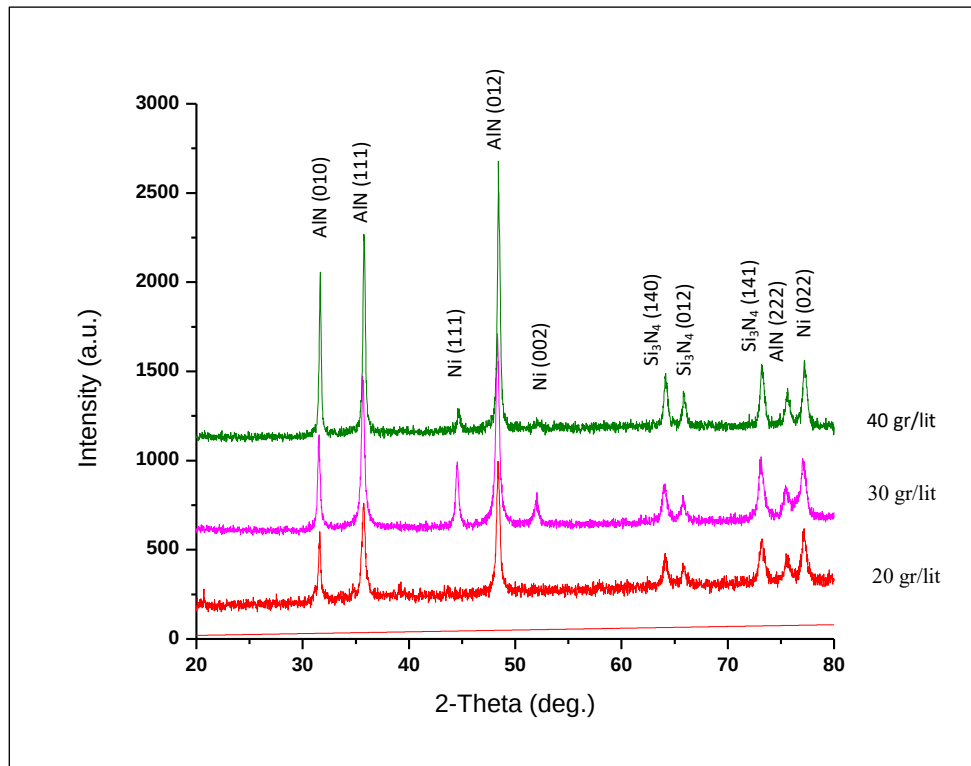


Figure 3. XRD spectrum pattern of Ni-AlN/Si₃N₄ composite coatings at different Si₃N₄ particles concentration.

It shows that, in general, the addition of Si₃N₄ particles concentration up to 30 gr/lit lead Ni (111) and Ni (002) crystals growth. However, the decrease of nickel (Ni) crystal size is occurred at further increasing Si₃N₄ particles concentration up to 40 gr/lit. It was reported that AlN and Si₃N₄ particles are mainly inert incorporated into the composite coatings due to its high melting point, thus both particles might be considered as obstacles for crystal growth [9,10]. In this study, the presence of AlN and Si₃N₄ particles may inhibit the Ni crystal growth. However, in this study, based on EDS result, the content of Al and Si decrease as the increase of Si₃N₄ concentration. Thus, the crystals growth is occurred especially Ni crystal growth as the increasing Si₃N₄ particles concentration.

3.4. Microhardness

Table 1 displays the microhardness measurement result of Si₃N₄ composite coating surface. In this study, the coating hardness increase as Si₃N₄ concentration is increased up to 30 gr/lit. Meanwhile, the microhardness decreases as further increasing Si₃N₄ particles concentration up to 40 gr/lit. An increase of coating microhardness with increasing Si₃N₄ particles concentration might be related to the AlN and Si₃N₄ dispersion-strengthening that hinder the dislocation [7]. The increase of Si₃N₄ particles concentration is followed by the increase of Ni crystal growth and microhardness. Two kinds of

strengthening mechanism in coating system, namely dispersion and particle strengthening [11]. Although the contribution from grain refinement strengthening is identical to agglomeration but the dispersion strengthening is larger [12]. In this study, the increase of Si_3N_4 particles concentration lead the decrease of Al and Si content. Therefore, AlN and Si_3N_4 particles contribute in only a very low volume percent in increasing microhardness of the coatings. However, as further increasing Si_3N_4 particles concentration followed by the increase of Ni crystal size, the microhardness decrease indicating that the AlN and Si_3N_4 phase is not effectively stopping the dislocation motion.

Table 1. Microhardness of Ni-AlN/ Si_3N_4 composite coatings at various Si_3N_4 particles concentration with load of 500 gram

Si_3N_4 concentration (gr/lit)	Indentation diameter average (mm)	Microhardness (Kgf/mm ²)
20	40.34	572.03
30	31.445	938.27
40	37.265	640.77

4. Conclusion

In this work, the development of Ni-AlN/ Si_3N_4 composite coatings at different Si_3N_4 particles concentration is described. It showed that Si_3N_4 particles were successfully deposited and dispersed homogeneously into Ni-AlN matrix. The incorporation of Si_3N_4 particles change the coating composition, surface morphology and crystal structure by promoting the agglomeration and crystal growth. The increase of coating microhardness is related to the particles dispersion-strengthening.

Acknowledgments

This work financially was supported by Ministry of Research, Technology and Higher Education with contract No. 55/SP2H/DRPM/LPPM-UNJ/II/2018 and 02/KOMP-UNJ/LPPM-UNJ/II/2018.

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