Review on Nickel Nitride Composite Coatings

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Abstract. Nickel based composite coating has been widely used in many field applications such as wear and corrosion resistance since it is possible to improve its properties by depositing a variety reinforcement and process parameters. In this work, a variety of reinforced nitride particles such as TiN, AlN and Si were used to enhance the microstructure, mechanical and oxidation properties of nickel nitride composite coating in improving the coating density and surface morphology. The electrodeposition method was used to develop the composite coating with variety process parameters including temperature and current density. The coating stress was calculated from crystal structure data to investigate its involvement in the mechanical properties of coatings. The high temperature oxidation behavior of nickel nitride composite coatings was studied by heating process. The review highlights the reinforced nitride particles and process parameters improve the hardness and wear resistance of nickel based composite coating, however, the improvement also involves the coating stress. From study result of high temperature oxidation process, it obtained that the aluminum oxide remains within the coating indicating its role play in protecting substrate from corrosion. Experimental study on surface modification and microstructure of nickel based composite coating are highly expected and meet a great challenge for different applications with superior mechanical and tribological properties.

1. Introduction

Nickel composite coating has been widely used in many field applications such as wear and corrosion since it is possible to improve its properties by depositing a great variety reinforcement micro-or nanoparticles [1-5]. The incorporating of reinforced particle such as nitride into nickel metal matrix to form a composite has significantly improved its properties [6-7]. The main goal in fabricating this composite is to enhance the mechanical properties as well as hardness, wear, and corrosion resistance of the coating. Compared to the pure metal or alloy coating, that need more expensive heat treatment, the composite coating is preferred for engineering application due to the significantly improvement in tribological and corrosion resistance properties [7-9]. The nickel composite coating with nitride, oxide or ceramic reinforced particles that combine the metal, covalent and ionic bonds attribute to the excellent high hardness and wear resistance.

The behavior of high temperature oxidation is the main factor for improvement of the material performance since it impacts tribological characteristics, especially a decrease in wear and corrosion resistance [10]. Nickel (Ni) is an industrial material that is widely used as coating material for wear and corrosion protection. However, at high temperatures, the pure nickel coating can be oxidized and reduce its applications performance. To enhance the mechanical properties as well as the corrosion resistance of the Ni coating, some complex inert particles are added as reinforcement particles into the Ni coating to form a Ni-composite coating system [3,8,11,12, 13,]. Introduction of the inert particles such as nitride within nickel coating, as reinforced phase from plastic deformation causes the improvement in hardness, wear, and corrosion.

The properties and performance of nickel composite coating depend on its composition and microstructure and can be controlled by process parameters. The uniform surface morphology is influenced by grain or crystallite size, particle dispersion and nucleation rate during deposition process [1,6]. The smooth surface morphology of composite coating is attributed by fine grain size of matrix due to the inhabitation of grain growth by reinforced particles. In the nickel composite system, the crystalline structure of nickel metal as the composite matrix is altered by embedding crystalline and or amorphous reinforced particles. The amorphous and crystalline phase of reinforced particles are effective to stop the dislocation motion [14-15]. Therefore, the composite strength is mainly contributed by the interface interaction strength between matrix and reinforcement.

However, in the coating system, the strength also is contributed by the interface strength of coating layer and substrate that is influenced by coating residual compressive stress. Usually, the tensile residual stress is generated in the coating during the deposition process due to the changing in the temperature leading to neutralize the compressive stress [17]. The tensile stress is also caused by the difference between thermal expansion coefficient of coating layer and substrate [16, 18]. Previous studies showed that the coating stress contributed to the mechanical properties that should be avoided since it would be released as the temperature operation is increased leading the decrease of mechanical properties. The formation of nanostructured coating became a main aim in producing the excellent performance of the coating for various applications.

Electrodeposition is one of the coating deposition methods for producing nickel composite coating since it is simple, low cost and rapid applicability [19,20]. This method requires an electrolyte solution (as an electrical conductor) and electrodes, both of which form an electrochemical cell system. The number of electrodes used can be two or three. For two electrodes, one electrode functions as a substrate (cathode/working electrode), namely the electrode that will be coated and the other electrode functions as a counter electrode. The two electrodes are connected to a power supply source. Most of the process parameters such as current, concentration, stirrer, temperature etc. influence the coating properties [21]. The current electrodeposition is one of the most significant parameters, however the pulse current played a key role in improving hardness, wear and corrosion resistance of the composite coating [2,22,23]. The microhardness, wear and corrosion resistance of composite coating that deposited by using pulse current is higher than that of using direct current electrodeposition. The pulse current is better and more effective in controlling adsorption particle and chemical composition of the coating.

Recently, the surface modification of composite coating is done by applying heating process using microwave induction technology [24, 25]. The microwave heating process involves a partial melting process in the formation of layers and the reaction of reinforcing particles in the composite matrix. The heating process at the molecular level makes heating uniform, fast and cheap compared to conventional heating. However, the interaction mechanism between microwaves and metal or ceramic materials has not been studied much. Modification of the coating surface microstructure can be carried out after the deposition process through the microwave induction heating stage. The application of microwave induction technology for surface modification of composite coating is used with consideration of molecular, uniform, and fast heating properties in improving the quality of the microstructure through involving partial melting of the reinforcing particles and composite matrix. This results in increased metallurgical bond strength between the reinforcement and the matrix, preventing cracks and porosity.

In this present study, an effort has been made to review the properties and performance of nickel nitride composite coating. The effect of parameter processes such electrodeposition temperature, particle concentration and current density is presented. The coating stress and its relationship with the mechanical properties are discussed. The oxidation behavior of nickel nitride composite coating at elevated temperature is presented.

2. Nickel Nitride Composite Coating

Nickel (Ni) is an engineered material that is widely used to protect a certain material from wear and corrosion in many industrial applications. Due to its ductility, nickel was also employed to tough the coating system of TiN/SiN [26,27]. To improve the strength of nickel coating properties, some reinforced particles were incorporated to the system such nitride (TiN, AlN and Si_3N_4) in formation of nickel-/nitride composite coating system. Nickel-nitride composite coating has been proven due to its excellent application performances due to high resistances in wear and corrosion [3,8,11]. The excellent performance of composite is attributed to the mixed combination of two or more constituent phases with different chemical compositions and structures, which are intentionally mixed and separated by different interfaces [28]. The phases mix of crystalline-crystallline or crystalline-amorphous was proved to provide excellent composite coating performances including at high temperature operation. Particulary, in the phase mix of crystalline-amorphous, the crystal growth at high temperature can be resisted by amorphous phase through nanostructure formation [14].

In nickel-based composite coating system, nickel played as matrix, while other components including TiN, AlN and Si_3N_4 played as reinforcement. Ni, TiN and AlN existed as crystalline phase with TiN and AlN were dispersed within Ni matrix [29]. Meanwhile Si_3N_4 existed as amorphous phase and it coexisted at grain boundary region or covered the crystal grains [30]. The mix phases between nickel and nitride are formed to improve the characteristics of pure nickel coating in some applications especially at high speed, high corrosive, and high temperature operation. To improve its performance, especially wear and corrosion resistance, some nitride particles are added to form nickel-nitride composite system. It is known that nitride has been used widely as coating system of MeN/Si_3N_4 such as TiN/Si_3N_4 and TiAlN/Si_3N_4 or TiN-AlN/Si_3N_4 since it shows a super hardness even at high temperature operation [30,31,32]. The high hardness strongly contributes to the excellent wear and corrosion resistance especially at high speed and high temperature operations. Based on this, the nitride particles have a potential material to enhamce a significant contribution in strengthening the nickel-nitride composite coating system as reinforcement phase.

Generally, the composite coating with particles incorporation such as nitride showed better oxidation resistance than pure element coating [33]. The nitride itself was commonly used as a coating system such as TiN. This coating showed excellent performance until degraded at operation temperature about 500-700 ^{0}C due to oxidation process. Meanwhile, AlN and TiAlN coatings firstly were oxidized at temperature about 700-900 ^{0}C [35,36,37]. On the other hands, Si_3N_4 particles was commonly employed as reinforced particles in a coating system and its remarkable performance was achieved when it was incorporated into TiN or TiAlN coatings to form a ultrahard and superhard coating system with high thermal stability up to 1100 ^{0}C [38,39]. The amorphous phase of Si_3N_4 contributed to the high oxidation resistance of the coating system due to its low electrical conductivity.

Surface morphology of Ni-TiN composite coating was compact with evaluation of nucleation and granular growth of TiN particles. The compactness changes as the particles grain further grow resulting agglomeration and pore on the surface. Meanwhile, the surface morphology of Ni-TiN-AlN/Si_3N_4 composite coating showed a disorder granular formation of particles providing the increase in surface roughness. The crystal structure of Ni-TiN-AlN/Si_3N_4 composite coating showed that Ni, TiN and AlN exist as crystalline phase while Si_3N_4 as amorphous phase. [40,41,50]. Peak of Ni reveal at $2\theta = 44.30^0$, 51.74^0 and 76.86^0 (ICDD 00-004-0850). The peak of TiN reveal at $2\theta = 35{,}43^0$ and $72{,}89^0$ (ICDD 01-

087-0631) meanwhile the peak of AlN reveal at 2θ = 31,30^0, 48.07^0 and 83.81^0 (ICDD 01-083-1287). No Si_3N_4 phase was observed due to its amorphous phase.

3. Electrodeposition

The electrodeposition process involves the deposition of reinforcing particles on a layer of metal matrix. Deposition involves the process of convection of particles on the surface of the substrate (cathode), mechanical particle capture and electrophoretic migration of particles into the metal matrix [19]. In general, the stages of the process of forming a thin composite layer using electrodeposition rules are as follows: the first is the formation of charged particles through the absorption of ions and surfactants on the surface of the particles. Next, the particles move physically towards the substrate through the convection layer (transport process) and the boundary layer (diffusion process) where both processes are controlled by the potential difference between the two electrodes. Then the final stage of this process is the settling of particles on the growing metal matrix [20].

The microstructure of nickel composite coating was strongly influenced by electrodeposition parameters such as current density, particle concentration, temperature etc. The current density influences the deposition of particles that ionize by a thin layer of negatively charged species such as OH^- from anionic surfactant that covered the particles [21]. Generally, the mechanism of deposition process consists of three steps which are exotic agitation, electrophoresis, and adsorption. The current density is directly associated to the electrophoresis rate thus the deposition rate increases as the current density is increased. However, the deposition rate will decrease as the current is further increased due to hydrogen reduction rate. With decreasing in particle deposition, the growth of nickel crystal as the composite matrix becomes favorable. Two typical currents used in electrodeposition are direct and pulse current. However, from the study results, it shows that the use of pulse current density produces a higher coating hardness than direct current density. Furthermore, the pulse current density directly correlates the electric potential that prominently influences the nucleation rate of nickel crystals as a composite matrix [23,42].

Meanwhile, the particle concentration influence to the particle content within the composite coating. However, as the particle concentration is further increased the nickel crystal growth is inhibited due to cathodic polarization. Simultaneously, the increase of particle concentration generates the agglomeration effect and subsequent gravitational sedimentation of particles. All these effects influence on the reduction of the deposited particles and eventually influence the coating properties and performance. However, due to steady state equilibrium where the number of co depositing particles equal the number of approaching the surface resulting the particle agglomeration in electrolyte solution [50].

Another important parameter of electrodeposition is temperature. The high electrodeposition temperature of 60 °C produced a compact morphology of Ni-TiN-AlN/Si_3N_4 composite coating due to the increase reinforced particles content. However, for Ni-TiN/Si_3N_4 composite coating, the high tlectrodeposition temperature resulted a rough morphology with a few agglomeration on the surface [50]. The deposited particles rate enhances as the electrodeposition temperature is increased due to the increase of thermodynamics mobility of particles and its dispersion within the electrolyte solution. As the temperature is increased the deposits become smother and more homogeneous and improve the crystalline [43]. However, as the temperature is further increased, the hydrogen molecule is generated and increases de-absorption process of particles.

4. High Temperature Oxidation

Generally, the high temperature oxidation mechanism is initiated by absorbing oxygen gas from atmosphere following the oxide nucleation and grow to form a metal oxide layer [44]. The mechanism is represented by equation as follows:

$$M - 2e^{-} \rightarrow M^{2+}$$
$$O_2 + 2e^{-} \rightarrow O^{2-}$$
$$M + O_2 \rightarrow MO$$

The oxidation resistance of composite coating at high temperature depends not only on its elemental composition but also on its crystallography phase and microstructure [10]. The microstructure is a main factor in oxidation behaviour since it controls the diffusion process and the amorphous phase at grain boundary can effectively hinder it. Two main decisive mechanism of diffusion for high temperature oxidation are outward diffusion of coating elements and inward diffusion of oxygen. The outward elements coating react with the inward oxygen forming an oxide layer on the top of the coating.

The oxidation of Ni and Ni-based coating at high temperature has become an essential issue for decades. The reason is that the increased number of applications for Ni-based coatings at high temperatures, thus the formation of NiO system with only one oxide stable at high temperatures has been considered as a scientific interest [45]. For a pure nickel metal, the protection against corrosion can not only through the formation of nanocrystals but also through the formation of a NiO oxide coating. However, at high temperatures about 700°C - 900°C, the formation of Ni crystal was reduced due to the crystal growth of NiO grains following the reduction of Ni elements to diffuse out through the NiO coating. At high temperature, as the nature of the nickel metal, it tends to be soft and eventually can potentially reduce its wear and corrosion resistance. Therefore, adding a complex noble compound such as nitride particles into the nickel coating could keep the resistance level of wear and corrosion, especially at high temperatures. Some particle nitride compounds that can be used were AlN-TiN [46,47].

Metal transition nitrides have received growing attention as coating material for material protection from wear and corrosion. The properties of metal transition nitride are mainly determined by the metal-nitrogen bonds. formation. Metal transition nitride such as Titanium Nitride (TiN) is a hard protective coating and commonly as the hard coating on the cutting tool. TiN are very hard and dense coatings with maintained hardness at high temperatures [48]. The oxidizing behavior of TiN hard coatings are a key role in tool applications since they are exposed to the oxidative atmospheres at high temperatures condition during its use as wear and corrosion resistant coatings [49]. However, at temperature of 500 °C - 700 °C, the oxidation process is occurred which oxygen gas molecule replace the nitrogen bonding followed by the out-gassing of nitrogen gas molecules. At temperature of 500 °C - 700 °C, the bonded nitrogen with oxygen molecules can be replaced by TiN as the nitrogen out-gassing leaving oxidized titanium (TiO_2) on the substrate. This oxide layer is ease to remove or delaminate leading to expose new TiN layer that is oxidized repeatedly. At the end, the TiN coating cannot protect the substrate anymore from oxidation, corrosion, and wear [50].

To improve the TiN coating performance, Aluminum (Al) element is added to form Titanium Aluminium Nitride (TiAlN) ternary system. Al is chosen since its mobility is higher than Ti then it is easier to be oxidized and effectively retard further penetration of oxygen molecules into the substrate surface [50]. Therefore, the TiAlN coating shows higher thermal stability than TiN coating. Another nitride such as AlN also was added to form TiN-AlN or TiAlN coating system that will start to oxidize at a temperature range of 700°C - 900°C [35]. At low temperature (< 700°C), a passive layer Al_2O_3 was formed preventing oxygen (O) atom entered and reacted with Ti to form TiO_2 (Figure 1). As temperature increased up to 700°C, the diffusion rate of oxygen atom enter the Al_2O_3 layer also increased, leading to the formation of a mixed oxide layer of Al_2O_3 onto the TiAlN coating surface [50]. As the temperature was further increased up to 800 °C, the mixed oxide layer changed into an oxide layer of Al_2O_3/TiO_2

structure. At temperature above 800°C, the diffusion rate of Al increased through Al_2O_3/TiO_2 layer and oxidized on the free surface while oxygen diffused into the Al_2O_3/TiO_2 layer and oxidized Ti to form oxide layer TiO_2 at TiN-TiO_2 interface.

Another nitride compound of Si_3N_4 particles were used as incorporated particles into coating due to its high melting point, hardness, and toughness [30]. It was known that the formation of TiN/Si_3N_4 composite coating exhibited excellent corrosion resistance and thermal stability up to 850 °C [31,32]. TiAlN/Si_3N_4 coating showed remarkably high thermal stability up to 1100°C due to self-organization to nanostructure [38,39]. The high thermal stability of the coating was attributed to the corporation of crystal TiAlN phase and amorphous Si phase within the coating system. The system also possessed high oxidation resistance.

The high temperature oxidation resistance of coating not solely depends on its elemental or compound compositions but also depend on its phase and structure. For TiAlN coating, its phase structure can be a TiAlN single cubic (NaCl structure) or mix of TiN cubic and AlN hexagonal (wurtzite structure) or AlN single wurtzite structure depending on Ti and Al content. Meanwhile, Si_3N_4 particle with high melting point, hardness and toughness, form an amorphous phase in TiN/Si_3N_4 and TiAlN/Si_3N_4 coating systems that collaborate with TiN crystal phase and TiAlN crystal phase, respectively, show high oxidation resistance and thermal stability at temperature of 850-1100 ^{0}C due to self-organization to nanostructure [50].

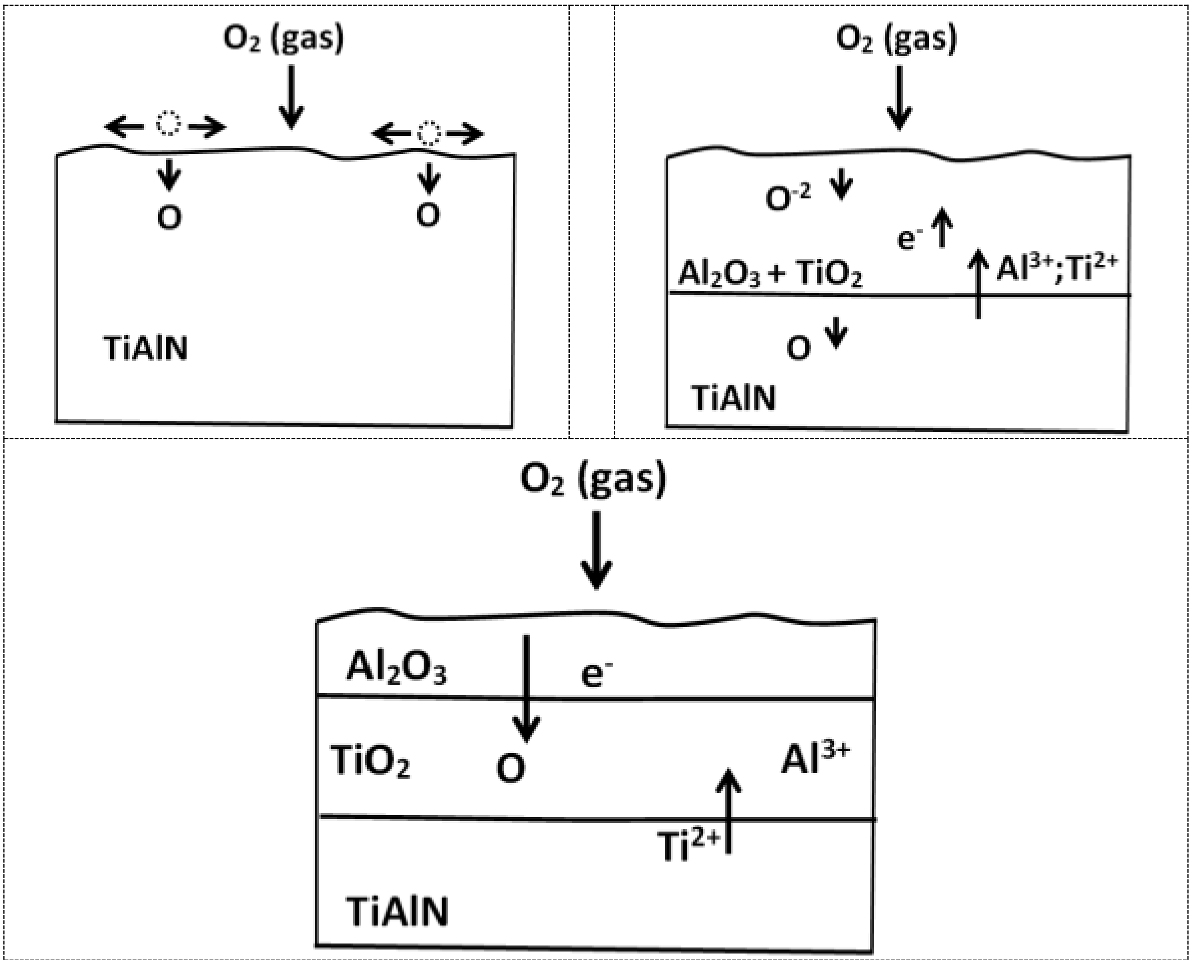

Figure 1. Illustration of high temperature oxidation of TiAlN coating at (a) initial adsorption, (b) $T \leq 700^0C$ (c) $T \geq 800^0C$ [44,50].

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The surface morphology of nickel-/nitride composite coating at high-temperature showed the presence of scale growth of coating grains and reduced cracks and pores. The crystal growth of scale on the coating is a characteristic of oxidation and indicates that a protective oxide coating has been developed after high-temperature oxidation. The pores on the coating surface are reduced because the pores have been oxidized on exposure to high temperatures.

Table 1. Metal element of Ni-TiN-AlN/Si_3N_4 composite coating before oxidation process that deposited at electrodeposition temperature of (a) 35 °C, (b) 40 °C and (c) 45 °C [56]

Element	% At.			
	Ni	Ti	Al	Si
(a)	20.46	0.01	0.37	0.26
(b)	17.28	0.39	0.63	0.37
(c)	21.11	0.64	2.61	1.64

Table 2. Metal element of Ni-TiN-AlN/Si_3N_4 composite coating after oxidation process that deposited at electrodeposition temperature of (a) 35 °C, (b) 40 °C and (c) 45 °C [57].

Element	% At.			
	Ni	Ti	Al	Si
(a)	8,47	-	9,46	-
(b)	11,77	-	13,06	-
(c)	3,25	-	2,44	-

The initial composition of Ni-TiN-AlN/Si_3N_4 composite coating before oxidation process is shown in Table 1. From the results of oxidation treatment, it shows that Ni and Al elements of Ni-TiN-AlN/Si_3N_4 composite coating still remain after oxidation process (Table 2). Meanwhile, element of Ti and Si disappear. It is known that the Gibbs free energy of Al oxide (and Ni) in the initial stage oxidation is lower than Ti-oxide [26]. Element of Ti and Si are consumed by oxygen faster than Al (and Ni) and since the Ti and Si content is low thus it is not possible for Ti and Si to form a compact subsurface layer.

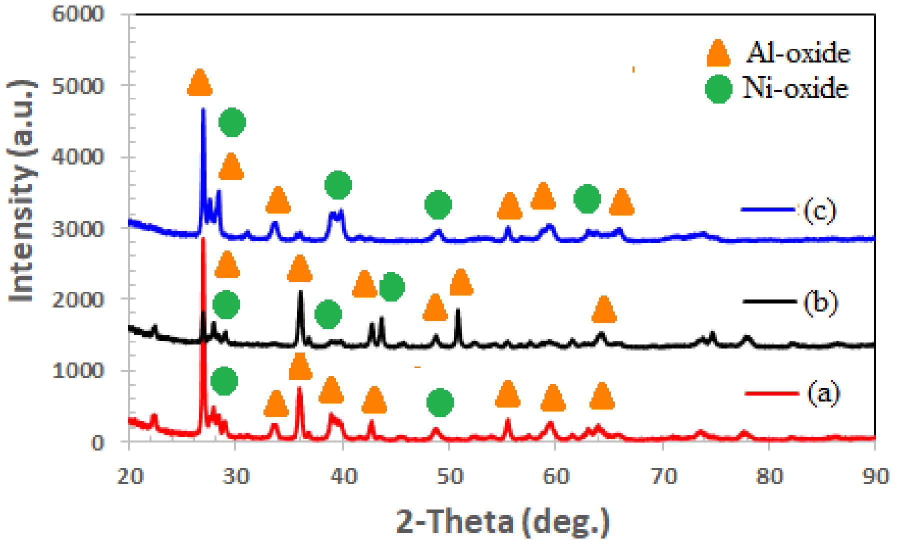

Figure 2. XRD spectrum of Ni-TiN-AlN/Si_3N_4 composite coating after oxidation process at temperature of 700 °C for 2 hours. The samples are deposited at various electrodeposition temperature of (a) 35 °C, (b) 40 °C, and (c) 45 °C [57]

From the XRD analysis result of Ni-TiN-AlN/Si_3N_4 composite coating, the high temperature oxidation process of Ni-TiN-AlN/Si_3N_4 composite was carried by heating at temperature of 700 °C for 2 hours (Figure 2). It showed that the oxidation process resulted in metal oxide formation in term of Aluminum oxide and Nickel oxide. At temperature of 700 °C, All metal elements (i.e. Al and Ni) of coatings diffuse outward and react to the oxygen molecule except Ti and Si. The morphology of the Ni-AlN-TiN/Si_3N_4 composite coating after oxidation process at temperature of 700 °C for 2 hours, showed the presence of scale growth of the surface followed by reducing cracks and pores [50].

5. Hardness and Wear Resistance

Vickers hardness test of Ni-TiN-AlN/Si_3N_4 composite coating was an average value of three points measurement. It showed that the hardness increased as increasing in electrodeposition temperature. The increase in hardness involved the inclusion of Si_3N_4 particles within Ni matrix [51,52]. the increase also was contributed by combining in crystalline-amorphous phase of nickel and nitride [30,41]. Therefore, in this study the coating hardness was addressed to the phase formation of Ni/TiN, Ni/AlN, TiN/Si_3N_4 or AlN/Si_3N_4. From the calculation of crystalline size, it was revealed that the increase of hardness was followed by the decrease in crystallite size. From the stress calculation showed that the lattice strain and stress of composite decease as increasing Si_3N_4 particles content thus the increase of Si_3N_4 particles concentration as strengthening particles effectively hindered the crystal growth of nickel crystal providing the increase of coating hardness [50]. The wear resistance of Ni-TiN-AlN/Si_3N_4 coated-WC drills showed that although the highest hardness of nickel composite coating was achieved for the sample that deposited at electrodeposition temperature of 40 °C however, the lowest wear rate as achieved for sample deposited at 45 °C with lowest wear value of ~30 µm. This in line with Archad's law of inverse relation between hardness and wear rate [53]. The lowest wear rate indicated good quality of adhesion strength of the coating [54] due to the ductile nickel layer.

6. Future Research and Applications of Nickel-based composite coating

The exploring on the formation of nanocomposite as coating to protect material from wear and corrosion is still interested to be investigated to find new material and technology for application in many industrial fields including in extreme condition or environment [25]. High temperature oxidation resistance of composite coating is a key to wider its applications especially in extreme conditions. It is important to understand the deeply insight into mass transport, chemical reaction, and diffusion during the oxidation process [57] as well as the deposition process. The post-heat treatment including microwave heating is an alternative option to modify the structure and properties of composite coating to enhance its performance through intermetallic formation and metallurgical bonding in the coating [58].

7. Summary

Deposition of nickel based composite coating were successfully accomplished by electrodeposition method. Varying process parameters such as dc or pulse current, electrodeposition temperature; particle concentration etc were applied to optimize the coating properties and performance. The reinforced nitride particles and process parameters improve the hardness and wear resistance of nickel based composite coating; however, the improvement also involves the coating stress. The aluminium oxide remains within the nickel nitride composite coating at high temperature oxidation process indicating its role play in protecting substrate from corrosion. Experimental study on surface modification and microstructure of nickel based composite coating post heating process are highly expected and meet a great challenge for different applications with superior mechanical and tribological properties.

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