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BRUSH ELECTROPLATING AND CHARACTERIZATION OF Ni/Si_3N_4 COMPOSITE COATINGS

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The paper presents the results of research on Ni/Si_3N_4 composite coatings produced by the brush plating method. The surface topography and morphology, structure, microhardness and the corrosion resistance of Ni and Ni/Si_3N_4 coatings were investigated. The composite coatings exhibited higher microhardness and corrosion resistance compared to pure nickel coatings. The average grain size decreased with increasing Si_3N_4 particle content in the plating bath. The study verified the feasibility of producing such coatings and determined the effect of the particle content on the properties of the nickel composite coatings.

Keywords: **composite coatings, silicon nitride, Ni/Si_3N_4, electroplating, brush plating**

INTRODUCTION

Brush plating is a modified electrodeposition technique used when it is not possible to apply a composite coating by conventional electroplating. In this method, the anode, wrapped in an absorbent material and soaked in the electrolyte, is moved over the surface to be coated – connected to the negative electrode. Deposition occurs only when the anode is in contact with the substrate [1–3]. Unlike tank electroplating, the brush plating technique is portable, therefore it is possible to perform on-site repairs and the plating of assembled parts [1–3], which is a great convenience for large items. The technique also requires significantly less solution compared to traditional methods, thereby reducing the amount of wastewater generated [1, 2].

In recent years, brush plating has been successfully employed for the deposition of composite coatings. This method utilizes specially formulated solutions that allow the application of coatings with high wear and corrosion resistance, increased microhardness, and good surface wettability [4]. The adhesion of coatings produced by this technique is comparable to or even better than those achieved by means of conventional electrodeposition. The corrosion resistance of layers deposited by brush plating is similar to that of coatings obtained by electroplating, while the microhardness is in most cases greater than that of metals electrodeposited from galvanic baths. For instance, 24-karat gold applied by brush plating exhibits a microhardness of 120–130 HK, compared to only 30–70 HK achieved by typical electroplating processes [5].

The effect of the deposition parameters – specifically, the applied voltage and the electrode travel speed relative to the coated surface – on the microhardness of nickel-based coatings was discussed in [5]. The highest microhardness, approximately 660 HV, was observed for coatings deposited at 10 V and an electrode travel speed of 11.3 m/min [5]. It is worth emphasizing that composite coatings with a metal matrix have attracted significant research interest due to their better mechanical, tribological and corrosion-resistant properties compared to pure metal coatings [2]. Nickel is widely used as a matrix material in such composites because of its ease of deposition and uniform distribution [1]. Nickel matrix composite coatings are extensively applied, especially in the automotive and aerospace industries owing to their very good mechanical properties and suitable corrosion resistance. Electrochemically deposited nickel coatings containing a dispersed phase are characterized by enhanced properties compared to conventional nickel coatings. The presence of embedded particles within the coating affects both the corrosion behavior of the layer, as well as its mechanical and physical properties.

The brush plating technique can be used to produce various composite coatings. Literature reports describe the fabrication of nickel matrix composite coatings reinforced with graphene oxide (GO) particles [1], aluminum particles [2], carbon nanotubes [6], carbon or copper particles [7], and molybdenum disulfide (MoS_2) combined with carbon particles [8]. Additionally, this method has been applied to produce Ni-Cr matrix coatings with Al_2O_3 or Cu particles [9], as well as Ni-P matrix coatings reinforced with WC particles [10]. Co-deposition via electrobrush plating is widely employed for the fabrication of particle-reinforced metal matrix composite coatings because of its operational simplicity, low cost and versatility [10]. Du et al. [11] stated that Ni/nano-Al_2O_3 composite coatings exhibited higher microhardness compared to pure nickel coatings. Jiang et al. [12] found that Ni/nano-ZrO_2 composite coatings provided a longer contact fatigue life than coatings made of pure nickel. The microhardness of nickel-carbon nanotube composite coatings produced by the brush plating method, with a dispersion phase content of 5 mg/dm^3, was 523 HV0.05, which was higher than the microhardness of the Ni coating (479 HV0.05) [6]. Furthermore, the research conducted by Wu et al. [13] demonstrated that the process of producing Ni/nano-Al_2O_3 composite coatings by the brush plating method can be automated, which can, for example, reduce the friction coefficient and improve the wear resistance of the resulting coatings.

Silicon nitride (Si_3N_4) is a high-performance ceramic material widely utilized in forging and surface hardening applications owing to its notable properties, including strong chemical inertness, excellent corrosion resistance, and high microhardness. Si_3N_4 nanoparticles can occupy voids and micrometer-sized pores within the coating structure, which strengthens the physical barrier effect and consequently enhances corrosion resistance [14].

The incorporation of dispersed phase particles may affect the material properties in different ways. According to Huang et al. [15], nickel-silicon nitride composite coatings with a high content of Si_3N_4 particles demonstrate the ability to achieve exceptionally low friction coefficients. In study [16], the authors produced Ni-B matrix coatings containing dispersed Si_3N_4 particles utilizing a chemical reduction method. The obtained Ni-B/Si_3N_4 coatings exhibited lower hardness (972 HK0.01) for the composite layer produced from a bath containing 10 g/dm^3 of the dispersion phase, compared to the Ni–B alloy coating (1126 HK0.01). The incorporation of the Si_3N_4 dispersion phase into the Ni-B matrix resulted in decreased wear resistance due to friction. In [14], the authors investigated the effect of the Si_3N_4 concentration in bath solutions on Ni-W/Si_3N_4 composite coatings. It was stated that the addition of Si_3N_4 to the Ni-W alloy matrix resulted in notable changes in both the morphology and phase composition. The crystallographic structure evolved from a dominant (111) orientation to a mixed phase consisting of (111), (200), and (220) planes, accompanied by a grain size reduction. A corresponding alteration in surface roughness (Ra) was also observed. The composite coatings demonstrated the most favorable combination of microhardness, wear resistance, and corrosion resistance at a Si_3N_4 concentration of 10 g/L, with microhardness showing an enhancement of up to 55% relative to the unreinforced Ni-W alloy matrix.

It is worth noting that during the deposition of composite coatings by the brush plating method, the movement of the electrode acts as a form of mechanical stirring, which can smooth the surface of the coating and prevent the agglomeration of dispersion particles added to the bath, even without additional surfactants [1].

The available literature related to the application of metal coatings by the brush plating method mainly presents general characteristics of the method, equipment, additional equipment and applications. However, there is no comprehensive data relating to the research of composite nickel coatings applied from various electrolytes. Many studies on the deposition of composite nickel coatings by the brush plating method rely on commercially available solutions. Therefore, in this work, a nickel solution developed by the authors was employed [17]. The objective of the presented investigations was to obtain Ni/Si_3N_4 composite coatings using the brush plating method and to determine the effect of the bath composition, specifically the dispersion particles content on the resulting material properties.

Ni/Si_3N_4 composite coatings combine the characteristics of a nickel matrix with the high mechanical and chemical stability of silicon nitride particles. As a result, such coatings may exhibit improved wear resistance, enhanced corrosion behaviour, and increased surface durability. Due to these properties, Ni/Si_3N_4 coatings produced by brush plating may find potential applications in the regeneration of worn machine elements, including shafts and bearing surfaces, as well as in components subjected to friction and abrasive wear. The possibility of localized deposition makes this technique particularly suitable for in-situ repair and surface modification in aerospace, mechanical, and energy industries.

EXPERIMENTAL PART

The nickel (Ni) and nickel/silicon nitride (Ni/Si_3N_4) composite coatings were produced by the brush plating method on copper plates with dimensions of 60 x 60 mm. Prior to the process, the substrate surface was degreased with calcium carbonate and then rinsed with deionized water.

The bath used in this study to produce Ni and Ni/Si_3N_4 coatings was developed at the Łukasiewicz Research Network – Warsaw Institute of Technology (formerly: The Institute of Precision Mechanics) with the following composition: nickel(II) sulphate(VI), nickel(II) hydroxide, acetic acid, citric acid. The solution exhibited a pH value of 4.2.

The Ni/Si_3N_4 composite coatings were produced in baths with varying Si_3N_4 powder content: 3, 7 and 15 g/dm^3 (denoted accordingly: $Ni/Si_3N_4(3)$, $Ni/Si_3N_4(7)$ and $Ni/Si_3N_4(15)$). The amorphous Si_3N_4 powder was supplied by Nanostructured&Amorphous Materials, Inc., with a particle diameter ranging from 15 to 30 nm. A graphite rectangular plate wrapped in absorbent cloth was utilized as the plating tool. During the plating process, the plating tool saturated with the electrolyte wiped the copper surface at a constant speed. Composite coatings and the nickel coating were deposited at a temperature of 298 K for 15 minutes. A voltage of 12 V was applied between the electrodes. Fig. 1 presents a diagram of the brush electroplating method.

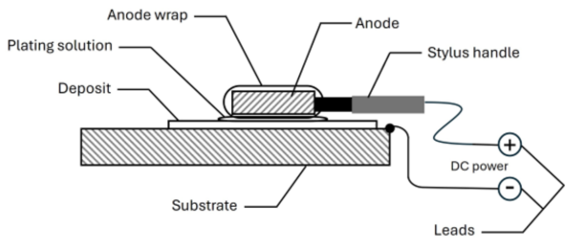


Fig. 1. Diagram of brush electroplating [18]

The dispersion phase used to produce the Ni/Si_3N_4 composite coatings was characterized by means of a JEOL JSM-IT100 LA scanning electron microscope (SEM) as well as surface morphology and topography studies of the coatings. The chemical composition was analyzed with the use of of a scanning electron microscope equipped with an energy-dispersive spectroscopy (EDS) detector. The thickness of the produced coatings was measured using an X-ray fluorescence spectrometer (XRF) X-RAY XDV-SDD from FISCHERSCOPE® manufacturer.

To investigate the crystalline structure of both the coatings and the dispersion phase, X-ray diffraction (XRD) analysis was performed utilizing a MiniFlex II diffractometer (Rigaku) with a copper anode lamp with the wavelength λ = 0.154 nm. The microhardness of the deposited coatings was measured by means of a Tukon 1202 hardness tester from WILSON-HARDNESS (BUEHLER) on cross-sections by the Knoop method under a load of 0.01 kG and load time of 10 s.

The corrosion properties of the coatings were investigated by the potentiodynamic polarization method using a Bio-logic SP-200 system. The tests were carried out in a three-electrode system in a 0.5 M NaCl solution at 293± 2 K in ambient atmosphere. The reference electrode was an $Hg/Hg_2Cl_2/KCl$ calomel electrode with a potential +244 mV and the counter electrode was a platinum electrode. The tests were conducted in the range of ±250 mV potential versus open circuit potential. The working surface of the electrode subjected to corrosion testing was 1 cm^2. The scanning speed was taken as 0.2 mVs^{-1}. The obtained Tafel curves were employed to calculate the corrosion current density and the corrosion potential.

RESULTS AND DISCUSSION

The nickel/silicon nitride composite coatings were deposited by the brush plating method. Electrolyte solutions containing different concentrations of the dispersion phase – 3, 7, and 15 g/dm^3 of Si_3N_4 particles - were used to produce the composite coatings. The coatings prepared from these baths were labeled as follows: Ni/Si_3N_4(3) for the coatings produced in baths containing 3 g/dm^3 silicon nitride particles, Ni/Si_3N_4(7) for the coatings produced in baths containing 7 g/dm^3 silicon nitride particles, and Ni/Si_3N_4(15) for the coatings produced in baths containing 15 g/dm^3 silicon nitride particles. A TEM micrograph of the Si_3N_4 powder particles is shown in Fig. 2.

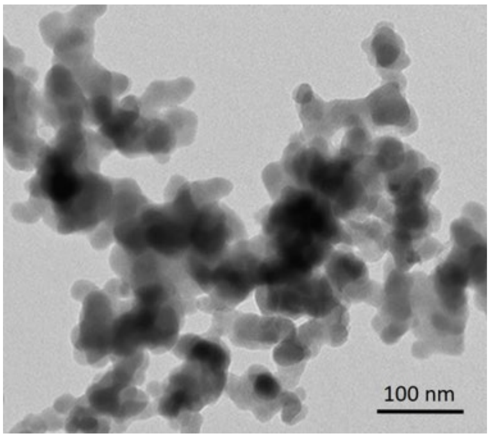
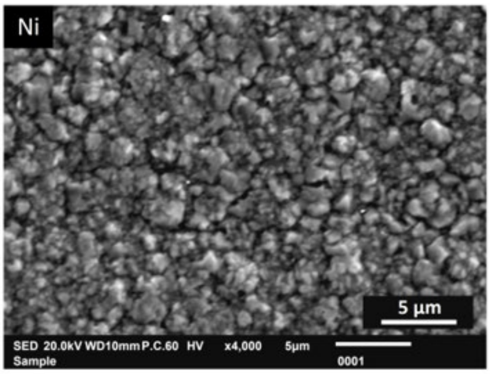


Fig. 2. TEM micrograph of Si_3N_4 powder used as dispersion phase in electrolytic bath

SEM micrographs of the surface topography and morphology of the Ni coating and Ni/Si_3N_4 composite coatings are shown in Fig. 3. The nickel coatings as well as the composite coatings are characterized by various grain sizes.



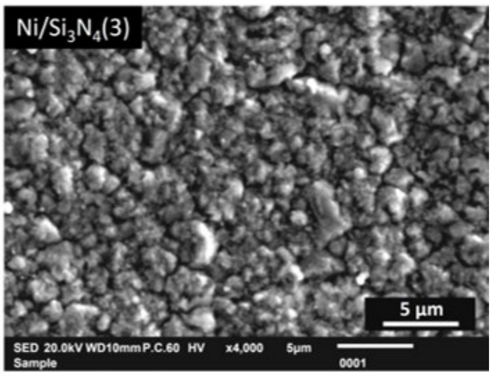


Fig. 3.

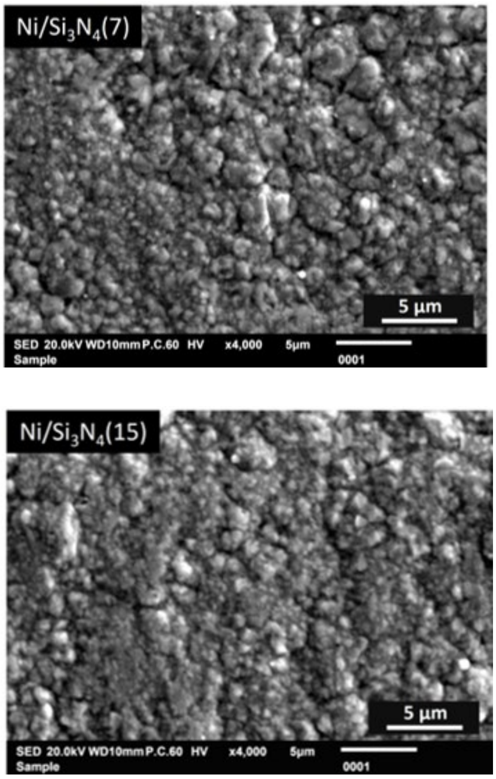


Fig. 3. SEM micrographs of surface topography and morphology of produced coatings

EDS analysis was performed to determine the chemical composition of the nickel/silicon nitride composite coatings, both at selected points and by means of elemental mapping. Table 1, Figs. 4 and Fig. 5 display the EDS results of the compositional analysis for the $Ni/Si_3N_4(15)$ composite coating.

EDS point analysis and elemental mapping confirm the composite nature of the Ni/Si_3N_4 coatings. The Ni-rich regions correspond to the continuous metallic matrix, while Si is distributed both uniformly and in localized regions, indicating the presence of dispersed particles. The point analysis revealed that Si-rich regions contain a high oxygen content and only a small amount of nitrogen, suggesting that the detected phase does not correspond to pure Si_3N_4. This is further supported by elemental mapping, where nitrogen exhibits a weak and diffuse signal that does not directly correlate with the silicon distribution. Such behavior can be explained by partial oxidation of the Si_3N_4 particles. It has been reported that silicon nitride reacts with oxygen to form SiO_2 and release nitrogen, even at room temperature [19]. Additionally, the low nitrogen content may be attributed to the known limitations of SEM-EDS in detecting light elements such as nitrogen. Nitrogen detection by SEM-EDS is known to be unreliable as a consequence of its low X-ray energy and absorption effects, and therefore should be treated as qualitative rather than quantitative [20]. These results indicate that the coating consists of partially oxidized Si_3N_4 particles within the Ni matrix, suggesting that the dispersed phase was successfully incorporated during the process.

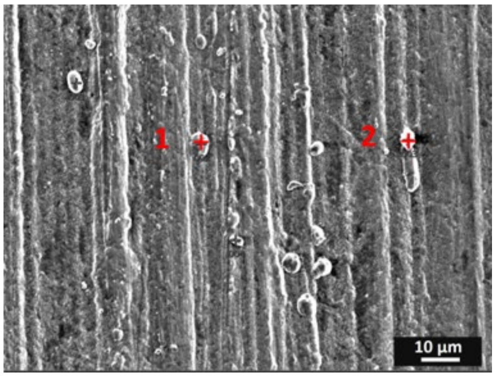


Fig. 4. EDS analysis points of $Ni/Si_3N_4(15)$ coating

TABLE 1. **Chemical composition of $Ni/Si_3N_4(15)$ coating**

Element	wt%	at.%	Element	wt%	at.%
1			2		
C	1.1	5.0	C	2.2	3.8
N	0.9	3.5	N	0.9	1.3
O	0.4	1.3	O	50.7	64.9
Si	0.8	1.6	Si	36.6	26.7
Ni	96.7	88.5	Ni	9.6	3.4

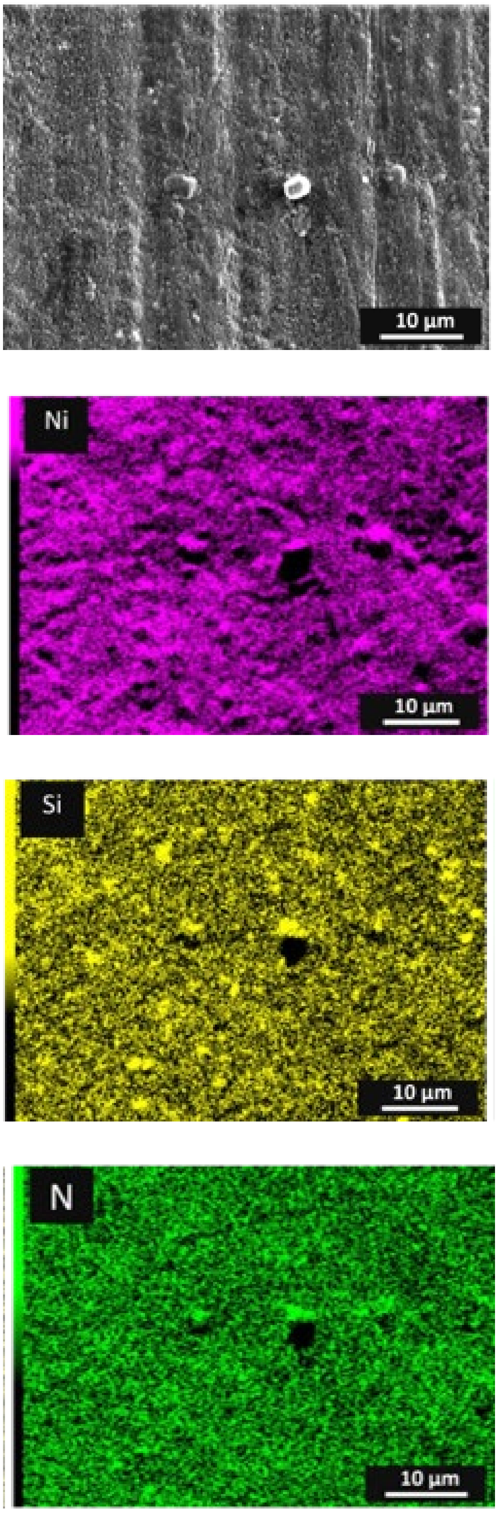


Fig. 5. Maps of elemental distribution on surface of Ni/Si_3N_4(15) coating

The cross-sections perpendicular to the surface of the deposited coatings are shown in Fig. 6. The Ni coating and Ni/Si_3N_4 coatings exhibit a compact structure and good adhesion to the substrate. The thickness of the deposited coatings was 18.23 µm for the Ni coating, 16.37 µm for Ni/Si_3N_4(3), 15.27 µm for Ni/Si_3N_4(7) and 15.97 µm for Ni/Si_3N_4(15).

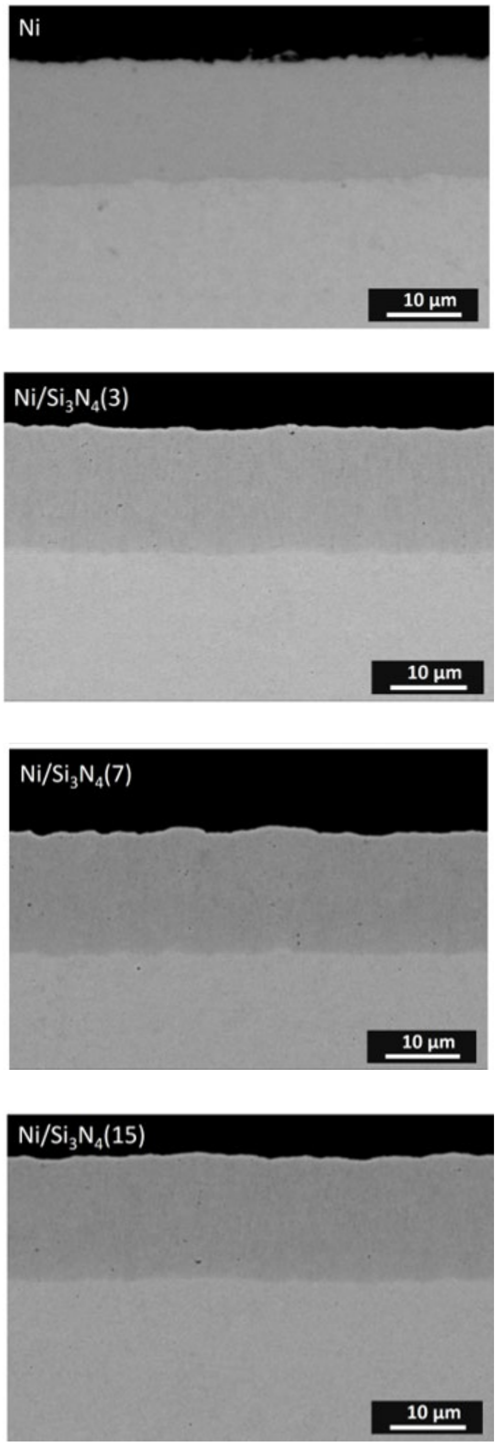
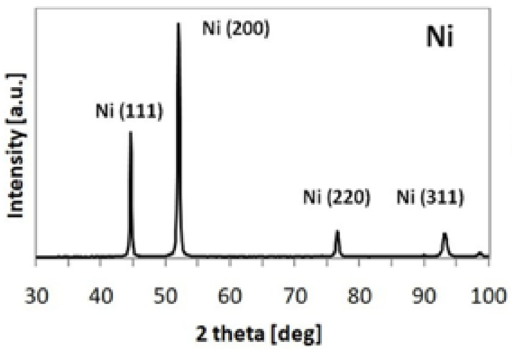


Fig. 6. Cross-sections of the Ni and Ni/Si_3N_4 coatings

The XRD patterns of the pure Ni and Ni/Si_3N_4 composite coatings produced from baths containing 3, 7 and 15 g/dm^3 particles, are presented in Fig. 7. The diffraction peaks at $2\theta = 44°$, 52°, 76°, 93° are assigned to the (111), (200), (220) and (311) crystallographic planes of Ni. These peak positions are in good agreement with those of the reference patterns for face-centered-cubic Ni. It can be concluded that there is a change of a preferred orientation of Ni growth in the composite coatings. The average crystallite size of the coatings was estimated using the Scherrer formula. The calculated grain size of the various samples is shown in Table 2 for the (111) and (200) crystallographic planes. The average grain size for Ni, $Ni/Si_3N_4(3)$, $Ni/Si_3N_4(7)$ and $Ni/Si_3N_4(15)$ decrease with an increase in the Si_3N_4 particle content in the bath.

A similar effect of microstructural refinement in Ni/Si_3N_4 composite coatings has been reported in the literature [21], where incorporation of the Si_3N_4 particles led to grain refinement and modification of the coating morphology. This behavior is commonly attributed to the inhibition of crystal growth and the increased number of nucleation sites caused by the presence of dispersed ceramic particles.



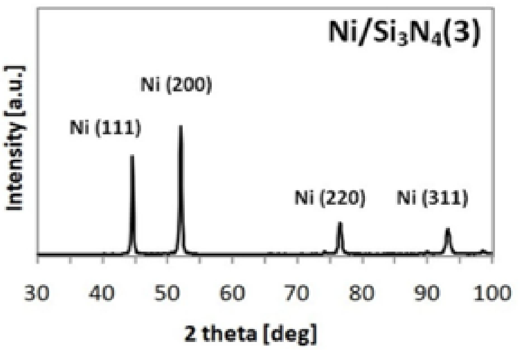
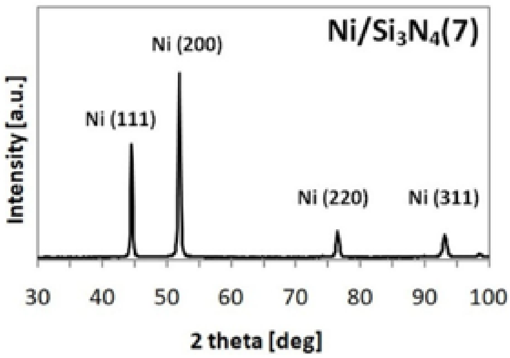


Fig. 7. →



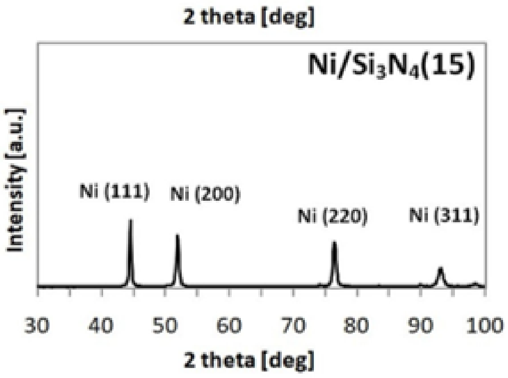


Fig. 7. Diffraction patterns of Ni and Ni/Si_3N_4 coatings

TABLE 2. **Crystallite size of Ni and Ni/Si_3N_4 coatings**

Crystallite size [nm]		
Coating	**Crystallographic planes**	
	(111)	(200)
Ni	26.9	32.3
$Ni/Si_3N_4(3)$	24.5	31.5
$Ni/Si_3N_4(7)$	24.2	28.6
$Ni/Si_3N_4(15)$	22.6	23.0

The results of the Knoop microhardness measurements for the Ni/Si_3N_4 composite coatings and Ni coating are shown in Fig. 8. The incorporation of Si_3N_4 particles into the nickel matrix led to growth in the microhardness of the coating material. The microhardness of the composite coatings ranged from 519 HK0.01 for $Ni/Si_3N_4(3)$ to 634 HK0.01 $Ni/Si_3N_4(7)$, whereas the Ni coating exhibited a microhardness of 463 HK0.01. Thus, the composite coatings demonstrated higher microhardness compared to the pure nickel coating. As the content of the dispersion phase particles in the plating bath increased, the microhardness of the coating material also rose, however, the hardness of $Ni/Si_3N_4(15)$ was slightly lower than that of $Ni/Si_3N_4(7)$.

The increase in microhardness of the composite material can be the result of particle strengthening, dispersion strengthening and grain refinement. Particle strengthening occurs in the case of the incorporation of hard particles of a volume fraction above 20%. In this mechanism, the load is carried by both the matrix and the particles while strengthening is achieved because the particles restrain matrix deformation. Dispersion strengthening is related to the incorporation of fine particles (<1 μm) of a volume fraction lower than 15%. In the case of dispersion strengthening, the dislocation motion is hindered by the dispersion phase particles embedded in the matrix material. Strengthening by grain refinement is related to the nucleation of small grains on the surface of the incorporated particles, resulting in general structural refinement. The occurrence of material smaller grains and thus, more grain boundaries, which block the dislocation mobility, results in a rise in microhardness [22].

The observed growth in the microhardness of the composite coatings shown in Fig. 8 can be explained by the dispersion strengthening and grain refinement mechanisms. The fine Si_3N_4 particles incorporated in the Ni matrix could restrain the growth of Ni crystals and hinder the motion of dislocations. These effects became more pronounced with the increase in the Si_3N_4 particle content in the bath from 3 to 7 g/dm^3, while no further improvement was observed at higher concentrations.

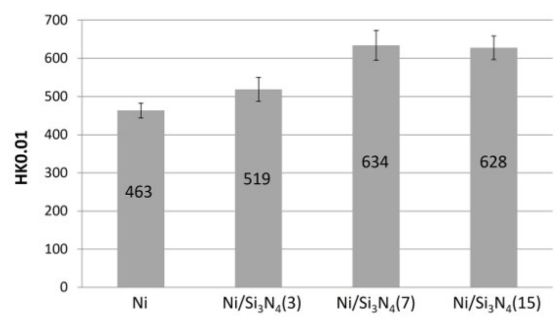


Fig. 8. Microhardness of Ni and Ni/Si3N4 coatings

The potentiodynamic polarization curves obtained from the investigation of the corrosion properties of the coatings produced by the brush plating method are presented in Fig. 9. The values of the corrosion potential and corrosion current density are presented in Table 3. Compared with the pure nickel coating, the corrosion potential of the composite coatings is shifted towards more positive values, which indicates their higher corrosion resistance. The content of the Si_3N_4 particles embedded in the nickel matrix has a significant influence on the corrosion resistance of the composite coatings. The results presented in Fig. 8 and Table 3 indicate that the corrosion resistance of all the composite coatings in 0.5 M NaCl was superior to that of the pure nickel coating. The enhancement in corrosion resistance can be attributed, in part, to the reduction in the surface area of the nickel-based material directly exposed to the corrosive environment. Furthermore, the incorporated nanoparticles serve as chemically inert physical barriers, hindering both the initiation and propagation of corrosion processes, thereby contributing to the improved protective performance of the coating. Additionally, the incorporation of nanoparticles into the Ni matrix could alter the crystallographic structure by refining the crystallite size, which further enhances the coatings' resistance to corrosion [23].

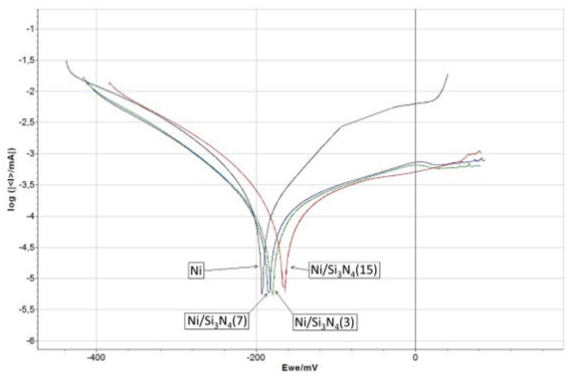


Fig. 9. Potentiodynamic polarization curves of deposited coatings

TABLE 3. **Corrosion parameters of Ni and Ni/Si_3N_4 coatings in 0.5 M NaCl solution**

Coating	E_{corr} [mV]	I_{corr} [μA/cm^2]
Ni	-192.6	0.52
Ni/Si_3N_4(3)	-180.2	0.14
Ni/Si_3N_4(7)	-184.3	0.14
Ni/Si_3N_4(15)	-165.2	0.21

CONCLUSIONS

The results demonstrated that Si_3N_4 particles can be incorporated into nickel coatings by means of the brush plating method. In the case of nickel coatings, the incorporation of silicon nitride into the matrix has a significant influence on the properties of the resulting material. The dispersion phase embedded in the nickel matrix reduces the average crystallite size and leads to growth in both the microhardness and corrosion resistance of the coating material. All the investigated Ni/Si_3N_4 composite coatings exhibited superior corrosion resistance compared to the pure nickel coating. The content of the dispersion phase in the plating bath strongly affects the properties of the produced coatings. The microhardness of the $Ni/Si_3N_4(3)$ coating was 519 HK0.01, whereas the $Ni/Si_3N_4(7)$ coating exhibited a higher microhardness of 634 HK0.01. In terms of corrosion resistance, the $Ni/Si_3N_4(15)$ coating demonstrated better performance than those produced from baths with lower concentrations of the dispersion phase.

Further research should be focused on detailed characterization of the mechanical and tribological properties of the obtained coatings. In particular, investigation of the wear resistance, friction behaviour, and long-term corrosion stability would allow a more comprehensive evaluation of the applicability of Ni/Si_3N_4 coatings produced by brush plating.

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