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Deposition of bright electrochemical nickel coatings on steel

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Abstract

The characteristics of nickel coatings obtained by electrodeposition on steel samples of known chemical composition were investigated, with a focus on the effects of current density and deposition time on the cathodic current efficiency, coating thickness, hardness, and adhesion. Nickel coatings were deposited onto steel samples from three baths, based on the Watts electrolyte: 30–50 g/dm³ NiCl₂·6H₂O, 15–30 g/dm³ H₃BO₃, and 200–250 g/dm³ NiSO₄·7H₂O. In Bath 1, in addition to the standard Watts electrolyte, basic additive 302 (5 cm³/dm³), wetting additive 304 (8 cm³/dm³), and ductility additive 305 (12 cm³/dm³) were added to the standard Watts electrolyte. Compared to Bath 1, Baths 2 and 3 contained brightness agents. In Bath 2, a high-brightness additive 301 (0.5 cm³/dm³) was added, while Bath 3 contained sulfosalicylic acid (0.5 cm³/dm³). The commercial additives used in the study were supplied by “Protekta”, Belgrade. Electrochemical nickel coatings were deposited for 10, 15, and 20 min at current densities of 1.0, 1.5, and 2.0 A/dm², at a bath temperature of 42 °C. It was observed that increasing the current density and deposition time increased the cathodic current efficiency and resulted in thicker nickel coatings. Under the same deposition conditions, nickel coatings of approximately the same thickness were obtained from all three baths, indicating that brightness additives do not significantly affect the thickness of electrochemical nickel coatings, but influenced coating compactness and brightness. Nickel coatings obtained from all three baths showed good adhesion to the steel substrate.

Keywords: nickel coatings, electrochemical deposition, roughness, current efficiency, hardness, adhesion

1. INTRODUCTION

Coatings are applied for the purpose of protecting the base material or for decorative purposes, most commonly on metal objects, through electrolytic deposition. Thanks to the good properties of nickel, it is possible to obtain coatings with various characteristics: black, matte, semi-bright, as well as mirror-bright finishes. Nickel coatings can be used as decorative coatings, but also as protective coatings that prevent corrosion, provided they have the appropriate thickness. Nickel is a silvery-white metal, highly polishable, and fairly resistant to air exposure. In electrolytes used in electroplating, it appears in a divalent state. It dissolves easily in diluted nitric acid (Jović, Lačnjevac, Jović, & Krstajić 2014; Dorđević 1970; Durović & Radović 2007; Wang et al. 2025).

Nickel behaves as a passive metal in many environments. The electrode potential of nickel is more positive than that of iron, and when directly applied to iron, it does not protect it from corrosion if the layer is porous or damaged. Nickel coatings of 1 to 3 µm are highly porous, and only coatings thicker than 50 µm are considered non-porous. Nickel coatings are usually not applied alone but in combination with other coatings, forming multilayer systems: copper-nickel-chromium, semi-bright nickel-bright nickel, nickel-chromium, or copper-semi-bright nickel-bright nickel. Copper is used as a sub-layer beneath nickel because it adheres better to steel than nickel does (Abbott, Ballantyne, Harris, Juma, & Ryder 2017; Boukhouiete & Creus 2015; Lamovec et al. 2018;

Mimani, Mayanna, & Munichandraiah 1993; Pavlović, Stanojević, & Mladenović 2012; Popov & Grgur 2002).

Nickel coatings can be obtained from sulfate, chloride, sulfamate, and fluoroborate electrolytes, which contain specific additives. The most well-known electrolyte for obtaining nickel coatings is the Watts electrolyte, whose basic components are nickel sulfate ($\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$), nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$), and boric acid (H_3BO_3). In addition to the basic components, the electrolyte may contain specific additives to enhance certain properties of the coating. The type of additive influences the properties of the coating. Wetting agents are added (to reduce surface tension and prevent pitting), as well as additives for ductility and various types of brighteners. The quality and properties of the coating are also influenced by current density, temperature, pH value, degree of cathode agitation, and electrolyte circulation (Mohanty, Tripathy, Singh, Keshavarz, & Iglauer 2019; Sadiku-Agboola, Rotimi-Sadiku, & Biotidara 2012; Schlesinger & Paunovic 2010; Schmitz, Quines, Garcia, de Souza, & Rodrigues 2016; Tomić, Pavlović, Pavlović, & Rakočević 2003; 2004; Zečević, Gojković, & Nikolić 2001).

The aim of this work is to examine the influence of current density and deposition time on current efficiency, thickness, roughness, and adhesion of electrochemically deposited nickel coatings in three baths with different combinations of additives. Furthermore, the aim is to investigate the influence of additives on the properties and quality of the coatings.

2. MATERIALS AND METHODS

Nickel coatings were electrochemically deposited at the Joint Stock Company for Production and Overhaul "Orao" a.d., Bijeljina. Steel cathodes ($5 \times 5 \times 0.2$ cm) of known chemical composition were used. The chemical composition of the samples was analyzed using the XRF method (X-ray fluorescence spectrometry), and the device used for this analysis was the X-MET 8000. Three different electrolyte baths were used for electrochemical deposition. Bath 1, which the company uses for industrial purposes, is a Watts electrolyte with the addition of a base additive and additives for ductility and wetting, and two additional baths of the same base composition, each containing different types of brighteners. The composition of the baths used and the working conditions for the electrochemical deposition of nickel coatings are presented in Table 1. Before deposition, all samples underwent mechanical preparation, i.e. sandblasting using 120–220 μm Al_2O_3 . This was followed by pickling (in $\text{HCl}:\text{H}_2\text{O}=1:1$ solution) and the application of a thin nickel film. Before the start of deposition, the cathodes prepared in this manner were placed in a heated electrolyte solution for a pre-

heating period of two minutes to reach optimal temperature before current was passed through the electrolyte. A nickel anode was used, and the holders to which the cathodes were attached were reinforced with copper wire to improve conductivity. Nickel coatings were deposited from the three baths in an electrochemical reactor with a volume of 64 dm^3 . The electrochemical nickel coatings were deposited for 10, 15, and 20 minutes at current densities of 1, 1.5, and 2 A/dm^2 , with the bath temperature maintained at 42 °C. After deposition, the samples were rinsed with running water, followed by dehydrogenation. The dehydrogenation process was carried out by heating the samples in a furnace at 200 ± 10 °C for 60 minutes, in order to remove hydrogen trapped within the coating during the electrolytic deposition process. All samples were then reweighed, and based on the increase in mass — that is, the difference in sample mass before and after coating — the cathodic current efficiency and coating thickness were calculated for each sample. The surface roughness of the coatings was measured using a "Mitutoyo" device (ISO 1302 standard), and hardness was measured using the Vickers method. Surface of the electrodeposited coatings was examined by Leica EZ4 HD optical microscope at 100 $\times$ magnification. The adhesion of the nickel coatings was tested using the bending method (DMP 14010 standard). Macro images of the specimens following the bending tests were captured using a Canon PowerShot SX280 HS digital camera with a resolution of 12.1 megapixels.

3. RESULTS AND DISCUSSION

Tables 2, 3, and 4 present the results of electrochemically deposited nickel coatings from the three different baths.

Figure 1 shows the dependence of average current efficiency on current density for all three baths at different nickel coating deposition times.

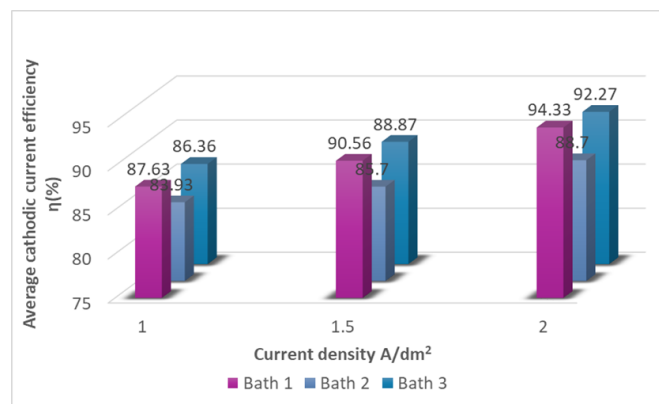


Figure 1. Dependence of average cathodic current efficiency on current density for baths 1, 2, and 3.

Table 1. Bath composition and operating conditions

	Bath 1	Bath 2	Bath 3
All three baths are based on Watts electrolyte with the same concentration: $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ 30–50 g/dm ³ H_3BO_3 15–30 g/dm ³ $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ 200–250 g/dm ³			
Composition	Base additive 302 (5 cm ³ /dm ³) Wetting additive 304 (8 cm ³ /dm ³) Ductility additive 305 (12 cm ³ /dm ³) (all additives by “Protekta,” Belgrade)	Base additive 302 (5 cm ³ /dm ³) Wetting additive 304 (8 cm ³ /dm ³) Ductility additive 305 (12 cm ³ /dm ³) High-gloss additive 301 (0.5 cm ³ /dm ³) (all additives by “Protekta,” Belgrade)	Base additive 302 (5 cm ³ /dm ³) Wetting additive 304 (8 cm ³ /dm ³) Ductility additive 305 (12 cm ³ /dm ³) Sulfo salicylic acid (0.5 cm ³ /dm ³)
Reactor volume	64 dm ³ (for all baths)		
pH	4.5–5.5		
Operating conditions	Current density: 1, 1.5, and 2 A/dm ² Deposition time: 10, 15, 20 minutes Temperature: 42°C		

Table 2. Results of electrochemical nickel coatings deposited from Bath 1

Bath 1: Watts electrolyte + base additive 302 (5 cm³/dm³), wetting additive 304 (8 cm³/dm³), and ductility additive 305 (12 cm³/dm³)

Current density j (A/dm ²)	Temp. T (°C)	Time t (min)	Current efficiency η (%)	Avg. current efficiency (%)	Coating thickness d (μm)	$v = d/t$ (μm/min)	V_{sr} (μm/min)
1	42	10	85.2	87.63	1.75	0.175	0.179
		15	87.6		2.69	0.179	
		20	90.1		3.65	0.183	
1.5	42	10	87.3	90.56	2.68	0.268	0.253
		15	90.6		4.18	0.276	
		20	93.8		4.33	0.217	
2	42	10	92.2	94.33	3.79	0.379	0.385
		15	94.7		5.82	0.388	
		20	96.1		7.80	0.390	

From Figure 1, it can be concluded that the cathodic current efficiency increases with the rise in current density for all three baths. The highest cathodic current efficiency is observed for Bath 1 (the bath without brighteners). This leads to the conclusion that brighteners slightly reduce current efficiency but have a positive effect on the appearance and quality of the coating. By observing the

results of the average current efficiency for baths 2 and 3, both of which contain brighteners, it can be seen that current efficiency is higher in Bath 3, where sulfosalicylic acid was used as a brightener, compared to Bath 2, which used the commercial high-gloss additive 301. This is a very favorable result, as sulfosalicylic acid can successfully replace the commercial brightener 301. It produces an

Table 3. Results of electrochemical nickel coatings deposited from Bath 2

Bath 2: Watts electrolyte + base additive 302 (5 cm³/dm³), wetting additive 304 (8 cm³/dm³), ductility additive 305 (12 cm³/dm³), and high-gloss additive 301 (0.5 cm³/dm³)

Current density j (A/dm ²)	Temp. T (°C)	Time t (min)	Current efficiency η (%)	Avg. current efficiency (%)	Coating thickness d (μm)	$v = d/t$ (μm/min)	V_{sr} (μm/min)
1	42	10	81.2	83.93	1.67	0.167	0.171
		15	83.5		2.57	0.171	
		20	87.1		3.53	0.175	
1.5	42	10	83.4	85.70	2.57	0.257	0.263
		15	85.7		3.95	0.263	
		20	88.2		5.37	0.268	
2	42	10	85.7	88.70	3.52	0.352	0.362
		15	89.3		5.49	0.366	
		20	91.1		7.40	0.370	

Table 4. Results of electrochemical nickel coatings deposited from Bath 3

Bath 3: Watts electrolyte + basic additive 302 (5 cm³/dm³), wetting agent 304 (8 cm³/dm³), and ductility additive 305 (12 cm³/dm³) + sulfosalicylic acid (0.5 cm³/dm³)

Current density j (A/dm ²)	Temp. T (°C)	Time t (min)	Current efficiency η (%)	Avg. current efficiency (%)	Coating thickness d (μm)	$v = d/t$ (μm/min)	V_{sr} (μm/min)
1	42	10	83.9	86.36	1.72	0.172	0.176
		15	86.5		2.66	0.177	
		20	88.7		3.61	0.181	
1.5	42	10	85.3	88.87	2.63	0.263	0.273
		15	89.1		4.11	0.274	
		20	92.2		5.62	0.281	
2	42	10	89.2	92.27	3.66	0.366	0.377
		15	92.5		5.69	0.379	
		20	95.1		7.72	0.386	

equally glossy nickel coating while achieving higher current efficiency.

Figure 2 shows the dependence of current efficiency on deposition time of the coatings for Baths 1, 2, and 3.

From Figure 2, it can be seen that the cathodic current efficiency increases with increasing deposition time and is highest after 20 minutes of deposition for all three baths (91–96.2%). The highest current efficiency is 96.1% for Bath 1, at a current density of 2 A/dm² and 20 minutes of deposition.

Figure 3 shows the nickel coating thickness as a function of deposition time for coatings electrodeposited at current densities of 1, 1.5, and 2 A/dm².

From Figure 3, it can be observed that the coating thickness increases with increasing deposition time, with the thickest coatings obtained after 20 minutes of deposition for all baths. The thickest coating, measuring 7.8 μm, was obtained from Bath 1 at a current density of 2 A/dm² and 20 minutes of deposition, which also corresponds to the highest current efficiency. In general, thicker coatings were obtained from Bath 1 (deposited without brighten-

ers) compared to baths 2 and 3 (with brighteners). Additionally, thicker coatings were obtained from Bath 3 (containing sulfosalicylic acid) compared to Bath 2 (with high-brightness additive 301).

Surface roughness was measured using a Mitutoyo device. Figure 4 presents the results of the measured roughness R_a (average profile deviation and maximum peak height, in μm). The measurement path length used to determine roughness was 0.8 mm.

The lowest surface roughness R_a was measured for coatings deposited from Bath 3 at all applied current densities, indicating that sulfosalicylic acid has a favorable effect on reducing surface roughness. Additionally, the resulting coatings exhibited excellent brightness.

To meet the conditions for good adhesion, the electroplated nickel coating must not detach from the base metal. The samples were bent using a bending tool, and observations were made to determine whether the coating cracked, peeled off, or if the bends remained coated with nickel. The obtained results are shown in Figures 5, 6, and 7.

For coatings obtained from all three baths, the bent surfaces remained coated with nickel, with no cracking or peeling of the coating from the base material. It can be concluded that the coatings from all three baths meet the

requirements for good adhesion. However, for coatings from baths 2 and 3, which contained brightening additives, a slight change in the shade of the coating was observed at the bend area. For the nickel coating deposited for 10 minutes from Bath 3 at 1.5 A/dm^2 , bubbles appeared, but the coating did not crack. Nevertheless, such a coating is unacceptable in terms of adhesion. This delamination may be due to somewhat poorer surface preparation before deposition compared to the other samples. All nickel coatings deposited from the three baths exhibit good adhesion except for the coating deposited for 10 minutes from Bath 3 at a current density of 1.5 A/dm^2 .

Samples were prepared for Vickers hardness testing using a diamond indenter in the shape of a true pyramid with a load of HV 0.2. Since the obtained coatings are very thin, the standard requirements for Vickers hardness measurement were not met.

Figures 8–10 show photographic images of the electrochemical nickel coatings taken with a Leica EZ4 HD optical microscope at $100\times$ magnification.

From Figures 8–10, it can be observed that the coatings obtained from all three baths exhibit similar structure and morphology. The surface coverage of the steel substrate is uniform and complete. However, it can be noted that the coatings from Bath 1 have slightly coarser

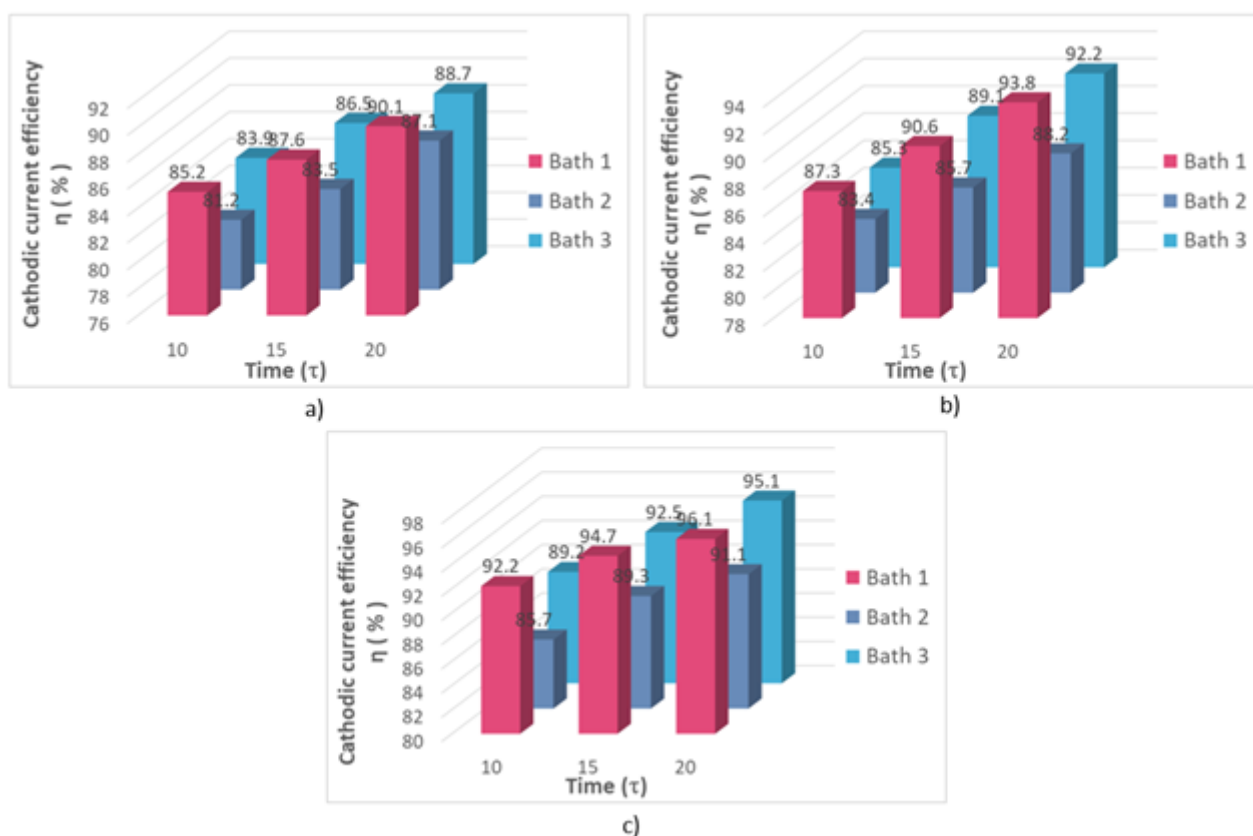


Figure 2. Dependence of current efficiency on deposition time at current densities a) 1 A/dm^2 , b) 1.5 A/dm^2 , and c) 2 A/dm^2 , for baths 1, 2, and 3.

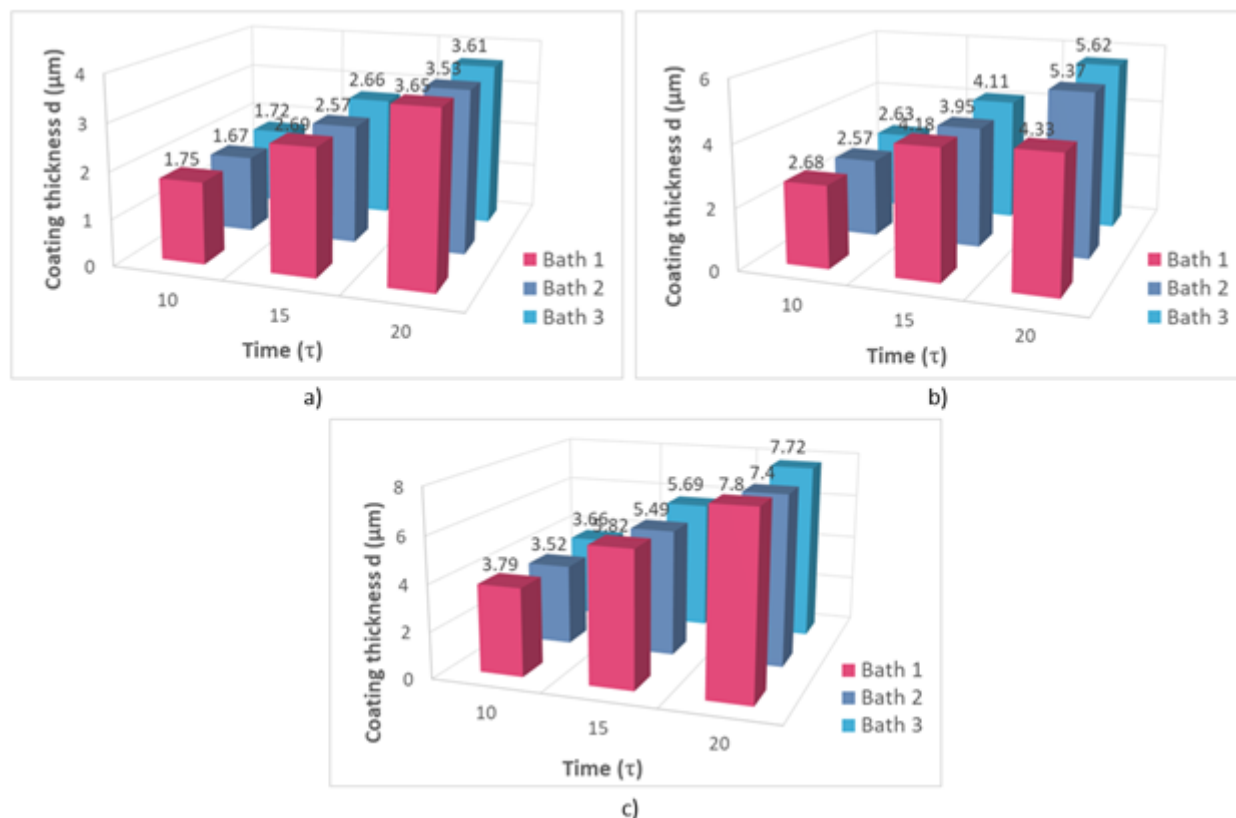


Figure 3. Dependence of coating thickness on deposition time for baths 1, 2, and 3 at current densities a) 1 A/dm², b) 1.5 A/dm², and c) 2 A/dm².

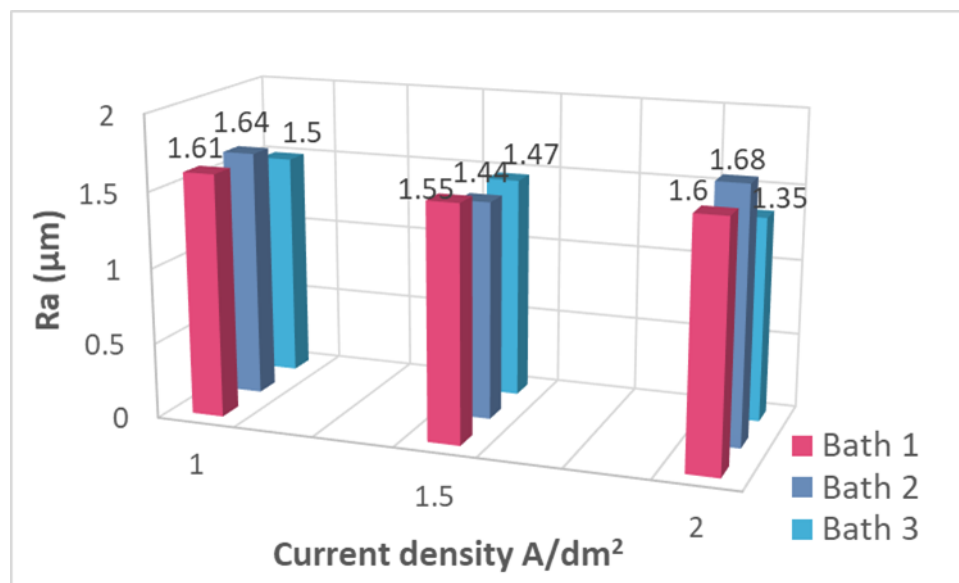


Figure 4. Average arithmetic profile deviation R_a as a function of current density.

grains compared to those from baths 2 and 3, and exhibit lower gloss. This is also confirmed by the surface roughness measurements (Figure 4), as coarser-grained coatings tend to have higher roughness. The nickel coatings obtained at all deposition times and current densities are homogeneous and compact. In the case of baths 2 and 3,

fine-grained and smoother coatings with a more attractive and shinier appearance can be observed, confirming that the addition of brighteners has a favorable effect on reducing surface roughness.

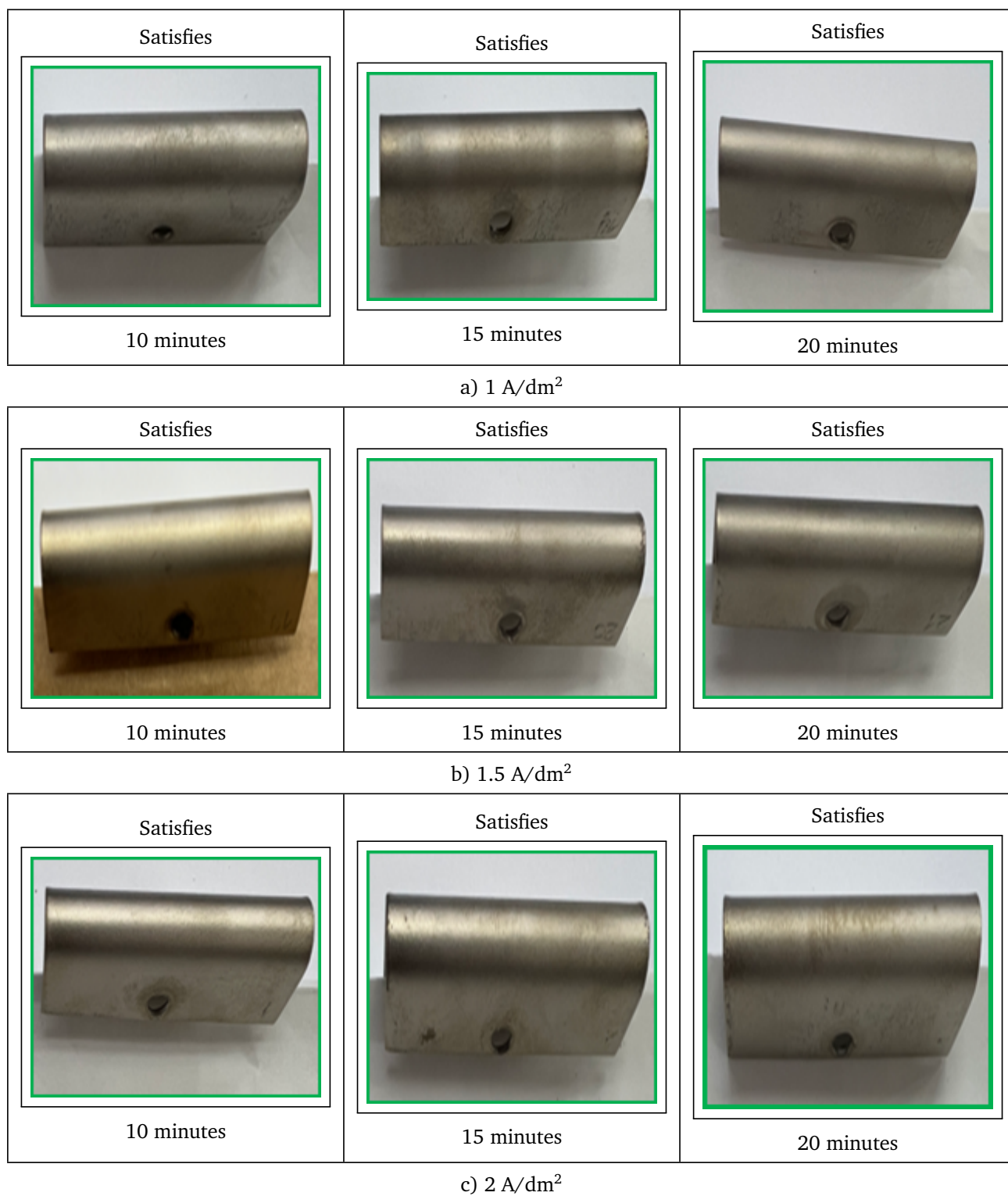


Figure 5. Photographic representation of the adhesion of nickel coatings deposited from Bath 1 at different current densities a) 1 A/dm², b) 1.5 A/dm², and c) 2 A/dm², and deposition times of 10, 15, and 20 minutes, at a temperature of 42 °C.

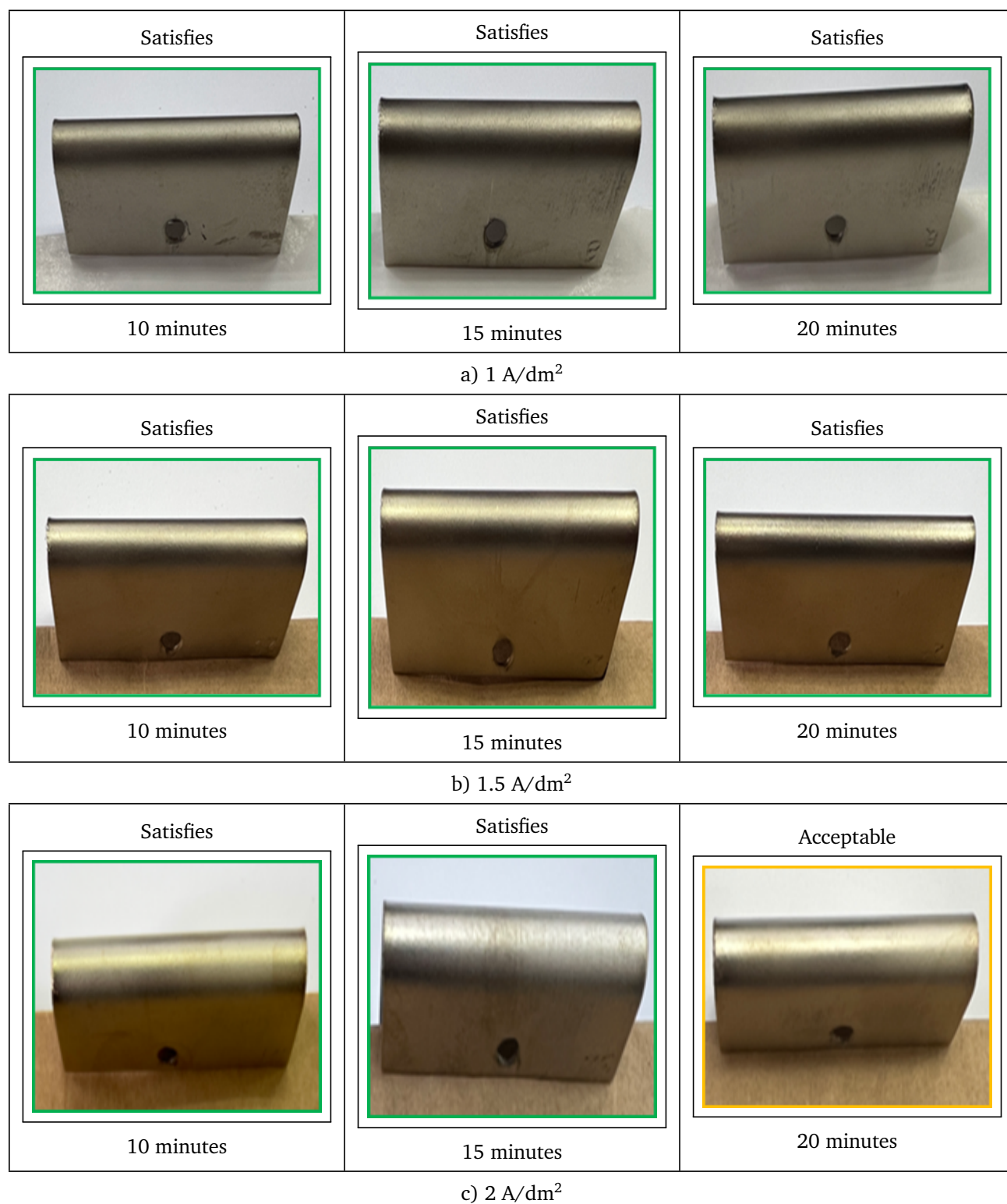


Figure 6. Photographic representation of the adhesion of nickel coatings deposited from Bath 2 at different current densities a) 1 A/dm², b) 1.5 A/dm², and c) 2 A/dm², and deposition times of 10, 15, and 20 minutes, at a temperature of 42 °C.

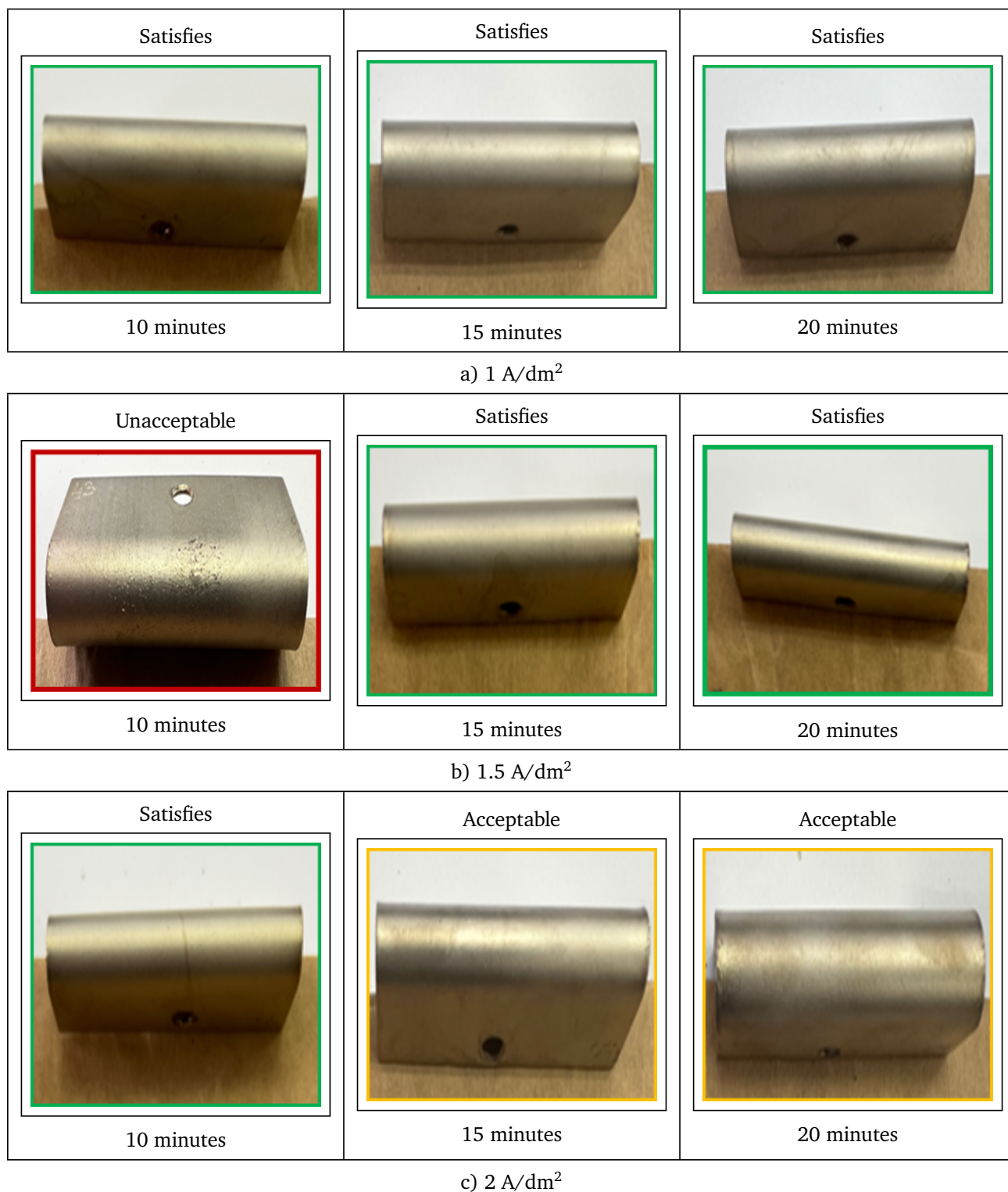


Figure 7. Photographic representation of the adhesion of nickel coatings deposited from Bath 3 at different current densities a) 1 A/dm², b) 1.5 A/dm², and c) 2 A/dm², and deposition times of 10, 15, and 20 minutes, at a temperature of 42 °C.

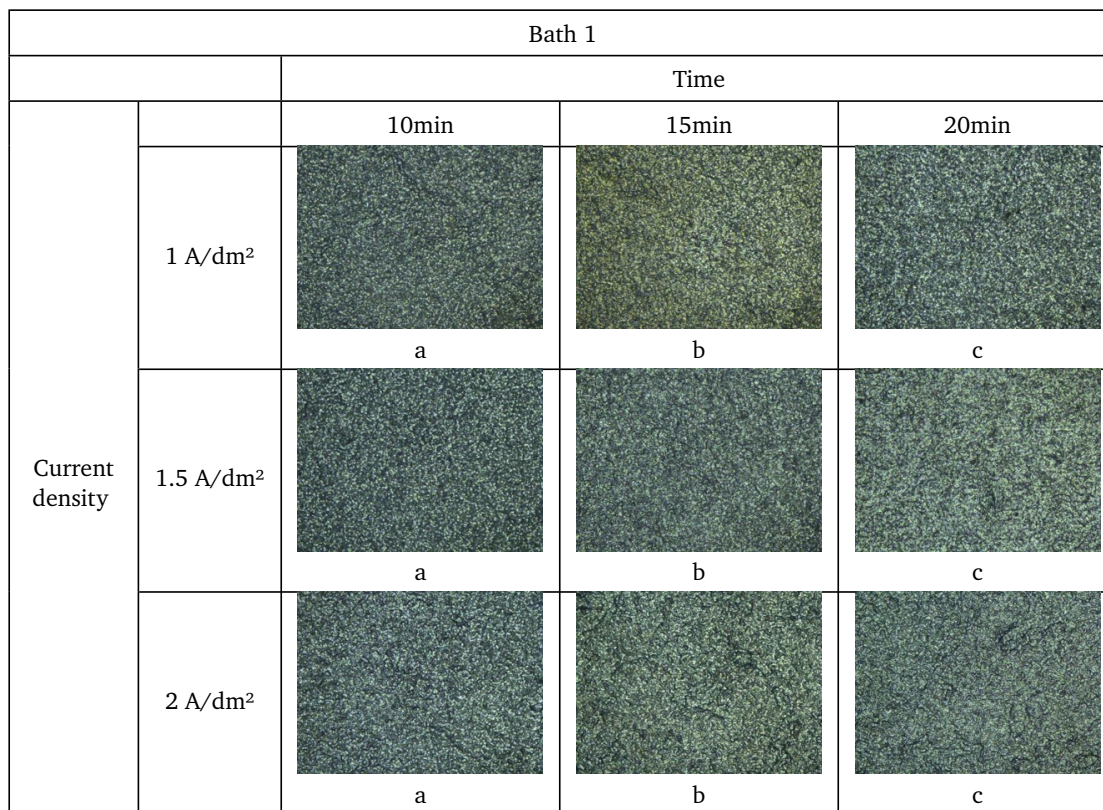


Figure 8. Photographic images of nickel coatings deposited from Bath 1: a) 10 min, b) 15 min, and c) 20 min, at current densities of 1, 1.5, and 2 A/dm². Magnification: 100×.

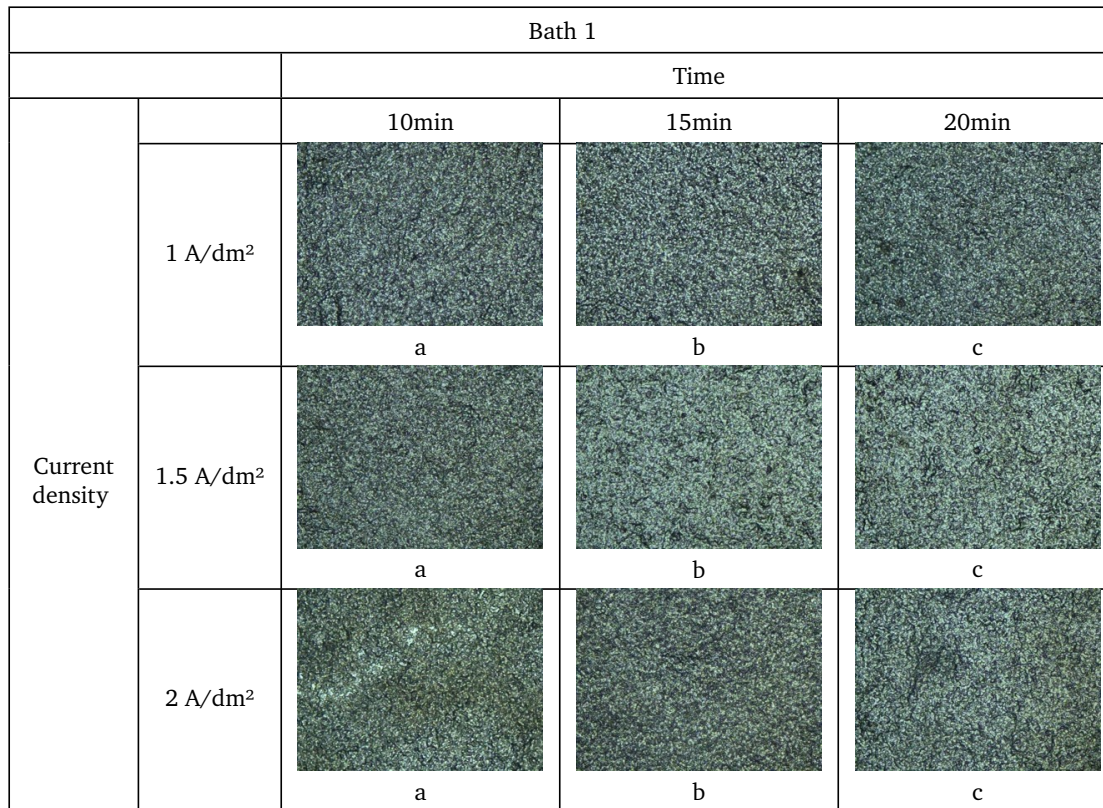


Figure 9. Photographic images of nickel coatings deposited from Bath 2: a) 10 min, b) 15 min, and c) 20 min, at current densities of 1, 1.5, and 2 A/dm². Magnification: 100×.

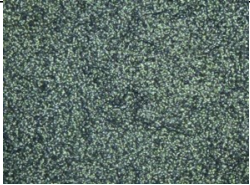
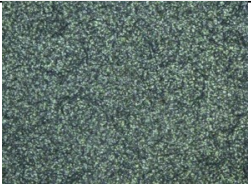
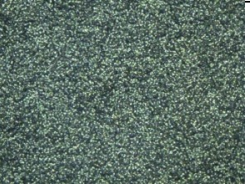
Bath 1				
		Time		
Current density		10min	15min	20min
	1 A/dm ²	 a	 b	 c
	1.5 A/dm ²	 a	 b	 c
	2 A/dm ²	 a	 b	 c

Figure 10. Photographic images of nickel coatings deposited from Bath 3: a) 10 min, b) 15 min, and c) 20 min, at current densities of 1, 1.5, and 2 A/dm². Magnification: 100×.

4. CONCLUSION

With the increase in current density, both the cathodic current efficiency and the thickness of the nickel coatings increase for all three baths under the given working conditions. Higher current efficiency and thicker coatings were obtained from Bath 1, which was used without brightening additives. The presence of brighteners reduces both the current efficiency and the coating thickness, but results in finer-grained, smoother, more attractive, and shinier coatings. The coatings obtained from Bath 3, which contained sulfosalicylic acid as a brightener, showed the lowest measured surface roughness values. Additionally, thicker coatings were obtained from Bath 3 (sulfosalicylic acid) compared to Bath 2 (containing high-brightness additive 301), indicating that sulfosalicylic acid is an effective brightener and may serve as a substitute for the commercial brightener 301. In general, the coatings obtained were of small thickness due to the short deposition times (10, 15, and 20 minutes), meaning that the conditions for Vickers hardness testing were not met. To perform hardness testing under the given conditions, the deposition time would need to be extended in order to produce thicker coatings that meet the required standard for this testing method. For most samples, adhesion was satisfactory — the coatings did not crack or

peel, and the samples remained coated with nickel after bending. Only one sample showed delamination (the sample from Bath 3, at a current density of 1.5 A/dm² and 10 minutes of deposition), and this coating is considered unacceptable.

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