

Electroless deposition of nickel boron coatings using morpholine borane as a reducing agent

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Nickel boron (NiB) coatings were deposited onto copper using a nickel-glycine (Ni-Gly) plating solution and morpholine borane (MB) as a reducing agent. It has been determined that using MB as a reducing agent in the Ni-Gly plating solution produces NiB coatings, which exhibit typical cauliflower-like textures. The deposition rate of the NiB coatings and their composition depend on the concentrations of the reducing agent (MB) and the ligand (Gly), in addition to the pH and temperature of the plating solution. The highest deposition rate ($3.42 \text{ mg cm}^{-2} \text{ h}^{-1}$) of the NiB coating was obtained when the plating bath was operated at pH 5 and 60°C temperature. Using this method, NiB coatings containing 10–20 at.% of boron can be obtained.

Keywords: electroless deposition, nickel, boron, morpholine borane, glycine

INTRODUCTION

Nickel and its alloy coatings obtained using the electroless metal deposition technique are widely used in many different areas of industry such as electronics, automotive, aerospace, medical, petrochemical and military applications. Such a wide field of application can be explained by a well-known combination of Ni and the properties of its alloys, such as high corrosion resistance, excellent wear resistance, uniformity of coating and magnetic properties [1–8]. Nickel-phosphorous alloys, which are obtained using sodium phosphinate as the reducing agent, are the most widely used and studied [9–12],

as they exhibit a very high corrosion resistance in very aggressive acidic media [13]. Ni coatings, containing boron, have also been widely investigated. Sodium and potassium borohydride are the most popular reducing agents used in the literature, but electroless plating with NaBH_4 solutions requires elevated temperatures (90–95°C) and a strong alkaline medium (pH 12–14) to achieve high deposition rates [14]. NiB alloys produced using NaBH_4 or amine borane compounds as the reducing agents are commonly reported in the literature; moreover, such NiB alloys are often described as high-performance coatings. The process of NiB coating deposition is simple and the chemicals used are relatively cheap, which makes NiB coatings more popular than other Ni alloy coatings used in industry. It is well known that boron containing compounds are

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stronger reducing agents as compared to phosphinates [15, 16]. Ni coatings containing boron exhibit higher hardness and better corrosion resistance as compared to those of Ni films with P [17–22]. The hardness of the NiB coatings increases with the amount of boron incorporated, while the corrosion resistance seems to be favourably influenced by higher boron concentrations [23]. As mentioned, electroless NiB coatings can be deposited using reducing agents such as borohydride [17, 24, 25] and amine boranes [26–30]. Amine boranes are effective reducing agents because they can be used in a wider range of pH, compared to the borohydrides. Ni plating baths employing amine boranes can operate in both acid and alkaline conditions. Dimethylamine borane (DMAB) is one of the most popular reducing agents among other amine boranes. NiB coatings fabricated via electroless deposition find extensive applications in different industrial fields due to simplicity of electroless deposition processes. DMAB, giving 6 electrons, is a stronger reducing agent in comparison to phosphinate, which gives only 2 electrons. The use of a stronger reducing agent, e.g. DMAB, for electroless metal deposition yields a self-activated (Pd-free) deposition [31, 32]. This process does not require the use of Pd nuclei for activation of the substrate due to the application of a stronger reducing agent [33]. Self-activated processes have been shown for cobalt deposition [31, 34–37]. As a result of these self-activated processes, highly selective Co-based metal films were formed directly on Cu lines when a DMAB was used as a reducing agent [37].

In the literature, some authors mention another boron-containing reducing agent – morpholine borane (MB), which has not been widely used in the electroless metal deposition to date. Previously, it was found that using MB as a reducing agent produced high-quality cobalt-boron coatings at a relatively low temperature of 30°C [16]. The suitability of MB to act as a reducing agent for the electroless deposition of metals is mentioned in some other works [16, 38–42]. However, there are practically no detailed data concerning this process or the properties of the coatings obtained. Compared to DMAB, MB is less volatile and toxic [43]. Therefore, the aim of this work was to investigate, in detail, electroless Ni deposition using MB as a reducing agent and plating solutions containing the additional ligand – glycine. We propose a new Ni plating bath using MB as a reducing

agent, which has not yet been thoroughly investigated. The main advantage of using MB as a reducing agent is the possibility to regulate and predict the composition (namely, the content of Ni and B) of the NiB coatings produced.

EXPERIMENT

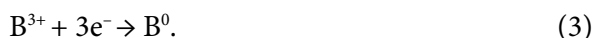
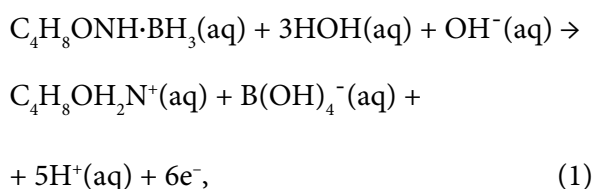
The NiB coatings were deposited on a 1 × 1 cm Cu sheet (model: C10200 pure copper plate, 99.90% purity, 0.4 mm thick). Prior to the electroless deposition of NiB coatings, the surface of the Cu sheet was pretreated with SiC emery paper (grade 2500) and MgO powder, then etched in 10% H₂SO₄ and rinsed with deionized water. After this degreasing procedure, Cu sheets were activated with Pd²⁺ ions by their immersion in a 0.5 g L⁻¹ PdCl₂ solution for 5 s, rinsed with deionized water and placed into an electroless Ni plating solution. The geometric area of the Cu sheet was 2 cm². The main composition of the electroless plating solution was the following (mol L⁻¹): NiSO₄ – 0.05, C₄H₈ONH·BH₃ (MB) – 0.02–0.30, NH₂CH₂COOH (glycine) – 0.40, CH₃COONa (sodium acetate) – 0.04. The deposition of coatings was performed from a freshly prepared solution at temperatures of 30 up to 60°C. The plating solution pH was in a range of 5–7. All chemicals used were of analytical purity grade. The thickness of the deposited films was determined from gravimetric data. The average deposition rate in mg cm⁻² h⁻¹ was calculated from at least three measurements. The surface morphology of the samples was characterized using a SEM/FIB Workstation Helios Nanolab 650 (FEI) arranged with an energy dispersive X-ray (EDX) spectrometer INCA Energy 350X-Max 20. TEM (XTEM) images of the cross-sections of the NiB coatings were done using an FEI TECNAI F20 field emission transmission electron microscope operated at 200 kV. The XTEM specimens were prepared by the FIB lift-out technique in the Helios NanoLab 650 dual beam microscope. Cross-sections were performed using a focused ion beam (FIB) with 30 keV energy Ga ions. Platinum was introduced on the top of the surface during sample cross sectioning.

The composition of the obtained NiB coatings was estimated using inductively coupled plasma optical emission spectroscopy (ICP-OES). ICP optical emission spectra were recorded using an ICP optical emission spectrometer Optima 7000DV (Perkin Elmer). The data on coating composition

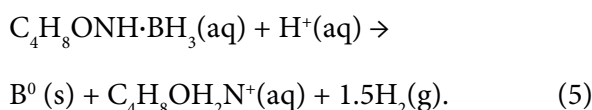
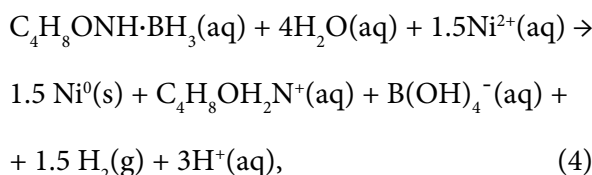
and deposition rates presented in this paper represent the average of at least three measurements. The variability of these measurements was found to be well within $\pm 2\%$ in all cases.

RESULTS AND DISCUSSION

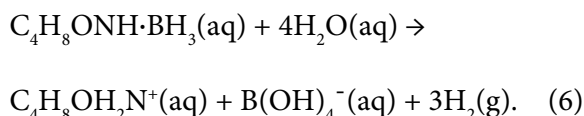
Electroless Ni plating solutions containing Gly, CH_3COONa and MB as a reducing agent are stable and may be used for deposition of the NiB coatings. Electroless deposition of the NiB coatings, using MB as a reducing agent, is a rather simple process; moreover, it can be viewed as the sum of two chemical reactions occurring simultaneously: the oxidation of MB on the catalytic surface (1) and the reduction of Ni^{2+} and B^{3+} ions (2, 3):



The electrons removed during the oxidation of MB are used in the reactions for reduction of Ni^{2+} and B ions. The overall reaction of electroless deposition of NiB coatings may be described as follows (Eqs. 4, 5):



At the same time, some quantity of MB may also be consumed in the following unproductive decomposition reaction (6):



NiB alloy coatings were deposited on the Cu surface using the plating baths operated at different pH values between 5–8 and different temperatures in the range 30–60°C. Figure 1 presents the dependence of the deposition rate ($\text{mg cm}^{-2} \text{h}^{-1}$) of the NiB coatings and their composition on the plating bath pH. The bath was operated at 30°C temperature and the concentration of MB was 0.02 mol L^{-1} . As seen from the data in Fig. 1a, the deposition rate decreases with an increase in solution pH over the whole investigated pH range. A significant decrease of the deposition rate is visible in a range of pH 7–8. The deposition rate decreases almost

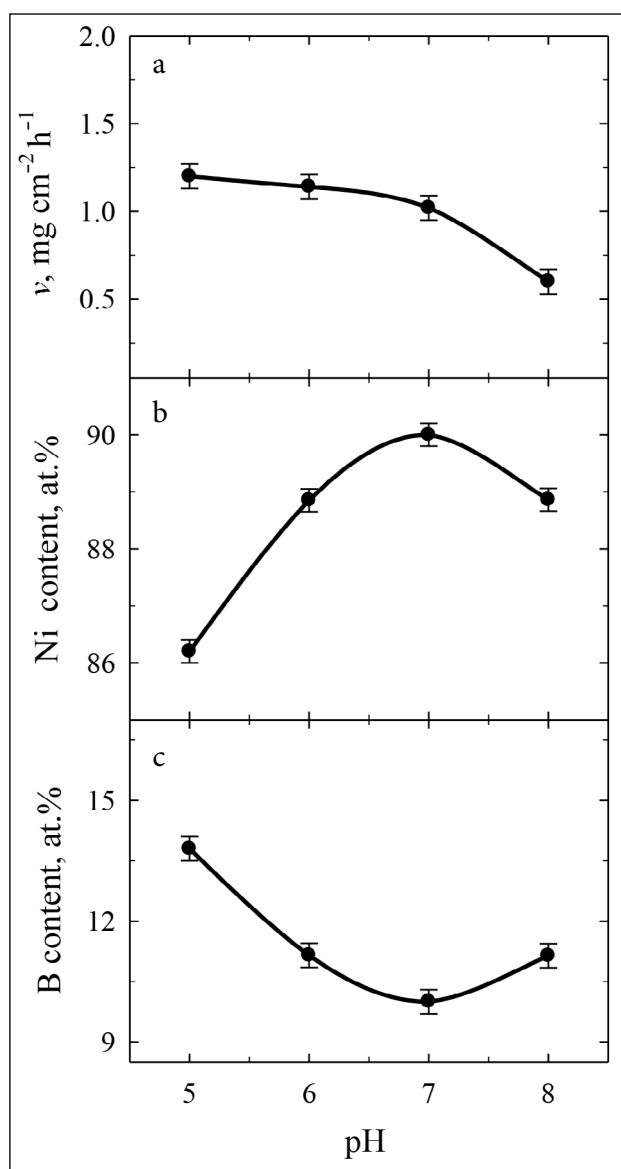


Fig. 1. Influence of plating bath pH on the deposition rate of NiB coatings and their composition. The solution composition (mol L^{-1}): $\text{NiSO}_4 - 0.05$, MB - 0.02 , $\text{CH}_3\text{COONa} - 0.04$, Gly - 0.40 , 30°C

twice. The highest NiB coating deposition rate of $1.20 \text{ mg cm}^{-2} \text{ h}^{-1}$ was obtained in the plating bath operated at pH 5. In addition, the highest boron content of 14.2 at.% was also achieved for the NiB coatings produced at pH 5 (Fig. 1c). With an increase in the plating solution pH from 5 to 7, the boron content in the NiB films was found to diminish, but a further increase in solution pH leads to a slight increase in the boron content (Fig. 1c). Furthermore,

the amount of Ni in the NiB coatings increases with the plating bath pH up to pH 7, followed by a decrease in the Ni content as the pH was increased further (Fig. 1b). It has been determined that the NiB coating with the highest Ni content of 90 at.% and the lowest boron content of 10 at.% were deposited from the plating bath operated at pH 7. Figure 2 shows the surface morphology of as-prepared NiB coatings deposited from the plating bath operated

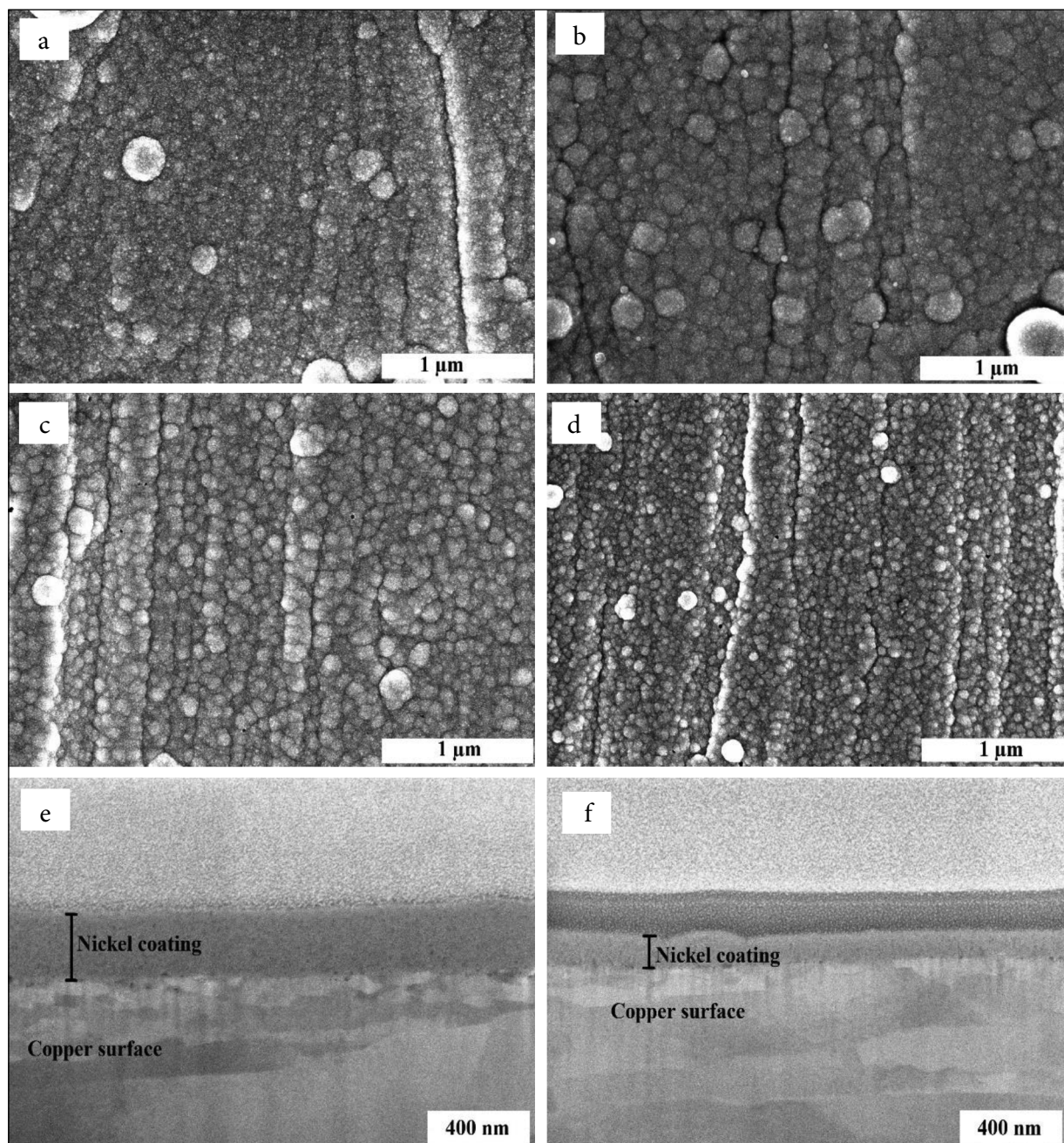


Fig. 2. SEM images of as-prepared NiB coatings deposited on the Cu surface from the solution containing (mol L^{-1}): $\text{NiSO}_4 - 0.05$, MB - 0.02, $\text{CH}_3\text{COONa} - 0.04$, Gly - 0.40, pH: 5 (a), 6 (b), 7 (c) and 8 (d), 30°C . Part of Fig. e and f represents the TEM images of the cross-section of the NiB coatings deposited on the Cu surface from the same solution at pH 5 and 7, respectively

at different pH values: 5 (a), 6 (b), 7 (c) and 8 (d). As evident from the data in Fig. 2, the surface morphology of all the coatings presents a typical cauliflower-like texture [44–48]; however, the surface morphology of the NiB coatings depends on the plating bath pH. It is clearly seen that all the coatings are nodulous, and with an increase in plating bath pH, the size of the surface features is significantly modified. Notably, at pH 5, the NiB coatings consisted of larger nodules ranging in size from approximately 130 to 340 nm and had the highest boron content (Fig. 2a), while coatings composed of smaller nodules of 50–160 nm in size were obtained from the solution at pH 8 (Fig. 2d).

TEM images of the cross-sections of the NiB coatings deposited from the plating bath operated at pH 5 and 7 are shown in Fig. 2e and f. It is evident that the NiB coatings deposited on the Cu surface are dense and continuous (Fig. 2e, f).

In spite of the fact that hydroxide ions influence oxidation of the reducing agent (MB), an increase in plating bath pH does not increase the plating rate of the NiB coating. It is thought that more stable Ni^{2+} complexes with Gly are formed in more alkaline media. As a result, the reduction of Ni^{2+} ions to Ni metal occurs via a more complicated pathway. The increase in plating bath pH from 5 to 8 results in a decrease in the plating rate of the NiB coatings. Moreover, a higher B content is incorporated in the coating as soon as the deposition rate begins to decrease (Fig. 1). A similar phenomenon was observed for the deposition of the NiP films from the Ni-Gly plating bath using sodium phosphinate as a reducing agent [49]. It should be noted that below pH 4, the Ni-Gly plating solutions are not stable, and the reduction of Ni^{2+} ions with MB occurs in the solution bulk.

The influence of the deposition rate of NiB coatings and their composition on the concentration of a reducing agent, e.g. MB, in the plating bath operated at pH 7 and at 30°C temperature is shown in Fig. 3. The results obtained have shown that the deposition rate of the NiB coatings remains practically unchanged with an increase in the concentration of MB from 0.02 to 0.10 mol L⁻¹ (Fig. 3a). With a further increase in the MB concentration from 0.10 to 0.30 mol L⁻¹, the deposition rate of the NiB coatings significantly decreases (Fig. 3a). The highest deposition rate of 1.02 mg cm⁻² h⁻¹ is obtained when the concen-

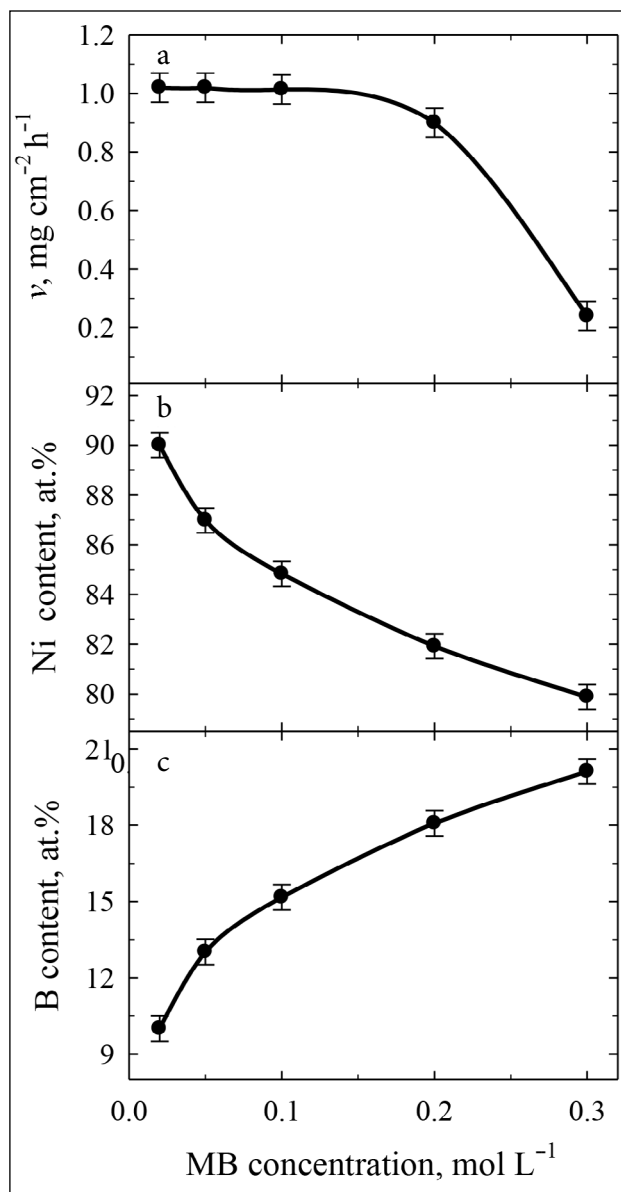


Fig. 3. Influence of the MB concentration on the deposition rate of the NiB coatings (a) and their composition (b, c). The solution composition (mol L⁻¹): $\text{NiSO}_4 - 0.05$, $\text{CH}_3\text{COONa} - 0.04$, Gly - 0.40, pH 7, 30°C

tration of MB in the plating solution is in a range of 0.02–0.10 mol L⁻¹. A general trend is observed: on the one hand, the Ni content in the coatings decreases from 90 to 80 at.% (Fig. 3b), on the other hand, the amount of boron simultaneously increases from 10 to 20 at.% (Fig. 3c), in the cases when the MB concentration in the plating solution is elevated from 0.02 to 0.30 mol L⁻¹.

The SEM images of the NiB coatings deposited on the Cu surface from the Ni-Gly plating baths, containing different concentrations of the reducing agent, MB, and operated at pH 7 and at 30°C temperature, are given in Fig. 4 (a–c). A cross-section

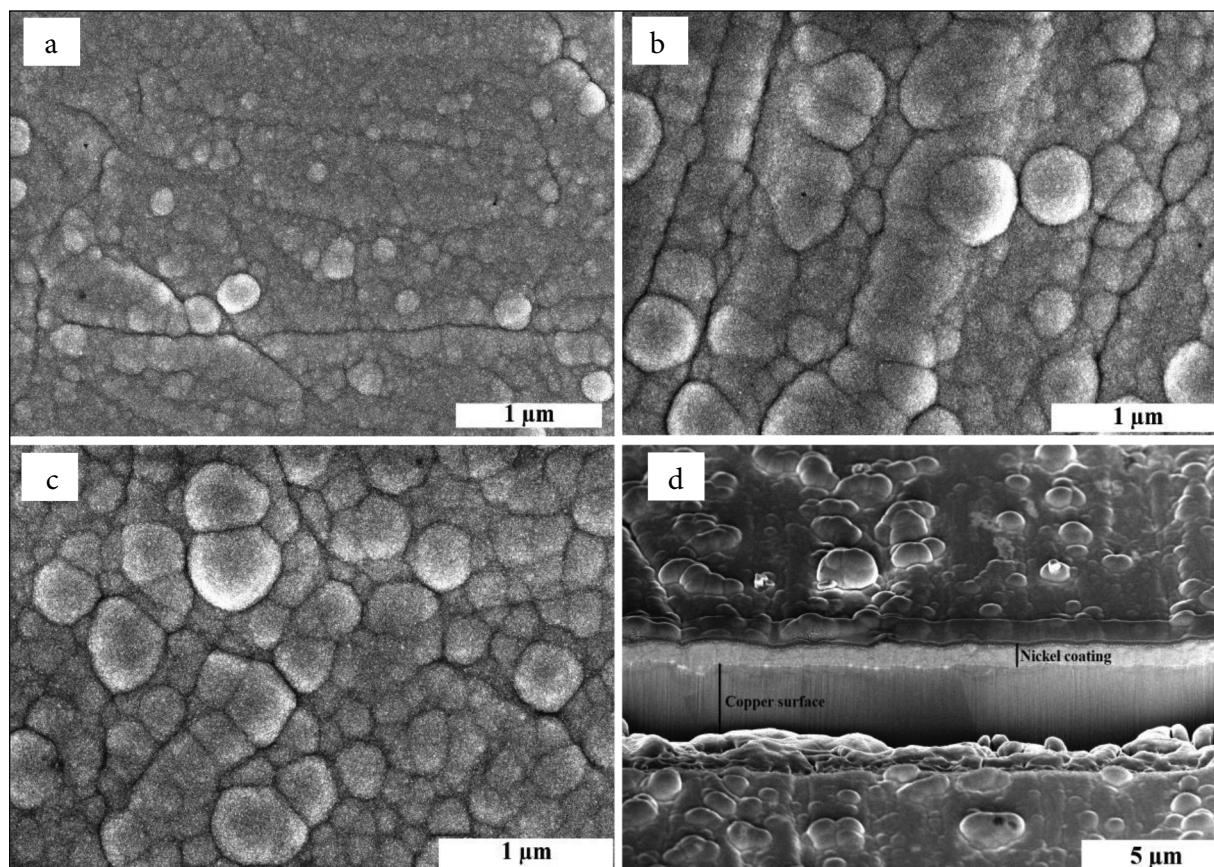


Fig. 4. SEM images (a–c) of as-prepared NiB coatings deposited on the Cu surface from the solution containing (mol L⁻¹): NiSO₄ – 0.05, CH₃COONa – 0.04, Gly – 0.40, MB – 0.05 (a), 0.1 (b) and 0.2 (c), pH 7, 30°C. Part of Fig. d represents the cross-section TEM image of the NiB coating deposited on the Cu surface in the same plating solution containing 0.05 mol L⁻¹ of MB

TEM image of the NiB coating, which was deposited on the Cu surface when the MB concentration in the plating bath was 0.05 mol L⁻¹, is shown in Fig. 4d. The deposited NiB coatings have also a cauliflower-like texture as shown in Fig. 4 (a–c). It can be noted that an increase in the MB concentration from 0.02 (Fig. 2c) to 0.20 (Fig. 4a–c) mol L⁻¹ in the plating solution caused a formation of more pronounced nodulus in the coatings obtained. A lower concentration of MB in the plating bath leads to the deposition of the NiB coating consisting of a smaller nodulus in size of approximately 80–260 nm and containing a lower B amount (Fig. 2c) as compared with that obtained from the plating bath containing a higher MB concentration (Fig. 4a–c). The sizes of nodules are significantly larger and are in a range of approximately 130–320, 225–600 and 240–660 nm for the NiB coatings obtained from the plating bath containing a concentration of MB equal to 0.05, 0.10 and 0.20 mol L⁻¹, respectively (Fig. 4a–c). As evident from the cross-section TEM im-

age in Fig. 4d, the adhesion of the NiB coating to the substrate seems to be quite good.

The influence of the deposition rate and changes in the composition of the NiB coatings, depending on the concentration of a reducing agent (e.g. MB), are shown in Fig. 3, for a plating bath operated at pH 7 and 30°C temperature. The results obtained have shown that the deposition rate of the NiB coatings remains practically unchanged with an increase in the concentration of MB from 0.02 to 0.10 mol L⁻¹ (Fig. 3a). With a further increase in the MB concentration from 0.10 to 0.30 mol L⁻¹, the deposition rate of the NiB coatings decreases significantly (Fig. 3a). The highest deposition rate of 1.02 mg cm⁻² h⁻¹ is obtained when the concentration of MB in the plating solution is in a range of 0.02–0.10 mol L⁻¹. A general trend is observed when the MB concentration in the plating solution is elevated from 0.02 to 0.30 mol L⁻¹: the Ni content in the coatings decreases from 90 to 80 at.% (Fig. 3b), while the amount of boron simultaneously increases from 10 to 20 at.% (Fig. 3c).

SEM images of the NiB coatings deposited on Cu from the Ni-Gly plating baths, containing different concentrations of MB, and operated at pH 7 and 30°C, are given in Fig. 4 (a–c). A cross-section TEM image of the NiB coating deposited on Cu at an MB concentration of 0.05 mol L⁻¹ is shown in Fig. 4 d. The deposited NiB coatings have a cauliflower-like texture as shown in Fig. 4 (a–c). It can be noted that an increase in the MB concentration from 0.02 (Fig. 2c) to 0.20 (Fig. 4a–c) mol L⁻¹ in the plating solution caused a formation of more pronounced nodules in the coatings obtained. A lower concentration of MB in the plating bath leads to the deposition of NiB coatings consisting of smaller nodules with sizes of approximately 80–260 nm and a lower boron content (Fig. 2c) as compared with that obtained from the plating bath containing a higher concentration of MB (Fig. 4a–c). NiB coatings obtained from the plating bath containing MB concentrations of 0.05, 0.10 and 0.20 mol L⁻¹ exhibited significantly larger nodules with sizes ranging from approximately 130–320, 225–600 and 240–660 nm, respectively (Fig. 4a–c). As evident from the cross-section TEM image in Fig. 4d, the NiB coating appears to be well adhered to the substrate.

The influence of the Gly concentration on the deposition rate of NiB coatings and their composition is shown in Fig. 5. When the concentration of Gly in the plating bath is increased from 0.10 to 0.40 mol L⁻¹, the deposition rate decreases sharply (Fig. 5a). Theoretically, the ratio of the Ni and Gly in the complex must be 1:2. An additional amount of Gly ensures a sufficient stability of the plating solution. An increase in the Gly concentration in the solution from 0.10 to 0.20 mol L⁻¹ leads to a decrease in the Ni content in the coatings obtained. In this case, the highest boron content was 11.8 at.%, observed in coatings deposited when the Gly concentration in the plating bath was 0.20 mol L⁻¹ (Fig. 5c). Furthermore, with an increase in the Gly concentration up to 0.40 mol L⁻¹, the Ni content in the coating increased slightly (Fig. 5b), while the B content decreased slightly (Fig. 5c).

The data on the dependence of the plating rate and the composition of the NiB coatings on temperature show that, in all cases, the Ni deposition rate increased with increasing the plating bath temperature (Fig. 6a). The highest coating depo-

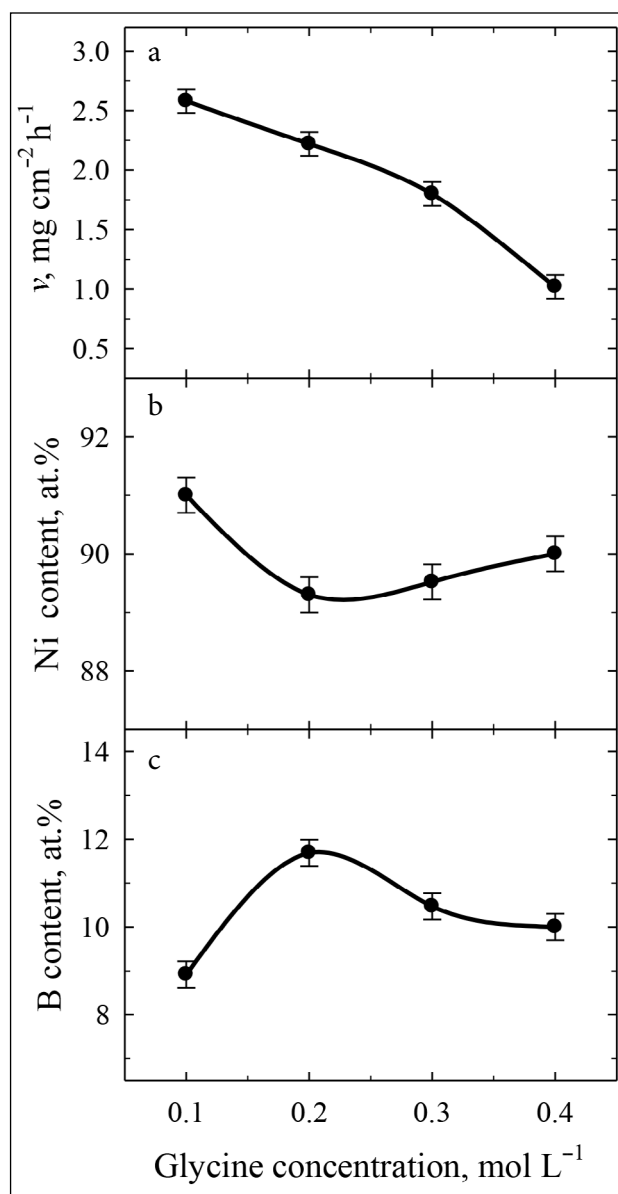


Fig. 5. Influence of the ligand (Gly) concentration on the deposition rate of the NiB coatings (a) and their composition (b, c). The solution composition (mol L⁻¹): NiSO₄ – 0.05, MB – 0.02, CH₃COONa – 0.04, pH 7, 30°C

sition rate was obtained using the plating bath at pH 5 and 60°C temperature (Fig. 6a). In this case, the deposition rate was 3.42 mg cm⁻² h⁻¹. Furthermore, an increase in temperature from 30°C to 60°C resulted in an increase in the deposition rate from 1.20 to 3.42 mg cm⁻² h⁻¹ at pH 5, from 1.14 to 3.30 mg cm⁻² h⁻¹ at pH 6, and from 1.02 to 3.00 mg cm⁻² h⁻¹ at pH 7. At pH 5, increasing the operating temperature of the plating bath led to an increase in the Ni content of the coating, whereas the B content decreased (Fig. 6b, c). ICP-OES analysis of the NiB coatings produced shows

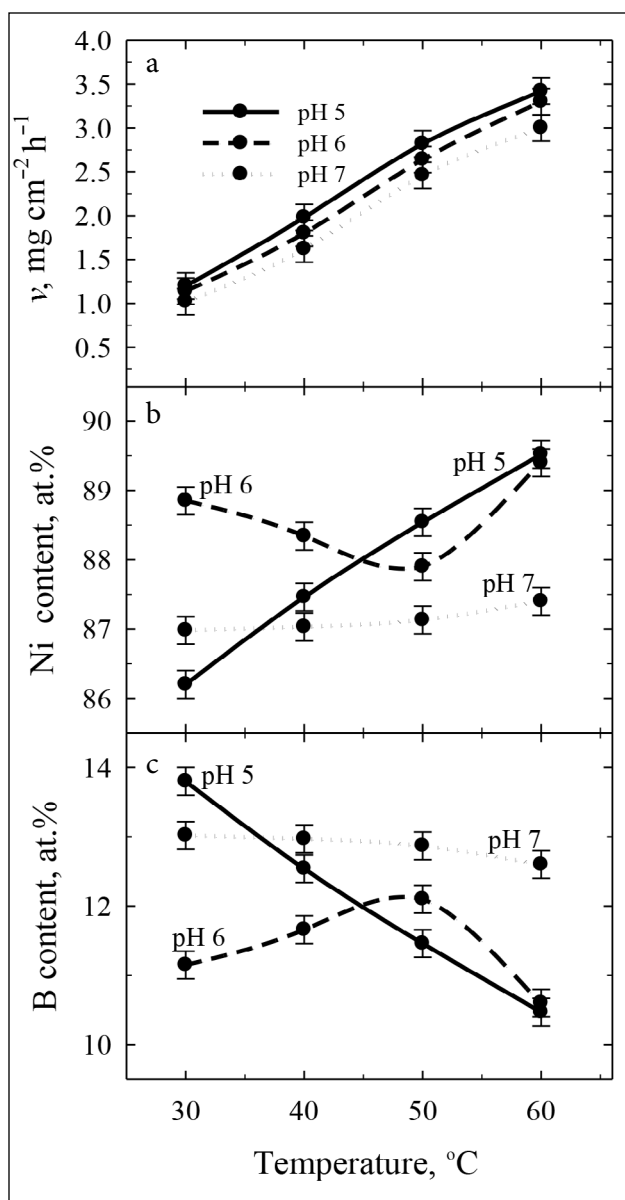


Fig. 6. Influence of temperature on the deposition rate of the NiB coatings (a) and their composition (b, c). The solution composition (mol L^{-1}): $\text{NiSO}_4 - 0.05$, MB - 0.05, $\text{CH}_3\text{COONa} - 0.04$, Gly - 0.40

that the coatings contained 86.2–89.5 at.% of Ni and 10.4–13.8 at.% of boron. At pH 6, an increase in the plating bath temperature from 30 to 50°C resulted in a diminished Ni content in the coatings obtained. Further increasing the temperature increased the Ni content, reaching 89.4 at.%; however, the B content of these coatings decreased. When the plating bath was operated at pH 7, the Ni and B contents in the coatings remained practically the same. In this case, the concentration of Ni detected in the coatings ranged from 86.9 to 87.4 at.%, while that of boron ranged from 12.6 to 13 at.% (Fig. 6b, c). It should be noted that at higher temperatures, the deposi-

tion of Ni occurred both in the solution bulk and on the walls of the vessel, indicating that the solutions were not adequately stable.

CONCLUSIONS

NiB coatings were deposited from a Ni-Gly plating solution onto Cu, using MB as a reducing agent. The effects of the MB reducing agent, the Gly ligand, solution pH and temperature on the electroless deposition rate of the NiB coatings have been investigated.

It has been determined that NiB coatings, which exhibit a typical cauliflower-like texture, can be obtained in the Ni-Gly plating solution using MB as a reducing agent. Moreover, the electroless deposition rate and composition of the NiB coatings depend on the concentrations of the reducing agent (MB) and the ligand (Gly), as well as the pH and temperature of the plating solution. The highest deposition rate ($3.42 \text{ mg cm}^{-2} \text{ h}^{-1}$) of the NiB coating was obtained when the plating bath was operated at pH 5 and 60°C temperature. Using the electroless plating deposition method described, NiB coatings containing 10–20 at.% of boron can be obtained.

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**CHEMINIS NIKELIO-BORO DANGŲ
NUSODINIMAS NAUDOJANT REDUKTORIUMI
MORFOLINO BORANĄ**

S a n t r a u k a

Nikelio-boro dangos buvo nusodintos cheminiu metalų nusodinimo metodu glicininiuose tirpaluose naudojant reduktoriumi morfolino boraną. Nustatyta, kad tokiu būdu galima gauti įvairios sudėties, kokybiškas nikelio-boro dangas vario paviršiuje. Ištirta, kad NiB dangų nusėdimo greitis ir jų elementinė sudėtis priklauso nuo reduktoriaus, ligando koncentracijos, nuo tirpalo pH ir temperatūros. Didžiausias dangų nusėdimo greitis nustatytas esant tirpalo temperatūrai 60 °C ir pH 5, kai pasiekiamas didžiausias dangų nusėdimo greitis – 3,42 mg cm⁻² h⁻¹. Taikant cheminį metalų nusodinimo metodą galima gauti NiB dangas, turinčias 10–20 at.% boro.