

MECHANICAL TESTING WITH MODELLING OF STRESS DISTRIBUTION IN NICKEL THIN COATINGS ON DIFFERENT SUBSTRATES AS A METHOD FOR ASSESSING ADHESION QUALITY

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ABSTRACT

Purpose: Covering materials with thin coatings is often necessary to protect them from wear and corrosion, ensure electrical conductivity, or form the structural microelectromechanical elements. For a composite system to maintain its integrity, it is necessary to understand its mechanical properties. The most important property that evaluates the integrity of composite systems is the adhesion strength between the substrate and the coatings. Poor adhesion can lead to the deterioration of the protected material or the failure of a component, or result in significant property damage, serious injuries, or loss of life. Analysing material adhesion problems and their causes of failure is crucial for forensic engineers.

Design/Methods/Approach: Thin foils of brass and stainless steel were chosen as the substrate materials. Nickel coatings were electrodeposited from sulphamate electrolyte. The bidirectional bending test, performed on a special structure, and the cross-hatching test were used for mechanical testing of adhesion. The number of bending cycles until material failure is determined. An analysis of stress distribution was conducted in addition to the bidirectional bending test. These results contributed to a better understanding of the mechanical behaviour of the system during bending, particularly in areas with high stress concentrations that may cause damage. After performing the cross-hatching test, the adhesion quality was assessed. A relation was made between adhesion quality criteria.

Findings: The crystallographic and mechanical properties of both the substrate and the coating, as well as the deposition process parameters and the thickness of the coatings, must be considered. Better adhesion is achieved with thin coatings and a similar crystallographic structure. Mathematical modelling of mechanical tests facilitates the monitoring of the testing process.

Originality/Value: Connecting the results of adhesion testing to the analysis of stress distribution will contribute to a better understanding of the phenomena that occur in composite systems. A quantitative relationship was established between the adhesion criteria.

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Keywords: coating adhesion, nickel electrodeposition, bidirectional bending test, the critical cycle number, crosshatch adhesion testing, stress distribution

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PURPOSE

Covering materials with thin coatings is often necessary for protecting them from wear and corrosion, ensuring electrical conductivity, and also in planar technologies for forming the structural elements of MEMS (microelectromechanical systems) components (Mittal, 1976; Medou, 1997; Ellahi, 2020). Substrates and one or more coatings on them constitute a composite material system. For the coating and substrate composite system to be of intact integrity, it is necessary to know and coordinate their mechanical properties. If substrates are not adequately chosen to provide coating support, there is a high risk of substrate and coating deformation under stress. The most important property that evaluates the integrity of the composite systems is adhesion strength between the substrate and coatings (Zhang et al., 2019; Chen et al., 2014). Adhesion strength is the maximum force per unit width of the sample that the coating/substrate system can withstand without showing coating failure.

Poor adhesion can lead to the deterioration of the protected material or the rapid failure of a component - that is, the separation or delamination of the coating from the substrate. Still, it can also lead to more serious accidents resulting in significant property damage, serious injuries, or loss of life. Therefore, analysing material adhesion problems and their causes of failure is crucial for forensic engineers (Peruzzi, 2021; Davis & Bond, 1999).

The adhesion of coatings on substrates is a peculiar and complex phenomenon that depends on surface energy, substrate cleanliness (presence of defects and impurities), elastic and thermal mismatch between the substrate and coating (mismatch of their elastic modulus and their coefficient of thermal expansion), roughness, and coating thickness, among other factors (Mittal, 1976; El-Shabasy, 1981; Chen et al., 2014; Rudawska, 2016; Lamovec et al., 2016, 2019; Mladenovic et al., 2017, 2025).

The importance of a thorough assessment of the adhesion strength of coatings on different substrates is evident from the considerable effort invested in developing various quantitative and qualitative methods for its evaluation. A broad classification of methods for characterizing the adhesion of coatings includes nucleation-based, mechanical, and other miscellaneous techniques. Nucleation methods for adhesion assessment often refer to methods that examine how coatings initiate bonding at the atomic or molecular level (e.g., during electrochemical deposition). Miscellaneous techniques typically include thermal, acoustic, and spectroscopic methods, as well as hybrid approaches that combine elements from multiple testing domains.

However, the most significant number of tests and adhesion assessment results in the scientific and professional literature were obtained through mechanical testing of composite coating systems on substrates (Chen et al., 2014; Valli, 1986; Hull et al., 1987; Rezaee et al., 2019). The mechanical methods that are most often used to assess the adhesion strength are the direct pull-off method (Valli, 1986), bending testing (Rezaee et al., 2019; ASTM International, 2022; Zhu et al., 2012), the scratch testing (Zhang et al., 2019; Konuru et al., 2021; Bull, 1997), and indentation testing (Zhang et al., 2019; Konuru et al. 2021; Magagnin et al., 2003; Mladenovic et al., 2019; Lamovec et al., 2018; Hou et al., 1999). The application of modern software packages and the possibility of modelling and simulating real engineering problems have greatly facilitated their understanding and solving (Jindra et al., 2021).

DESIGN/METHODS/APPROACH

Electrochemical deposition is a widely used technology for producing high-quality metal coatings on substrates, with nickel being one of the most commonly deposited metals. Electrochemical deposition techniques leave much room for manoeuvring process parameters, enabling precise control over the



resulting coating microstructure. The microstructure directly affects the mechanical properties of adhesion in coating/substrate composite systems. Composite systems are made by electrochemically depositing nickel of varying thicknesses onto brass and stainless-steel substrates, and they represent material systems with different structural and mechanical properties (Ebrahimi et al., 1999; Lamovec et al., 2008).

Bidirectional bending and cross-hatching tests were employed to evaluate the adhesion of nickel films on various substrates; both techniques are classified as destructive methods for assessing adhesive properties. The bidirectional bending adhesion testing involves the dynamic cyclic bending of a composite system, comprising nickel coatings on brass and stainless-steel substrates, using a specially designed splint. The number of cycles until the appearance of the first signs of coating separation, i.e., the occurrence of delamination of the coating from the substrate, is determined, and this number is called the critical number of cycles (Zhu et al., 2012; Yu et al., 2012; Mušicki et al., 2025). According to the applicable standard, the cross-hatching method evaluates the adhesion quality of the nickel coating by determining the fraction of the surface that detaches from the substrate. The adhesion quality is quantified as the percentage of the coating removed after a specialized adhesive tape is applied to and pulled from the hatched area (ASTM International, 2022). Simulation of the bidirectional bending process enabled the analysis of stress distribution within the coatings during deformation, providing insight into the experimental results.

Thin foils of brass and stainless steel were prepared for use as substrate materials. Dimensions of the samples prepared for different adhesion tests were 10 mm x 100 mm and 20 mm x 100 mm. The adhesion quality of coatings to the substrate depends on thorough substrate preparation. The preparation process involves mechanical polishing followed by degreasing and concludes with thorough rinsing and drying of the substrates. Brass and stainless-steel substrates were polished using silicon carbide (SiC, P600-1200) sandpaper of varying grit sizes, followed by sequential rinsing with acetone, ethyl alcohol, and distilled water.

Electrically non-conductive and thermally insulating tape was used to define the specific areas of the substrate surface designated for electrochemical deposition of nickel coatings. Surface areas measuring 10 mm x 60 mm and 20 mm x 20 mm were defined for the bidirectional bending and the cross-hatching testing, respectively.

Nickel coatings were electrochemically deposited from a sulphamate-based electrolyte composed of 300 g/L $\text{Ni}(\text{NH}_2\text{SO}_3)_2 \cdot 4\text{H}_2\text{O}$ (Sigma Aldrich, CAS Number: 124594-15-6), 30 g/L $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (Sigma Aldrich, CAS Number 7791-20-0), 30 g/L H_3BO_3 (Supelco, CAS Number: 10043-35-3), and 1 g/L saccharin (Sigma Aldrich, CAS Number: 81-07-2). The electrochemical deposition process was carried out in DC galvanostatic mode at a constant current density of 33 mA/cm². The electrolyte temperature and pH were maintained at 50 °C and 4.20, respectively. The electrolyte was constantly stirred with a magnetic stirrer to prevent the inclusion of hydrogen, which is released simultaneously at the cathode, into the nickel coatings. The duration of electrodeposition was calculated based on the selected deposition area, applied current density, and projected coating thicknesses of 5, 10, and 20 µm. For microstructural analysis, a sample with a 10-µm-thick nickel coating was etched in a solution containing 25 ml H_2O , 25 ml glacial CH_3COOH , and 50 ml HNO_3 for 60 seconds to reveal the grain boundaries (Lamovec et al., 2008).

Topographic examination of the brass and stainless-steel substrate surfaces and composite systems with electrochemically deposited nickel coatings was performed using optical and atomic force microscopy (AFM, AutoProbe CP Research; TM Microscopes, Veeco Instruments, Santa Barbara, CA, USA). Based on topographic AFM images, it was possible to obtain information about the roughness of the substrate surface, which is an important parameter for assessing adhesion. Performing bidi-



rectional bending tests for adhesion assessment required developing a specially designed structure (splint) based on literature references, as presented in Figure1 (Zhu et al., 2012).

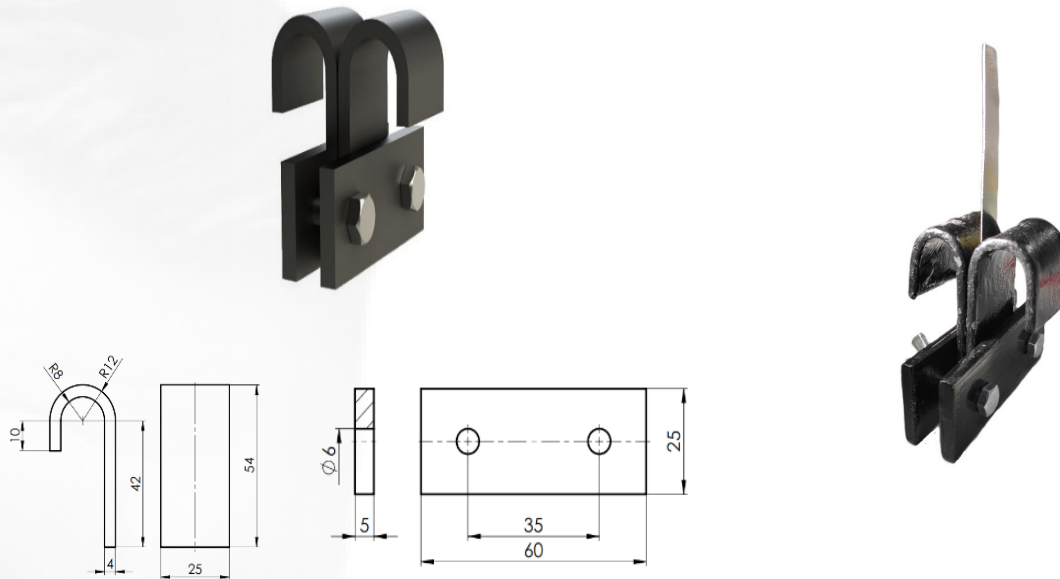


Figure 1: Computer-aided design and realisation of bending supports for the bidirectional testing. The starting position of the test specimen fixed in the splint is shown.

The samples prepared for this test measured 10 mm $\times$ 100 mm, with a nickel-coated area of 10 mm $\times$ 60 mm. Adhesion assessment was carried out as follows: each sample was inserted and securely clamped between bending supports within a specially designed splint, which was fastened using screws and nuts. While holding the free end of the sample, it was bent firmly to one side and held in position for several seconds. The procedure was then repeated in the opposite direction to complete the bidirectional bending cycle. This procedure constitutes one bending cycle. The test determines the critical number of cycles, defined as the point at which adhesion failure occurs and the coating begins to delaminate or peel off. The selected maximum number of bending cycles was 100.

The cross-hatching method for evaluating coating adhesion was performed following ASTM D3359-22, Test Method B (ASTM International, 2020). A cutting tool with a sharpened edge was used to create the grid pattern manually. Parallel cuts were made in one direction, followed by a second set of cuts perpendicular to the first, forming a cross-hatch grid. Each cut was 20 mm in length and spaced 1 mm apart. Cutting was performed by pulling in one smooth motion, using sufficient pressure to reach the substrate as shown in Figure 2.

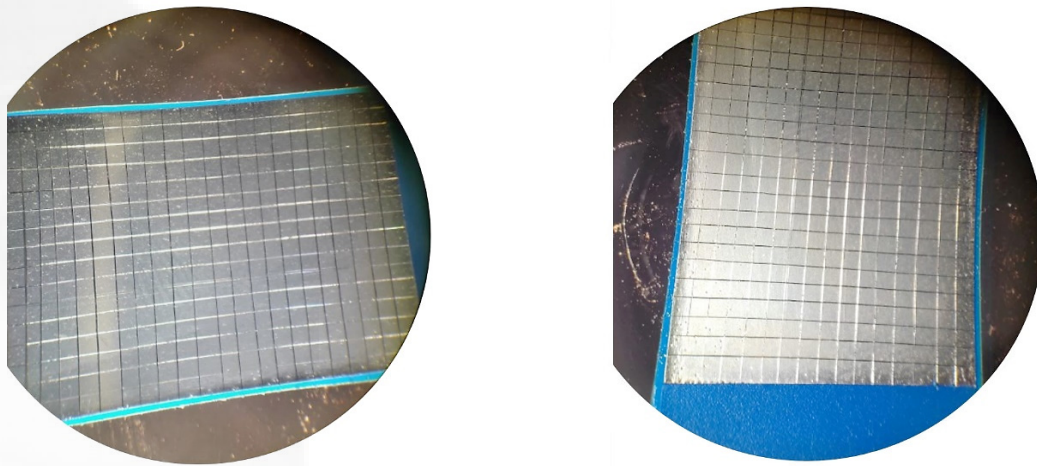


Figure 2: *The initial phase of cross-hatch testing, the process of drawing a cross-hatch on a nickel coating*

Detached portions of the coating were gently removed with a soft brush to minimise any influence on the test results. A pressure-sensitive tape (type 3M duct tape, Deutschland GmbH) was applied by placing its centre directly over the cross-hatch grid. The tape was gently smoothed to eliminate any air pockets between the tape and the nickel coating surface. After a waiting period of 90 seconds, the tape was removed by quickly pulling it back from the free end at a consistent angle. Adhesion was evaluated based on the percentage of the coating removed from the substrate, using the classification system defined in ASTM D3359-22, as follows:

- 5B: 0% area removed
- 4B: Less than 5% area removed
- 3B: 5–15% area removed
- 2B: 15–35% area removed
- 1B: 35–65% area removed
- 0B: More than 65% area removed

Modelling and simulation of the bidirectional bending testing was performed in the ANSYS 2025 R2 program. The model was formed according to the real dimensions of the splint for bidirectional bending testing.

FINDINGS

Adhesion performance was analysed for composite systems of electrochemically deposited nickel coatings of varying thicknesses on brass and stainless-steel substrates, fabricated at a constant current density. The resulting composite systems were evaluated using bidirectional bending and cross-hatching adhesion tests. Brass and stainless steel, which differ in microstructure and surface topography, were used as substrate materials. Due to the minor mismatch in crystallographic structure between nickel and brass, strong adhesion was observed on brass substrates. In contrast, nickel coatings exhibited weak adhesion on stainless steel substrates. Reduced film thickness and increased substrate surface roughness enhance the adhesion of nickel coatings, with a particularly notable effect observed on stainless steel substrates. Simulation of the bidirectional bending test revealed the stress distribution within the coatings and identified critical points of maximum stress, which were consistent with the results obtained from the experimental testing.



RESULTS AND DISCUSSION

Topographic Characterization of Substrates and Electrodeposited Ni Coatings

The surface topographies of both substrates and nickel coating were analysed using stereomicroscopy and the Atomic Force Microscopy (AFM) technique to enhance the understanding and interpretation of the results obtained from adhesion testing using dynamic two-directional bending and static cross-hatching methods. Figure 3 presents stereomicroscopic and AFM images of the sandpaper-polished surfaces of brass and stainless-steel substrates.

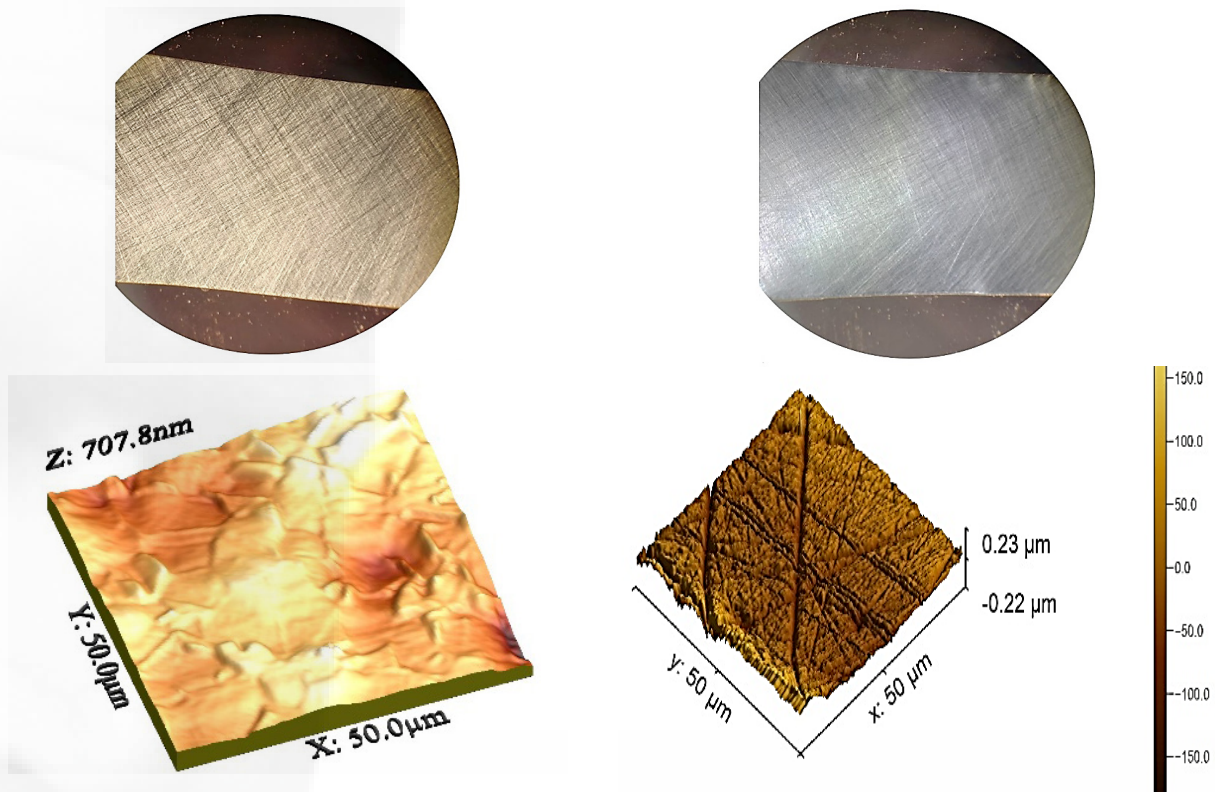


Figure 3: The stereomicroscopic and AFM topography images of the sand-polished and etched brass (left) and the stainless steel (right) substrates

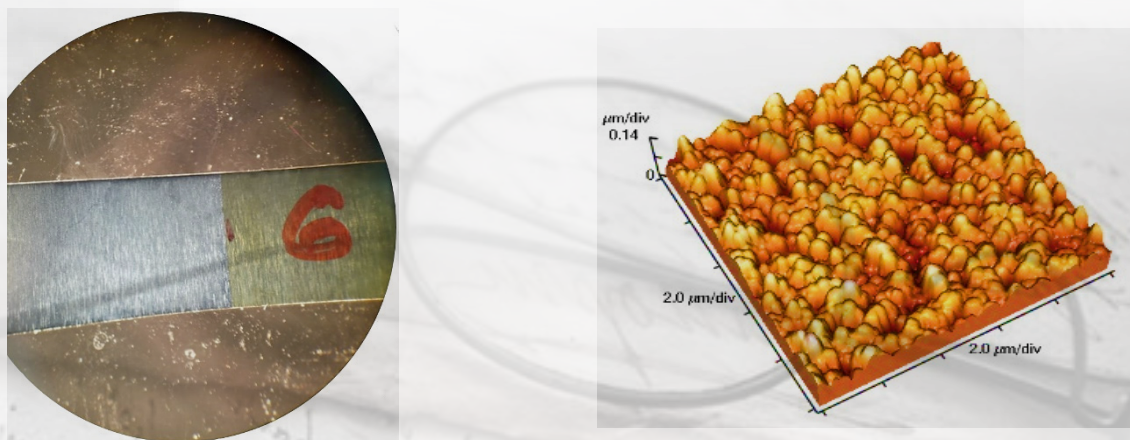


Figure 4: Stereomicroscopic image of the as-electrodeposited nickel coating ($10\mu\text{m}$ thick) on brass substrate and an AFM topography after etching in a grain-boundary-revealing solution.

Figure 4 shows the optical image of the as-electrodeposited nickel coating with a thickness of 10 μm , alongside the AFM image obtained after etching in a grain-revealing solution for 60 seconds.

Adhesion Characterization of Electrochemically Deposited Nickel Coatings on Brass and Stainless-Steel Substrates

The characterization of adhesion quality for nickel coatings on brass and stainless-steel substrates began with drawing a cross-hatch pattern on the coating surface, as shown in Figure 5.

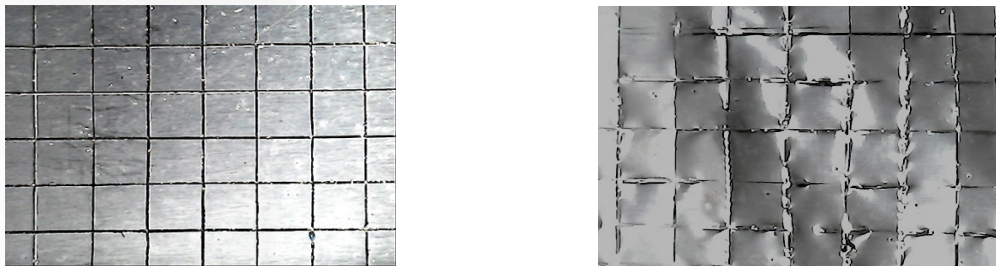
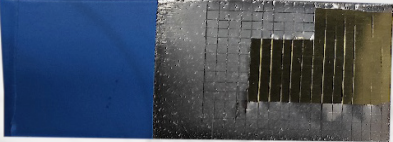
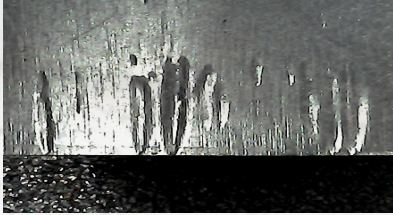
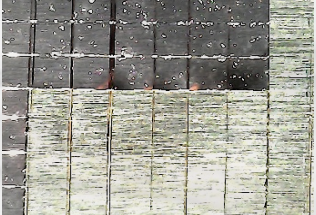


Figure 5: *The process of drawing a cross-hatch on nickel coating on brass substrate (left), and nickel coating on stainless steel substrate (right)*

The result images of the chosen samples of composite systems of nickel coatings on brass and stainless-steel substrates, exhibiting the most characteristic behaviour after the cross-hatching adhesion test (Method B), along with the two-directional bending test, are presented in Table 1 and quantitatively summarized in Table 2.

Table 1: *Representative images of the test results highlighting characteristic features of nickel coatings electrodeposited on brass and stainless-steel substrates.*

	Coating thickness (in μm)	Bending test	Cross-hatching test
Brass substrate	5		No coating delamination.
	10		No coating delamination.
	20		
			

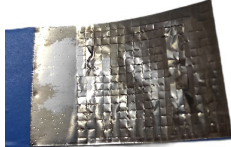
Stainless-steel substrate	5		
			
	10		
			
	20		Complete coating delamination
			

Table 2: The results of the adhesion tests of nickel coatings with different thicknesses electrodeposited on brass and stainless-steel substrates

	Coating thickness (in μm)	The critical cycle number	The value of the cross-hatch test, method B
Brass substrate	5	/	5B
	10	/	5B
	20	47	1B
Stainless-steel sub- strate	5	8	0B
	10	2	0B
	20	2	0B

Simulation and analysis of the two-directional bending testing was performed using the ANSYS 2025 R2 software package, focusing on the behaviour of sheet metal samples coated with a protective film under bending loads. The simulation results are presented as stress distribution contours, with maximum stress indicated in red and minimum stress in blue, as presented in Figure 6.

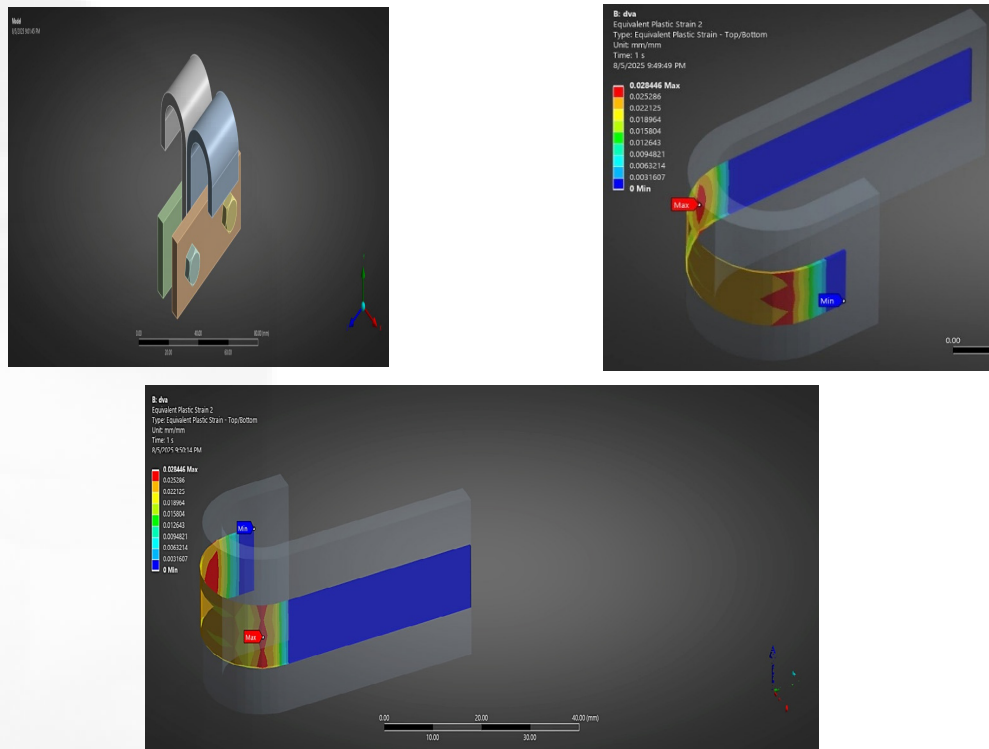


Figure 6: Results of the simulation of the two-directional bending test according to the presented experimental conditions

ANALYSIS OF THE PRESENTED RESULTS

The adhesion of nickel coatings electrochemically deposited on brass and stainless-steel substrates has been analysed. The substrates were prepared using a consistent procedure, which included degreasing and mechanical polishing with sandpaper. As shown in Figure 3, the substrate surfaces exhibited a noticeable degree of roughness, which can contribute to improved coating adhesion. Both substrates belong to alloys whose crystallographic structure depends on their composition. Due to the potential crystallographic similarity (the same type of FCC crystal lattice is expected for the used brass substrate) between nickel and brass, improved adhesion performance of nickel coatings on brass substrates was anticipated. Nickel coatings were deposited from a sulfamate electrolyte solution with the addition of saccharin. This electrolyte composition, combined with applying the higher current densities, enables the formation of fine-grained coatings with reduced internal stresses — factors that are particularly beneficial for achieving high-quality adhesion. Figure 4 shows the fine-grained columnar microstructure of the nickel coating after etching in a grain-boundary-revealing solution.

Adhesion performance testing was performed with cross-hatching and bidirectional bending tests. Visually, the adhesion quality could already be compared while drawing a cross-hatch as part of the cross-hatching test. Figure 5 (image on the right) clearly shows the loosening of the nickel coating adhesion on the stainless-steel substrate during the cross-hatch drawing process.

In addition to the type of substrate, it has been shown that the quality of adhesion of coatings on substrates depends a lot on the coating thickness. Nickel coatings with thicknesses of 5 μm and 10 μm on brass substrates exhibited excellent adhesion during the cross-hatching test. After testing with a special adhesive tape, no signs of delamination were observed, and the coatings were classified as cat-

egory 5B according to the standard. The coating with a greater thickness of 20 μm exhibited reduced adhesion, with partial delamination observed after application of the adhesive tape. Due to the significant portion of the coating surface that became detached, the adhesion performance was classified as category 1B. In the crosshatching adhesion test, the adhesion results of the nickel coatings on stainless steel were poor, regardless of the thickness of the coating, and classified as category 0B. This category indicates that more than 65% of the coating was removed from the substrate and adhered to the adhesive tape (Table 1, 10- μm -thick nickel coating electrodeposited on stainless-steel substrate transferred to the adhesive tape). Similar adhesion quality results were obtained through dynamic bidirectional bending testing, as shown in Table 2. The selected maximum number of bending cycles was 100. Nickel coatings of 5 μm and 10 μm thickness on the brass substrate showed excellent adhesion quality, where no signs of delamination were observed after 100 bending cycles. The thicker nickel coating (20 μm) on the brass substrate yielded after 47 cycles, as shown in Tables 1 and 2. The critical number of bending cycles for nickel coatings on stainless steel substrate was extremely low. This test also confirmed the assumption that the adhesion quality will depend on the thickness of the nickel coating. For a coating with a thickness of 5 μm , the critical number was 8 cycles. For coatings with a thickness of 10 μm and 20 μm , the critical cycle number was only 2. The experimental findings demonstrated a strong correlation with the simulation results. Based on the simulated stress distribution, it was possible to identify critical points where worsening of the nickel coating's adhesion was most likely to occur. These predicted failure points aligned well with the areas where delamination was observed during experimental testing, thereby validating the accuracy of the numerical model.

ORIGINALITY/VALUE

Identifying and evaluating the factors that influence the adhesion strength of coatings on substrates is crucial, as the structural integrity of components, devices, or entire constructions often depends on the adhesion quality. Material system failures due to inadequate adhesion are a common cause of structural or functional breakdowns, making adhesion a critical consideration in forensic engineering analyses. Each composite system is unique and must be analysed individually because many variable parameters are involved. The results presented in this paper provide valuable insights into predicting the factors that influence the behaviour of composite material systems and their integrity. Combining adhesion test simulations with experimental results allows for a reduction in both time and material resources - an important advantage, given that most adhesion testing methods are inherently destructive.

CONCLUSION

This study evaluated the adhesion performance of electrochemically deposited nickel coatings on brass and stainless-steel substrates through experimental mechanical testing and finite element simulation. Nickel coatings were deposited from a sulphamate-based electrolyte with saccharin as a stress-reducing agent. Key parameters such as coating thickness, substrate surface roughness, and failure behaviour under mechanical stress were examined to establish correlations and assess the reliability of the applied testing methods. Two destructive testing methods, bidirectional bending and cross-hatching, were employed, alongside non-destructive numerical modelling to analyse stress distribution and predict failure initiation zones.

The results demonstrated that the substrate material's microstructure, surface roughness parameters, and coating thickness significantly influence the adhesion quality of the nickel coatings.



Based on the results obtained, we can highlight the following:

- Nickel coatings on brass substrates exhibited excellent adhesion for both tests, particularly at lower thicknesses (5 μm and 10 μm), with no delamination observed even under extended mechanical stress.
- Increasing the nickel coating thickness on the brass substrate to 20 μm demonstrated lower adhesion performance, suggesting that increased coating thickness can negatively influence interfacial bonding integrity.
- In contrast, nickel coatings on stainless steel substrates showed poor adhesion performance across all thicknesses, with early onset of delamination during both tests. These disparities are primarily attributed to differences in microstructural compatibility and substrate surface roughness.
- Adhesion bending test simulations closely matched the experimental results and identified critical stress regions associated with coating failure.

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