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# CHARACTERIZATION OF ARC-PVD NICKEL COATINGS USED FOR VACUUM BRAZING OF JOINTS IN ACCELERATING STRUCTURES

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In this research, the structure and properties of multilayered nickel coatings obtained by vacuum-arc deposition (Arc-PVD) have been investigated. The influence of Ni layered coatings after being brazed using silver-based brazing solder on vacuum joints has been determined. The Ni coatings are usually applied to improve adhesion between accelerating structures' components made of various materials and to ensure the vacuum tightness of the entire structure. The Ni layers with a thickness of 4.7 μm and a maximum roughness of $R_{max} \approx 3.29$ μm were deposited on substrates made of ceramic, steel, and copper. The contact angle (θ) examinatios have demonstrated a significant effect of the substrate type on surface properties. The contact angle values ranged from 59 to 78°, with the best wettability observed for steel substrates comprised 59°. The SEM analysis confirmed a homogeneous layer morphology formation with oxygen content not exceeding 3.87%. Vacuum tightness tests of brazed joints revealed that the applied combination of thickness and surface roughness of the Ni layer meets the operational requirements for accelerating structures.

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## INTRODUCTION

The accelerating structures designed and manufactured at the National Centre for Nuclear Research (NCBJ) are complex assemblies composed of several dozen precisely machined resonant cavities, operating with a standing electromagnetic (EM) wave at a frequency of 3 GHz in the π/2 mode [1]. The resonant cavities are produced with an accuracy up to 0.01 mm from oxygen-free high conductivity copper (OFHC), which exhibits very high electrical and thermal conductivity [2]. The accelerating structures are being processed under high vacuum conditions [3], which reduces electron beam scattering on gas particles and ensures effective particle acceleration. Fully accelerated electrons can be used directly as an ionizing radiation beam or directed onto a conversion target. Upon rapid deceleration in the target material, electrons emit their energy in the form of photons, generating bremsstrahlung X-rays. Such an ionizing radiation of photon beam has higher penetration capability compared to the electron beam. However both electron and photon beams find applications in radiotherapy, non-destructive testing, and border cargo inspection.

Modern designs of ionizing radiation accelerator structures impose stringent requirements on materials and joining technologies, both in terms of mechanical properties and vacuum tightness. One of the key steps in the manufacturing process of such structures is brazing the components using silver-based hard solders. To ensure high-quality joints, surfaces intended for brazing are coated with a layer of nickel. These coatings not only improve solder adhesion but also play a crucial role in maintaining the structural integrity under high vacuum conditions and varying operating temperatures.

The aim of this research was to develop optimal technological parameters for nickel layer deposition and to analyze their futher impact on the brazing process with silver-based solder under vacuum conditions. Particular emphasis was placed on investigating wetting properties, durability of brazed joints, and the significance of these factors in the context of high-vacuum applications.

In the next stage of research, it is planned to extend the analysis to brazed joints involving ceramic substrates, which impose additional challenges due to joining materials with different physical-chemical properties.

## 1. EXPERIMENTAL SETUP

Nickel coatings were deposited using vacuum-arc deposition technique (Arc-PVD) [4, 5]. The facility and experimental setup for synthesis are shown in Fig. 1. The cathode was made of a pure nickel rod (at purity 99.9%) with a diameter of 50 mm. Copper, steel, and ceramic substrates were used to provide coatings on elements with different compositions. The substrates were mounted in a holder approximately at a distance of 150 mm from the cathode. The deposition process was conducted at a base pressure reaching $3.3 \cdot 10^{-8}$ mbar. The cathode current was maintained at 72 A, and the arc voltage was kept at 72 V. The samples were biased at a voltage ($I_{bias}$) of 250 mA. Nickel deposition was performed cyclically – each cycle lasted 30 s, followed by a 5-min technological pause. A total of 12 cycles were completed, resulting in a layered thickness of approximately 4.7 μm. This deposition sequence was designed to minimize thermal effects.

The surface morphology and cross-section of the deposited Ni layers were examined using a Zeiss EVO® MA10 scanning electron microscope (SEM) operated at an accelerating voltage of 10 kV. Chemical composition was analyzed by EDX (elemental mapping and line scan).

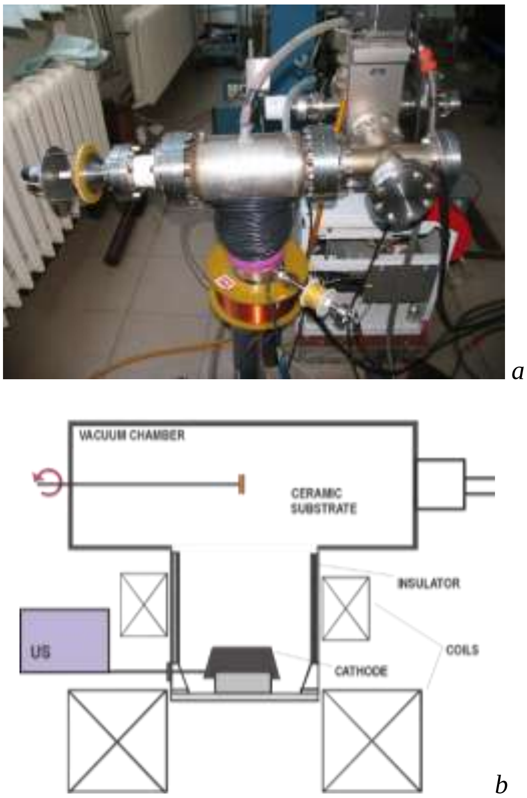



*Fig. 1. Design (a) and schematic (b) of the device for nickel layer synthesis*

Surface roughness was measured using a Keyence VK-X3000 series laser scanning microscope.

Water contact angles on the Ni layers were determined using the sessile drop method. Samples were placed on a rotating stage to ensure that the sample surface was always aligned with the optical axis of the digital camera. A 0.1 ml water droplet was gently deposited on the layer surface using a micro-syringe. The contact angle (θ) was calculated as the average of six independent measurements based on analysis of the recorded images for each layer.

## 2. RESULTS AND DISCUSSION

SEM and EDS investigations revealed that the nickel coating exhibits a typical surface morphology for Arc-PVD process [6–9]. The coating is characterized by a microstructure with a uniform distribution of elements wirhout microcracs and various microparticles of various size (Fig. 2). The recorded oxygen content in the layer did not exceed 3.87 at.%, suggesting limited surface oxidation and good coating purity. The presence of oxygen at this level may result from natural surface passivation upon exposure to air after the deposition process. Such an oxidation level does not negatively affect the brazing properties of the layer and is acceptable for applications in accelerating structures.

One of the key factors is the appropriate selection of layer thickness. Both an excessively thin and an excessively thick Ni layer can negatively affect joint strength. A layer that is too thin does not act as an effective diffusion barrier [10], while one that is too thick may lead to stresses and cracking. Based on multiple experiments, it was determined that the proposed parameters of the Arc-PVD deposition process make it possible to obtain a Ni layer with a thickness of 4.7 µm. The research showed that such a layer is optimal for use in vacuum brazing of components for accelerating structures.

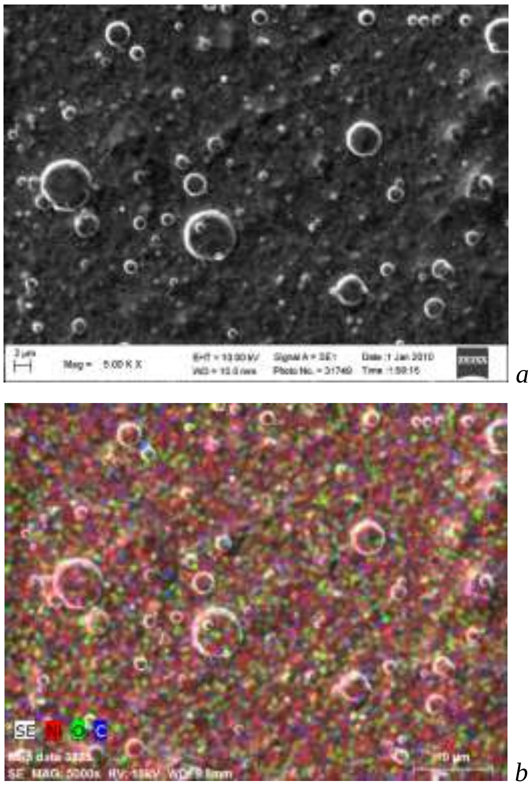


*Fig. 2. SEM surface morphology (a) and EDS maping (b) of Ni layer*

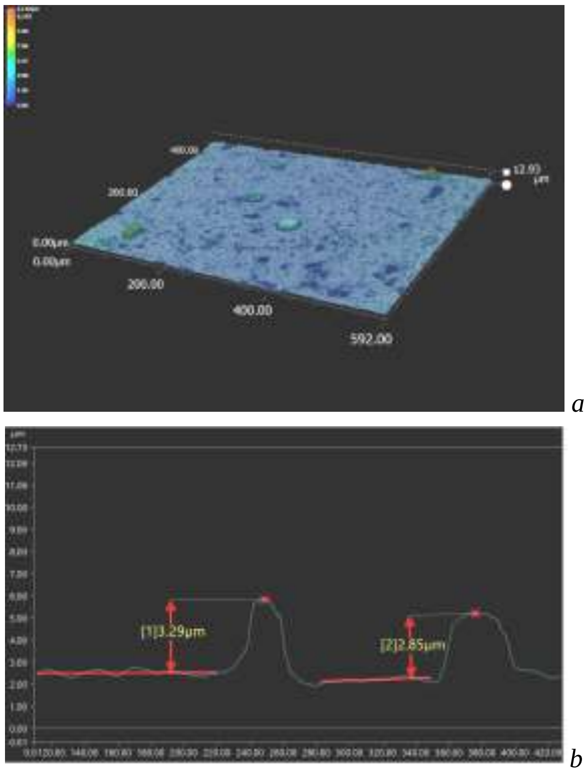
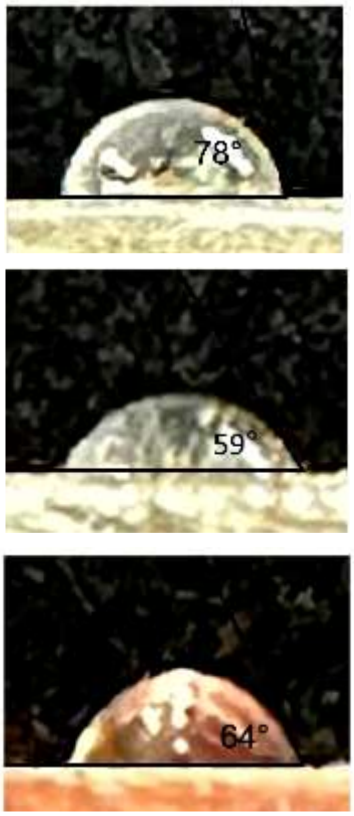



*Fig. 3. 3D morphology (a) and surface roughness (b) of the Ni coating*

Surface topography analysis showed that these coatings are characterized by a maximum roughness of $R_{max} \approx 3.29$ μm (Fig. 3). Such a surface profile positively affects solder droplet wettability and increases the effective contact area, which promotes the formation of strong joints.

Measurements of the water droplet contact angle on Ni layers deposited by the Arc-PVD method revealed significant differences depending on the type of substrate (Fig. 4). The contact angle ranged from 59 to 78°. Better wettability parameters were recorded for Ni on steel (59°), which suggests higher surface energy and more favorable initial conditions for brazing. The highest contact angle was observed for the Ni layer on ceramic (78°). This phenomenon can be explained by differences in the micro-roughness inherited from the substrate, local oxidation of nickel, and the influence of the base material type on the microstructure and adhesion of the deposited layer.

In the studies conducted on Ni layers, a characteristic spreading of silver-based solder on the surface was observed, consistent with our predictions (Fig. 5). This confirms the beneficial effect of moderate roughness on wettability and the quality of brazed joints.



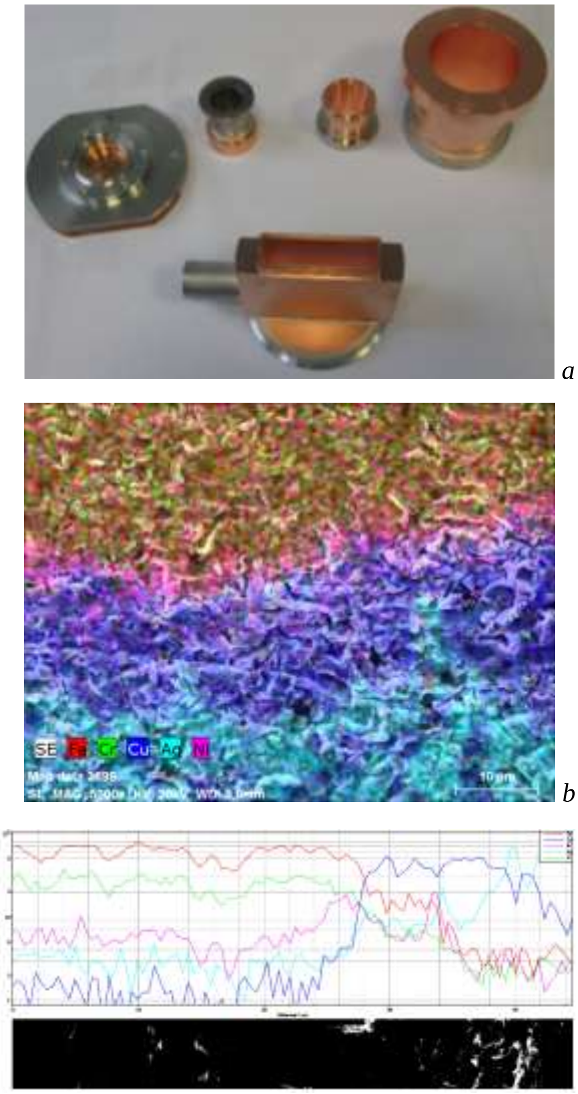

*a*

*b*

*c*

*Fig. 4. Contact angle on different types of substrates coated with nickel: a – ceramic; b – steel; c – copper*

Investigations of the joint regions demonstrated that the selected combination of nickel layer thickness and surface roughness provides optimal vacuum tightness of the brazed joints, thereby fulfilling the operational requirements of accelerating structures. The absence of microcracks, good adhesion, and uniform solder distribution confirm the effectiveness of the proposed deposition parameters for high-vacuum applications.




*a*

*b*

*c*

*Fig. 5. Brazed components of accelerating structures (a), cross-section of the brazed joint with EDS mapping (b), and elemental concentration variation (c)*

In the next stages, studies are planned to apply the developed Ni layer deposition technology to brazed joints between ceramic and metallic components, which will allow for assessing its suitability for a broader range of accelerating structures' components.

## CONCLUSIONS

The developed Arc-PVD deposition parameters allow to obtaine Ni coatings with a thickness of 4.7 μm and a maximum roughness of $R_{max} \approx 3.29$ μm. The coatings are characterized by this maximum roughness value. Measurements of the water droplet contact angle on Ni layers deposited by the Arc-PVD method revealed significant differences depending on the substrate type, ranging from 59 (steel) to 78° (ceramic). The best wettability was observed for coatings on steel, which favors the formation of high-quality brazed joints. Experimental studies of the joined components showed that the applied combination of Ni layer thickness and roughness ensures sufficient vacuum tightness of the brazed joints.

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## ХАРАКТЕРИЗАЦІЯ НІКЕЛЕВИХ ПОКРИТТІВ, НАНЕСЕНИХ ВАКУУМНО-ДУГОВИМ МЕТОДОМ, ЩО ВИКОРИСТОВУЮТЬСЯ ДЛЯ ВАКУУМНОГО ПАЯННЯ З'ЄДНАНЬ У КОНСТРУКЦІЯХ ПРИСКОРЮВАЧІВ

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Представлено результати дослідження структури та властивостей багатошарових нікелевих покриттів, отриманих методом вакуумно-дугового осадження (Arc-PVD). Визначено вплив шаруватих нікелевих покриттів на вакуумні з'єднання, паяні припоєм на основі срібла. Нікелеві покриття зазвичай наносяться для покращення адгезії між компонентами прискорювача, виготовленими з різних матеріалів, та для забезпечення вакуумної герметичності всієї конструкції. Шари нікелю товщиною 4,7 мкм та максимальною шорсткістю $R_{max} \approx 3{,}29$ мкм були нанесені на підкладки з кераміки, сталі та міді. Дослідження кута змочування (θ) продемонстрували значний вплив типу підкладки на властивості поверхні. Значення кута змочування коливалися від 59 до 78°, причому найкраща змочуваність, що спостерігалася для сталевих підкладок, становила 59°. СЕМ-аналіз підтвердив формування однорідної морфології шару з вмістом кисню, що не перевищує 3,87%. Випробування на вакуумну герметичність паяних з'єднань показали, що застосоване поєднання товщини та шорсткості поверхні шарів нікелю відповідає експлуатаційним вимогам до конструкцій прискорювачів.