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INFLUENCE OF PRETREATMENT METHODS ON THE FORMATION OF THIN-FILM NICKEL ELECTRODES FOR CAPACITIVE SENSORS

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Annotation. The paper investigates the influence of pretreatment methods for sitall ST-50-1-1-0,6 substrates surface on the subsequent formation of thin-film nickel electrodes for capacitive sensors for heavy metal ions detection in water. A comparison of two cleaning methods was carried out: ion-beam cleaning in vacuum and treatment in a dielectric barrier discharge plasma torch at atmospheric pressure. Optimal cleaning regimes ensuring maximum adhesion and quality of the formed films were established. Nickel films with a thickness of 100 nm were obtained by ion-beam sputtering; their optical properties and surface morphology were studied.

Keywords: thin films, nickel, sitall, ion-beam sputtering, atmospheric pressure plasma, surface cleaning, capacitive sensors.

Introduction. Rapid population growth and industrial development lead to pollution of water resources with heavy metals (Hg^{2+}, Pb^{2+}, Cu^{2+}, etc.), which are characterized by persistence and bioaccumulation ability. Existing analytical methods (liquid chromatography, atomic absorption) have high sensitivity but require complex sample preparation and expensive equipment. An alternative is electrochemical sensors that provide rapid real-time analysis [1].

The quality of thin-film electrodes for such sensors critically depends on the substrate surface condition before deposition. The presence of contaminants and oxides degrades adhesion and stability of characteristics. Surface cleaning in intense ion beams allows obtaining an atomically clean surface, removing both physical and chemical contaminants, and simultaneously activates the surface, improving adhesion of the deposited coating [2].

The aim of this work is to study the influence of pretreatment methods (ion-beam in vacuum and atmospheric pressure plasma) on the properties of sitall substrates and the subsequent formation of thin-film nickel electrodes by ion-beam sputtering.

Experimental Methods. Sitall ST-50-1-1-0,6 was used as substrate material, and nickel as electrode material. The choice is justified by the proximity of thermal expansion coefficients (TEC of nickel $1.5\cdot10^{-6}$ 1/°C, TEC of sitall $0.5–3\cdot10^{-6}$ 1/°C) and high chemical resistance of sitall, as well as the optimal combination of corrosion resistance, resistivity, and cost of nickel [3].

Substrate pretreatment was carried out by two methods:

Ion-beam cleaning in vacuum – in a modified VU-1BSp setup with an ion source based on an anode layer accelerator. Cleaning regime: argon flow rate – 1.24 l/h, voltage – 2220 V, current – 32 A, power – 50 W, distance to substrate – 200 mm, treatment time varied from 15 to 960 s.

Atmospheric pressure plasma cleaning – in a dielectric barrier discharge plasma generation system of coaxial type. Cleaning regime: argon flow rate – 142 l/h, distance to substrate – 10 mm, treatment time – 15–120 s.

Formation of nickel thin films was carried out by ion-beam sputtering in the VU-1BSp setup. Deposition regime: preliminary pressure in the chamber – $4\cdot10^{-3}$ Pa, working pressure – $2\cdot10^{-2}$ Pa, argon flow rate – 0.34 l/h, source voltage – 3240 V, discharge current – 42–52 mA, distance from target to substrate – 200 mm, deposition time – 60 min.

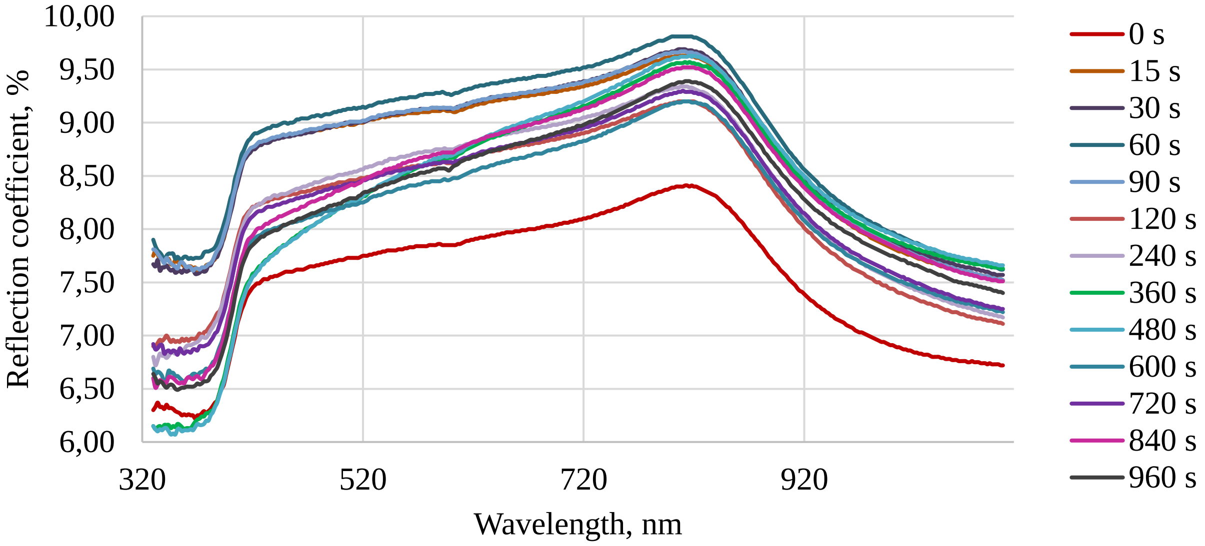
The following methods and equipment were used for research:

– spectrophotometer MS-122 (measurement of reflection coefficient in the range 330–1100 nm);

– atomic force microscope NT-206 (surface morphology);

– sessile drop method using a Lenpipet LAIT dispenser (contact angle, liquid – distilled water).

Effect of Cleaning on the Reflection Coefficient of Sitall. Figure 1 shows the dependences of the reflection coefficient of sitall substrates on wavelength for different treatment times with ion source and atmospheric pressure plasma.



a

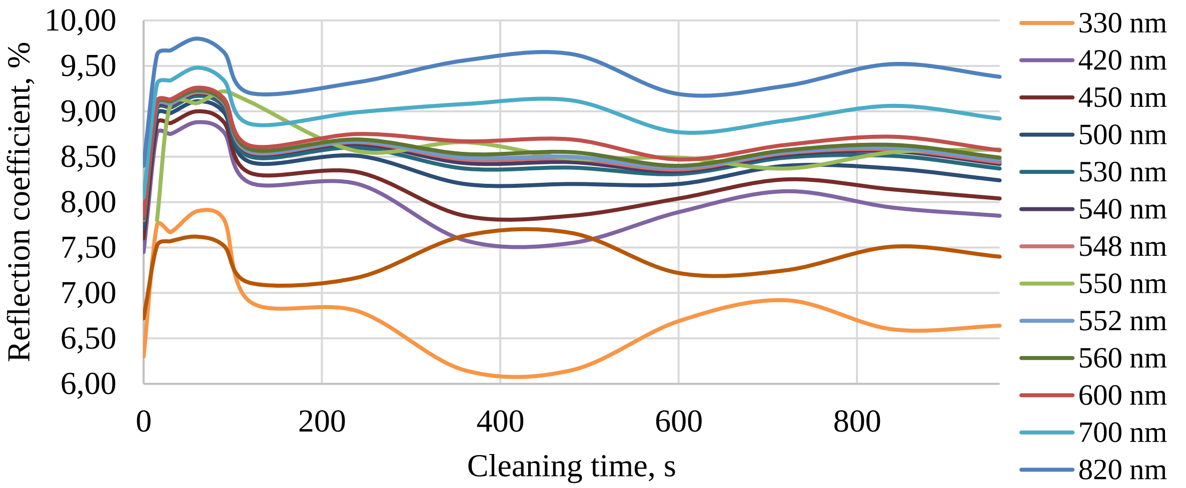


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Figure 1 – Dependence of reflection coefficient on wavelength at different cleaning times: a) ion source; b) atmospheric plasma

Analysis of the spectra shows that the initial surface (0 s) has low reflectivity (6.2–8.4 %), which is associated with the presence of contaminants and oxide films. Dusring ion-beam cleaning, the most significant change is observed in the first 60 seconds: the reflection coefficient at a wavelength of 820 nm increases from 8.4 % to 9.8 %. Further treatment (up to 960 s) leads to a slight decrease in reflectivity, indicating saturation and possible onset of etching of the glazed sitall layer.

During atmospheric pressure plasma cleaning, an increase in the reflection coefficient is also observed, with maximum values achieved at 120 s of treatment (from 7.11 % to 9.02 %). However, unlike ion cleaning, plasma treatment does not cause significant changes in surface morphology (confirmed by AFM data, not shown).

Effect of Cleaning on Surface Wettability. To assess adhesive properties, the contact angle was measured using the sessile drop method. The results are presented in Table 1 and Figure 2.

Table 1 – Dependence of contact angle on cleaning time

Cleaning time, s	Contact angle (ion source), °	Contact angle (atmospheric plasma), °
0	69,52	69,52
15	24,05	9,16
30	18,69	7,27
60	11,91	6,6
90	13,72	5,75
120	14,23	4,48

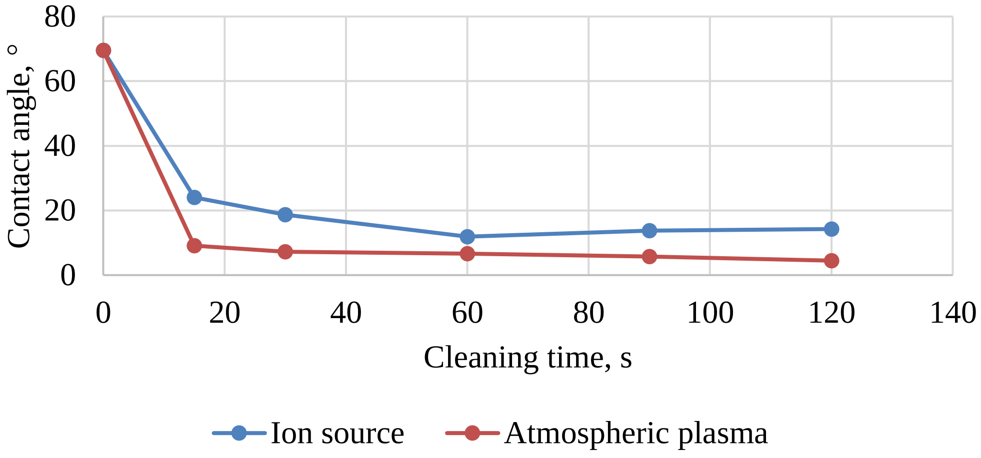


Figure 2 – Dependence of contact angle on cleaning time

The initial contact angle is 69.52°, corresponding to moderate wetting. During ion-beam cleaning, the most dramatic decrease occurs in the first 15 s (to 24.05°), with the minimum value reached at 60 s (11.91°). Further treatment leads to a slight increase in the angle, associated with the onset of surface etching.

Treatment in atmospheric pressure plasma demonstrates more effective reduction of the contact angle: already at 15 s the value drops to 9.16°, and at 120 s reaches 4.48°, indicating almost complete wetting and, consequently, high adhesive ability of the surface. It is important to note that plasma treatment does not cause etching of the glazed layer, confirmed by the preservation of morphology.

Formation and Study of Nickel Thin Films. Deposition of nickel films was carried out on substrates that underwent optimal pretreatment.

Figure 3 shows the reflection spectra of nickel films formed on substrates with different pretreatment (ion source cleaning for 60 s and atmospheric plasma cleaning for 120 s).

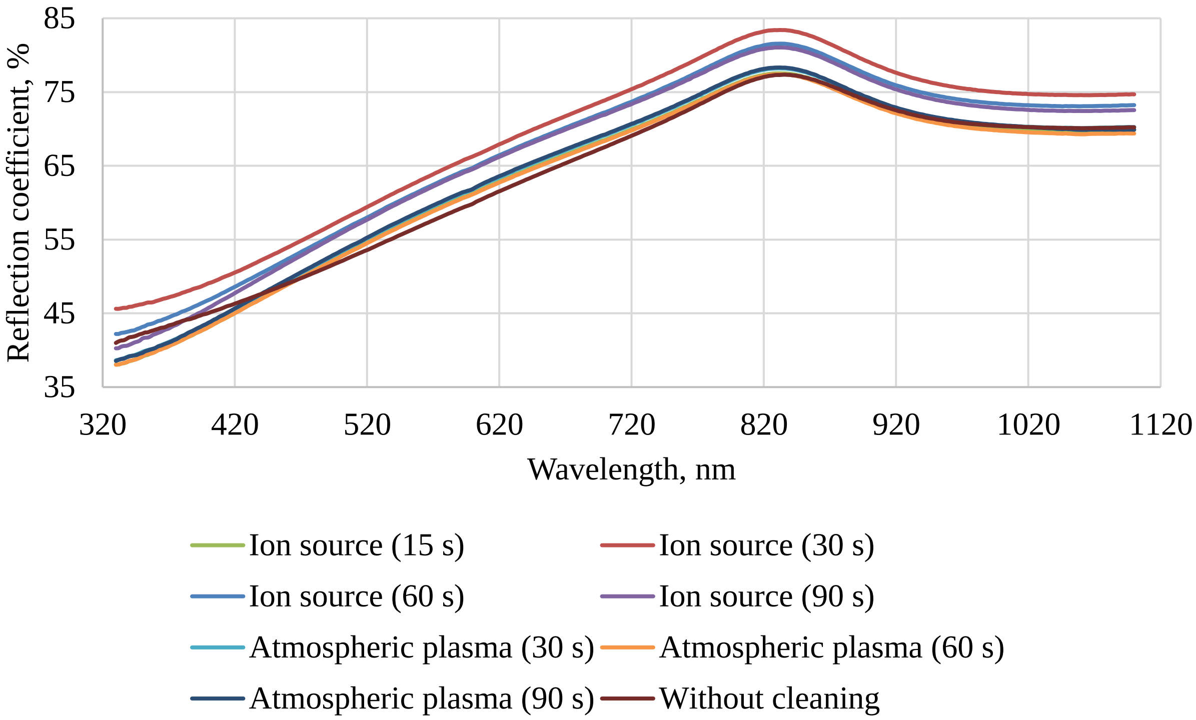
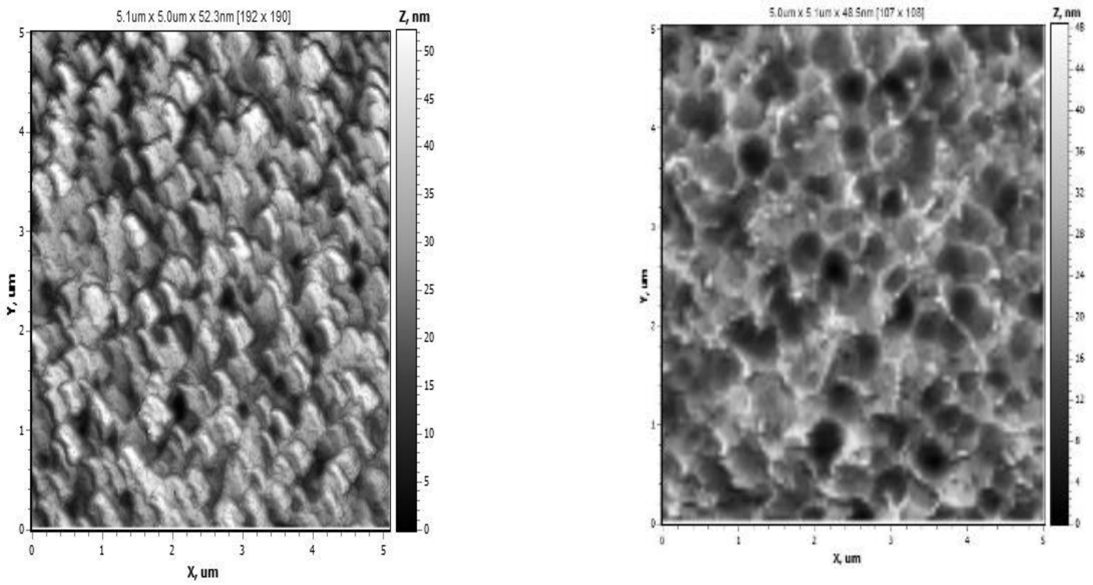


Figure 3 – Dependence of the reflection coefficient of nickel films on wavelength for different substrate cleaning methods

It can be seen that pretreatment in atmospheric pressure plasma provides a higher reflection coefficient of the formed films compared to ion cleaning, which correlates with the wettability data and indicates better adhesion and a more perfect film structure.

Surface morphology study using an atomic force microscope (Figure 4) showed that the nickel film has a uniform granular structure with individual grain sizes up to 450×800 nm. The surface is homogeneous, without visible defects.



a b

Figure 4 – AFM images of the surface: a) initial sitall; b) nickel film

Optimal Regimes. Based on the conducted research, the following optimal regimes were established:

1 Pretreatment: treatment in atmospheric pressure plasma (dielectric barrier discharge) for 120 s at an argon flow rate of 142 l/h, distance to substrate 10 mm. This regime provides a contact angle of 4.48° and does not cause surface etching.

2 Deposition of nickel films: ion-beam sputtering at a pressure of $2 \cdot 10^{-2}$ Pa, voltage 3240 V, current 42–52 mA, distance to target 200 mm for 60 min, which allows obtaining films of 100 nm thickness with uniform structure and high optical characteristics.

Conclusion. A comparative study of two methods for pretreatment of sitall substrates before the formation of thin-film nickel electrodes was carried out. It was established that treatment in dielectric barrier discharge plasma at atmospheric pressure provides better adhesive properties (contact angle 4.48°) compared to ion-beam cleaning in vacuum, while not causing etching of the glazed substrate layer. Nickel films of 100 nm thickness were obtained by ion-beam sputtering, and their optical properties and morphology were studied. The developed regimes can be used for the formation of electrodes for capacitive sensors for heavy metal content in water, confirming the relevance and practical significance of the work.

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