

Experimental study of the interaction of a jet high-frequency inductive discharge of low pressure with a copper surface

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The article presents the results of a study of the interaction of a low-pressure jet high-frequency inductive discharge (HFID) with a liquid plasma-forming medium on the surface of an M1 copper plate. The discharge was generated in a quartz tube with a spiral inductor. The electrophysical characteristics, as well as the types and shapes of plasma structures formed during material processing, were studied. The surface morphology of the samples was examined before and after processing, and contact angle measurements were conducted to assess changes in the surface energy of the copper. Thermographic measurements allowed us to determine the temperature distribution in the HFID discharge combustion zone. The obtained results demonstrated the potential of the jet HFID discharge for modifying the copper surface and open up prospects for its application in hardening and protecting metallic materials.

Keywords: high-frequency inductive discharge, plasma-liquid systems, discharge combustion, copper.

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Introduction

A low-temperature plasma is one of the most versatile tools of modern science and engineering. Its use covers metallurgy, mechanical engineering, additive technologies, microelectronics, medicine, environmental processes and power engineering [1,2]. The recent decades have seen formation of some fields that are related to interaction of the plasma and liquid, which are referred to plasma-liquid systems. The liquid medium in them can perform several functions: to act as a plasma-forming phase, an electrode or a coolant. This combination paves a unique way for generating chemically active radicals, transfer of charged particles and modification of properties of a material surface [3–8].

The most detailed are direct current discharges and pulse discharges in the liquid electrodes. These systems demonstrated efficiency in cleaning, disinfection, activation and strengthening of a metal surface [9–15]. However, a remaining significant limitation is erosion of the solid electrodes and ingress of their destructibles into the plasma, thereby reducing reproducibility and controllability of the processes.

In this regard, researchers pay more and more attention to high-frequency inductive (HFI) discharges excited by a variable electromagnetic field. The HFI discharge does not require direct contact of the electrodes with the plasma, since it provides non-contact energy input into the system and excludes electrode wear. The key advantages of the HFI discharges lie in high stability, operability within a

wide pressure range and medium compositions as well as in formation of a volume plasma with high concentration of charged particles [16].

Special interest is paid to jet configuration of the lower-pressure HFI discharges, when the liquid is supplied as a thin jet into a working chamber. This geometry has a number of unique properties:

- formation of a directed plasma flow,
- localizability of a heat impact area,
- adjustment of plasma parameters by jet hydrodynamics (its speed, flowrate, diameter),
- simplification of control of the plasma composition by varying the electrolyte composition.

Despite the fact that the classic volume HFI discharges in gases are quite detailed [16], there is almost no systematic study of jet HFI discharges with the liquid plasma-forming medium. Only separate publications of the recent years [17–19] address issues of setup experiments and demonstrate that it is possible to excite the discharge in flow-through configurations. At the same time, it is obvious that mechanisms of plasma formation, its stability and influence on metal materials in these conditions are understudied.

Scientifically, the jet HFI discharge is interesting as an example of a non-equilibrium plasma under lower-pressure conditions, which simultaneously has electrophysical, hydrodynamic and thermal-physical processes. This case includes a unique combination of factors: high frequency of an electromagnetic field, low pressure, narrow jet geometry

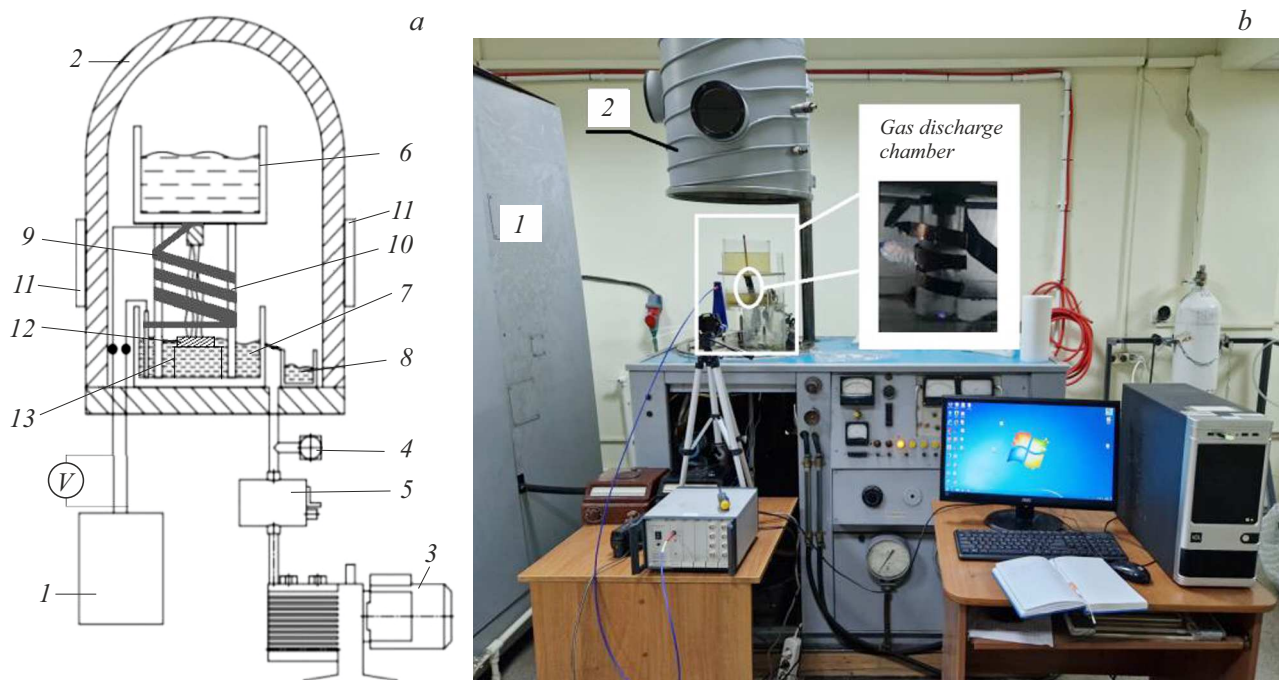


Figure 1. Functional diagram (a) and a photo (b) of the experimental unit for ignition of the lower-pressure jet HFI discharge with the liquid plasma-forming medium: 1 — the high-frequency power supply; 2 — the vacuum chamber; 3 — the sliding-vane pump 2NVR-5DM; 4 — the vacuum gauge; 5 — the pressure adjustment valve; 6 — the electrolytic cell of electrolyte jet supply; 7 — the main electrolytic cell for drain; 8 — the additional electrolytic cell for drain; 9 — the quartz chamber, whose surface had a 7 mm-wide copper tape screwed on, which coincides with an external diameter of the quartz tube and forms a three-coil solenoid; 10 — the spiral inductor, 11 — the sight glasses; 12 — the treated part; 13 — the support for the treated part.

and presence of the liquid plasma-forming medium. This combination forms a rich physical-chemical pattern, in which interaction of the plasma with the surface can substantially differ from known modes of arc or glow discharges.

Practically, relevance of the study is determined by a need for new method of treating copper and its alloys. Copper (the M1 grade) is widely used in electrical engineering, power engineering, mechanical engineering due to high thermal conductivity, electrical conductance and plasticity. But copper is susceptible to oxidation and corrosion, its surface is subject to wear and mechanical properties limit durability of products. At the same time, using the jet HFI discharge makes it possible to locally affect the surface without excessive superheating, which is especially important for copper with its high thermal conductivity.

Studying of interaction of the lower-pressure jet HFI discharge with the liquid plasma-forming medium and the copper surface is an urgent problem at a junction of fundamental plasma physics and applied materials science. It will make it possible:

- to extend representations about the mechanisms of formation of the jet plasma structures,
- to study influence of the discharge on the morphology and the physical-chemical properties of copper,
- to estimate changes of wettability after treatment. The present study is aimed at comprehensive experimental

investigation of the lower-pressure jet HFI discharge with the liquid plasma-forming medium and its impact on the surface of the M1 grade copper samples, including analysis of the electrophysical parameters of the discharge, the surface morphology and a contact angle.

1. Experimental unit

In order to ignite and sustain the lower-pressure HFI discharge, we have created an experimental unit with the working chamber, whose schematic diagram is shown in Fig. 1.

The power source was a high-frequency oscillator UGPN-1. It was powered by a three-phase network of 380 V voltage, 50 Hz (an allowable deviation was $\pm 5\%$). Nominal oscillatory power was 5 kW, while output power at the operating frequency of 1.76 MHz was at least 4 kW. Oscillator efficiency is at least 50%, full efficiency of the unit was about 30%. Anode voltage was adjusted within 8–9.5 kV at the current 0.2–1.5 A; a grid current did not exceed 1.5 A. The oscillator was cooled using a flow-through water system (the flowrate is at least $0.2 \text{ m}^3/\text{h}$ at pressure $(1.5 \pm 0.2) \text{ kgf/cm}^2$).

The quartz tube had the 0.2 mm-wide copper tape of the 7 mm width screwed on, which coincides with the external diameter of the quartz tube and forms the three-

coil solenoid. End of the three-coil solenoid were connected to the HF oscillator.

Reproducibility of the parameters of the HFI discharge was provided using a closed electrolyte circulation system with a thermostatic bath. The solution temperature was maintained by means of a thermostat and it was cooled using a refrigeration cooler, thereby making it possible to stabilize a thermal mode of the experiment. The medium composition was renewed by supplying and pumping out the solution; a circuit additionally includes a coarse filter.

The range of the operating pressures was provided by the pump 2MVR-5DM that had a limit pressure of $6.7 \cdot 10^{-2}$ Pa and air performance of 5 L/s. Residual pressure was maintained by the vacuum gauge and adjusted by a metering valve, thereby providing stability of the parameters during the experiment.

Pressure was measured by means of the vacuum gauge of the OBV1-100 grade.

A set of modern diagnostics methods was used for analysis:

1. High-rate video recording. Plasma formation dynamics and development of plasma structures was recorded by a camera Casio EX-F1 with a rate of 600–1200 fps. This range made it possible to register fast non-stationary processes, including fluctuations of microdischarges and motion of glowing structures along the jet. Obtained frame sequences were processed in the software packages HX Link and Movavi Video Editor 14 Plus, thereby enabling to construct frame-by-frame trajectories and qualitative analysis of a fluctuation frequency. Additionally, a morphology of plasma formation on the surface of copper was studied using an optical microscope SP-52 designed to estimate local areas of interaction of the plasma with the solid body.

2. Electric measurements. Voltage was measured by an electrostatic voltmeter S95, which was connected in series. Current was measured by a Rogowski coil.

3. In order to estimate thermal impact on elements of the unit and the studied samples, Infrared Thermography was used. The measurements were made by a thermal imager Testo 875 with a radiation coefficient $\varepsilon = 0.95$ (it is a typical value for quartz) at the reflected temperature of 20 °C. Thermographic data made it possible to plot charts of temperature distribution on the external surface of the media.

4. Microstructure studies. The morphology of the copper surface was analyzed before and after treatment by Confocal Laser Scanning Microscopy (Olympus LEXT OLS4100). This method provided obtaining three-dimensional images of the surface with high spatial resolution, measurement of roughness parameters (average roughness R_a and a — is a maximum height of irregularities R_z) and identification of typical traces of plasma impact, including micromelts, defects and discharge channels. Comparative analysis made it possible to specify correlation between discharge conditions and surface microrelief variation.

5. Wettability. Changes in surface energy of copper were estimated by determining a static contact angle by

Parameters of the jet HFI discharge during interaction with the copper sample

№	Parameter	Value
1	Jet length l_c , mm	35
2	Jet diameter d_c , mm	1.5
3	Jet speed v_c , m/s	0.89
4	Electrolyte flowrate Q , mL/s	1.57
5	Pressure P , Pa	10 000
6	Anode current I_a , A	0.25
7	Grid current I_c , A	0.4–0.6
8	Discharge voltage U_p , kV	0.56–0.83
9	Discharge current I_p , A	0.8–1.35

the sessile drop technique. Hysteresis was not measured. This parameter is a sensitive indicator of modification of a chemical state of the surface and presence of oxide films. The contact angle is considered as an indicator of variation of surface energy and wettability. Reduction of the angle is interpreted as an increase of hydrophilicity and potential cohesion of polar liquid systems (glues, lacquers, fluxes) with the copper surface [20,21], while the increase thereof is interpreted as hydrophobization. Adhesion herein means cohesion of external liquid systems with the copper surface, rather than strength of the oxide film to copper.

The parameters of the jet HFI discharge with the liquid plasma-forming medium during jet treatment of the surface of the M1 grade copper plate (the length is 10 mm, the width is 10 mm) are given in Table.

The electrolyte jet length was 35 mm with the 1.5 mm diameter. The jet flowrate $v_c = 0.78$ m/s and its flowrate $Q = 1.5$ mL/s provided stable formation of the liquid jet with the HFI discharge. The pressure in the working chamber was 10 000 Pa. The electric parameters: the anode current $I_a = 0.25$ A, the grid current $I_c = 0.4–0.6$ A, voltage $U_p = 0.56–0.83$ kV, the discharge current $I_p = 0.8–1.35$ A. A plasma-forming solution was a 3% solution of $(\text{NH}_4)_2\text{SO}_4$ in purified technical water. The inductor was made as a spiral copper ring set around the quartz tube of the 22.3 mm diameter.

2. Results and their discussion

It is found in the performed experiments that combustion of the lower-pressure jet HFI discharge with the liquid plasma-forming medium at the 10 000 Pa pressure is of a filamentous-jet nature and can be implemented in several modes that depend on the electric parameters and the hydrodynamic conditions (Fig. 2, a).

The initial stages of ignition exhibited a micro-discharge mode manifested as a series of short-lived glowing channels

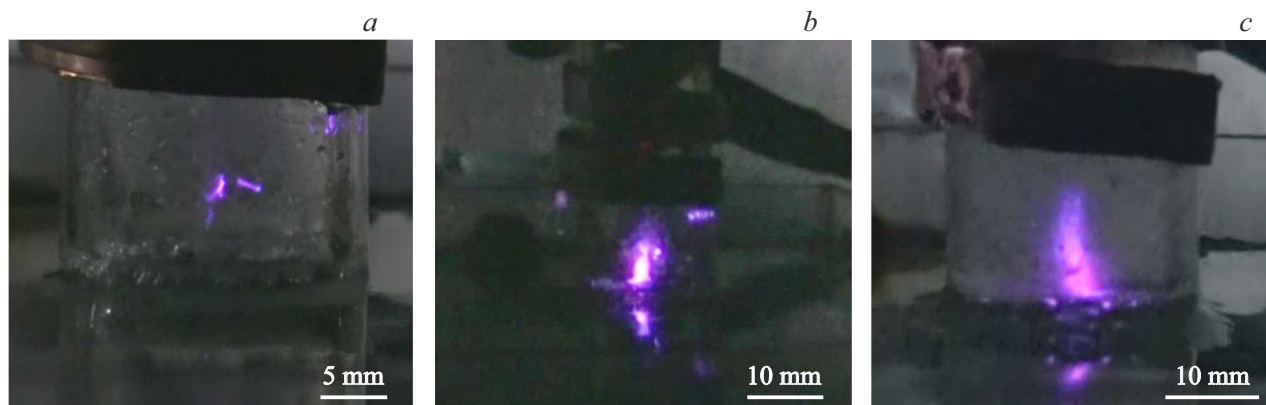


Figure 2. Photos of the jet HFI discharge with the liquid plasma-forming medium when $p = 10^4$ Pa; *a* — $U_p = 0.56$ V, $I_p = 0.8$ A; *b* — $U_p = 0.7$ V, $I_p = 1.1$ A; *c* — $U_p = 0.8$ V, $I_p = 1.3$ A.

that are localized predominantly in areas of narrowing and disturbances of the jet surface. These micro-discharges were characterized by a fluctuating nature of combustion and unstable dynamics: separate channels appeared and disappeared at a high rate, thereby resulting in flickering luminescence and heavy spatiotemporal instability of the plasma.

With an increase of the current and the discharge power, the system was transiting into a more stable filamentous-continuous mode (Fig. 2, *b*). In these conditions, a beam of stable plasma filaments was formed along the jet axis and they were uniformly distributed along the channel length. Glowing was getting a uniform nature and high reproducibility of the discharge shape was observed, thereby indicating predominance of inductive energy input over spurious capacitance pumping. A transition into this mode was accompanied by stabilization of the electric parameters and reduction of fluctuations of the amplitude of current and voltage.

With further amplification of energy deposition and development of jet disturbances, a quasi-mode of combustion was observed (Fig. 2, *c*). It was characterized by general exposure of a jet core and pronounced localization of radiation in a subsurface area of the copper sample. In these conditions, the plasma was becoming shaped as a truncated cone and the periphery exhibited developed filamentation. This mode was accompanied by more intense heat-mass transfer and electrolyte boiling, thereby amplifying interaction of the plasma with the copper surface and forming complex dynamic structures that included stable luminescence in a central part as well as fluctuating filaments on boundaries of the plasma channel.

Formation of the filaments was determined by a joint effect of the electromagnetic field and jet hydrodynamics. For the jet of the 1.5 mm diameter, at the speed of 0.8–0.9 m/s the Reynolds number was about 10^3 , thereby corresponding to the mode of a laminar flow with surface disturbances. The disturbances created local heterogeneities of the density and electrical conductance, at which the

microdischarges were initiated. Further on, accumulation of energy in the channels and local Joule heating resulted in development of stable filaments, which stabilized with a balance achieved between inductive energy input, convective cooling by the liquid jet, processes of evaporation and partial boiling of the electrolyte as well as radiation-induced losses.

Calculated estimates of the electrophysical parameters demonstrate that with a jet cross-section of about $1.77 \cdot 10^{-5} \text{ m}^2$ the current density reached $4.5 \cdot 10^5$ – $7.6 \cdot 10^5 \text{ A/m}^2$ (453–764 kA/m²). The respective specific power across the jet cross-section was $2.5 \cdot 10^8$ – $6.3 \cdot 10^8 \text{ W/m}^2$. These values indicate extremely high concentration of energy in the plasma channel, thereby explaining the localized nature of thermal impact on the surface of the copper sample. At the same time, the external elements of the unit were moderately heated, which is confirmed by data of infrared thermography: the distribution of the temperatures on the quartz tube and the inductor remained quite uniform and there was no superheating. It indicates that the main part of energy is spent directly within the plasma volume and for interaction with the copper surface, rather than thermal losses to a unit structure.

The lower-pressure jet HFI discharge has a multi-stage nature of combustion, which exhibits a successive transition from fluctuating microdischarges to the stable filaments and then to the cone-like dynamic structures. Formation of these modes is based on a complex combination of inductive input of energy, hydrodynamic specific features of the jet and the heat-mass transfer processes. The high energy concentration in the plasma channel defines efficiency of interaction with the copper surface and is a key factor of modification of its physical-chemical properties.

With the increase of stability of combustion from the micro-discharge mode to the filamentous and the quasi-conical mode, effective voltage at the active length was increasing from 0.56 to 0.83 kV (Fig. 3, *a*).

It reflects the transition to the more conductive plasma, wherein higher energy deposition is required for sustaining

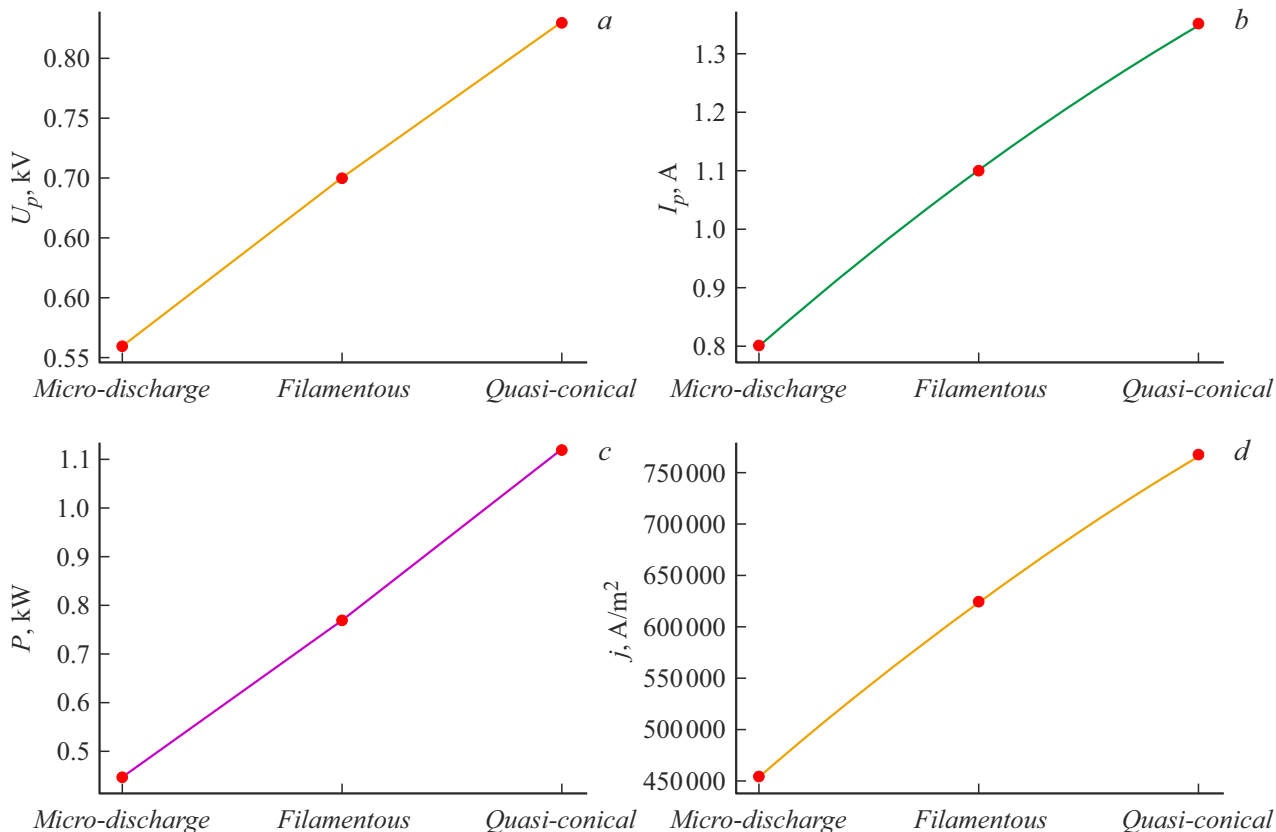


Figure 3. Dependences: *a* — discharge voltage U_p on the mode of combustion of the jet HFI discharge (micro-discharge → filamentous → quasi-conical); *b* — discharge current I_p on the mode of combustion of the jet HFI discharge; *c* — discharge power P during the transition from the micro-discharge mode to the filamentous and the quasi-conical mode; *d* — the current density j in a jet cross-section on the mode of combustion of the HFI discharge.

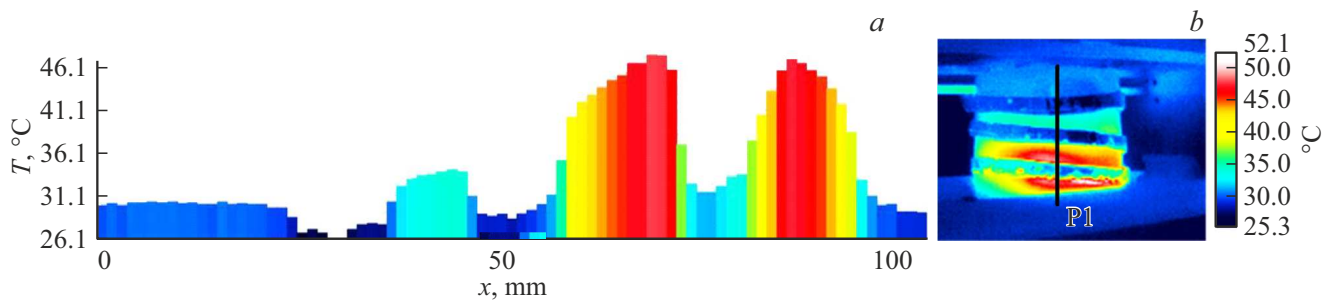


Figure 4. Distribution of the temperature (*a*) along the black line on the surface of the quartz tube and the inductor (*b*) and a thermogram of the quartz tube and the inductor (*b*) under conditions of combustion of the lower-pressure jet HFI discharge with the liquid plasma-forming medium.

volume ionization. Amplification of the inductive contribution (H-mode) is accompanied by reduction of a role of the spurious capacitance component and stabilization of the filaments.

The increase of current from 0.80 to 1.35 A indicates an increase of conductance of the plasma channel and narrowing of the effective area of current-carrying areas (Fig. 3, *b*). This dynamics indicates suppression of fluctuations of capacitance pumping and the transition to stable

filamentous combustion that is accompanied by formation of the stable beam of the plasma channels.

Integral power increased from 0.45 to 1.12 kW (Fig. 3, *c*). The observed power increase directly correlated with variation of the discharge morphology: from the fluctuating microdischarges through the stable filaments to a quasi-conical torch associated with the copper surface. Thus, the transition to the more intense modes was accompanied by the increase on energy deposition to the plasma.

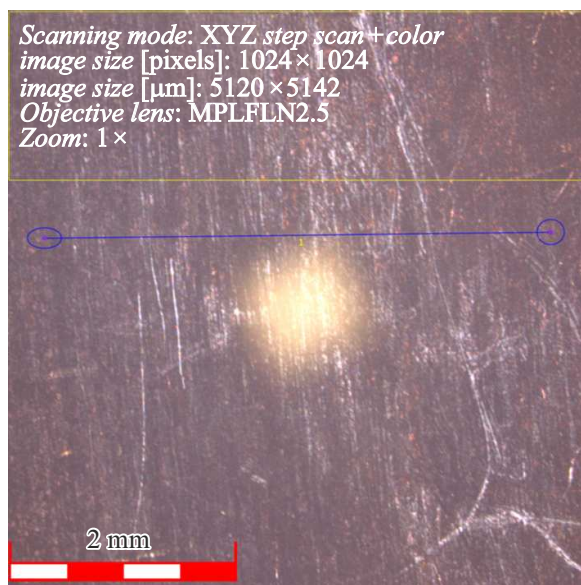


Figure 5. Photo of the surface of the copper plate with a local area treated with the lower-pressure jet HFI discharge.

At the jet diameter of 1.5 mm and the currents 0.8–1.35 A, the current density was up to $4.5 \cdot 10^5$ – $7.6 \cdot 10^5$ A/m² (Fig. 3, *d*). These values are typical for the compressed plasma channels and provide the high energy concentration within a limited area. This is exactly what explains the local nature of thermal impact on the copper surface without significant superheating of the unit elements.

Results of the thermographic studies demonstrated a pronounced heterogeneity of the temperatures in the discharge chamber (Fig. 4, *a*).

The maximum temperature recorded on the surface of the quartz tube near combustion of the HFI discharge was 47.6 °C (320.75 K). The minimum values of the temperature were observed on the surface of the copper inductor — 26.2 °C (299.35 K), which is related to its effective cooling by convection of ambient air and a thermal barrier in the form of a vacuum interlayer. The average temperature along the studied portion on the surface of the quartz tube with the inductor (Fig. 4, *b*) was 34.1 °C (307.25 K).

A temperature difference between the hottest area and relatively cold elements of the unit reached ~ 21 °C, thereby indicating high efficiency of heat removal. The main part of thermal energy was taken away by the circulating electrolyte passing through the interaction area and scattered by removal into copper and plasma radiation. Limitation of heating of structural elements was additionally contributed by lower pressure that decreased a role of convective thermal exchange between the plasma and walls of the quartz tube.

It is important to underline that the temperature of the surface of the inductor (26.2 °C) almost coincided with the temperature of the environment. It means that input

energy of the HF oscillator is basically spent for maintaining the plasma torch, heating and partially evaporating the electrolyte as well as for processes of ionization and dissociation, rather than for useless heating of the structural elements.

Thermal imaging control has confirmed that at the high temperatures in the area of interaction of the jet with the copper surface the external elements of the unit are slightly heated. The maximum temperature on the chamber surface did not exceed 48 °C, thereby indicating an effective thermal mode of system functioning and safety of its operation.

Fig. 5 shows a photo of the copper plate after treatment: its center exhibits a light round spot — the area of impact by the jet HFI discharge.

Clarification is explained by variation of the topography and the chemical composition of the surface: removal of adsorbed contaminants and redistribution of oxide layers, thereby affecting reflectivity of the metal.

Fig. 6, *a* shows a SEM image of the morphology of the copper surface before treatment. The surface is covered with parallel scratches that form typical depressions and protrusions. According to the results of measurements of the roughness parameters: $R_a = 0.746 \mu\text{m}$, $R_z = 2.992 \mu\text{m}$. According to tabular values of the roughness classes as per GOST 2789-73/GOST 2789-59 this combination of the parameters approximately corresponds to the class 5–6 ($R_a \approx 0.63$ – $1.6 \mu\text{m}$, $R_z \approx 2.5$ – $5 \mu\text{m}$).

After impact by the HFI discharge, the surface was greatly flattened (Fig. 6, *b*). Large defects and scratches are smoothed and the measurements demonstrated that $R_a = 0.166 \mu\text{m}$ and $R_z = 0.822 \mu\text{m}$. These values already fall within a range of the class 8–9 ($R_a \approx 0.16$ – $0.25 \mu\text{m}$, $R_z \approx 0.8$ – $1.6 \mu\text{m}$).

Reduction of the roughness parameters almost in five times in terms of R_a and in ~ 3.6 times in terms of R_z indicates great flattening of the relief. This effect may be caused by local melting and remelting of a microrelief and ion bombardment. Impact by the jet HFI discharge shifted the copper surface from the class ~ 5–6 into a region of the classes ~ 8–9 as per GOST, thereby significantly improving physical-mechanical properties of the surface, reducing microcontacts, being able of affecting cohesion of the coatings (an effect direction depends on the „coating-copper“ system and the treatment modes) and reducing susceptibility to corrosion.

In order to estimate influence of the lower-pressure jet HFI discharge on the physical-chemical properties of copper, we have studied a contact angle of distilled water. This parameter is a sensitive indication of the surface state, since it depends on its morphology, chemical composition and presence of oxide or organic films.

Before treatment, the copper surface was characterized by the contact angle of 68°, thereby corresponding to a moderately hydrophilic state (Fig. 7, *a*). This value is related to presence of natural oxide layers that are formed during contact of copper with the atmosphere as well as to pronounced microroughness of the surface, which enhances

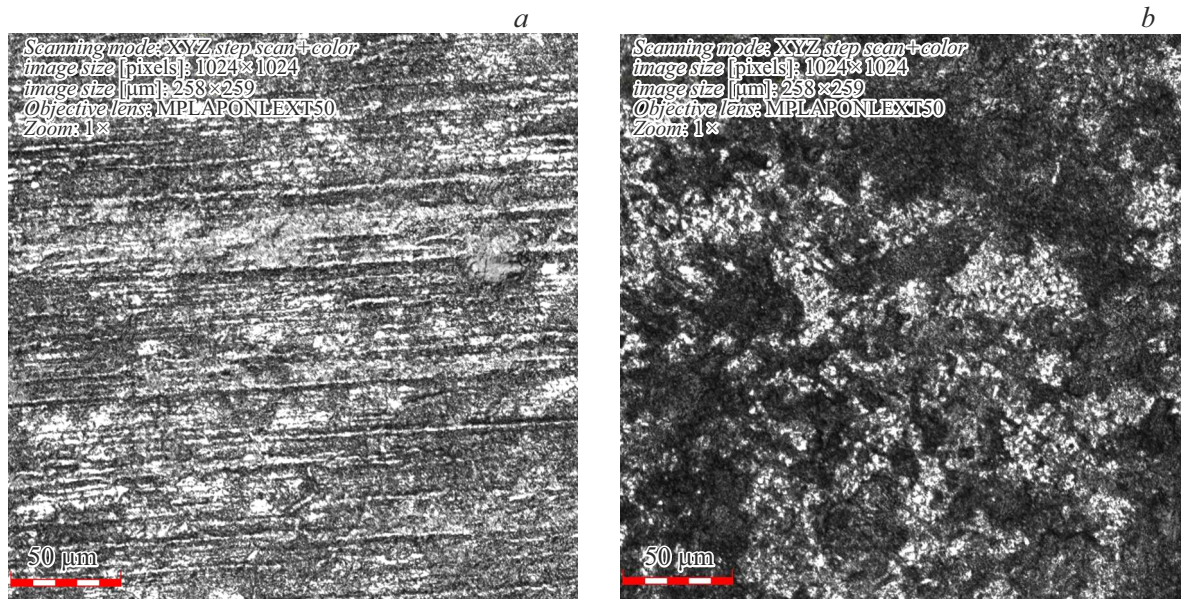


Figure 6. SEM image of the copper surface morphology: *a* — before treatment, *b* — after treatment.

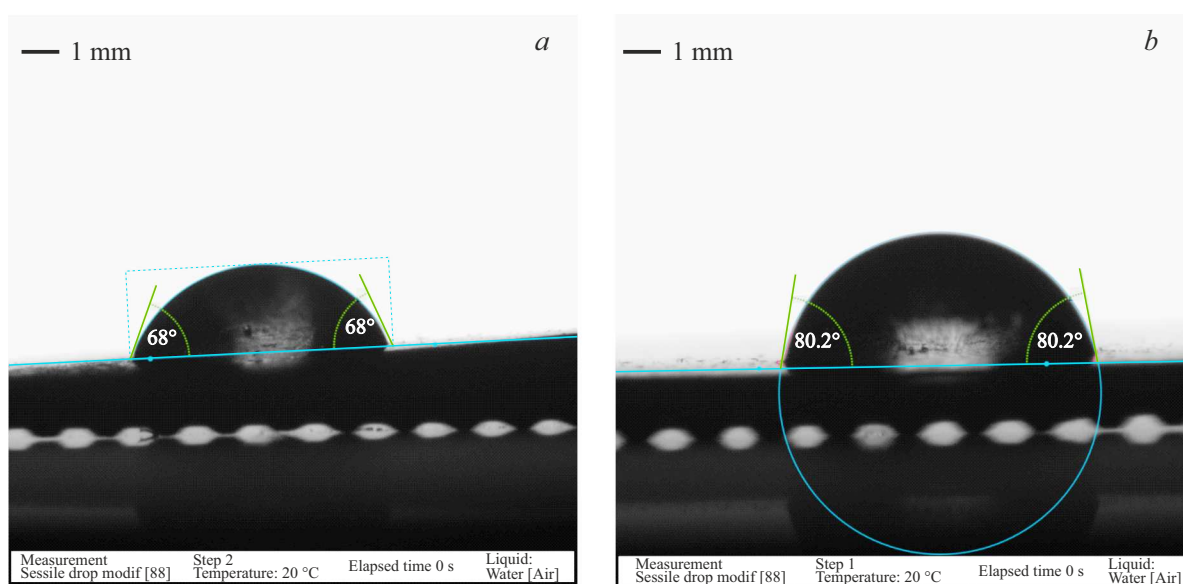


Figure 7. Contact angle of the copper surface: *a* — before treatment, *b* — after treatment.

a capillary effect and contributes to spill of a drop over the surface. Mechanical traces of treatment, which were shaped as parallel scratches, additionally created channels, through which the liquid could propagate, increasing effective wetting.

After impact by the plasma discharge, the contact angle increased to 80.2° , thereby indicating reduction of hydrophilicity and the transition of the surface to a more hydrophobic state (Fig. 7, *b*). This effect is explained by a combination of several factors. First of all, significant smoothing of the relief: the roughness parameters decreased from $R_a = 0.746 \mu\text{m}$ and $R_z = 2.992 \mu\text{m}$ to $R_a = 0.166 \mu\text{m}$

and $R_z = 0.822 \mu\text{m}$, thereby reducing the area of actual contact of the liquid with the surface and limiting capillary spill. Secondly, plasma impact results in modification of the chemical state of copper: formation of new oxide structures, redistribution of oxygen and copper on the surface, thereby changing its surface energy. It is also contributed by thermal impact of the discharge, which facilitates partial recrystallization and compaction of the surface layer.

The observed increase of the contact angle from 68° to 80.2° indicates reduction of water wettability. Conclusions on strength of cohesion of the coatings were not made and they require separate mechanical tests. The present

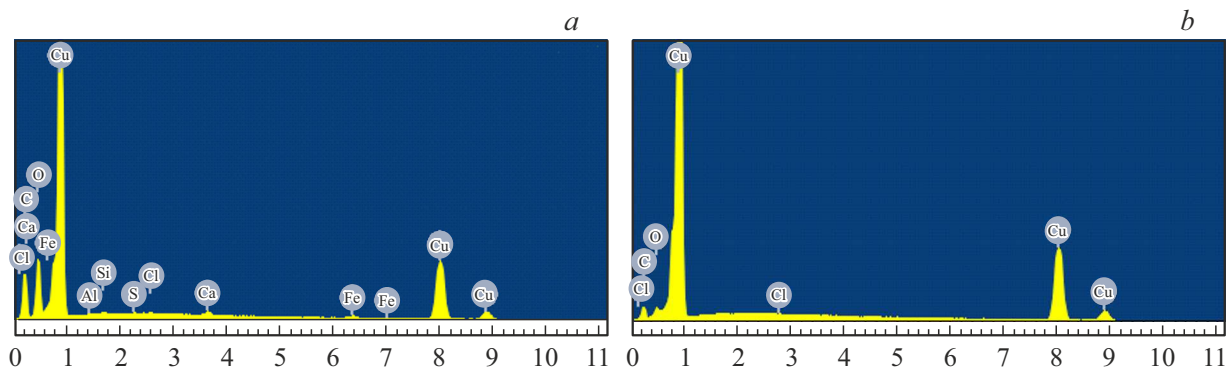


Figure 8. Elementary composition of the sample surface: *a* — before treatment, *b* — after treatment.

study recorded controlled modification of wettability. Before treatment, the surface had pronounced susceptibility to liquid spill, after treatment it demonstrates higher resistance to liquid spill. This effect is related to reduction of roughness and the change of the chemical composition of the surface layer (Fig. 8). This effect can also have important application significance: higher hydrophobicity contributes to reduction of a corrosion rate in aggressive media, reduces probability of formation of galvanic cells in presence of humidity and can be used for improving operational characteristics of the copper parts. Along with the morphology change and reduction of roughness, modification of the contact angle confirms that impact of the jet HFI discharge is an effective tool of controlling physical-chemical properties of the copper surface, enabling its purposeful functionalization for various technical applications.

Conclusion

1. We have experimentally studied a process of interaction of the lower-pressure jet HFI discharge with the surface of the M1-grade copper plate. It is found that combustion of the discharge is of the filamentous-jet nature and can be implemented in the micro-discharge, filamentous and quasi-conical modes.

2. The electro-physical characteristics of the discharge have been determined: voltage $U_p = 0.56–0.83$ V, current $I_p = 0.8–1.35$ A, the current density $j = 4.5 \cdot 10^5–7.6 \cdot 10^5$ A/m². It is demonstrated that the increase of the current and the current density correlate with the transition from the fluctuating micro-discharge mode to the stable filamentous mode.

3. The thermal imaging measurements have demonstrated that a maximum of the temperature of the quartz tube limiting the area of combustion of the HFI discharge was 47.6 °C, whereas the inductor temperature did not exceed 26.2 °C.

4. The morphological analysis has identified significant smoothing of the relief: the average roughness decreased from $R_a = 0.746$ to 0.166 μm, while the maximum height of irregularities decreased from $R_z = 2.992$ to 0.822 μm. The

copper surface transited from the range of the classes 5–6 into the region of the classes 8–9 as per GOST 2789-73.

5. The contact angle has been measured to demonstrate that it increased from 68 to 80.2°, thereby indicating reduction of hydrophilicity and the increase of hydrophobicity of the treated surface. This effect is related to reduction of roughness and the change of the chemical composition of the surface layer.

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Conflict of interest

The authors declare that they have no conflict of interest.

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