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Thermal regimes of high-temperature oxidation of tungsten wires

Research on tungsten oxidation is essential for creating functional oxide materials, developing next-generation photocatalysts, and constructing modern models of high-temperature heat and mass transfer and phase formation. Heat and mass transfer processes during tungsten oxidation significantly influence the properties of tungsten oxides at virtually all levels — from phase composition to electrophysical characteristics. It is heat and mass transfer that determines the morphology of oxides and surface self-organization.

This work is dedicated to a detailed analysis of the influence of various heat and mass transfer mechanisms and phase transitions on the low- and high-temperature steady-state and critical regimes of tungsten oxidation. Physical and mathematical modeling was carried out to study the heat and mass transfer processes and the oxidation kinetics of tungsten wires heated by an electric current in air.

The conducted studies showed that in steady-state low-temperature oxidation regimes, as well as near the critical current value corresponding to the ignition of the tungsten wire, heat losses due to the evaporation of the oxide layer are negligible and can be omitted. However, at wire temperatures exceeding 1500 K, oxide evaporation processes become crucial and significantly affect the formation of dendritic branched structures of tungsten trioxide on the conductor surface.

The role of radiative heat loss from the surface of the heated wire to the walls of the reaction chamber was investigated in steady-state and critical regimes corresponding to wire ignition and extinction. It was established that for wires with a diameter of less than 100 μm, thermal radiation to the apparatus walls can be neglected. For wires with a diameter exceeding 100 μm, accounting for radiative heat losses in the physical and mathematical model leads to an increase in the critical current value at which high-temperature oxidation regimes occur.

It is demonstrated that natural convection substantially influences the stable and critical oxidation regimes of tungsten wires. It was found that the intensification of heat transfer due to natural convection causes an increase in the critical current value by approximately 20%.

Keywords: *heat and mass transfer, oxidation, tungsten, tungsten oxides, evaporation, radiation, convection.*

Introduction. The relevance of researching tungsten oxidation processes has significantly grown in recent years due to the development of thermonuclear energy, nanotechnology, photocatalytic systems, and functional oxide materials [1-3]. Tungsten is characterized by an extremely high melting point, high thermal conductivity, and excellent erosion resistance, which leads to its widespread use in high-temperature technologies, particularly as a material for plasma-facing components in thermonuclear reactors [4]. Tungsten trioxide (WO_3), with its sensitivity to visible light, reliable stability, adaptive morphology, and tunable electronic structure, has become a primary material for the photocatalytic production of hydrogen and hydrogen peroxide [5]. At the same time, when interacting with oxygen-containing environments, tungsten oxidizes intensively, forming multiphase oxide layers and volatile WO_3 oxides, which can lead to surface degradation, loss of material mass, and the formation of oxide dust.

Significant attention is also paid to the photoelectrochemical properties of tungsten oxides. Due to its relatively narrow bandgap and high chemical stability, WO_3 is considered one of the promising materials for photocatalytic water splitting processes [6], hydrogen generation, and environmental remediation. Current research is aimed at modifying the oxide structure through doping, creating heterostructures, and managing oxygen vacancies to increase charge transfer efficiency and photocatalytic activity [7].

A distinct modern direction is the study of the morphology of tungsten oxide structures. The formation of dendritic, porous, needle-like, and nanocrystalline structures during thermal oxidation is of considerable interest for surface physics and the theory of self-organization [8–10]. Analyzing such structures using fractal geometry methods and electron microscopy allows for establishing the relationship between oxidation conditions, oxide growth mechanisms, and surface evolution.

One of the methods for obtaining tungsten oxides is the controlled high-temperature oxidation of a metallic conductor heated by an electric current in a regulated gaseous medium (electrothermal heating) [8–12]. The advantages of this method lie in its cost-effectiveness, simplicity of implementation, and the possibility of simultaneous measurement of electrical and temperature parameters, as well as the study of oxide structures on the metal surface.

High-temperature tungsten oxidation is accompanied by phase transformations on the metal surface: melting and evaporation of the oxide film. These processes occur with the absorption of heat. Therefore, phase transitions can affect the quantitative characteristics of high-temperature oxidation.

The aim of this work is to investigate, through physical and mathematical modeling, the influence of various heat and mass transfer mechanisms and phase transitions on the steady-state and critical regimes of heat and mass transfer and oxidation of a tungsten wire.

Physical and Mathematical Modeling. Let us consider the heat and mass transfer and the oxidation kinetics of a tungsten wire heated by an electric current in air. An increase in the wire temperature leads to the activation of the chemical oxidation reaction on its surface. Let us assume that an oxide film consisting predominantly of tungsten trioxide WO_3 is formed on the wire surface according to the equation $W+O_3 \rightarrow WO_3$. Utilizing the physical and mathematical model [10], we determine the heat fluxes that describe the thermal balance of the wire.

Since the oxidation reaction proceeds according to a parabolic law, its rate is limited by the thickness of the oxide film [11], and the chemical heat release density on the conductor surface q_{ch} is defined by the expression:

$$q_{ch} = Q\frac{D}{h}C_{O_2S}, \tag{1}$$

where Q is the thermal effect of the reaction, J/kgO_2; h is the oxide film thickness, m; C_{O2s} – is the oxygen concentration at the metal surface, kg/m^3; D is the diffusion coefficient of oxygen through the oxide layer to the metal surface, m^2/s.

The diffusion coefficient depends on temperature according to the Arrhenius law: $D = D_0 \exp\left(-\frac{E}{RT}\right)$, where E is the activation energy, J/mol; and T is the wire temperature, K.

The oxygen concentration at the wire surface is determined from the condition that the oxygen mass flux to the surface equals the rate of its consumption during oxidation:

$$C_{O_2S} = C_{O_2\infty}\left(1 + \frac{D}{h\beta}\right)^{-1}, \qquad \beta = \frac{Sh \cdot D_g}{d},$$

where $C_{O_2\infty}$ is the mass concentration of oxygen in the air, kg/m^3; d is the conductor diameter, m; β is the mass transfer coefficient, m/s; Sh is the Sherwood number; and D_g is the diffusion coefficient of oxygen in the air, m^2/s.

For the molecular-convective heat transfer of the heated conductor with air, we write the expression:

$$q_\alpha = \alpha\left(T - T_g\right), \ \alpha = \frac{Nu \cdot \lambda_g}{d}, \qquad (2)$$

where T_g is the gas temperature, K; α is the heat transfer coefficient, W/(m^{2}K); λ_g is the thermal conductivity coefficient of the gas (air), W/(m·K) and Nu is the Nusselt number.

It is well known that for thin wires, the condition Nu = Sh=0.5. However, to calculate the heat transfer coefficients under natural convection for wires of small diameter (d=0.2÷1mm), the following empirical formula [12] can be used:

$$\mathrm{Nu} = 1.18(\mathrm{Gr}\,\mathrm{Pr})^{1/8} \qquad (3)$$

$$Gr = \frac{g(T - T_g)d^3\rho_g}{273\mu_g^2}, \quad \mathrm{Pr} = \frac{\mu_g c_g}{\lambda_g},$$

where Gr and Pr are the Grashof and Prandtl numbers, respectively; and μ_g, c_g ρ_g are the dynamic viscosity, specific heat capacity, and density of air, respectively (Pa·s, J/(kg·K), kg/m^3).

The radiative heat flux between the conductor and the walls of the reaction apparatus is accounted for by the Stefan-Boltzmann law:

$$\mathrm{q_w} = \varepsilon\sigma\left(\mathrm{T}^4 - \mathrm{T_w^4}\right), \qquad (4)$$

Where ε is the emissivity of the oxide film; and T_W is the temperature of the reaction apparatus walls, K. In our case, $T_W = T_g = T_r$, where T_r is the room temperature of the air, K.

Tungsten oxide is volatile, and upon reaching high temperatures, its sublimation and evaporation occur. Intensive evaporation of the WO_3 oxide begins after its melting. Tungsten trioxide melts at a temperature of 1470°C, but it sublimates relatively quickly even at lower temperatures [13]. In the thermal balance of the conductor, we account for the heat losses due to oxide evaporation:

$$q_{ev} = L_{ev}\frac{ShD_{WO_3}}{d}\frac{M_{WO_3}P_S}{RT}, \tag{5}$$

where D_{WO3} is the diffusion coefficient of WO_3 vapor in the air, m^2/s; P_S is the saturated vapor pressure of tungsten trioxide near the wire surface, *Pa*; and M_{WO3} is the molar mass of WO_3.

At the connection points where the tungsten wire attaches to the terminals, a conductive heat flux arises:

$$q_\lambda = \frac{\alpha \cdot S_L \cdot \left(T - T_g\right)}{S_d \cdot \theta}, \tag{6}$$

where S_d, S_L are the cross-sectional area and the lateral surface area of the wire, respectively, m^2; and θ is a dimensionless parameter that depends on the thermal conductivity coefficient of the metal, the conditions of heat transfer with the environment, and the geometric dimensions of the conductor. In this case, we assume that the temperature of the current-carrying contacts equals the ambient temperature T_g.

The heat flux density that heats the conductor due to the flow of an electric current (according to the Joule–Lenz law), taking into account its dependence on the geometric dimensions of the wire and temperature, can be expressed as:

$$q_{DL} = \frac{4I^2}{\pi^2 d^3}\rho_m, \qquad \rho_m = \rho_0\left(1+\gamma\left(T-T_0\right)\right), \tag{7}$$

where I is the electric current, A; ρ_m and ρ_0 are the electrical resistivity of tungsten at temperatures T and T_0, respectively (T_0=273 K), Om·m; and γ is the temperature coefficient of resistance, K^{-1}.

Steady-state regimes of high-temperature heat and mass transfer and tungsten wire oxidation are realized under the condition:

$$q_{ch} + q_{DL} = q_\alpha + q_w + q_\lambda + q_{ev}, \tag{8}$$

Equation (8), along with expressions (1)–(7), determines the stable high- and low-temperature regimes of heat and mass transfer and wire oxidation, as well as the critical conditions for transitioning between these regimes (ignition and extinction) under various heat and mass transfer mechanisms and heating currents.

From the steady-state condition (8), we obtain the dependence I^2 (T):

$$I^2 = \frac{\pi^2 d^3}{4\rho}\left[q_\alpha + q_w + q_\lambda + q_{ev} - q_{ch}\right], \tag{9}$$

which allows for the determination of the steady-state temperature regimes of the wire at various heating currents.

Let us analyze the influence of oxide film evaporation and radiative heat transfer on the stable and critical oxidation regimes of a tungsten wire by examining the calculated dependences of the steady-state temperature on the current intensity (Fig. 1).

The extrema on the T(I) curves characterize the critical regimes of wire ignition (point I - maximum) and extinction (point E - minimum) at the corresponding critical current intensities I_I and I_E. The curve up to point I defines the stable low-temperature oxidation regimes, while the curve after point E defines the stable high-temperature

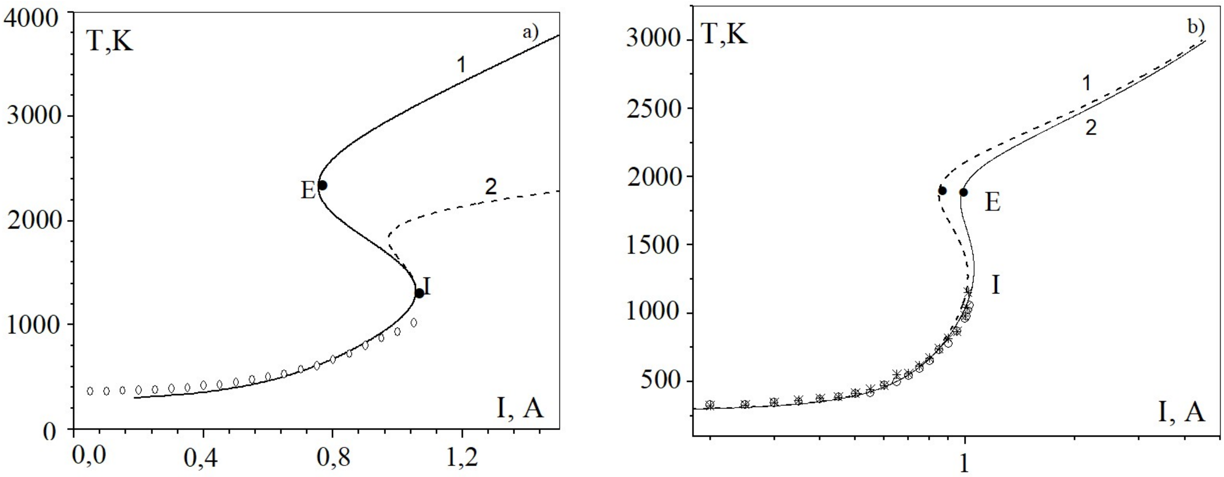

Fig. 1. Dependences T(I) for a tungsten wire with a diameter of d = 70 μm and a length of L = 0.1 m: a) 1 - excluding oxide evaporation, 2 - taking into account; b) 1 - excluding radiative heat transfer; 2 - including radiative heat transfer; circles and asterisks indicate experimental data. T_g=T_w=288 K.

oxidation regimes. As the current intensity in the low-temperature regime increases to the critical value I_I (point I), a jump-like transition to the high-temperature state occurs. Conversely, a transition from the high-temperature state back to low-temperature oxidation can be achieved by decreasing the current intensity to the value I_E. Thus, within the range $I_I > I > I_E$, hysteretic regimes of heat and mass transfer and wire oxidation ar e observed. Within the hysteresis region, a transition to the high-temperature state can be induced by increasing the initial wire temperature within the interval $T_E > T > T_I$. For currents $I < I_E$, wire ignition is impossible regardless of its initial temperature.

It follows from Figure 1a that the heat losses due to the evaporation of the oxide film do not affect the low-temperature oxidation regimes or the critical current intensity I_I at which the wire ignition occurs. A significant effect of evaporation occurs at high temperatures, when the wire temperature reaches the melting point of tungsten trioxide and exceeds it. This is evident from a comparison of the high-temperature oxidation regimes with (curve 2) and without (curve 1) accounting for evaporation. It is the evaporation of the WO_3 oxide that constitutes one of the key links in the mechanism of oxide crystal growth on the surface of the primary oxide layer [9–10].

Let us consider the influence of radiative heat losses on the steady-state and critical oxidation regimes of a thin tungsten wire (Fig. 1b). It is apparent that for low-temperature regimes, this influence is insignificant. For high-temperature oxidation, lower temperatures and a lower critical current intensity at which the oxidation reaction extinction occurs are observed. Thus, for small wire diameters, radiative heat transfer can be neglected when modeling low-temperature oxidation regimes.

Let us analyze the influence of radiation, natural convection, and the chemical oxidation reaction on the steady-state heat and mass transfer regimes of a tungsten wire with a diameter of 200 μm. This is practically three times larger than the diameter of the previous conductor (70 μm).

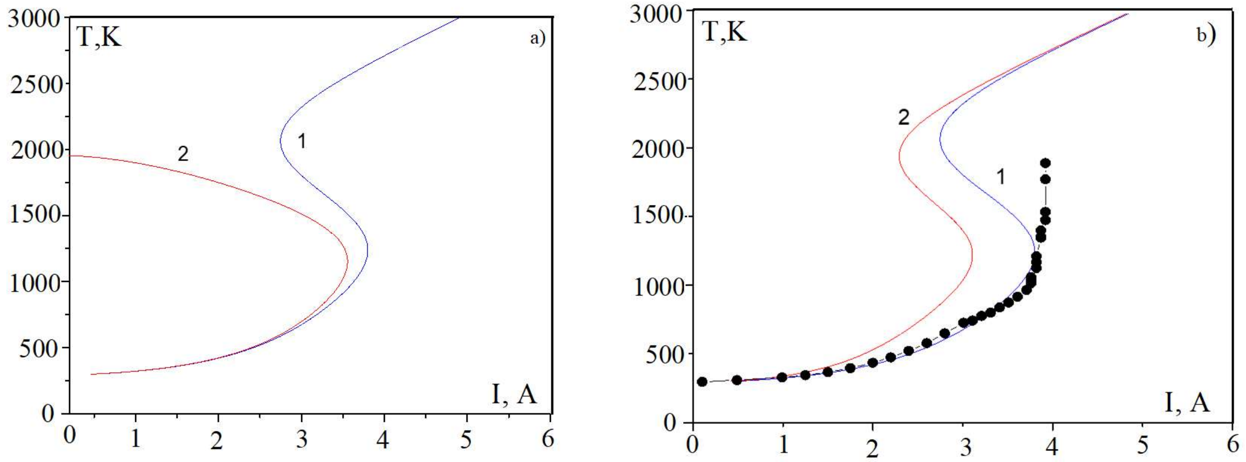

Fig. 2. Influence of radiative heat transfer (a) and natural convection (b) on the steady-state regimes of a tungsten wire heated by an electric current in air. d=200 μm, L=0.115 m, $T_g = T_w = 293$K. a) 1 – accounting for radiative heat losses; 2 – without accounting for radiative heat losses; b) (in the presence of radiative heat transfer): 1 – accounting for natural convection; 2 – without accounting for natural convection; • – experimental data.

It can be seen from Figure 2a that without accounting for radiative heat losses, the critical current intensity I_I decreases by approximately 7%, and the critical extinction regime is not observed at all. For such diameters, incorporating radiation into the calculations is essential.

The calculations presented in Figure 2b illustrate the significant influence of natural convection on the steady-state and critical regimes of heat and mass transfer and tungsten wire oxidation. Natural convection was accounted for by introducing the semi-empirical Nusselt correlations (3) into the Newton–Richman law (2). Consequently, the molecular-convective component of the wire's thermal balance increased due to the convective heat and mass fluxes from the electrically heated conductor. This led to a decrease in the wire temperature and a significant increase in the critical current intensity at which the conductor ignites and transitions to high-temperature oxidation.

It should be noted that the steady-state high-temperature regimes (the upper branches of the T(I) dependences) obtained by calculations are not realized in experimental studies. In the experiment, the following pattern of ignition and transition to high-temperature wire oxidation is observed. Upon reaching the critical current intensity, the chemical oxidation reaction on the wire surface becomes rapid according to the Arrhenius law. This releases a large amount of heat (described by formula (1)), which, together with Joule heating, heats the conductor to high temperatures. Melting and evaporation of the oxide layer occur. At this stage, oxide crystals (branched dendritic structures) actively grow on the surface of the primary oxide layer [10], and new condensation centers arise at the sites of local oxide melting, impurities, and mechanical stresses within the oxide film. Due to the condensation of trioxide molecules onto these condensation centers, the oxide crystals grow in both longitudinal and transverse directions, frequently coalescing with each other. A further increase in the wire temperature due to chemical heat release leads to the onset of a transient (non-steady-state) oxidation regime. The molten oxide film evaporates rapidly, facilitating oxygen access

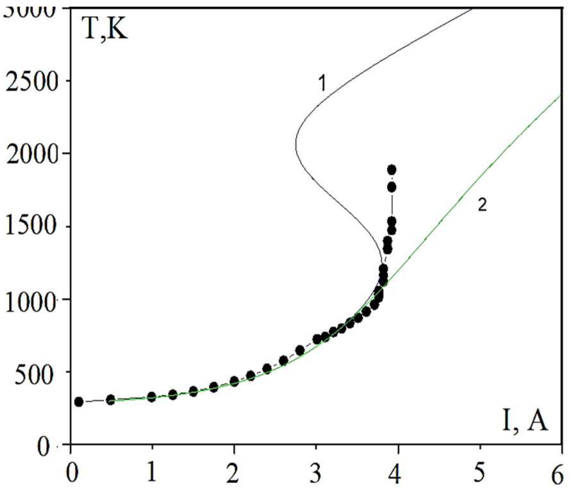

Fig. 3. Dependence T(I) for d=200μm, L=0.115m. 1 - $q_{ch}\neq 0$; 2 - $q_{ch}=0$; • – experiment.

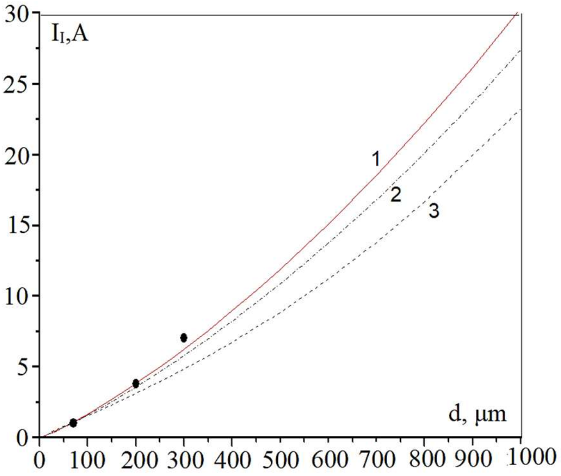

Fig. 4. Dependences of the critical current intensities at which the wire ignition occurs on its diameter. L = 0.115 m, T_w = T_g = 293K. 1 – accounting for all heat transfer mechanisms; 2 – without accounting for radiative heat losses; 3 – without accounting for natural convection; • – experiment.

to the conductor surface. This, in turn, accelerates the oxidation rate and increases the amount of heat released. Due to these chemical and phase processes, the conductor rapidly thins at its center. The ends of the conductor heat up insignificantly due to thermal conduction to the cold current-carrying contacts. The transient high-temperature heat and mass transfer terminates with the melting of the metal itself and the destruction of the wire at its center [14]. Thus, near the critical current intensity I_I , one can speak of quasi-steady-state conductor oxidation, which over time transforms into a transient process.

Fig. 3 illustrates the fact that it is precisely the presence of the chemical oxidation reaction that causes the hysteresis of heat and mass transfer processes. For an inert wire (q_{ch}=0) monotonic heating (curve 2) is observed due to the electric current power.

Fig. 4 presents the critical conditions for tungsten conductor ignition in the form of the dependence of the critical ignition current intensity on the conductor diameter.

The calculations were performed for cases accounting for all types of heat losses from the conductor: convection, radiation, and thermal conduction to the cold contacts, as well as in the absence of radiative heat transfer and natural convection. It is apparent that the experimental data fit well with the curve that accounts for all heat transfer mechanisms. The analysis shows that for thin conductors d<100 μm the influences of natural convection and radiation can be neglected. For diameters exceeding this value, incorporating natural convection is necessary. For conductor diameters greater than 100 μm it is also essential to account for radiative heat losses in the physical and mathematical model.

Conclusions. Detailed physical and mathematical modeling of heat and mass transfer processes and oxidation kinetics of tungsten wires heated by an electric current in air has been performed. It is shown that heat losses due to the evaporation of the oxide layer can be neglected in steady-state low-temperature states and within the range of the critical current intensity that determines the wire ignition. However, at wire temperatures above 1500K oxide evaporation plays a key role in the formation of branched dendritic tungsten trioxide structures on the wire surface.

The influence of radiative heat losses from the heated wire surface to the reaction vessel walls on the steady-state and critical states determining the wire ignition and extinction has been analyzed. It is proven that for wires with diameters d < 100 μm radiative heat losses to the walls of the experimental setup can be neglected. For d > 100 μm incorporating radiative heat losses into the physical and mathematical model leads to an increase in the critical current intensity I_I at which high-temperature oxidation processes occur.

The significant influence of natural convection on the stable and critical oxidation regimes of a tungsten wire has been demonstrated for d > 100 μm. It has been established that the enhancement of heat transfer from the wire due to natural convection results in a 20% increase in the critical current intensity I_I.

It is shown that the calculations based on the physical and mathematical model accounting for heat losses via convection, radiation, and thermal conduction to the contacts agree well with the experimental data regarding the critical current intensities at which the tungsten wire ignites and transitions to transient high-temperature oxidation.

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Теплові режими високотемпературного окислення вольфрамових дротиків

Анотація

Дослідження окислення вольфраму мають важливе значення для створення функціональних оксидних матеріалів, розробки фотокаталізаторів нового покоління та побудови сучасних моделей високотемпературного тепломасопереносу й фазоутворення. Процеси тепломасообміну при окисленні вольфраму дуже суттєво впливають на властивості оксидів вольфраму, причому фактично на всіх рівнях — від фазового складу до електрофізичних характеристик. Саме тепломасообмін визначає морфологію оксидів та самоорганізацію поверхні при нагріванні.

Дана робота присвячена детальному аналізу впливу різних механізмів переносу тепла і маси, фазових переходів на низько- та високотемпературні стаціонарні і критичні режими окислення вольфраму. Проводилось фізико-математичне моделювання процесів тепломасообміну та кінетики окислення вольфрамових дротиків, які нагрівались електричним струмом в повітрі.

Проведені дослідження показали, що в стаціонарних низькотемпературних режимах окислення, а також поблизу критичного значення сили струму, яке відповідає запаленню вольфрамового дротика, втрати теплоти на випаровування оксидного шару є незначними і можуть не враховуватись. Разом з тим при температурах дротика понад 1500 К процеси випаровування оксиду набувають визначального значення та суттєво впливають на формування дендритних розгалужених структур триоксиду вольфраму на поверхні провідника.

Досліджено роль радіаційних тепловтрат від поверхні нагрітого дротика до стінок реакційної камери в стаціонарних і критичних режимах, що відповідають запаленню та згасанню дротика. Встановлено, що для дротиків діаметром менше 100 мкм тепловим випромінюванням до стінок установки можна знехтувати. Для дротиків більшого діаметру врахування радіаційних тепловтрат у фізико-математичній моделі приводить до зростання критичного значення сили струму, за якого реалізуються високотемпературні режими окислення.

Показано, що природна конвекція істотно впливає на стійкі та критичні режими окислення вольфрамових дротиків. Встановлено, що інтенсифікація тепловіддачі внаслідок природної конвекції спричиняє збільшення критичного значення сили струму приблизно на 20%.

Ключові слова: *тепломасообмін, окислення, вольфрам, оксиди вольфраму, випаровування, випромінювання, конвекція.*