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INFLUENCE OF GAMMA-RADIATION ON CURRENT DENSİTY AND VOLT AMPERE CHARACTERISTICS OF METALLIC ZIRCONIUM

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Abstract:

The effect of preliminary radiation-oxidative treatment on the relative change in the resistance ($\Delta\rho$ / $\Delta\rho0$) of metallic zirconium has been studied. The contribution of preliminary radiation-oxidative treatment to the change in the electro physical characteristics during thermal and radiation-thermal tests in the contact of zirconium with water is revealed. The effect of preliminary radiation-oxidative treatment on the current density and current-voltage characteristic of metallic zirconium has been studied. The completion of the protective oxide film during the radiation-oxidative treatment of zirconium ($D \geq 80$ kGy) is accompanied by a decrease in electrical conductivity and current values in the study of their current-voltage characteristics. When these samples are tested in the processes of radiation-thermal and thermal decomposition of water, a partial destruction of the protective oxide film occurs as a result of which additional charge carriers accumulate on the surface. The rate of destruction of the surface oxide film and the accumulation of charge carriers during radiation-thermal processes are higher than during thermal processes.

Keywords: Metallic zirconium; γ-radiation; current-voltage characteristics; electro-physical; radiation-oxidatively.

1.Introduction

Radiation-heterogeneous processes in contact of preliminarily radiation-oxidative treated zirconium with water causes a change in the amount of surface oxide film. The formation of an oxide film, in turn, changes the radiation-catalytic activity and physicochemical properties, which affect the kinetic parameters. One of them, the most sensitive is the electro physical and optical properties of metal surfaces. Therefore, after testing zirconium samples in the process of water decomposition, the study of the electro physical and optical properties is of great interest (Liu and Han, 2022; Zhu et al., 2016; Ma, Yuan & Sehgal, 2016; Wang et al., 2021; Frano, Ciolini & Pesetti, 2020; Du, Bernat & Jay Gerin, 2000; Kang et al., 2009; Guello et al., 2000; Amblard et al., 2006).

The study of the kinetics of radiation, radiation-thermal and thermal processes shows that in metals used directly in a nuclear reactor, there is not only a change in natural defect states, but

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also the appearance of new defects - displaced interstitial atoms, as well as a change in surface properties due to adsorption, insertion other substances (for example O_2, H_2), corrosion, etc.

As is known, the accumulation of point defects and impurities introduced during irradiation (for example, O_2, H_2) strongly affect the physical properties of the metal (Zongyang et al., 2021). Thus, a consistent study of the physical properties before and after thermal and radiation-thermal treatment makes it possible to judge changes in electronic and structural defects and draw certain conclusions about the processes taking place (Garibov et al., 1992; Garibov, 2004; Garibov, 2005; Imanova, Agayev & Jabarov, 2021; Ali et al., 2021; Imanova & Hasanov, 2020; Imanova, 2021; Agayev, Musayeva & Imanova, 2021; Imanova, 2020; Imanova et al., 2021).

The purpose of this work is to change the resistance and thermo EMF. metallic zirconium, occurring in samples pretreated and tested under conditions of radiation-thermal and thermal effects. The purpose of this work is to change the current density and current-voltage characteristic of metallic zirconium, which occur in samples pretreated and tested under conditions of radiation-thermal and thermal effects.

2. Experimental

Investigated plates of reactor zirconium (purity 99.99%) with a thickness of d = 0.012 - 0.20 mm; width b = 2.0 - 4.0 mm and length l = 20 - 25 mm. The samples were preliminarily cleaned with ethyl alcohol, acetone and distilled water, dried first in air, then in vacuum ($1 \cdot 10^{-3}$ Pa) at T = 300 K, and then at T = 473 K. Then the samples were placed in ampoules with 30% - solution of hydrogen peroxide (CH_2O_2 = 9 mol/L) and subjected to preliminary exposure to gamma rays (absorbed dose rate D = 1.14 Gy / s) at different exposure times. Then the samples were dried and their electro physical parameters were measured. Then the samples were placed in special ampoules to test their radiation-catalytic activity in the processes of radiolysis decomposition of water. The required amount of water was introduced into ampoules with samples by condensation of water vapor from a graduated volume of a vacuum adsorption unit. The accuracy of introducing water into ampoules with samples from a vacuum adsorption installation in the investigated range of water vapor density values was ± 5%. During the experiments, the temperature was maintained with an accuracy of ± 1^0C. Radiation-oxidative treatment and radiation-thermal tests were carried out on an isotope source of γ-radiation ^{60}Co. Dosimetry of the source was carried out with chemical dosimeters - ferrosulfate, cyclohexane and methane. Measurements of electrical resistivity and measurement of current-voltage characteristics were carried out using four probe point contacts by the method of compensation at constant voltage (Gunel & Kaya, 2021). We used power supplies of the TES-41 brand, a universal

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voltmeter of the V7-21 and V7-21A brands (for measuring the voltage drop), and a combined digital device Sh4313 (for measuring current).

The electro-physical properties of samples subjected to preliminary radiation-oxidative treatment were also determined under conditions of radiation-thermal and thermal tests in contact with a heat transfer water (density ρ=5 mg/cm^3) at T=673K, D=1.14 Gy/s.

The kinetics of radiation decomposition of hydrogen peroxide in aqueous solutions has been studied in many works. Some of the dependencies established in them are consistent with each other. At the same time, there are some discrepancies. In (Imran et al., 2022; Gunel, 2022; Imran et al., 2020; Murat et al., 2021; Hokman et al., 2021), the kinetics of the reaction of radiation decomposition of hydrogen peroxide under the action of radiation in the concentration range 0.5-18 mol $\cdot L^{-1}$ was investigated. At concentrations up to 4 mol $\cdot L^{-1}$, the reaction was first order with respect to hydrogen peroxide; at concentrations above 4 mol$\cdot$ L^{-1}, the reaction order is higher. The reaction rate is proportional to the square root of the radiation intensity; the activation energy is 5.1 kcal$\cdot$mol^{-1}.

In the radioactive decomposition of H_2O_2, ions play a certain role. Based on this, the following process scheme was proposed;

$$H_2O_2 \rightarrow 2\dot{O}H$$

$$\dot{O}H + H_2O_2 \rightarrow H_2O + H\dot{O}_2$$

$$H\dot{O}_2 + H_2O \rightarrow H_3O^+ + O_2^-$$

$$H_2O_2 + O_2^- \rightarrow HO^- + \dot{O}H + O_2$$

$$H_2O_2 + H\dot{O}_2 \rightarrow H_2O + O_2 + \dot{O}H$$

$$\dot{O}H + H\dot{O}_2 \rightarrow H_2O + O_2$$

Since the radical is inactive, the reaction should proceed relatively slowly. The reaction processes should proceed quickly, since the transitions of the proton and the electron proceed without a significant energy barrier.

3. Results and discussion

The paper presents the results of a study of changes in the electrophysical properties of preliminarily radiation-oxidatively treated zirconium samples as a result of their testing during

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thermo- and thermoradiolytic processes of water decomposition. At the same time, special attention is paid to the following aspects;

- ✓ During the operation of radiation-oxidatively treated zirconium samples under real operating conditions of water-cooled nuclear reactors, all parameters of these samples change, including surface physicochemical parameters, corrosion resistance, and electrical properties. A special place in the change of these properties belongs to the processes of defect formation in the Zr-ZrO_x system.
  The study of changes in the electrophysical properties of pretreated zirconium samples allows us to judge the mechanism of defect formation and, ultimately, about all radiation-heterogeneous processes in the Zr-ZrO_x-H_2O system. Therefore, we have carried out studies of changes in resistivity-, thermo emf-of preliminarily radiation-oxidatively treated zirconium samples after testing them in contact with water at T = 673K, at various absorbed doses.
- ✓ The current-voltage characteristics of these samples were studied in order to clarify the state and quantitative features of charge carriers formed in the Zr-ZrO_2 system under the influence of radiation-heterogeneous processes in contact with water.

Based on the experimental results, generalizations are made about the mechanism of defect formation and their effect on the electrophysical properties of the Zr-ZrO_2 system.

When irradiated with γ-quanta, which create damage in metals and alloys, complex processes occur that lead to the formation of defect structures in the form of electronic and lattice defects, pores, precipitates, etc. This is the reason for the application of the method of resistivity and thermoelectric power (Agayev, Imanova & Imran, 2021; Gunel & Bekpulatov, 2021; Sami & Imanova, 2022; Imran & Imanova, 2022). Publications in recent years indicate an increased attention to this method, due to its high sensitivity to smaller defect structures and in solving many problems in damage physics and radiation materials science.

In the present work, are investigated the possibilities of methods for studying the resistivity ($\Delta\rho / \Delta\rho 0$) and thermo-emf ($\alpha$) in the study of the processes of radiation oxidation of the surface of zirconium in contact with H_2O_2. During the radiation-oxidative treatment of metals, an oxide phase is formed on the surface, which affects the resistivity of materials. Therefore, electrophysical methods began to be widely used as a method for obtaining information on the state of structural materials in nuclear technologies.

Figure 1 shows the dependences of the resistivity of preliminarily radiation-oxidatively treated samples on the time of γ-irradiation at. As can be seen from the figure, at low values of the absorbed dose D≤20kGy, the resistivity of the samples decreases in comparison with the initial state. The observed decrease in the resistivity in the initial regions of the time of radiation-

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oxidative treatment of zirconium is associated with surface radiation-heterogeneous processes. In the initial doses of radiation-oxidative treatment of zirconium, there is an accumulation of defect states in the surface oxide phase.

When irradiated with γ-quanta, which create damage in metals and alloys, complex processes occur that lead to the formation of defect structures in the form of electronic and lattice defects, pores, precipitates, etc. This is the reason for the application of the method of resistivity and thermo-emf (Barkaoui et al., 2022). Publications in recent years indicate an increased attention to this method, due to its high sensitivity to smaller defect structures and in solving many problems in damage physics and radiation materials science.

In the present work, the possibilities of methods for studying the resistivity ($\Delta\rho$ / $\Delta\rho 0$) and thermo-emf are investigated. In the study of the processes of radiation oxidation of the surface of zirconium in contact with H_2O_2. During the radiation-oxidative treatment of metals, an oxide phase is formed on the surface, which affects the resistivity of materials. Therefore, electrophysical methods began to be widely used as a method of obtaining information on the state of structural materials of nuclear technologies.

Figure 1 shows the dependences of the resistivity of preliminarily radiation-oxidatively treated samples on the time of γ-irradiation at. As can be seen from the figure, at small values of the absorbed dose D≤20kGy, the resistivity of the samples decreases compared to the initial state. The observed decrease in the resistivity in the initial regions of the time of radiation-oxidative treatment of zirconium is associated with surface radiation-heterogeneous processes. In the initial doses of radiation-oxidative treatment of zirconium, there is an accumulation of defect states in the surface oxide phase:

$$Zr\text{-}ZrO_x \rightarrow Zr \cdot ZrO^*$$

where - Zr-ZrOx is the initial state of the surface with a protective oxide phase, $Zr \cdot ZrO^*$ is the defect state formed as a result of the action of γ-quanta (the defect state can be attributed to the localized state of nonequilibrium charge carriers, vacancies of anions, surface O- holes) .

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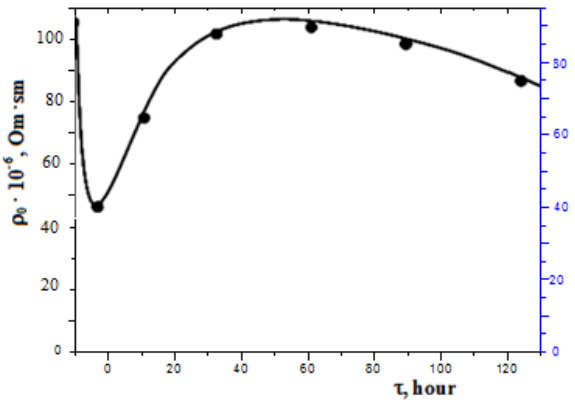

Figure 1. Dependence of the resistivity of zirconium samples on the time of preliminary irradiation at T = 300K, D=1.14 Gy/s in an H_2O_2 medium.

As a result of the interaction of surface defect states with the products of H_2O_2 radiolysis, hydroxyl-containing states of zirconium atoms are formed and dissolved in the contacting medium. Partial destruction of the biographical oxide film and the formation of charged states lead to a decrease in the resistivity of the metal. With a further increase in the time of radiation-oxidative treatment, an oxide film is formed. As a result of the interaction of charged and coordination-unsaturated metal atoms by the products of H_2O_2 radiolysis, a new oxygen-containing state of the zirconium surface is formed.

After D≥123 kGy, the predominance of the formation of the oxide phase begins during radiation-heterogeneous processes in the Zr-H_2O_2 system, therefore, the resistivity of the radiation-oxidatively treated samples begins to increase.

The obtained results show that a stable protective oxide film can be formed in a certain amount on the zirconium surface. Its properties depend on the methods of its formation. It was revealed that a stable state of a protective oxide film is formed on the surface of zirconium during radiation-oxidative treatment. The stability of the surface oxide state remains in the range of values of the absorbed radiation dose D≈123 ÷ 290 kGy. The observed decrease in the resistivity of the samples preliminarily radiation-oxidatively treated in an H_2O_2 medium in the region of the absorbed radiation dose D≥290 kGy is apparently associated with the accumulation of stable charged states in the Zr-ZrOx system.

In order to elucidate the nature of the dependence of the resistivity ρ(τ) on the preliminary oxidative treatment of the metal surface, the relative change in the resistance and thermo emf of the samples preliminarily radiation-oxidatively treated at times corresponding to a minimum (5 hours) was studied.

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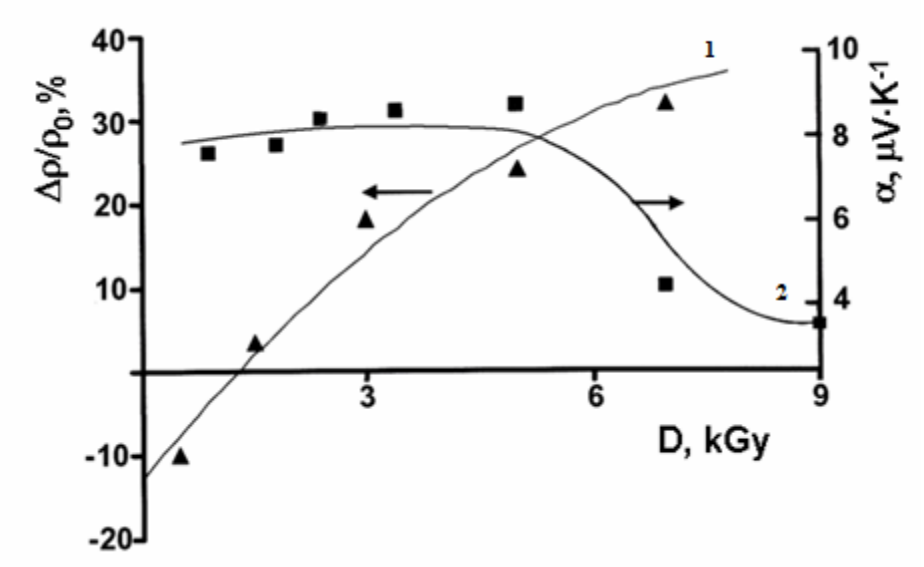

Figure. 2. Dependence of Δρ / Δρ0 = f (D) and α = f (D) of metallic zirconium (Zr) on the absorbed dose.

Figure 2. (curve 1) shows the dependences of the relative change in resistance Δρ / Δρ0 and thermo-emf (α) of radiation-thermally tested plates on the absorbed radiation dose. It can be seen from the figure that $\Delta\rho / \Delta\rho_0$ initially decreases and at low doses increases intensively, and at relatively large doses (D≥3.5 kGy) the growth of $\Delta\rho / \Delta\rho_0$ weakens and slowly approaches saturation.

As a result of the interaction, Zr-ZrO_2 appears, the localization of which proceeds until the surface is saturated with them. The relative resistance in the samples after the radiation-heat treatment slowly increases (Fig. 2, curve 1), and the value of the thermo-emf (curve 2) decreases. As is known, the thermo emf (α) is very sensitive to changes in the Fermi properties of the metal surface and therefore, by measuring the thermo emf of the samples, we obtain additional information on the localization of charge carriers and the defectiveness of the tested samples. With an increase in the absorbed dose D>50 kGy, the value of thermoelectric power decreases, which directly confirms an increase in the concentration of emitted charge carriers.

The article presents the results of experimental studies by measuring the current-voltage characteristics (CVC) of zirconium plates, pre-irradiated in an H_2O_2 medium at various doses, and then tested in the process of radiation-thermal and thermal decomposition of water at T=673K, $\rho_{H_2O} \approx 5mG/sm^3$, $\dot{D} = 1.14Gy/s$.

As is known, the appearance on the metal surface of various point defects, vacancies and surface oxidation processes strongly affect the metal conductivity. To identify certain factors

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affecting the conductivity of metallic zirconium, we studied the current-voltage characteristics (CVC) and changes in the current density of thin zirconium plates 80-200 μm thick, depending on the absorbed dose of gamma quanta.

Figure 3 (τ=5 hour) shows the CVC characteristics of the initial (curve a), pre-irradiated (curve b) in an H_2O_2 medium at T=300K, $\tau = 5$hour., $\dot{D} = 1.14 Gy/s$. samples of zirconium plates and CVC characteristics of the same samples tested in the process of thermal (curve c) and radiation-thermal (curve d) decomposition of water at T=673K, τ= 30 min, $\rho_{H_2O} \approx 5 mG/sm^3$.

The figure shows that at the same electric voltage (for example, at $U=8\cdot10^{-5}$ V/sm), the current increases by

$$\frac{J_{pr.}-J_0}{J_0}=\frac{9-3{,}4}{3{,}4}=1{,}45;\ \frac{J_T-J_{0..}}{J_0}=\frac{7-3{,}4}{3{,}4}=1{,}06 \text{ and } \frac{J_{RT}-J_{0.}}{J_0}=0{,}15.$$

where: J_0 - is the current strength of the original samples, $J_{pr.}$ - current strength of pre-irradiated samples, J_T and J_{RT} - current strength of the same samples thermally and radiation-thermally tested in the process of water decomposition. As can be seen from the figures, to obtain the same current strength, for example $J = 4\cdot10^{-3}$A, the following electric voltages will be required accordingly: for the initial sample $10.5\cdot10^{-5}$ V, for the preliminary radiation-oxidative treated sample (curve b) - 3, $5\cdot10^{-5}$ V, i.e. almost three times less, for a preliminary radiation-oxidatively treated sample tested at thermal (curve c) $9.5\cdot10^{-5}$ V and radiation-thermal U = $4.5\cdot10^{-5}$ In the processes of water decomposition, i.e. approximately 2.3 times less.

Hence, it can be seen that when measuring a preliminarily radiation-oxidatively treated zirconium sample and the same sample after testing in the process of radiation-thermal decomposition of water to obtain the same current, the applied electric voltages decrease by almost 3 and 2.3 times, respectively. This is due to an increase in the concentration of current carriers.

Figure 3 (τ=15 hour) shows the I - V characteristics of the same conditions at the time of preliminary irradiation with gamma quanta $\tau_{irra.}$=15 hours, where it is shown that there is a change in the current strength in the same value of the electric voltage ($U=8\cdot10^{-5}$ V/sm). As can be seen after thermal and radiation-thermal tests, the J of the samples increases during the decomposition of water. It can be seen from the figure that the properties of the CVC characteristics of preliminarily radiation-oxidatively treated samples strongly depend on the absorbed dose of γ-quantum, since in the sample irradiated during τ= 15 hours at the same electrical voltage (eg $U=8\cdot10^{-5}$V) J - the current decreases $\frac{(J)_{\tau=5}}{(J)_{\tau=15}}=\frac{9\cdot10^{-5}}{3{,}5\cdot10^{-5}}\approx 2{,}5$ times . In the

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samples tested in radiation-thermal processes, J (U) changes in the same way as in the samples pre-irradiated during τ = 15 hours.

From Figure 3 (τ=30 hour) where the CVC characteristics of the above processes are shown: a - initial samples; b - pre-irradiated in an H_2O_2 environment; c - thermally and d - radiation-thermally tested samples, it can be seen that the current strength of the samples after preliminary irradiation in an H_2O_2 medium at an irradiation time $\tau_{irra.}$=30 h., increased by 14.7%, and after the thermal test by 29.4% and, accordingly, after the radiation-thermal test, it doubled.

With a further increase in the pre-irradiation time, similar results were obtained.

Figure 3 (τ=50 hour) also shows the results of studies of the CVC characteristic in the same sequence of zirconium samples preliminarily radiation-oxidatively treated (curve b) in an H_2O_2 medium during sample. τ=50 hours and the same samples tested in the processes of thermal (curve c) and radiation-thermal (curve d) decomposition of water (T = 673K, τ= 30 min, $\rho_{H_2O} \approx 5 mG/sm^3$).

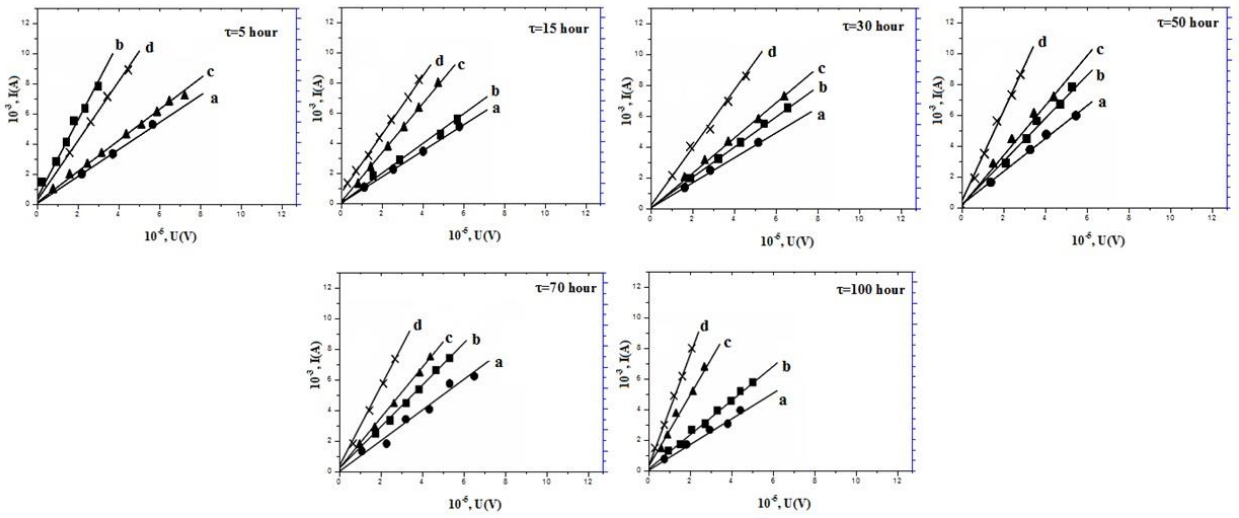

Figure 3. Current-voltage characteristic of zirconium samples: τ=5, 10, 30, 50, 70 and 100 hours, $\dot{D} = 1.14 Gy/s$

a- original sample

b- preliminarily irradiated sample in H_2O_2 medium at T=300K

c- thermally and

d-radiation-thermally tested samples after preliminary irradiation in H_2O_2 environment.

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It can be seen from the figure that at the same electric voltage, the current strength of the samples tested in thermal and radiation-thermal processes, respectively, increases by $2.1 \cdot 10^{-2}$ and $U=6.6 \cdot 10^{-2}$ A, i.e. increases approx. 1.7 and 2.2 times.

Figure 3 (τ=70 hour) shows the CV characteristics of the initial (a) samples and the CV characteristics of the same samples tested in the process of thermal (c) and radiation-thermal (d) decomposition of water after their preliminary irradiation in an H_2O_2 medium at T=300K for τ= 70 hours. As can be seen from the figure, after preliminary irradiation, the current increased by 8.94%. After heat treatment of the same samples in the process of water decomposition, the current increased by 26.5% (Fig. 3 (τ=70 hour), curve c), and after the radiation-thermal process, it increased by 1.5 times (curve d), i.e. the current strength increased by the same percentage.

Figure 3 (τ=100 hour) also shows the CV characteristics of the initial (a) samples and the CV characteristics of the same samples tested in the process of thermal (c) and radiation-thermal (d) decomposition of water (T=673K, $\rho_{H_2O} \approx 5 mG/sm^3$, $\dot{D} = 1.14 Gy/s$) after their preliminary irradiation in an H_2O_2 medium at T=300K for τ=100 hours. As can be seen from the figure, under these conditions, the CV characteristics of the samples are very different from the previous ones. It was revealed that the current strength of the thermally treated samples increased by ≈35.5% and in the samples tested during radiation-thermal processes almost 3 times.

Comparison of the results of the study $\sigma=f(D_{irra.})$ and the current-voltage characteristic shows that there are satisfactory coincidences between them. Since during radiation-oxidative treatment at low values of the irradiation dose $D \leq$ 20-25 kGy, charged states are formed and the samples have a relatively high electrical conductivity. These samples are characterized by a relatively high concentration of charge carriers. Therefore, at the same voltage values, the values of the current strength of the samples preliminarily radiation-oxidatively treated at $D \leq$20-25 kGy are higher than others. With a further increase in the absorbed radiation dose of radiation-oxidative treatment, a protective oxide film is formed and therefore the current strength of the samples processed at the values of the radiation dose $D \geq$30 kGy is less than that of the samples tested during thermal and radiation-thermal processes of water decomposition (Fig. 3).

After testing the zirconium samples preliminarily radiation-oxidatively treated at $D \geq$25 kGy in the processes of radiation-thermal and thermal decomposition of water, the protective oxide film is partially destroyed and, as a result, charged states are formed, which cause an increase in the current strength. The concentration of charge carriers in the samples tested during the radiation-thermal process of water decomposition is higher than that in the samples tested during the thermal process of water decomposition. Therefore, the current strength of these

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samples in all ranges of values of the radiation dose is greater than that of the original, radiation-oxidative treated and samples tested during thermal processes of water decomposition.

4. Conclusions

The completion of the protective oxide film during the radiation-oxidative treatment of zirconium (D≥80 kGy) is accompanied by a decrease in electrical conductivity and current values in the study of their current-voltage characteristics. The rate of destruction of the surface oxide film and the accumulation of charge carriers during radiation-thermal processes are higher than during thermal processes. Analyzing the results of studies of the electrophysical properties of the initial, radiation-oxidatively treated and tested during radiation-thermal and thermal processes of water decomposition of zirconium samples, the following conclusion can be drawn. When these samples are tested in the processes of radiation-thermal and thermal decomposition of water, a partial destruction of the protective oxide film occurs as a result of which additional charge carriers accumulate on the surface. The rate of destruction of the surface oxide film and the accumulation of charge carriers during radiation-thermal processes are higher than during thermal processes.

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