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## INFLUENCE OF $\text{TiOSO}_4$ ON THE KINETICS OF FORMATION AND PROTECTIVE PROPERTIES OF OXIDE COATINGS ON AISI 304 STEEL

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**ABSTRACT** The influence of titanium thiosulfate, introduced into an aqueous electrolyte, on the features of electrochemical formation of oxide coatings on AISI 304 stainless steel has been studied, with a focus on changes in anodic process kinetics, electrochemical passivation parameters, and the protective properties of the resulting layer. The changes in the transition potential to the passive state, the width of the passive region, and the passivation current density depending on the titanium concentration in the electrolyte were analyzed. It was shown that increasing the content of titanium thiosulfate in the solution promotes a decrease in the passivation onset potential and the formation of a denser protective layer, as evidenced by an increase in polarization resistance and a decrease in anodic current density. Emphasis was placed on the optimal concentration range of  $\text{TiOSO}_4$  (20 g/L), which ensures maximum efficiency in the formation of the passive layer. Results of potentiodynamic polarization studies in a 3% sodium chloride solution were analyzed, indicating a significant influence of the titanium-containing component on the electrochemical behavior of the steel. It was shown that samples with oxide coatings formed in modified electrolytes are characterized by a shift of the corrosion potential toward more electropositive values by 500–800 mV compared to untreated steel. It was confirmed that the obtained coatings effectively reduce the electrochemical activity of the metal in a chloride environment, as evidenced by a significant reduction in corrosion current density and an increase in polarization resistance. The results of the study highlight the potential of controlled electrolyte modification for the electrochemical formation of stable oxide layers with enhanced corrosion resistance, which is important for the protection of steel structures, particularly under high-temperature conditions and exposure to aggressive environments. This research lays the foundation for further improvement of electrolyte compositions and anodizing regimes for steels, aimed at creating stable protective films for operation in aggressive environments without the need for complex surface post-treatment.

**Keywords:** anodizing of steel; titanium thiosulfate; polarization behavior; passivation; chronopotentiometry; corrosion resistance; oxide coating

## ВПЛИВ $\text{TiOSO}_4$ НА КІНЕТИКУ ФОРМУВАННЯ ТА ЗАХИСНІ ВЛАСТИВОСТІ ОКСИДНИХ ПОКРИТТІВ НА СТАЛІ AISI 304

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**АНОТАЦІЯ** Розглянуто вплив тіосульфату титану, введеного до складу водного електроліту, на особливості електрохімічного формування оксидних покриттів на нержавіючій сталі марки AISI 304, з акцентом на зміну кінетики анодних процесів, електрохімічних параметрів пасивації та захисних властивостей сформованого шару. Проаналізовано зміну потенціалу переходу в пасивний стан, ширини пасивної області та густини струму пасивації залежно від концентрації титану в електроліті. Показано, що підвищення вмісту тіосульфату титану у розчині сприяє зниженню потенціалу початку пасивації та формуванню щільнішого захисного шару, що проявляється у зростанні поляризаційного опору та зниженні густини анодного струму. Акцентовано увагу на оптимальному діапазоні концентрацій  $\text{TiOSO}_4$  (20 г/л), за якого забезпечується максимальна ефективність формування пасивного шару. Проаналізовано результати потенціодинамічних поляризаційних досліджень у 3%-му розчині натрій хлориду, що вказують на суттєвий вплив титановмісного компонента на електрохімічну поведінку сталі. Показано, що зразки з оксидними покриттями, сформованими у модифікованих електролітах, характеризуються зсувом корозійного потенціалу в область більш електропозитивних значень на 500–800 мВ порівняно з необробленою сталлю. Підтверджено, що одержані покриття ефективно знижують електрохімічну активність металу в хлоридному середовищі, що проявляється в істотному зменшенні густини корозійного струму та збільшенні поляризаційного опору. Результати дослідження розкривають перспективність керованого модифікування електролітів для електрохімічного формування стабільних оксидних шарів з підвищеною корозійною стійкістю, що є важливим для захисту сталевих конструкцій, зокрема в умовах високих температур і дії агресивних середовищ. Дані дослідження є основою для подальшого удосконалення складу електролітів і режимів анодування сталей із метою створення стабільних захисних плівок для роботи в умовах агресивного середовища без потреби у складному післяобробленні поверхні.

**Ключові слова:** анодування сталі; тіосульфат титану; поляризаційна поведінка; пасивація; хронопотенціометрія; корозійна стійкість; оксидне покриття

### Introduction

Steel structures operating under nuclear power conditions, particularly in the coolant environment of

VVER-1000 reactors, are subjected to intense exposure to high temperatures and aggressive media, necessitating highly effective corrosion protection. One of the

promising solutions is the formation of oxide coatings with predetermined functional properties on the steel surface through electrochemical deposition [1–3].

Currently, particular attention is being paid to the modification of electrolytes through the introduction of metal cations capable of stabilizing the coating structure and enhancing its thermal and chemical resistance [4]. Recent studies demonstrate the possibility of forming self-organized porous oxides on the surface of stainless steel through anodizing in organic and hybrid media, particularly with the involvement of glycerol, ethylene glycol, and ammonium fluorides [5–7]. It has been established that the morphology and corrosion behavior of such coatings significantly depend on the electrolyte composition, particularly the content of water, fluoride ions, and metal cations. The work by Wang et al. [7] showed that changing the concentrations of  $\text{NH}_4\text{F}$  and  $\text{H}_2\text{O}$  allows control over the thickness and uniformity of the oxide layer on AISI 304, while studies [8,9] confirm that anodized porous films on AISI 316L may demonstrate satisfactory corrosion resistance only after additional heat treatment aimed at removing residual fluorides.

Of particular interest is the use of valve metal cations such as titanium, zirconium, and aluminum in electrolytes, which are capable of forming a mixed oxide phase with enhanced stability. For instance, the incorporation of  $\text{TiO}_2$  nanoparticles into Ni–Pd coatings contributes to improved corrosion resistance [10,11], while the addition of Zr(IV) compounds enables the formation of reinforced, chemically resistant films on steel substrates [9]. It has also been shown that using aqueous-ethanolic or organic electrolytes with fluorine-containing components allows the creation of controlled structures based on metal oxides; however, this approach requires further refinement in terms of reducing toxicity and environmental impact.

Against the backdrop of recent advancements, the use of titanium thiosulfate ( $\text{TiOSO}_4$ ) as an inorganic electrolyte modifier for forming oxide films on steel appears promising. However, the influence of this compound's concentration on the anodic process kinetics and the protective properties of the resulting coatings remains insufficiently studied, which defines the scientific novelty and practical significance of the proposed research.

#### Purpose of work

The aim of this work is to study the effect of  $\text{TiOSO}_4$  concentration in the electrolyte on the kinetic patterns of metal-oxide coating formation on steel and to determine their protective properties using electrochemical analysis.

#### Presentation of the main material

The formation of metal-oxide coatings on AISI 304 steel was carried out in galvanostatic mode using

electrolytes containing sulfuric acid (300 g/L), sodium chloride (50 g/L), and various concentrations of titanium thiosulfate ( $\text{TiOSO}_4$ ) at 0, 5, 10, 15, and 20 g/L. A steel plate with a surface area of  $4 \text{ cm}^2$  served as the working electrode, and a platinum wire was used as the counter electrode. Electrolysis was conducted in a 200 mL volume with continuous stirring. The oxidized samples were rinsed with distilled water and air-dried [12].

The anodic behavior of the steel and the corrosion resistance of both the steel and the oxide coatings were studied in a three-electrode electrochemical cell using an IPC-Pro potentiostat-galvanostat. Steel samples with and without metal-oxide coatings, each with a surface area of  $1 \text{ cm}^2$ , were used as the working electrode. The potential of the tested steel electrode was measured relative to a silver/silver chloride reference electrode (EVL-1M1). The auxiliary electrode was platinum. The potential scan rate was  $2 \text{ mV/s}$ . Experiments were conducted under natural convection at an electrolyte temperature of  $25 \text{ }^\circ\text{C}$  [13].

The polarization curves (Fig. 1) show regions of active dissolution, active-passive transition, and oxygen evolution. It is shown that the addition of titanium compounds shifts the potential  $E_j=0$  toward more electropositive values, and the current values in the passive region are significantly higher than those in the solution of sulfuric acid and sodium chloride. This is likely due to structural changes in the coating during formation and the active influence of oxygen-containing components in the electrolytes.

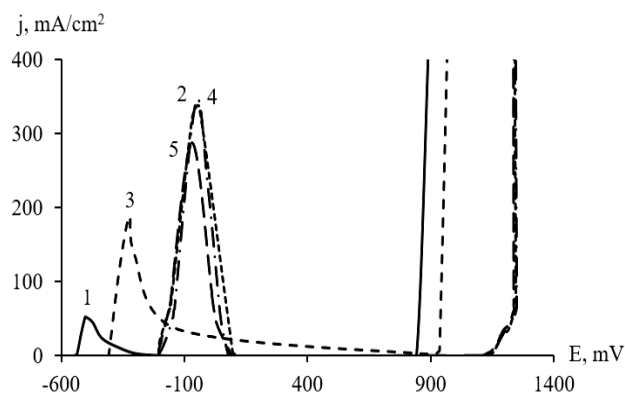


Fig. 1 – Anodic voltammograms of AISI 304 steel in solutions containing 300 g/L  $\text{H}_2\text{SO}_4$  and 50 g/L  $\text{NaCl}$  with the addition of  $\text{TiOSO}_4$ , g/L: 0 (1); 5 (2); 10 (3); 15 (4); 20 (5)

With an increase in the concentration of titanium compounds in the sulfate-chloride electrolyte from 5 to 10 g/L, the passivation potential shifts in the electronegative direction, indicating that the transition of the metal to the passive state becomes easier. However, the addition of  $\text{TiOSO}_4$  at concentrations of 5, 15, and 20 g/L leads to an expansion of the passive region, and the onset of oxygen evolution occurs at 1135 mV (Table 1), which significantly affects the properties of the oxide layers.

Table 1 – Electrochemical parameters of the anodic behavior of steel in titanium-containing electrolytes

Electrolyte composition, g/L	Electrochemical parameters					
	$E_{j=0}$ , mV	$E_p$ , mV	$j_p$ , mA·cm <sup>-2</sup>	$E_{pp}$ , mV	$j_{pp}$ , mA·cm <sup>-2</sup>	$E_{O_2}$ , mV
H <sub>2</sub> SO <sub>4</sub> – 300, NaCl – 50	-550	-501	52,3	-286	0,1	841
H <sub>2</sub> SO <sub>4</sub> – 300, NaCl – 50, TiOSO <sub>4</sub> – 5	-203	-68	335,7	87	3,3	1135
H <sub>2</sub> SO <sub>4</sub> – 300, NaCl – 50, TiOSO <sub>4</sub> – 10	-407	-364	118,6	-130	35,9	920
H <sub>2</sub> SO <sub>4</sub> – 300, NaCl – 50, TiOSO <sub>4</sub> – 15	-203	-55	342,7	61	3,13	1135
H <sub>2</sub> SO <sub>4</sub> – 300, NaCl – 50, TiOSO <sub>4</sub> – 20	-203	-40	288,1	70	3,11	1135

The change in the potential of the steel electrode over time in titanium-containing electrolytes is shown in Fig. 2.

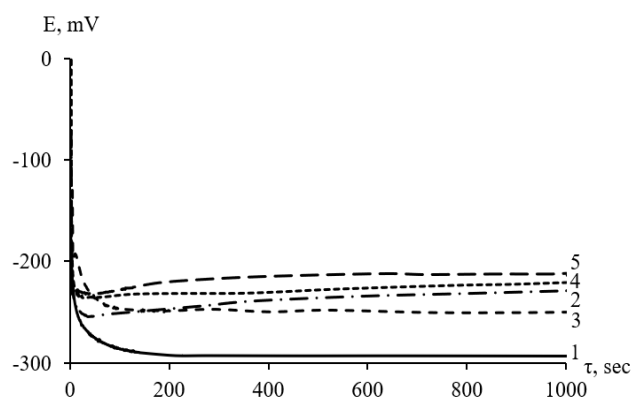


Fig. 2 – Chronopotentiograms of AISI 304 steel in solutions containing 300 g/L H<sub>2</sub>SO<sub>4</sub> and 50 g/L NaCl with the addition of TiOSO<sub>4</sub>, g/L: 0 (1); 5 (2); 10 (3); 15 (4); 20 (5)

The change in the steady-state potential depends on the concentration of titanium compounds and shifts it in the more electropositive direction.

Corrosion tests of the formed coatings were conducted in a 3% sodium chloride solution. Polarization of the working electrode was carried out within ±100 mV from the steady-state potential after being held in the solution for 1 hour. The potential scan rate was 10 mV/s.

From the polarization curves (Fig. 3), it can be concluded that the addition of a titanium-containing compound to the coating composition results in a shift of the potential toward more electropositive values by 500–800 mV and a reduction in the corrosion current.

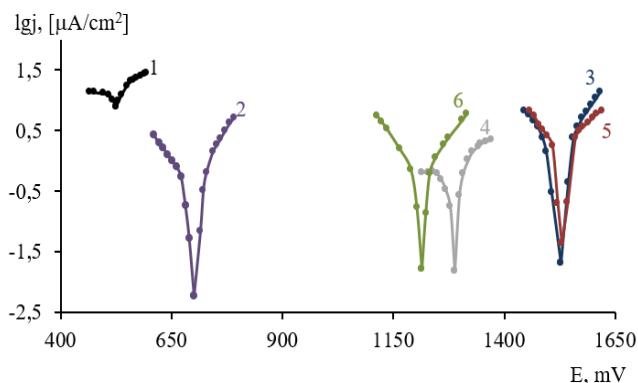


Fig. 3 – Polarization curves in 3% NaCl solution for AISI 304 steel (1) and metal-oxide coatings obtained in titanium-containing electrolytes (Table 2): No.1 (2), No.2 (3), No.3 (4), No.4 (5), No.5 (6)

Based on the experimental data (Table 2), it was determined that increasing the concentration of titanium thiosulfate enhances the polarization resistance of the coating, thereby providing the metal-oxide coatings with improved protective properties in a chloride environment.

Table 2 – Corrosion characteristics of AISI 304 steel with oxide coatings formed in titanium-containing solutions

Electrolyte composition, g/L	$R_p$ , kΩ·cm <sup>2</sup>	$b_k$ , mV	$b_a$ , mV	$B$ , mV	$k_j$ , A/m <sup>2</sup>
Unoxidized steel	3,0	84	91	19	1,24·10 <sup>-6</sup>
H <sub>2</sub> SO <sub>4</sub> – 300, NaCl – 50	28,0	91	80	18	1,33·10 <sup>-7</sup>
H <sub>2</sub> SO <sub>4</sub> – 300, NaCl – 50, TiOSO <sub>4</sub> – 5	45,6	74	82	17	7,45·10 <sup>-8</sup>
H <sub>2</sub> SO <sub>4</sub> – 300, NaCl – 50, TiOSO <sub>4</sub> – 10	50,6	68	28	8	3,4·10 <sup>-8</sup>
H <sub>2</sub> SO <sub>4</sub> – 300, NaCl – 50, TiOSO <sub>4</sub> – 15	68,8	89	58	15	4,44·10 <sup>-8</sup>
H <sub>2</sub> SO <sub>4</sub> – 300, NaCl – 50, TiOSO <sub>4</sub> – 20	72,2	86	88	18	5,25·10 <sup>-8</sup>

### Discussion of results

Analysis of the anodic polarization curves indicates a significant influence of titanium thiosulfate on the mechanism of passive layer formation on AISI 304 steel. The shift of the passivation onset potential in the electronegative direction at a TiOSO<sub>4</sub> concentration of 10 g/L suggests a reduction in the energy barrier for oxide layer formation, which

may be associated with the catalytic effect of titanium-containing cations on the oxidation processes of iron and chromium.

Further increase in concentration to 15–20 g/L leads to an expansion of the passive region and a rise in the oxygen evolution potential, which is likely due to the formation of a denser and electrochemically more stable coating that more effectively blocks the access of aggressive ions to the metal substrate. The increase in polarization resistance of the coatings to 72.2 kΩ·cm<sup>2</sup> at the maximum TiOSO<sub>4</sub> concentration confirms the enhanced barrier effect of the formed layer.

The nearly one-order-of-magnitude reduction in corrosion current density indicates the effective inhibiting action of the titanium-containing phase within the coating, which aligns with current understanding of the ability of oxygen-containing Ti(IV) compounds to stabilize the film structure through the formation of mixed-type oxides that exhibit high electrical resistance and chemical inertness.

A change in the nature of cathodic and anodic processes in 3% NaCl solution was also observed, where oxidized samples exhibit a shift of the corrosion potential toward more positive values, indicating a decrease in the thermodynamic probability of corrosion dissolution. The lowest value of  $k_j = 3.4 \cdot 10^{-8}$  A/m<sup>2</sup> was obtained at a TiOSO<sub>4</sub> concentration of 10 g/L, which correlates with the maximum reduction in corrosion activity in the presence of a stabilized Ti phase.

Thus, the results indicate that the optimal concentration of TiOSO<sub>4</sub> in the electrolyte is 20 g/L, which ensures effective steel passivation, the formation of a dense metal-oxide layer, and a reduction in the corrosion dissolution rate in aggressive environments.

### Conclusions

Based on the results of the conducted research, the influence of titanium thiosulfate concentration in the electrolyte on the electrochemical patterns of oxide coating formation on AISI 304 steel was established. It was shown that the addition of TiOSO<sub>4</sub> promotes a decrease in the passivation potential and the formation of a protective layer with increased polarization resistance. The optimal concentration of TiOSO<sub>4</sub> in the electrolyte is 20 g/L, which ensures the greatest reduction in corrosion current density and expansion of the passive region. It was found that modification of the electrolyte with titanium thiosulfate significantly improves the protective properties of the coatings in chloride-containing environments.

The obtained results indicate the potential of using titanium-containing components for the controlled formation of functional metal-oxide coatings on stainless steel in order to enhance its operational durability under aggressive conditions.

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