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Patterns and Mechanisms of Pitting of Heat Exchangers Made of AISI 321 and 08Kh18N10T Steels During their Operation in Recycled Water

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Abstract

The article examines the patterns and mechanisms of pitting corrosion in AISI 321 and 08Kh18N10T stainless steels under conditions simulating recycled waters used in heat exchanger operation. It was established that the critical pitting temperature (CPT) of these steels increases significantly with increasing pH and decreasing chloride concentration in model solutions. The influence of the structural components of the steels was found to be less significant than that of the chloride-containing environment and to depend strongly on its parameters (pH and chloride concentration). In acidic model recycled waters (pH 4), AISI 304 steels – which contains minimal amounts of titanium nitrides, aluminum oxides, and carbon – exhibits the highest CPT values, regardless of the chloride concentration in the range of 350–600 mg/L. In contrast, AISI 321 and 08Kh18N10T steels, characterized by the highest volume fractions of titanium nitrides, as well as melt 2 of AISI 321 steel and 12Kh18N10T steel with elevated carbon content, show the lowest CPT values. Based on an analysis of the literature, it is hypothesized that this behavior may be attributed to the increased overvoltage of hydrogen ionization on titanium nitrides and carbonitrides, which act as initiation sites for pit formation and propagation. Additionally, the corrosion rate was found to correlate with the specific paramagnetic susceptibility χ_0 of austenite, a parameter reflecting the atomic magnetic state and determined by the chemical composition and phase structure of the alloy.

1. Introduction

Stainless steels AISI 321 and 08Kh18N10T are widely used in the manufacture of heat exchangers, which often operate under recycled water conditions. However, the presence of chloride ions and other local corrosion activators can lead to pitting corrosion [1–3]. To mitigate local corrosion of heat-transfer elements, various protective approaches have been proposed, including composite coatings applied by the cut-off method [4, 5], polymer films based on nanomaterials [6], adsorbed layers formed from chromium-containing electrolytes [7, 8], and coat-

ings incorporating refractory chemical elements [6]. Despite these measures, the assessment and prediction of the resistance of these steels to localized corrosion remain important challenges. Recycled water used in industrial facilities represents a low-mineralized, chloride-containing environment; therefore, in [9, 10], the pitting resistance of corrosion-resistant stainless steels and alloys was evaluated using the critical pitting temperature (CPT). It was shown in [9–11] that pitting nucleation and growth on the surface of these steels are associated with inclusions of specific nature and size, and that the CPT decreases markedly with an increasing volume fraction of inclusions involved in pitting. Mathematical models developed in [2, 9, 10, 12–14], based on multifactorial regressions of different orders, showed that pitting resistance depends more strongly on the

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parameters of the medium than on the structural components of the steel. The influence of chromium content within its standard range was found to be only half as significant as the influence of structural components. These mathematical models are used to evaluate and predict the pitting resistance of heat exchangers during operation in recycled water, but they do not clarify how the structural components of AISI 321 and 08Kh18N10T steels, and under what specific conditions, affect their CPT. This limitation may constrain the optimization of technological operating modes. Therefore, the present study investigates the effect of the structural components of AISI 321 and 08Kh18N10T steels on their critical pitting temperature in model recycled water.

2. Materials and methods

Five industrial melts of AISI 321 steel and one melt of 08Kh18N10T steel were investigated. Their

chemical composition (Table 1), structure components (Table 2), and critical pitting temperatures (CPT) in model recycled waters with pH values ranging from 4 to 8 and chloride concentrations of 350, 400, 500, 550, and 600 mg/L were previously determined in [15]. Corrosion losses of the investigated steels due to pitting on their surfaces were determined by the gravimetric method after testing in a 3% FeCl₃ chloride-containing solution [16]. Graphical relationships between CPT and the pH of model recycled waters at chloride concentrations of 350, 400, 500, 550, and 600 mg/L were analyzed, as it has been shown [9–11, 17] that the influence of steel parameters and environmental conditions on pitting resistance increases in the following order: chromium content, structural parameters of the steel, chloride concentration, and pH of the medium. The specific paramagnetic susceptibility of austenite (χ_0) in the investigated steels was determined in [18] and is presented in Table 2.

Table 1. Chemical composition of investigated steels, wt.%

Steel	Melt No.	C	Mn	Si	S	P	Cr	Ni	Ti	N
AISI 321	1	0.035	1.66	0.54	0.001	0.026	17.10	9.10	0.32	0.012
	2	0.060	1.59	0.66	0.002	0.027	16.43	9.14	0.34	0.011
	3	0.064	1.22	0.52	0.001	0.026	17.43	9.70	0.41	0.012
	4	0.030	1.62	0.41	0.002	0.028	17.41	9.24	0.31	0.013
	5	0.040	1.70	0.49	0.001	0.026	17.70	9.10	0.35	0.013
08Kh18N10T	6	0.070	1.70	0.49	0.007	0.027	17.97	10.46	0.46	–

Table 2. Specific paramagnetic susceptibility of austenite (χ_0), content of Ti nitrides and Al oxides, austenite grain size of investigated steels

Steel	Melt No.	χ_0 , 10 ⁻⁸ m ³ /kg	Ti nitrides, vol.%	Al oxides, vol.%	Austenite grain, μ m
AISI 321	1	2.55	0.475	0.0065	312
	2	2.56	0.401	0.0116	240
	3	2.59	0.314	0.0104	278
	4	2.68	0.234	0.0050	211
	5	2.54	0.298	0.0066	196
08Kh18N10T	6	2.52	0.467	0.0027	264

3. Results and Discussion

Analysis of the dependences shown in Fig. 1 demonstrated that, in model recycled waters with a chloride concentration of 350 mg/L, the CPT of AISI 321 steels increases as the pH rises from 4 to 6. At pH 4 and 5, the CPT values of the melts increase in the following order: melts No. 1 → 2 → 3 → 4 → 5.

With a further increase in pH to 6, a more pronounced increase in CPT is observed for melts No. 4 and 5 compared to melts No. 1–3.

This behavior is most likely associated with the lower volume fractions of titanium nitrides (0.234 and 0.298 vol.%), aluminum oxides (0.0065 and 0.005 vol.%), as well as the smaller mean austenite grain diameters (211 and 196 μ m) in these melts (Table 2).

At the same time, it should be noted that these melts also contain the lowest C (0.03 and 0.04 wt.%), Si (0.41 and 0.79 wt.%) and the highest N (0.013 wt.%) and Cr (17.7 wt.%) in melt No. 5 and Ni (9.24 wt.%) in melt No. 4 (Table 1).

Summarizing the above data, it can be noted that the less carbide phase in AISI 321 steel in the vicinity of titanium nitrides where pitting occurs and develops [15], the higher its CPT in model recycled water. It is known [19] that the less C in steels and alloys, the less carbides they contain, and Si accelerates its precipitation from the austenite solid solution [20]. It should be noted that stable pitting occurs in the vicinity of these inclusions, which intersect with the boundaries of austenite grains. It is known [1, 21] that the smaller their mean diameter, the higher the probability of this process and the greater the number of pitting on their surface. This reduces the likelihood of metastable pitting turning into stable pitting due to a decrease in the density of currents in them due to its redistribution among a larger number of metastable pittings [2, 11]. At the same time, Cr increases the protective properties of oxide films against the activating effect of chlorides [12, 22, 23], but, as established in [8, 9, 11, 19, 20], it is half as much on the CPT of stainless steels and alloys as its constituent structures. It should be noted that in model recycled waters with a pH of 4 to 5, the CPT of 08Kh18N10T steel even slightly decreased with increasing pH, while that of five AISI 321 steel melts increased (Fig. 1). Most likely, this is due to the fact that they are alloyed with nitrogen (Table 1), since it is known [23–25] about the positive effect of N on the pitting resistance of austenitic steels in chloride-containing media. Further, we observed an increase in the CPT of melts No. 2–4 of AISI 321 steel and 08Kh18N10T steel and a decrease in melts No. 1 and 5 with an increase in the pH of the model recycled water up to 7 (Fig. 1). This showed that in neutral chloride-containing media, the less carbide phase in steels, the lower their CPT. After all, its decrease was recorded in the melts No. 1; 5 with the lowest C content (Table 1). However, increasing the pH of the model recycled water up to 8 contributed to the growth of the CPT of AISI 321 steels; only in 08Kh18N10T steel not alloyed with N this trend was not observed (Fig. 1). In addition, the increase was most pronounced in melt No. 3 (Fig. 1), which may be attributable to its highest C and Ni contents and lowest Mn content (Table 1).

Analysis of the data presented in Fig. 2 showed that in model recycled waters with a chloride concentration of 400 mg/L, a rapid increase in the CPT with

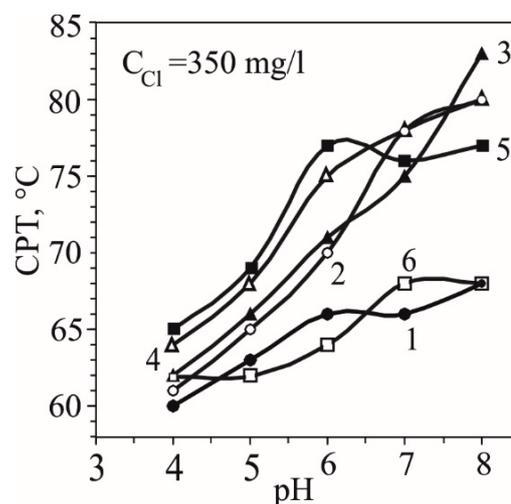


Fig. 1. Dependence of the CPT of melts of AISI 321 (melts No. 1–5) and 08Kh18N10T (melt No. 6) steels on the pH of model recycled water with a chloride concentration of 350 mg/L (numbers at points are melt number).

increasing pH from 4 to 8 was observed only for melt No. 4 (AISI 321 steel). In contrast, under the same conditions at pH 6–7, the CPT values of melts No. 1–3 and 5 remained essentially unchanged, whereas a noticeable increase was observed for 08Kh18N10T steel (Fig. 2). This tendency may be due to the highest value of the specific paramagnetic susceptibility of austenite in melt No. 4 ($\chi_0 = 2.68 \times 10^{-8} \text{ m}^3/\text{kg}$; Table 2), its lowest volume of Al oxides ($V_{\text{ox}} = 0.0050 \text{ vol.}\%$) and the content of C (0.030 wt.%) (Tables 1 and 2) and the absence of N and a smaller volume of Al oxides in 08Kh18N10T steel than in melt 4 (Tables 1 and 2). At the same time, a rapid increase in the latter's CPT in model recycled waters with a chlo-

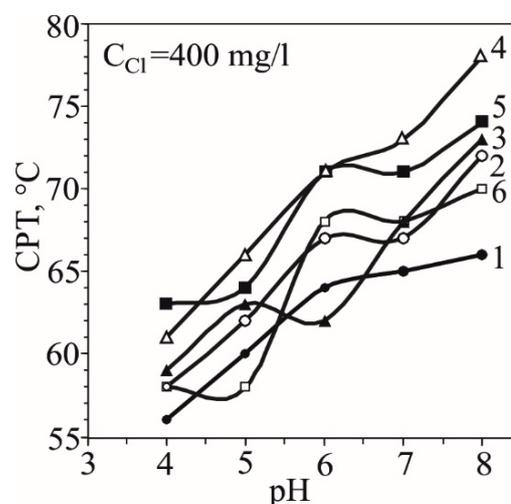


Fig. 2. Dependence of the CPT of 5 melts of AISI 321 (melts No. 1–5) and 08Kh18N10T (melt No. 6) steels on the pH of model recycled water with a chloride concentration of 400 mg/L (numbers at points are melt number).

ride concentration of 400 mg/L and pH from 5 to 6 should be noted (Fig. 2). In addition, at pH 6 to 7, it was even higher than in melts No. 1–3 (Fig. 2). This may indicate that in neutral chloride-containing media with such a chloride concentration, the positive effect of nitrogen on the pitting resistance of AISI 321 steel is somewhat less than in acidic and slightly alkaline model recycled waters.

In general, the established dependencies are consistent with the findings of [24–32], which report an improvement in the pitting resistance of austenitic stainless steels upon nitrogen alloying. This is attributed to an increase in the passivity of the oxide film [22]. The mechanisms proposed by the authors of these papers to improve the passive stability of films as a result of alloying these steels with nitrogen include the formation of ammonium ions [22, 24, 25] or nitrate (nitrite) ions [13, 24, 26, 27], segregation of nitrogen on the surface during anodic dissolution [24, 26–34], and the formation of N bonds on the surface [32], and the authors of [33] believe that the positive effect of nitrogen is due to its influence on the anodic dissolution of steel. It was found in [2, 11, 17, 18] that the pitting resistance of AISI 304 and AISI 321 steels, which was assessed by their CPT, mainly depends on nitrogen dissolved in austenite in solid solution. It was slightly higher in AISI 321 than in AISI 304, but corrosion losses from pitting on its surfaces were higher in AISI 321, which has a nitrogen content more than three times higher than in AISI 304. Therefore, in our opinion, the positive effect of nitrogen on the pitting resistance of these steels is associated with its influence on the anodic dissolution of metals within pits. This process, as established in [16, 18], depends on the specific paramagnetic susceptibili-

ty of austenite (χ_0), which characterizes the atomic magnetic state of the steel [36]. In addition, it was shown in [16, 18] that higher χ_0 values correspond to lower corrosion losses due to pitting in these steels when exposed to chloride-containing media (Fig. 3).

It should be noted that in model recycled water with a chloride concentration of 400 mg/L, an increase in CPT over the pH range of 4–8 was observed only for melt No. 4, which exhibits the maximum value of specific paramagnetic susceptibility, $\chi_0 = 2.68 \times 10^{-8} \text{ m}^3/\text{kg}$. This observation suggests that the paramagnetic state of austenitic steel may exert a non-unambiguous effect on the properties of the oxide film responsible for counteracting the activating influence of chloride ions. Such behavior is most likely related to the semiconducting properties of the oxide film, which depend on numerous factors, primarily the presence of so-called “valve” metals in steels and alloys [37, 38].

In particular, some researchers suggest [17] that oxide films on the surface of stainless steels change from p-type at room temperature to n-type at higher temperatures. The outer layer of the passive film behaves as a p-type semiconductor, whereas the inner layer exhibits n-type conductivity [39]. In [40], a correlation was established between the pitting resistance of stainless steel in a chloride-containing media and the semiconducting properties of the passive film on its surface [16]. When determining the characteristics of passive films by the pitting potential of steel, which depends on temperature, pH, chloride concentration, and other factors [36], it was found that stainless steels with passive films with a closer donor bond are more susceptible to pitting in chloride-containing media [40].

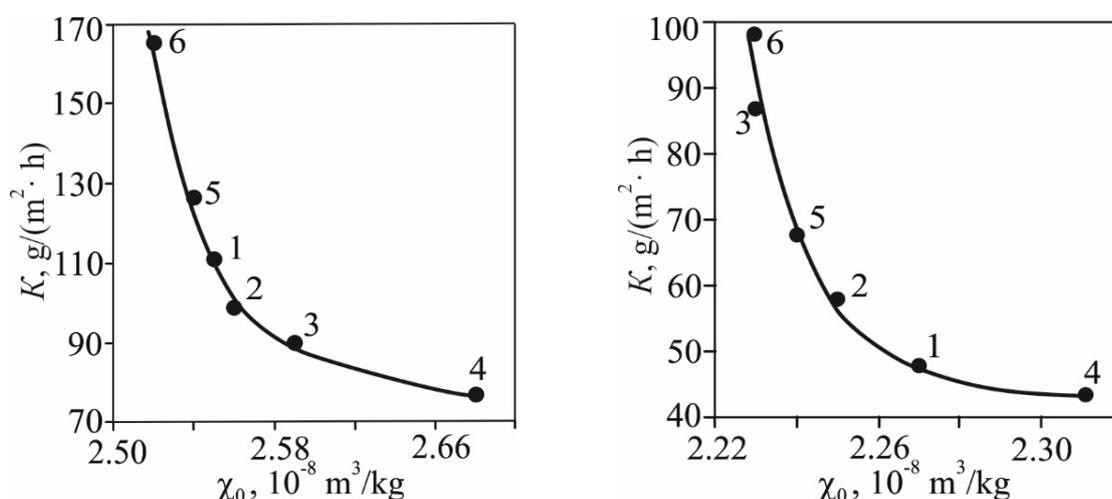


Fig. 3. Dependence of corrosion losses K from pitting on the surface of AISI 321 (a) and AISI 304 (b) steels on its specific paramagnetic susceptibility χ_0 of austenite in a chloride-containing aqueous solution of 3% FeCl_3 (numbers at points are melt number) [18, 35].

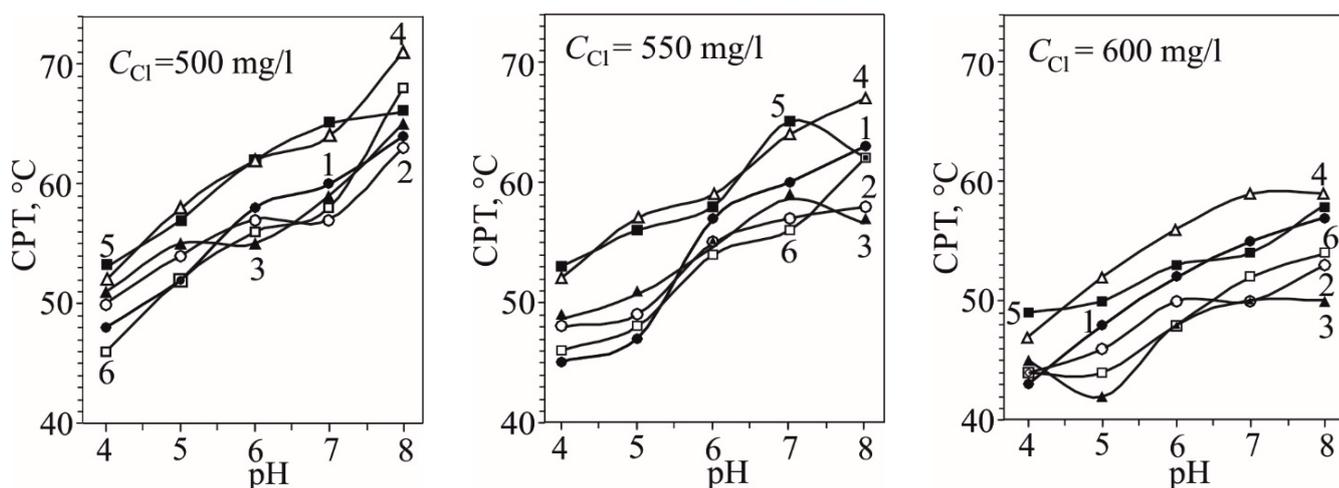


Fig. 4. Dependence of the CPT of 5 melts of AISI 321 (melts 1-5) and 08Kh18N10T (melt 6) steels on the pH of model recycled water with chloride concentration C_{Cl} : (a) – 500, (b) – 550, (c) – 600 mg/L (numbers at points are melt number).

The authors of [10, 16] established complex dependencies between the limiting electrochemical parameters of AISI 304 steel in a chloride-containing medium (3% NaCl) and the specific paramagnetic susceptibility of austenite, χ_0 . It can be noted that surface phenomena occurring on the oxide films of AISI 321 and 08Kh18N10T steels are more strongly influenced by the adsorption of chloride ions than by χ_0 , which correlates well with the structural components of the steel [39]. In particular, [9, 40] reported that in model recycled waters with pH values of 4–8 and chloride concentrations ranging from 350 to 600 mg/L, the CPT of AISI 321 steel and the 06KhN28MDT alloy decreased proportionally with increasing volume fraction of inclusions involved in pitting. Based on an analysis of multifactor mathematical models that establish the relationship between the CPT of AISI 321 and AISI 304 steels, as well as the 06KhN28MDT alloy, to their chemical composition, structural components, and environmental parameters (pH, chloride concentration) of model recycled water, the authors of [16, 17, 41] concluded that pitting resistance is primarily determined by environmental factors, while the influence of structural components and chromium content is 2 and 4 times lower, respectively. This trend is consistent with the results of the present analysis (Figs. 1–4). Therefore, it can be concluded that pitting of AISI 321 steel in the investigated model recycled waters is consistent with the adsorption theory.

With an increase in chloride concentration in the model recycled water up to 500 mg/L, the rate of CPT increase for AISI 321 and 08Kh18N10T steels was observed to rise with increasing pH from 4 to 8

(Fig. 4a). It should be noted that in model recycled waters with pH 4, irrespective of chloride concentration, the CPT of AISI 321 steel melts increased in the following order: melts No. 1 → 2 → 3 → 4 → 5 (Figs. 1, 2, and 4).

In 08Kh18N10T steel, which is not alloyed with nitrogen (Table 1), the CPT in model recycled water with a chloride concentration of 350 mg/L was similar to that of melt No. 3 of AISI 321 steel, but higher than that of melts No. 1 and 2. This may be due to the larger volume of Al oxide inclusions in melts No. 1–3 (0.0065, 0.0116, 0.0104 vol.%, respectively, Table 2) and the lower content of Cr (17.1, 16.43, 17.43 wt.%) and Ni (9.1, 9.14, 9.7 wt.%, respectively) than in steel 08Kh18N10T (volume of oxides – 0.0027 vol.%, content of Cr – 17.97 wt.% and Ni – 10.46 wt.%, Table 1). However, under the same conditions, the role of titanium nitrides in the investigated steels is the largest, since the composition of sections 4 and 5 of AISI 321 steel has the lowest volume of titanium nitrides (0.2336 and 0.2974 vol.%, respectively, Table 2) and C content (0.03 and 0.04 wt.%, respectively, Table 1) and the highest CPT (Fig. 1).

In model recycled water with pH 4 and 400 mg/L, it was found that the CPT of 08Kh18N10T steel is the same as in melt No. 2, but higher than in melt No. 1 (Fig. 2). Considering the data in Tables 1 and 2, this highlights the increasing role of titanium nitride inclusions in the pitting behavior of these steels. A further increase in chloride concentration to 500 mg/L resulted in the CPT of 08Kh18N10T steel becoming the lowest among the investigated samples (Fig. 4a). This may be attributed to intensive adsorption of chloride ions in the vicinity of titanium carboni-

trides, as this steel has the highest carbon content (Table 1) and volume fraction of nitrides (Table 2). It is known [39] that the electrode potentials of certain carbides, particularly TiC, MoC, and NbC, are associated with very low hydrogen overvoltages and can thus accelerate hydrogen ion discharge. When steel is corroded with hydrogen depolarization, the accumulation of carbides on its surface accelerates cathodic depolarization processes, which leads to a shift in the pitting potential to the positive side and increases the rate of anodic processes in the pitting.

In model recycled water with pH 4 and chloride concentrations of 550 and 600 mg/L, the CPT of the investigated steels (Fig. 4b, c) was found to increase in the same sequence as observed at 400 mg/L (Fig. 2).

Neutral chloride-containing media are considered to be the most hazardous for pitting corrosion of stainless steels and passivated alloys [42]. This is attributed to a decrease in the number of stable pits on the surface accompanied by their intensive growth, resulting from an increase in current density due to its redistribution among a smaller number of pits. In such conditions, anodic processes within the pits are supported by cathodic oxygen reduction reactions on the steel surface according to $O_2 + 2H_2 + 4e^- \rightarrow 4OH^-$ [43, 44]. However, it was shown in [7] that the participation coefficients of titanium nitrides in the pitting of AISI 321 steel decrease monotonically with increasing chloride concentration in model recycled water from 350 to 600 mg/L. The authors of [40, 41] attribute this trend to the destruction of "covers" over pitting from the remnants of the oxide film under the influence of chlorides. At the same time, according to [9], the most intensive reduction of the amount of nitrides in titanium involved in the pitting of AISI 321 steel was observed in model recycled waters with pH 4 and pH 5, and the slowest – with pH 8. It was also established that, in model recycled waters with pH 4 and 5, the CPT values of AISI 321 and 08Kh18N10T steels were the lowest, while at pH 8 they were the highest, regardless of chloride concentration (Figs. 1, 2, and 4). Nevertheless, analysis of the data indicates that the CPT of the investigated steels is significantly affected by their structural features, and that this effect depends on both the pH and chloride concentration of the model recycled water. Indeed, in model recycled waters with pH 4, regardless of the chloride concentration in them, the CPT of AISI 321 steels increased in the following order: melts No. 1 → 2 → 3 → 4 → 5 (Figs. 1, 2, and 4). In contrast, in chloride-containing media with pH 7, the CPT values followed different trends depending on chloride concentration:

- at 350 mg/L: melts No. 1 → 3 → 5 → 4 → 2;
- at 400 mg/L: melts No. 2 → 3 → 1 → 4 → 5;
- at 550 mg/L: melts No. 2 → 3 → 1 → 4 → 5;
- at 600 mg/L: melts No. 2 → 3 → 5 → 1 → 4.

These results indicate that, in model recycled waters with pH 7 and chloride concentrations of 500–600 mg/L, melts No. 1, 4, and 5 of AISI 321 steel exhibit the highest CPT values. This behavior may be associated with their lower carbon and silicon contents (Table 1). At the same time, it should be noted that melt No. 1 demonstrated the lowest CPT in model recycled water with pH 4 (Figs. 1, 2, and 4), which is most likely related to its highest content of titanium nitrides (Table 2).

Analysis of the data presented in Figs. 1, 2, and 4 showed that, in model recycled waters at pH 8 the CPT increased with increasing melt number in the following order depending on the chloride concentration:

- at 350 mg/L: melts No. 1 → 5 → 4 → 2 → 3;
- at 400 mg/L: melts No. 1 → 2 → 3 → 5 → 4;
- at 500 mg/L: melts No. 2 → 1 → 3 → 5 → 4;
- at 550 mg/L: melts No. 3 → 2 → 5 → 1 → 4;
- at 600 mg/L: melts No. 3 → 2 → 1 → 5 → 4.

This indicates that in model recycled water with pH 8 and chloride concentrations of 400, 500, 550, and 600 mg/L, melts No. 4 and 5 of AISI 321 steel exhibit the highest CPT values, while at 550 and 600 mg/L, the CPT of melt No. 1 approaches these values.

Summing up the above, it can be noted that the parameters of model recycled water (pH and chloride concentration) have the greatest impact on the CPT of AISI 321 and 12Kh18N10T steels. In particular, they increase with increasing pH and decreasing chloride concentration. It was found that the components of the structure of the studied steels have less influence on their CPT, and their corrosion behavior depends on the parameters of the environment media. Indeed, in acidic chloride-containing media with pH values of 4–6, regardless of chloride concentration, the maximum CPT was observed in melts No. 4, 5, and 3, which have the lowest volumes of titanium nitrides and aluminum oxides, the smallest mean austenite grain diameters, and the lowest carbon contents in melts No. 4 and 5. In neutral model recycled water with pH 7, the influence of steel structure components depends on the chloride content; in particular, in media with 400 mg/L, the maximum CPT was found in the melts with the minimum volume of titanium nitrides, which are mainly involved in pitting of steels, and with 500, 550, 600 mg/L with the minimum C content, which created a carbide phase in its vicinity. In the slightly

alkaline model recycled water with pH 8, a similar trend was found, but the same behavior of the melt No. 1 with low C content was observed in solutions with chloride concentrations of 550, 600 mg/L. This is due to more intensive processes of pitting repassivation due to the destruction of “covers” from the remnants of the oxide film above them and repassivation due to free access of oxygen to them and the inhibitory effect of hydroxides in the medium.

4. Conclusions

The results of the study indicate that the parameters of the model recycled water – specifically, pH and chloride concentration – have the greatest influence on the pitting resistance of AISI 321 and 08Kh18N10T steels, while the effect of structural components is smaller and depends on the characteristics of the medium. It has been found that the CPT of the steels increased with an increase in the pH of the model recycled water and a decrease in the concentration of chlorides in it. It was shown that in acidic chloride-containing media with a pH of 4–6, regardless of the chloride concentration, the maximum CPT was observed in melts with a smaller volume of titanium nitride involved in its pitting and lower carbon content, which created a carbide phase in its vicinity. It has been found that in neutral (pH 7) and slightly alkaline acidic (pH 6) model recycled waters, the effect of structure on the CPT of the investigated steels depends on the concentration of chlorides in them. In particular, in solutions with pH 7 and 400 mg/L, they are maximum in the melts with the minimum volume of titanium nitride, and with 500, 550, 600 mg/L also with the maximum C content. In slightly alkaline model recycled waters (pH 8), a similar trend was observed only at chloride concentrations of 550 and 600 mg/L. It was also shown that nitrogen alloying of AISI 321 steel does not significantly enhance its pitting resistance in the studied model recycled waters. The CPT of 08Kh18N10T steel was only slightly lower than that of AISI 321 steel in acidic media and exhibited intermediate values in neutral and slightly alkaline environments, regardless of chloride concentration.

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