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FIELD APPLICABLE METHODS FOR INTERGRANULAR CORROSION TESTING OF STAINLESS STEELS STRUCTURES

METODE ISPITIVANJA INTERKRISTALNE KOROZIJE NA KONSTRUKCIJAMA OD NERĐAJUĆIH ČELIKA NA TERENU

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- intergranular corrosion
- test methods

Ključne reči
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- metode ispitivanja

Abstract

Determination of the susceptibility of austenitic stainless steel X5CrNi18-10 to intergranular corrosion (IGC) is performed by measuring the corrosion potential E_{corr} in a drop of a test solution with specific chemical composition. It has been shown that a good correlation exists between the results obtained by E_{corr} measurements in a drop of the test solution and the results obtained by the double loop electrochemical potentiokinetic reactivation method (DL EPR). The results are confirmed by SEM analysis of the stainless steel surface after the IGC test. The method of E_{corr} measurements is a simple non-destructive method that provides qualitative information on the susceptibility of the stainless steel to IGC. The DL EPR method is a quantitative method that can determine small differences in the susceptibility of stainless steel to IGC. Simple and cheap equipment is required to perform E_{corr} measurements. The method is easy to perform on stainless steel structures in the field.

Izvod

Određivanje sklonosti austenitnog nerđajućeg čelika X5CrNi18-10 prema interkristalnoj koroziji izvršeno je na osnovu merenja korozionog potencijala E_{kor} u kapi rastvora definisanog sastava. Pokazano je da postoji dobra saglasnost dobijenih rezultata sa rezultatima ispitivanja interkristalne korozije primenom metode elektrohemijske potenciokinetičke reaktivacije sa povratnom petljom (DL EPR). Dobijeni rezultati su potvrđeni SEM analizom površine čelika posle ispitivanja interkristalne korozije navedenim metodama. Metoda merenja E_{kor} u kapi rastvora je jednostavna, nerazarajuća metoda koja daje kvalitativne podatke o sklonosti čelika prema interkristalnoj koroziji, dok je DL EPR metoda kvantitativna, kojom se mogu odrediti male razlike u sklonosti nerđajućih čelika prema interkristalnoj koroziji. Za izvođenje ispitivanja metodom merenja E_{kor} u kapi rastvora potrebna je znatno jeftinija i jednostavnija oprema. Ova metoda se lako izvodi na gotovim konstrukcijama, na terenu.

INTRODUCTION

IGC is a form of localized corrosion of stainless steels which is manifested by dissolution of grain boundary areas (GBA). During slow cooling or heating, in the temperature range from 420 to 820 °C, the chromium-rich carbides precipitate in the GBA, mainly $M_{23}C_6$ /1-10/. Their precipitation causes the depletion in the chromium content of GBA. If the chromium content in these areas is less than the content necessary for maintaining the protective passive film, these areas become sensitized and susceptible to IGC. This is due to the slow diffusion of chromium in austenite in the specified temperature range. The GBA depleted in chromium have a higher dissolution rate as compared to the grain interior, /2-4/.

Sensitization to IGC is most common in welded joints of stainless steels, in the heat affected zone (HAZ), parallel to the weld metal, or during residual stress annealing. The susceptibility to IGC is greater after welding of thick plates than thin sheets, as a result of different cooling rates, /11/.

Different procedures may be applied to increase the resistance of stainless steels to IGC /1, 3-5, 12-14/. It is possible to apply a stabilizing heat treatment of welded structures in order to dissolve chromium-rich carbides and to homogenize the chromium content in the stainless steel. Stabilization heat treatment is usually performed at temperatures from 950 to 1050 °C. The susceptibility to IGC can be significantly reduced using stainless steels with low carbon content (< 0.04 %C) or by application of stabilized stainless steels, i.e. austenitic stainless steels alloyed with Ti or Nb.

Traditionally, testing of the sensitization to IGC is carried out by chemical treatment of stainless steel samples in boiling solutions of various inorganic acids, such as Strauss method, Streicher test, or Huey test /1, 3-5, 14/. Testing time is relatively long and can be up to 10 days, depending on the test method.

Testing time can be significantly reduced when using the DL EPR method /15-18/. The test is performed in a solution of sulphuric acid and potassium thiocyanate. The potential

of the sample gradually shifts from E_{corr} to the positive potential region, where sample passivation occurs, and then it reverses to E_{corr}. If the stainless steel is susceptible to IGC, the GBA are activated in the reverse part of the loop. The ratio of the current peak value in the reactivation (reverse) part of the loop and the current peak value in the passivation (anodic) part of the loop is a measure of stainless steel susceptibility to IGC. The thiocyanate ion is a depassivator that increases anodic dissolution during the anodic sweep, as well as the grain boundary attack during the reactivation sweep /5/. The DL EPR method was developed by V. Číhal /15, 18/. The method has been applied for testing the susceptibility to IGC, not only of austenitic stainless steels /19-21/, but also ferritic stainless steels /22/ and duplex stainless steels /23/.

Tomashov and co-workers /24-26/ developed a qualitative method for testing stainless steels susceptibility to IGC. The method is based on E_{corr} measurements in a drop of the test solution with a specific chemical composition. During the occurrence of electrochemical corrosion reactions in a drop of the solution, the E_{corr} is established on the stainless steel surface. The E_{corr} value depends on the nature of a metal, state of the surface, chemical composition and concentration of the electrolyte and temperature. A stainless steel in the active (sensitized) state has a negative E_{corr} value, while in a passive state it has a positive E_{corr} value.

IGC testing by E_{corr} measurements is performed in a solution of HNO_3, containing a strong oxidizing agent $FeCl_3$ $6H_2O$ and HCl as an activator. Due to a decrease in the concentration of chromium in the vicinity of grain boundaries, IGC and pitting corrosion occur in the presence of activator (HCl). Grain interiors with a higher concentration of chromium are not subjected to the corrosion attack. Test solution contains HNO_3 as a passivator, which allows the formation of a stable passive layer on the stainless steel surface. The passive layer is resistant to IGC. The activation of GBA with a lower concentration of chromium occurs in the mentioned test solution.

Width of the sensitized area depleted in chromium, on each side of the grain boundary is ~ 0.5 μm, according to ASTM G108 /27/. Total surface area of sensitized regions along all grain boundaries on the sample surface SGBA can be determined using the following equation, in accordance with ISO 12732 /18/:

$$S_{GBA} = A_s \left(10^{-3} \cdot \sqrt{2^{G+5}}\right) \quad (1)$$

where: A_s is the sample surface area; G is the grain size, according to ISO 643 /28/.

The susceptibility of stainless steels to IGC is often examined after sensitization heat treatment. Testing of susceptibility to IGC for stainless steel X5CrNi18-10 after sensitization heat treatment is performed in this work, using E_{corr} measurements in a drop of the test solution and the DL EPR method. The sensitization heat treatment of the stainless steel is carried out at 630°C for 90 min., in accordance to the peak position on the C-curve. In the case of stainless steel with 0.04 %C, the peak for the chromium-rich carbide precipitation is at 630°C, on the C-curve (Fig. 1).

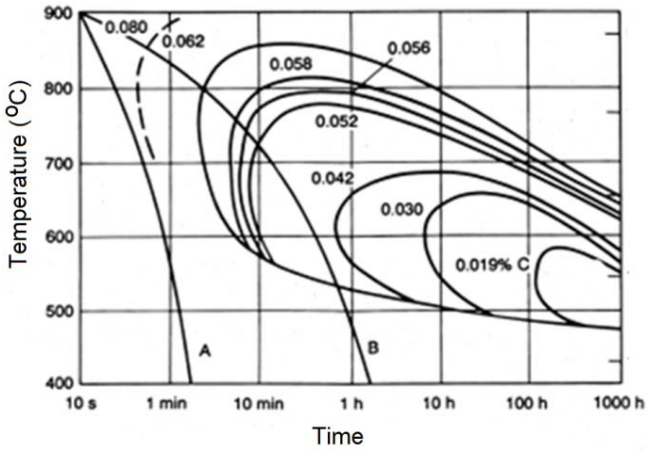

Figure 1. C-curves for CrNi18-10 stainless steel (AISI 304) with different carbon content. A - high cooling rate, B - low cooling rate, according to /9/.

Testing of the stainless steel susceptibility to IGC is also carried out after the stabilization heat treatment, as well as on stainless steel samples without heat treatment. Stabilization heat treatment is performed at a considerably lower temperature (730°C/90 min) than usually applied temperature for stabilization heat treatment (950 to 1050 °C), also in accordance with the C-curve for the given stainless steel (Fig. 1).

The aim of this work is to apply E_{corr} measurements in a drop of the test solution for determining IGC susceptibility of stainless steel X5CrNi18-10 and to compare the obtained results with the results of the DL EPR method and the SEM analysis.

EXPERIMENTAL

Material

Chemical composition of stainless steel X5CrNi18-10 is given in Table 1.

Table 1. Chemical composition of X5CrNi18-10, mass.%

Element	C	Cr	Ni	Mo
X5CrNi18-10	0.04	18.8	9.5	0.22

Before testing, stainless steel samples are ground by grinding paper of progressively finer grade (from 600 to 1500 grits). After that, the samples are polished on the polishing cloth, using an aqueous Al_2O_3 suspension (with particle size of 5 μm). The samples are then degreased with ethanol, washed with distilled water and air-dried.

E_{corr} measurements in a drop of the test solution

Measurements of E_{corr} are carried out on the stainless steel surface, in a drop of the test solution. The chemical composition of the test solution is $FeCl_3$ $6H_2O$ + HNO_3 + HCl. The test solution is prepared with analytical grade chemicals and double-distilled water, according to /26/. The test surface is limited with PVC insulating tape. PVC tape with holes (6 mm in diameter) is attached to the stainless steel surface, and 1-2 drops of the test solution is carefully poured into holes on the PVC tape.

Figure 2. SCE with double mantle for IGC testing by E_{corr} measurements in a drop of the test solution.

E_{corr} measurements are carried out using a saturated calomel electrode SCE with a double mantle. A ceramic frit is placed at the bottom of the inner mantle, while the outer mantle has a hole (1 mm in diameter) at the bottom. Electrical contact between the drop of the test solution on the stainless steel surface and SCE is achieved through the hole and the ceramic frit (Fig. 2). A saturated solution of KCl is inside the double mantle, while the test solution is outside the mantle.

SCE with a double mantle is placed near the sample, so that the electrical contact between the sample (stainless steel) and the SCE is achieved through the test solution drop. A multimeter with internal impedance of 10 MΩ is connected to the sample and the SCE. E_{corr} is measured after 5, 15, and 30 s and then after every 15 s to 120 s. Measurements are performed with the sensitized and stabilized sample, and with a sample without heat treatment. Measurements are taken at 3 different places on each sample. E_{corr} values measured vs. SCE are converted to E_{corr} values vs. a saturated silver/silver-chloride electrode (Ag/AgCl), in accordance with /26/.

A stainless steel is resistant to IGC if the measured values of E_{corr} are positive ($E_{corr} > 0$). A stainless steel is not resistant to IGC if the measured E_{corr} are negative ($E_{corr} < 0$).

Double loop electrochemical potentiokinetic reactivation method (DL EPR)

DL EPR tests are performed on sensitized and stabilized samples, as well as on the sample without heat treatment, in a solution of 0.5 mol·dm^{-3} H_2SO_4 + 0.01 mol·dm^{-3} KSCN /18/.

The tests are carried out in the usual electrochemical cell with SCE as a reference electrode and a platinum foil as a counter electrode. The working electrode (test sample) with 0.785 cm^2 area is placed in a special holder. The relatively stable E_{corr} is established in the test solution. The value of E_{corr} is in the required range of corrosion potentials, /18/. The sample was held for 5 min at the E_{corr}, and then the potential was moved in the positive direction to the passivation range (+300 mV) at a scan rate 1.67 mV·s^{-1}. Immediately after reaching the passivation potential (+300 mV) the direction of polarization is changed and the potential of the sample is returned to the E_{corr}. If a stainless steel is susceptible to IGC, GBA are activated in the reverse part of the loop. The ratio of the charge density Q_r that is spent during reactivation (i.e. during the dissolution of GBA) and the charge density Q_p that is consumed during activation (i.e. during dissolution of grains and GBA) represents an indicator $(Q_r/Q_p)_{GBA}$ of the IGC susceptibility:

$$\left(\frac{Q_r}{Q_p}\right)_{GBA} = \frac{Q_r}{Q_p\left(10^{-3}\cdot\sqrt{2^{G+5}}\right)} \tag{2}$$

where: G is grain size, according to ISO 643 /28/.

SEM analysis

A scanning electron microscope JEOL JSM-5800, operating at 20 keV, is used to analyse the morphology of the stainless steel surface after testing the IGC susceptibility. The grain size G, required for calculating the sensitization degree of stainless steel to IGC (Eq.(2)) is determined by SEM. Determination of the grain size is performed on sensitized and stabilized samples, as well as on the sample without heat treatment. In all cases the grain size is G9, according to ISO 643 /28/.

RESULTS AND DISCUSSION

Results of IGC testing by E_{corr} measurements in a drop of the test solution are compared with results obtained by the DL EPR method.

E_{corr} measurements in a drop of the test solution

Typical results of E_{corr} measurements on the stainless steel sample without heat treatment, and on the sensitized and stabilized sample are shown in Fig. 3a-c. In case of the stabilized stainless steel, the E_{corr} value rapidly increases in the first 45 s, after which it reaches an approximately constant value (~ +300 mV vs. Ag/AgCl). In case of the sensitized stainless steel, the value of E_{corr} drops sharply during the first 45 s, and then it reaches an approximately constant value (~ –300 mV vs. Ag/AgCl).

SEM micrographs of the stainless steel surface after E_{corr} measurements in a drop of the test solution are shown in Fig. 4. The presence of corrosion damages was not observed on the sample surface without heat treatment (Fig. 4a). A significant corrosion damage of local nature (pitting and partial dissolution of GBA) can be seen on the sensitized sample (Fig. 4b). As a consequence of corrosion processes, the sensitized sample surface is activated and E_{corr} is shifted in the region of negative potentials (Fig. 3b). In case of the stabilized sample surface and the sample surface without heat treatment, the presence of corrosion damages is not noticed. These surfaces are passivated and E_{corr} is shifted in the region of positive potentials (Fig. 3a,c).

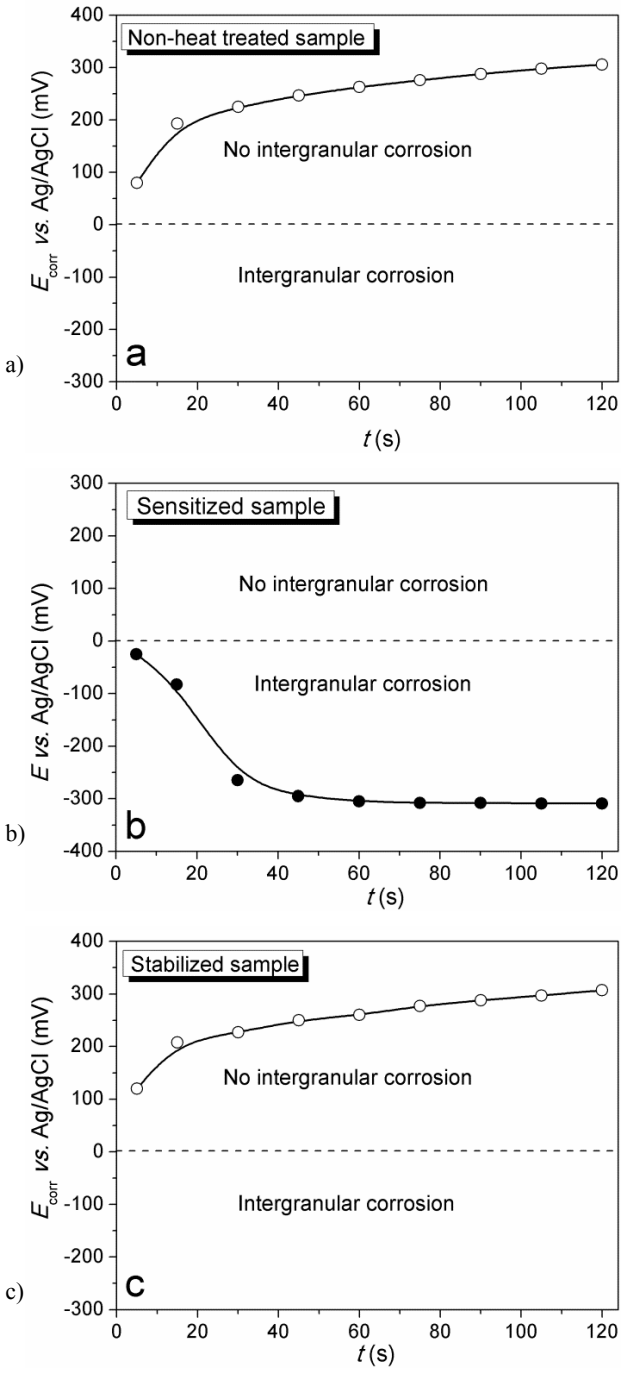

Figure 3. Results of IGC testing by E_{corr} measurements in a drop of the test solution: a) sample without heat treatment, b) sensitized and c) stabilized sample.

Table 2 shows the results of E_{corr} measurements in a drop of the test solution, after 120 s.

Table 2. Results of E_{corr} measurements after 120 s.

	E_{corr} (mV)		
	No. 1	No. 2	No. 3
Non-heat treated sample	303	306	305
Sensitized sample	–306	–309	–305
Stabilized sample	309	307	308

Three E_{corr} measurements are performed at different places on each sample of stainless steel. Obtained results are very reproducible (Table 2). Negative values of E_{corr} indicate that the stainless steel after sensitization heat treatment is susceptible to IGC. Positive values of E_{corr} indicate that the stainless steel without heat treatment and the stabilized stainless steel are not susceptible to IGC.

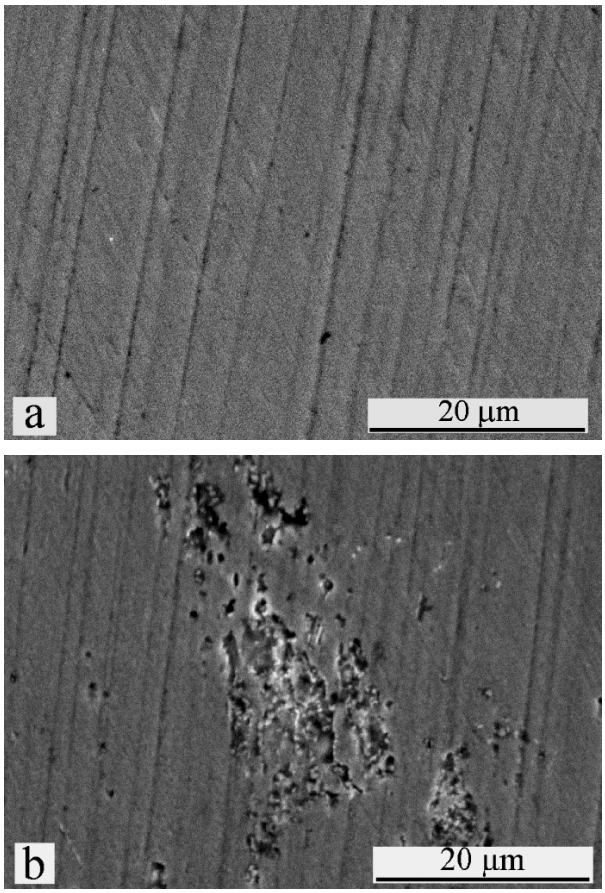

Figure 4. SEM micrographs of stainless steel X5CrNi18-10 after E_{corr} measurements in a drop of the test solution: a) sample without heat treatment, and b) sensitized sample.

Results of IGC susceptibility testing for stainless steel X5CrNi18-10, using the DL EPR method, are shown in Fig. 5a-c. It can be seen that the value of the reactivation charge density Q_r is the lowest in case of stabilized sample (Fig. 5c). The value of Q_r is slightly higher for the sample without heat treatment (Fig. 5a), and significantly higher for the sensitized sample (Fig. 5b). Calculated value of Q_r is 35 times higher for the sensitized sample than for the sample without heat treatment, and 49 times higher than for the stabilized sample. The value of maximum reactivation current I_r is 29 times higher for the sensitized sample than for the sample without heat treatment, and 116 times higher than for the stabilized sample. This indicates IGC susceptibility of the sensitized stainless steel. IGC susceptibility can be also estimated on the basis of the maximum reactivation current I_r and the maximum passivation current I_p (Fig. 5a-c).

SEM micrographs of the stainless steel surface after IGC testing by DL EPR method are shown in Fig. 6. A visible dissolution of the GBA on the sensitized sample (Fig. 6b) can be noticed. A much less dissolution of the GBA can be seen on the sample without heat treatment (Fig. 6a).

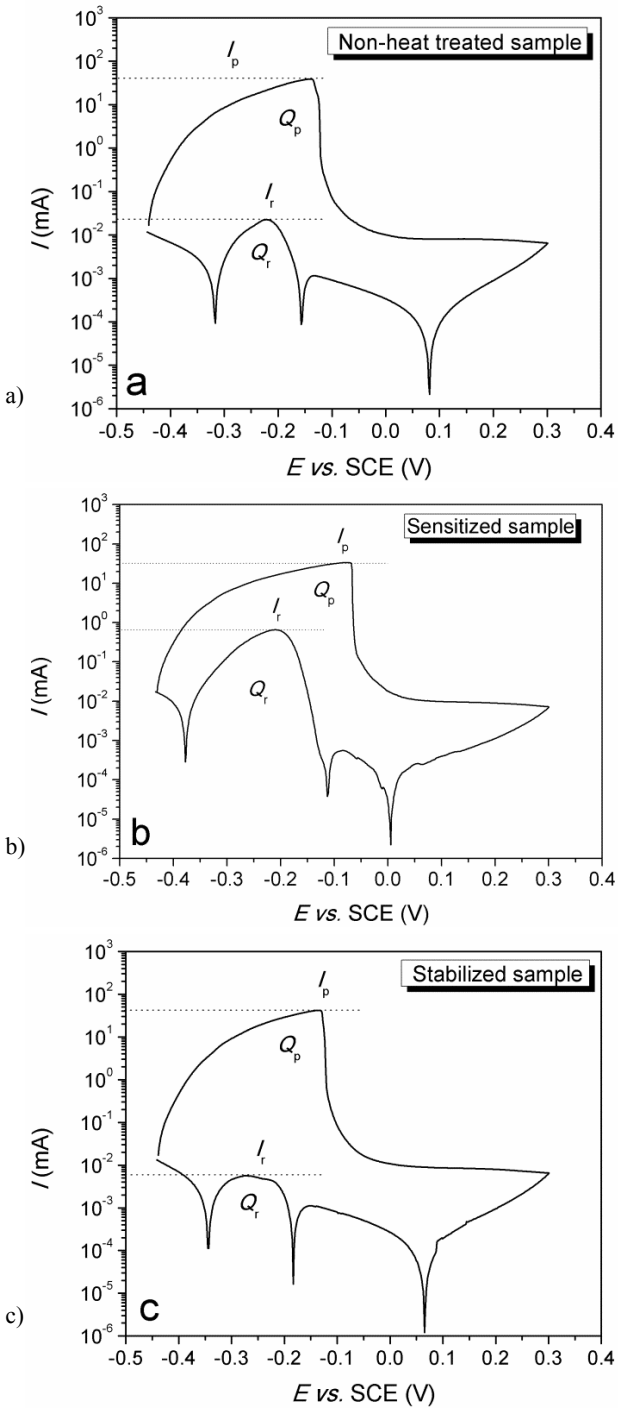

Figure 5. Results of IGC testing by DL EPR method: a) sample without heat treatment, b) sensitized and c) stabilized sample.

Results of IGC testing by DL EPR method are shown in Table 3.

Table 3. Results of IGC testing by DL EPR method.

	I_r (μA)	I_p (μA)	Q_r (mC)	Q_p (mC)	$(Q_r/Q_p)_{GBA}$ (%)
Non-heat treated sample	22.62	38797	0.992	2690	0.288
Sensitized sample	650,1	33625	35.1	2941	9.324
Stabilized sample	5.611	41988	0.713	2960	0.188

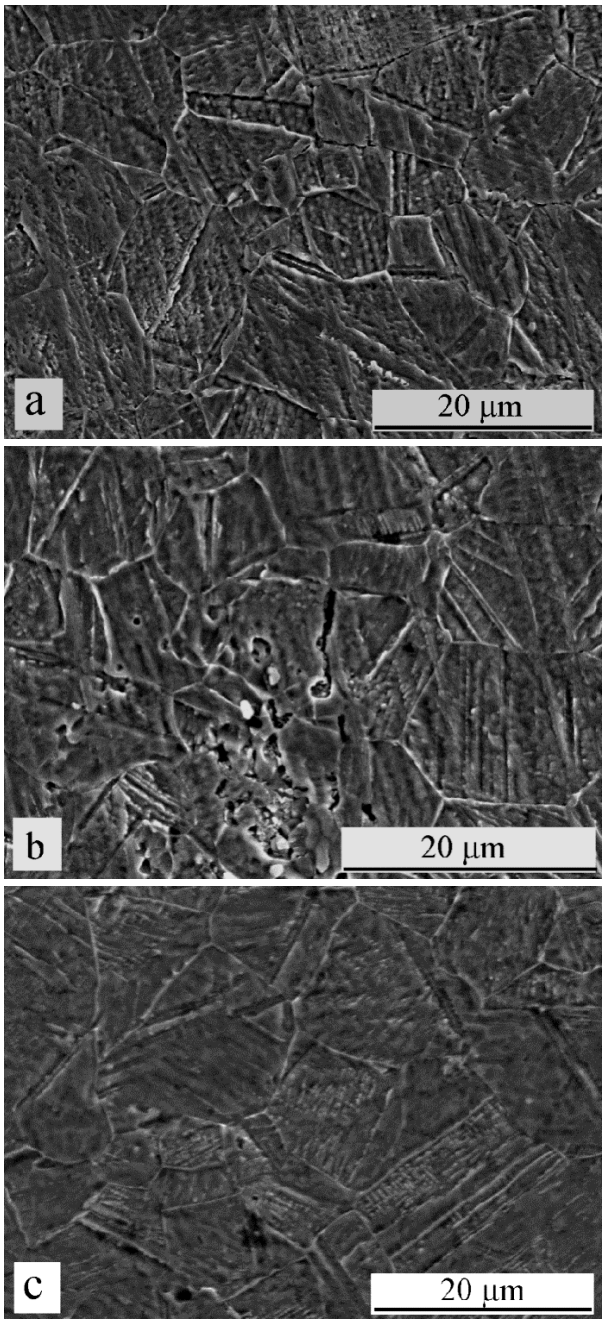

Figure 6. SEM micrographs of stainless steel X5CrNi18-10 after testing by DL EPR method: a) sample without heat treatment, b) sensitized and c) stabilized sample.

Quantitative indicators of IGC susceptibility are shown in Table 3. Their values are calculated using Eq.(2). Very low value of the indicator $(Q_r/Q_p)_{GBA}$ is obtained for the stabilized sample which indicates its complete resistance to IGC. A slightly higher value of $(Q_r/Q_p)_{GBA}$ for the sample without heat treatment does not indicate its susceptibility to IGC. A significantly higher value of $(Q_r/Q_p)_{GBA}$ indicates that the sensitized sample is more susceptible to IGC than the sample without heat treatment and the stabilized sample. Indicator $(Q_r/Q_p)_{GBA}$ is 32 times higher for the sensitized- than for the sample without heat treatment, and it is 50 times higher than for the stabilized sample.

According to the results shown in Figs. 3-6 and in Tables 2 and 3 it can be concluded that the stabilized stainless steel is not susceptible to IGC. The stainless steel without heat treatment is also not susceptible to IGC. The stainless steel after sensitization heat treatment is more susceptible to IGC. The results of testing by E_{corr} measurements, DL EPR method and SEM analysis confirm these statements.

A stainless steel which is susceptible to IGC is also susceptible to intergranular stress corrosion cracking (IGSCC), so that E_{corr} measurements in a drop of the test solution and DL EPR method can be used to assess the susceptibility of the stainless steel to IGSCC, /18/.

As previously mentioned, the applied temperature and time of sensitization heat treatment for stainless steel X5CrNi18-10 are in accordance with the C-curve shown in Fig. 1. During sensitization heat treatment of stainless steel X5CrNi18-10 the chromium-rich carbides $M_{23}C_6$ were precipitated. Due to this a depletion in chromium content in GBA has occurred. The actual formula of $M_{23}C_6$ carbide is $(Cr,Fe)_{23}C_6$ as some Cr atoms are replaced with Fe atoms in this carbide.

Comparison of test results obtained by E_{corr} measurement in a drop of the test solution (Fig. 3a-c and Table 2) and by the DL EPR method (Fig. 5a-c and Table 3) shows that these results are in a good agreement. Method of E_{corr} measurements is a qualitative method, while the DL EPR method is a quantitative method, which can determine small differences in IGC susceptibility of a stainless steel. Both methods can be applied for testing IGC susceptibility of stainless steel structures on site, but the E_{corr} measurement testing is more easier and cheaper. To perform E_{corr} measurements, a multimeter and a reference electrode are required, as well as the test solution. Testing by the DL EPR method requires more complex and more expensive equipment. Preparation of the surface of a stainless steel structure before E_{corr} measurements is similar to the preparation when making a replica for metallographic tests on site. The preparation of replicas is described in ASTM E351, /29/.

CONCLUSIONS

Testing of IGC susceptibility of the austenitic stainless steel X5CrNi18-10 is carried out by E_{corr} measurements in a drop of the test solution and by DL EPR method. The tests were performed on samples without heat treatment, after sensitization heat treatment (630°C/90 min) and after stabilization heat treatment (730°C/90 min).

According to the results obtained by E_{corr} measurements the stainless steel X5CrNi18-10 is susceptible to IGC after the sensitization heat treatment. The stainless steel without heat treatment and stabilized stainless steel are not susceptible to IGC.

According to the results obtained by DL EPR method, the indicator $(Q_r/Q_p)_{GBA}$ is 32 times higher for sensitized- than for the sample without heat treatment, and 50 times higher than for the stabilized sample. This means that sensitized stainless steel is considerably more susceptible to IGC than the stabilized- and non-heat treated stainless steel.

The results obtained by E_{corr} measurements are in a good agreement with results obtained by DL EPR method. All results are confirmed by SEM analysis of the stainless steel surface after IGC tests.

The method of E_{corr} measurements in a drop of the test solution is a qualitative method that determines if a stainless steel is susceptible to IGC. The DL EPR method is a quantitative method that enables determination of small differences in susceptibility of a stainless steel to IGC. The method of E_{corr} measurements is a simple, non-destructive method that does not require expensive equipment, while the application of DL EPR requires more complex and expensive equipment. Both methods can be applied for in-situ testing of IGC susceptibility on stainless steel structures, but the E_{corr} measurements are easier and cheaper.

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27. Electrochemical Reactivation (EPR) for Detecting Sensitization of AISI Type 304 and 304L Stainless Steels – ASTM G 108.
28. Micrographic determination of the apparent grain size - ISO 643.
29. Standard Practice for Production and Evaluation of Field Metallographic Replicas – ASTM E1351.

ESIS ACTIVITIES

CALENDAR OF CONFERENCES, TC MEETINGS, and WORKSHOPS

June 18-23, 2017	ICF14-Fourteenth International Conference on Fracture	Rhodes, Greece	http://www.icf14.org/
June 27-29, 2017	LCF8-Eighth International Conference on Low Cycle Fatigue	Dresden, Germany	http://www.lcf8.de/
July 3-5, 2017	VHCF7-Seventh International Conference on Very High Cycle Fatigue	Dresden, Germany	http://www.vhcf7.de/
September 4-7, 2017	2nd Int. Conf. on Structural Integrity (ICSI2)	Madeira, Portugal	http://icsi.inegi.up.pt/
September 10-14, 2017	ESIS TC-4 Meeting	Les Diablerets, Switzerland	
September 19-22, 2017	3rd Int. Symp. on Fatigue Design and Material Defects	Lecco, Italy	http://www.fdmd3.polimi.it
September 25-27, 2017	ESIS TC-5 Meeting	St. Petersburg, Russia	
August 26-31, 2018	22nd European Conference of Fracture (ECF22)	Belgrade, Serbia	
September 19-21, 2018	CP 2018- 6th International Conference on 'Crack Paths'	Verona, Italy	