

## CORROSION EFFECT OF 65% NITRATE ACID ON X4CrNi1812 AT 333 K

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**Abstract:** Austenitic stainless steels are often used for a materials in the construction of machines and equipment for agricultural and for industrial construction. One of the most important factors constructional material is corrosion resistance. Equipment with austenitic stainless steel can be easy join by quickly welding at a not to high construction price, but one with the serious problem in aggressive environment is their corrosion resistance.

A few corrosion processes in crevices and awkward corners can be avoided at the design stage (low roughness parameters, round-section and other). But still the construction material is exposed to corrosion. These steels often come into contact with an aggressive environment based on nitric acid.

The main aim of this research is to investigate corrosion resistance in different time (48, 96, 144, 192, 240, 288, 336 hours). For this used weight loss of test samples and its profile roughness. The research was conducted on austenitic stainless steel in grade in Nitrate acid at 333 K.

Corrosion tests confirmed that the research this steel in 65% nitrate acid as a corrosive environments is characterized through proportionate to time corrosion process whose measure may be surface roughness. In industrial practice roughness parameters for all the research times can be used for determine the stage and size of steel corrosion.

**Keywords:** steel, stainless steel, corrosion, corrosion rate, roughness

### 1. INTRODUCTION

The stainless steels are very popular in industry and in home (Pramanik and Basak, 2015). The austenitic steels are the most widely used grade of stainless steel. They contain between about 16 and 25% chromium and about 8-10% nickel which helps to stabilize their austenitic structure and low levels of carbon. This steels has very high formability and resistance to corrosion. The corrosion resistance of austenitic stainless steels is attributed to the formation of a thin, adherent and self-healing passive layer formed on the surface in most environments. The stainless steels are non-magnetic.

The austenitic stainless steel has low fatigue endurance limit is only about 30% of the tensile strength. The low fatigue endurance are problem in machine parts working with variable loads, first of all with high temperature (the steels has high thermal expansion coefficients). The parameters makes them especially susceptible to thermal fatigue.

One with the popular grade austenitic stainless steel is. If the chloride and salt contents are low the X4CrNi1812 steel is resistant to corrosion in most urban and rural atmospheres. According to the standard EN 10088-1:2014 "Stainless steels – Part 1: List of stainless steels" (EN 10088-1:2014, 2014), this grade of steel is susceptible to intergranular corrosion in the as-welded condition. This grade of stainless steel is not high resistant on salt and acid in high temperatures.

The purpose of this work was to ascertain how 35-minutes isothermal heat treatments at 1373 K and corrosion time effect the relative mass loss and profile roughness parameters of X4CrNi18-12 stainless steel.

## 2. METHODOLOGY

The experiment was performed with austenitic stainless steel X4CrNi18-12 (1.4303). The samples were held at a temperature 1373 K by 35 minutes and cooling down on water at room temperature. Accordance with standard PN-EN ISO 3651-1:2004, Determination of resistance to intergranular corrosion of stainless steels. Part 1: Austenitic and ferritic-austenitic (duplex) stainless steels. Corrosion test in nitric acid medium by measurement of loss in mass (Huey test), corrosive media were represented by boiling nitric acid V 65% at 333 K.

Before experiments, the specimens with an area of 16 cm<sup>2</sup> (4 x 1 x 0.8 cm) were successively polished with emery paper to roughness no more than Ra=0.35 µm of, next mechanically cleaned with 95% alcohol.

The corrosion rare of the 1.4162 steel measured in mm/year was calculated with the use of the below formula (1), but measured in g/m<sup>2</sup> were calculated with the use of the below formula (2):

$$r_{corm} = \frac{8760 \cdot m}{S \cdot t \cdot \rho} \quad (1)$$

$$r_{corg} = \frac{1000 \cdot m}{S \cdot t \cdot \rho} \quad (2)$$

where:

t – time of treatment in a corrosive solution of boiling nitric acid [hours],

S - surface area of the sample [cm<sup>2</sup>],

m – average mass loss in boiling solution [g],

ρ - sample density [g/cm<sup>3</sup>].

The holding times of the samples in nitric acid were 48, 96, 144, 192, 240, 288, 336, 384 hours. The influence of boiling nitric acid on the X4CrNi18-12 austenitic steel corrosion resistance was investigated using weight loss. The mass of samples were measured by Kern ALT 3104AM general laboratory precision balance with accuracy of measurement 0.0001 g.

Profile roughness parameters were analyzed according to the PN-EN 10049:2014-03 standard (Measurement of roughness average Ra and peak count R<sub>Pc</sub> on metallic flat products) by the Diavite DH5 profilometer.

### 3. RESULTS AND DISCUSSION

The real chemical composition tested steel is presented in Table 1.

Table 1  
Chemical composition of the X4CrNi18-12 steel

Mean chemical compositions [wt. %]								
C	Si	Mn	Ni	P	S	Cr	N	Fe
0.04	0,6	1.43	11.05	0.018	0.01	17,6	0.09	bal.

Average mechanical properties at ambient temperature tested steel are presented in Table 2.

Table 2  
Mechanical properties at ambient temperature of the X4CrNi18-12 steel

Mechanical properties			
R <sub>02</sub>	R <sub>m</sub>	A	HB
MPa	MPa	%	HB
346	642	47	195

Influence time of soaking the X4CrNi18-12 stainless steel in nitric acid V 65% at temperature 333 K on the relative mass loss (RML) is presented in Fig. 1, regression equation and correlation coefficient  $r$  at (3).

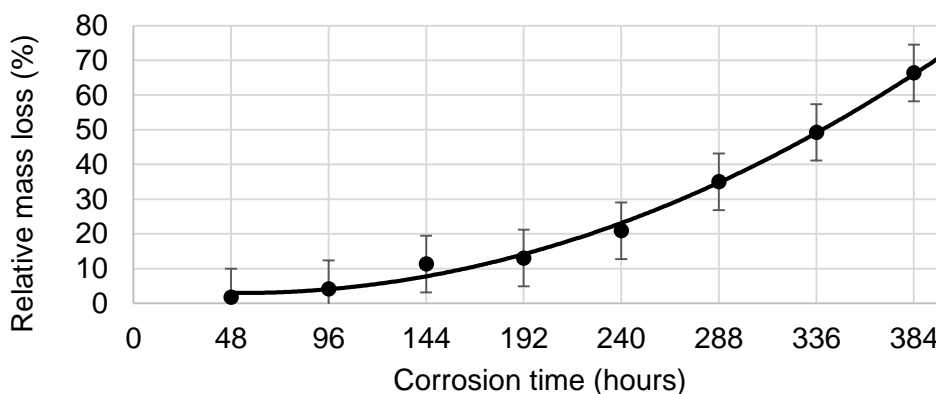


Fig. 1. Influence of immersion time in boiling nitric acid on the relative mass loss of X4CrNi18-12 after solution heat treatment in 1373 K by 35 minutes and cooling down on water

$$\text{RML} = 0.0006 t^2 - 0.06 t + 4.561 \text{ and } r = 0.9972 \quad (3)$$

For the first 186 hours a proportional (linear) increase in relative mass loss was observed. In the next stage, RML accelerated to 288 hours, and for next this corrosion times increase is linear again (Fig. 1).

Influence time of soaking the X4CrNi18-12 stainless steel in nitric acid V 65% at temperature 333 K on the corrosion rate ( $r_{\text{corrm}}$ ) measured in mm per year is presented in Fig. 2, regression equation and correlation coefficient  $r$  at (4).

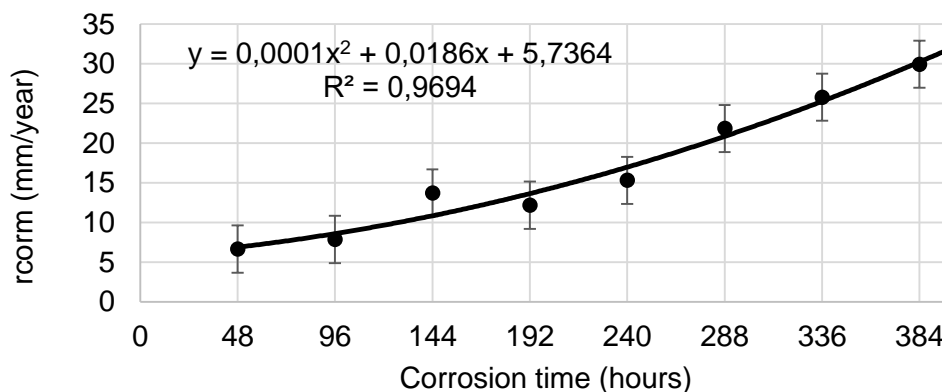


Fig. 2. Influence of immersion time in boiling 95% nitric acid on the corrosion rate measured in mm per year of X4CrNi18-12 after solution heat treatment in 1373 K by 35 minutes and cooling down on water

$$r_{\text{corrm}} = 0.0001 t^2 + 0.0186 t + 5.7364 \text{ and } r = 0.9845 \quad (4)$$

Influence time of soaking the X4CrNi18-12 stainless steel in nitric acid V 65% at temperature 333 K on the corrosion rate ( $r_{\text{corrm}}$ ) measured in gram per  $\text{m}^2$  is presented in Fig. 3, regression equation and correlation coefficient  $r$  at (5). By analyzing the course of the curves of Fig. 2 and Fig. 3, a proportional corrosion course was observed up to 240 hours. For longer soaking times, the process has accelerated.

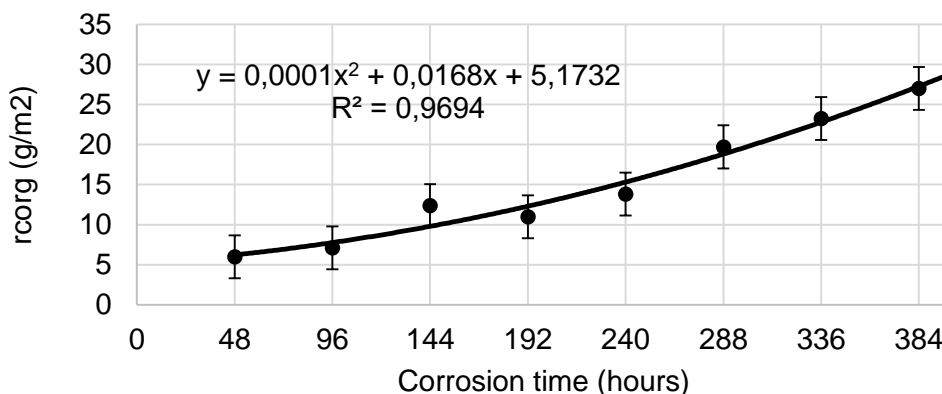


Fig. 3. Influence of immersion time in boiling 95% nitric acid on the corrosion rate measured in gram per  $\text{m}^2$  of X4CrNi18-12 after solution heat treatment in 1373 K by 35 minutes and cooling down on water

$$r_{\text{corg}} = 0.0001 t^2 + 0.0168 t + 5.732 \text{ and } r = 0.9845 \quad (5)$$

Profile roughness parameters of X4CrNi18-12 steel after corrosion tests in nitric acid V 65% at temperature 333 K is presented in Fig. 4 with:  $R_a$  - arithmetical mean roughness value [ $\mu\text{m}$ ],  $R_q$  - mean peak width [ $\mu\text{m}$ ], for time range: 48, 96, 144, 192, 240, 288, 336

and 384 hours of soaking. The regression equations and correlation coefficients  $r$  is presented in (6) and (7).

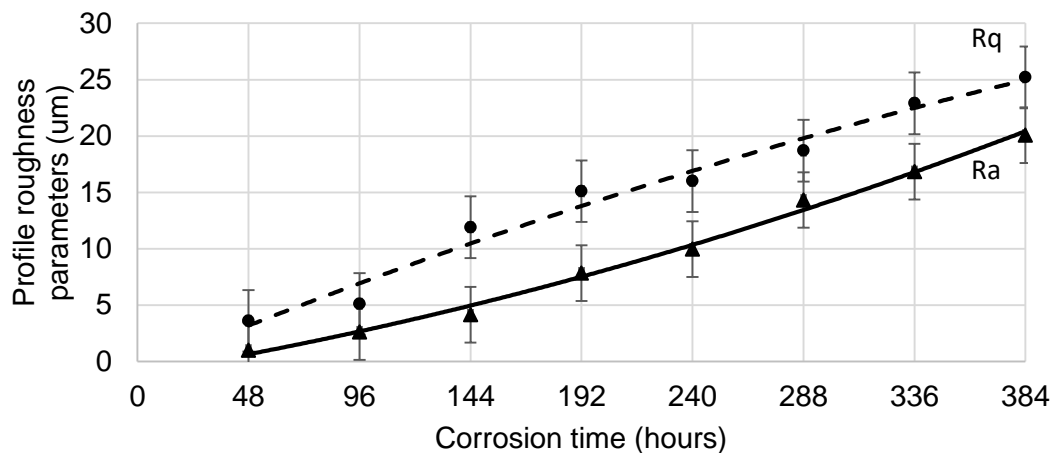


Fig. 4. Profile roughness of X4CrNi18-12 steel heat treatment in 1373 K by 35 minutes and cooling down on water after corrosion tests in boiling 95% nitric acid for different corrosion time: Ra - arithmetical mean roughness value [ $\mu\text{m}$ ], Rq - mean peak width [ $\mu\text{m}$ ]

$$R_a = 6 \cdot 10^{-0.5} t^2 + 0.0338 t - 0.7768 \text{ and } r = 0.9971 \quad (6)$$

$$R_q = 5 \cdot 10^{-0.5} t^2 + 0.0846 t - 1.1245 \text{ and } r = 0.9985 \quad (7)$$

The X4CrNi18-12 steel after heat treatment in 1373 K by 35 minutes and cooling down on water after corrosion tests in boiling 95% nitric acid for 240 hours is presented in Fig. 5.

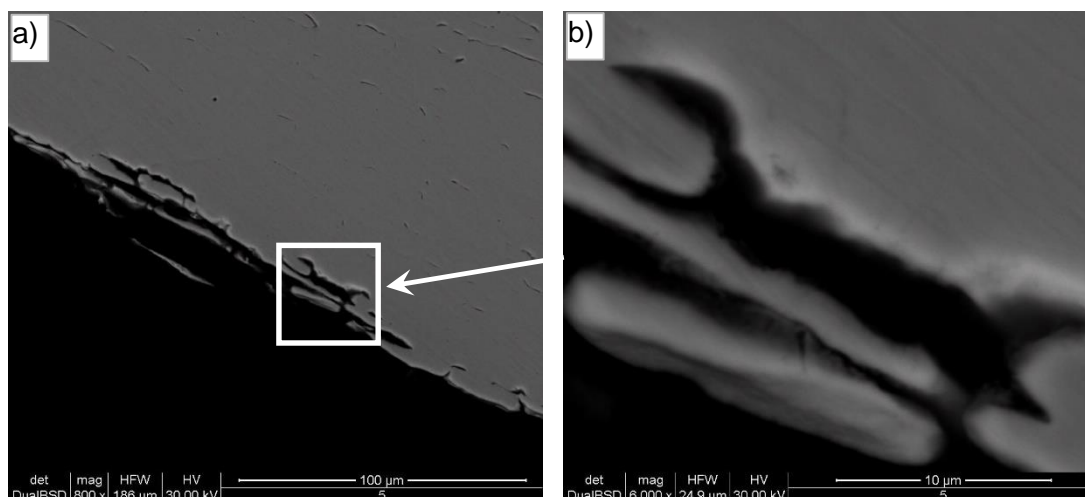


Fig. 5. The surface of X4CrNi18-12 steel after heat treatment in 1373 K by 35 minutes and cooling down on water after corrosion tests in boiling 95% nitric acid for 240 hours

Profile roughness X4CrNi18-12 steel after heat treatment in 1373 K by 35 minutes and cooling down on water after corrosion tests in boiling 95% nitric acid for 240 hours soaking is presented in Fig. 6.

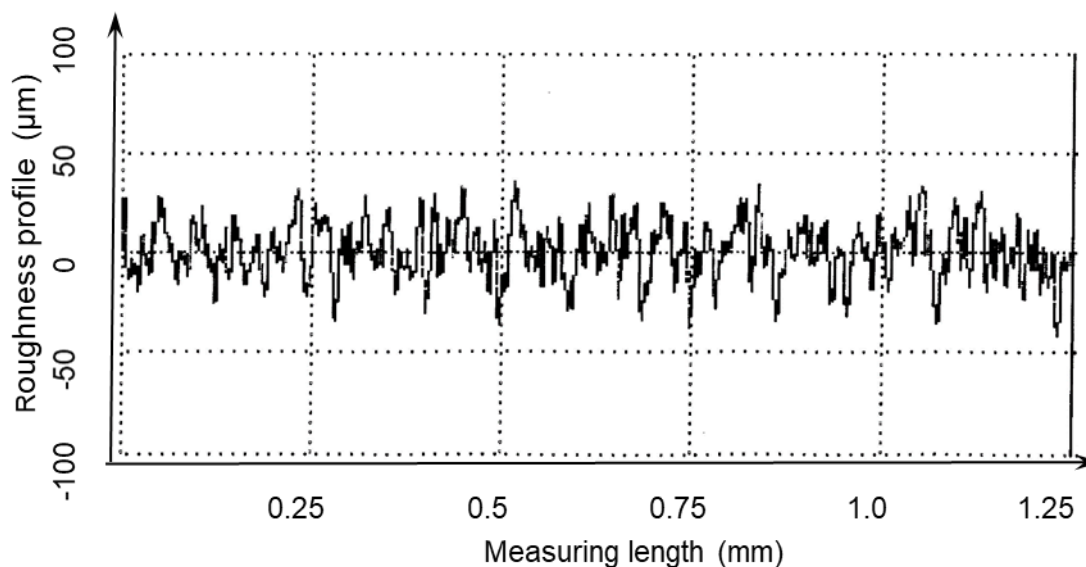


Fig. 6. Roughness profile of X4CrNi18-12 steel after heat treatment in 1373 K by 35 minutes and cooling down on water after corrosion tests in boiling 95% nitric acid for 240 hours

The performed surface roughness tests of steel after 240 hours of corrosion in boiling 95% nitric acid (Fig. 6) do not indicate appearance of subsurface slots (Fig. 5).

#### 4. CONCLUSION

The steel under investigation in the nitric acid environment is resistant to some time. After this corrosion time is exceeded, this steel is not eligible for further use. Obviously this critical time must be determined individually for each corrosive agent, for example by scheduled periodical measurements of surface roughness.

For the corrosion progress determined by the relative mass loss, the curve can naturally be derived from  $t = 0$  and  $RMS = 0$ . For the other two methods for determining the corrosion rate (1) and (2), the change in the corrosion rate in the time interval from 0 to 48 hours is not proportional. Thus, the equations (1) and (2) recommended for determining the corrosion rate by the standard do not reflect the course of corrosion for the first period.

During the first 180 hours of steeping steel in nitric acid, the process is linear. In the time interval of 180-240 hours, the surface of the metal creates gaps, which after washing off the upper layers who are shield for them creates a surface development, which significantly accelerates the course of the corrosion process. Until the top shield of the gaps has been removed, the roughness tests show no disturbing changes.

These results appear to be interested in heavy-duty machines applications, especially in hydraulic components (Pobedza and Sobczyk, 2013; Krawczyk and Sobczyk, 2018), where typical medium is chemically aggressive (Fabis-Domagala et al., 2018; Domagala et al., 2018a; Domagala et al., 2018b).

The obtained results may be also interesting for more exotic equipment applications like in phytochemistry (Skrzypczak-Pietraszek and Pietraszek, 2009) and biotechnology (Skrzypczak-Pietraszek and Pietraszek, 2012; Skrzypczak-Pietraszek et al., 2018a; Skrzypczak-Pietraszek et al., 2018b) where environments may be very aggressive.

It should be also inspiring for researchers focused on protective coatings (Włodarczyk et al., 2011; Bara et al., 2016; Korzekwa et al., 2018; Radek et al., 2018), especially in implants (Dudek and Włodarczyk, 2010; Dudek, 2011) and surface layers modifications (Skoneczny et al., 2007; Bara et al., 2009; Kmita and Bara, 2012; Skoneczny et al., 2018; Szczotok et al., 2018; Bara et al., 2019) investigated by image analysis methods (Gadek-Moszczak et al., 2014; Gadek-Moszczak et al., 2015).

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