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## CORROSION ANALYSIS OF THE X2CrNiMoN25-7-4 SUPER DUPLEX STAINLESS STEEL

### ANALIZA KOROZJI STALI ODPORNEJ NA KOROZJĘ SUPER DUPLEX X2CrNiMoN25-7-4

#### Abstract

Super duplex stainless steels present excellent corrosion resistance of austenite steel and a high mechanical behaviour of ferrite steel. However, performance presented by super duplex stainless steels can be drastically reduced if undesirable phases, such as sigma phase, chi phase, secondary austenite and a lot of rich chromium and carbides precipitate. The purpose of this work was to ascertain how 30-minutes isothermal heat treatments at 530°C and corrosion time effect the relative mass loss and profile roughness parameters of the X2CrNiMoN25-7-4 super duplex stainless steel. The influence of boiling nitric acid on the steel corrosion resistance was investigated using weight loss and roughness parameters.

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

#### Streszczenie

Stale super duplex posiadają doskonałą odporność na korozję dziedziczną ze stali austenitycznych i wysokie właściwości mechaniczne właściwe dla stali ferrytycznych. Jakkolwiek właściwości stali odpornej na korozję super duplex mogą ulegać gwałtownemu obniżeniu wraz z powstaniem niepożądanych faz, takich jak faza sigma, faza chi, austenit wtórny i wielu innych wydzieleni bogatych w chrom, jak również węglików. Celem tego artykułu było określenie, jak 30 minutowa izotermiczna obróbka cieplna w temperaturze 530°C i czas przetrzymywania materiału w ośrodku korozyjnym wpływa na względny ubytek masy i parametry chropowatości stali odpornej na korozję super duplex X2CrNiMoN25-7-4. Badania wpływu wrzącego kwasu azotowego na odporność korozyjną badano, wykorzystując ubytki masowe i parametry chropowatości.

*Słowa kluczowe: stale odporne na korozję, stale duplex, korozja, szybkość korozji, chropowatość*

**DOI: 10.4467/2353737XCT.16.118.5729**

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## 1. Introduction

Duplex stainless steels are among the most popular construction materials. They are used in a wide range of industrial applications, but their properties are continuously studied to improve their qualities. Low maintenance costs, very high material circulation and good environmental reasons are further important arguments for enhancing the selection of these steels. For the most part, the corrosion resistance of a welded joint is slightly lower than the parent material. It is of great importance to have duplex steels readily available to fabricators and end users. Because of this skilled technical support, their well known properties in different temperatures are required to widen the application areas of duplex steels [1–9].

The microstructure and utility properties of steels are determined by phase transformation during its thermal processing [7]. While the phase relationships during the manufacturing process are user-independent, the range of stability or volume fraction of each phase depend of individual maintenance (mainly thermal conditions). The percentage of each phase depends on the composition, technological processes, heat treatments and critical cooling rate obtained by means of the manufacturing method applied [11–12].

Duplex steels are more prone than austenitic steels to the precipitation of phases causing embrittlement and reduced corrosion resistance. The formation of intermetallic phases such as sigma phase occurs in the temperature range of 600–950°C and the reformation of ferrite occurs in the 350–525°C range (475°C embrittlement). However, the performance presented by super duplex stainless steels can be drastically reduced if undesirable phases, such as sigma phase, chi phase, secondary austenite and a lot of rich chromium and carbides precipitate. The sigma phase is rich in chromium and molybdenum and is formed by ferrite decomposition, in the temperature of over 500°C. In normal alloying, heat-treatment or welding processes, the risk of embrittlement is not too high [6]. However, a risk exists for example in a failure that can arise during its operation causing overheating, especially if the cooling is slow. Generally, the higher the superheating temperature the higher the ferrite content. However, steel must be heated to a very high temperature to become completely ferritic. Then heat treatment process for both solution annealing and stress relieving is advisable at certain temperatures with subsequent rapid cooling in water [1, 6]. A lot of authors report that corrosion resistance of stainless steels depends on rich chromium precipitates in microstructure [1, 2, 12].

The purpose of this work was to ascertain how 30-minutes isothermal heat treatments at 530°C and corrosion time affect the relative mass loss and profile roughness parameters of the X2CrNiMoN25-7-4 super duplex stainless steel.

## 2. Materials and Methods

The experiment was performed with the super duplex stainless X2CrNiMoN25-7-4 steel. The chemical composition of the X2CrNiMoN25-7-4 steel is presented in Table 1.

Table 1

**Chemical composition of the X2CrNiMoN25-7-4 steel**

Meanchemicalcompositions [wt. %]									
C	Si	Mn	P	S	Cr	Mo	Ni	Cu	N
0.02	0.29	0.5	0.02	0.0005	25.37	3.74	6.82	0.17	0.269

Before experiments, the specimens with the area of 13 cm<sup>2</sup> (4 × 1 × 0.5 cm) were successively polished with 400 grades of emery paper and mechanically cleaned with 95% alcohol.

The samples were held at the temperature of 530°C for 30 minutes and cooled down in the open air, in accordance with the PN EN ISO 3651-1 standard. Determination of stainless steel resistance to intergranular corrosion. Part 1: Austenitic and ferritic-austenitic (duplex) stainless steels. Corrosion test in nitric acid medium by the measurement of loss in mass (the Huey test), corrosive media were represented by the boiling V 65% nitric acid.

The samples of X2CrNiMoN25-7-4 steel (about 10 mg) were analyzed by means of the Dynamic Scanning Calorimetry measurement by NEITSH DSC204 F1 Phoenix and DSC/dt in a nitrogen atmosphere (with the constant flow of 20 ml/min) by means of Neitsch-Proteus 5.1 software. DSC measurements were carried out in the temperature range of 20–-610°C and with the heating rate of 10°C/min.

The corrosion rate of the X2CrNiMoN25-7-4 steel measured in mm/year was calculated by means of formula (1), but measurements in g/m<sup>2</sup> were calculated by means of formula (2):

$$r_{\text{corn}} = 8760 \text{ m/Std} \quad (1)$$

$$r_{\text{corg}} = 10000 \text{ m/St} \quad (2)$$

where:

- m – average mass loss in the boiling solution [g],
- S – surface area of the sample [cm<sup>2</sup>],
- t – time of treatment in the corrosive solution of a boiling nitric acid [hours],
- d – sample density [g/cm<sup>3</sup>].

The influence of boiling nitric acid on the X2CrNiMoN25-7-4 steel corrosion resistance was investigated using the loss of weight. The mass of samples was measured by Kern ALT 3104AM general laboratory precision balance with the accuracy of measurement 0.0001 g.

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

### 3. Results

The Dynamic Scanning Calorimetry curves of heating measurement from 400 to 600°C (according to literature – embrittlement temperature of 475°C) the super duplex steel for heating rate 10°C/min is presented as an example in Fig. 1.

The endothermic peak (Fig. 1) involving 8.155 J/g of reaction enthalpy with the Tonset at about 465°C,  $T_{end}$  at about 566°C and  $T_{peak}$  about 532°C according to literature [3, 4] represents dissolving of chromium-rich  $\alpha$ -phase. The reformation of ferrite which occurs in the range of 350–525°C is determined as the 475°C embrittlement. For this sample Cp is about 0.146 J/(gK).

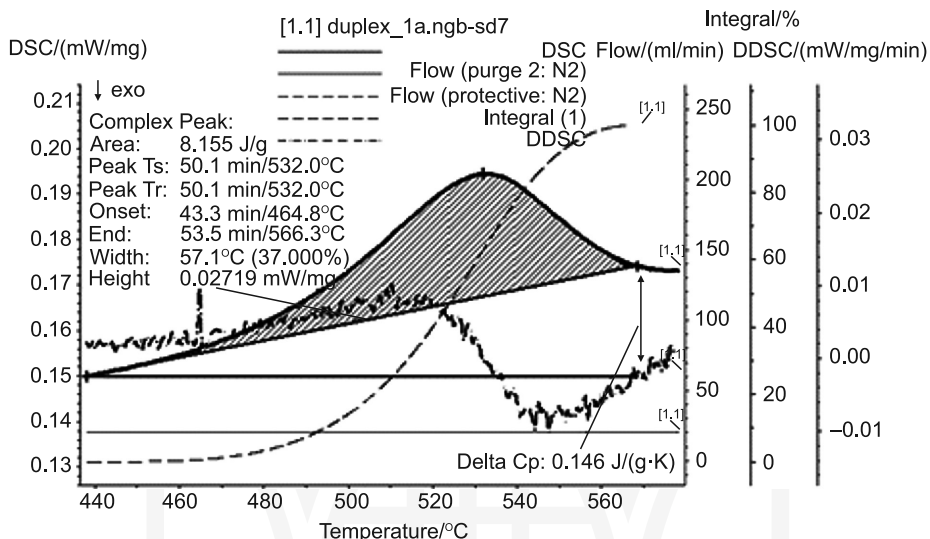


Fig. 1. DSC and DDSC curves of heating measurement the X2CrNiMoN25-7-4 steel. Heating rate 10°C/min

Profile roughness parameters of X2CrNiMoN25-7-4 steel with:  $R_a$  – arithmetic average of absolute values [ $\mu\text{m}$ ],  $R_p$  – maximum peak height [ $\mu\text{m}$ ],  $R_q$  – root mean squared [ $\mu\text{m}$ ],  $R_t$  – Maximum Height of the Profile [ $\mu\text{m}$ ] after corrosion tests in boiling  $\text{HNO}_3$  for different boiling time is presented in Fig. 2, regression equation and correlation coefficient  $r$  at (3)–(6).

$$R_a = 0.0055t + 2.37 \quad \text{and} \quad r = 0.95 \quad (3)$$

$$R_p = 0.0002t^2 - 0.057t + 23.34 \quad \text{and} \quad r = 0.82 \quad (4)$$

$$R_q = 0.0074t + 2.94 \quad \text{and} \quad r = 0.96 \quad (5)$$

$$R_t = 0.0002t^2 - 0.057t + 23.52 \quad \text{and} \quad r = 0.88 \quad (6)$$

Percentage effects of corrosion time on the relative mass loss (RML) of X2CrNiMoN25-7-4 steel annealed at 530°C for 30 minutes and cooling down in the open air are presented in Fig. 3, regression equation and correlation coefficient  $r$  at (7).

$$\text{RML} = 0.21t + 18.3 \quad \text{and} \quad r = 0.98 \quad (7)$$

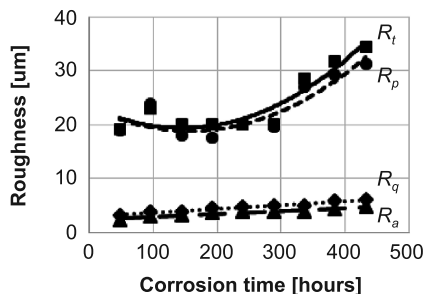


Fig. 2. Profile roughness parameters of X2CrNiMoN25-7-4 steel annealed at 530°C for 30 minutes and cooling down in the open air after corrosion tests in boiling HNO<sub>3</sub> for different boiling time

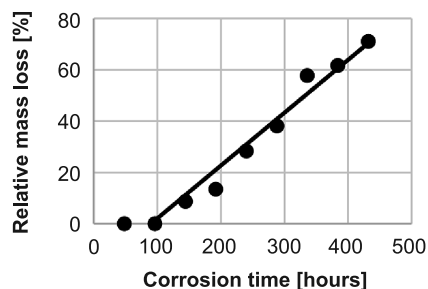


Fig. 3. Percentage effects of corrosion time on the relative mass loss (RML) of X2CrNiMoN25-7-4 steel annealed at 530°C for 30 minutes and cooling down in the open air

Effects of corrosion time on the corrosion rate measured in mm per year of X2CrNiMoN25-7-4 steel annealed at 530°C for 30 minutes and cooling down in the open air are presented in Fig. 4, regression equation and correlation coefficient  $r$  at (8).

$$r_{\text{corr}} = 0.062t - 2.69 \quad \text{and} \quad r = 0.96 \quad (8)$$

Effects of corrosion time on the corrosion rate measured in gram per m<sup>2</sup> of X2CrNiMoN25-7-4 steel annealed at 530°C for 30 minutes and cooling down in the open air are presented in Fig. 5, regression equation and correlation coefficient  $r$  at (9).

$$r_{\text{corr}} = 0.056t - 2.46 \quad \text{and} \quad r = 0.96 \quad (8)$$

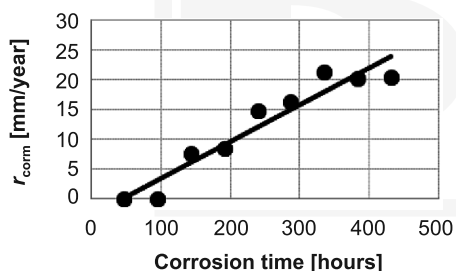


Fig. 4. Effects of corrosion time on the corrosion rate measured in mm per year of X2CrNiMoN25-7-4 steel annealed at 530°C for 30 minutes and cooling down in the open air

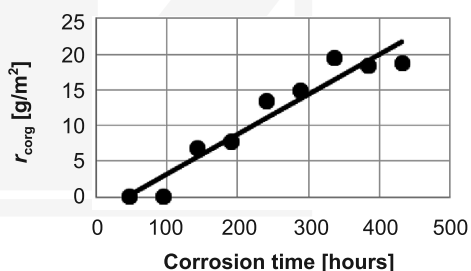


Fig. 5. Effects of corrosion time on the corrosion rate measured in gram per m<sup>2</sup> of X2CrNiMoN25-7-4 steel annealed at 530°C for 30 minutes and cooling down in the open air

#### 4. Conclusions

1. Annealing duplex steels in temperatures from 465°C to 565°C causes dissolving of chromium-rich  $\alpha'$ -phase.
2. The results of the tests indicate that the loss of weight of X2CrNiMoN25-7-4 steel annealed at 530°C for 30 minutes and cooling down in the open air is proportional to the time of corrosion.
3. Profile roughness parameters such as  $R_a$  and  $R_q$  increase with the increase of the time of corrosion process while  $R_t$  and  $R_p$  in the first stage of corrosion did not change and in the second period increased exponentially.
4. Based on the profiles of roughness parameters every research study can determine the size of duplex steel corrosion.

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