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THE INFLUENCE OF MOLIBDENUM ON CORROSION RESISTANCE OF SINTERED AUSTENITIC STAINLESS STEELS

WPŁYW MOLIBDENU NA ODPORNOŚĆ NA KOROZJĘ SPIEKANYCH AUSTENITYCZNYCH STALI NIERDZEWNYCH

Abstract

Molybdenum was introduced into sintered austenitic stainless steels to improve their corrosion resistance. The influence of different Mo contents on the pitting corrosion resistance of AISI 304L and 316L steels in 0.1 M sodium chloride solution has been investigated. The corrosion behaviour was evaluated by potentiodynamic polarisation method. Sintered Mo-containing 304L steel is less prone to pitting corrosion in environment containing chlorides than 316L with Mo addition. The introduction of molybdenum to 304L steel resulted in an improvement of corrosion resistance. This steel exhibits the highest value of polarisation resistance as well as the lowest value of corrosion rate.

Keywords: *sintered austenitic stainless steel, molybdenum addition, pitting corrosion, potentiodynamic polarization*

Streszczenie

Molibden wprowadzono do austenitycznych stali nierdzewnych w celu poprawy ich odporności na korozję. Badano wpływ różnej zawartości molibdenu na odporność na korozję wżerową stali AISI 304L i 316L w 0.1 M roztworze chlorku sodu. Zachowanie korozyjne oceniano metodą polaryzacji potencjodynamicznej. Spiekana stal 304L zawierająca molibden jest mniej podatna na korozję wżerową w środowisku zawierającym chlorki niż 316L z dodatkiem Mo. Wprowadzenie molibdenu do stali 304L spowodowało poprawę odporności na korozję. Stal ta wykazuje najwyższą wartość oporu polaryzacji, jak i najniższą wartość szybkości korozji.

Słowa kluczowe: *spiekana austenityczna stal nierdzewna, dodatek molibdenu, korozja wżerowa, polaryzacja potencjodynamiczna*

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1. Introduction

Stainless steel is the common name of a large family of steels that are resistant to destruction under the influence of chemical or electrochemical reaction with the surrounding environment. Corrosion resistance of stainless steels is the result of the passive state. In order to ensure corrosion resistance, the content of chromium should be at least about 11% wt. [1–5]. Due to presence of this element in chemical composition of stainless steel, a thin, chemically stable, invisible, durable, extremely adherent, self-healing, and passive film of chromium oxide is formed on the surface of steel [3, 5–8]. This film is formed in oxidising environments, whereas steel may be subjected to corrosion in anaerobic environments and in the presence of aggressive ingredients (chlorides, sulphur compounds). Then corrosion can lead to perforation, reduction in mechanical strength, deterioration of the surface appearance or contaminant of the manufactured products [7]. When steels contain chromium in an amount of more than 13 weight %, they are resistant to corrosion in oxidising environments, such as nitric acid. These steels are definitely less resistant in reducing environments, such as hydrochloric acid or sulphuric acid. It is well known that higher content of chromium must be added to protect against pitting and rusting in more hostile environments than, for instance, relatively pure and dry air [3, 7]. Because of austenitic phase stability considerations, the content of chromium in austenitic stainless steels is generally kept in the range of 17–18 weight % [3–4].

Besides chromium and nickel, modern austenitic stainless steels contain other alloying elements. For example, the introduction of molybdenum addition to stainless steel improves the corrosion resistance of the passive layer in the presence of chloride ions (derived from chemicals, road salt, sea water, etc.), so steels with the addition of this element have a higher resistance to pitting and crevice corrosion than conventional chromium-nickel steels [3].

The AISI 300 series includes austenitic stainless steels containing chromium and nickel. The chromium-nickel steels are mostly resistant to electrochemical corrosion in an environment of inorganic and organic acids, nitrogen compounds, salt solutions and aggressive food products. They have excellent corrosion resistance in diverse environments, but their mechanical properties are not satisfactory in some applications. Moreover, their tribological properties are rather poor. Chromium-nickel steels exhibit a high coefficient of friction and low wear resistance. Austenitic stainless steels are non-magnetic [2–4, 7]. The most commonly used steels are 304L and 316L. They belong to the group of austenitic stainless steels. The chemical composition of both these grades is similar. It should be pointed that the main difference between them is the absence of molybdenum in 304L. Thanks to the content of molybdenum, corrosion resistance of 316L steel is increased compared to 304L steel (especially pitting corrosion in the presence of chlorides) [7]. Generally, the addition of molybdenum increases corrosion resistance of wrought austenitic stainless steels. It lowers the critical passivation current and the current in the area of passive, shifts pitting potential towards positive values, raises the temperature of the critical pitting, and also reduces the number and size of pitting [7, 9–12]. Because of excellent corrosion resistance, 316L steel is used for equipment in the food industry and marine industry, for surgical implants, the nuclear fuel processing installations [4, 7].

Sintered austenitic stainless steels are increasingly used in many industrial branches, for instance, in the automotive industry, electromechanical industry or in medicine. They are used as components of engines, drive systems, braking systems, exhaust systems or suspensions (for example, fuel pumps, self-lubricating bearings, brake pads, oxygen concentration sensors or ABS sensors). In medicine, they are used as bone implants and elements of the hocks endoprostheses [13].

P/M stainless steels are usually solid-state sintered and have usually worse mechanical properties and corrosion resistance as compared to their wrought counterparts. This is mainly due to inherent porosity [4, 8, 14–16]. The austenitic stainless steels fabricated by powder metallurgy are economically attractive because there are no metal losses during machining and finishing [17].

Corrosion behaviour of sintered stainless steels is dependent on many factors, such as: their chemical composition (effect on the durability and quality of the passive layer, assurance of corrosion protection); the state of the surface; the interconnected pores (which considerably increase the total reactive surface); the morphology of the pores; and others [6, 13, 17–19]. It is known that low porosity and the presence of intermetallic precipitation, compounds, or phases, do not favour the corrosion resistance of stainless steels. It has been reported that precipitation of undesired phases may cause drastic decrease of mechanical properties as well as corrosion resistance [3, 4, 13, 14].

In recent years, it can be seen the development of sintered austenitic stainless steels towards an improvement of corrosion resistance through variation in the compacting pressure, sintering parameters (temperature, time, atmosphere), heating mode (conventional, microwave) or use of alloying additions (such as copper, boron, phosphorus), which favour liquid phase sintering [13–19]. Besides corrosion resistance, the attention has been directed towards improvement of density, the hardness and mechanical properties of these steels.

It has been reported that the increase of sintering time caused slight improvement of strength, however, it led in grain growth and particle coarsening [17]. Whereas the higher sintering temperature, the greater corrosion resistance. Steels sintered in a hydrogen atmosphere avoid any chromium depletion phenomena.

Many studies have investigated on the corrosion behaviour of austenitic stainless steels [4, 6, 7, 9–22]. The effect of reduction of anodic area on the surface of the modified sintered austenitic stainless steel in neutral media (with and without chlorides) has been observed. It caused a decrease in the i_{corr} and an increase in E_{corr} [17].

Pardo et al. [9, 12] investigated the effect of introduction of Mo and Mn additions to AISI 304 and AISI 316 on the pitting corrosion resistance in chloride-containing media. They found out that Mo additions slightly shifted the corrosion potential to more noble values and increased the pitting corrosion resistance. It has been reported that the presence of Mo modified the passive film on the surface of steel. This film rendered more stable against breakdown because of the attack of aggressive Cl^- ions. The Mo addition significantly improved the repassivation behaviour [9–12].

The austenitic stainless steel type Cr18Ni10 with different molybdenum content (in the range up to 0.13 to 6.1 wt. %) was investigated to determine the effect of molybdenum on the corrosion and passivation of this steel. The author has proposed the synergistic mechanism of nitrogen-molybdenum interaction [7].

In this study, molybdenum was introduced into austenitic stainless steels (AISI 304L and AISI 316L) to improve their corrosion resistance. These steels were obtained through conventional pressing and sintering. The corrosion behaviour of austenitic stainless steels (with and without molybdenum addition) were investigated. The influence of different Mo contents on the pitting corrosion resistance of AISI 304L and AISI 316L steels in 0.1 M sodium chloride solution has been investigated. The corrosion behaviour was evaluated by potentiodynamic polarization method. The electrochemical behaviour has been correlated with the densification.

2. Materials for research

Commercially available AISI 316L and AISI 304L (corresponding with standard UNS S31603 and S30403, respectively) stainless steel powders (provided by Höganäs) were used in the study. These powder grades were produced by water atomisation. The chemical compositions of these powders are listed in Table I. The physical properties of used powders are given in Table 2. The powders of both grades had a nominal particle size of < 150 µm.

Table 1

Chemical composition (% wt.) of tested powders

Powder grade	Cr	Ni	Mo	Si	Mn	C	Fe
AISI 316L	16.8	12.3	2.2	0.8	0.12	0.02	bal.
AISI 304L	18.5	11.2	–	0.8	0.14	0.02	bal.

Table 2

Properties of stainless steels powders (according Höganäs)

Powder grade	Flow [s/50g]	Apparent density [g/cm^3]
AISI 316L	31	2.67
AISI 304L	31	2.88

Molybdenum in the form of elemental powder (product of Sigma-Aldrich, average particle size of 10 µm, purity of 99.95%) was used. The powders mixtures of the austenitic stainless steels with different molybdenum content in the range from 0 to 4.4% were prepared using the powders of AISI 304L, AISI 316L and molybdenum (Table 3).

Table 3

Components of powder mixtures used in this research work

Designation	304L	304L/316L	316L	304L Mo	316L Mo
Base powder	AISI 304L	AISI 304L, AISI 316L	AISI 316L	AISI 304L	AISI 316L
Mo additive	no	no	no	yes	yes
Mo [%]	0	1.1	2.2	4.4	4.4

3. Experimental procedure

All powder mixtures were prepared by mixing in Turbula. The time of mixing was 120 minutes. Then, the powders were uniaxial pressed in a rigid matrix at a pressure of 600 MPa. In this way, the cylindrical samples of ∅20×5 mm size were obtained. The zinc stearate was used to minimise the friction along the walls of die. These samples were sintered in Nabertherm furnace in pure (99.9992%), dry (dew point below –60°C) hydrogen atmosphere. The temperature of isothermal sintering was 1240°C. The sintering time was 45 minutes. The samples were slowly heated to the sintering temperature at a rate of 10°C/min. The same rate was applied during cooling from the sintering temperature to the ambient temperature.

Green and sintered densities of investigated steels were determined. For compacted specimens, the density measurements were carried out by geometrical method. Whereas the density and porosity of sintered samples were measured by the water-displacement method, in accordance with Standard PN-EN ISO 2738:2001.

To estimate the sinterability, the densification parameter (DP) was used. The DP is calculated as follows [16]:

$$\text{DP} = (\text{sintered density} - \text{green density})/(\text{theoretical density} - \text{green density}) \quad (1)$$

Metallographic cross sections were prepared. The microstructural study of the sintered steels was done with Nikon Eclipse ME 600P Light Optical Microscopy and SEM microscope JSM550LV produced by Joel.

Corrosion resistance test of investigated stainless included open-circuit potential and potentiodynamic polarization measurements. They were performed using the ATLAS 0531 Electrochemical Unit (ATLAS – SOLLICH), controlled by AtlasCorr05 software. During the electrochemical measurements, platinum was used as the counter electrode, saturated calomel electrode (SCE), as the reference electrode, and austenitic stainless steels samples as the working electrode. All potentials were measured versus SCE. Before starting the measurement, the surface of the working electrode was grinded (600 grade of silicon carbide paper), then washed by distilled water, degreased in acetone and dried in air. Corrosion behaviour of the sintered steels was investigated in 0.1 M NaCl solution at room temperature. The open circuit potential was measured in function of immersion time (about 3 hours). When the open circuit potential became stabilised, the potentiodynamic polarisation was performed. A polarisation curve was obtained at a potential scan rate of 1.0 mV/s from – 0.8 V to + 1 V. The corrosion current density (i_{corr}), corrosion potential (E_{corr}), cathodic Tafel slope (b_c), and anodic Tafel slope (b_a) were determined from the Tafel plot (potential versus logarithm of corrosion current density). The electrochemical parameters such as, polarisation resistance (R_{pol}) and corrosion rate were determined based on polarisation curve. The polarisation resistance was evaluated using Stern method as well as Stern-Geary method. According to standard ASTM G 102, the polarisation measurements can be used to calculate the corrosion rate, either in terms of penetration rate (*CR*) or mass loss rate (*MR*).

PREN (pitting resistance equivalent number) is commonly used to compare the corrosion resistance of various types of stainless steels in a chloride-containing environment. The pitting corrosion resistance of stainless steel depends on its chemical composition. Because elements such as chromium, molybdenum, and nitrogen have significant influence on pitting resistance, the most frequently used formula of PREN is following:

$$\text{PREN} = 1\%\text{Cr} + 3.3\%\text{Mo} + 16\%\text{N} \tag{2}$$

It is well known that the higher value of PREN, the greater corrosion resistance of stainless steel.

4. Results and discussion

The calculated values of densification parameter and relative density of sintered austenitic steels are presented in Fig. 1. Fig. 2 shows the results of measurements of an open and closed porosity obtained for all investigated steels.

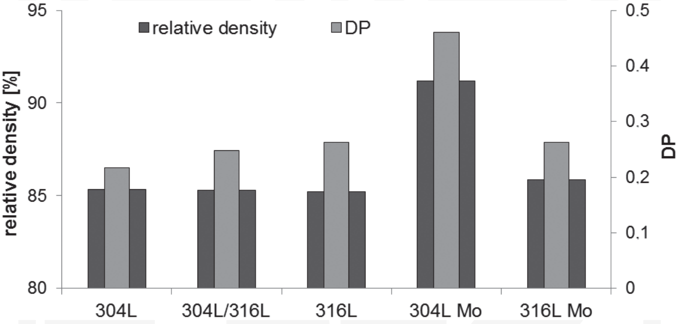

Fig. 1. Relative density and densification parameter (DP) of tested stainless steels

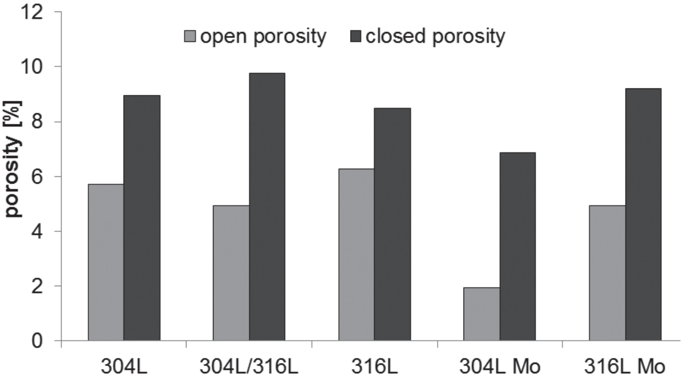

Fig. 2. Open and closed porosity of tested stainless steels

It can be generally seen that the value of densification parameter increases with increasing of molybdenum content in investigated steels. Furthermore, the DP value of 316L Mo steel is significantly lower than 304L Mo for the same amount of molybdenum in chemical composition. It should be pointed that the compressibility of 316L is lower than 304L. It was similar when molybdenum was added to these powders of steel. The highest relative density and the lowest values of open and closed porosity were obtained in the case of 304L with molybdenum addition. The relative density of 304L/316L steel (made with the mixture of 304L and 316L powders) remained at a similar level as sintered 304L and 316L steels.

The variations in the open circuit potential were monitored for all investigated steels (immersed in 0.1 M NaCl solution). The obtained results are presented in Fig. 3. Based on these results, values of open circuit potential were determined at the beginning of the test as well as after 3 hours of immersion in 0.1 M NaCl solution. They are presented in Table 4.

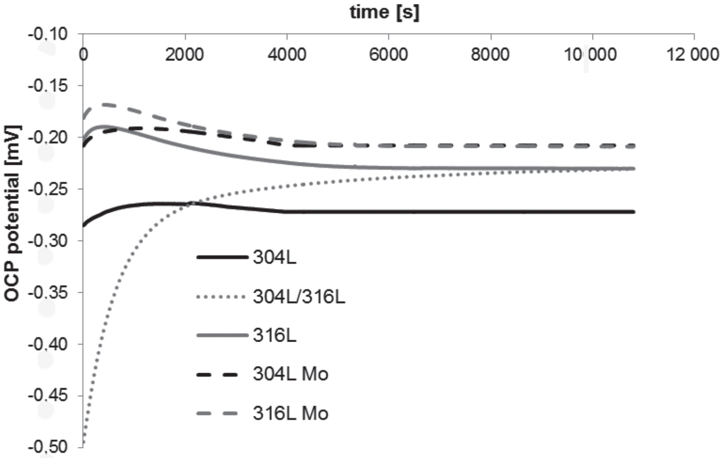

Fig. 3. Variation of OCP for investigated stainless steels immersed for 3 hours in 0.1 M NaCl solution

Table 4

OCP values of the investigated steels: initially and after 3 hours of immersion in 0.1M NaCl solution

Designation of samples	OCP [V vs. SCE]	
	Initial	After 3 hours
304L	–0.285	–0.272
304L/316L	–0.484	–0.231
316L	–0.191	–0.229
304L Mo	–0.208	–0.208
316L Mo	–0.181	–0.209

It can be observed that the evolution of the OCP with time is virtually the same for tested steels. At the beginning, the OCP showed a tendency to slightly reduce with time. After a few minutes of exposure in 0.1 M NaCl solution, it started to shift towards the lower values, and then it stabilised. Only for 304L/316L steel the evolution of the OCP with time is different. Namely, potential increases all the time during measurement. When OCP potential is shifted with time towards more positive value, it indicates the formation of a protective passivation layer on the surfaces. The steel without molybdenum had the lowest value of OCP. It might suggest that 304L steel has the highest tendency to corrode in the studied medium. The OCP potential of 316L steel is more positive. From the analysis of presented characteristic, it can be generally concluded that higher molybdenum content in steel leads to potential increase (shift to more positive values).

In order to estimate the pitting corrosion behaviour of the sintered stainless steels, potentiodynamic polarisation study in 0.1 M NaCl solution was performed. Fig. 4 shows the registered potentiodynamic curves for the tested steels.

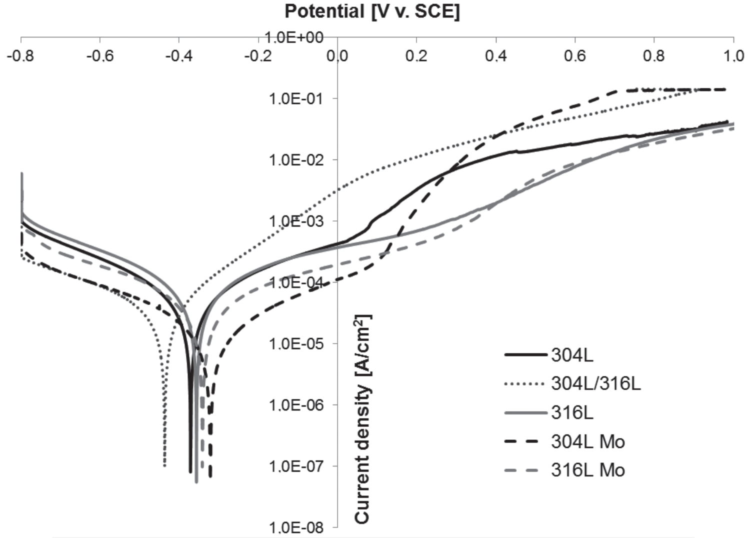

Fig. 4. Potentiodynamic polarisation curves of investigated stainless steels in 0.1 M NaCl solution

The parameters such as corrosion potential (E_{corr}), corrosion current density (i_{corr}), polarization resistance (R_{pol}) as well as corrosion rate (*CR* and *MR*) have been calculated for all investigated steels. The values of these corrosion parameters are summarized in Table 5.

Figure 5 shows the values of polarisation resistance and corrosion rate for the sintered austenitic steels with different Mo contents in sodium chloride solution.

As expected, the corrosion parameters are dependent on the chemical composition of investigated steels. It should be pointed out that there is no linear dependence between molybdenum content in alloy and polarisation resistance (or other corrosion parameters).

Table 5

The values of the corrosion parameters of investigated stainless steels

Designation	Corrosion parameters					
	E_{corr} [V vs. SCE]	i_{corr} [A/cm^2]	R_{pol} [Ω·cm^2]		*CR* [mm/y]	*MR* [g/m^{2}d]
			Stern method	Stern-Geary method		
304L	–0.371	$4.39 \cdot 10^{-5}$	1520	1549	0.539	9.701
304L/316L	–0.437	$3.36 \cdot 10^{-5}$	1834	1936	0.414	7.507
316L	–0.359	$6.87 \cdot 10^{-5}$	1185	1186	0.851	15.359
304L Mo	–0.322	$1.35 \cdot 10^{-5}$	5072	5276	0.157	3.022
316L Mo	–0.342	$2.95 \cdot 10^{-5}$	2065	2104	0.365	6.633

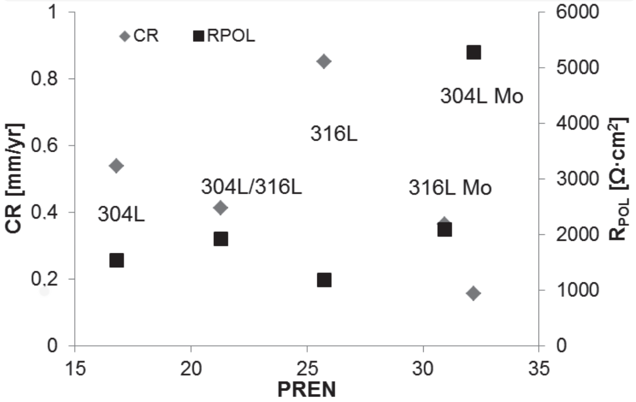

Fig. 5. The dependence of corrosion rate and polarisation resistance on PREN for investigated stainless steels

Generally, the highest value of polarisation resistance as well as the lowest values of *CR* and *MR* parameters were obtained for 304L Mo steel. This alloy is characterised by the highest molybdenum content (4.4% wt.) as well as the total content of elements such as Cr, Ni and Mo. It is no surprise that among all investigated steels 304L Mo has the highest value of the PREN. Therefore, greater corrosion resistance can be obtained in the case of this stainless steel. Although 316L Mo steel exhibits the same molybdenum content, the total content of Cr and Mo is lower in comparison to 304L Mo steel. And therefore, the value of PREN as well as polarisation resistance of this steel is lower while the corrosion rate is higher. As already stated, the introduction of molybdenum to 316L steel did not result in an improvement of the corrosion behaviour as significant as that for 304L with Mo. Although both steels contain molybdenum (in amount of 4.4% wt.) and total content of chromium and nickel is almost the same, they differ in regards to alone chromium content as well as nickel content in chemical composition. It seems that higher chromium content in 304L Mo steel has contributed towards its better corrosion resistance in comparison to 316L Mo.

The obtained results pointed out that the pitting resistance of Mo-containing 304L stainless steel is superior to that of non-Mo-containing one. It can be seen that 316L steel exhibits the lowest polarisation resistance and the highest values of corrosion rate although it contains molybdenum in chemical composition.

Taking into account the corrosion resistance in chloride-containing environment (0.1 M NaCl solution), 304L steel modified molybdenum (4.4% wt.) has turned out to be the best steel.

5. Conclusions

The pitting corrosion resistance plays an essential role in the properties of stainless steels. In this study, the pitting corrosion resistance of austenitic stainless steels with different Mo contents was investigated in 0.1 M sodium chloride solution. Electrochemical corrosion measurement (OCP and potentiodynamic polarisation) have been performed. The influence of molybdenum addition on pitting corrosion resistance of austenitic steels has been studied.

It is generally known that in the case of sintered materials corrosion pits initially are formed in open pores and then proceed into interior of them. That is why the open porosity is an important parameter. In regards to the effect of chemical composition of steel on properties, such as density and open porosity, it can be concluded that 304L and 316L steels with introduced molybdenum addition exhibit higher relative density and lower open porosity than Mo-containing ones. The steel designated as 304L Mo shows the highest density and lowest open porosity.

Considering the influence of molybdenum addition on the pitting behaviour of austenitic steels, it can be concluded that:

- sintered 304L Mo steel is less prone to pitting corrosion than 316L Mo in environment containing chlorides,
- sintered steels (304L, 316L) containing molybdenum behaves better than the steel without Mo. It can be stated that molybdenum additive with presence of chromium and nickel improves corrosion resistance of investigated austenitic steels,
- steel designated as 304L Mo exhibits the highest corrosion resistance in 0.1 M NaCl solution (the lowest values of corrosion rate (*CR* and *MR*) and corrosion current density, the highest values of PREN and polarisation resistance).

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