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Influence of chemical composition on the pitting corrosion resistance of non-standard low-Ni high-Mn–N duplex stainless steels

R. Merello ^{a,1}, F.J. Botana ^{b,*}, J. Botella ^a, M.V. Matres ^a,
M. Marcos ^b

^a ACERINOX, S.A. Departamento Técnico. Apdo. 83, E-11370, Los Barrios, Cádiz, Spain

^b Departamento de Ciencia de los Materiales e Ingeniería Metalúrgica y Química Inorgánica, Facultad de Ciencias del Mar., Universidad de Cádiz, Polígono Río San Pedro s/n Apdo 40, Avda. República Saharaui s/n, E-11510, Puerto Real, Cádiz, Spain

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Abstract

The pitting corrosion resistance of a new family of duplex stainless steels has been evaluated. These non-standard duplex stainless steels are characterised by low Ni content and high N and Mn levels. Potentiodynamic polarisation scans in NaCl solution have been carried out to determine pitting potentials. A crevice-free cell has been used to perform the electrochemical tests.

An exponential equation is obtained in the regression analysis between the pitting potential and chemical composition which allows an estimate of the pitting resistance of these new duplex stainless steels.

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1. Introduction

Duplex stainless steels are characterised by a two-phase structure comprising a mixture of ferrite (α) and austenite grains (γ). The relationship between both phases

* Corresponding author. Tel.: +34-956-016154; fax: +34-956-016140.

E-mail addresses: jbotella@acxgroup.com (R. Merello), javier.botana@uca.es (F.J. Botana).

¹ Tel.: +34-956-629375.

is usually about 50% (by volume) although it can vary between 30% and 70% and more frequently between 40% and 60% [1,2]. These materials have excellent corrosion resistance and higher mechanical strength than austenitic stainless steels (AISI 300 grade).

Duplex stainless steel development has been bound to the demand of more resistant materials in chemical industries and petrochemicals, in those of energy generation, construction of oil platforms and pulp and paper industries [1,3,4]. New duplex grades have appeared, with higher levels of Cr, Mo and N. These materials have an increased localised corrosion resistance and better mechanical properties. Nitrogen is essential in the behaviour of these second-generation duplex stainless steels and in the so-called superduplex steels. When the nitrogen content increases, the pitting corrosion resistance improves and it influences positively the high temperature duplex structure stability, particularly in the heat-affected zones of welds [5].

Some new duplex stainless steels have been designed in the Research Department of Acerinox in order to generate stainless steels with higher features [6]. They have balanced two-phase structures (40% α –60% γ up to 60% α –40% γ) and are highly alloyed with nitrogen.

This new duplex family has lower nickel level (2.5%, in weight), according to the purpose of an economic design. These materials, as with standard duplex stainless steels, can substitute austenitic stainless steels in many applications where high mechanical resistance and enhanced outstanding localised corrosion resistance are required [7].

This paper shows the results of research carried out on the susceptibility of the new duplex family to pitting corrosion. The work has been focused, towards the determination of the experimental conditions that guarantee the validity of measures. The work aims to find a correlation among the susceptibility to pitting corrosion in chloride media and the chemical composition of the prepared duplex.

2. Studied alloys

Thirty-three non-standard low-Ni high-Mn–N duplex alloys have been designed which have the same chemical composition except for Cr, Mo, N and Ni levels. The content of Ni has been used to adjust the ferrite volume fraction to 40–60% after the heat treatment. Table 1 shows the chemical composition range of these new alloys.

A two-fold double objective has been aimed when preparing the materials [6]. These stainless steels should be highly nitrogen alloyed and have a well balanced two-phase structure (40% α –60% γ up to 60% α –40% γ). Nitrogen besides being

Table 1
Compositional range of the experimental stainless steels

% α	Weight percentage								
	Si	Mn	Ni	Cu	Cr	Mo	C	S	N
40–60	0.5	8	0.5–2.7	1	18–24	0.3–4	≤ 0.03	≤ 0.002	0.09–0.34

Table 2
Chemical compositions of standard duplex stainless steels

Materials	Weight percentage								
	Cr	Mo	N	Si	Mn	Ni	Cu	C	S
2304	22.55	0.29	0.0815	0.52	0.97	4.03	0.31	0.023	0.0017
2205	21.06	2.46	0.1011	0.68	1.66	5.52	0.30	0.025	0.0010

essentially economic, significantly retards the formation of harmful intermetallic phases, such as σ , χ , etc, whose development is stimulated by high Cr and Mo levels [9,10]. However, nitrogen alloying has practical difficulties derived from its rather low solubility in austenite and almost nil in ferrite [8,11–13]. On the other hand, it increases the probability of nitride precipitation like that of chromium [15,16]. To improve nitrogen solubility, the chromium level is increased [17,18] and, especially, that of manganese [12,19]. The decrease of the content of an element such as nickel, essentially γ -stabiliser, is compensated with an increase of nitrogen content. Nitrogen is an austenite former and stabiliser [20]. Mn has an unclear role—neither α -former nor γ -former—at rather high levels. Therefore, it does not practically influence phase distribution [21,22]. Nevertheless, it is an important γ -stabiliser and it also contributes to improve nitrogen solubility. Cr levels are similar to those of duplex standard 2205, avoiding higher levels because of the tendency of Cr to stimulate deleterious intermetallic phases. Moreover, Cr increases N solubility and therefore reduces the susceptibility of the materials to pitting corrosion [14,23]. Molybdenum is an essential element for the improvement of pitting corrosion resistance, since it acts synergistically with chromium and nitrogen. Mo content is restricted to <4% to avoid the formation of intermetallic phases [22,23].

Additionally, two standard duplex stainless steels have also been prepared to be used as references, Table 2.

Non-standard low-Ni high-Mn–N and standard duplex stainless steels were prepared as 50 g castings. They were vacuum melted and centrifugally cast in a high frequency induction furnace. The alloys were heat treated at a temperature of 1100 °C for 30 min.

3. Experimental

Potentiodynamic polarisation scans were used to evaluate the alloys pitting corrosion susceptibility. A PC driven EG&G PAR model 263A potentiostat was used to perform these electrochemical tests. A crevice-free cell based on the Avesta design was used. The advantage of this cell is that permits the accurately determination of pitting potential, since it avoids the interference from crevice corrosion. This type of localised corrosion can take place at the seal between the specimen and the *o*-ring.

The fundamental principle of this cell is the continuous rinsing of the area of contact between the sample and the *o*-ring, using a small flow of distilled water,

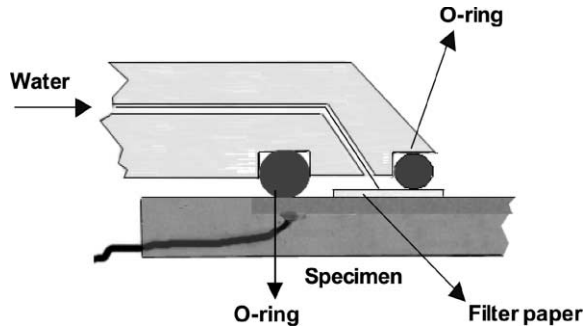


Fig. 1. Typical structure of a low-Ni duplex stainless steels.

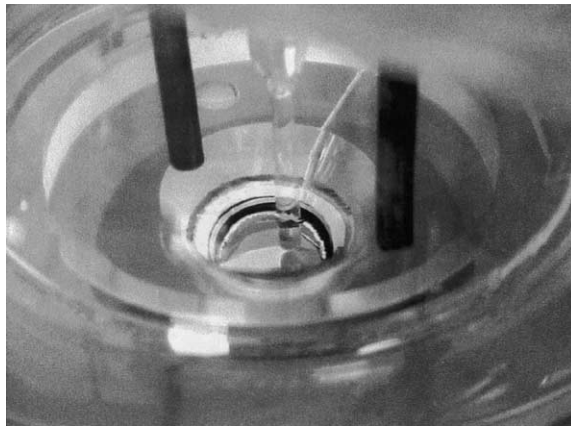


Fig. 2. Scheme of the electrochemical cell.

Fig. 1. This way, the concentration of aggressive ions and the acidification process inside the crevice is avoided [24] (Fig. 2).

Distilled water was conducted to the cell through a plastic tube, using a peristaltic pump. Water was pumped to a circular chamber in the base plate of the cell, limited by two concentric *o*-rings. A small disc of filter paper (Whatman 44) was located between the specimen and the *o*-rings, allowing a continuous circulation of the distilled water in the crevice flowing and it flows towards the interior of the cell, Fig. 3. Once the water is inside, it flows immediately upwards because of its lower density.

Most authors [25] suggest that a water flow of 4–5 ml/h is enough to avoid the concentration of aggressive ions in the crevice. With this small volume of water, the test solution is not significantly diluted. Qvarfort [26] documented that the pitting potential in 0.63 M NaCl solution is independent of the distilled water flow rate in the range 1–12 ml/h. If the specimen is too tight to the cell, a continuous flow of

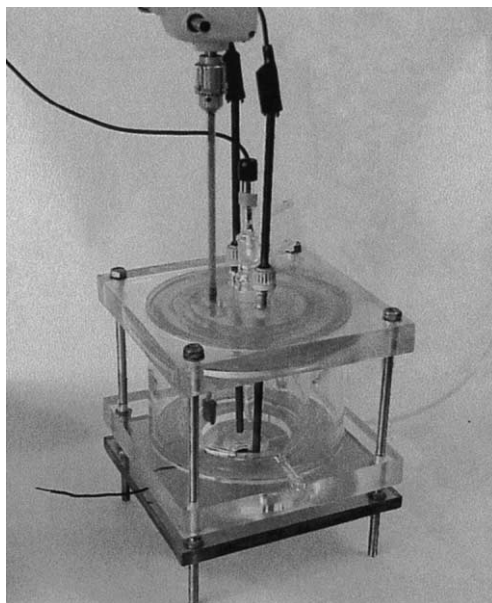


Fig. 3. Details of the cell design.

water towards the interior is difficult. This could lead to stagnant areas under the filter paper, causing aeration cells and therefore crevice corrosion. If the water flow is too high, the test surface can be flushed by water and would inhibit pitting corrosion.

Another effect attributed to the presence of distilled water, is the contribution to the anodic current coming from the part of the specimen located under the filter paper. However, since this current corresponds to the passive current in a non-aggressive environment (distilled water), the effect would be seen only at very low current densities. This means that the detection of pitting is unaffected [24]. Considering all these factors, it has been experimentally determined that the best results are obtained using a water flow of approximately 5–6 ml/h.

Additionally, the cell has a water jacket through which water can be circulated from a thermostat bath, Fig. 3, which allows carrying out tests at different temperatures. A saturated calomel electrode (SCE) is used as a reference and the counter electrodes are two graphite bars.

The specimens were mounted in epoxy resin and the test surface (1 cm^2) was wet ground with 600 grit abrasive paper immediately before immersion. The electrolyte was a 35 g/l NaCl solution at 50°C which was mechanically stirred and deaerated with pure nitrogen (0.8 l/min).

Before starting the test, the specimen was immersed in the deaerated solution for 30 min so that the open circuit potential was stabilised. Then, the specimen was cathodically polarised to -1300 mV (SCE) for 3 min. The potential scan began at -1100 mV (SCE) with a scan rate of 0.17 mV/s .

4. Results and discussion

Potentiodynamic polarisation curves were obtained in sodium chloride solution for all the prepared alloys. Fig. 4 shows an example of the curves obtained for one of the alloys. The pitting potential, E_p , is defined in terms of the potential at which the current density has shown an increase, typically $100 \mu\text{A}/\text{cm}^2$ [27,28]. After the test, the specimens were observed in an optical microscope to check pitting attack.

Fig. 5 shows the E_p values obtained for some non-standard low-Ni high-Mn-N duplex alloys in 50 g/l NaCl solution at different temperatures. These results show

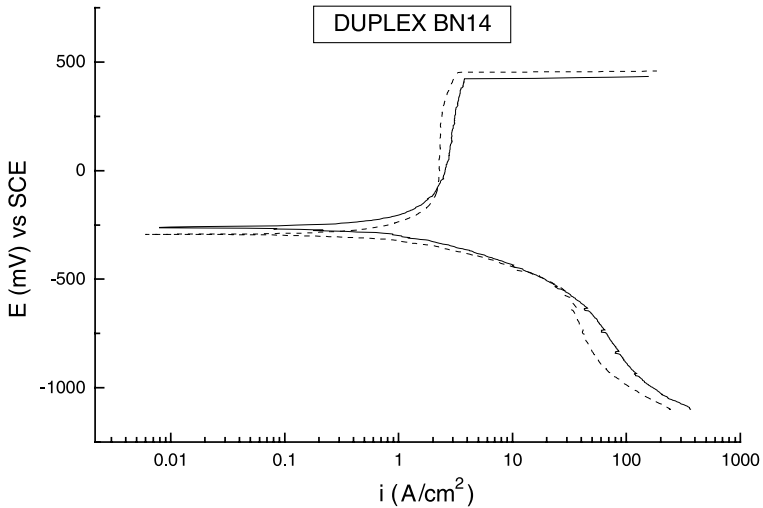


Fig. 4. Polarisation curves of BN14 specimen in NaCl solution.

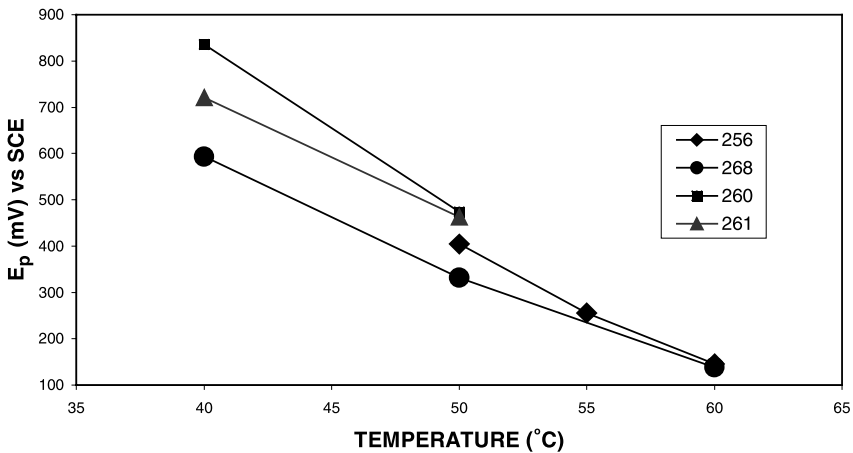


Fig. 5. Variation of E_p with temperature.

that as has been reported for other stainless steels [16,23], these new alloys are less pitting corrosion resistant as temperature increases.

Some authors [29,30] have tried to correlate pitting corrosion resistance with the chemical composition of stainless steels. It has been suggested that pitting corrosion depends basically on the content of Cr, Mo and N. According to this hypothesis, pitting resistance equivalent number (PREN) is defined by the following Eqs. [31, 32]:

$$\text{PREN}_{16} = \%Cr + 3.3\%Mo + 16\%N \quad (1)$$

$$\text{PREN}_{30} = \%Cr + 3.3\%Mo + 30\%N \quad (2)$$

The PREN subscript indicates the nitrogen coefficient.

In the particular case of stainless steels, the results included in [1,8,12,13,33] show that an expression of the type PREN_{16} would allow establishing a correlation among the corrosion resistance and the chemical composition of the alloy. Nevertheless, some authors [23] suggest using a coefficient of 30 for the nitrogen to emphasise the importance of this element in the behaviour of the alloy.

To consider the harmful effect of elements like S, P and Mn, some authors suggest including negative terms in the expression of PREN [34,35]. The alloys designed for this research, have S levels lower than 0.002%, since this is the usual content in duplex stainless steels. Although the Mn content is 8%, the formation of MnS inclusions is not very likely considering the low content of S in these materials and the solubility of this element in the ferritic phase. Nevertheless, the influence of S in pitting corrosion behaviour of the prepared alloys would not be taken into account, since the content of this element is practically the same in all the materials. Some authors argue that the negative effect of Mn associated to the formation of manganese sulfide inclusions, is balanced in these steels because of the higher N contents. Mn increases N solubility and therefore its beneficial effect in the pitting corrosion resistance [35–37]. Lastly, P contents in the new alloys are residual. Therefore, these three elements (S, Mn and P) have not been considered in the study carried out to determine a relationship between chemical composition and pitting corrosion resistance of the new alloys.

Using Eqs. (1) and (2) PREN_{16} and PREN_{30} values have been calculated for non-standard low-Ni high-Mn–N duplex alloys. Table 3 includes these values, Cr, Mo and N contents and the mean pitting potential values.

In Fig. 6 the mean pitting potential values against the values of PREN_{16} and PREN_{30} of each non-standard duplex alloys are plotted. These data confirm that an increase in the PREN value produces higher pitting potentials. The relationship found between these two variables is exponential.

Table 4 shows the results of the regression analysis for each series of PREN values (PREN_{16} and PREN_{30}), the intercept and slope parameters as well as the standard deviations of both. The “Student *t*” values and their associated probability are calculated. In both series, the *P*-value in the ANOVA table is lower than 0.01, therefore there is a statistically significant relationship between E_p and PREN at the 99% confidence level. However, comparing the standard deviation values of the

Table 3

Cr, Mo and N contents, PREN_{16} and PREN_{30} values calculated with Eqs. (1) and (2) and mean pitting potential values of non-standard low-Ni high-Mn–N

Alloy	Weight percentage			PREN_{16}	PREN_{30}	E_p (mV vs SCE)
	Cr	Mo	N			
BN1	17.97	1.98	0.0893	25.94	27.19	112
BN2	18.97	1.02	0.0998	23.93	25.32	111
BN3	19.05	1.06	0.1066	24.24	25.74	84
BN4	19.36	1.17	0.1132	25.04	26.62	128
BN5	19.95	1.03	0.1388	25.58	27.52	124
BN6	19.86	2.98	0.1747	32.50	34.95	372
BN7	20.01	3.00	0.1804	32.79	35.32	329
BN8	20.86	0.51	0.2123	25.94	28.91	119
BN9	20.98	1.00	0.1803	27.15	29.67	143
BN10	20.92	2.00	0.2305	31.22	34.45	306
BN11	21.00	2.00	0.2358	31.37	34.67	291
BN12	20.82	2.00	0.2487	31.38	34.86	278
BN13	20.96	1.99	0.2568	31.64	35.23	272
BN14	21.00	2.96	0.2558	34.85	38.43	445
BN15	20.92	3.93	0.2744	38.27	42.11	500
BN16	21.96	0.29	0.2209	26.46	29.55	118
BN17	21.89	0.29	0.2355	26.62	29.92	99
BN18	21.92	0.99	0.2213	28.73	31.83	187
BN19	21.89	0.98	0.2218	28.67	31.77	151
BN20	21.98	1.90	0.2309	31.95	35.18	335
BN21	21.95	3.01	0.2360	35.65	38.95	426
BN22	22.10	2.98	0.2561	36.02	39.61	439
BN23	22.02	3.04	0.2586	36.18	39.80	533
BN24	22.46	2.61	0.2613	35.25	38.91	418
BN25	21.97	3.00	0.3411	37.33	42.10	496
BN26	21.89	3.97	0.2468	38.95	42.40	971
BN27	21.77	4.03	0.2863	39.66	43.67	979
BN28	22.90	1.03	0.2172	29.77	32.81	170
BN29	22.89	1.00	0.2434	30.10	33.51	233
BN30	22.89	2.00	0.2406	33.34	36.71	393
BN31	22.86	2.99	0.3066	37.64	41.93	569
BN32	22.99	3.01	0.3191	38.03	42.49	638
BN33	23.96	3.04	0.2896	38.61	42.66	881

residual S_y (standard error of the estimate) and r^2 (correlation coefficient) for the two series, a better correlation between E_p values and PREN_{16} results. This regression gives a lower standard deviation value of the residual, S_y , and a correlation coefficient, r^2 , whose value indicates that the model as fitted explains 95.32% of the variability in E_p after transforming to a logarithmic scale to linearise the model. The results included in Table 4 show that, the expression of PREN with a coefficient of 16 for the nitrogen defines better the pitting corrosion behaviour of this type of materials.

The following exponential equation is established by means of the regression analysis between E_p (mV vs SCE) values and PREN_{16} ($\text{Cr} + 3.3\text{Mo} + 16\text{N}$) that

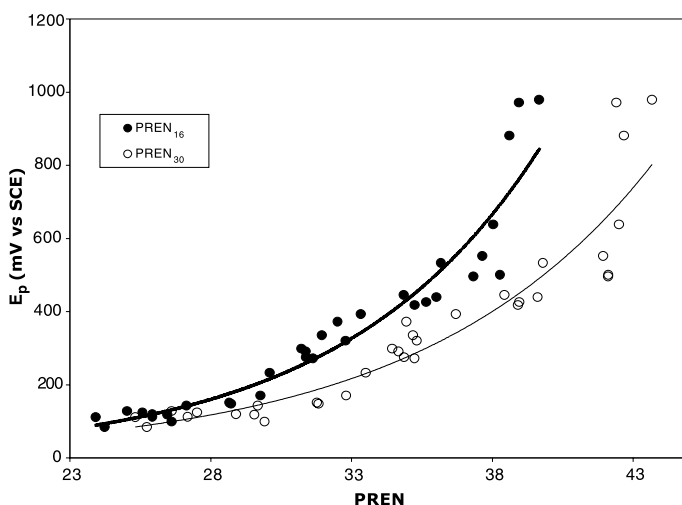


Fig. 6. Pitting potentials obtained for non-standard duplex alloys against $PREN_{16}$ and $PREN_{30}$ values.

Table 4

Results of the regression analysis between pitting potential values (E_p) and the series $PREN_{16}$ and $PREN_{30}$

	Parameter	Estimate	Error	“Student t ”	Probability
$PREN_{16}$	Intercept	1.08509	0.182601	5.94238	0.0000
	Slope	0.142552	0.0055671	25.1384	0.0000
	r^2	95.3239			
	S_y	0.156078			
$PREN_{30}$	Intercept	1.33354	0.213493	6.24629	0.0000
	Slope	0.122615	0.0060268	20.3449	0.0000
	r^2	93.0324			
	S_y	0.190519			

allows estimate of the pitting potential from the contents of the decisive alloying components:

$$E_p = 2.94 \exp(0.14 PREN_{16}) \quad (3)$$

where the units of the coefficient 2.94 are (mV vs SCE). Using this equation, new non-standard low-Ni high-Mn–N duplex alloys could be designed. By varying the contents of Cr, Mo and N, it would be possible to prepare this type of alloys with pitting corrosion resistance equivalent to those of the standard duplex stainless steels.

The standard duplex alloys prepared as references have been tested in the same conditions that the non-standard duplex. The mean pitting potential values obtained are included in Table 5. $PREN_{16}$ expression is used to compare the pitting resistance of these standard duplex with non-standard duplex alloys. It has been previously shown that this parameter defines better the pitting corrosion behaviour of the

Table 5
Cr, Mo and N contents, $PREN_{16}$ values and E_p means values for the standard duplex alloys prepared

Alloy	Weight percentage			PREN	E_p (mV vs SCE)
	Cr	Mo	N		
DC1	22.55	0.29	0.0815	24.82	282
DC2	21.06	2.46	0.1011	30.78	594
DCS	21.80	2.96	0.1279	33.61	–
AC1	16.87	2.11	0.0387	24.45	274

non-standard alloys and it is frequently used by different authors to predict the pitting corrosion resistance of standard duplex alloys [1,8,12,13,33].

A first conclusion that can be drawn from the results included in Table 5 is related with the pitting corrosion behaviour of the standard duplex alloys compared with that of the austenitic stainless steels. It can be observed in this table, that the standard duplex alloy DC1 (2304) and the austenitic stainless steel AC1 have PREN values practically similar, 24.82 and 24.45 respectively. Therefore, both materials have nearly identical pitting corrosion susceptibility. However, the duplex alloy DC1 has a residual Mo content (0.29%), while that of the austenitic AC1 is 2.11%, which means an increment in cost.

Alloys DC2 and DC3 have chemical compositions included in the compositional range of standard duplex 2205. These stainless steels are higher alloyed than alloy 2304, having higher Mo content. The pitting corrosion resistance of these steels is higher than those of the austenitic stainless steel AC1 and duplex steel DC1. The higher Mo and N contents of alloy DC3, would justify that its pitting potential could not be obtained in these experimental conditions.

The pitting potentials of all the prepared alloys have been plotted against $PREN_{16}$ values in Fig. 7 to determine the influence of the chemical composition in pitting corrosion resistance. It can be observed from this figure that for the non-standard duplex alloys, an increase in the $PREN_{16}$ value means higher pitting potentials. Therefore, as it has been previously demonstrated, the tendency is not linear but it is fitted to an exponential mathematical model, Eq. (3). A detailed analysis of Fig. 7, shows that the results of the non-standard duplex alloys can be divided in three groups, depending on the relation between E_p and PREN, Table 6. This way, alloys included in Group I are characterised because there is not a significant increase of pitting potentials as the materials are higher alloyed and therefore their PREN values are higher. All the materials included in this group have Mo contents $\leq 1\%$. Therefore when Mo content is less than 1%, an increase of Cr or N contents (higher PREN value), would not mean a significant increase of pitting potential in the test conditions (50 °C and 50 g/l of NaCl).

Group II is characterised because for a variation of PREN similar to that previously mentioned for Group I, a significant increase of pitting potential takes place. In this group, Mo contents are between 2% and 4% (weight). In such a case, an increase of Cr and N contents means an improvement of the pitting corrosion behaviour. Therefore, an increase of one unit in the PREN value means an increment of 52 mV in E_p .

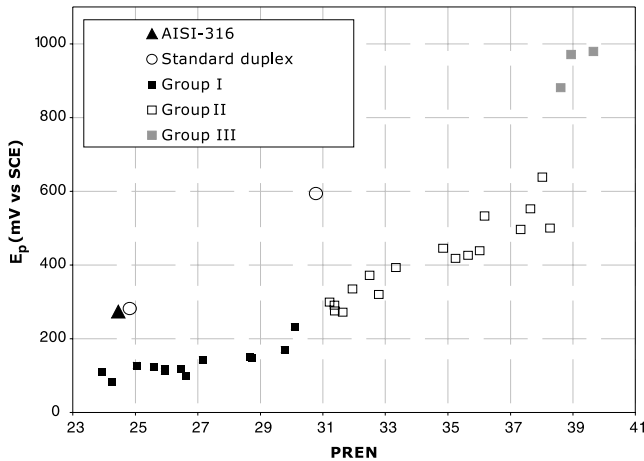


Fig. 7. E_p values for all prepared alloys against PREN.

Table 6

Variations of PREN and E_p for the three groups

Group	Variation of PREN	Variation of E_p	mV/PREN
I	6.17	149	24
II	7.05	366	52
III	1.05	98	93

Non-standard duplex alloys with Mo contents between 3% and 4% are included in Group III. These materials are characterised because an increment of one unit in the PREN value, means an increment of 93 mV in E_p .

These results show the importance of the Mo content in the pitting corrosion behaviour of these new alloys. This way, to get new non-standard duplex alloys with pitting corrosion resistance similar or higher to that of the standard duplex alloys, Mo contents greater than 1% are necessary. Moreover, these new alloys should be higher alloyed than standard duplex and therefore their PREN values would be higher.

5. Conclusions

A new family of duplex stainless steels have been prepared and characterised. These alloys have a balanced two-phase relation (α/γ). They are also strongly N alloyed, which reduces the economic cost. The results obtained show that these alloys have a higher pitting corrosion resistance in chloride environments than equivalent standard duplex or austenitic stainless steels. An exponential relationship between PREN_{16} and pitting potentials for these new alloys has been determined. These materials have also a strong dependence between E_p and Mo content. In order

to achieve improved pitting corrosion resistance, Mo content should be higher than 1%. The best results are obtained for alloys with Mo content between 3% and 4%. Lastly, Eq. (3), which correlates pitting potential with chemical composition of non-standard duplex alloys, is an essential metallurgical tool in the design of these alloys. This way, it is possible to design a non-standard duplex alloy with a pitting corrosion resistance equivalent to that of standard duplex but with less economic cost.

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