Effects of low temperature aging on the mechanical properties and corrosion resistance of duplex and lean duplex stainless steels UNS S32205 and UNS S32304

Efeitos de envelhecimentos em baixas temperaturas nas propriedades mecânicas e resistência à corrosão dos aços duplex e lean duplex UNS S32205 e UNS S32304

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Abstract

Duplex stainless steels (DSS) with austenite-ferrite microstructure are high strength and corrosion resistant steels frequently used as pipes and accessories in chemical/petrochemical on/and off-shore industries. Low temperature heat treatments (400-475°C) may increase the hardness and wear resistance of duplex steels, due to a spinodal decomposition reaction of the ferrite, whose small particles α' precipitate in the matrix. In this work, several heat treatments at 400°C and 475°C with short duration (<24h) were performed on UNS S32304 and UNS S32205 grades. The kinetics of α' precipitation was studied by hardness measurements. Tensile tests were also carried out for some heat treatment conditions to evaluate the effect of the aging on the ductility of both grades. The hardening of duplex UNS S32205 was more intense than that of UNS S32304, although both steels have shown an interesting increase in tensile strength. Specimens of duplex steel heat treated at 475°C for 4 h and 8 did not show any detectable decrease of corrosion resistance in anodic polarization and critical pitting temperature tests were carried out in NaCl media.

Keywords: Duplex stainless steels, aging, mechanical properties.

Resumo

Os aços inoxidáveis duplex (AID) com microestrutura austeno-ferrítica são aços de alta resistência mecânica, sendo utilizados em tubos e componentes nas indústrias química e petroquímica. Tratamentos térmicos em baixas temperaturas (400-475°C) podem aumentar a dureza e a resistência ao desgaste, devido à reação de decomposição espinoidal da ferrita, quando finas partículas de α' se precipitam na matriz. Nesse trabalho, diversos tratamentos térmicos de curta duração a 400°C e 475°C foram realizados nos aços UNS S32304 e UNS S32205. A cinética de precipitação foi estudada por meio de medidas de dureza. Ensaios de tração foram *realizados em algumas condições para se avaliar o efeito do envelhecimento na ductilidade dos dois aços. O endurecimento do aço UNS S32205 é mais pronunciado do que o do aço UNS S32304, embora ambos os aços apresentem um ganho considerável de resistência com tratamentos térmicos de curta duração. Amostras de aço duplex tratadas a 475°C por 4 h e 8 h não apresentaram qualquer prejuízo na resistência à corrosão nos ensaios de polarização cíclica e temperatura crítica de pites em meio contendo NaCl.*

Palavras-chave: Aços inoxidáveis duplex, envelhecimento, propriedades mecânicas.

1. Introduction

Austenitic-ferritic stainless steels present an excellent combination of corrosion resistance and mechanical properties in critical operation conditions. The higher mechanical and corrosion resistance is responsible for the increasing substitution of traditional austenitic grades by austentic-ferritic grades. The steels with austenitic-ferritic microstructure may be divided into *lean duplex*, *duplex*, *superduplex*, and, more recently, *hyperduplex* (Tavares et al., 2010). The steels with a low Mo content (<0.60%) are called lean duplex. Typical lean duplex steels have a corrosion resistance comparable to the most popular traditional austentic grades (AISI 316L and AISI 317L), but with superior yield limit and ultimate strength. The increase of Cr and N, and the addition of Mo in the chemical composition created the more corrosion resistant duplex and superduplex steels. The parameter used to distinguish these two sub-groups is the pitting resistance equivalent (PRE), which gives an indication of the pitting corrosion resistance of corrosion resistant alloys (CRA). The most used formulae for this parameter are the PRE_N and PRE_W (ASM, 1994):

$$\text{PRE ou } PRE_N = \%Cr + 3{,}3(\%Mo) + 16(\%N) \quad (1)$$

$$PRE_W = \%Cr + 3{,}3(\%Mo+\%W) + 16(\%N) \quad (2)$$

Austenitic-ferritic steels with PRE ≥ 40 are called "superduplex". The elements Cr, Mo and N are the most important for pitting corrosion resistance. Tungsten is also mentioned in Equation (2), and is used in some superduplex steels. These steels contain high contents of Cr, Mo and N, and also Ni, which are added to maintain the ferrite/austenite ideal proportion.

It is important to note that the PRE number is only a reference parameter. For instance, superduplex and hyperduplex steels may present very poor pitting corrosion resistance, if undesirable phase transformations take place during the manufacturing process (Gunn, 2003; Linton et al., 2004; Hitchcock et al., 2001). Intermetallic compounds (sigma and chi), chromium nitride (Cr_2N), and α' chromium rich precipitates are all prejudicial to corrosion resistance (Nilsson, 1992).

One of the most studied phase transformations in ferritic-astenitic steels is the spinoidal decomposition of ferrite into fine α' chromium rich precipitates and a Cr-depleted matrix ($\delta \rightarrow \alpha' + \alpha''$). This reaction occurs in the 350-550°C range, and is faster at 475°C. As the prolonged aging causes hardening and decrease of toughness, this phenomenon is often referred to as 475°C embrittlement. The α' precipitates can only be directly observed by transmission electron microscopy, as shown in some previous works (Otárola et al., 2005; Solomon et al., 1979; Padilha et al., 2004). The α' precipitation has also been studied by other indirect methods, such as electrochemical tests (Yi et al., 1996), hardness tests (Tavares et al., 2000), Mossbauer spectroscopy (Lemoine et al., 1998; Solomon et al., 1978), and magnetic measurements (Tavares et al., 2000; Maeda et al., 1997; Isobe et al., 1996). The decrease of corrosion resistance, due to prolonged aging in the 350-550°C range, was investigated by double loop electrochemical potentiodynamic reactivation tests (DL-EPR), and with the proper choice of the test solution, an increase of the degree of sensitization with aging time could be observed (Tavares et al., 2005).

In this work, the use of α' precipitation as a hardening mechanism for lean duplex UNS S32304 and duplex UNS S32205 steels was studied. Short duration heat treatments at 400°C and 475°C were performed to evaluate the benefits and prejudicial effects.

2. Materials and methods

The UNS S32304 and UNS S32205 steels studied were purchased as sheets of 1.8 mm thickness, both in the annealed condition. Chemical compositions are presented in Table 1.

The annealing of this class of material is a type of solution treatment in which the material must be soaked at 1000-1050°C and rapidly cooled to avoid deleterious phase precipitation on cooling. Light optical microscopy (LOM) was used to determine ferrite and austenite percentages, and also to investigate about the presence of deleterious tertiary phases. For austenite/ferrite quantification, the specimens were prepared with Behara's etching (80ml H_2O + 20ml HCl + 0,3g potassium metabissulfite), and the

Grade	Cr	Ni	Mo	Mn	Si	N	Cu	C	P	S
UNS S32304	23.0	4.50	0.30	1.00	0.50	0.100	0.30	0.030	0.04	0.040
UNS S32205	22.5	5.30	2.90	1.85	0.32	0.166	0.03	0.023	0.03	0.001

Table 1
Chemical composition of UNS S32304 and UNS S32205 studied.

analysis was performed by image analysis using Image Tool software (UTHSCSA, 2002). For deleterious phase investigation 5-10%KOH electrolytic etching (3V, 15 s) was used.

Specimens with 12 x 12 mm were cut and heat treated at 400°C and 475°C for 2, 4, 6, 8, 10, 12, 14, 16, 18, 20, 22 and 24 hours. These specimens were put through Vickers hardness tests with a load of 30 kgf.

For some heat treatment conditions uniaxial tensile tests were performed to evaluate the variation of mechanical properties in tension. The specimens for uniaxial tension tests were cut and machined according to ASTM E370 standard (ASTM, 2009), with initial length (l_o) equal to 75 mm. The tests were carried out at room temperature with velocity at 12 mm/minute.

Three types of corrosion tests were also carried for some heat treatment conditions. Double loop electrochemical potentiodynamic tests (DL-EPR) are used to detect and quantify the sensitization of stainless steels (Cihal et al., 2001). In this work the tests for lean duplex and duplex steels were carried out at ambient temperature, using a potentiostat-galvanostat micro-AUTOLAB. For lean duplex steel, the test solution was 2M H_2SO_4 + 0.01KSCN, and for duplex steel, the test solution was 2M H_2SO_4 + 0.5M NaCl + 0.01M KSCN. The DL-EPR tests were conducted in a conventional three-electrode cell using a Pt foil as the auxiliary electrode and a saturated calomel electrode (SCE) as the reference one. The working electrode was constructed using the lean duplex or duplex specimens embedded in epoxy resin, ground with 400 emery paper, degreased with alcohol and cleaned in water. The tests were initiated after nearly steady-state open circuit potential (E_{oc}) had been achieved, followed by the potential sweep in the anodic direction at 1 mV.s^{-1} up until the potential of 0.3 V_{SCE} was reached. The scan was then reversed to the cathodic direction until the E_{oc}. The loss of corrosion resistance due to the chromium-depleted regions, i.e. the degree of sensitization (DOS), was evaluated from the ratio I_r/I_a, where I_a is the activation peak current of the anodic scan and I_r is the reactivation peak current in the reversed scan.

The two other corrosion tests were the cyclic polarization test and the critical pitting temperature (CPT) measurement. These tests were carried out only in some heat treatment conditions for duplex stainless steel UNS S32205.

Cyclic polarization tests were carried out at 40°C in 3.5% NaCl solution, according to ASTM G61 standard (ASTM, 1998). The CPT measurement was performed by the potentiostatic method. The potential was fixed in 0.700 V_{SCE} and the temperature was raised in a constant rate of 3°C/min. The CPT was the temperature correspondent to a current density of 100μA/cm². The method was similar to the ASTM G150 standard (ASTM, 2004), except for the higher heating rate applied in this work.

3. Results and discussion

Figures 1(A-B) shows the microstructures of the materials in the as-received condition. Lean duplex UNS S32304 has 63% ferrite and duplex UNS S32205 has 65% ferrite. Deleterious phases such as chi (χ) and sigma (σ) were not detected with KOH electrolytic etching (not shown).

Figures 2 and 3 show the hardness *versus* aging time curves at 475°C and 400°C for duplex UNS S32205 and lean duplex UNS S32304, respectively. Some important observations can be made in the light of these two figures. First, the aging at 475°C is more pronounced in both steels, as expected. In the first 10 hours of aging at 475°C the hardness of duplex steel was raised from 252HV to

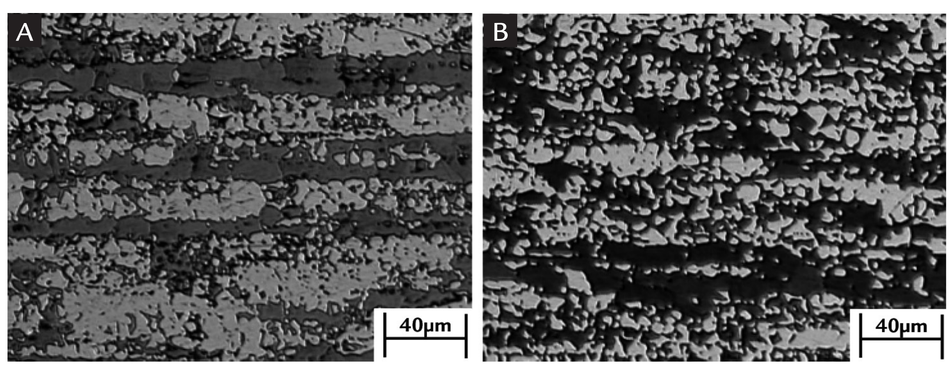

Figure 1
Microstructures of UNS S32304 (A) and UNS S32205 (B) in the as-received condition.

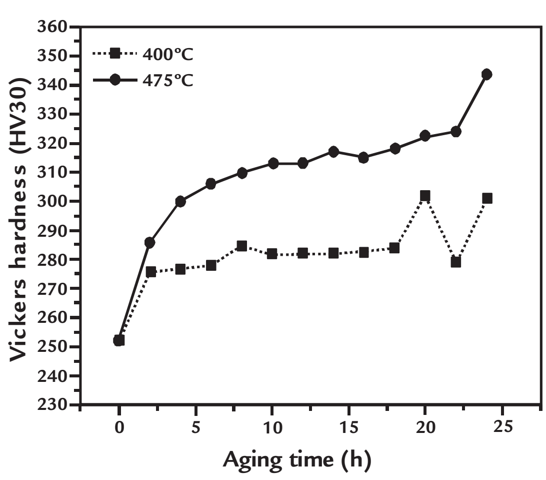

Figure 2
Hardness *versus* aging time curves for duplex UNS S32205 steel.

313 HV (ΔHV = 61 HV). In the lean duplex steel the variation of hardness in the same period and temperature is from 234 HV to 286 HV (ΔHV = 53 HV). Figure 4 compares the hardness variation of duplex and lean duplex steels during aging at 475°C, where it is possible to note that the hardening is more intense in the duplex steel than in the lean duplex. This can be attributed to the higher Mo content of the duplex steel, which enhances the kinetics of α' precipitation.

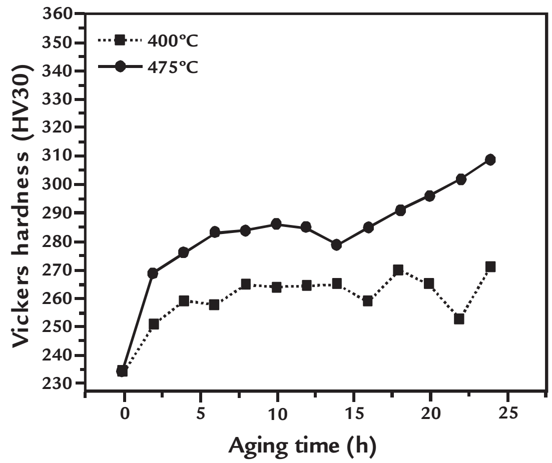

Figure 3
Hardness *versus* aging time curves for lean duplex UNS S32304 steel.

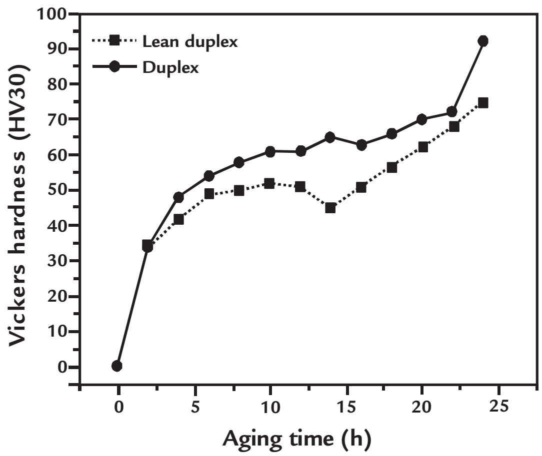

Figure 4
Variation of hardness during aging lean duplex UNS S32304 steel and duplex UNS S32205 steels.

The age hardening curves may be roughly divided into two parts. The initial stage is that of aging time up to 14 hours. At this initial stage the increase of hardening can be described by Equation 3:

$$HV_{(t)} = \frac{HV_i}{1 - a.\ t^b} \tag{3}$$

Where:
$HV_{(t)}$ – Hardness as function of time (HV)
HV_i – Initial value of hardness (HV)
a – constant
b – aging exponent
t – aging time (h)

Figure 5(A-B) shows the curves adjusted for lean duplex and duplex steels aged at 475°C, respectively. Table 2 shows the values of the a and b constants and the correlating coefficients obtained.

The interest in the initial stage of age hardening is the possibility to perform heat treatments capable of hardening the material, with minimum prejudicial effects on corrosion resistance, ductility and toughness. In order to evaluate this possibility, two short duration heat treatments were chosen for a more deep analysis by tensile and corrosion tests in both steel grades (duplex and lean duplex). The heat treatments chosen were 475°C/4 hours and 475°C/8h. These heat treatments promote significant hardness variations (ΔHV), as

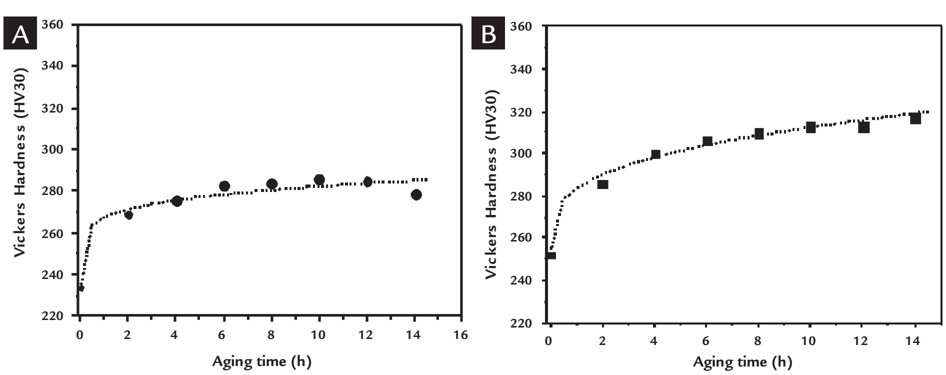

Figure 5
Curves fitted with equation (3) and parameters of Table 2:
(A) lean duplex.
(B) duplex.

Table 2
Constants of Equation (3) adjusted for lean duplex UNS S32304 and UNS S32205 aged at 475°C.

Material	Constants of Equation (1)			Correlation coefficients (R)
	HV_i	a	b	
UNS S32304	233	0.128899	0.132986	0.996
UNS S32205	252	0.110621	0.242834	0.977

shown in Figure 4.

Two tensile test specimens were produced per condition. Average values of yield limit (σ_{YL}, 0.2%), ultimate strength (σ_{UTS}), total elongation (El.), and nominal uniform ductility ($\varepsilon_{unif.}$) are presented in Table 3.

Table 3 also contains the values of toughness measured in the tensile tests. Toughness is commonly measured by impact tests, such as Charpy and Izod. However, as the sheets of duplex and lean duplex studied have thickness 1.88 mm, Charpy impact tests could not be done. The area under the curve tension-deformation curve gives a measure of the energy necessary to fracture per unit of area. Since the tests were conducted with the same experimental parameters (strain rate, temperature and specimens size), the toughness values obtained can be used to compare the different heat treatment applied.

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The data exposed in Table 3 shows that a simple heat treatment at 475°C for 4 hours increases the yield limit of about 100 MPa in both steels. The ultimate strength increases 55 MPa in lean duplex and 69 MPa in duplex steel.

Table 4 shows the percentage variations of each tensile property based on the data of Table 3. The spinodal decomposition of ferrite works as a typical hardening mechanism which decreases ductility and toughness. However, it seems that the choice for short duration heat treatments may increase the mechanical resistance with a small decrease of toughness and ductility. It must be pointed out that all fractures presented a typical ductile behavior.

Another important aspect to be evaluated is the effect of the heat treatments at 475°C for 4h and 8h on the corrosion resistance of steels. The corrosion tests were more concentrated on duplex steel because lean duplex has a corrosion resistance significantly inferior to duplex, due to its lower PRE value.

Figure 6(A) shows the anodic polarization curve of UNS S32205 in the

Table 3
Tensile properties and hardness values for lean duplex steel UNS S32304 and duplex UNS S32205 in the heat treatment conditions indicated.

Properties	Lean duplex UNS S32304			Duplex UNS S32205		
	annealed	475°C/4h	475°C/8h	annealed	475°C/4h	475°C/8h
σ_{YL}, 0.2% (MPa)	534	632	660	573	676	706
σ_{UTS} (Mpa)	722	779	803	798	867	891
Hardness (HV)	234	276	284	252	300	310
El. (%)	29.7	27.1	23.9	28.2	24.2	23.5
$\varepsilon_{unif.}$	22.2	18.5	17.0	21.1	18.6	18.4
Toughness (10^6 J/m^3)	216.0	209.8	203.4	205.5	195.3	182.0

Table 4
Variations of tensile properties with the short duration treatments at 475°C.

Tensile Properties	Lean duplex UNS S32304		Duplex UNS S32205	
	475°C/4h	475°C/8h	475°C/4h	475°C/8h
σ_{YL}, 0.2%	+ 18.4	+ 23.6	+ 18.0	+ 23.2
σ_{UTS}	+ 7.9	+ 11.2	+ 8.6	+ 11.7
Elongation	- 8.8	- 19.5	- 14.2	- 16.7
Toughness	- 2.9	- 5.8	- 5.0	- 11.4

as-received condition, i.e. with the annealing treatment. Figure 6(B) is from the same material aged at 475°C for 8 hours. Both curves were constructed at 40°C. The material aged at 475°C for 8 hours maintains high values of pitting and repassivation potentials, denoting that the critical pitting temperature is kept above 40°C after the treatment.

A second set of experiments was done specifically to determine the effect of the aging at 475°C on the critical pitting temperature (CPT) of duplex UNS S32205. Figure 7(A-C) shows the potentiostatic curves for the determination of CPT in the specimens that were un-aged, aged at 475°C for 4 hours, and aged at 475°C for 8 hours, respectively. The CPT for the un-aged specimen is 52°C, which is in accordance to other previous results (Gunn, 2003). The aging for 4 hours promotes a slight increase of CPT, which can be associated to diffusion of Cr, Mo and N in the material, eliminating the local gradients of these elements. The specimen aged for 8 hours has a CPT = 53°C, which means that this aging time is not sufficient to decrease the pitting resistance of the material.

DL-EPR tests were applied in duplex and also lean-duplex steels. Figures 8(A-B) and 9(A-B) show the DL-EPR curves for duplex and lean duplex, un-aged and aged at 475°C for 8 hours, respectively. Table 5 shows the I_r/I_a values obtained in each test. The results for the duplex steel show that aging for 8 hours was not sufficient to create Cr-depleted zones and provoke detectable sensitization. The results for the lean duplex are quite different. The curve of the un-aged material presents a reactivation peak, which indicates that the test solution is still very aggressive to this class of material. The aging at 475°C for 4 h and 8 h promotes a further increase of the I_r/I_a ratio, indicating that the material becomes sensitized with these treatments.

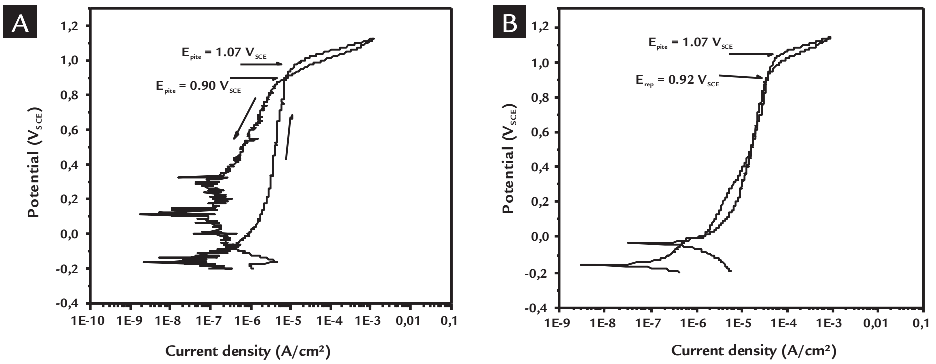

Figure 6
Cyclic polarization curves for duplex UNS S32205:
(A) Annealed.
(B) Treated at 475°C for 4 hours.

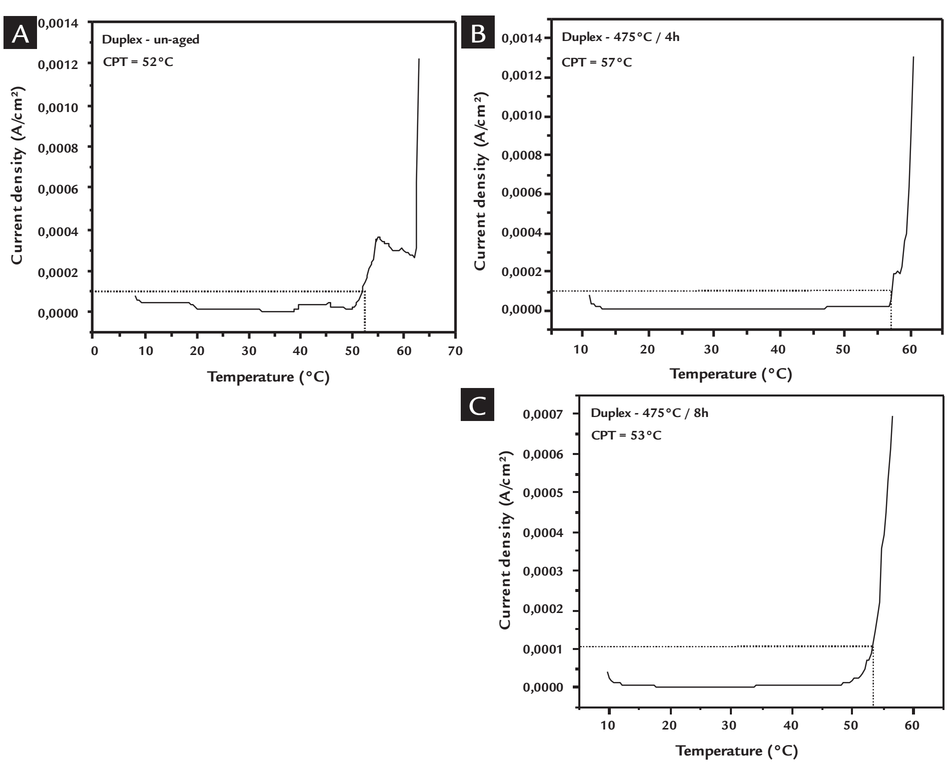

Figure 7
Density current x temperature curves for E = 0.700V_{SCE} for determination of CPT in duplex UNS S32205:
(A) Annealed.
(B) Treated at 475°C for 4 hours.
(C) Treated 475°C for 8 hours.

4. Conclusions

The results obtained in this work show that low temperature (475°C) and low duration (4h and 8h) heat treatments may be employed to increase the yield limit and ultimate strength of duplex and lean duplex steels with minimum effects on ductility and toughness. For instance, the heat treatment of duplex UNS S32205 steel at 475°C for 4 hours promoted an increase of 103 MPa in the yield limit and 69 MPa in the ultimate strength.

The aging at 475°C for 4 h and 8h did not cause any detectable damage to the corrosion properties of duplex UNS S32205 steel. Lean duplex, on the other hand, became more susceptible to sensitization according to the DL-EPR tests.

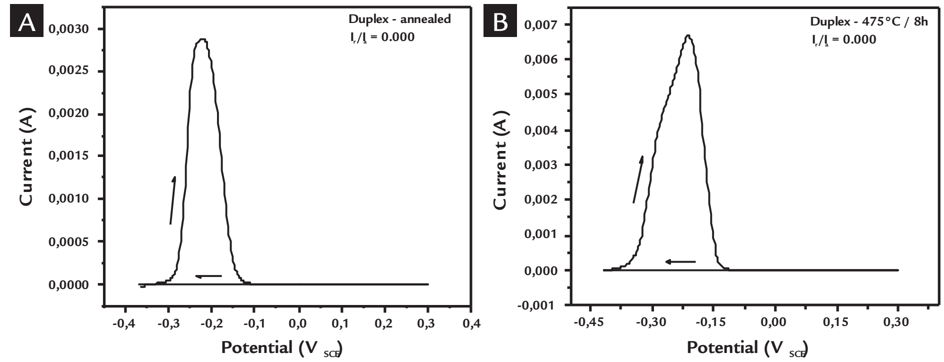

Figure 8
DL-EPR curves of duplex UNS S32205:
(A) un-aged (annealed).
(B) 475°C / 8 hours.

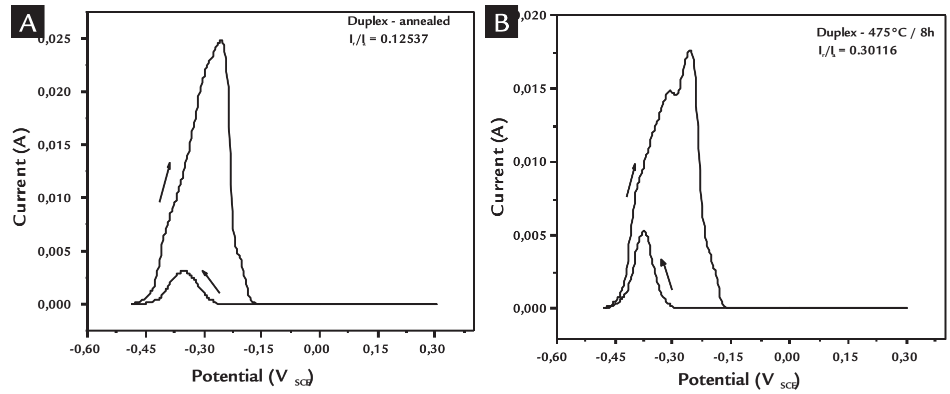

Figure 9
DL-EPR curves of lean
duplex UNS S32205:
(A) un-aged (annealed).
(B) 475°C / 8 hours.

Table 5
I_r/I_a obtained from DL-EPR curves.

Material	Annealed	475°C / 4 h	475°C / 8h
Lean duplex UNS S32304	0.125	0.280	0.301
Duplex UNS S32205	0.000	0.000	0.000

5. Acknowledgements

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