

Effect of brief heat treatments performed between 650 and 850 °C on corrosion behaviour of a lean duplex stainless steel.

F. Zanotto^{a,b}, V. Grassi^b, M. Merlin^b, A. Balbo^b, F. Zucchi^b*

^aTerra&AcquaTech Laboratory, University of Ferrara, Via Luigi Borsari 46, 44121 Ferrara, Italy

^bCorrosion and Metallurgy Study Centre “Aldo Daccò”, Engineering Department, University of Ferrara G. Saragat 1, 44122 Ferrara, Italy.

* Corresponding author: phone +39 0532 455195, fax +39 0532 455011, e-mail: federica.zanotto@unife.it.

Abstract

In this study the effect of brief heat treatments within the 650-850 °C range on pitting corrosion and intergranular corrosion resistance of a lean duplex stainless steel was investigated. Pitting potentials (Epitt) and critical pitting temperature (CPT) were determined in sodium chloride solutions. The degree of sensitisation (DOS) to intergranular corrosion (IGC) was evaluated by DL-EPR method application. The most critical treatment conditions were observed at 650 °C and at 750 °C. A recovery of the pitting and IGC resistance of the studied lean duplex was noted at 850 °C due to the redistribution of chromium towards the depleted zones.

Keywords

Stainless steel; Polarisation; Pitting corrosion; Intergranular corrosion.

Introduction

The manganese-containing duplex stainless steel LDX 2101® was commercially introduced in 2002. This lean duplex stainless steel has higher mechanical properties and resistance to stress corrosion cracking than those of AISI 304, together with a pitting resistance equal or better than that of AISI 316 [1,2].

The performance of conventional ferritic-austenitic Duplex Stainless Steels (DSS) can be adversely affected by the precipitation and transformation phenomena in the 650-950 °C temperature range, which is the most critical for the mechanical and corrosive behaviour of these alloys [3]. Several investigations highlighted the changes of the mechanical [4-7] and corrosion [8-16] resistance performances caused by the precipitation of sigma phase (σ), which is brittle and determines the formation of Cr-depleted zones [8-10]. Few works have been carried out showing the effect of the microstructural modifications on the corrosion resistance of lean duplex stainless steels [1,17-20]. In the lean duplex the precipitation of the σ phase is kinetically very slow due to a minor molybdenum content [21]. However the formation of chromium carbides and nitrides is possible at

the ferrite (α) and austenite (γ) grain boundaries also for a brief time permanence in the 650-950 °C temperature range [17,22] occurring, for example, during welding procedures [23]. It is reported that a typical problem in the heat affected zone (HAZ) of as-welded low nickel DSS submitted to multipass weldings or post-weld heat treatments is the precipitation and/or dissolution of nitrides within ferrite and at ferrite-austenite interfaces with the generation of a new austenite phase (called secondary austenite, γ_2) [24]. This latter phase has lower chromium, molybdenum and nitrogen contents than the primary austenite phase γ . These microstructural changes can determine a loss of corrosion resistance and/or toughness in the HAZ [23,25].

Concerning the effect of isothermal ageing in the 650-950 °C range on mechanical and corrosion resistance of the lean duplex 2101, Berner et al. [17] documented a decreasing of its impact toughness for isothermal treatments of few minutes between 700 and 750 °C. The same authors evidenced also a correlation between the impact toughness, the Critical Pitting Temperature (CPT) and the pitting potentials (E_{pitt}) in the 0.1 M NaCl solution. The pitting potentials showed a better agreement with Charpy test data than the CPT. Liu et al. [18] observed that a consistent reduction of toughness and CPT after few minutes of ageing was caused by the precipitation of $M_{23}C_6$ -type chromium carbides and Cr_2N -type nitrides. Zhang et al. [19] evidenced that the microstructural modifications induced by Cr-rich precipitates along α/γ and α/α grain boundaries affected the pitting resistance of a 2101 lean duplex. E_{pitt} and CPT data significantly decreased after treatments at 700 °C performed for periods lower than 30 min. Moreover, the potentiostatic CPT measurements resulted to be more sensitive than the potentiodynamic tests to the presence of small amount of precipitates.

The Double Loop Electrochemical Potentiokinetic Reactivation (DL-EPR) method is described in the ISO 12732 standard [26] and mainly proposed to detect the degree of sensitization (DOS) to intergranular corrosion (IGC) of stainless steels. However some recommendations have been indicated for the effective application of DL-EPR to duplex and superduplex stainless steels, i.e. hydrochloric acid (HCl) as depassivator and scan rate ranges between 2V/h to 15 V/h [27]. Recently, many papers adopted this technique to detect intergranular corrosion susceptibility of duplex stainless steels [1, 16, 28-31]. In particular, Deng et al. [1] carried out the DL-EPR tests in a 33 % sulphuric acid (H_2SO_4) solution with the addition of HCl as depassivator in order to find the DOS to IGC of a heat treated lean duplex 2101 (treatment time between 3 min up to 300 h). Optimum test conditions were obtained for a HCl concentration of 0.1 %, with a temperature of 20 °C and a scan rate of 2.5 mV/s. This study showed that DL-EPR tests conducted in these conditions were able to characterise the correlation between secondary phase precipitation, Cr depleting and IGC of a 2101 lean duplex with high accuracy and reproducibility.

The aim of this work was to determine the influence of brief heat treatments between 650 and 850 °C on the corrosion pitting behaviour and susceptibility to IGC of a LDX 2101®. This was achieved by E_{pitt} and CPT measurements in sodium chloride solutions and by the DL-EPR method application in a 33 % H₂SO₄ solution with the addition of a proper HCl concentration. The correlation between the microstructural modifications and the electrochemical test results was studied with the support of the optical and scanning electron microscopy observations.

Experimental

Tests were performed on the LDX 2101® stainless steel (supplied by Outokumpu Company) with the nominal chemical composition (wt.%) showed in table 1. Specimens with a 15x15 mm square surface were cut from the as received 1.5 mm thick sheet. The specimens were thermally treated at 650°C for 5, 10, 30 and 60 min and at 750 and 850°C for 5, 10 and 30 min and successively air cooled. The presence of secondary phases after the thermal treatments was documented by scanning electronic microscopy (SEM) with back-scattered electron detector (BSE) and by analysis with energy dispersive X-ray spectroscope (EDS).

The electrochemical measurements were performed on electrodes embedded in an epoxy resin in order to have a 2.25 cm² surface exposed to the solution. The surface of the electrodes was ground to 2500 grit emery papers, polished with diamond paste (from 6 to 1 µm), rinsed with deionized water and finally degreased with acetone.

The pitting potentials (E_{pitt}) were determined by anodic polarization curves recorded after 1 h of immersion in a 0.1 NaCl solution at 10 and 20°C or in a 1 M NaCl solution at 10°C, starting from the corrosion potential and with a scan rate of 0.1 mV/s. The results were presented as the average of three tests.

CPT measurements were performed in a 0.1 M NaCl solution. In order to deoxidize the surface, the working electrode was firstly cathodically polarized at -0.9 V_{SCE} for 5 min. Secondly, the specimen was allowed to stabilise, in the solution thermostated at 3°C, at the open circuit potential for 30 min. The CPT was determined by applying an anodic polarization of +0.75V_{SCE} and, at the same, by increasing the solution temperature of 1°C/min. The experiment was stopped when the current density reached a value of 400 µA/cm² and the CPT was defined as the temperature for which the current density was equal to 100 µA/cm². The CPT values were the average of three tests.

For DL-EPR tests a 33% H₂SO₄ solution, at 20°C, was adopted with controlled addition of different HCl concentrations (0.1; 0.35; 0.45 and 0.6%) as depassivator and with a potential scan rate of 2.5 mV/s [16]. The DSS samples were cathodically polarized at -0.6 V SCE for 3 min in order to improve the reproducibility. After a stabilisation of the electrode surface at the open circuit

potential (E_{ocp}) for 10 min, a potential sweep in the anodic direction was performed, up to a potential value of $+0.3 V_{SCE}$. At this point, the scan was reversed in the cathodic direction until E_{ocp} . The degree of sensitisation (DOS) to intergranular corrosion (IGC) was evaluated as the percent ratio $(I_r/I_a) \times 100$, where I_a is the peak of current in the anodic scan and I_r is the peak of current in the reverse scan. The sensitisation limit value was 1% [32].

After the DL-EPR tests, the morphologies of the intergranular corrosion attack were observed by optical microscope (OM) and SEM.

Microstructure

Fig. 1 (a-f) presents the microstructure of the LDX 2101® heat treated for different times at 650, 750, and 850 °C. Elongated austenitic grains (lighter phase) are distinguishable in the ferritic matrix (darker phase). At the SEM in BSE observation the austenitic phase, due to its higher nickel content [17,24], appeared brighter than the ferritic phase. No χ and σ secondary phases were detected in this alloy after the performed thermal treatments, due to the lower content of molybdenum [1]. Aging at 650°C for 5 min seems to not cause the presence of precipitates at the grain boundaries of the biphasic microstructure. Very small black precipitates (indicated by black arrows) were observed at the α/α grain boundaries in the sample aged for 5 min at 750 °C. In literature [1,21] these precipitates were principally identified as chromium nitrides (Cr_2N), however the presence of some chromium carbides is also possible [21,22]. With the increasing of aging time (30 min for the various temperatures) the presence of these particles became more evident.

SEM examination by line-profile analysis attested the presence of chromium nitrides at the α/α , α/γ and γ/γ grain boundaries in the LDX 2101® exposed for 30 min at 750 °C (Fig. 2 e 3) and for 5 min at 850°C (Fig. 4).

Anodic polarization behavior

As an example of polarisation behaviour, Fig. 5 collects the anodic polarisation curves recorded in the 0.1 M NaCl solution at 10 °C on the LDX 2101® non-sensitised and thermally treated at 750 °C for different times. The non-sensitised specimen was immune to pitting corrosion in this environment. In fact, starting from a potential value of $0.925 V_{SCE}$, a transpassive behaviour was observed. The samples aged for 5, 10 and 30 min at 750 °C showed an important decrement of E_{pitt} to values of 0.430, 0.374 and 0.343 V_{SCE} , respectively.

Anodic polarisation curves were also performed by increasing solution temperature to 20 °C or by increasing NaCl concentration to 1 M (Table 2). With a solution temperature of 20 °C the non-sensitised LDX 2101® showed an E_{pitt} average value of 0.566 V_{SCE} . A drop of about 200 mV was

observed after a treatment of 5 min at 750 °C, however these test conditions were not able to significantly differentiate E_{pitt} values for the longest treatment times (10 and 30 min at 750 °C). On the contrary a good differentiation was achieved in the 1 M NaCl solution at 10 °C.

The histogram in Fig. 6 collects the E_{pitt} average values obtained by anodic polarisation in 1 M NaCl solution at 10 °C also on the LDX 2101® electrodes thermally treated at 650 and 850 °C.

A treatment of 5 and 10 min at 650 °C led to a moderate E_{pitt} decrease (of about 200 mV_{SCE}) in comparison to that of the non-sensitised sample, whereas by prolonging the treatment time up to 30 and 60 min a noticeable decrement (of about 500 mV_{SCE}) was observed. A treatment of only 5 min at 750 °C significantly reduced the resistance to pitting corrosion of the LDX 2101®, with an E_{pitt} value drop of 350 mV. The increasing of treatment time at 750 °C led to a further small decrease of E_{pitt} . By increasing the treatment temperature to 850 °C a small increment of the E_{pitt} value in comparison to that measured for the sample thermally treated at 750°C was obtained. Fig. 7 shows that the localised corrosive attack started at the austenite and ferrite interface and propagated within the ferritic phase [17,33,34].

CPT results.

Fig. 8 shows as an example the current density vs. temperature curves obtained for the LDX 2101® electrodes thermally treated at 850°C and for the non-sensitized sample. In agreement with the potentiodynamic test, an anodic polarisation of the non-sensitised sample above the E_{pitt} measured in the 0.1 M NaCl solution, determined a quick current density increase when test temperature approached 20 °C, so that the measured CPT value resulted 19.7 °C. The temperature for which an abrupt current density increase was observed decreased significantly for the sensitised samples. The CPT decreased also by increasing the treatment time from 5 to 30 min, whereas the sample aged for 10 min at 850 °C showed a pitting resistance slightly better than that aged for only 5 min.

The histogram of Fig. 9 shows that the trend of the CPT values are in agreement with that of the E_{pitt} values, i.e when the CPT decreased the E_{pitt} values also decreased, however this electrochemical test seems to better differentiate the corrosion pitting behaviour for the different treatment conditions than the anodic polarisation tests. The CPT values for the samples thermally treated for 30 and 60 min at 650 °C and for 30 min at 750 °C are around 3 °C, which is the initial temperature of the 0.1 M NaCl solution. This means that at the starting test conditions the passive film formed on the samples was not stable and a current density equal or higher than 100 $\mu\text{A}/\text{cm}^2$ was measured.

DL-EPR test results

The determination of the optimal depassivator concentration, which is the most significant factor affecting the sensitivity of the DL-EPR test, was performed on the LDX 2101® thermally treated at 750 °C. In Fig. 10 are reported the Ir/Ia % ratio obtained with different depassivator concentrations on the LDX 2101® sensitised at 750 °C for different treatment times. For a HCl concentration above 0.35 % the value of Ir/Ia % for the non-sensitised specimens were higher than 1 %, indicating the occurrence of generalised corrosions together with IGC [35], whereas a HCl concentration of 0.1 % was not sufficient to detect the IGC susceptibility of the aged specimens. When HCl concentration was set to 0.35% the non-sensitised specimen retained a Ir/Ia % value lower than 0.1% (i.e. 0.08 %), a result indicating good experimental conditions [27], and the samples aged for different times presented a proper Ir/Ia % ratio differentiation, evidence of a good selectivity of the attack.

The 0.35% HCl concentration was finally selected as the optimal depassivator concentration and was used to detect the DOS of the specimens thermally treated at 650 and 850°C, too.

The DOS obtained with DL-EPR tests performed on the LDX 2101® for all the tested sensitisation conditions are collected in the histogram reported in Fig. 11. A treatment time of 5 and 10 min at 650 °C determined a moderate DOS, in fact values of 1 and 1.8%, respectively, were calculated from the DL-EPR curves. A treatment time of 30 min at 650 °C produced an increase in the Ir/Ia % value of about one order of magnitude in comparison to that obtained with a treatment of 10 min. By doubling the treatment time to 60 min, a little decrease of the DOS was observed, however susceptibility to IGC was still very high. In agreement with CPT and E_{pitt} results, the Ir/Ia % determined for the samples aged at 750 °C increased by increasing the treatment time, however retaining a DOS lower than that observed after treatments performed for 30 and 60 min at 650 °C. Finally, the samples aged for 5 and 10 min at 850 °C showed a susceptibility to IGC similar to that of the correspondent samples at 650 °C. But a sensitisation of 30 min at 850 °C produced a DOS that was 1/5 of that obtained at 650 °C.

In Fig 12 some of the optical micrographs acquired after the DL-EPR tests performed on the LDX 2101® electrodes are reported. The non-sensitised sample showed no evident signs of IGC attack. A selective attack localised at grain boundary started to be clearly observable for the 5 min at 650°C treated sample. The attack became more evident for treatment conditions of 30 min at 850 °C or of 10 min at 750 °C. A diffuse and deeper selective attack of the grain boundary was observed for the most sensitised specimens (30 min at 650 and 750 °C).

Discussion

The results of this investigation shows that heat treatments performed in the 650-850 °C temperature range, also for brief treatment times, cause a reduction of the LDX 2101® resistance to pitting corrosion and IGC. From a microstructural point of view, SEM-EDX observations evidence the presence of small chromium rich precipitates, essentially chromium nitrides (Cr_2N), at the α/α , α/γ and γ/γ grain boundaries, depending on treatment conditions. Wei et. al. [21] reported the presence of mainly Cr_2N together with some Cr_{23}C_6 at the α/γ interface and α/α grain boundaries of a lean duplex 2101, after ageing for 240 min at 700 °C. Other authors [17, 18] identified as M_{23}C_6 type carbides the fine particles formed after very long aging times (10 and 100 h) between γ and secondary austenite (γ_2) in a LDX 2101, whereas Cr_2N were found only at the ferrite grain boundaries or within ferrite grains. It was confirmed in literature [35] that Cr_2N -type nitride precipitation occurs simultaneously with M_{23}C_6 within the interval of 550-1000 °C, however they have the highest kinetics formation in the 700-900 °C temperature range. In this research the Cr_2N -type nitrides (probably with M_{23}C_6) precipitation is observed to occur between the austenite grains after ageing at 850 °C for 5 min (Fig. 4). This was also noted by Deng et al. [1] for aging conditions of 48 h at 700 °C.

As already discussed, the low molybdenum content that characterizes the LDX 2101 alloy makes the formation of σ phase a kinetically very slow process. Thus, also the longest treatments (30 or 60 min) within the 650-850 °C temperature range were not sufficient to allow the precipitation of this deleterious phase. Other authors observed in the lean duplex stainless steel 2101 the presence of σ phase only after 168 h at 700 °C [1] or after 100 h at 700 °C [18].

The precipitation of Cr_2N at the grain boundary determines the formation of Cr-depleted zones in which the passive film is less stable and becomes a preferential site of pitting attack in the chloride solutions [17-19] and/or areas subjected to the IGC during reactivation in the DL-EPR tests [1,20,36]. A research performed with scanning Kelvin probe force microscope (SKPFM) [37] showed that Cr_2N is a nobler phase in comparison with the surrounding austenite, since it exhibits a higher Volta potential. Moreover, it is well known that nitrogen has a beneficial effect on the passive film and corrosion resistance of stainless steels [38], thus a depletion of nitrogen in the austenite phase makes the boundary area surrounding the Cr_2N more prone to localized corrosion.

In this investigation pitting resistance and DL-EPR test results are almost in agreement. This is probably due to the use of HCl as depassivator agent in the DL-EPR test, thus chloride ions takes place in the mechanism of attack during pitting and IGC.

In Fig. 13 are reported the CPT and Ir/Ia % data in function of the sensitisation temperature for all the performed treatment times. The DOS obtained after a short sensitisation time, 5 or 10 min in the

650–850 °C temperature range, shows a maximum of DOS at 750 °C and the same behaviour for the sample treated at 650 and 850 °C. Also for the CPT values obtained with treatments of 5 and 10 min a minimum of the pitting resistance at 750 °C can be observed. The treatment performed at 650 and 850 °C determines a quite similar pitting behaviour only for the 10 min ageing time, whereas the treatment performed for 5 min at 850 °C causes a reduction of pitting resistance higher than that performed at 650 °C (CPT = 14.9 °C for 5 min at 650 °C and CPT = 9.3 for 5 min at 850 °C). For the longer treatment periods (30 min between 650 and 850 °C) the resistance to IGC and pitting corrosion increases by increasing the sensitisation temperature. In this case there is a maximum of the DOS (14.5%) and a minimum of the CPT (< 3 °C) for the samples sensitised 30 min at 650 °C. Roncery et al. [39] performed thermodynamic and diffusion simulation of the nucleation and growth of $M_{23}C_6$ and M_2N in an austenitic stainless steel during isothermal ageing at temperatures between 850 and 900 °C. They observed that at the higher ageing temperatures Cr-depletion occurs in a wider area than that obtained at the lower temperatures, but with a higher level of the content of Cr. This observations can sustain the hypothesis of a thermal activated rediffusion of chromium from the grains inner towards the depleted zones [40,41] at the highest performed sensitisation temperature (850 °C) in our investigation. This effect determines the improvement of the pitting and IGC resistance of the LDX 2010® at this temperature. In Fig. 14 images acquired by SEM in BSE of the electrodes surface aged for 30 min in the considered temperature range after the DL-EPR tests are showed. The percentage of area interested by the IGC attack was estimated by an image analysis software applied on the acquired SEM-BSE micrographs. The results (Fig. 15) confirms that for a treatment time of 30 min the amount of area in which a deeper IGC attack occurred (darker areas in the SEM-BSE micrographs) decreased by the increasing of the sensitisation temperature.

Conclusions

1. Brief heat treatments performed in the 650–850 °C range determined a reduction of the pitting resistance and an increasing of IGC susceptibility of the lean duplex LDX 2101®.
2. In general, the $E_{pitt.}$ and the CPT average values decreased by increasing the treatment time, however a little recovery was observed for a treatment of 60 min at 650 °C or of 10 min at 850 °C.
3. The DL-EPR test results were almost in agreement with $E_{pitt.}$ and CPT test results.
4. The DOS increased by increasing the treatment time, even if a little reduction for the treatment of 60 min at 650 °C was observed.

5. The most critical treatment conditions resulted the 30 and 60 min at 650 °C and the 10 and 30 min at 750 °C.
6. A recovery of the pitting and IGC resistance of the LDX 2010® was observed for ageing performed at 850 °C, this was due to the redistribution of chromium towards the depleted zones.

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