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Effect of brief thermal aging on stress corrosion cracking susceptibility of LDSS 2101 in the presence of chloride and thiosulphate ions

F. Zanotto^{a,*}, V. Grassi^b, A. Balbo^b, C. Monticelli^b, C. Melandri^c, F. Zucchi^b^a Terra & AcquaTech Laboratory, University of Ferrara, Via Saragat 1, 44122 Ferrara, Italy^b Corrosion and Metallurgy Study Centre “Aldo Daccò”, University of Ferrara, Via G. Saragat 4A, 44122 Ferrara, Italy^c ISTE-CNR, Via Granarolo 64, 48018 Faenza, Italy

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ABSTRACT

In this paper, the effect of brief aging within the 650–850 °C temperature range on the resistance of lean duplex stainless steel (LDSS) 2101 to stress corrosion cracking (SCC) was discussed. The thermal aging induced a decrease in Vickers microhardness and an increase in alloy ductility. This behaviour was connected to depletion of interstitial nitrogen in austenite. LDSS 2101 was found to suffer SCC in NACE TM-0177 solution, particularly under specific thermal aging and in the presence of thiosulphate ions ($S_2O_3^{2-}$) at 10^{-4} and 10^{-3} M. Crack nucleation and propagation were linked to microstructural variations connected to thermal aging.

1. Introduction

In the last 20 years, the development of austenitic-ferritic stainless steels, more commonly known as duplex stainless steels (DSS), has followed two different paths: the improvement of corrosion performances by increasing chromium, molybdenum and nitrogen content (superduplex and hyperduplex grades) and the developing of the so called “lean” duplex family [1]. The lean duplex stainless steels (LDSSs) were conceived to lower the molybdenum content, which can cause dangerous secondary phase formation during welding operations, and to reduce the nickel content in order to limit raw material costs in duplex production. In 2002, LDX 2101[®] (a low nickel grade) was commercially introduced with the aim of advantageously replacing austenitic stainless steels (AISI 304 and 316), in applications where high mechanical and localized corrosion resistance are required [2–5], but also, to some extent, to substitute carbon steels, where maintenance costs are significant [1].

The mechanical properties and the corrosion resistance of ordinary DSS can be adversely affected by aging within the 650–950 °C range [6], mainly due to σ phase formation [7–15]. However, in LDSS the growth of this compound is very slow, because of the low molybdenum content [4,16], and may affect the performances of these alloys only after long aging time (on the order of 100 h [4,17]).

LDSS 2101 contains high amounts of manganese (5 mass%) and nitrogen (0.22 mass%) [18] added with the aim to stabilize the austenite phase. The influence of N content is quite complex. In fact, it improves the pitting corrosion resistance and stimulates cracks

repassivation [19,20]. It also enhances the mechanical strength, even at low concentrations (i.e. 0.1–0.2%) [21,22]. In DSS, it increases the microhardness of both ferrite (α) and austenite (γ) phase, with a more pronounced effect on γ [22], because this phase can dissolve higher nitrogen amounts. For this reason, in DSS the γ phase becomes the strongest one at N concentrations higher than 0.12 wt.% [23,24]. On the other side, it is reported to reduce the stacking fault energy (SFE) of austenite phase, so promoting planar slip and easier passive film breakdown, when slip/dissolution mechanism is involved in SCC [25,26].

Other problems connected to high nitrogen levels occur after thermal aging because precipitation of nitrides (e.g. Cr_2N) at the grain boundaries occurs already after few minutes of aging in the 650–850 °C interval [27–30]. During a previous research carried out in our laboratory on LDSS 2101 [30], these microstructural modifications are found to determine a worsening of pitting and intergranular corrosion (IGC) resistance. In particular, a very low resistance to pitting corrosion and high values of the degree of sensitization (DOS) to IGC were obtained, particularly after 30 and 60 min of aging at 650 °C and 10 and 30 min at 750 °C. On the contrary, aging at 850 °C for up to 30 min essentially did not modify the resistance to IGC and only slightly decreased that to pitting corrosion, likely due to chromium replenishment around nitride precipitates. Liu et al. [17] documented the structural evolution of LDSS 2101 for thermal aging at 600–850 °C and looked for a connection between variations in mechanical properties and pitting resistance. In agreement with Berner et al. [27], they found a significant reduction of both critical pitting temperature (CPT) and impact

* Corresponding author.

E-mail address: federica.zanotto@unife.it (F. Zanotto).

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toughness after few minutes of aging at 750 °C, which resulted the temperature leading to the most severe degradation of the alloy performances. All quoted studies evidenced that these short-time thermal aging provoked formation of Cr-rich precipitates (mainly Cr₂N but also M₂₃C₆-type carbides) at the phase grain boundaries and Cr-depletion in the adjacent zones, determining an easier localization of corrosion attack.

Oil and gas equipment often operate in contact with chloride- and sulphide-rich media. Therefore, the employed materials have to resist to pitting corrosion and stress corrosion cracking (SCC) in these environments. LDSS 2101 has a resistance to pitting and crevice corrosion, which is higher than that of austenitic 304 L and is comparable to that of 316 L [2,5]. Even SCC resistance is superior to that of standard austenitic grades. This makes LDSS 2101 a suitable candidate for oil and gas industry applications, in mild well conditions and topside components [18,31] and the study of its corrosion performances in simulated oil and gas conditions is reputed quite important.

Several works in literature concern the SCC resistance of ordinary DSS 2205 [32–36] or superduplex stainless steels [37–39] in environments containing hydrogen sulphide (H₂S). Few data are reported about the SCC behaviour of lean duplex 2304 and LDSS 2101 in the presence of H₂S [18,40]. In our laboratory, the slow strain rate technique was applied to LDSS 2101 during exposures to 20% NaCl solution at 80 °C and to standard NACE TM-0177 solution at 25 °C, in the presence of thiosulphate ions (S₂O₃²⁻) at various pH and temperatures [40]. In fact, as proposed by Tsujikawa et al. [41,42], it is possible to substitute H₂S with S₂O₃²⁻ ions in laboratory studies aiming to estimate the sulphide SCC susceptibility of alloys in sour environments. The use of S₂O₃²⁻ ions in replacement of H₂S gas can minimize the health hazards in laboratory tests and therefore can reduce the costs to ensure safe working conditions [43]. Our research [40] showed that LDSS 2101 was susceptible to SCC even in the presence of small amounts of S₂O₃²⁻. However, in dependence of S₂O₃²⁻ concentration, pH and temperature of the solution, also a diffuse selective attack of ferrite phase occurred during slow strain rate tests (SSRT). Actually, selective dissolution of ferrite in LDSS 2101 was detected also in the presence of chlorides and H₂S [18] and this behaviour must be taken into account when these alloys are used in oil and gas field.

In the present paper, the influence of brief aging performed between 650 and 850 °C on SCC susceptibility of LDSS 2101 was studied in standard NACE TM-0177 solution at pH 2.7 and 25 °C, in the absence and presence of S₂O₃²⁻ at 10⁻⁴ and 10⁻³ M.

2. Experimental

Tests were performed on LDX 2101[®] stainless steel (supplied by Outokumpu Company under annealed conditions) with nominal chemical composition (mass%) reported in Table 1.

Tensile samples with an overall length of 23 cm and a gauge portion of 20 × 5 × 1.5 mm were machined from a 1.5 mm thick steel sheet. The samples were aged at 650 °C for 30 and 60 min and at 750 and 850 °C for 5, 10 and 30 min and then air cooled. The resulting microstructures were observed by Zeiss EVO MA15 scanning electron microscope (SEM) in back-scattered electron (BSE), in order to reveal the presence of secondary phases.

Before and after thermal aging, Vickers microhardness (HV) measurements were carried out to evaluate the alloy hardness, while nanoindentation tests were performed to determine the hardness of the single ferrite and austenite phases. Both tests were carried out on

Table 1
Nominal chemical composition (mass%) of LDSS 2101.

| Elements | C | Mn | Cr | Ni | Mo | N | Fe |
|----------|------|-----|------|-----|-----|------|---------|
| % | 0.03 | 5.0 | 21.5 | 1.5 | 0.3 | 0.22 | balance |

samples ground to 2500 grit emery papers, polished with diamond pastes (from 6 to 1 μm), rinsed with deionized water and finally degreased with acetone. HV values were the average of 5 measurements obtained with an applied load of 10 N. In order to measure the dispersion of each set of HV data, standard deviations were calculated.

Load-controlled nanoindentation tests were performed by employing a commercial nanoindenter (Nanoindenter XP MTS, Oak Ridge, TN, USA) using a Berkovich diamond tip. The peak load was 5 mN. The indenter was continuously loaded up to the peak load and immediately unloaded with no holding time. From each load-displacement curve the maximum contact depth (h_{max}) and the maximum indentation load (P_{max}) parameters were derived, together with the estimated area of contact (A). The latter was determined by Oliver and Pharr method [44], directly from the geometry of the Berkovich indenter and h_{max} (nanoindenter software TestWorks TM ver. 4.06A). The nanoindentation hardness was finally computed as the ratio between the measured P_{max} and the corresponding calculated A . The area function of the indenter tip was calibrated on a standard silica sample for a contact depth range corresponding to those measured on the LDSS samples. A 10 × 4 grid of indentations, horizontally spaced 5 μm and vertically spaced 4 μm, was performed on each sample. After nanoindentation tests, samples were observed with SEM in BSE modality, in order to classify each indentation as located in either ferrite or austenite grains or discharged, due to proximity to an interface boundary. The accepted ferrite and austenite hardness data (expressed in GPa) were separately averaged to obtain the mean hardness values of each phase and data dispersion was quantified by calculating standard deviation.

The susceptibility to SCC was evaluated by SSRT, with a strain rate of 1 × 10⁻⁶ s⁻¹ [45]. After thermal aging, the samples were ground parallel to the stress direction down to 800 grit emery papers and screened by a two-component epoxy varnish, so leaving only the gauge portion exposed to the solution. SSRT were performed by inserting the sample in an electrochemical cell, which was filled by deaerated 5% NaCl + 0.5% CH₃COOH solution (the basic standard solution NACE TM-0177 [46], without saturated H₂S gas) in the absence and presence of 10⁻⁴ or 10⁻³ M Na₂S₂O₃, at 25 °C and pH 2.7. During each test, the stress – strain curve was recorded. Stress (in MPa) is the ratio of the applied load to cross section area of the gauge portion (5 mm x 1.5 mm) in tensile samples, while strain is the ratio of the elongation of the sample to its original length, as evaluated from the relative movement of the tensile machine crosshead. The obtained strain values are unitless, but they were expressed in percentage, as they were multiplied by 100. In parallel to stress-strain curve recording, corrosion potential (E_{cor} , versus Saturated Calomel Electrode (SCE)) values were measured. Reference SSRT in air at 25 °C were also carried out. Each test was performed in triplicate.

The SCC susceptibility was evaluated by the ratio, R , between the percentage fracture elongation ($\epsilon_f\%$) of samples exposed to the test solution and the corresponding $\epsilon_f\%$ in air. At the end of the tests, side surfaces of the samples (sections parallel to load direction in SSRT) were polished and examined by optical stereomicroscope, and by SEM, with the purpose to analyze crack initiation and morphology.

3. Results

3.1. Microstructure

The microstructural evolution due to thermal aging of the studied LDSS was discussed in a previous work [30]. As an example, Fig. 1 compares the microstructures of the cross section (load in SSRT was applied in the direction perpendicular to the shown section) of the as-received sample (a) and those of samples aged for 30 min at 650 °C (b) and for 5 (c) and 30 min (d) at 850 °C. In general, brief aging within the 650–850 °C range produced no evidence of χ and σ secondary phases. However, very fine chromium carbides and especially nitrides (as those indicated by blue arrows in Fig. 1) precipitated at the α/α and γ/γ grain

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