Effect of 600°C reversion treatment to reabsorb α′ forged components made of F51 DSS

S. Barella, A. Gruttadauria, C. Mapelli, P. Cetto, M. Mogliazzi

Duplex stainless steels encounter the precipitation of several secondary phases that are detrimental to both mechanical and corrosion properties when exposed to temperatures above 300°C. When embrittlement is caused by the α′ phase, heat treatment just above the miscibility gap responsible for α′ separation is sufficient to restore the lost properties, avoiding the most common solubilization at 1050°C. The aim of this study was to determine the optimal holding time for 600°C reversion treatment of 2205 DSS affected by α′. Optimization was performed by characterizing the material treated for different periods of time. To verify the soundness of this treatment, an analogous experimental campaign was conducted on the same material that was reverted at 550°C for different periods. From the research, the most effective holding time for 600°C reversion was found to be 2 h, as it gave the best results for elongation, corrosion rate, ductility, UTS, and Rp02. Lower times do not allow for complete recovery from embrittlement, whereas longer times cause the precipitation of detrimental secondary phases. In this study, conventional characterization techniques were coupled with double-loop electrochemical potentiodynamic reactivation to verify the reliability of this technique for the detection of detrimental secondary phases.

KEYWORDS: DUPLEX STAINLESS STEEL, 475 EMBRITTLEMENT, ALPHA PRIME, SIGMA PHASE REVERSION TREATMENT, DLEPR, POTENTIODYNAMIC CHARACTERIZATION

INTRODUCTION

Duplex stainless steels (DSS) are high-strength corrosion-resistant alloys that meet the requirements for structural applications in highly aggressive environments, such as petrochemical industries and marine atmospheres. When exposed to temperatures between 300 and 1000°C, these alloys encounter the precipitation of harmful secondary phases that affect both mechanical properties and corrosion resistance [1]–[5]. The time–temperature precipitation diagram (TTP) allows the identification of two different precipitation fields in this temperature range, one between 300°C and 550°C, and the other between 550°C and 1000°C. Among low-temperature precipitates, α′ is the most common and dangerous phase precipitating in these alloys. α′ is a chromium-enriched phase that is generated intragranularly in ferrite grains because of the miscibility gap between 300 and 500°C, which mostly affects material toughness and corrosion resistance [1]–[5].

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It was found that heat treatment just above the miscibility gap is sufficient to reabsorb the precipitated α′ without the need for a traditional solution treatment above 1050°C. This treatment is called the reversion treatment and relies on overcoming the miscibility gap and on the presence of a gap between the two precipitation fields in the TTP diagram between 500°C and 600°C. This temperature range is where secondary phases present the highest incubation time and allow performing short treatments before encountering further precipitation of harmful secondary phases [4], [6]–[9].

The focus of this work was to test the soundness of 600°C reversion on S32205 duplex stainless steel affected by 475°C embrittlement and consequent α′ precipitation. This was achieved through the experimental investigation of an embrittled material that was reverted for different times at 600°C. For comparison with the more traditional 550°C reversion treatment, an analogous experimental campaign was conducted on samples reverted at 550°C. The entire study was paired with double-loop electrochemical potentiodynamic reactivation analysis for each condition experienced by the material. The objective of this investigation was to study the effectiveness of a specific setup for detecting α′.

MATERIALS AND METHODS

The material under investigation was SS32205 obtained from a forged flange. The material was obtained from a hot-rolled bar, flanged at approximately 1100°C, and then solution-treated at 1050°C for 2 h per inch. The chemical composition of the as-received materials is listed in Tab. 1.

Tab.1 - Chemical composition of the as received material.

%wt.	C	Si	Mn	Cr	Mo	Ni	Cu	N	Fe
S332205	0.015	0.407	1.858	22.98	3.306	4.898	0.181	0.283	65.78

Severe embrittlement at 475 °C for 76 h was accomplished on the entire material to obtain α′ separation. The proportion of material that was exposed to this treatment was named 475. Subsequently, the 600°C and 550°C reversion treatments were performed in a resistance oven for different holding times, as shown in Tab. 2. The treatments were followed by water-quenching to prevent secondary precipitation during cooling.

Tab.2 - Samples nomenclature.

	30 min	1h	2h	3h	4h
600°C	600_30	600_1h	600_2h	600_3h	600_4h
550°C	-	550_1h	550_2h	-	550_4h

Three Charpy tests were conducted for each condition according to ASTM E23 [10]. Before the test, the specimens were soaked for 1 h at -46°C (ISO 17781) [11]. Vickers microhardness tests were performed on all the metallographic samples with a load of 25 gf. Mechanical characterization included tensile tests following ASTM E8 only on 475 and 600°C reverted samples with a crosshead speed of 2 mm/min [12].The microstructure was observed by optical microscopy (OM) and scanning electron microscopy (SEM) after polishing and subsequent Bereha etching. A local chemical composition analysis by EDS probe (energy-dispersive X-ray spectroscopy (EDS) was performed on the secondary phases. The ASTM G 48 test was conducted at 25°C for 24 h to characterize the pitting corrosion behavior [13]. Potentiodynamic analyses were performed using a three-electrode configuration potentiostat (AMEL 2553) following the standard ISO 12732 [14]. The setup was envisaged as an electrolyte,

700 ml solution of 3,7 M hydrochloric acid, and a potential scan rate of 0,5 mV/s.

RESULTS AND DISCUSSION

The characterization performed on the 475 material highlighted severe embrittlement with a complete loss in toughness, corrosion resistance, and elongation at the UTS, paired with the hardening of the ferrite phase. Considering the exposure temperature, this property loss can be ascribed to α′ [1]–[4].

Optical microscopy revealed no significant differences in the material microstructure after the reversion treatment. The only considerable evidence of secondary phase precipitation is the grain boundary broadening visible on the 600_4h sample. The SEM investigation confirmed this feature, showing progressive coarsening of the incoherent phases in the grain boundary region, starting from a holding time of 1 h. At 600_4h precipitation became more consistent, and EDS analysis highlighted the enrichment of chromium and molybdenum in this phase. The chemical composition, together with the 600°C temperature, makes it reasonable to identify the secondary phase as the σ phase [1]–[4] [15]. The analysis conducted on the 550°C-reverted samples showed slightly different results, with less pronounced precipitation. This could be due to slower kinetics at lower temperatures and the progressive increase in incubation time associated with lowering the temperature [1]–[5].

The Charpy test conducted on the reverted material showed a complete restoration of toughness at both temperatures even after 1 h, achieving values far above the limit imposed by the standard ISO 17781 (Fig.1) [11]. Samples treated for 1 h and 2 h at both temperatures exhibited similar average values of approximately 170 J, with great dispersion. This scatter could be linked to the beginning of the high-temperature secondary phase precipitation. The holding time chosen in this experimental campaign was within the range of the incubation time for these phases, which led to more inhomogeneous and unpredictable precipitation and its relative consequences on material properties [1]–[5]. This hypothesis is supported by the results obtained by the 4 h treatment samples, which show a reduction in the average toughness but a lower dispersion, possibly due to a more stable and prolonged σ phase formation [16]–[18].

Tensile tests performed on the 600°C reverted samples showed that UTS and Rp02 did not experience a significant variation both before and after the heat treatment, while elongation at UTS, on the other hand, revealed an important result for this study. Starting from a value well below the ASTM A182 standard shown by the 475 sample, the elongation at UTS values increased with holding time until 2 h of treatment, which resulted in the maximum values obtained in the experimental campaign (Fig.1) [19]. 600_2h is also the only condition exhibiting elongation that satisfies the value imposed by the standard. Upon increasing the holding time to 4 h, a new drop in elongation was observed, which is in accordance with the results obtained by the Charpy test and the hypothesis of σ-phase precipitation [15]–[18].

A decreasing trend was observed in the ferrite microhardness between 475 and reverted conditions. Samples treated for 1 h showed a higher ferrite hardness than those treated for 2 h, confirming that one hour of reversion does not allow for complete α′ reabsorption. The trend followed by the 550 °C and 600°C reverted samples is the same, but a faster decrease in ferrite microhardness can be seen in the 600_1h sample compared to the 550_1h sample, confirming that at lower temperatures, the reabsorption of α′ needs longer time.

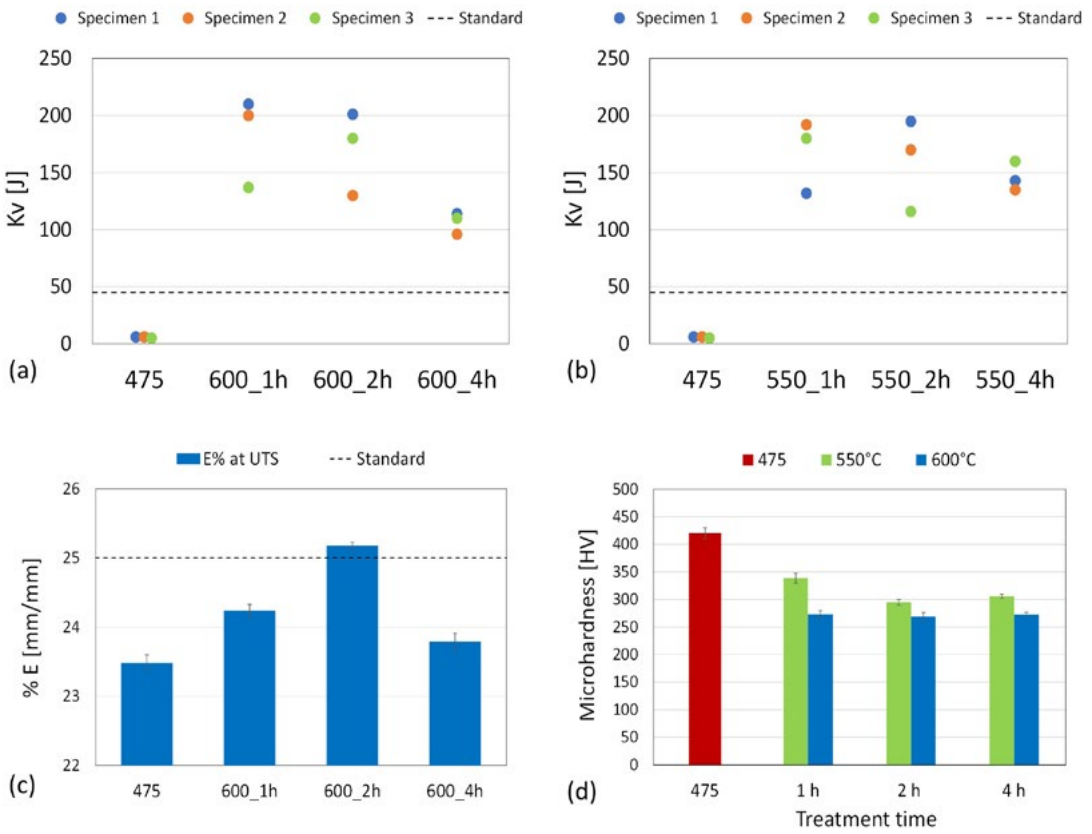

Fig.1 - (a) Charpy test results after 600°C reversion. (b) Charpy test results after 550°C reversion (c) Elongation at UTS (d) Ferrite microhardness.

For both reversion temperatures, the weight loss decreasing trend started from the 30 min treatment and continued until 2 h, which were the only samples satisfying the ISO 17781 standard of 4 g/m2 [11]. On the other hand, the samples treated for 4 h showed this trend, showing an increase in weight loss, which could be ascribed to σ-phase precipitation, as this phase also affects the corrosion behavior [15]–[18]. 550_30, 550_1h, and 550_2h experienced slightly higher weight loss than that at 600 °C, possibly because of the increased difficulty in reabsorbing α′. An inversion of this trend was observed for the 4 hours samples, where the 600_4h sample showed a higher weight loss than the 550_4h sample. This follows the hypothesis of a lower secondary-phase precipitation at 550°C.

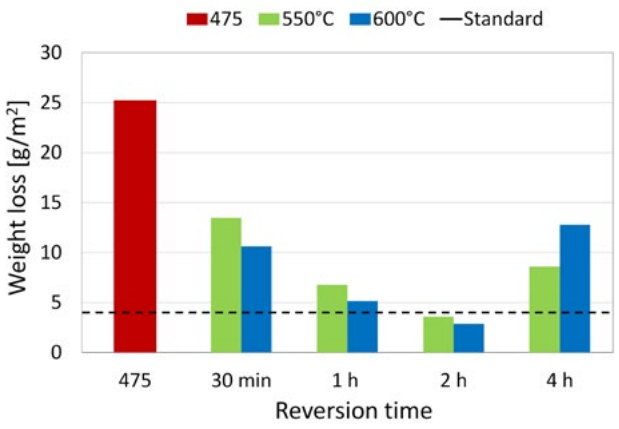

Fig.2 - Weight loss measured after ASTM G48 A.

The main features of the potentiodynamic curves considered in this study were the curve shape and the ratio between the reactivation current density and passivation current density, named the degree of sensitization (DOS). Regarding the shape of the curves, duplex stainless steel typically presents only one peak in the passivation curve and two peaks in the reactivation curve. Secondary phases can affect this behavior by erasing one peak or creating a second peak in the passivation curve [20], [21]. Considering the DOS, an increase in this parameter is commonly linked to the presence of secondary phases that affect corrosion resistance. The absolute DOS value is strongly influenced by the setup features, such as the potential scan rate, electrolyte nature, and material analyzed [22]. In this study, DOS variation under different conditions in the same experimental setup was considered.

The 475 curves are completely different from the as-received curves (Fig.3). The 475 passivation curve showed a typical peak, whereas the reactivation curve showed only one peak. The explanation is not certain, as the literature is not unambiguous regarding this phenomenon, but this could be due to ferrite chromium depletion caused by α′ and the consequent faster ferrite corrosion. The DOS increased significantly from the as-received condition, starting from 0,7 and reaching 1,17 in the 475 sample, confirming the loss in corrosion resistance.

Reverted samples showed an immediate return to the as-received behaviour, with two peaks and a DOS in the same range even in the sample treated for 30 min. Given that α′ had not been completely reabsorbed even after 1-hour treatment, the obtained result strengthens the hypothesis that α′ can be detected by this technique only when present in large amounts, as shown by the 475 sample. Again, the only difference between the two reversion treatments was in the 4 h treatment. While sample 550_4h is completely in line with the as-received and other reverted samples, 600_4h gave completely different results. Two well-distinguished peaks are present in the passivation curve, and the DOS increases, reaching a value of 1,17. This behavior can be associated with the presence of the σ phase, which reportedly affects the corrosion behavior and potentiodynamic response [15],[16]. The reason why the 550_4h scan did not highlight any difference compared to the as-received condition could be the lower extent of precipitates that also led to the softer embrittlement seen in the traditional characterization. This indicates that the sensitivity of this instrument setup for the detection of secondary phases is too low to detect small microstructural changes that still affect material properties.

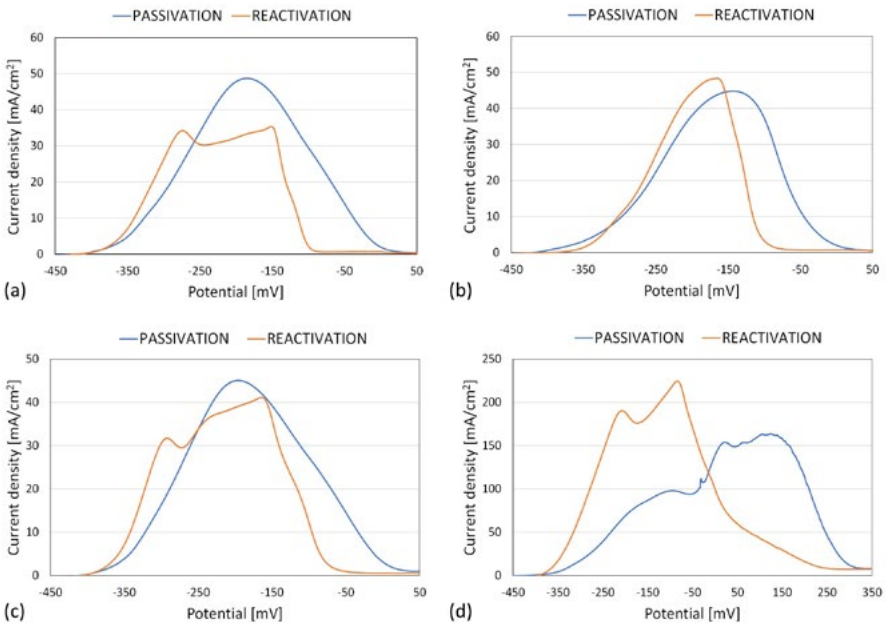

Fig.3 - DLEPR current density vs potential curves: (a) As received material (b) 475 (c) 600_2h (d) 600_4h

CONCLUSIONS

According to the findings of this study, the 600°C reversion treatment seems to be a promising alternative to the 550°C treatment for restoring the mechanical properties and corrosion behavior. The optimal time for this treatment was 2 h, which meets all the requirements set by the standards. On the other hand, the 550°C treatment failed to adequately restore the mechanical properties and did not reabsorb α′ in a time useful to avoid the precipitation of embrittling secondary phases. The main drawback of this treatment is that high-thickness components would require high holding times to restore the mechanical properties at the heart of the piece, whereas on the surface, σ-phase precipitation degrades the corrosion properties. Therefore, it is recommended that a solution treatment at 1050°C be used to safely reabsorb α′; however, for a limited thickness, a 600°C reversion treatment for 2 h could be a suitable option. The DLEPR method with the configuration used in this study was effective in detecting embrittling secondary phases, such as α′ and σ, when they were present in large quantities. However, to consider DLEPR a reliable alternative to traditional characterization methods, further optimization of the electrolyte composition and potential scan speed is necessary.

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