Fatigue crack propagation micromechanisms in high temperature embrittled duplex stainless steels

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Abstract. Austenitic-ferritic (duplex) stainless steels are successfully used in chemical, nuclear, oil and gas industries, due to their good mechanical properties and excellent generalized and localized corrosion resistance in many environments and operating conditions (for example, chloride induced stress corrosion). The aim of this work is the analysis of the fatigue crack propagation resistance in air of three austenitic-ferritic stainless steels:

- a "lean" duplex stainless steel 21 Cr 1 Ni;
- a "standard" duplex stainless steel 22 Cr 5 Ni;
- a "superduplex" stainless steel 25 Cr 7 Ni.

Two different heat treatments were considered:

- solution annealed 1050°C for 1 h (as received; $R = K_{min}/K_{max} = 0.1; 0.5; 0.75$)
- tempering at 800°C up to 10 h (only R = 0.5)

A scanning electron microscope (SEM) fracture surface analysis has been performed to investigate the fatigue crack propagation micromechanisms in the investigated superduplex stainless steel. Experimental results allow to evidence the influence of secondary phases, carbides and nitrides on fatigue crack propagation resistance that depends on the duplex stainless steel chemical composition.

Introduction

Duplex stainless steels (DSSs) are in between the austenitic and the ferritic grades, combining the best mechanical and corrosion resistance properties of both. As a result of their high mechanical strength, good thermal conductivity and excellent corrosion resistance DDSs are extensively used both in pulp and paper industries, in chemical and petrochemical plants. They also find some applications in food and biomedical fields as well [1–3]. The wide use of DSSs is closely connected to their specific microstructure, formed by roughly equal percentages of austenite and ferrite. Such an austenite-ferrite ratio gives a higher yield and ultimate tensile strength than the austenitic grades, with good ductility and toughness. On the other hand, the marked microstructural anisotropy of these hot rolled materials can result in variability of mechanical properties, such as tensile strength and fracture toughness [4,5]. The high chromium (between 21 and 27 wt.%) and molybdenum (up to 4.5 wt.%) contents allow the use of DSSs under conditions of pitting, crevice and, above all, stress corrosion cracking that would be critical for the traditional AISI 304 and 316. Finally, some economical advantages follow as a result of lower nickel content than the austenitic grades. The aforementioned mechanical and corrosion resistance properties are achieved in commercially wrought DSSs after hot rolling followed by a solution annealing and quenching. Hot rolling and solution annealing parameters (e.g. temperatures, times and strain reductions) for DSS depend on the chemical composition, the desired ferrite/austenite volume ratio, the final plate thickness [6].

Partition coefficients for a given element do not vary with the steel chemical composition: ferrite grains result to be enriched in P, W, Mo, Si and Cr, whereas austenite grains are enriched in N, Ni, Cu and Mn.

Considering that DSSs are characterized by really interesting resistance to pitting and intergranular corrosion, a practical classification criterion of various DSS is based on their pitting index, or pitting resistant equivalent (e.g. PRE= %Cr + 3.3 (%Mo + 0.5%W) + 16 %N). Among duplex stainless steel, at least three different types can be identified:

- "lean" duplex, that are characterized by very low Mo and Ni content, with a PRE that is about 25(they can be considered as valid substitute of AISI 304);
- duplex with a PRE of about 35; 22 Cr 5 Ni duplex stainless steel can be considered as the standard alloy;
- "superduplex" stainless steels having PRE values greater than 40 (they are characterized by a corrosion resistance that is comparable to superaustenitic steels and can be used in very aggressive environment).

Depending on their chemical composition, these steels are prone to age hardening and embrittlement over a wide temperature range [7, 8]. DSSs are characterized by two embrittling temperature ranges (C-shaped curves) which exhibit several secondary phases, carbides and nitrides precipitation at different holding times. A representative TTT diagram showing the above mentioned phenomena for SAF 2304, 2205 and 2507 grades is reported in Fig. 1 [9].

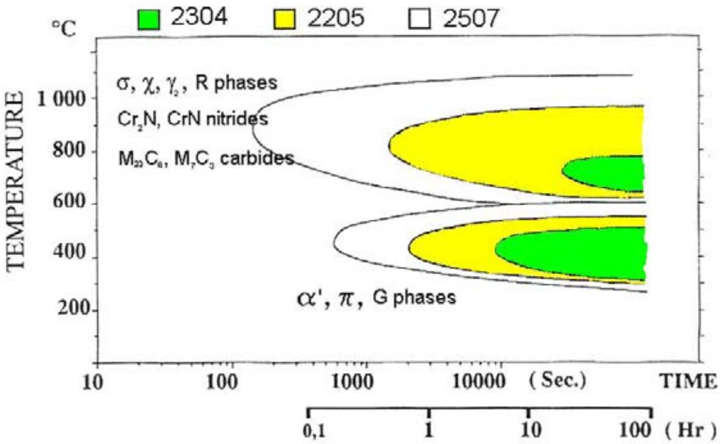

Fig. 1: TTT diagram for DSSs: chemical composition influence [9].

The first critical temperature range is situated between 500° and 1100°C, and it involves the formation of carbides (M_7C_3 and $M_{23}C_6$), nitrides (Cr_2N and π), secondary austenite γ_2, χ, R, and σ phases depending upon the steel composition and its thermal conditions [10]. σ phase, as a result of the fact it is present in a large volume, is the most important phase besides ferrite and austenite.

Second critical temperature range is between 350°C and 500°C with a nose at about 475°C. In this temperature range two mechanisms can be related to the so called "475°C embrittlement" of the steel [8, 11]:

- a spinodal decomposition of the a ferrite in two phases: an a9 Cr-rich phase and an a Fe-rich phase.
- nucleation and growing of Ni-Si-Mo rich f.c.c. G phase, characterized by a very slow precipitation kinetic (the overall concentration in "G-forming" elements increases from 40 to 60% between 1000 and 30000 h at 350°C).

Mechanical properties are strongly influenced by these changes in microstructure [12].

In this work three different austenitic stainless steel were considered, ranging from "lean" chemical composition to "super" duplex. Fatigue crack propagation resistance was investigated in air considering stress ratio influence and high temperature tempering treatment (800°C), ranging from 1 to 10 hours. Crack propagation micromechanisms were investigated by means of a scanning electron microscope (SEM) fracture surface analysis.

Material and experimental procedure

Investigated rolled stainless steels chemical composition and tensile properties are shown in tables 1-3. All the investigated steels are characterized by analogous ferrite/austenite ratio (α/γ = 1) and show a rolling texture. Fatigue crack propagation tests were run according to ASTM E647 standard [13], using 10 mm thick CT (Compact Type) specimens and considering three different stress ratio values ($R = K_{min}/K_{max}$ = 0.1; 0.5; 0.75). Tests were performed using a computer controlled Instron 8501 servohydraulic machine in constant load amplitude conditions (sinusoidal loading waveform) at room temperature, with a loading frequency of 30 Hz. Crack length measurements were performed by means of a compliance method using a double cantilever mouth gage. Different heat treatments were considered:

- solution annealed 1050°C for 1 h (as received);
- 800°C for 1, 3 and 10 h (only R=0.5).

Fatigue crack propagation micromechanisms were investigated by means of SEM fracture surface analysis, considering loading conditions (R and applied ΔK) influence.

Table 1: 21 Cr 1 Ni "lean" DSS chemical composition (wt%) and tensile properties (PRE = 26); EN 1.4162.

C	Mn	Cr	Ni	Mo	N
0.03	5.00	21.5	1.5	0.3	0.22

YS [MPa]	UTS [MPa]	A%
483	700	38

Table 2: 22 Cr 5 Ni DSS chemical composition (wt%) and tensile properties (PRE = 35); EN 1.4462.

C	Mn	Cr	Ni	Mo	N
0.019	1.51	22.45	5.50	3.12	0.169

YS [MPa]	UTS [MPa]	A%
565	827	35

Table 3: 25 Cr 7 Ni superduplex stainless steel chemical composition (wt%) and tensile properties (PRE = 42); EN 1.4410.

C	Mn	Cr	Ni	Mo	N
0.019	0.80	24.80	6.80	3.90	0.30

YS [MPa]	UTS [MPa]	A%
556	814	31

Experimental results and comments

Fig. 2 shows chemical composition influence on DSS fatigue crack propagation in air. Duplex 2205 and superduplex 2507 stainless steels are characterized by an analogous fatigue crack propagation resistance, for all the investigated stress ratio values (ranging from 0.1 to 0.75). "Lean" DSS 2101 is characterized by lower threshold values (for all the investigated R values); furthermore, 2101 DSS is characterized by higher crack growth rate values, for all the investigated loading conditions (R and ΔK values), up to five times with respect to crack growth rate values obtained with duplex or superduplex stainless steels. Considering the same loading conditions, SEM fracture surface analysis confirm that crack propagation micromechanisms are the same in duplex and superduplex stainless steel, with striation and some secondary cracks for lower ΔK and R values (Fig. 3; crack propagates from left to right) and an increasing importance of ferrite grain cleavage corresponding to more critical loading conditions (higher ΔK and R values; Fig. 4). Solution annealed “lean” DSS fracture surfaces are characterized by a more evident importance of cleavage (Figs. 5 and 6) , also considering less critical loading conditions (lower ΔK and R values). Striations are also observed. Embrittling conditions influence on fatigue crack propagation resistance is shown in Figs. 7-9.

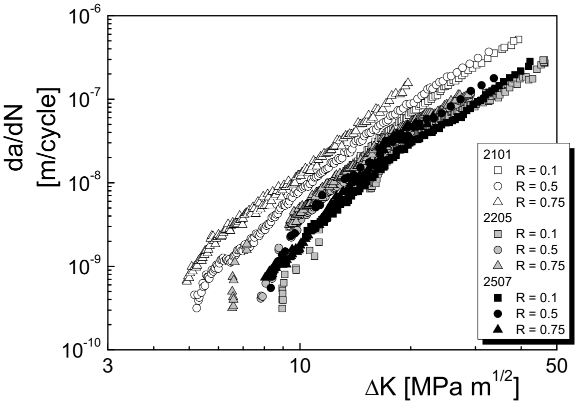

Fig. 2: Chemical composition influence on DSSs fatigue crack propagation resistance (solution annealed conditions).

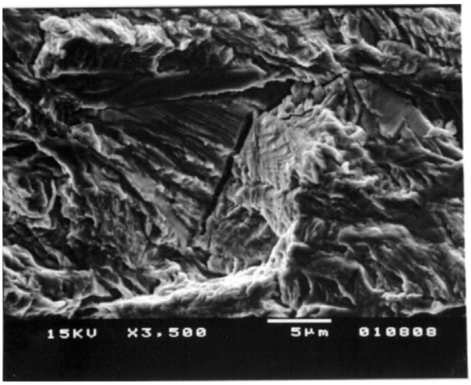

Fig. 3: Solution annealed 2205 DSS fracture surface (ΔK = 15 MPa√m; R = 0.5).

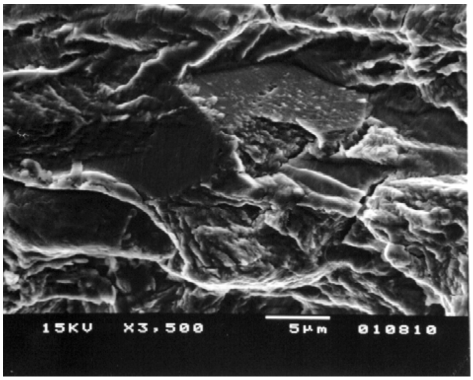

Figure 4: Solution annealed 2205 DSS fracture surface (ΔK = 25 MPa√m; R = 0.5).

Heat treatment influence on investigated DSSs is strongly influenced by DSS chemical composition:

- 2101 DSS shows an increase of the threshold value ΔK_{th}, from about 5 MPa√m (solution annealed) to about 10 MPa√m (800°C-3h); longer tempering duration at 800°C is characterized by a decrease of ΔK_{th}; final rupture values decrease with the increase of the tempering duration at 800°C (highest value is obtained with the solution annealed steel).

- 2205 DSS shows threshold values ΔK_{th} that practically do not depend on the heat treatment (about 7 MPa√m for all the investigated heat treatment); the increase of the duration of the 800°C tempering implies a strong increase of the crack growth rate obtained for the some loading conditions (e.g., for ΔK = 10 MPa√m, differences between solubilized DSS and the 800°C-10 hours embrittled DSS are higher than a factor of one).
- 2507 DSS is characterized both by a strong decrease of the threshold value ΔK_{th} and by a strong increase of crack growth rate values, with the increase of the 800°C tempering duration; worst fatigue crack propagation behavior is obtained after 10 hours at 800°C (lowest threshold value ΔK_{th}, highest crack growth rates).

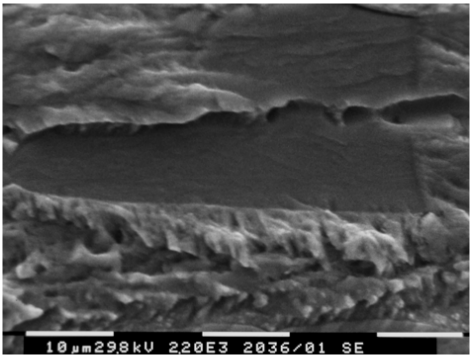

Fig. 5: Solution annealed 2101 "lean" DSS fracture surface (ΔK = 6 MPa√m; R = 0.5).

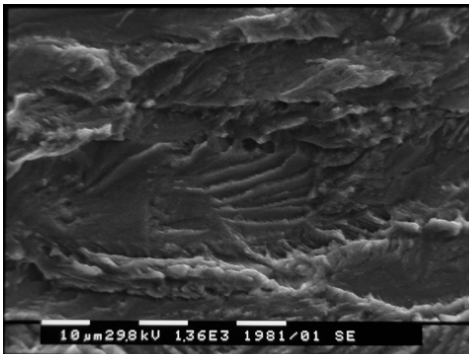

Fig. 6: Solution annealed 2101 "lean" DSS fracture surface (ΔK = 11 MPa√m; R = 0.1).

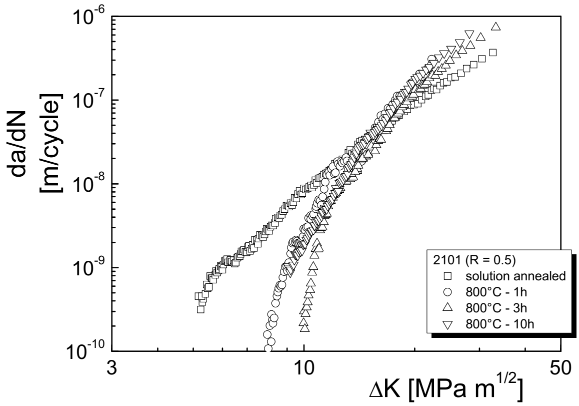

Fig. 7: 2101 "lean" DSS fatigue crack propagation resistance: heat treatment influence (R = 0.5).

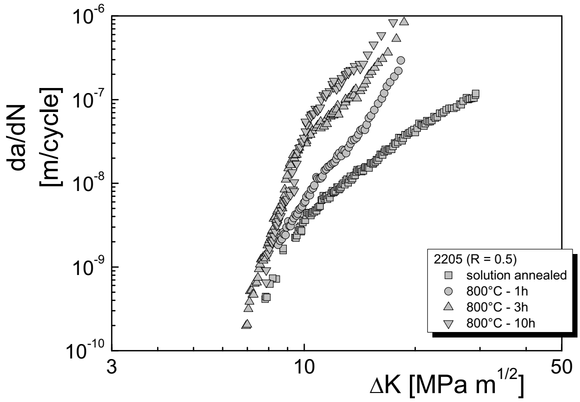

Fig. 8: 2205 “standard” DSS fatigue crack propagation resistance: heat treatment influence (R = 0.5).

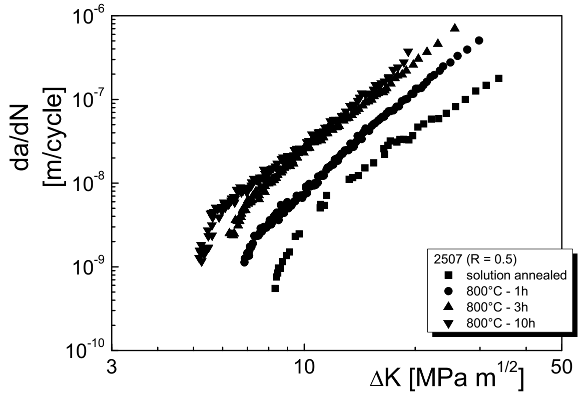

Fig. 9: 2507 “super” DSS fatigue crack propagation resistance: heat treatment influence (R = 0.5).

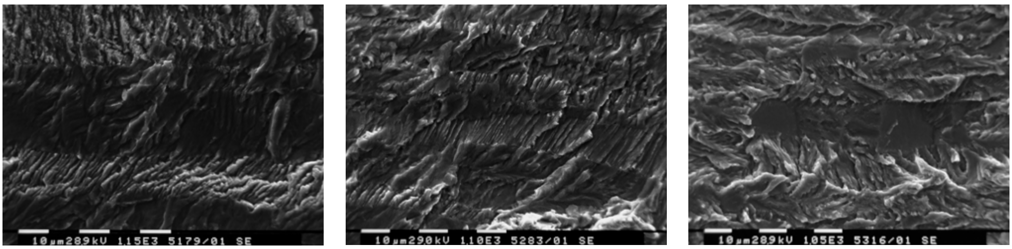

Fig. 10: 2101 DSS fracture surface analysis (threshold conditions). From left to right: 800°C -1h: 800°C – 3h; 800°C – 10h.

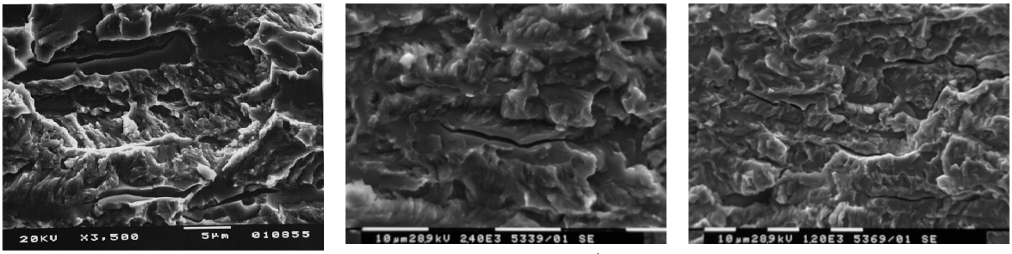

Fig. 11: 2205 DSS fracture surface analysis (ΔK = 12 MPa√m; R = 0.5). From left to right: 800°C - 1h: 800°C – 3h; 800°C – 10h.

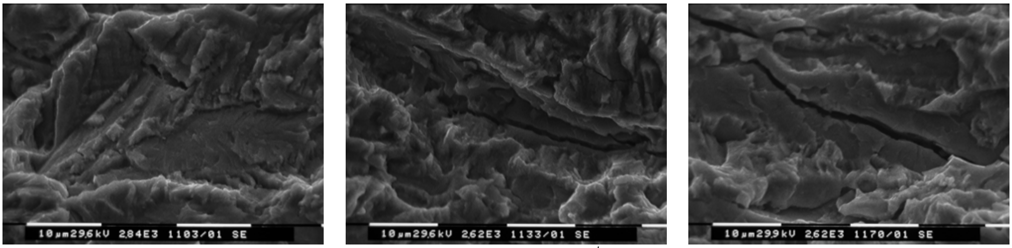

Fig. 12: 2507 DSS fracture surface analysis (ΔK = 12 MPa√m; R = 0.5). From left to right: 800°C - 1h: 800°C – 3h; 800°C – 10h.

Also crack propagation micromechanisms are influenced by heat treatment conditions. Considering near threshold conditions, 2101 tempered at 800°C for 1 hour and 3 hours are characterized by fatigue striations, without cleavage and secondary cracks (Fig. 10). Longest duration of 800°C tempering treatment imply an increase of cleavage importance with some secondary cracks.

Considering both 2205 and 2507 DSS(Figs. 11 and 12, respectively), ferrite grains cleavage and secondary cracks are more and more evident with the increase of the 800°C tempering duration.

The different influence of 800°C tempering on fatigue crack propagation resistance is probably due to different secondary phases, carbides and nitrides precipitation kinetics.

Although 2101 TTT diagram is not available, the analysis of Fig. 1 allow to hypothesize that the really low Ni content and the high Mn content that characterize 2101 "lean" DSS imply a strong TTT diagram modification, with different precipitation kinetics if compared to the "standard" 2205 and the "super" 2507 DSS. Fatigue crack propagation micromechanisms in 800°C tempered 2101 DSS do not correspond to a very embrittled steel: higher critical temperature range in "lean" DSS should be more deeply investigated.

"Standard" 2205 DSS and "super" 2507 DSS are prone to be embrittled due to tempering treatments at 800°C: fatigue crack propagation micromechanisms are strongly influenced, with an evident increase of the importance of ferrite cleavage and secondary cracks propagation in ferrite grains or in ferrite/austenite grain boundaries.

Conclusions

Austenitic-ferritic (duplex) stainless steels are successfully used in chemical, nuclear, oil and gas industries, due to their good mechanical properties and excellent generalized and localized corrosion resistance in many environments and operating conditions (for example, chloride induced stress corrosion). In this work, three austenitic-ferritic stainless steels were investigated (2101, 2205 and 2507) focusing the influence of chemical composition and high temperature tempering (800°C, from 1 to 10 hours).

On the basis of the fatigue crack propagation results and of the SEM fracture surface analysis , the following conclusions can be summarized:

- considering solution annealed conditions, “lean” 2101 DSS is characterized by the worst fatigue crack propagation behavior for all the investigated loading conditions, with a decrease of the threshold values and higher crack growth rates;
- considering 800°C tempered steels, both “standard” 2205 and “super” 2507 DSSs are prone to be more and more embrittled with the increase of the tempering duration: as a consequence an increase of cleavage and secondary crack importance on fracture surfaces is obtained with the increase of the tempering duration;
- 2101 “lean” DSS does not seem to be prone to be embrittled by 800°C tempering heat treatment; this is probably due to evident differences in secondary phases, carbides and nitrides precipitation kinetics.

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