

FATIGUE OF DUPLEX STEELS IN CORROSIVE ENVIRONMENT

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Abstract: Duplex stainless steels (DSS) are prime candidates for paper and pulp, off shore and mining industry due to their high corrosion fatigue resistance accompanied by good mechanical properties. The aim of the study is to obtain the fatigue data for two powder metallurgical (P/M) DSS steels in modified TIP 0402-09 type II corrosive environment resembling white water found in pulp and paper industry. The specimens were manufactured from two types of commercial DSS one hot rolled type and one P/M hot isostatically pressed (HIP-ed) type. Axial fatigue testing method at 15 Hz on a servo-hydraulic testing machine was utilized for determining the fatigue limit and S/N curve. Fracture surfaces were examined using scanning electron microscope and light optical microscope.

Key words: Duplex steel, corrosion, fatigue, fracture.

1. INTRODUCTION

In pulp and paper industry duplex stainless steels (DSS) are prime candidates for use in paper machine suction rolls [1]. Suction rolls are used in “white water” removal and to control the wet paper web during the paper making process [2]. “White water” is corrosive agent containing Chloride (Cl^{-1}), Thiosulphate ($\text{S}_2\text{O}_3^{2-}$) and Sulphate (SO_4^{2-}) ions [6].

Fatigue is progressive, localized, permanent structural change that occurs in materials when subjected to fluctuating

stresses and strains that may result in development of cracks or fracture after sufficient number of cycles of cracks or fracture after sufficient number of cycles of fluctuations [3]. Fatigue testing can be carried out in several control regimes - stress control and strain control. Former is used in case of high cycle fatigue, latter low cycle fatigue. There are several loading types possible: axial, torsion, rotating [4].

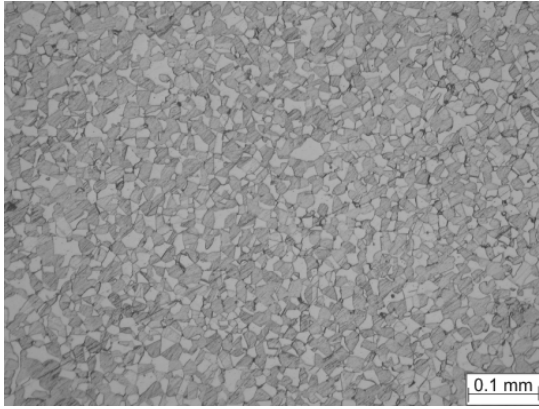
The aim of the study is to obtain the fatigue data for two commercial DSS steels in modified TIP 0402-09 type II corrosive environment and to give a reference to literature results obtained in TIP 0402-09 type I corrosive environment.

2. DUPLEX STEELS

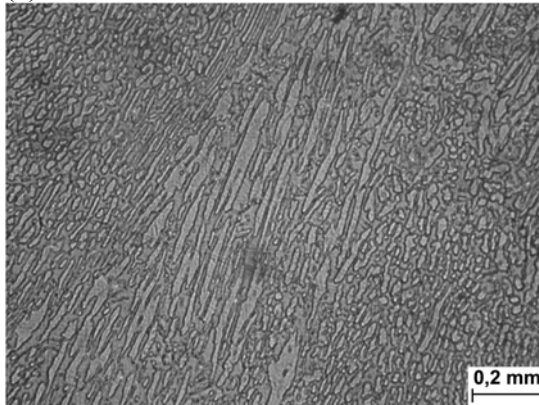
Tested Duplok22 and 3RE60 SRG are low alloy DSS-s and were supplied by Metso Powdermet OY.

They contain approximately equal separate volume fraction of ferrite (α) and austenite (γ), which grant them unique corrosion resistance along with good mechanical properties [3]. In modern DSS the α and γ ratio can be high as 40/60 respectively [7].

DSS are manufactured as forged, cast, wrought and P/M products. The initial powder size before hot isostatic pressing (HIP-ing) is $<250 \mu\text{m}$ in the case of powder metallurgy (P/M) HIP-ed Duplok22 [3]. Fine and homogeneous microstructure is preserved during the HIP-process (Fig.1a). The hot rolled 3RE60 SRG microstructure shows that the ferrite and austenite distribution is not as fine as in Duplok22 (Fig. 1b).



(a)



(b)

Fig. 1 Microstructure of tested steels

2.1 Chemical composition

DSS-s contain Ni to stabilize the austenite and to form a duplex structure, Cr to give them corrosion resistance and stabilize ferrite, Mo to enhance corrosion resistance and stabilize ferrite, N and Mn to stabilize austenite [3]. N is almost completely dissolved in the austenite [3,5]. Duplok22 has higher alloying element concentration compared to 3RE60 SRG. The tested material chemical composition is shown in Table 1.

2.2 Corrosion resistance

DSS have excellent resistance to stress corrosion cracking compared to conventional austenitic stainless steels [3]. This can be explained by the alloying elements especially Cr, Mo and N and unique behaviour and portioning of the alloying elements of the two phases [3].

When testing DSS in corrosive environment the fatigue crack could initiate at the pitting marks, which act as a stress concentrators. Pitting marks occur when localized corrosion takes place.

Resistance to pitting corrosion can be evaluated by the pitting corrosion resistance number (PREN), which is calculated in literature [5] according to Eq.(1)

$$\text{PREN} = \text{wt\% Cr} + 3.3 (\text{wt\% Mo}) + 16 (\text{wt\% N}) \quad (1)$$

DSS-s are divided into two groups according to PREN. Duplex PREN <35 and super duplex PREN >40.

Both 3RE60 SRG and Duplok22 belong to the first group with PREN number about 34 and about 29 respectively [3,5].

Table 1. Chemical composition of the tested DSS-s [2,3].

Steel grade	C, %	Mn, %	Cr, %	Ni, %	Mo, %	N, %
3RE60 SRG	0.02	1.50	18.50	4.90	2.8	0.08
Duplok22	0.03	0.60	22.0	6.10	3.05	0.15

2.3 Mechanical properties

Low-nitrogen DSS good mechanical strength originates from the ferrite phase and high impact toughness from the austenite phase compared to conventional austenitic stainless steels [3,5]. The mechanical properties are not affected by the thickness or the orientation of the microstructure in P/M HIP-ed DSS-s [3]. Austenite is reported to be the weaker phase even when N content is as high as 0.32%.

The mechanical properties of the tested DSS-s are shown in Table 2.

Table 2. Mechanical properties of tested DSS-s [2,3].

Steel grade	Tensile strength, MPa	Yield strength, MPa
3RE60 SRG	719	440
Duplok22	739	499

3. EXPERIMENTAL

3.1 Test method and equipment

To determine the fatigue limit of DSS-s axial push-pull method with constant stress ratio $R = -1$ was used. The testing was carried out under load-controlled regime at 15 Hz frequency. Instron 8516 type 100 kN servo-hydraulic test machine was used.

Test chamber was made of stainless steel (Fig. 2) and coated with Teflon. Test chamber window is made from acrylic plastic. A membrane pump was used to circulate the test solution about twice an hour (test chamber volume about 0.7 l). Specimens were submerged by the parallel part into the test solution.

Test end criterion was failing of the specimen at any given stress level or reaching 10^7 cycles.

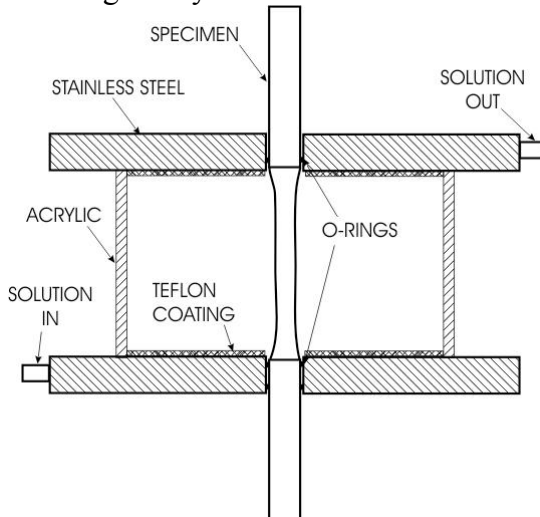


Fig. 2 Test cell

The variable was the maximum stress level S_{max} , and output cycles count. The S_{max} values were chosen to cover the S/N curve from 10^4 to run out (10^7) cycles.

The specimens were round \varnothing 10 mm at the grips, \varnothing 6 mm at the 24 mm long parallel part and 150 mm in total length.

The specimens were mechanically polished at the parallel part to remove scratches using a polishing wheel and abrasive. Diameter and surface roughness Ra was measured. The required Ra was <0.6 .

3.2 Testing environment

Testing environment was a modified TIP 0402-09 type II test solution. Solution containing NaCl, $Al_2(SO_4)3 \cdot 18H_2O$ and $Na_2S_2O_3 \cdot 5H_2O$ dissolved in distilled water.

The test solution pH was adjusted to 3.50 by adding 1 vol% H_2SO_4 .

The test solution composition was following (prior to pH adjustment):

Cl^{-1} – (1000 ppm);

SO_4^{2-} – (800 ppm);

$S_2O_3^{2-}$ – (200 ppm).

The expiration time for a solution was three days.

The test solution pH tended to increase about 0.1 pH units per 24 hours and then stabilise at about pH 3.70.

4. TEST RESULTS

According to the material specification [2] the fatigue limit for 3RE60 SRG is 200 MPa at 5Hz and 265 MPa at 25 Hz, obtained with test solution containing 400 ppm Cl^{-1} , 250 ppm SO_4^{2-} ions and pH 3.50.

Fatigue limit, as it follows from Fig. 3, for the 3RE60 SRG was 250 MPa is mean value when compared to the literature results when taking account the testing frequency.

Fatigue limit, as it follows from Fig. 4, for the tested Duplok22 was 350 MPa.

According to the material specification [2] the fatigue limit for Duplok22 is 275 MPa (10^9 cycles).

This result is obtained with less corrosive test solution containing 100 ppm Cl^{-1} , 1000 ppm SO_4^{2-} ions and pH 3.5 (TIP 0402-09 type I).

Duplok22 higher fatigue limit compared to 3RE60 SRG is due to its fine microstructure, which in inhibits the growth of a fatigue

crack and also due its higher alloying element content [3]. Fatigue cracks tend to grow in ferrite, austenite phase tends to retard the crack [3].

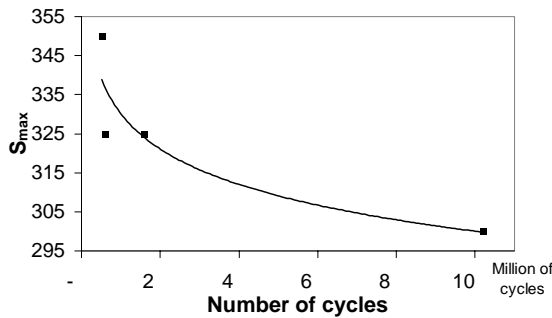


Fig. 3 3RE60 SRG S/N curve

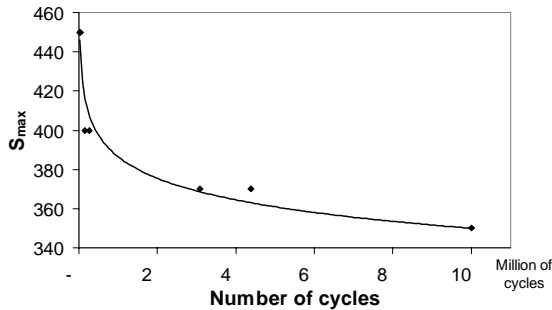


Fig. 4 Duplok22 S/N curve

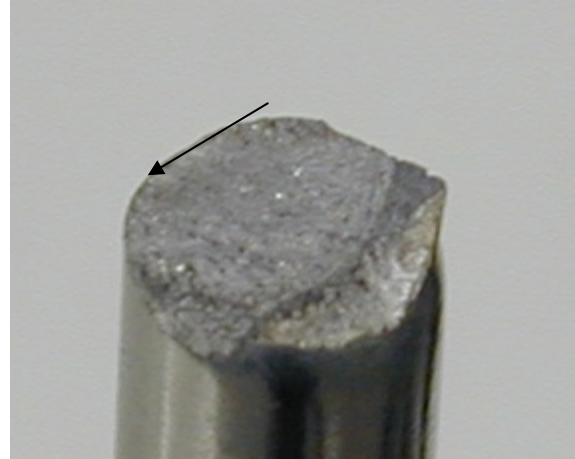
5. FRACTURE SURFACE ANALYSIS

In the case of high-cycle fatigue the initiation of cracks takes mainly place at non-metallic inclusions situated at or near the surface of a specimen [3]. In corrosive environment pitting marks may enhance the initiation of cracks [3].

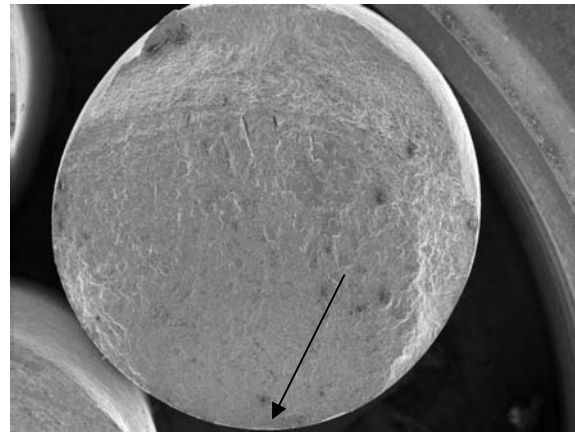
The influence of inclusions is more important in the case of P/M HIP-ed DSS-s like Duplok22 [3]. HIP-ing process is used to obtain fully dense materials [8]. Certain period of time is needed for corrosion to occur. Under high frequency testing the crack tip may not be exposed to corrosive media for significant time [9]. In the tested materials first signs of pitting marks occurred within 24 hours of being exposed. Fracture surfaces were examined using scanning electron microscope (SEM) and light optical microscope (LOM).

Fracture surface analysis revealed that fatigue crack initiation could be traced to one certain initiation point in all the tested specimens (Fig. 5, 6).

In the case of low cycles counts, 10^4 and 10^5 cycles, the initiation site was an inclusion situated on or near the surface (Fig. 5, 6).



a) 3RE60 SRG $5 \cdot 10^5$ cycles (LOM)



b) 3RE60 SRG $5 \cdot 10^5$ cycles (SEM).

Fig. 5 Crack initiation point

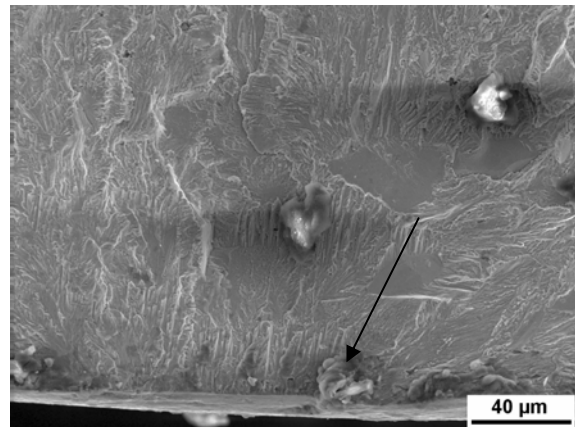


Fig. 6 Crack initiation point 3RE60 SRG $6 \cdot 10^4$ cycles (SEM)

In case of 10^6 cycles the effect of corrosion is more pronounced. A pitting mark may act as a crack initiation point (Fig. 7).

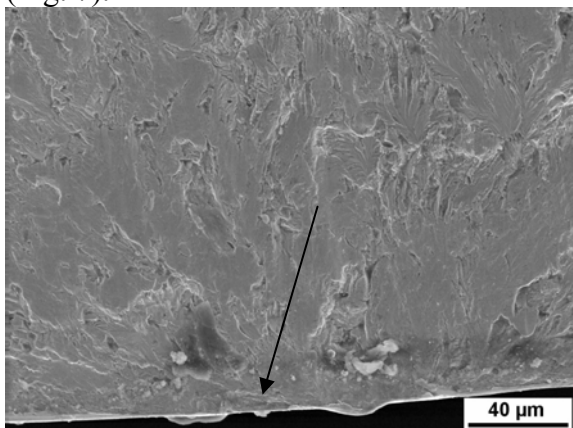


Fig. 7 Pitting mark as crack initiation point 3RE60 SRG $1.5 \cdot 10^6$ cycles (SEM)

6. CONCLUSION

In the case of corrosion fatigue testing of the duplex stainless steels the test parameters have significant influence on the test results especially the testing frequency.

Corrosion needs time to form pitting marks that could act as fatigue crack initiation spots.

Both tested steels (3RE60 SRG and Duplok22) were affected by the pitting corrosion when exposed to the test solution for at least 24 hours.

Duplok22 higher fatigue limit is due to its fine microstructure in both TIP 0402-09 type I and type II solution.

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