Fatigue behaviors in duplex stainless steel studied using in-situ SEM/EBSD method

Guocai Chai\textsuperscript{1,2}, Ru Lin Peng\textsuperscript{2} and Sten Johansson\textsuperscript{2}

\textsuperscript{1}Sandvik Materials Technology, SE-811 81 Sandviken, Sweden
\textsuperscript{2}Linköping University, Engineering Materials, SE-58183 Linköping, Sweden

Abstract

Austenite and ferrite in duplex stainless steels have different physical and mechanical properties. They can behave different during cyclic loading. To understand the fatigue behaviors of these two phases, an in-situ SEM/EBSD fatigue test has been performed. Flat specimens made from the specimens of pre-fatigue tested with three point bending were cyclically loaded in a scanning electron microscope via a compact test rig. By in situ/ex situ SEM/EBSD examination, slip activities and propagation of the fatigue cracks have been studied. Microstructures along the path of the fatigue crack were characterized. The different phase properties seem to lead to certain difference in the slip activity and formation of PSBs. Inhomogeneous slip activities and local strain concentrations were also found, which developed with increasing number of load cycles. Crack propagation behaviors in grain and cross the grain or phase boundaries have been discussed. Crack deflection occurs at the phase boundaries, but crack branching occurs mainly in the grains due to the dislocation slip. In-situ SEM/EBSD fatigue test confirms that crack propagation deflection and formation of crack branches can significantly reduce the crack propagation rate.

© 2014 Elsevier Ltd. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/3.0/).
Selection and peer-review under responsibility of the Norwegian University of Science and Technology (NTNU), Department of Structural Engineering

Keywords: Fatigue, Fatigue crack propagation, in-situ SEM/EBSD, crack branching, crack deflection.

* Corresponding author. Tel.: +0-000-000-0000 ; fax: +0-000-000-0000.
E-mail address: author@institute.xxx
1. Introduction

Duplex stainless steels (DSSs) are a group of steels that contain almost equal amount of ferritic ($\alpha$) and austenitic ($\gamma$) phase. These materials have a good combination of excellent corrosion resistance and mechanical properties. Since the two phases in the steel have different crystallographic and mechanical properties, the behavior of the material are strongly dependent on their microstructure and micromechanical behavior of the constitutive phases. Material damage and propagation of short cracks in DSSs under cyclic loading has been studied by a number of researchers who combined fatigue tests and microstructure studies, by Alvarez-Armas et al. (2007), (2008), (2009), or Christ et al. (2008), or Balbi et al. (2008), or Callone et al. (2004). Their work indicates the complex nature of fatigue process in such materials and the important role of microstructure in the crack initiation and early propagation. Influenced by the properties of the constitutive phases and the type and amplitude of cyclic load, fatigue damage in the form of microcrack initiation may occur inside $\alpha$, $\alpha/\alpha$ or $\alpha/\gamma$ phase boundaries. As the analyses in these studies has revealed, the slip activities and localization of cyclic plastic strains and crystallographic orientations of the involved grains affect significantly the fatigue process.

It is known that fatigue cracking can significantly deviate from the propagation directions that should be perpendicular to the main stresses in mode I. This may lead to the formation of crack deflection, kinking or branching, by Lankford et al. (2004) or Suresh (1983), and consequently cause significant retardation or even arrest of the subsequent crack propagation by reducing crack driving force, by Suresh (1983) or Meggiolaro et al. (2003). Although it is very difficult to analyze propagation behavior of branched cracks, several analytical solutions have been developed to predict the propagation path of a branched crack and to calculate the mode I and II stress intensity factors. They confirm that the formation of fatigue crack branches reduce the stress intensity factors and therefore the crack propagation rate. Less work could be found on the interaction of the microstructure and a growing macroscopic crack in DSSs. The aims of the current work are to investigate the crack initiation behavior using local cyclic plasticity and growth behavior of fatigue crack under cyclic loading in one duplex stainless steel of austenite and ferrite, SAF 2507 using an in-situ SEM/EBSD fatigue tester and a conventional da/dN test. Slip activity in the area near the crack tip and advancing of the crack tip were studied first under in situ cyclic loading and then by ex situ cyclic loading. Three imaging techniques, namely secondary electrons (SE), electron backscatter diffraction (EBSD), and electron channeling contrast (ECC) mode were employed.

2. Materials and experimental

The material used was a hot rolled bar material made of one austenitic and ferritic duplex stainless steel SAF 2507 (equivalent to UNS S32750) with a diameter of 80 mm (AD) and a nominal chemical composition as shown in Table 1 and 45% volume fraction of ferritic phase. The tensile properties in the rolling direction and the hardness of the austenitic phase and the ferritic phase (Vickers hardness) are also shown in Table 1. The two phases have closer hardness.

| C    | Si  | Mn  | Cr | Ni | Mo | N  | Fe  | $\sigma_y$ (MPa) | $\sigma_t$ (MPa) | Elongation (%) | $H_v,T$ | $H_v,N$ |
|------|-----|-----|----|----|----|----|-----|----------------|----------------|---------------|---------|---------|
| 0.03 | 0.8 | 1.2 | 25 | 7  | 4  | 0.3| bal.| 625            | 820            | 41.7         | 272     | 286     |

A conventional fatigue crack growth (FCG) test was performed using 18 mm thick compact C(T) specimens with TS orientation in an MTS servo-hydraulic machine (50kN). The main tests were performed using K-gradient constant value of $C_g=-0.08$ mm$^{-1}$ in air at RT. The mean stress ratio $R$ is 0.1 and the frequency is 10 Hz. The test was stopped when the crack propagation rate below $10^{-7}$ mm/cycle was reached.

To study the micro fatigue crack initiation and propagation behavior, an in-situ SEM/EBSD fatigue tester was used. The in-situ fatigue testing was performed inside a HITACHI SU-70 FEGSEM scanning electron microscope (SEM) using a specially designed Gatan microtoste-tensile test stage. A small specimen with a thickness of 1.513 mm and a width of 5 mm was machined from a three point bending fatigue pre-cracked specimen with the crack at the mid-length of the gauge section. The geometry of the specimen is shown in Fig. 1b. The length of the crack is...
1.18 mm. The specimen was mounted on the microtester which was then inserted into the sample chamber of the scanning electron microscope. With the build-in pre-tilting fixture the carefully prepared surface of the specimen was in 70° tilt with respect to the incident electron beam. The EBSD detector and Channel 5 software from the Oxford Instrument were used for the in-situ EBSP mapping.

Fig. 1. (a). Microstructure of SAF 2507 in the longitudinal direction, α: ferritic phase, γ: austenitic phase, (b). The geometry of the specimen for the in-situ SEM/EBSD examination.

For fatigue crack propagation test, a load varying from 1800 N to 2800 N was carried out first to find a suitable growth rate which allows the in-situ experiment to be performed within a reasonable time frame. Finally, a cyclic load between 2800 and 280 N (R=0.1) was induced with a frequency of 0.0125Hz. The fatigue test was stopped after loading to different number of cycles for imaging by SEM or EBSP.

3. Results and discussion

3.1. Fatigue crack propagation behavior in CT specimen

Fig. 2 shows the fatigue crack growth (FCG) rate (da/dN) versus stress intensity factor range (ΔK) curves from a conventional fatigue crack propagation test using a CT specimen in this study. A summary of the results is shown in Table 2.

Fig. 2. Crack growth rate versus stress intensity factor range for SAF 2507 tested with K-gradient constant Cg=-0.08.

Table 2 shows a summary of the fatigue crack propagation results from the conventional da/dN test. The Paris law constants, C_m and n, were evaluated. ΔK_{cl,th} and ΔK_{eff,th} are also compared. The results show that this material has a very high closure threshold, ΔK_{cl,th}, which is even higher than the effective thresholds, ΔK_{eff,th}. The thresholds
evaluated by analyzing the load versus crack opening displacement (COD) curves (sum of $\Delta K_{cl,th}$ and $\Delta K_{eff,th}$) are comparable to those determined from the da/dN versus $\Delta K$ curves, $\Delta K_{th,exp}$.

### Table 2. Paris law’s constants and threshold values.

| $C_m$ (mm/cycle) | n  | $\Delta K_{th,exp}$ (MPa$\sqrt{m}$) | $\Delta K_{cl,th}$ (MPa$\sqrt{m}$) | $\Delta K_{eff,th}$ (MPa$\sqrt{m}$) | $\Delta K_{th,exp} + \Delta K_{eff,th}$ (MPa$\sqrt{m}$) |
|------------------|----|-------------------------------|-------------------------------|-------------------------------|-------------------------------|
| $5.35 \times 10^{-10}$ | 3.52 | 8.64 | 5.35 | 3.90 | 9.25 |

#### 3.2. Fatigue crack initiation behavior during in-situ fatigue testing

The crack tip in the SAF 2507 specimen for in situ test was located in an austenite dominant area but oriented almost perpendicular to the loading axis (Fig. 3). The EBSD map in Fig. 3b shows similar plastic strain distribution around the crack tip. The load was cycled between $K = 2.1$ and $20.9$ MPa$\sqrt{m}$, giving a $\Delta K$ of $18.8$ MPa$\sqrt{m}$. No visible slip traces appeared at $K = 14.4$ MPa$\sqrt{m}$ but at $16.7$ MPa$\sqrt{m}$ the first slip systems became activated in grain 1 (Schmid factor, SF=0.45) and 2 (SF=0.43). One more loading step to $18.9$ MPa$\sqrt{m}$ induced gliding in grain 1 (SF=0.36) and 2 (SF=0.32). At the peak load of the first cycle, $20.9$ MPa$\sqrt{m}$, slip traces became obvious in grain 3 (SF=0.5 and 0.46). With increased number of load cycles,

![Fig. 3. Slip traces in SAF 2507 viewed in SEM at peak load at the first load cycle (a) and the 50th cycles (b). (c) An image of the same area taken in SE mode after 60 load cycles and removing of the specimen from the in situ stage. Only highest SF was given. For grain boundaries in (b), see captions in Fig. 3.](image)

PSBs developed and strong PSBs were observed near the crack tip in grains 1 and 2 after unloading from 60 cycles. Closer examination also found a third slip system operated in grain 1 (SF=0.32), 3 (SF=0.36) and 4 (SF=0.45). Except for the one in grain 3, they left very weak markings in the surface. In spite of a lower $\Delta K$, surface markings at the PSBs were more profound.

Ex situ experiment was carried out under the same $\Delta K$, $21$ MPa$\sqrt{m}$. Examination of the crack tip was done at 0 cycle and after 1, 11 and 44 cycles. In grain 4, PSBs with a very low SF, 0.16, were also visible. Secondary cracks developed under cyclic loading in grain 1, not far away from the main crack, and grew along a few PSBs with SF=0.36. The small cracks merged to form a longer crack, which finally joined the main crack. In the meantime, growth of the main crack halted, possibly because of tip blunting by the formation of a secondary crack at the tip. Changes in the local strain fields were also obvious with the development of local plasticity and crack formation and propagation. The high concentration of local strains near the tip gradually decreased and finally relaxed. In the meantime, new strain concentrations developed in grain 3 and 4 close to their twin boundary.

#### 3.3. Fatigue crack propagation behavior during in-situ fatigue testing

The in-situ fatigue test was terminated after reaching 5100 cycle. The specimen was unloaded and the surface was cleaned with replica and acetone. Fig. 4a is a backscatter electron image, which reveals the crack geometry. Both crack deflection and branching can be observed. The first one appears at about 500 cycles (A) and the second one at about 3800 cycles (B), while the significant deflection is marked by 980 cycles (C). Fig. 4b is an EBSD image of the region taken before the fatigue crack passed through, in which the microstructure of the duplex stainless steel is shown. The high angle grain boundaries, plotted in thin black lines, are defined as larger than 10 degrees. A microstructure with fine austenitic grains (red) distributed in a matrix of relatively large ferritic grains.
(blue) is clearly shown. Furthermore, the distribution of austenite is not homogenous. In certain regions, bands containing mainly austenitic grains are observed.

Fig. 4. (a) Backscatter electron image taken after the in-situ fatigue test was terminated and unloaded; (b) EBSD image with crack growth path indicated by thick white line (the main crack) and thin white lines (branches). The dark line is the pre-fatigue cracking. The loading direction is horizontal. blue: ferritic phase, red: austenitic phase.

The fatigue crack path is also shown in Fig. 4b. The main crack, marked with thick white line, grew from the pre-cracking (thick black line). Obviously, the observed branching and deflection behaviors are dependent on the microstructure. Within the ferritic grains, the crack propagates in a transgranular manner. In the austenitic grains both intergranular and transgranular cracking can be observed. A local change of the microstructure, namely the existence of an austenite dominant region in the front of the crack tip causes a large deflection, see for example C in Fig. 4a, or branching (A and B). A closer observation reveals extensive plastic deformation and dense slip lines around the crack tip. Slip on multiple systems is dominant. For example, in the F1 grain two groups of slip bands, determined from the EBSD data to form on the (-112)[1-11] and (1-12)[-111] system, respectively, are observed at the crack tip. Both slip systems have a comparable Schmidt’s factor of 0.47 but operate in different directions.

The main crack length is plotted as a function of the number of loading cycles. The average crack propagation rates between two measure points can then be calculated, which are shown in Fig. 4c. As can be seen the crack growth rate changes at 500 cycles or 3800 cycles (regions A or B) and becomes lower (Fig. 4c). This can correlate to the formation of crack branching. The formation of crack branches will reduce the mode I crack driving force, which leads to a crack growth retardation for the main crack path, by Suresh (1983) or Meggiolaro et al. (2003). On the other hand, some crack branches propagate almost transversely to the main crack direction with a mode II SIF. In the beginning, they propagate with a rate that is much higher than that of the main crack (Fig. 4c), and then the growth will stop somewhere in spite of that its length is longer than that in the main crack path. This contradicts to the result from the analytical solution, by Suresh (1983). Fig. 4d shows a comparison of fatigue crack propagation rates from a conventional fatigue crack propagation test and that of in-situ test. They are comparable, but the micro fatigue crack propagation rates with the formation of branches are much lower.

Fig. 5. Crack propagation behavior in the duplex stainless steel, (a). Crack in the austenitic and ferritic phase, position C in Fig. 4. (b). Crack in the austenitic phase, position B in Fig. 4. (c). Crack in the ferritic phase, position D in Fig. 4.
Fatigue damage (dislocation slipping) and crack propagation behaviors in the austenitic and ferritic phase in this duplex stainless steel were found to be very heterogeneous as shown in Fig. 5. In the austenitic phase, the dislocation slipping is mainly planar (Fig. 5a) or cross planar slipping (Fig. 5b). The crack propagation path is the interaction area of the cross planar slipping (Fig. 5b). In the ferritic phase, the slipping looks like wave slipping in one slipping system or direction and planar slipping in another slipping system or direction (Fig. 5a). In the crack front, river shaped cracks can be observed (Fig. 5c) due to the interactions of dislocation slipping in different directions. At the phase boundary, the crack initiates due to the impingement of dislocation slipping from both the austenitic and ferritic phase. More damage in the ferritic phase can be observed. This is similar to the earlier observations since the ferritic phase can be a weaker phase after cyclic loading, by Lillbacka (2007).

4. Conclusion

Duplex stainless steel SAF 2507 has a high fatigue crack propagation threshold value, especially a very high closure threshold value.

In-situ fatigue testing shows that cyclic loading can promote dislocation glide on slip systems of very low SF. Crack initiation and propagation may not always occur on the slip systems oriented favorably for dislocation gliding. The heterogeneity of slip activities, such as that different slip systems operate in different parts of the same grain, can affect directly crack formation and growth.

Cyclic plastic strains are inhomogeneous, which are manifested not only by the formation of PSBs, but also by the different distribution and density of the PSBs. PSBs which are oriented transverse to the growing tip may also halt its growth by blunting the tip.

In-situ observation confirms that the formation of crack branches can significantly reduce crack propagation rate that leads to crack growth retardation in the main crack path. The crack branches formed are usually not ideal. They can propagate almost transverse to the main crack direction with a mode II SIF and a rate that is much higher than that of the main crack.

The branching behavior in the material leads to a high fatigue threshold value since they have caused high crack growth retardation and high crack closure.

5. Acknowledgements

This paper is published by permission of Sandvik Materials Technology. The assistance of the fatigue testing by Mr Dan-Erik Gräll and Mr Gunnar Svensk is gratefully acknowledged.

References

Alvarez-Armas, I., Marinelli, M.C., Malaria, J.A., Degallaix, S., Armas, A.F., 2007, Microstructure associated with crack initiation during low-cycle fatigue in a low nitrogen duplex stainless steel, Int. J. Fatigue. 29, 758-64.

Alvarez-Armas, I., Krupp, U., Balbi, M., Herenu, S., Marinelli, M.C., Knobbe, H., 2012, Growth of short cracks during low and high cycle fatigue in a duplex stainless steel, Int. J. Fatigue. 41, 95-100.

Balbi, M., Avalos, M., El Bartali, A., Alvarez-Armas, I., 2009, Microcrack growth and fatigue behavior of a duplex stainless steel, Int. J. Fatigue. 31, 2006-2013.

Callone, V., Gourgues, A.F., Pineau, A., 2004, Fatigue crack propagation in cast duplex stainless steels: thermal ageing and microstructural effects, Fatigue and Fracture of Engineering Material and Structures. 27, 31-43.

Christ, H., Duber, O., Knobbe, H., Fritzcn, C., Krupp, U., Kunkler, B., Koster, P., 2008, Propagation behaviour of microstructural short fatigue cracks - experimental characterization and mechanism-based simulation, Materialwissenschaft und Werkstofftechnik. 39, 688-93.

Lankford, J., Davidson, D.L., 1981, The Effect of Overloads upon Fatigue Crack Tip Opening Displacement and Crack Tip Opening/Closing Loads in Aluminum Alloys. Advances In Fracture Research, Perg. Press, 2, 899-906.

Lillbacka, R., Chai, G., Ekh, M., Liu, P., Johnson, E., Runesson, K., 2007, Cyclic stress–strain behavior and load sharing in duplex stainless steels: Aspects of modeling and experiments, Acta Materialia, 55, 5359-5368.

Marinelli, M., El Bartali, A., Signorelli, J.W., Evrard, P., Aubin, V., Alvarez-Armas, I., Degallaix-Moreuil, S., 2009, Activated slip systems and microcrack path in LCF of a duplex stainless steel, Materials Science & Engineering A. 509, 81-8.

Meggiolaro, M.A., Castro, J.T.P., 2003, On the dominant role of crack closure on fatigue crack growth modeling. Int. J. of Fatigue, 25, 843-854.

Suresh, S., 1983, Micromechanisms of Fatigue Crack Growth Retardation Following Overloads, Engineering Fracture Mechanics, 18, 577-593.