Microstructural aspects of duplex steel during high cycle and very high cycle fatigue

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Abstract. Fatigue experiments were conducted up to \( N = 10^8 \) cycles on a duplex stainless steel. The investigations revealed important information: (i) most of the slip markings on \( \{111\} \)-slip planes form during the early stage of fatigue (~10⁴), although many grains stay active in developing more and/or longer slip bands (investigations with SEM/EBSD). It was found that the damage in these grains often has its origin at grain or phase boundaries, growing into the interior of the grains; (ii) in most of the recrystallisation twins slip markings are present in all parts of the grain leading to the assumption that just tilted grain boundaries are not very effective as microstructural barriers. In cases where the twin grain boundary is favorably oriented, it even can promote microcrack initiation; (iii) phase boundaries are most effective in restricting dislocation movement to the softer austenite, since no evidence of growing fatigue damage was found in ferritic grains. These findings lead to the conclusion that for the material studied an endurance limit exists despite occurring plastic deformation. This behaviour is a consequence of the barrier effect induced by phase boundaries against short crack propagation and holds true as long as no inclusions are present.

1. Introduction

During the last years much attention has been drawn to the investigation of metallic materials’ fatigue behaviour in the very high cycle regime, implying numbers of cycles to fracture ranging from \( 10^6 \) up to \( 10^{10} \) or \( 10^{12} \) cycles [1,2]. In consequence of the first results obtained mostly on high strength steels and aluminium alloys the ongoing discussion whether a fatigue limit exists or not was raised. Several authors found that many different influencing factors such as microstructural condition, prehistory, notches and experimental setup may have a crucial effect on the fatigue life [3]. However, it was discovered that materials can fail below the conventional fatigue limit [4], but most of the work concentrates on single-phase materials [5]. Few papers are available covering the complex situation of two-phase materials such as two-phase titanium alloys and duplex steels [6-8]. The crack initiation sites can be shifted from the surface in case of HCF to subsurface for VHCF loads. The latter can occur at inclusions or non-defect sites. In this study, only inclusions were found to promote subsurface crack initiation in some rare cases.
2. Material and Experimental

The present material X2 CrNiMoN 22-5-3 consists of a strong phase (α ferrite) and a soft phase (γ austenite). The strength distribution strongly depends on the alloying condition, primarily the nitrogen content [9]. The chemical composition for the material studied is given in table 1.

Table 1. Chemical composition of duplex steel in wt. %.

|   | Fe   | Cr   | Ni   | Mo   | Mn   | Si   | P    | S    | Cu   | Ti   | Co   | N    | C    |
|---|------|------|------|------|------|------|------|------|------|------|------|------|------|
| bal. | 21.91 | 5.58 | 3.08 | 1.83 | 0.53 | 0.023| 0.002| 0.11 | 0.03 | 0.07 | 0.19 | 0.02 |

The material was heat-treated in order to obtain a homogeneous microstructure. Heat treatment was done in laboratory air at 1250°C for 4h followed by controlled cooling to 1050°C in 3h leading to a phase fraction of 1:1. Afterwards the material was water-quenched to prevent the formation of brittle phases such as the σ phase. After heat treatment the surface oxide scale was mechanically removed.

Fatigue experiments were carried out at a stress ratio of R = -1 with a servohydraulic fatigue testing system (MTS) capable of frequencies up to 1000 Hz. The occurrence of excessive damping heat requires the use of compressed air cooling and restricts the testing frequency to 300 Hz. The cylindrical specimens had a diameter of 5mm with a gauge length of 10 mm in order to test a relatively large volume. All experiments were performed in stress control with a sinusoidal waveform and were stopped at 1x10^8 cycles. The damage evolution was followed by interruption of some tests after reaching defined numbers of cycles. The specimens used for these experiments contained a shallow-notch in the middle of the gauge length in order to localize the damage. All specimens were electrolytically polished prior to testing to obtain a surface without residual stresses allowing to measure orientation data. The EBSD technique was used to determine the distribution of the austenite and ferrite phases and the crystallographic orientations.

3. Results

The S-N-data of the material is published in ref. [10]. In summary, three different situations were observed at stress levels in the order of the endurance limit at 10^8 cycles: (i) failure of the specimens in the classical high cycle fatigue regime (~10^6 cycles) with crack initiation at the surface, (ii) failure with crack initiation at non-metallic inclusions with fish eye formation (~10^7 cycles) and (iii) no failure after 10^8 cycles (run-out specimens). The existence of slip markings at run-out specimens together with the measurable temperature increase during the experiments lead to the assumption that the occurring plastic slip is present during the whole experiment but reversible to some extent. In order to gain more precise information on the evolution of fatigue damage with ongoing cyclic loading, different areas were selected on the shallow notch and analyzed by means of SEM after certain numbers of cycles. Figure 1 shows the evolution of slip traces in one austenite grain, marked by the orange circle. Two important observations can be made: (i) the cyclic deformation starts at or near the phase boundary in the upper part of the grain and grows into the interior of the grain (this may be explained by incompatibility stresses raised by the different strengths of both phases [11]); (ii) after reaching the phase boundary at the lower part of the grain more and more other parts of the grain are involved, so no saturation can be found up to 2x10^7 cycles. This indicates a continuous plasticity during the whole experiment, which is in good agreement with the measured temperature increase. Orientation measurements by EBSD reveal that slip most frequently occurs on the {111} slip planes, but the phase boundaries obviously are strong enough to prevent the dislocations from crossing into the ferritic phase. Figure 2 shows another example of slip trace evolution for two interesting sites. The orange circle marks an austenite grain containing a twin boundary. This boundary is oriented in such a way that slip is induced on it. Two important observations can be made: (i) the cyclic deformation starts at or near the phase boundary in the upper part of the grain and grows into the interior of the grain (this may be explained by incompatibility stresses raised by the different strengths of both phases [11]); (ii) after reaching the phase boundary at the lower part of the grain more and more other parts of the grain are involved, so no saturation can be found up to 2x10^7 cycles. This indicates a continuous plasticity during the whole experiment, which is in good agreement with the measured temperature increase. Orientation measurements by EBSD reveal that slip most frequently occurs on the {111} slip planes, but the phase boundaries obviously are strong enough to prevent the dislocations from crossing into the ferritic phase. Figure 2 shows another example of slip trace evolution for two interesting sites. The orange circle marks an austenite grain containing a twin boundary. This boundary is oriented in such a way that slip is induced on it. It is well known for pure austenitic steels that twin boundaries act as crack initiation sites [12]. However, another slip system is also activated. In this case the slip traces become more pronounced during the fatigue process, but they do not cross the twin boundary, because the plastic deformation on the boundary, together with the unfavorable orientation of the grain, prevents slip into the other part of the grain. The grain highlighted by the blue circle is also a twin but...
shows another situation. Here, slip markings have crossed the boundary very early in the fatigue process leading to the assumption that generally twin boundaries do not have a strong influence on cyclic slip. This is in good agreement with the idea of special (coincident site lattice, CSL) grain boundaries. In this case CSL-boundaries of type $\Sigma 3$ are found, meaning that every 3rd atom contained in the boundary is shared by both sides of the twin, making it easy for the dislocations to move over the boundary. Furthermore this observation gives rise to the assumption that tilted boundaries are not very effective as barriers. In other words high twist angles are required in order to gain more efficiency against dislocation movement.

**Figure 1.** Slip traces after (a) $5 \times 10^4$, (b) $10^5$, (c) $5 \times 10^5$, (d) $10^6$, (e) $2 \times 10^6$, (f) $5 \times 10^6$, (g) $10^7$ and (h) $2 \times 10^7$ cycles showing that cyclic deformation starts at grain or phase boundaries.

**Figure 2.** Slip traces after (a) $5 \times 10^4$, (b) $10^5$, (c) $5 \times 10^5$, (d) $10^6$, (e) $2 \times 10^6$, (f) $5 \times 10^6$, (g) $10^7$ and (h) $2 \times 10^7$ cycles showing that cyclic deformation occurs throughout the whole experiment.

Figure 3 indicates that areas near the phase boundaries do not only induce cyclic slip but can also promote microcrack initiation. These findings could be linked to incompatibility stresses together with elastic anisotropy. In this case a stage I crack has formed on a single slip system in the austenite, but obviously the crack is not able to grow (a,b). Figure 3 c) shows a detailed micrograph of the slip bands with considerably strong intrusions and extrusions. But still there are areas in between the slip traces that are not deformed up to this stage of fatigue. The observations above led to the assumption that these areas will be involved in the cyclic deformation with higher numbers of cycles. Finally, figure 3 d) gives an insight into the microcrack formation on the slip band. It is clearly visible that the crack started from an intrusion growing with a certain angle inside the material. This is typically observed
for stage I microcracks [13]. The situation of non-growing cracks can be governed by two factors: (i) the phase boundary in the lower part (figure 3 a,b) simply arrests the crack. In other words the crack-driving force is not high enough to exceed the critical stress in the ferrite since this is the stronger phase; (ii) the incompatibility stresses which are superimposed to the external loading are released by the crack initiation. Once the crack has formed and the microstructural stresses are gone, the external stress is not high enough to cause further crack growth and thus an endurance limit can be defined, at least as long as no inclusions are present in the tested volume.

![Figure 3](image)

**Figure 3.** Microcrack formation near the phase boundary on a slip band after (a) \(N=5\times10^6\), (b) \(N=2\times10^7\), (c) \(N=2\times10^7\) (intrusions and extrusions in slip bands), (d) \(N=2\times10^7\) (microcrack initiation at intrusions).

### 4. Conclusions
The fatigue behaviour of duplex steel in the very high cycle fatigue regime was investigated with special emphasis on fatigue damage evolution. It was found that cyclic slip is present during the whole experiment in the softer austenite. Twin boundaries play an important role because they either act as damage initiation sites or promote slip across the boundary. However, microcracks predominantly form near phase boundaries due to incompatibility stresses. Such cracks do not grow further as soon as the microstructural stresses are reduced. This leads to the definition of an endurance limit despite ongoing plastic deformation if no inclusions are existent.

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