Microstructure characterization in fatigue analyses

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Contents

- Introduction: How to characterize a fatigue test specimen?
- Case I: Round-robin on striation spacing evaluation (INCEFA SCALE)
- Case II: Microstructural analysis on effects of PWR water, surface treatment and strain amplitude on the fatigue behaviour of SS304L (INCEFA PLUS)
- Case III: Influence of mean stress and LWR environment on fatigue life and dislocation microstructures of SS316L

Introduction: How to characterize a fatigue test specimen?

1 Macroscale Characterization of Gauge Section and Fracture Surface

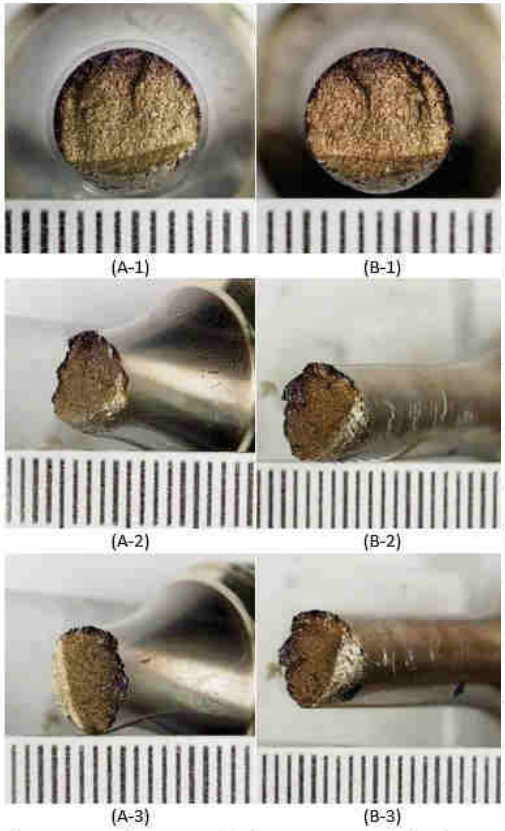


Figure 1 Example macroscale documentation of the fracture surfaces of a failed solid specimen. A and B are referring to different halves.

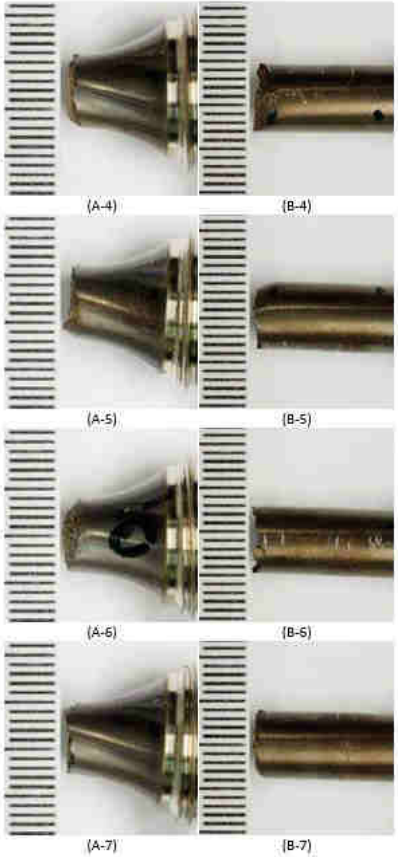


Figure 2 Example macroscale documentation of the gauge section of a failed solid specimen (both halves).

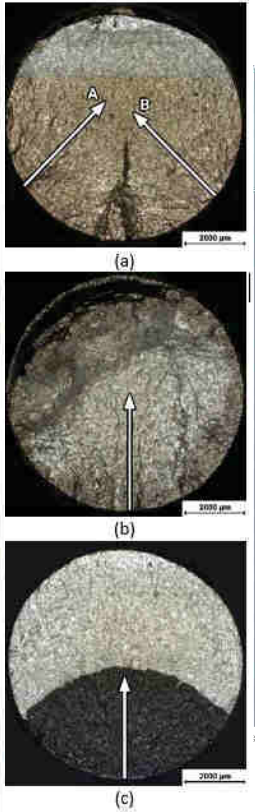


Figure 3 Example identifications of crack paths from macroscale images of failed solid specimen fracture surfaces.

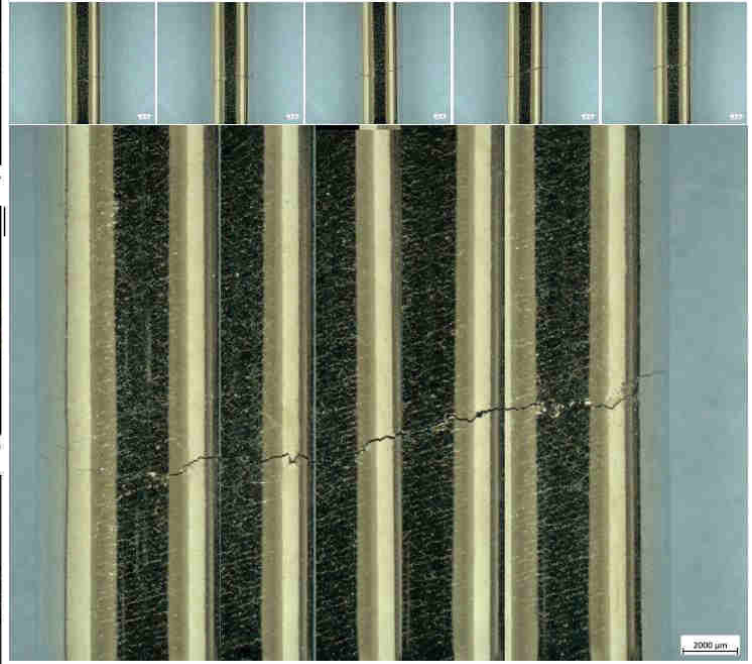


Figure 4 Example result for stiching together five images of the gauge section of a failed solid specimen.

2 SEM Fractography and Striation Spacing Evaluation

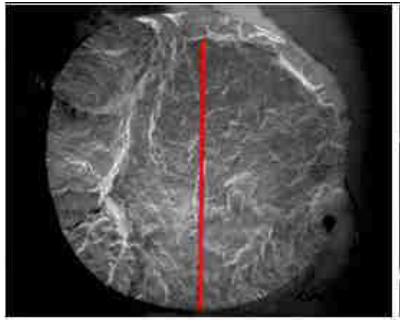


Figure 5 Example of choosing the most dominant crack associated with the largest area on the fracture surface of a failed solid specimen.

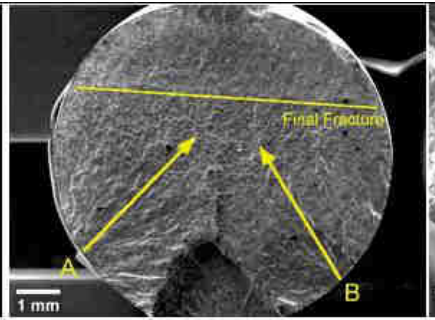


Figure 6 Example identification of two "main" crack intiation sites and paths, and the region of final fracture on the fracture surface of a solid specimen.

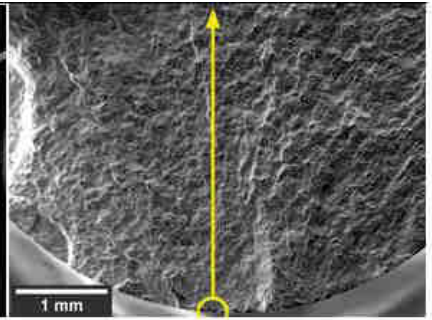


Figure 7 Example identification of a initiation site and a suitable crack path for striation counting on the fracture surface of a failed solid specimen.

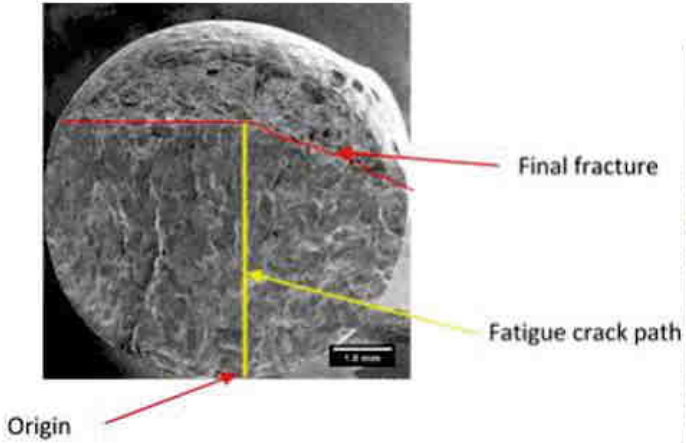


Figure 8 Example identification of a crack path from initiation site to the region of final fracture on the fracture surface of a failed solid specimen.

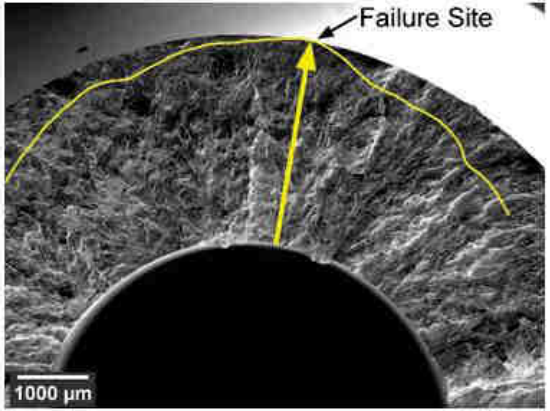


Figure 9 Example identification of the "main" crack path, from initiation site to the failure site on the fracture surface of a failed hollow specimen.

2 SEM Fractography and Striation Spacing Evaluation

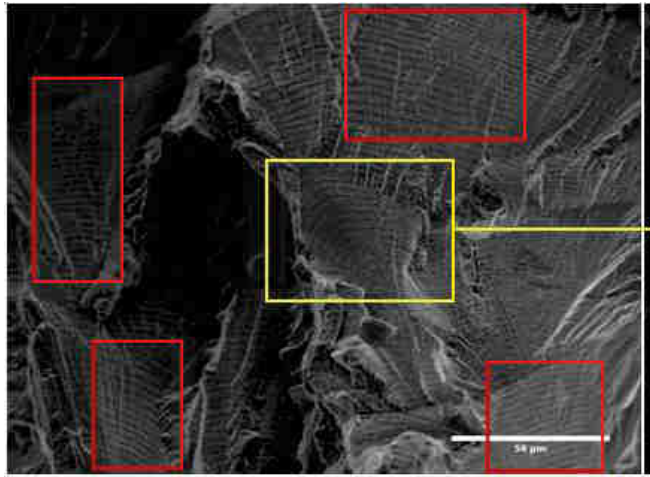


Figure 10 Example reference image taken with a suitable magnification so that at least three striation patches can be observed.

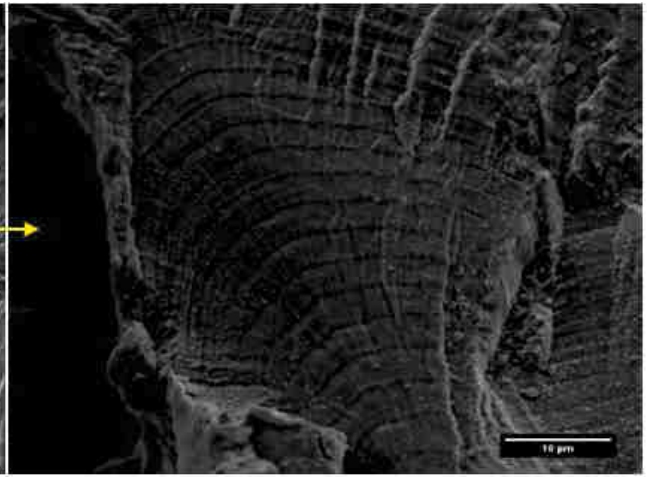
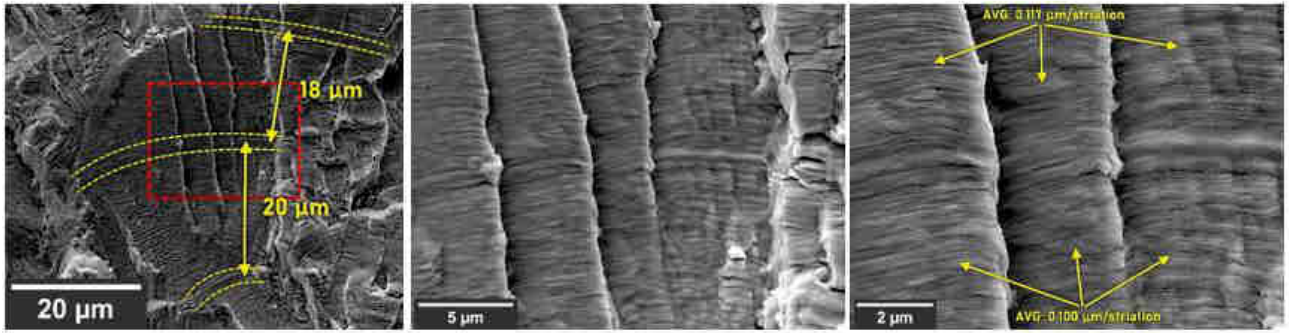


Figure 11 Example higher magnification image of a individual striation patch that is suitable for striation counting.



(a) (b) (c)

Figure 12 Example micrographs of location with evidence of two overload cycles.

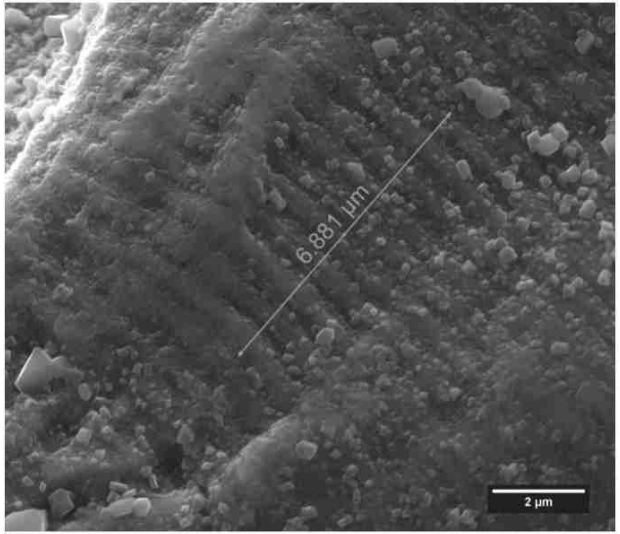


Figure 13 Example of a measurement line perpendicular to striations. The line starts and ends at a through, and extends over 10 striations.

3 Cross-Sectional Characterization of Near-Surface Region and Crack Tips

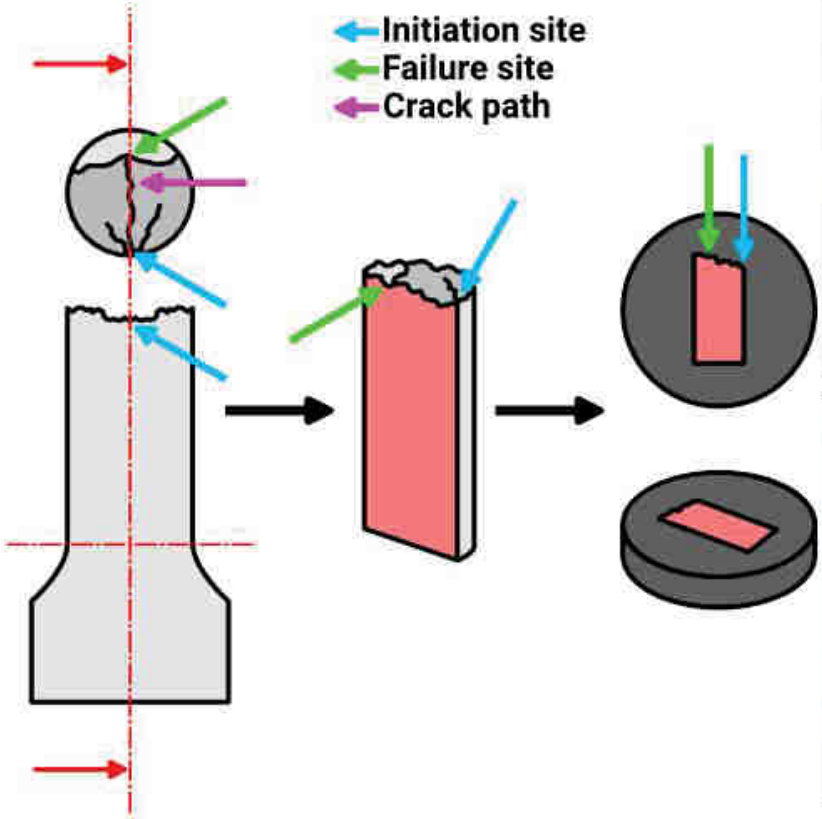


Figure 14 Cross-sectioning of a failed solid specimen.

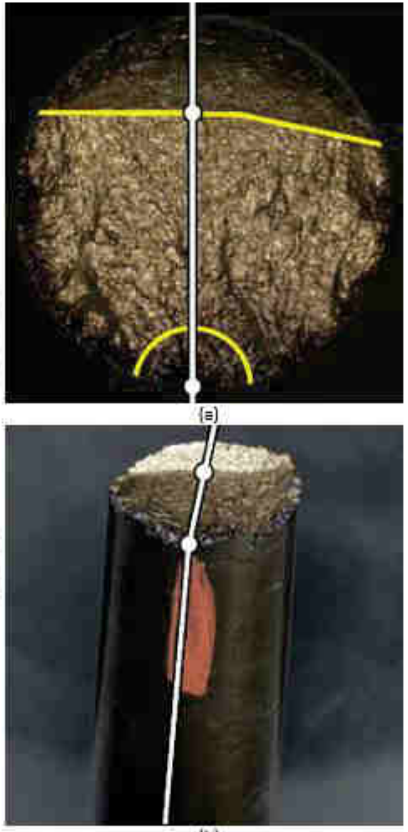
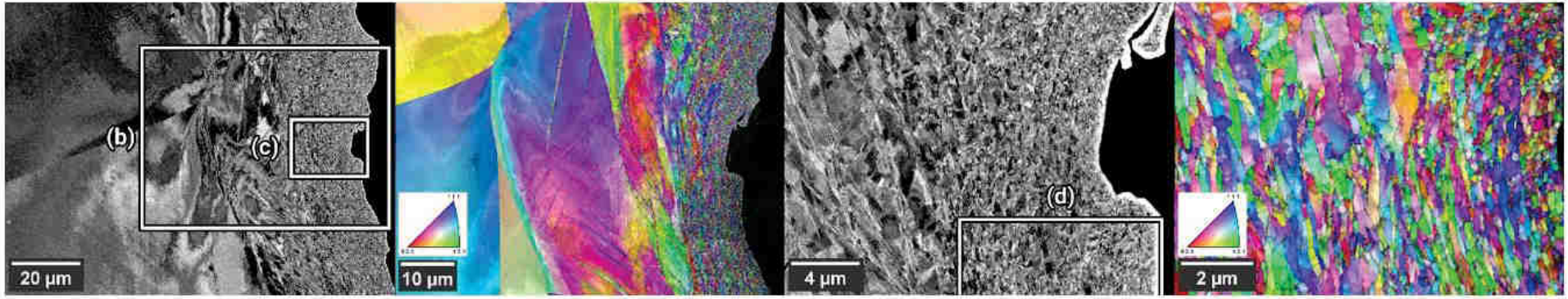


Figure 15 Example of cutting lines/cross-sectioning of a failed solid specimen.

3 Cross-Sectional Characterization of Near-Surface Region and Crack Tips



(a) (b) (c) (d)

Figure 17 Example BSE images and EBSD IPF maps of near-surface region showing the layered microstructure.

Refs:

- A. Vainionpää, J. Spadotto, Z. Que, B. Connolly, A. Mclennan, S. Arrieta, J. Huret, T. Damiani, in: Proceedings of the ASME Pressure Vessels & Piping Conference (PVP2023), PVP2023-105888, Atlanta, USA, 2023.

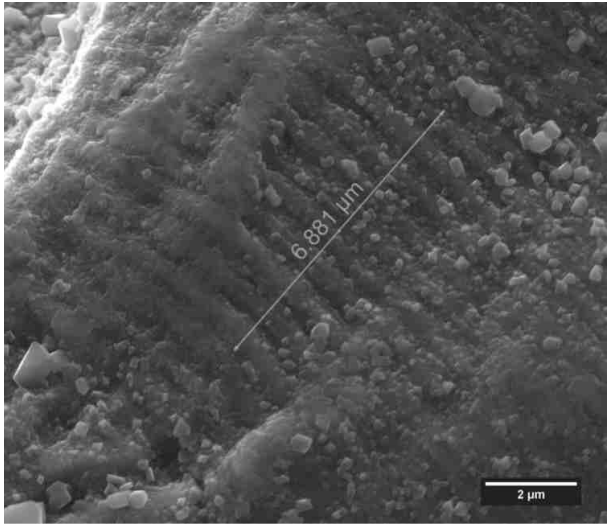
Case I: Round-robin on striation spacing evaluation (INCEFA SCALE)

Fatigue crack

- The process of accumulating fatigue damage can be separated into a series of stages that are each governed by different physical mechanisms. In a nominally uncracked specimen that is subjected to suitable cyclic loading, fatigue damage first develops as an accumulation of plastic strain in the microstructure, typically in areas surrounding geometric discontinuities such as at the surface of the specimen and around defects such as pores or inclusions. Eventually, this leads to the local separation of material and nucleation of a microstructurally short crack (MSC).
- Once a crack has been established, if the loading is sufficient to overcome intrinsic microstructural barriers (e.g. grain boundaries) the crack will propagate as a microstructurally short crack, with growth rates being heavily influenced by the local microstructure and difficult to predict using standard engineering methods. As the crack grows, the relative influence of the microstructure becomes less important as the crack front samples a larger volume of the material. At this stage the crack can be described as being mechanically short (100-500 μm), with growth rates become more stable and able to be described with improved confidence using elastic plastic fracture mechanics techniques.

Fatigue striation

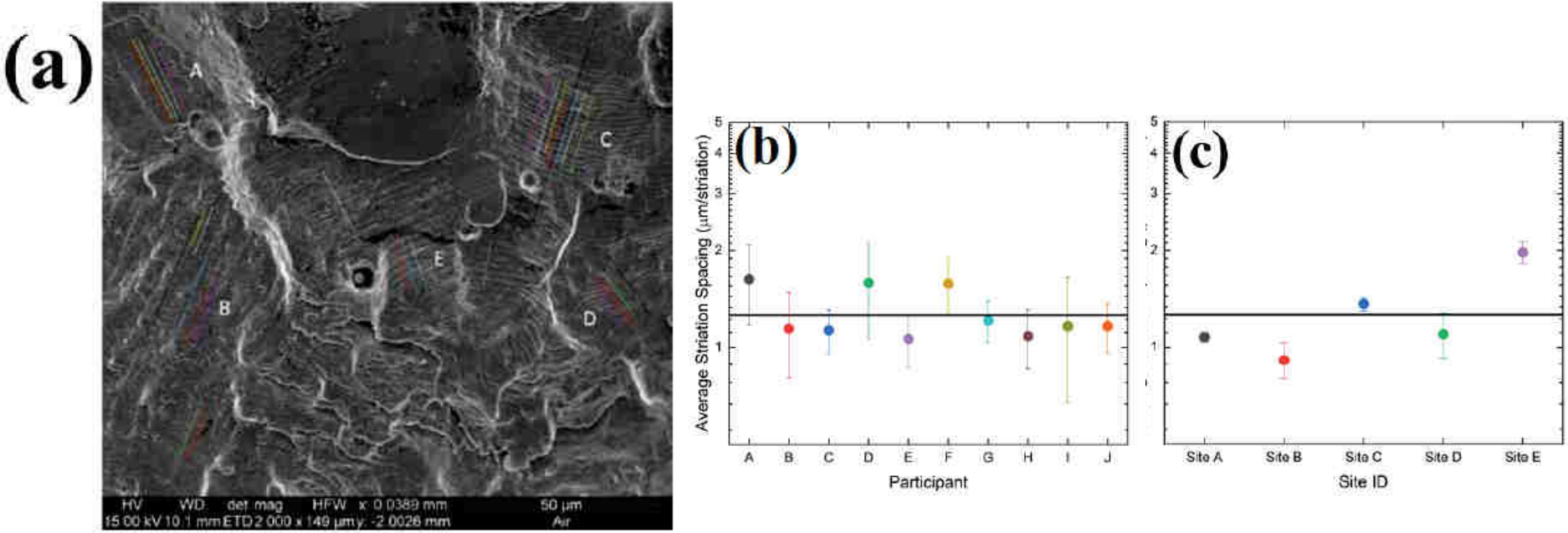
- As the crack continues to grow, the relative size of the plastic zone at the tip of the crack becomes smaller compared to the overall component size and crack growth rates become readily predictable using standard linear elastic fracture mechanics techniques. At this point (1-2 mm) the crack can be considered to be propagating as a long crack and will continue to grow until some failure criterion such as plastic collapse or fracture leads to the failure of the component.
- During crack propagation stages, plastic deformation at the crack tip can lead to the formation of local features on the fracture surface called striations. Striations are typically characterised as linear features that are parallel to the propagating crack front with a spacing that is proportional to the rate of crack advance.



Fatigue striation

- A key enabler for the development of improved analytical methods and mechanistic understanding is the ability to separate out and understand crack propagation throughout each of the individual stages of fatigue. In the absence of in situ monitoring of crack growth during an endurance test (which is experimentally complicated to implement) striation data represents a valuable way of reconstructing the crack history following the conclusion of the experiment.
- Whilst striation counting is a commonly used technique in failure investigations, to the authors' knowledge there are no standardised procedure s or universal guidelines for the measurement and evaluation of fatigue striations on endurance specimens exposed to a simulated PWR primary environment. In recognising the challenges associated with striation counting, it was decided that a standardised procedure should be developed to ensure common working practices.

Fatigue striation evaluation



Striation Counting Protocol

The procedure described below has been developed within the framework of the INCEFA-SCALE Programme for the evaluation of striation spacing's on fatigue endurance specimens tested in a simulated PWR environment and RT or HT air. This procedure can be applied after the identification of a single main crack on the fracture surface. This procedure should be applied with caution to specimens with multiple crack pathways. The procedure described can be applied to both hollow and solid LCF specimens.

For conciseness, this procedure is presented in an abbreviated form in this document with only the main points highlighted. Additional guidelines and detailed instructions have been omitted at this point in time. The procedure is undergoing continuous development within the INCEFA-SCALE Programme.

NOMENCLATURE[1]

FoV Field of View

STRIATION CAPTURING PROCEDURE

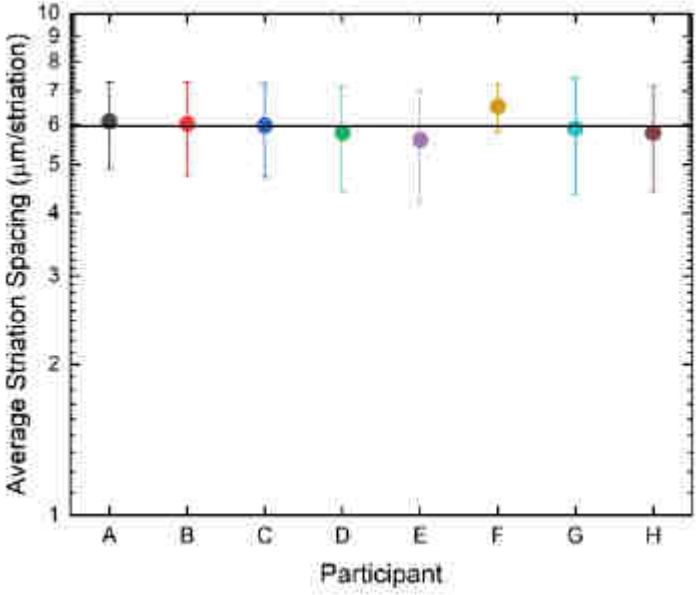
The following describes the procedure for capturing the variation in striation spacing along the fracture surface:

1. Orientate the specimen within the SEM so that crack path is aligned with the Y (vertical) axis of the microscope.
2. Centre the Field of View (FoV) onto the initiation site and record the X and Y stage coordinates. This represents the initiation site co-ordinates from which all subsequent crack depths will be measured.
3. Centre the FoV onto the final failure site and record the X and Y stage coordinates. This will provide the final crack depth when the test was stopped.
4. Starting from the initiation site, select a suitably high magnification and move along the crack path in small increments until striations can be clearly observed. The precise magnification and movement step will be dependent on the loading conditions and may vary for different specimens. The operator should use their best judgement and draw on previous experience for selecting the magnifications and displacements.
5. Identify a FoV and magnification where approximately five (5) striation patches can be observed. The aim is to obtain an average of several striation patches for a given crack depth to minimise bias.
6. Iterate through the following steps until no less than ten (10), but ideally more, sites across the crack path have been captured.
   a. Record X and Y stage coordinates at centre of FoV.
   b. Capture image(s) according to principles described in Section 4.
   c. Advance FoV by at least a distance that prevents overlapping of adjacent FoV's.
7. For most FoVs it is expected that a single image will be suitable for performing the striation counting. However, at smaller crack depths it may be difficult to accurately resolve striations in the reference image due to obfuscation by the oxide and smaller spacings. In these cases, additional higher magnification images of individual striation patches can be taken but the calculated depth from the original FoV should be assumed for each image.

STRIATION COUNTING

The following describes how striations should be counted from the images captured on the SEM. It is recommended that some sort of image processing software be used to perform this analysis.

1. Measure the scale by setting the number of pixels per micron, this can be done by measuring the scale bar.
2. Identify a clearly defined patch of striations
3. A measurement line should be drawn perpendicular to the striations. This line should aim to cover at least a minimum of ten (10) striations. The line should start and end at the same feature, either in a trough or at a striation peak. An example of this is shown in Fig. 1 of the main publication.
4. Where it is not possible to resolve ten (10) striations but striations are clearly visible, it is permissible to measure a smaller number but this should be documented in the results file.
5. Label each line clearly on the image with a letter (e.g. "a", "b", etc.).
6. A marked up version of the image featuring these measurement lines should be recorded along with the raw image.



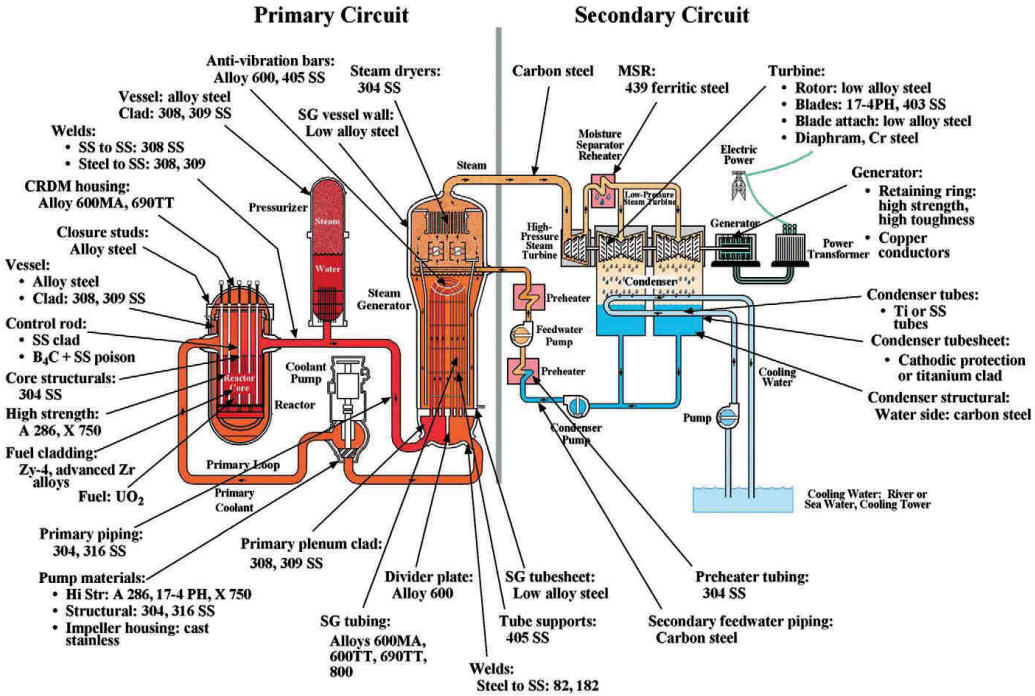
Refs:

- B. Howe, J. Mann, Z. Que, C. Huotilainen, F. Scenini, G. Burke, Development of a Robust Procedure for the Evaluation of Striation Spacings in Low Cycle Fatigue Specimens Tested in a Simulated PWR Environment, in: Proceedings of the ASME Pressure Vessels & Piping Conference (PVP2022), PVP2022 84027 Las Vegas, Nevada, USA, 2022.

Case II: Microstructural analysis on effects of PWR water, surface treatment and strain amplitude on the fatigue behaviour of SS304L (INCEFA PLUS)

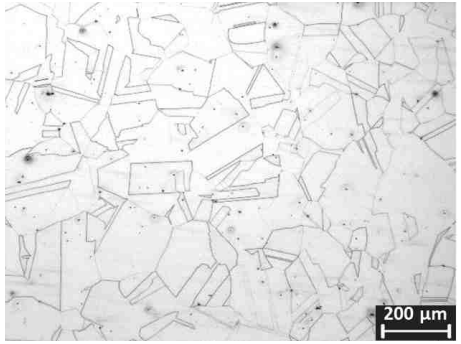
Background & Motivation

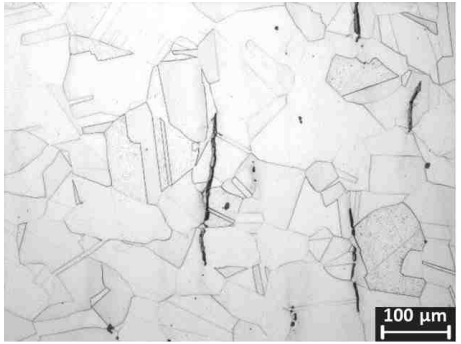
- Austenitic stainless steels are a material of choice for primary circuit pipelines of pressurized water reactors (PWR)
- Exposure to PWR primary environment leads to a decreased fatigue life than air
- The effect of surface treatment on CF?
- The effect of environment on fatigue crack tip microstructure?



Materials & Methods

- Solution annealed NG 304L
  - Longitudinal grain size: ASTM 3-4
  - Transverse grain size: ASTM 3
  - Approx. 3% δ-ferrite in rolling direction
  - Two surface finishes
    - polished (laboratory finish) 2700 FEPA and fine axial polishing marks at a 45° angle to the loading/unloading direction
    - ground (worst case plant finish) using an automated grinding bench for cylindrical fatigue test specimens, resulting in circumferential grinding marks along the entire gauge length and transition radius of the fatigue test specimen





	Fe	C	Mn	Si	S	P	Ni	Cr	Mo	Cu	N
SS 304L	bal.	0.029	1.86	0.37	0.004	0.029	10.0	18.00	0.04	0.02	0.056

Materials & Methods

- Fatigue testing
  - high temperature air
  - simulated PWR primary site environment
  - Nominal strain amplitude (R = −1): 0.3% and 0.6% (strain rate of 1×10^{-4} s^{-1})
- Post mortem characterizations
  - Fractography & fatigue striation spacing evaluation
  - Microstructural investigations of cross-sections
    - near surface microstructure
    - crack tip microstructure

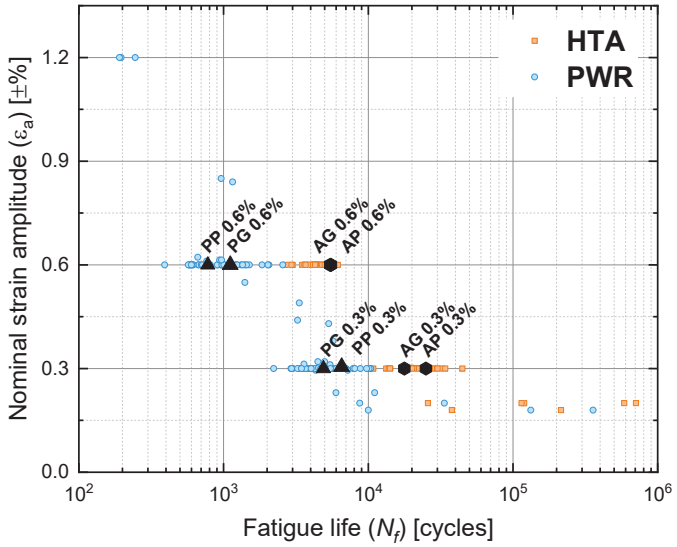
		Air	PWR
T	°C	300 ± 2	300 ± 2
Relative humidity	%	< 30	-
P	bar	atmospheric	150 (min)
[Li] as LiOH	(ppm)	-	(2 ± 0.2) ppm
[B] as $B(OH)_3$	(ppm)	-	1000 ± 100
O_2	(ppb)	-	< 5
H_2	($cc \cdot kg^{-1}$)	-	25 - 30
$pH_{300°C}$		-	6.95

N_f

- Eight representative specimens selected
- The fatigue life (N_f) criteria for failure was defined as the cycle at which the tensile peak stress of a cycle drops by 25 % from its steady state tensile peak stress
- Polished specimens tested in air or PWR water were referred to as AP and PP, while ground specimens tested in air or PWR water were referred to as AG and PG

Sample name	N_f (cycles)	R_a (µm)	R_t (µm)
AP 0.3 %	25020	0.04[i]	0.76[i]
AG 0.3 %	17768	2.18	25.96
AP 0.6 %	5450	0.04[i]	0.76[i]
AG 0.6 %	5525	5.20	44.60
PP 0.3 %	6547	0.04[i]	0.76[i]
PG 0.3 %	4897	1.46	13.65
PP 0.6 %	778	0.04[i]	0.76[i]
PG 0.6 %	1112	1.74	19.28

[i] indicates nominal value



N_f

- Specimens tested in HTA exhibit longer fatigue lifetimes, compared to those exposed to a simulated PWR primary environment.
- Negative impact of high strain amplitude and a ground surface finish, particularly at low εa, on fatigue lifetime were found

Sample name	N_f (cycles)	R_a (µm)	R_t (µm)
AP 0.3 %	25020	0.04[i]	0.76[i]
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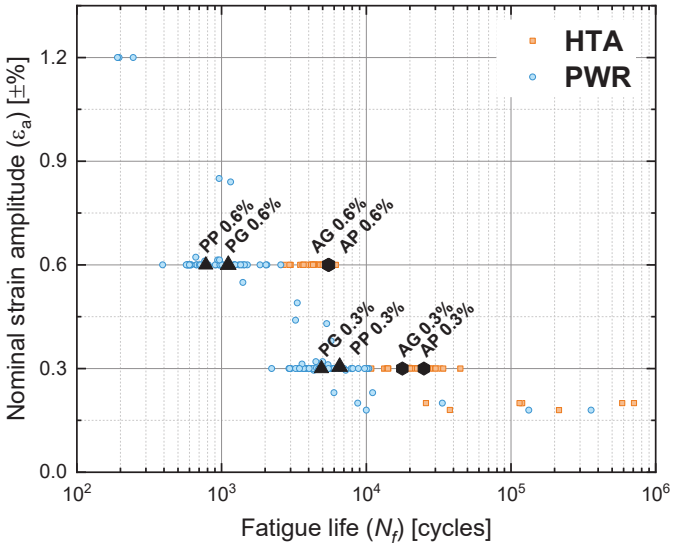
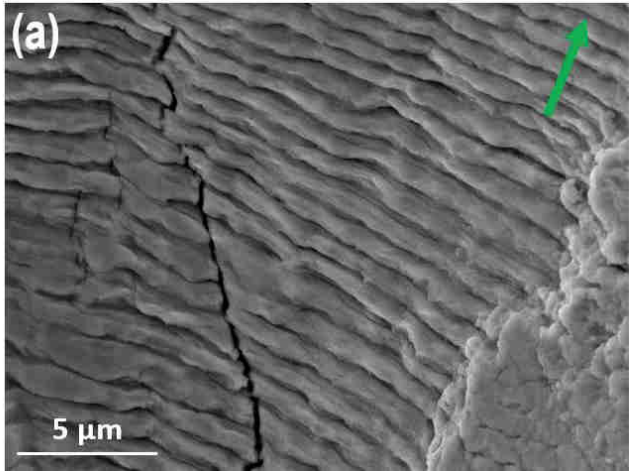
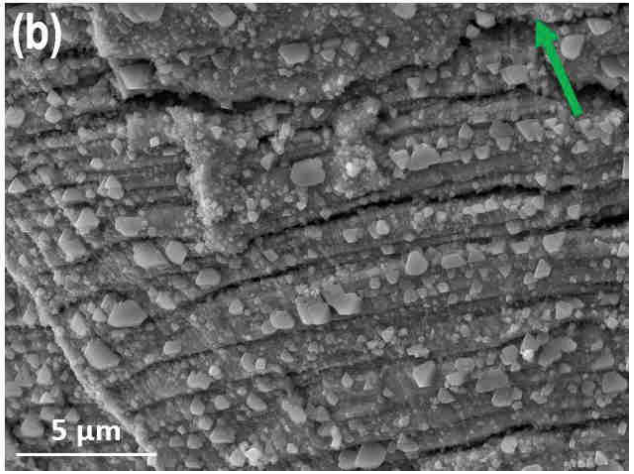


Fatigue striations

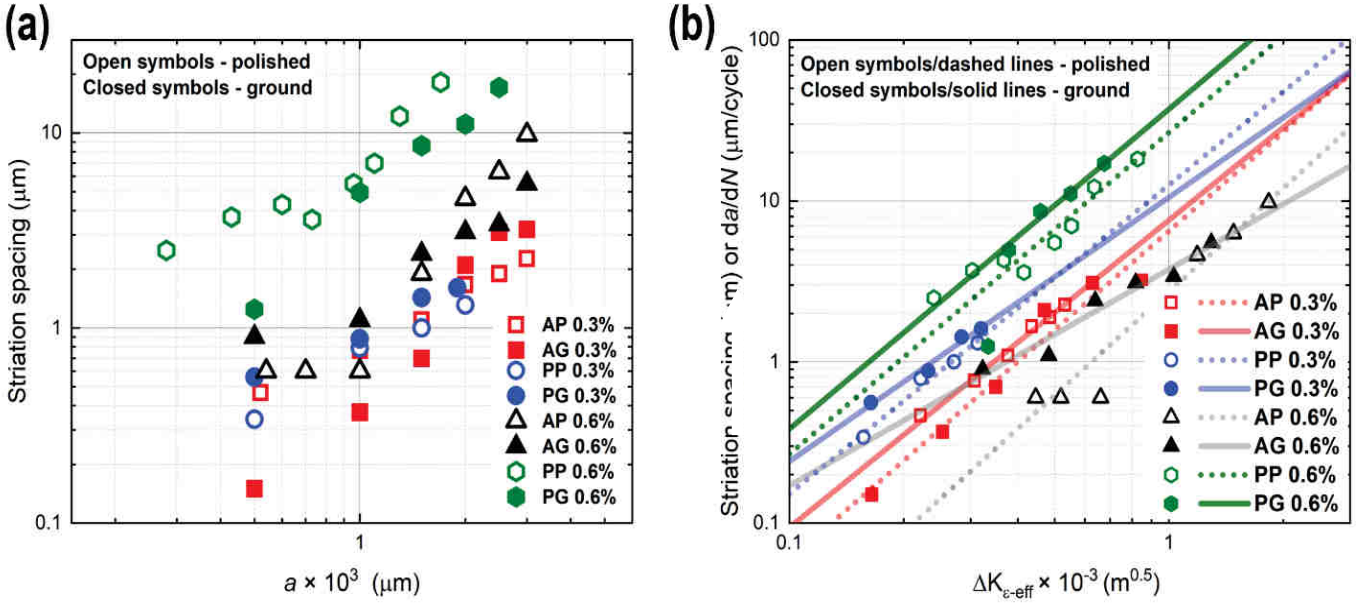
- Fatigue striations formed on polished specimens tested in (a) air and (b) PWR environment (AP 0.6 % and PP 0.6 %) at a = 500 µm





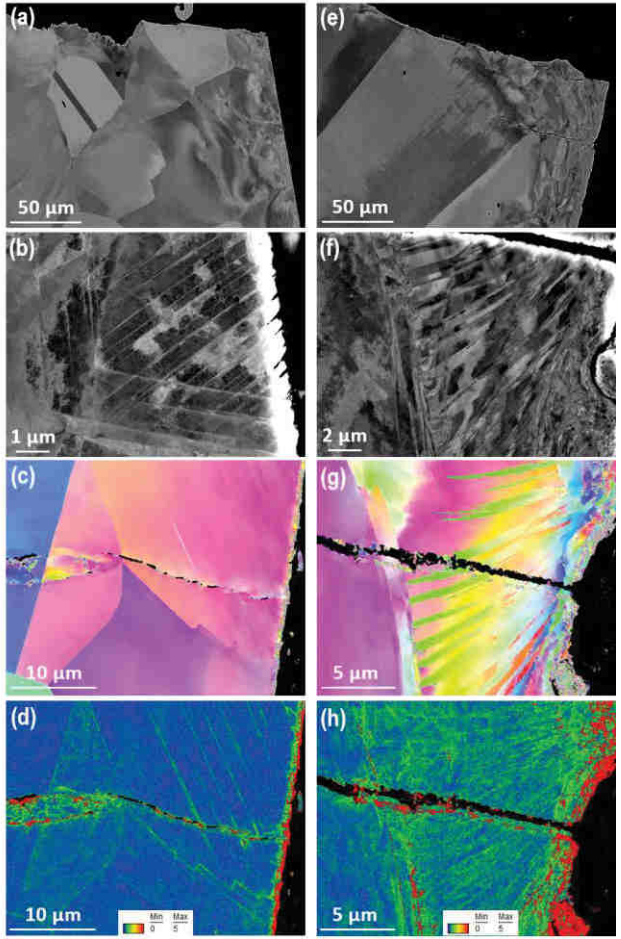
Striation spacing

- Striation spacing as a function for (a) crack depth and (b) effective strain intensity factor for short crack depths (≤ 3 mm, validity region of the stress intensity factor)
- The increased strain amplitude and the exposure to PWR environment result in a higher FCGR of 304L.
- No effect of surface finish on striation spacing, since surface finish mainly affects crack initiation and MSC formation.



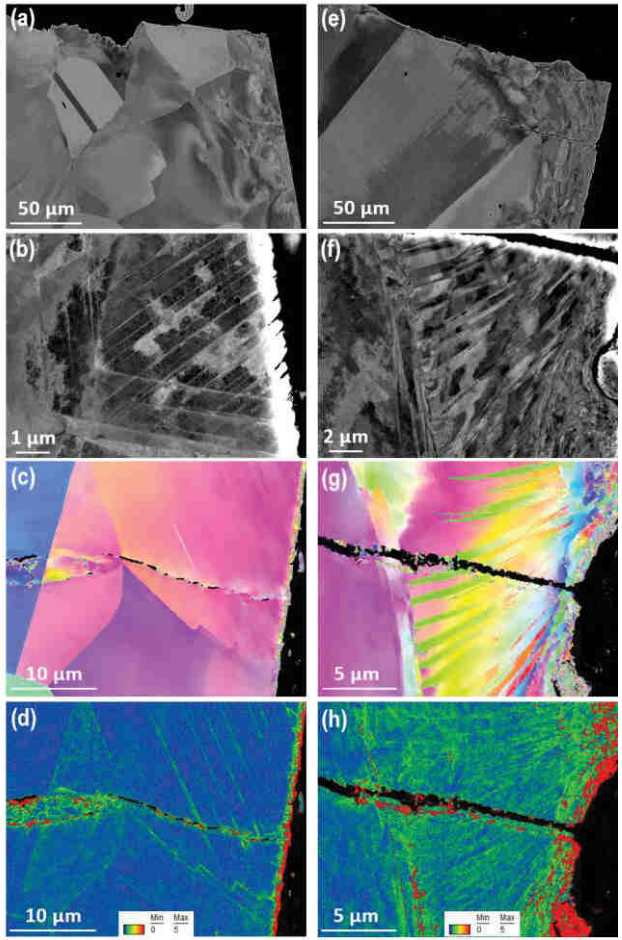
Near-surface observations

- The polished surface finish resulted in an ultrafine-grained layer and thin deformation layer extending around 1 μm into the specimen. Nanometric, dense, transgranular cracks, which are parallel to the deformation induced twins, were observed at the sub-surface.
- Grinding operations resulted in an outer ultrafine-grain layer with a deformation layer extending 10–20 μm into the bulk and a set of deformation bands.
- The grinding process resulted in a higher average KAM value and higher surface roughness.

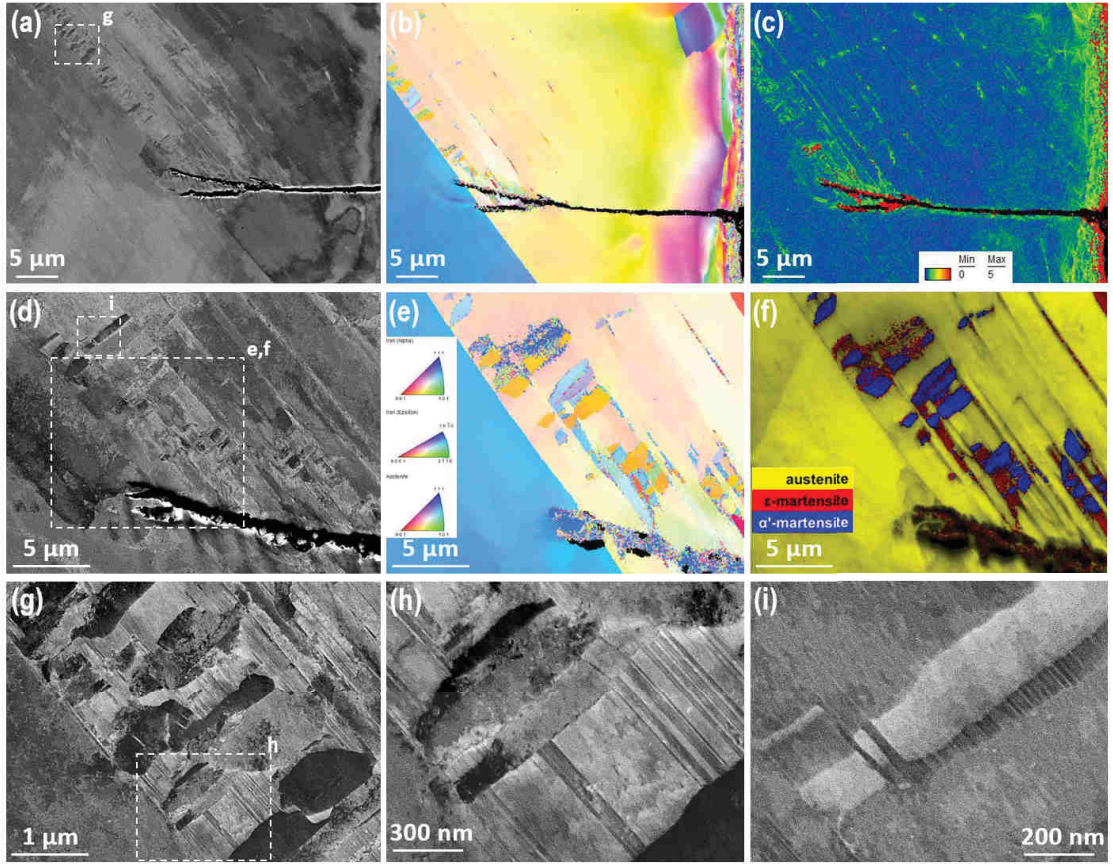


Near-surface observations

- In a polished specimen, the UFG layer formed below the specimen surface promotes faster oxidation in a PWR water, leads to passivation/crack arrest and increases the cracking initiation resistance, which hinders the initiation and short crack growth of EAF cracks.
- Grinding introduces a deformed layer with a steep microstructural gradient in the material and the localisation of plastic deformation and higher ratio of HAGB and more extensive slip bands beneath the surface. Cracking takes place preferentially in high-energy structural defects, like HAGBs and deformation bands with a high-density of dislocations. Dissolution along the slip bands can also promote micro-crack initiation through a crevice corrosion effect.



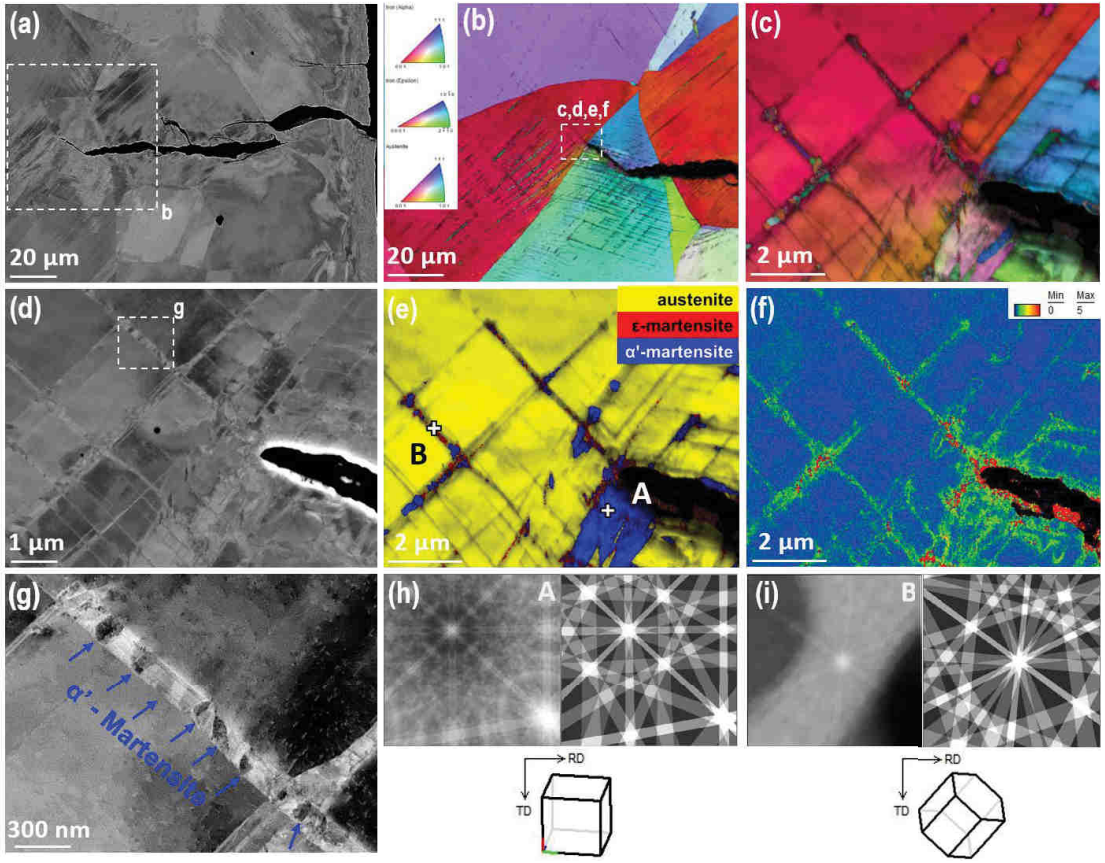
Crack tip investigations



- Deformation bands formed parallel to the intersecting GB during cycling.
- The nucleation of α'-martensite occurs perpendicular to the ε-martensite bands.
- ε-martensite bands are forming deformation channels
- The cyclic plastic strain is localised in the volumes between the ladder-like α'-martensite islands

(PG 0.3 %)

Crack tip investigations

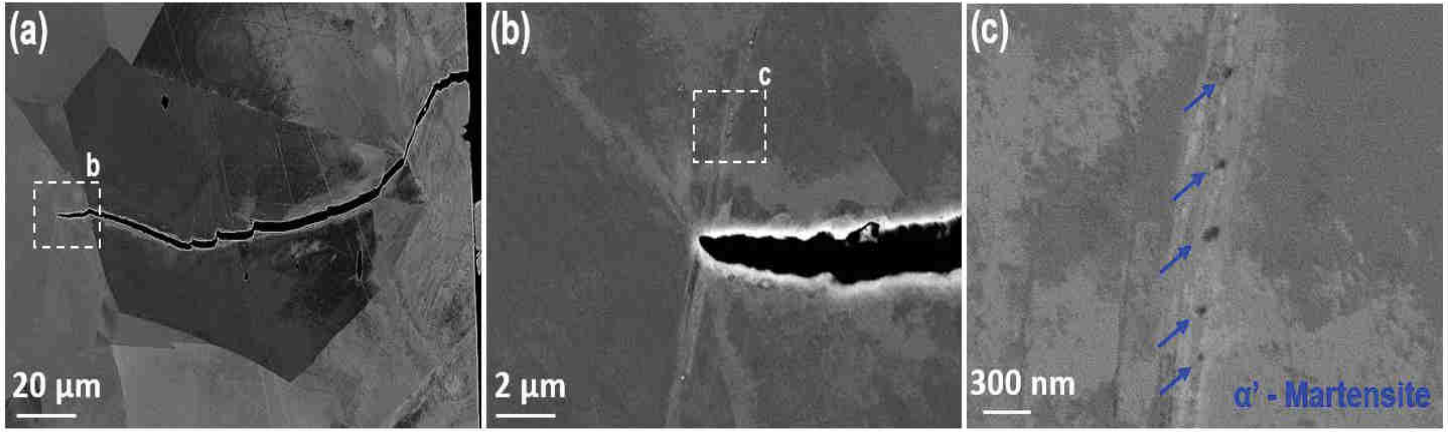


- Deformation band boundaries were characterised by a parallel set of nano twins.
- The hcp ε-M phase is observed inside the deformation band and α′-M nucleation occurs at hcp ε-martensite band intersections

(PG 0.3 %)

Crack tip investigations

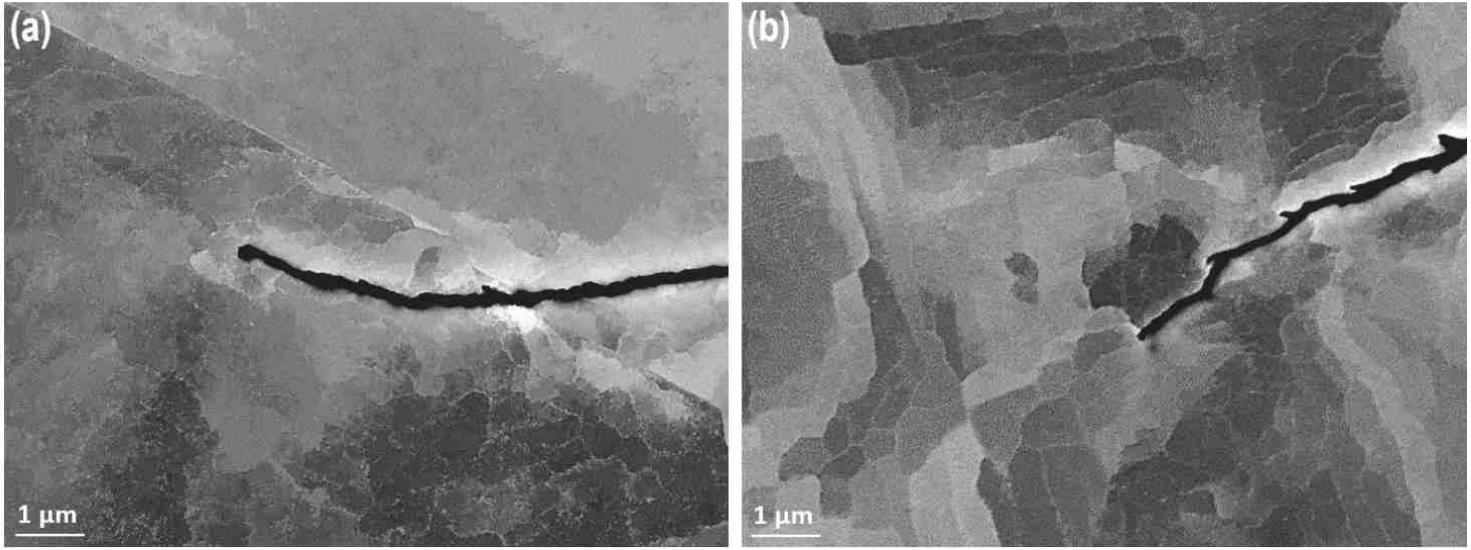
- The amount of secondary fatigue cracks on the surface of specimens tested in PWR environment is higher than that tested in HTA. A very limited amount of phase transformation in the vicinity of the fatigue crack tip was observed for specimens tested in HTA at εa of 0.3 % or 0.6 %, i.e. a very small amount of martensite was observed (only in one of the ~40 investigated crack tips), unlike frequently observed for specimens tested in a simulated PWR environment (martensite was observed in one third of ~35 investigated crack tips).



(AP 0.3 %)

Crack tip investigations

- The crack tip in the majority of HTA specimens is characterised by mixed planar dislocation structures with poorly-defined wavy boundaries at low strain amplitude (εa = 0.3 %), while more homogeneous dislocation cell/wall structures are formed at increased strain amplitude (εa = 0.6 %) The transition from mixed wavy/ planar dislocation structures to dislocation cells occurs as a result of the increasing plastic deformation and cyclic loading.

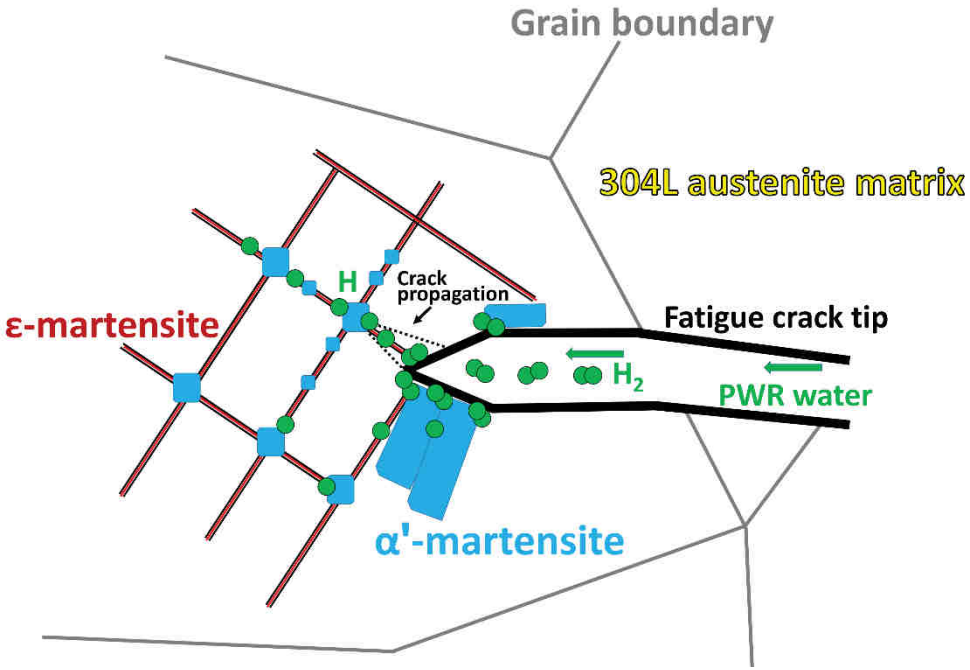


AG 0.3 % and AG 0.6 %

Interaction of martensite with hydrogen

- The fatigue life reduction by the exposure to a simulated PWR is in part caused by the formation of martensite phases in the vicinity of the fatigue crack tip, and thus the possible effect of hydrogen should be discussed.
- The fatigue cracking is enhanced by the deformation inconsistence between austenite and α’-martensite during cyclic loading.
- In addition to being at the root of martensite transformation at the crack tip, the possible accumulation of hydrogen at the transformed martensite/austenite interfaces may lead to an increased FCGR in the material.
- Local strain-induced martensitic transformation in the vicinity of crack tips provides a potential hydrogen diffusion passage. Due to the high solubility and low hydrogen diffusivity in austenite matrix and the comparatively low hydrogen solubility and high diffusivity in α'-martensite, the hydrogen can accumulate at the phase boundary or interface between the austenite and α'-martensite.
- Consequently, the crack may continue to propagate along the α'-martensite/austenite interface, resulting from the weak interatomic bonding subject to exterior stress in the presence of hydrogen and the activation of hydrogen induced slip deformation.

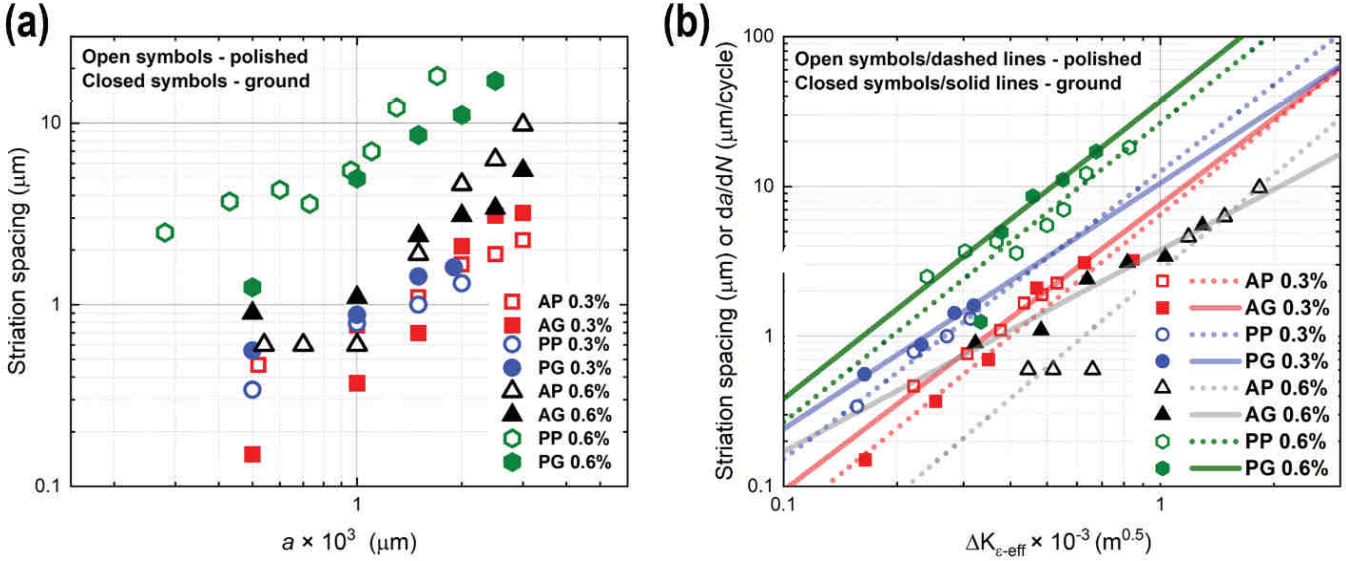
Interaction of martensite with hydrogen



α'-martensite initiates at the intersections of ε-martensite. After the dissociation of molecular hydrogen due to the corrosion reaction, atomic hydrogen will be absorbed by and diffuse in the material, where it will accumulate at the phase interfaces, e.g. martensite/austenite boundaries, and other microstructural heterogeneities. The LCF crack can continue to propagate along the martensite/austenite interface.

Additional discussion

- The PWR environment can influence both the MSCs initiation (up to about 250 μm) and mechanically small crack growth (beyond 250 μm). For an estimation of the effect of PWR environment on these two stages, the pair PP 0.3% (Nf = 6547 cycles and da/dN = 3.1 μm/cycle when $\Delta K_{\varepsilon-eff}$ is 0.5×10^{-3} $m^{0.5}$) and AP 0.3% (Nf = 25020 cycles and da/dN = 1.6 μm/cycle when $\Delta K_{\varepsilon-eff}$ is 0.5×10^{-3} $m^{0.5}$) was taken as an example. The mechanically small crack growth rate is only about a factor of 2 greater in PWR than in air. Thus, the environmental effect on MSCs needs to be >>2 as the experimental F_{en} for engineering crack initiation (approximately 3 mm) is roughly 4.



Summary

- Ground surface finish, high strain amplitude and the PWR environment lead to a decrease in fatigue lifetime, specifically with
  - the ground surface finish and simulated PWR environment enhancing fatigue crack initiation and
  - the simulated PWR environment and increased strain amplitude resulting in an increased FCGR.
- Phase transformation was observed in the vicinity of the fatigue crack tip of specimens tested in a simulated PWR primary environment.
  - Phase transformation, from γ-austenite to α'-martensite occurred via the intermediate ε-martensite phase.
  - Phase formation was rarely observed in specimens tested in HTA.
- The enhanced fatigue crack propagation rate in the hydrogen-containing simulated PWR primary environment is potentially due to the aggregated presence of α'- and ε-martensite in the vicinity of the crack tip and their susceptibility to hydrogen-assisted fatigue cracking.

Refs:

- Z. Que, C. Huotilainen, T. Seppänen, J. Lydman, U. Ehrnstén, Effect of machining on near surface microstructure and the observation of martensite at the fatigue crack tip in PWR environment of 304L stainless steel, Journal of Nuclear Materials 558 (153399) (2022).

Case III: Influence of mean stress and LWR environment on fatigue life and dislocation microstructures of SS316L

TEM for dislocation structure

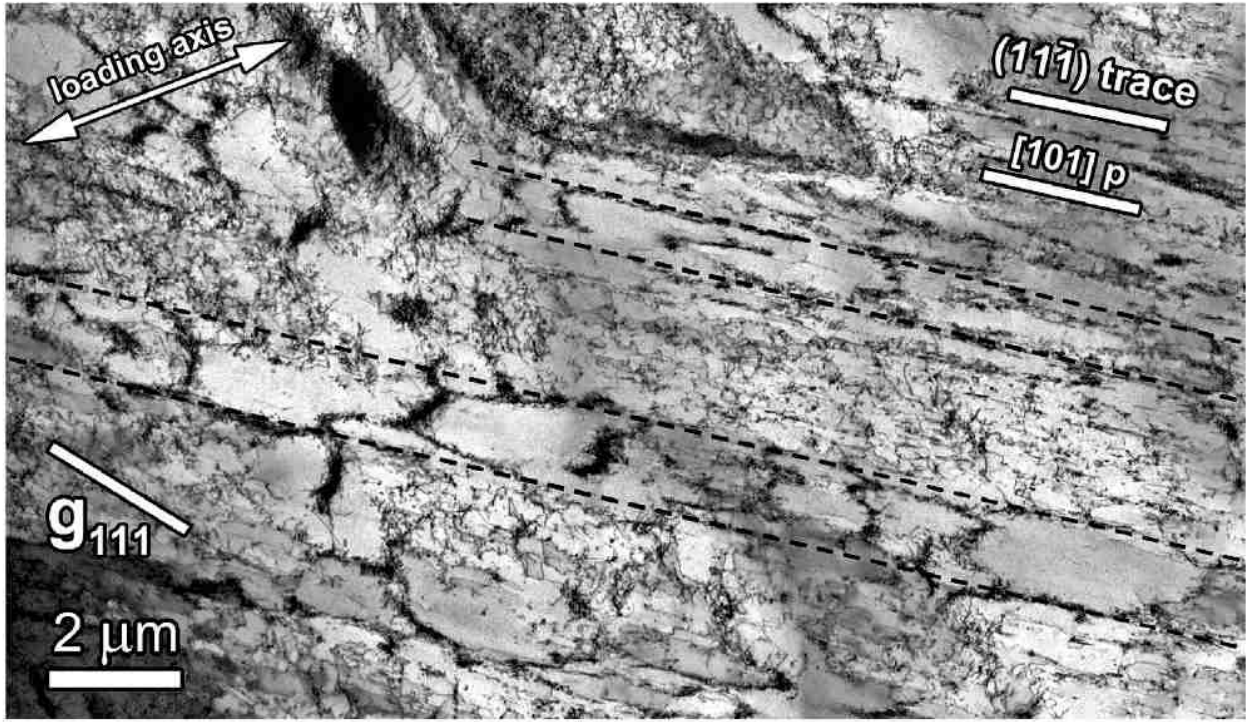


Fig. 22. Sample loaded with stress amplitude 190 MPa and no mean stress. Cyclic plastic deformation is localized in the persistent slip bands having typical ladder-like dislocation structure with alternating dislocation rich walls and dislocation poor channels (highlighted by black dashed lines). Edge dislocations forming dipole configuration in the walls have Burgers vector ½[101]. Gliding on the ($\bar{1}$11) plane, they form the slip system with the Schmid factor = 0.454. Grain was oriented close to [001] zone axis relative to loading direction facilitating activation of three slip systems.

TEM for dislocation structure

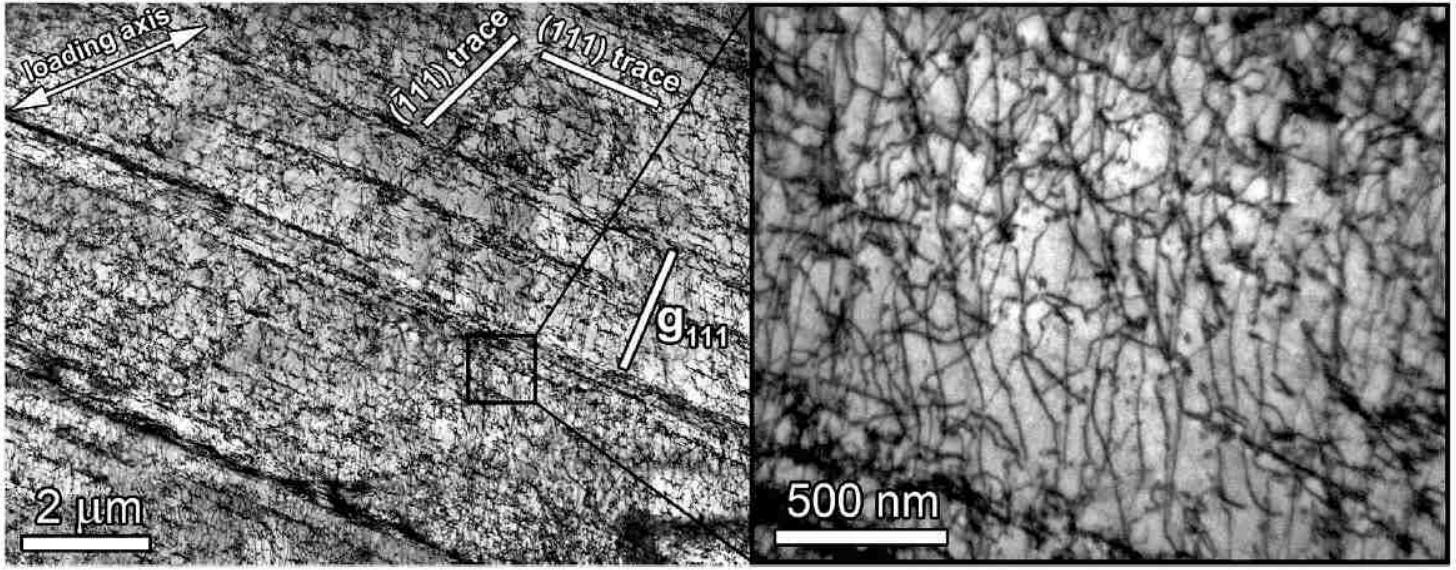


Fig. 24. Characteristic microstructure observed in sample loaded with stress amplitude 190 MPa and 50 MPa tensile stress. High density of homogeneously dispersed dislocations is observed. Dislocations from three slip systems with the highest Schmid factors were determined. Detail in black inset shows high density of dislocations with high amount of dislocation-dislocation interactions.

TEM for dislocation structure

- The TEM observations of the dislocation structures at the end of life of the tests with stress amplitude of 190 MPa and zero mean stress revealed that the microstructures are in good agreement with previous studies made on 316 L in standard conditions (strain controlled tests at room temperature in air). The plastic strain amplitude of these tests is about 0.28% in the mid-life, which is low enough to produce planar structures in some grains. However, most of the grains exhibit imperfect veins, wall/channel and persistent slip bands, which are typical features of intermediate plastic strain amplitudes (between 0.1% and 0.5%) in strain-controlled tests and are usually correlated to the localization of plastic deformation and cyclic softening and stabilized response to the external load.
- The microstructure resulting from the tests with stress amplitude of 190 MPa and + 50 MPa mean stress showed a much higher dislocation density, distributed homogeneously within the grains and with no notable signs of recovery processes. Such a microstructure is characteristic by high amount of dislocation-dislocation interactions leading to increased flow stress as described well by Taylor equation for dislocation-forest hardening. These microstructural changes and corresponding differences in cyclic plastic response of material were tentatively attributed to the large plastic strain to set the mean stress during the first cycles.