Proposal Code	**MA-3355**
Proposal Title	Analysis of VHCF damage in duplex stainless steel using microbeam Laue diffraction and a pnCCD detector

Experimental Report

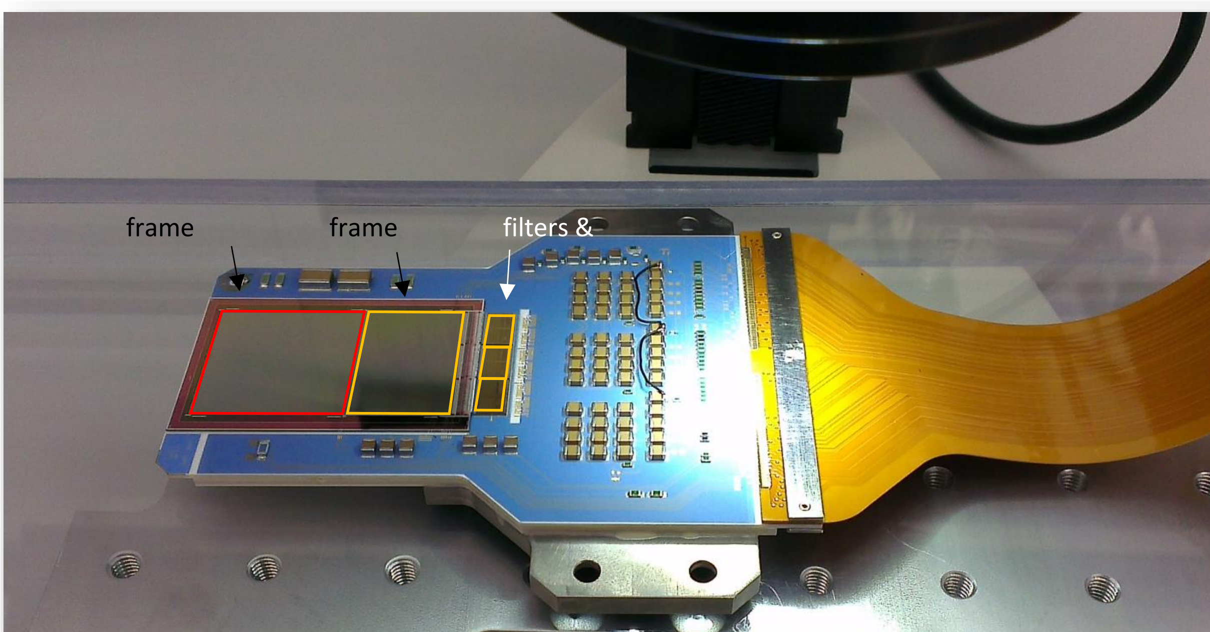
Contents:

- Motivation and Aim
- Properties of Duplex Steel
- Microbeam X-ray Laue Diffraction @the ERSF
- Results and Discussion
- Conclusion & Outlook

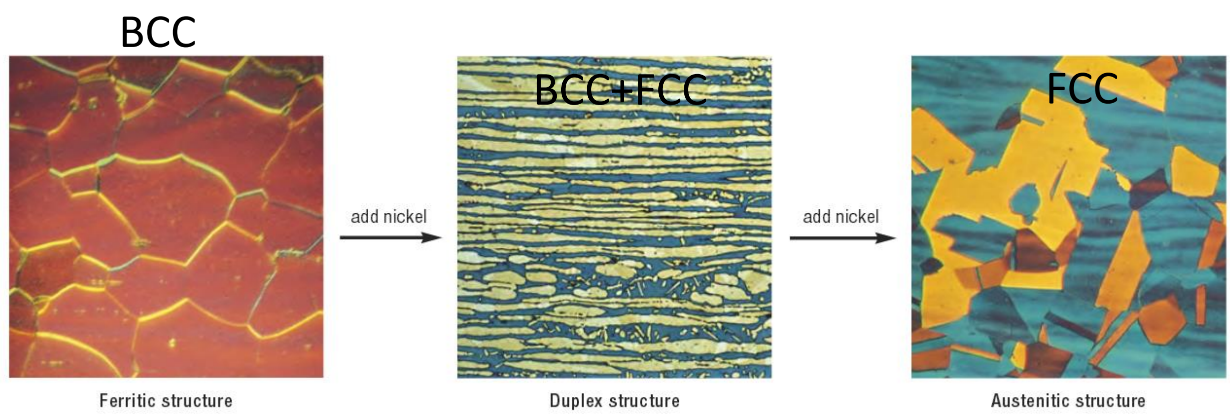
Motivation and Aim: Duplex stainless steel is of generic interest. It is used in various components and applications overs a spread of length scales ranging from meter sized structures to nano-sized structures.

One of the open question in this field is the material behavior at very high cyclic fatigue, by this I mean in the region of 10e9 cycles and above. We would like to investigate crack formation at VHCF below the stress critical point.

For this study we will use microbeam Laue X-ray diffraction analysis with a 2D energy dispersive pnCCD as our primary tool. The detector consists of 384x384 pixels each of 75um size.

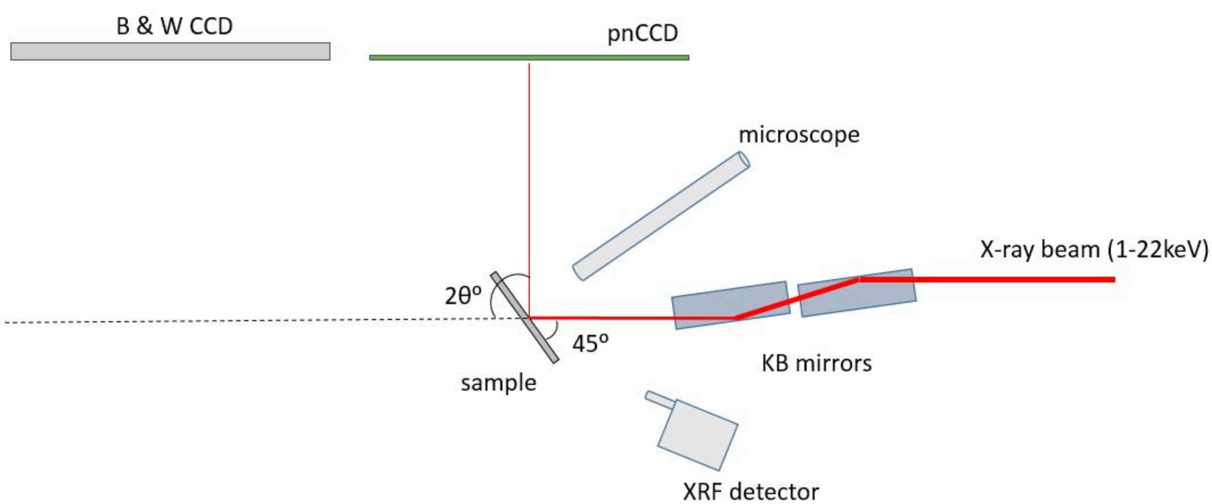


The detector has an optimal QE (~100%) between 1 and 20keV and with the possibility of subpixel resolution and the experiment angular resolution can 0.004 degrees at a sample to detector distance of 10cm

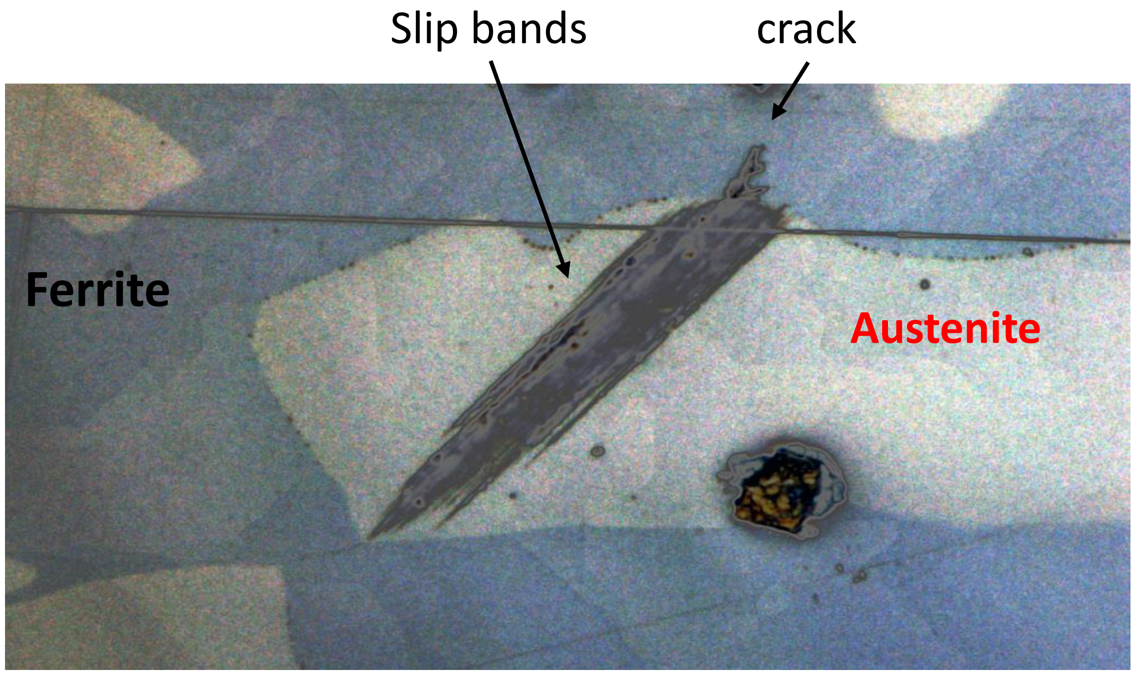


What is DSS? Its main elemental component is iron with ratios of Cr, Mo, Ni and N. The 2 phase structure is a combination of Ferrite which is BCC and Austenitic that is FCC. The two phases are distributed in a 50:50 ratios. The addition of Cr and Mo makes DSS corrosion resistive, shows high strength and ease of fabrication, and chloride stress corrosion and cracking resistance making it suitable to many applications such as:

<u>BM32 @ ESRF</u>

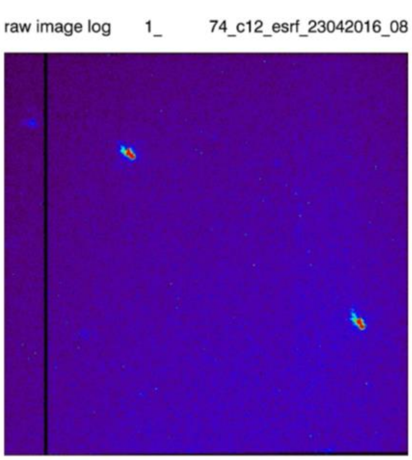


The experiment was carried out at the microbeam Laue diffraction setup beam line (BM32) at the ESRF. The hutch is specifically equipped with x-ray focusing optics that produces x-ray beams with a size of 0.5x0.5µm. The x-ray photon energies range between 1-22keV. The Setup is equipped with a B &W CCD for fast raster scans and was used to calibrate the detector distance. A 1D XRF detector was used to draw a map of the region of interest of the sample based on the different composition of Ferrite and Austenite grains. The sample mounted at 45 degrees w.r.t the incident beam and can be translated in the x,y and z direction. The pnccd detector was mounted at a 90 degrees to the incident x-ray in a reflection geometry.

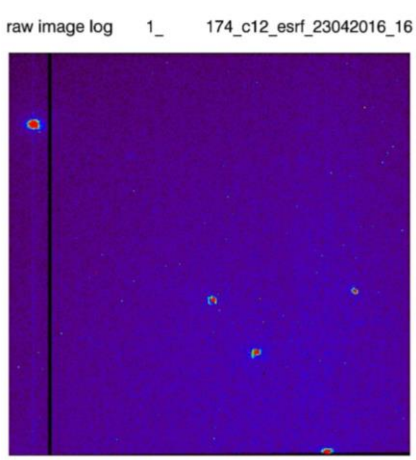


Measurement Plan: The measurement plan consists of identifying a region of interest on the sample and to perform Laue x-ray diffraction. The optical microscope images taken in Siegen shows an Austenite grain surrounded by Ferrite phase. Within the Austenite slip bands are visible that extend to one side initiating a crack in the ferrite phase. Because the optical microscope at the beamline could not resolve such a reasonable resolution image we had to use the XRF map to determine the location of the beam on the sample. And as a result we reproduced the surface of the sample with the signal from the point detector. The measurement plan consists of scanning the sample and recording data in the vicinity of the SBs and the crack.

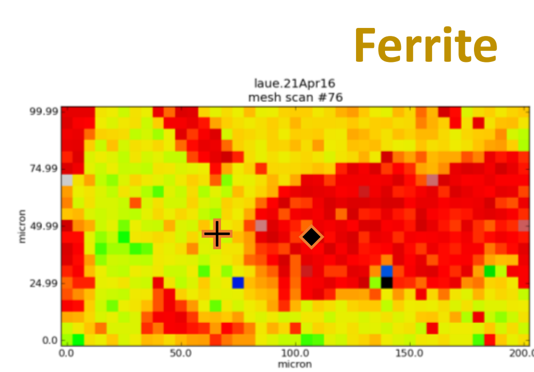
A first attempt to characterize the Austenite and Ferrite phases would be to record Laue patterns at the interesting points on the sample. In these two Laue patterns I show the signal from the ferrite (taken in the vicinity of the cross signal) and that of the Austenite. Each of such Laue patterns contains the (energy of the Laue spot and the Bragg angle) and hence with a few permutations the hkl indices can be determined.



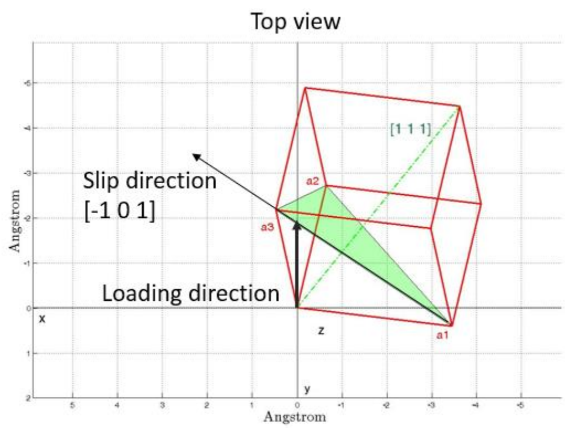
Ferrite signal



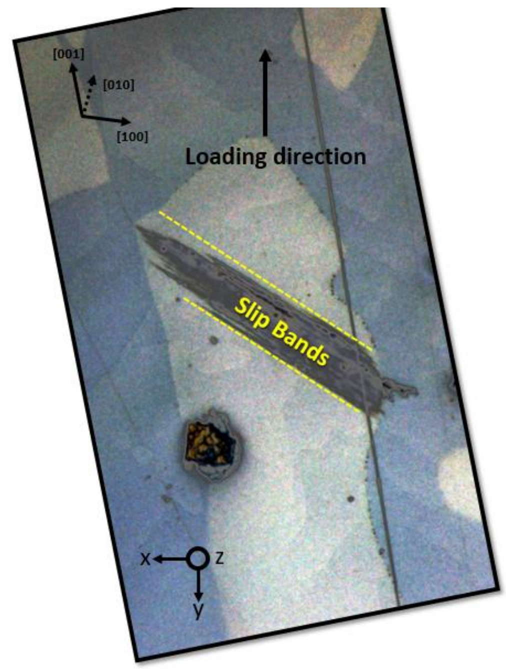
Austenite signal



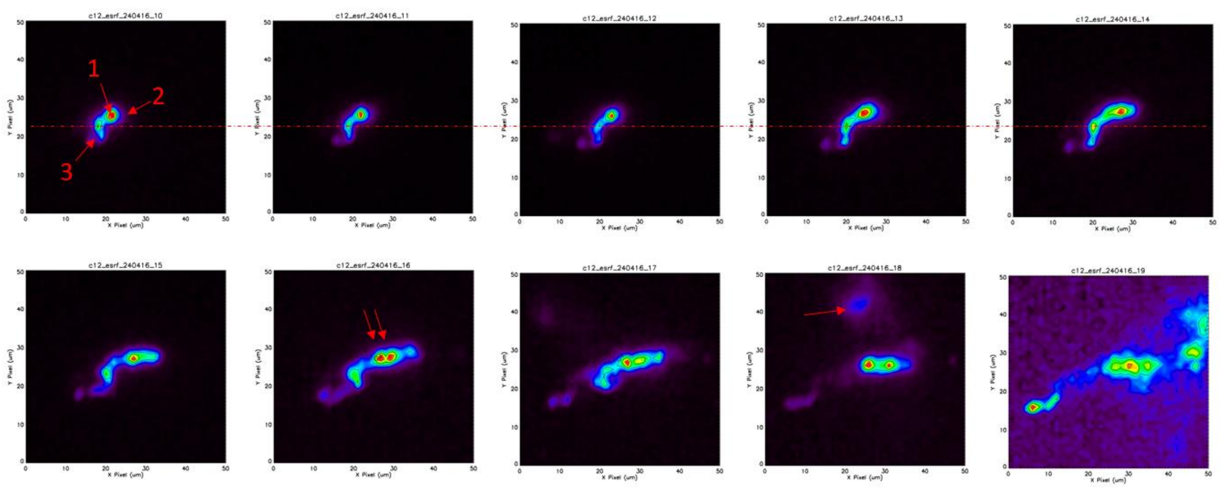
Using at least two Laue reflections, with their energy and position known, the crystal orientation with respect to a predefined lab system can be obtained. In this example, the crystal orientation of the austenite grain was determined. The red cube shows the lattice unit cell as supposed to be oriented w.r.t. to the lab system. Within we can see the most dominant slip system the 111 plane with different slip directions. The Grain orientation agrees well with the optical microscope image. Indeed, the slip bands are aligned at approx. 45 degrees with respect to the loading direction. Such a system is the one with highest probability to be active in case of an external load. The x-ray data goes well with the predictions.



Grain orientation agrees well with the optical microscope images and crystallographic features such as slip bands direction.
The Schmid factor =0.436, the highest Schmid factor slip system for this crystal orientation.



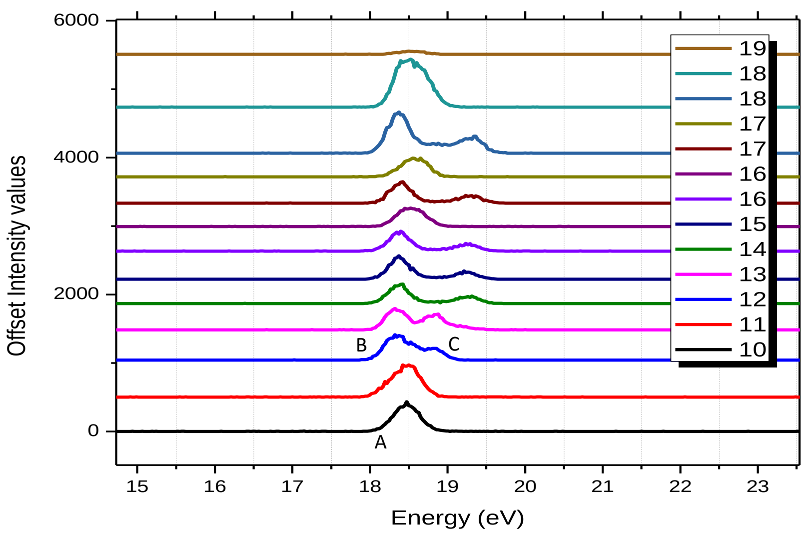
Scanning the crack: We take a closer look by plotting the variations of the Laue spot along the crack next to each other.



Example of intensity distributions for a selected Ferrite peak are provided as a function of scanning position.

The closer we get to the grain boundary the more changes are visible to the intensity maps.

- Starting from a relatively far position w.r.t to the GB (20um, crack is 6um in length) the intensity maps shows a confined high intensity peak with a tail of less intensity. The sharp peak originates from a region in the ferrite grain with non or at least very few dislocations. the blurred peak a region of increased dislocation density.
- Moving closer to the crack tip, the blurred region around the sharp peak increases while the sharp peak position changes indicating lattice rotation. At approx. the tip of the crack, the sharp peak splits into two distinguishable peaks that migrate away from each other further down the crack. This indicates the formation of sub grains within the ferrite grain which are separated by regions of dislocation boundaries.
- The appearing and disappearing of the particular features in the vicinity of the Laue spots provide insight into the dynamics of dislocations movements. As we approach the base of the crack the number of sub grains increase surrounded by large number of dislocation broadening the envelopes and lowering the relative intensity of the Laue spot.



The energy spectra of the Laue spot complete the image and complement those of the intensity maps.

The change in the energy of the Laue spot help explain the way the crack propagates.

The image to the left shows the energy spectra of the most pronounced peak taken from an area of 4x4 pixels. The overlap in the numbers show the positions where the Laue spot splits. X-ray diffraction without the precise knowledge of the energy of the reflection is blind to changes in the volume.

If we trace the energy spectrum of the most prominent peak we see that it has a single energy peak, for positions closer to the crack tip we notice the following:

The peak splits into two parts in energy, this is a signature of a change in the lattice spacing (volume of the unit cell is changing) with a tilt in the Bragg angle. At one stage the prominent Laue peak dissolves in a cloud indicating a high dislocation density region.

If we want to translate this into the real space image. The once perfect ferrite grains undergo a change of lattice parameter before splitting into two separate grains takes place. The process is accompanied by the formation of perfect sub grains and an increase in the dislocation density.

Conclusion: The experiment performed sheds light on the process that accompanies crack propagation

- The formed sub grains are dislocation free. They form islands in high dislocation regions.
- The density of dislocation increases towards the crack tip.
- The sub-grain formation is proceeded by change in the volume of the unit cells.