Dislocation patterns in Cu-OFE by EBSD and their relation with BD

Advanced studies

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(CERN & University of Vigo)

Outline

Introduction	Context		
	Aim of the study		
Protocol	Sampling	Heat treatment	
		Tensile test	
		Fatigue Test	
	Characterization method	EBSD (KAM)	
Results	Tensile test		
	Fatigue tested	0.2 µm stepsize	Reference
			Fatigue 83% LT
			Fatigue 100% LT
			Summary of results
		15 µm stepsize	Reference
			Fatigue 65% LT
			Summary of results
Perspective			
Conclusions			

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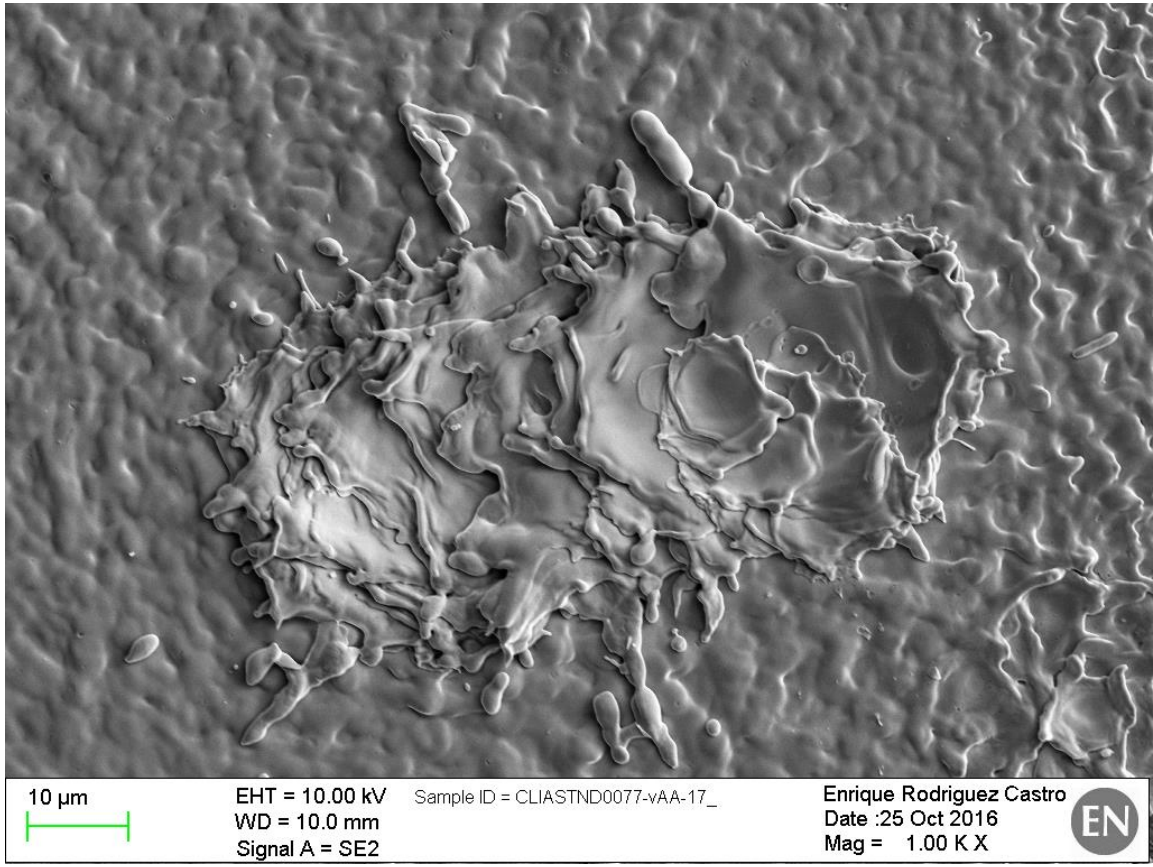
Context:

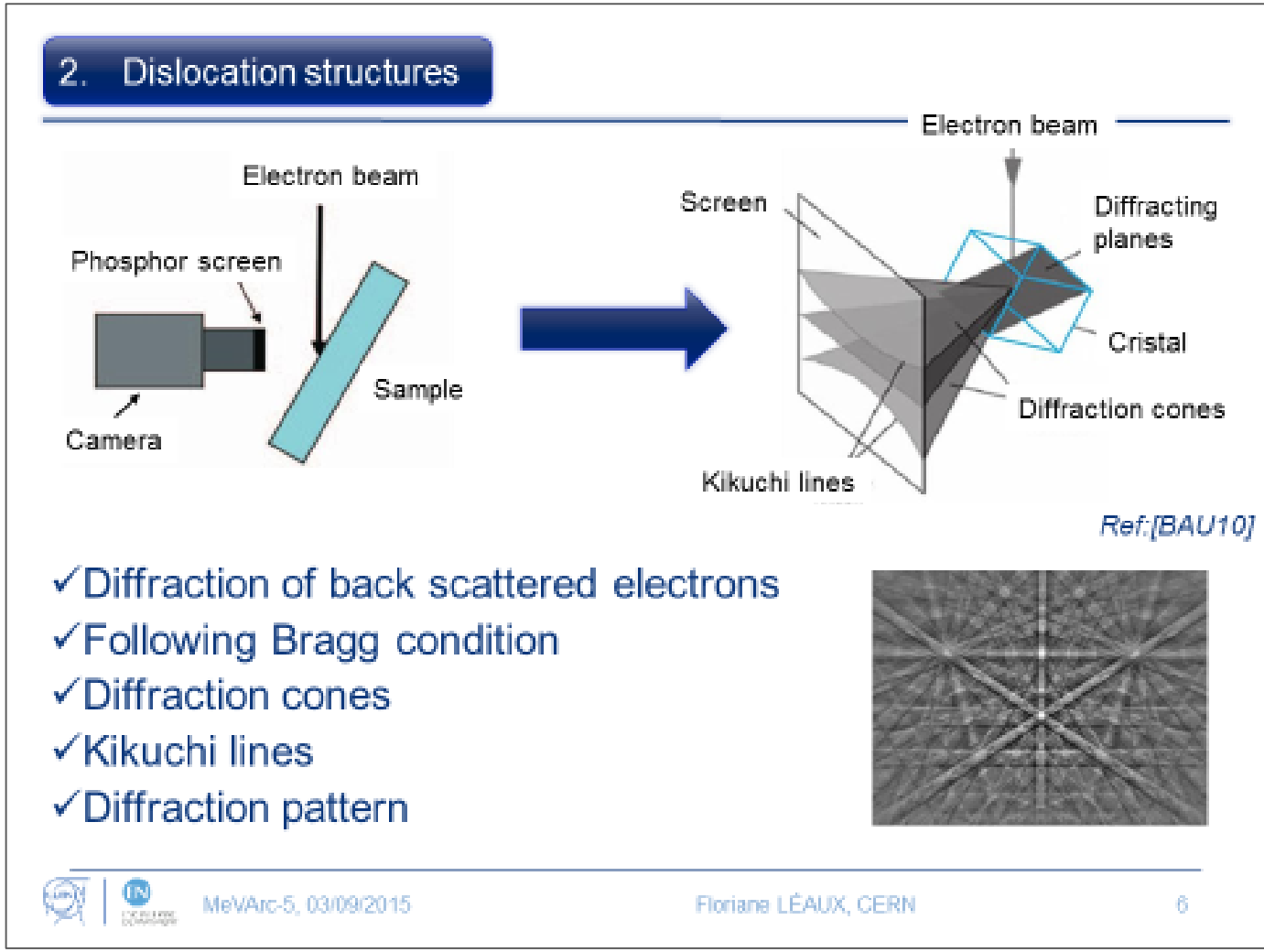
- CLIC have especial interested in understanding BD phenomena
- BDs develop in isolated or clusters of craters in the surface of CLIC-AS
- Dislocations seem to play an important role in the BD phenomena
- Find a non-destructive diagnostic technique which determines dislocation presence, density and patterns.



Aim of the study

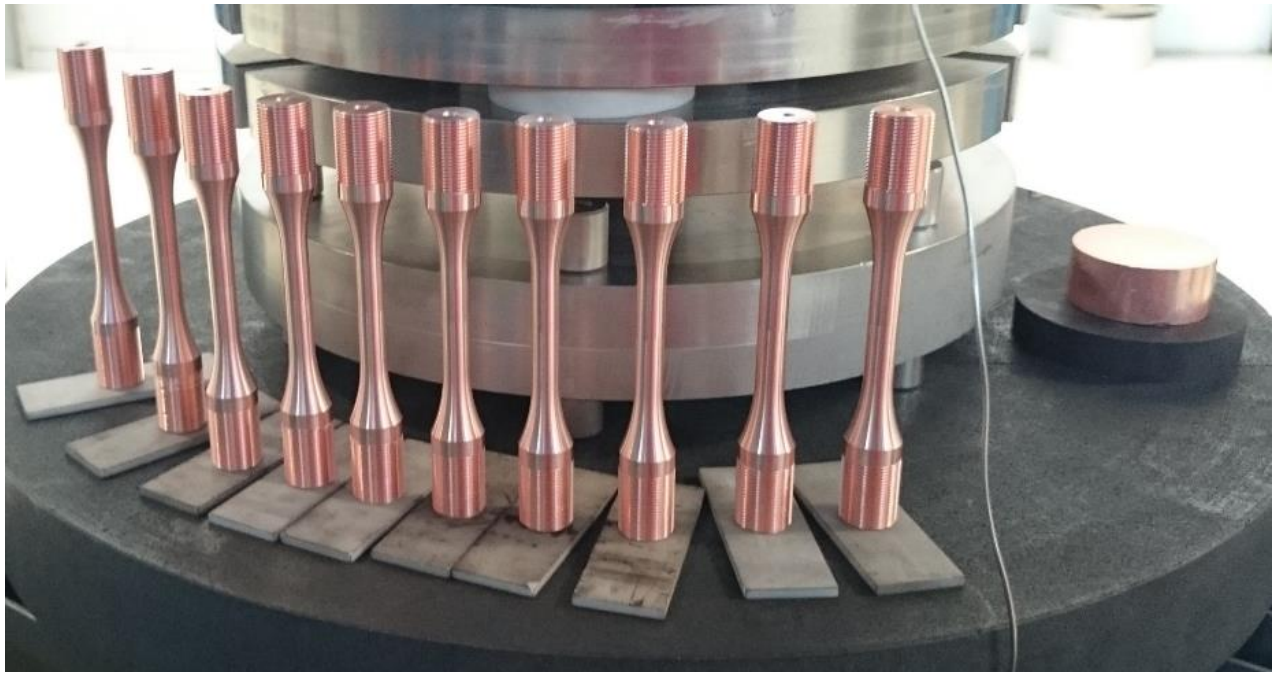
- Two objectives:

1. Establishing EBSD as a valid diagnostic ***non destructive*** technique which determines the presence of dislocations and/or density in Cu-OFE used in CLIC-AS
2. Study dislocations role in the locations of FE/BD sites



Aim of the study:

- To study the relation
  - Sample free of dislocation, or as few as possible
  - Sample with dislocations
- To have sample “free of dislocation” → heat treatment
- To introduce dislocation
  - Tensile test
  - Fatigue test
- To study and quantify the dislocations
  - Non destructive way
  - Large area
  - EBSD



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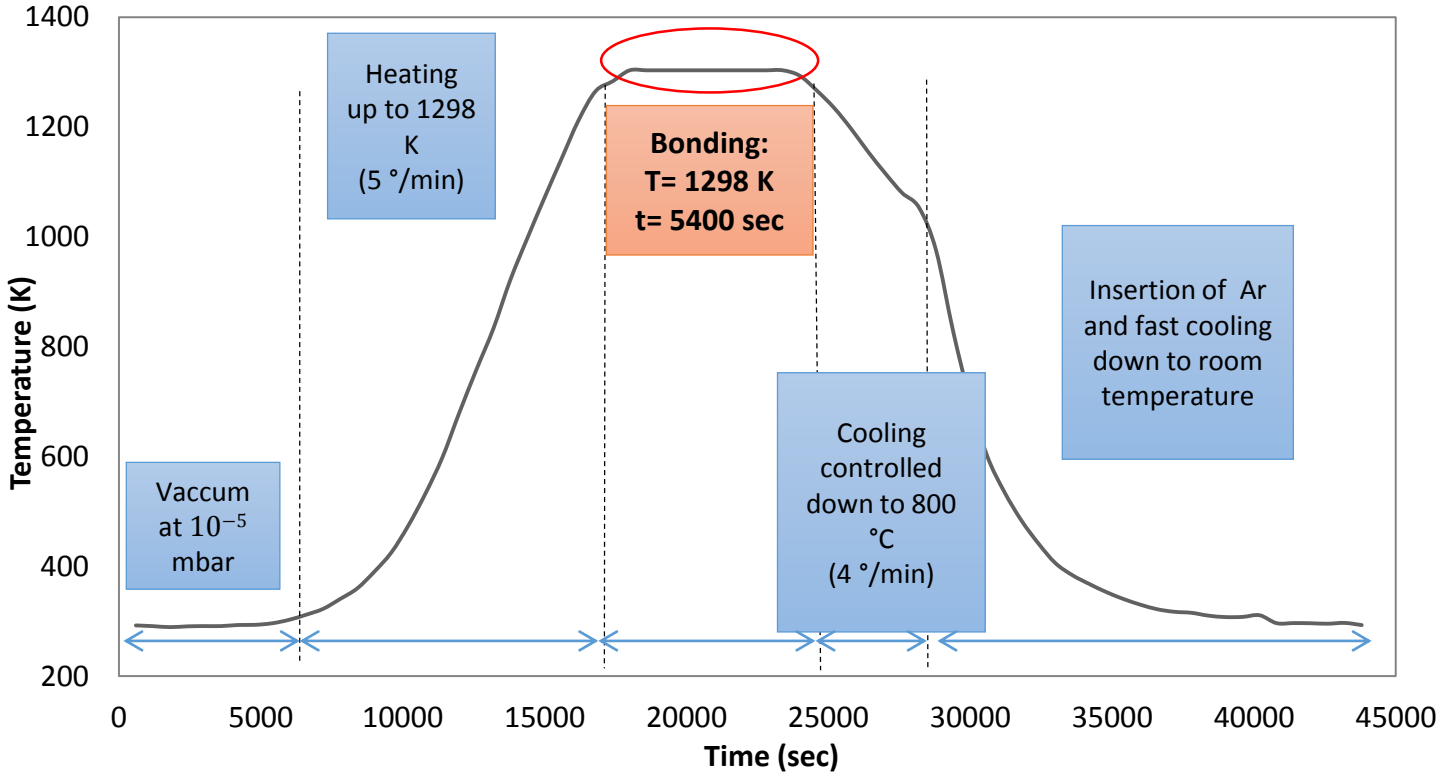
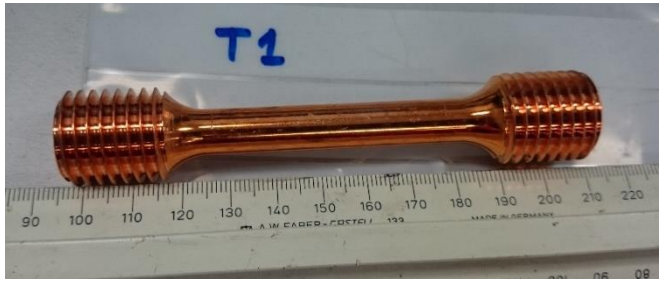
History of samples and sample preparation

- Tensile and fatigued specimens
  - Same Cu-OFE CLIC-AS
  - Heat treated:
    - Same heating cycle as real structures
    - No H_2
    - But Ar for cooling



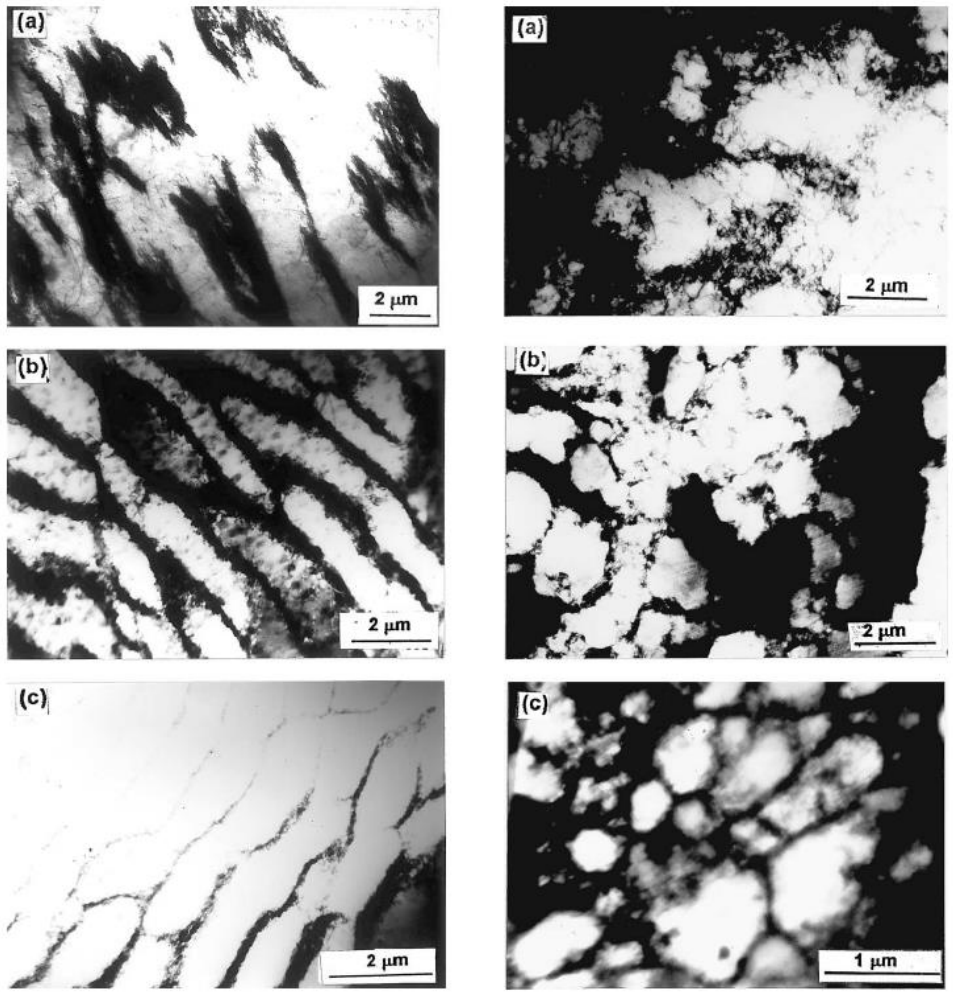


Fatigue and Tensile tests

- Fatigue
  - Pros:
    - Creation of dislocation structure (ladder type)
  - Cons:
    - Not possible to do it in house at CERN
    - Difficulties to determine the stress and number of cycles (stress control mode)

- Tensile
  - Pros:
    - Easy repeatability
    - Equipment in house
    - Creation of dislocation structures (cell type)
  - Cons:
    - EBSD not successful… yet

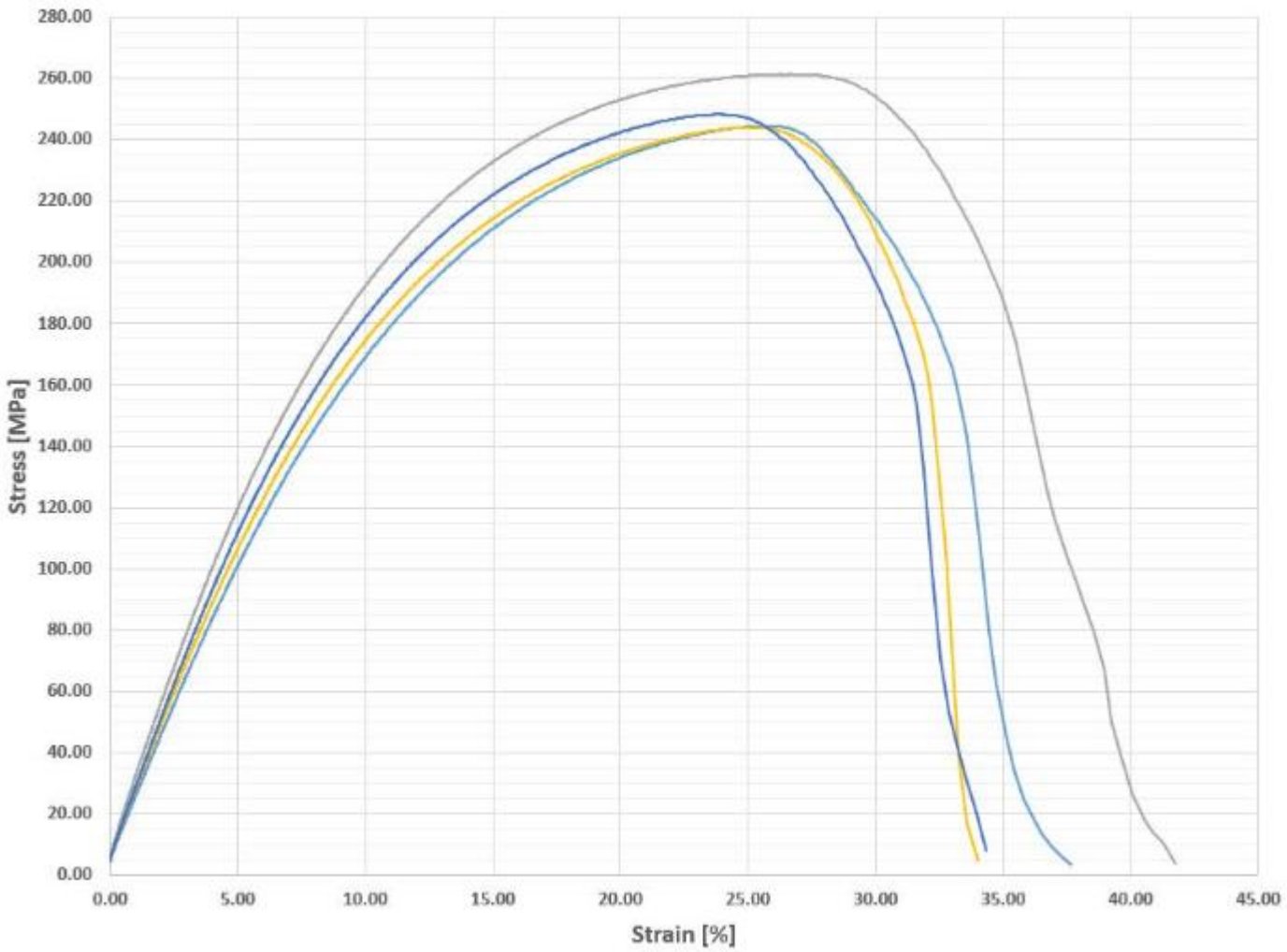
Fatigue | Tensile



TEM image of Cu samples (Mechanical behavior and the evolution of the dislocation structure of copper polycrystal deformed under fatigue-tension and tension fatigue sequential strain paths – W.P. Jia, J.V. Fernandes)

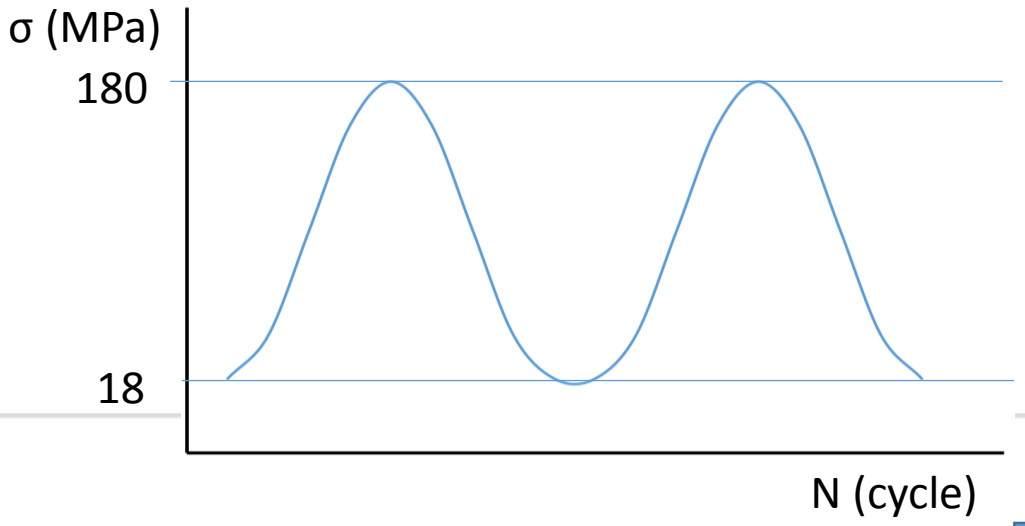
Tensile test

- Fracture
- 35% strain
- 30% strain
- 20% strain
- 0% → reference

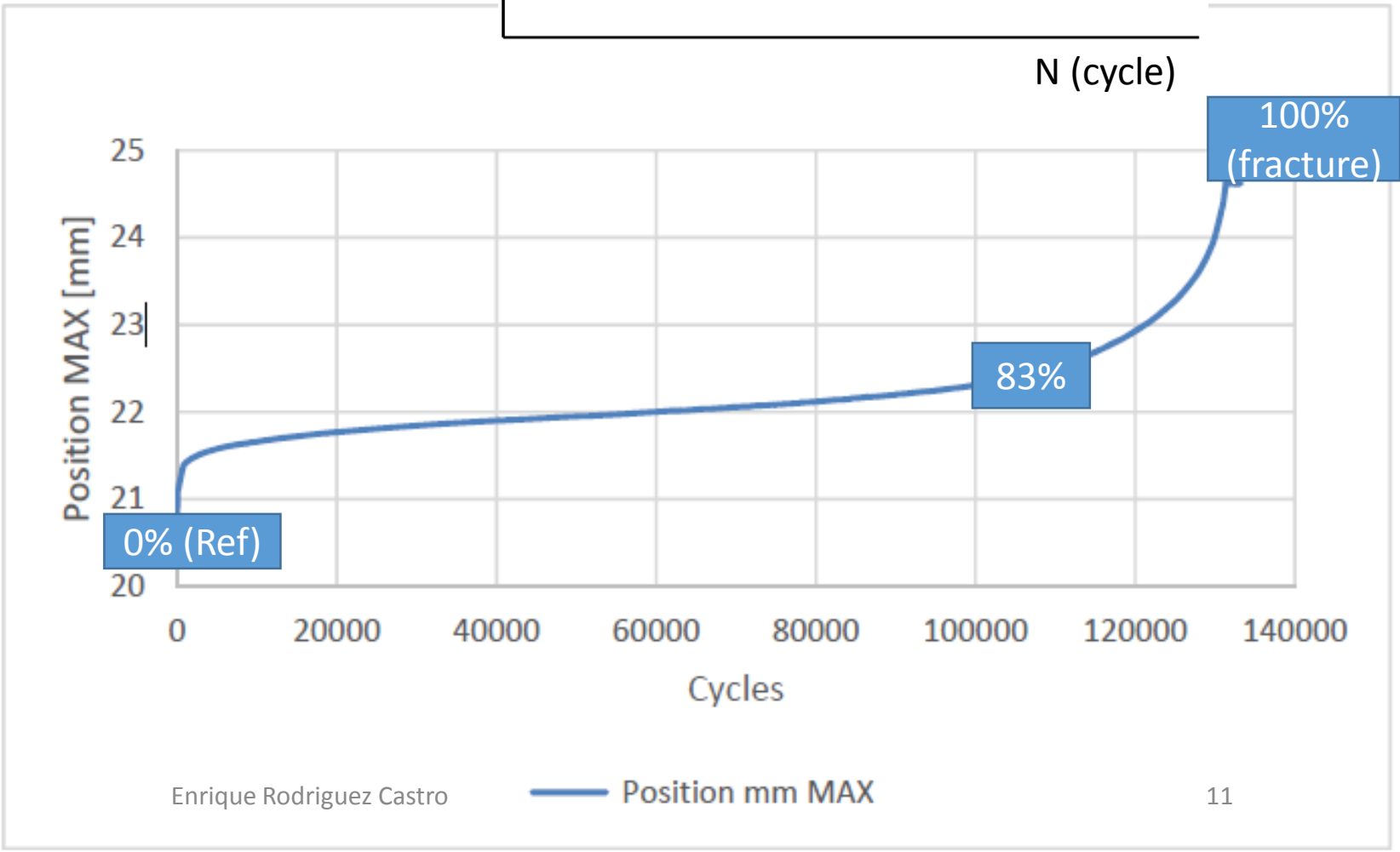


Fatigue test

- R=0.1
- σ_{Max}=180 MPa

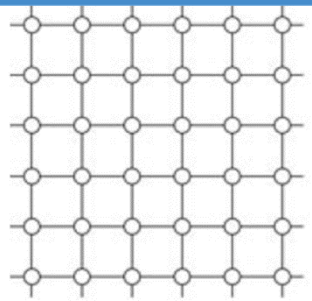


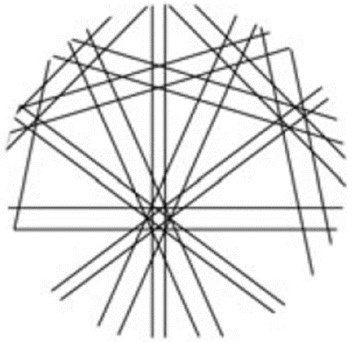
- Fracture: 100% of life time = 131700 cycles
- 83% of life time = 109700 cycles
- Reference: 0% of life time = 0 cycles



Mis-orientation and dislocation

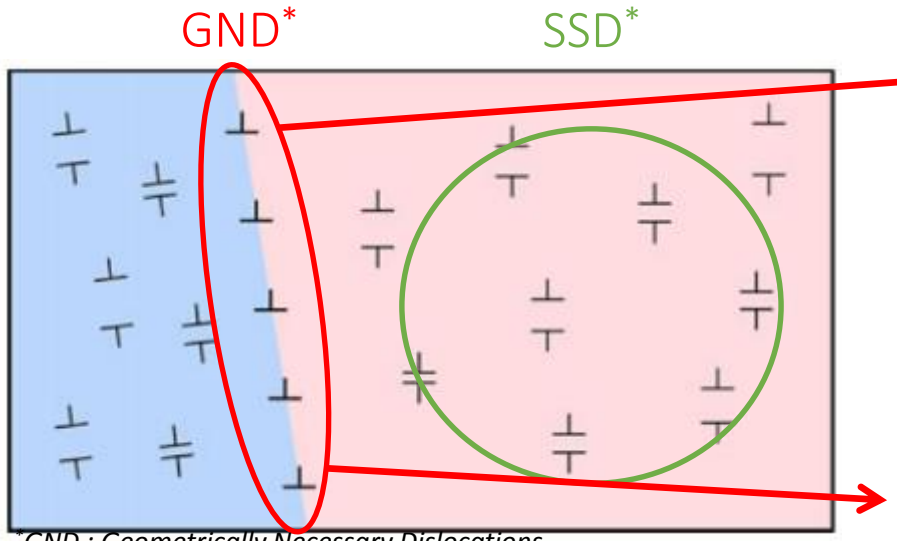
Perfect crystal



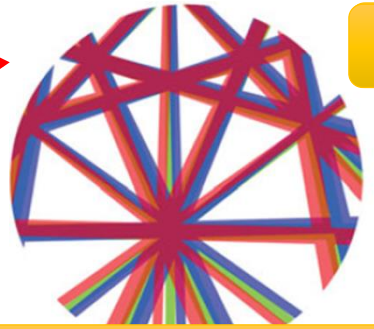


Theoretical diffraction pattern

KAM = 0



*GND : Geometrically Necessary Dislocations
*SSD: Statistically Stored Dislocations



Local misorientation

➔ Rotation of the pattern

LAGB : Low Angle Grain Boundaries

KAM : Kernel Average Misorientation

KAM > 0

Due to GND

EBSD study - KAM

- Kernel Average Mis-orientation
  - Can be used as a measure of local grain mis-orientation
  - Generally, higher in grains with higher dislocation density
- Compare every point with neighbor points
  - Assigns a value
  - 3x3 size
  - Comparison:
    - Mis-orientation maps (colour scale)
    - Histogram
- Two different step size
  - 0.2 µm
  - 15 µm

ω_{i8}	ω_{i1}	ω_{i2}
ω_{i7}	i	ω_{i3}
ω_{i6}	ω_{i5}	ω_{i4}

$$KAM_i = \frac{1}{K}\sum_{j=1}^{K} \Delta\Theta_{ij}, \Delta\Theta_{ij} < \Delta\Theta_{lim}$$

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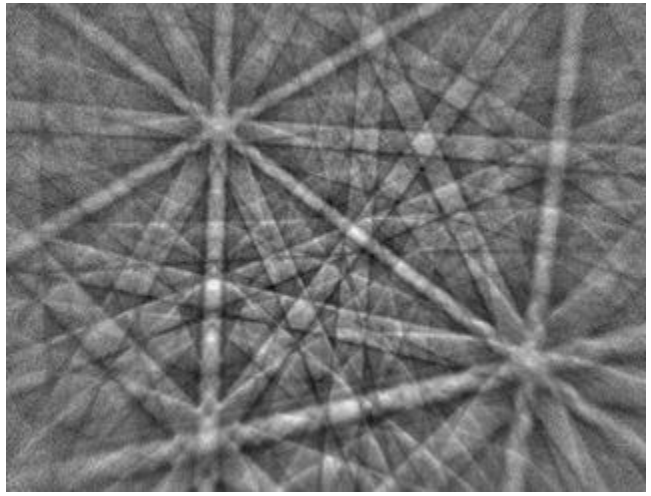
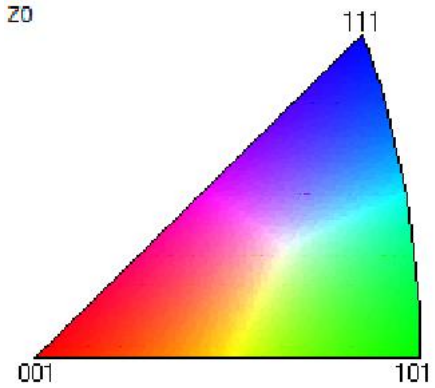
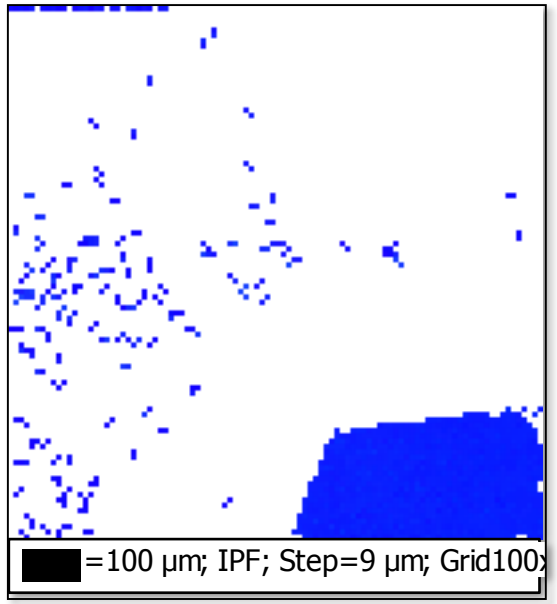
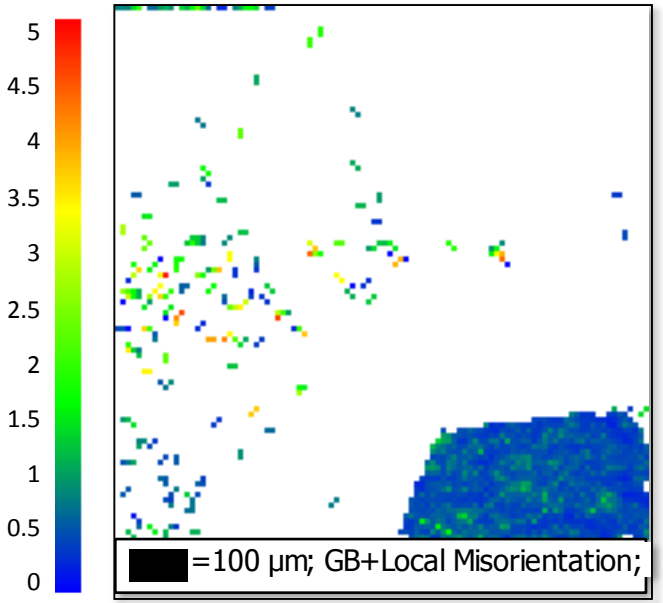
Tensile

- It was found that tensile sample are not suitable for our analysis
  - High strain → deformation of Kikuchi bands → difficult to index



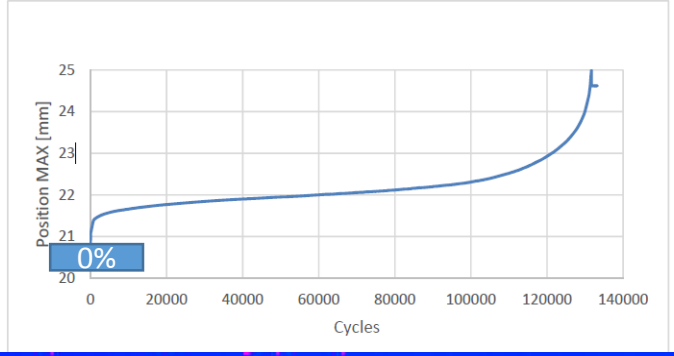


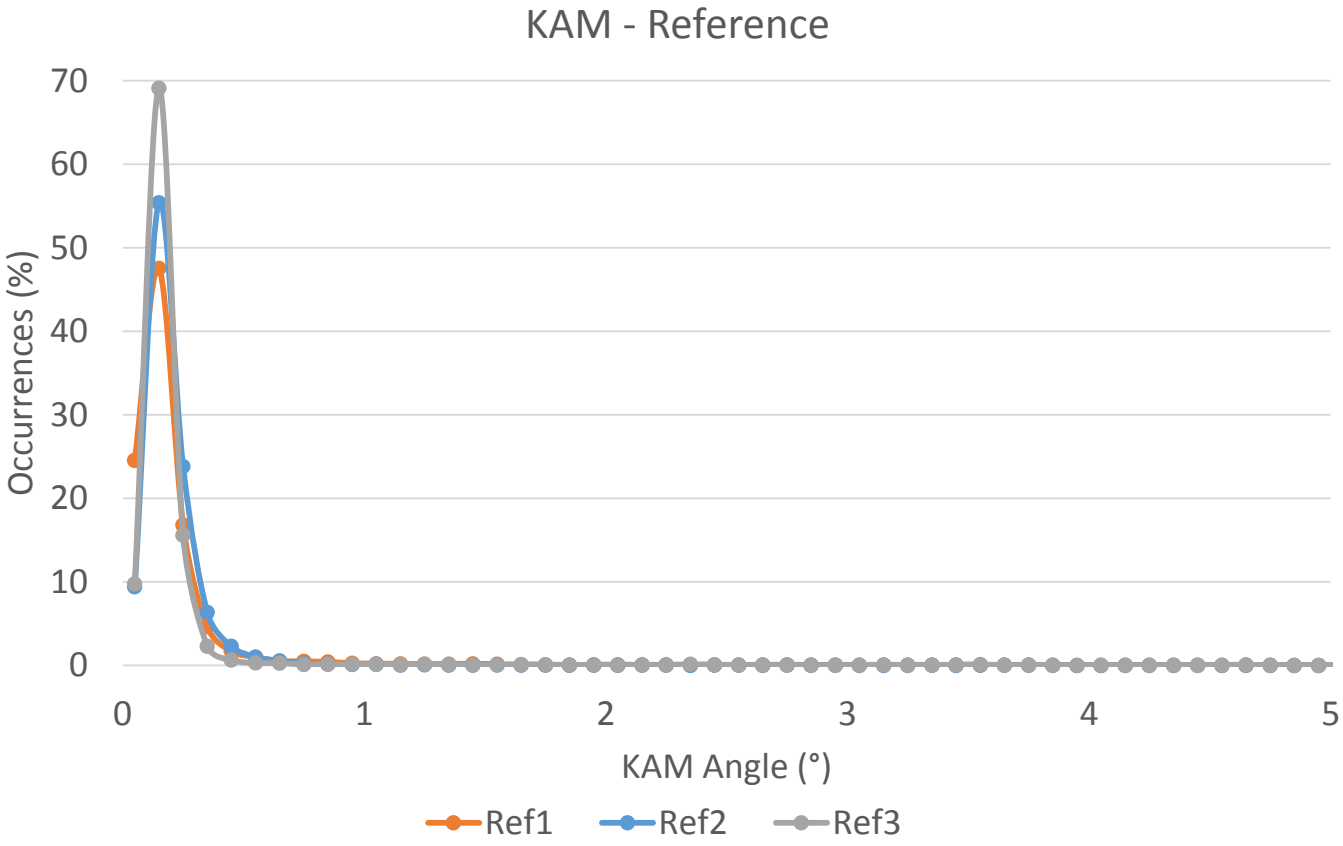


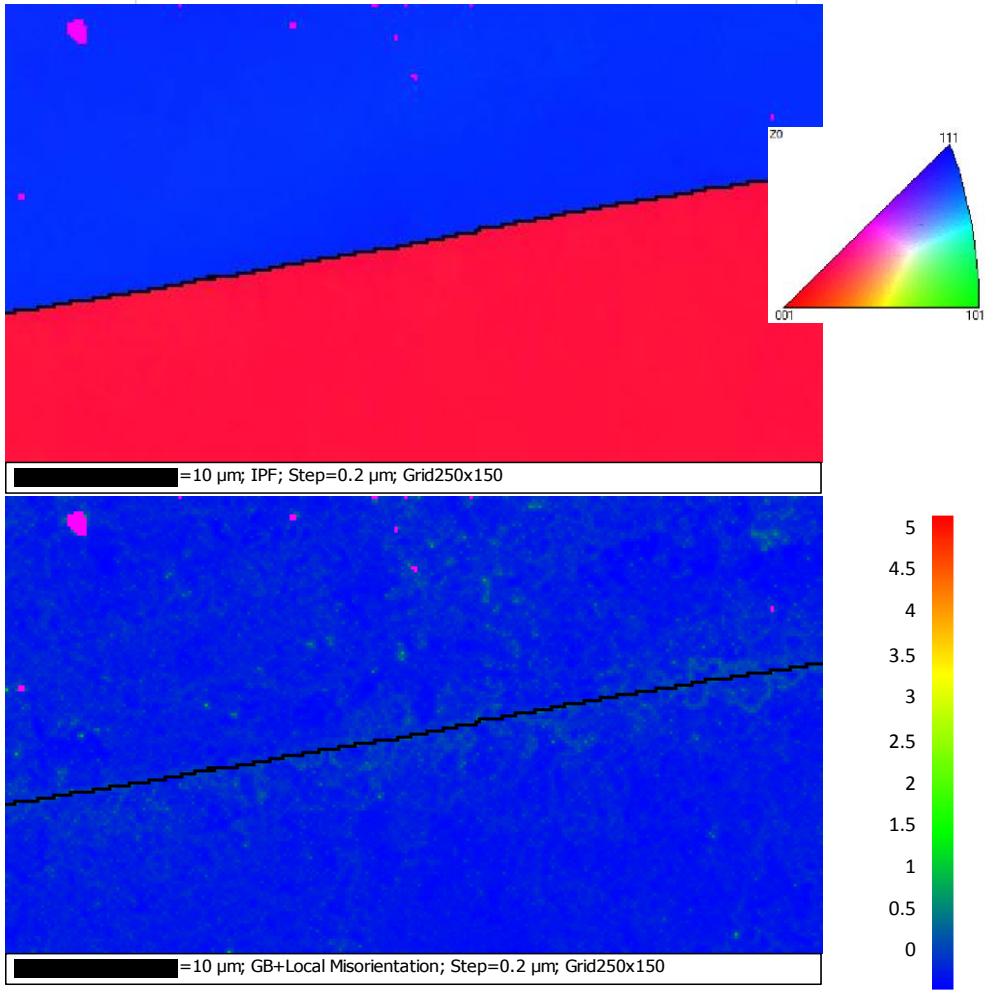


Fatigue - Reference (0% LT)

- Narrow distribution
- Low KAM values

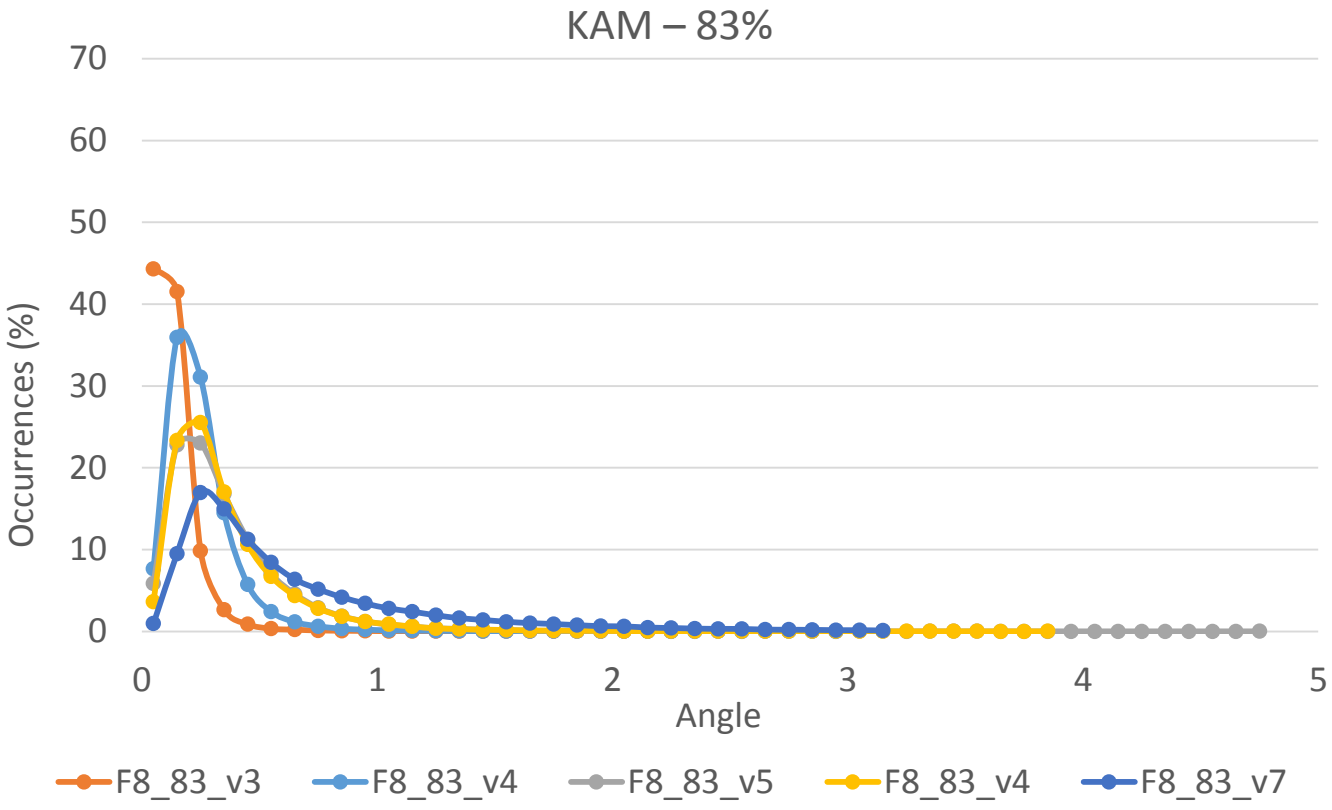


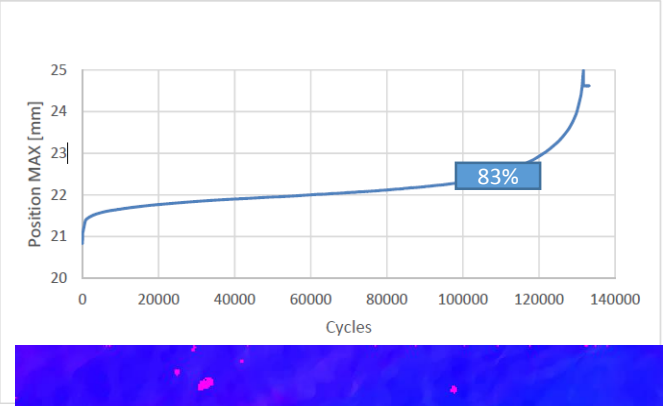


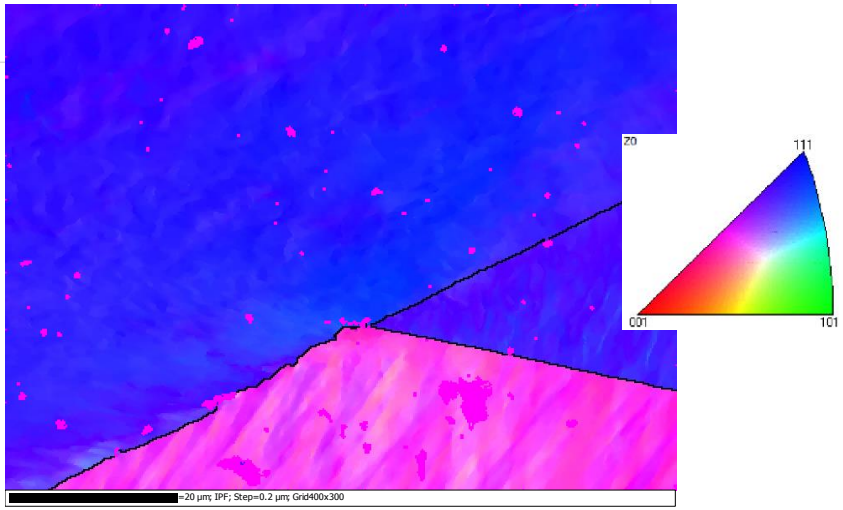


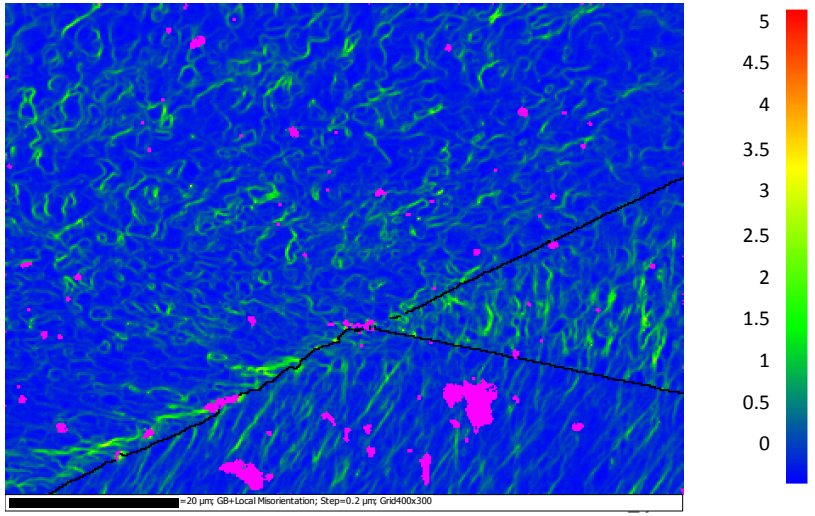
Fatigued (83% LT)

- Broadening of the distribution
- Shift to higher value
- Higher dispersion









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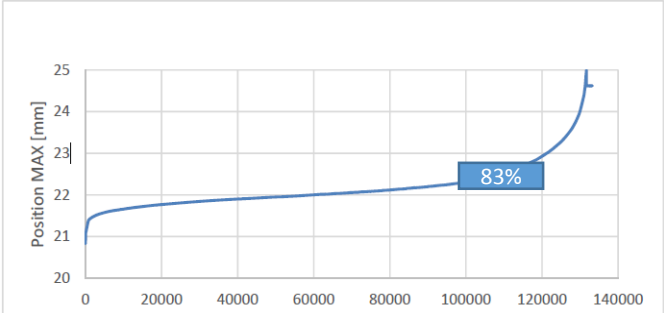
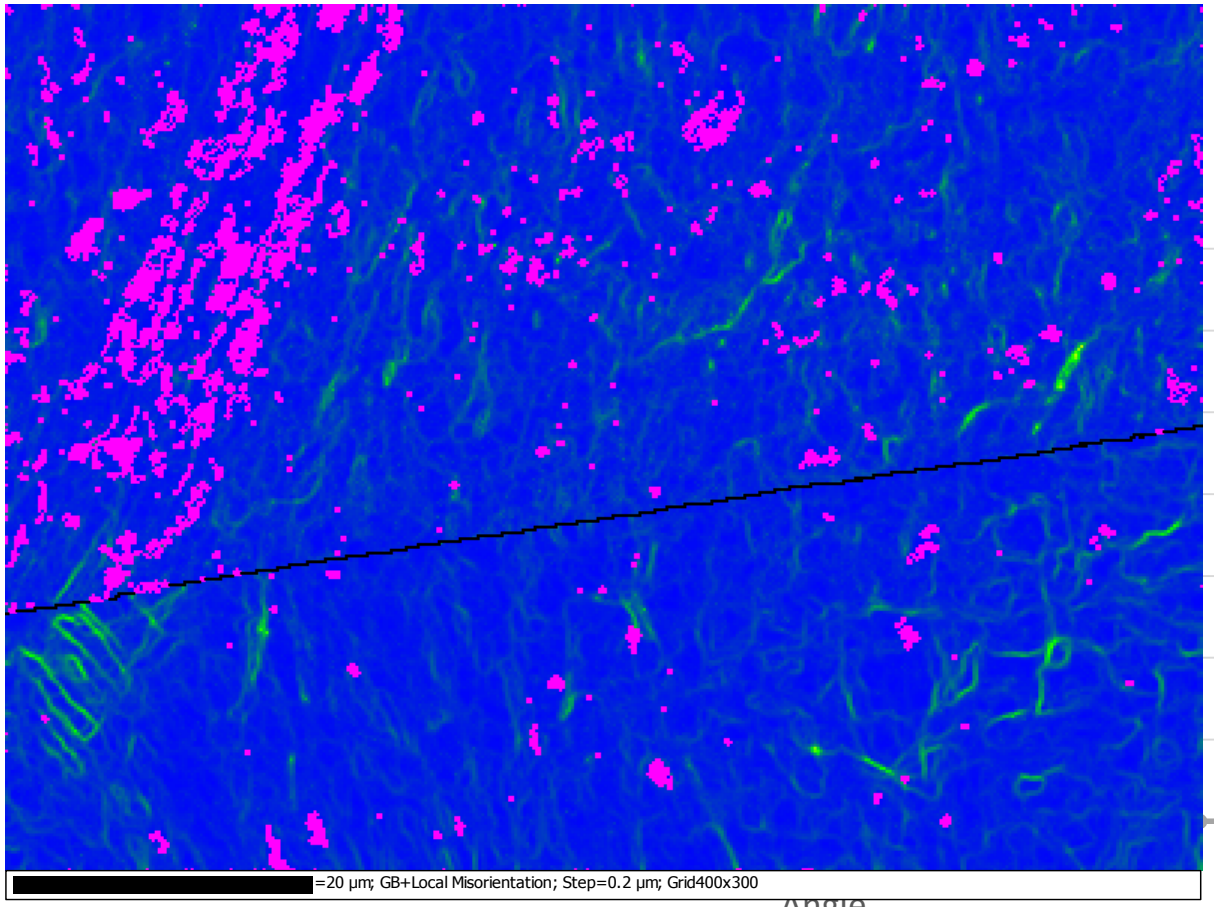
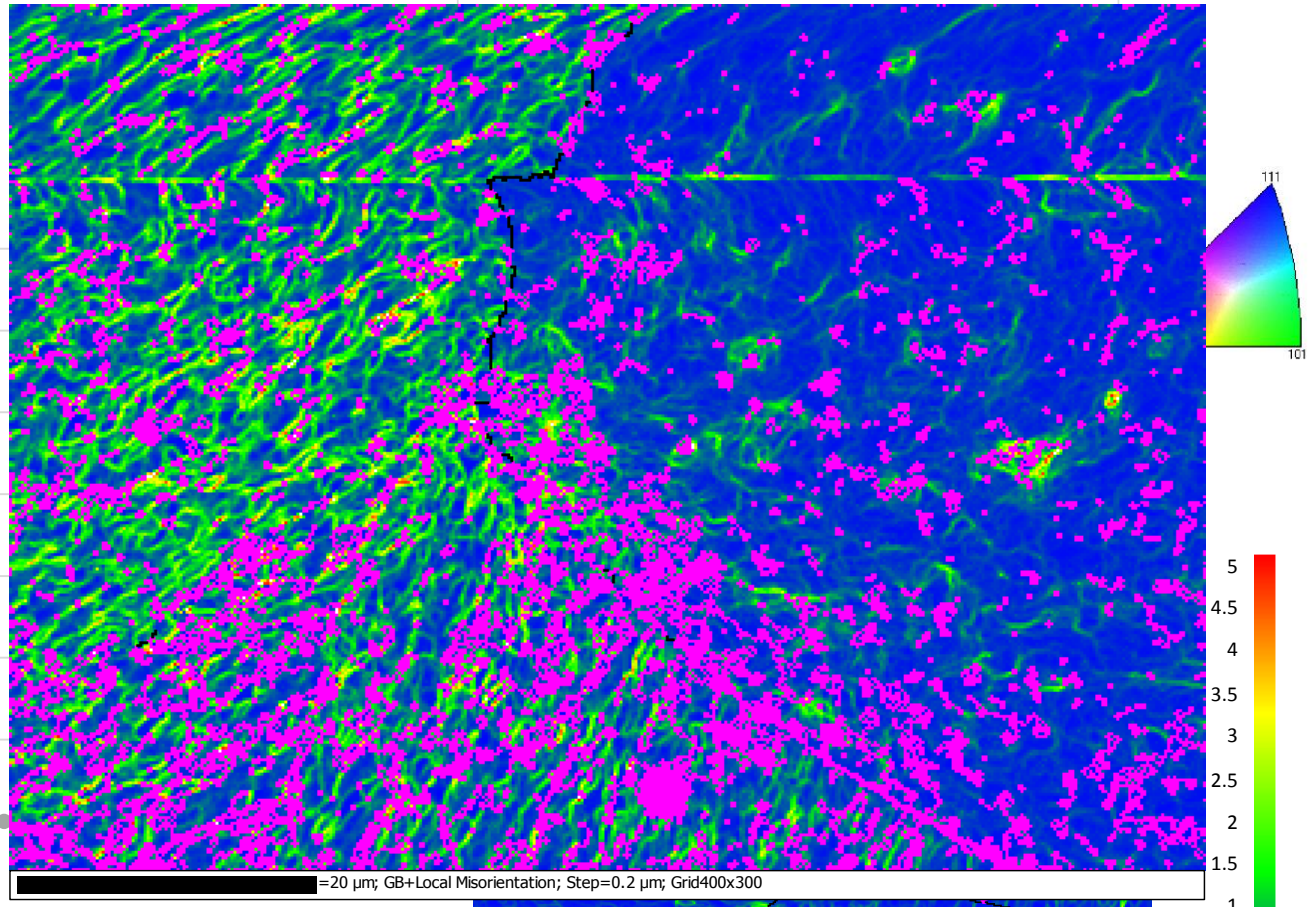
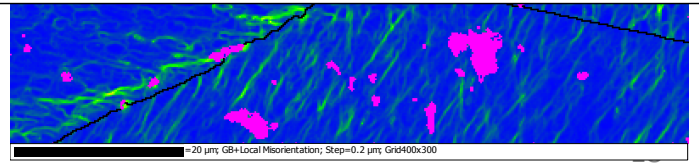






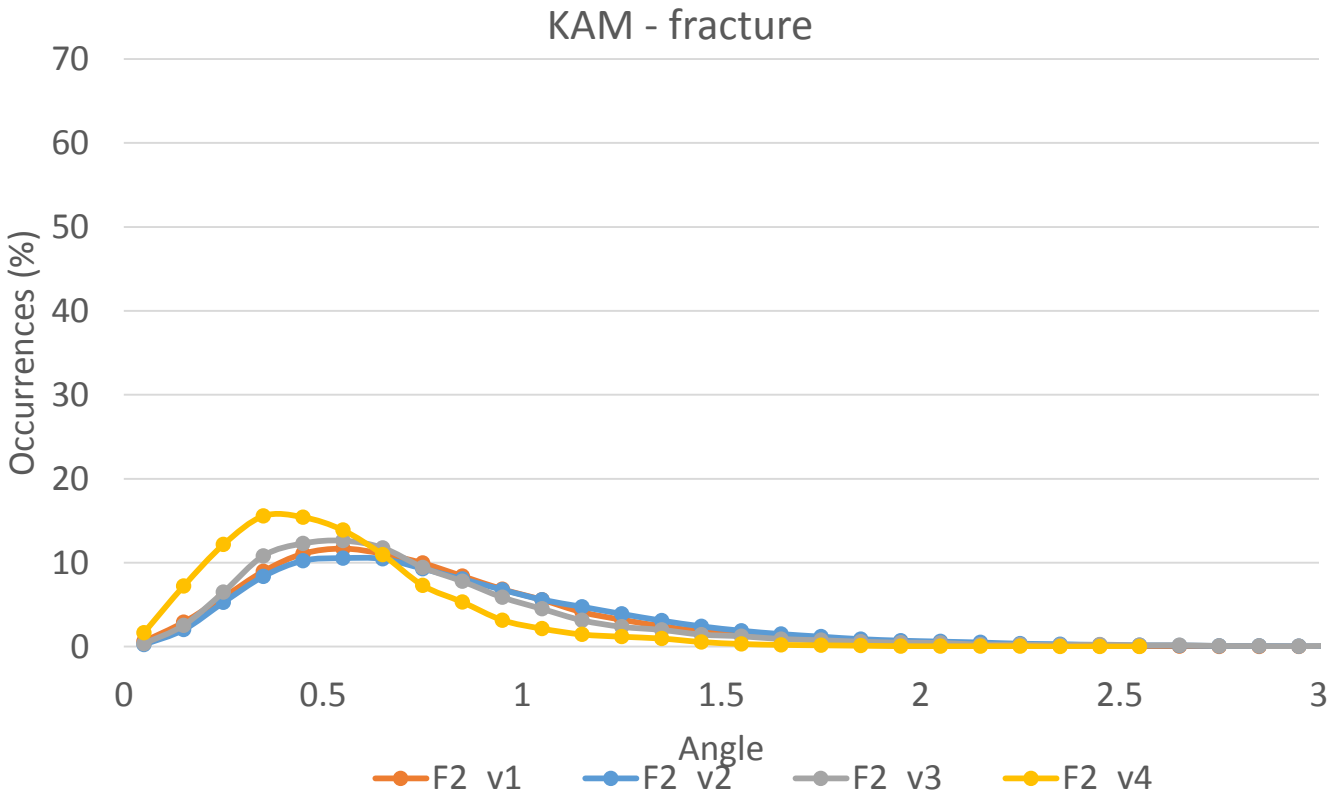
Angle

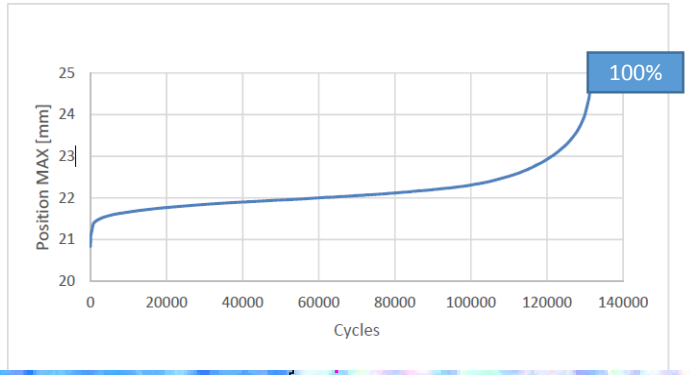
F8_83_v3 F8_83_v4 F8_83_v5 F8_83_v4 F8_83_v7

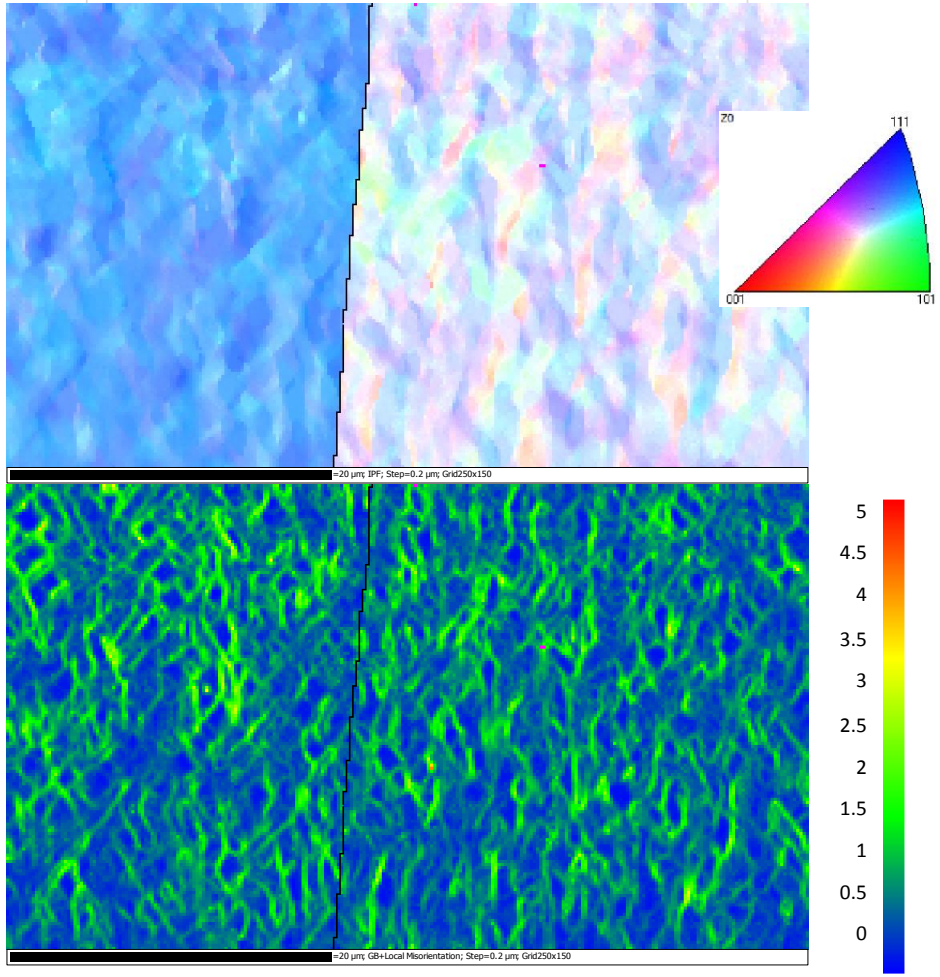


Fatigued (100% LT)

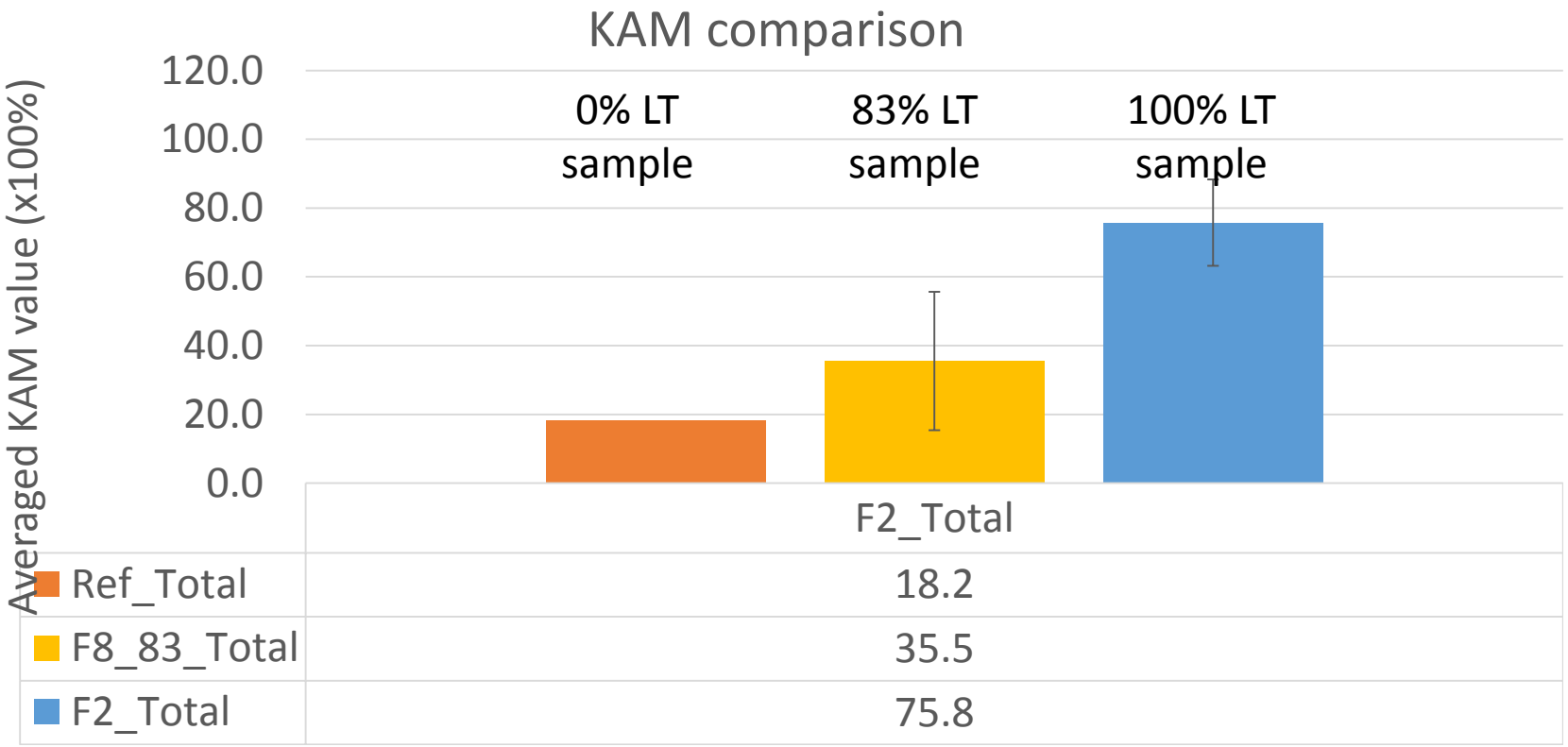
- Strong broadening of the distribution
- Shift to even higher value

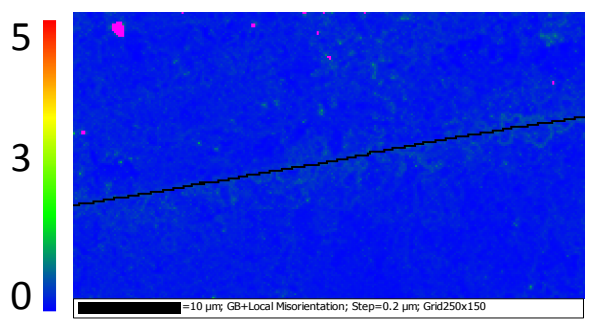


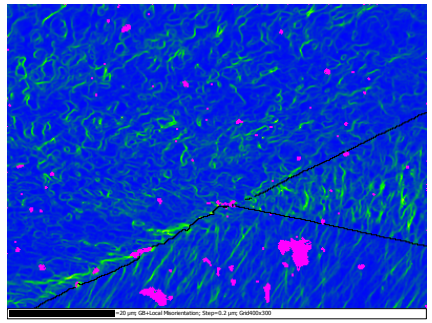


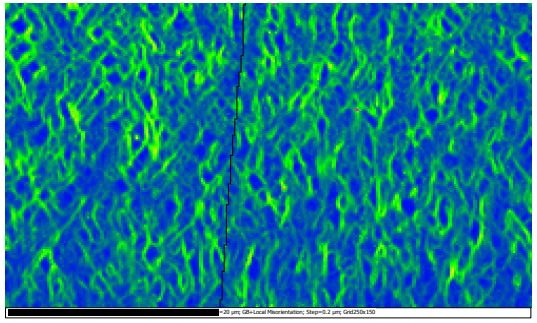


Summary of EBSD observations



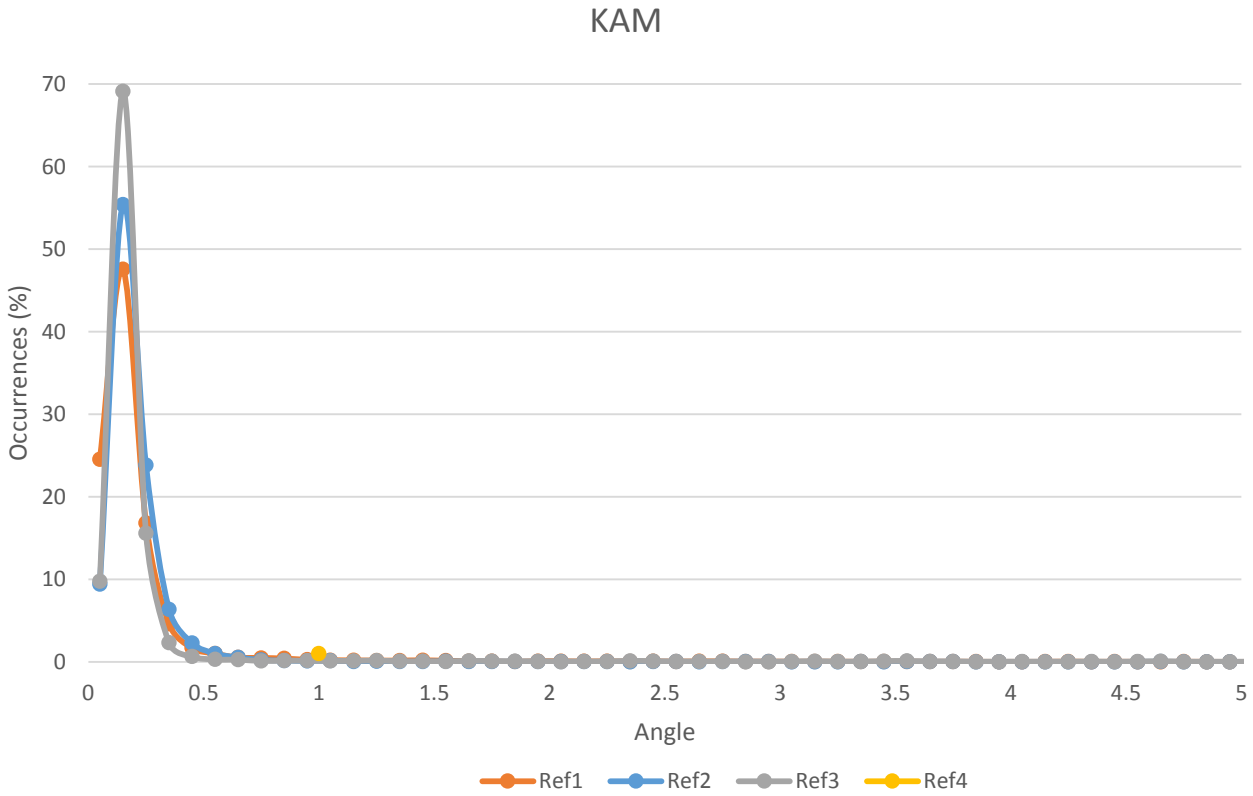


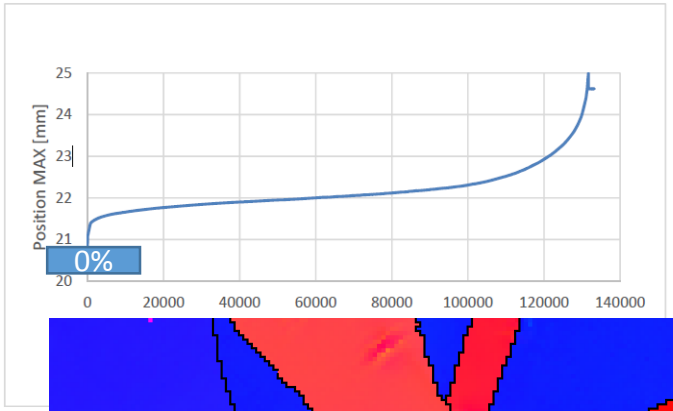


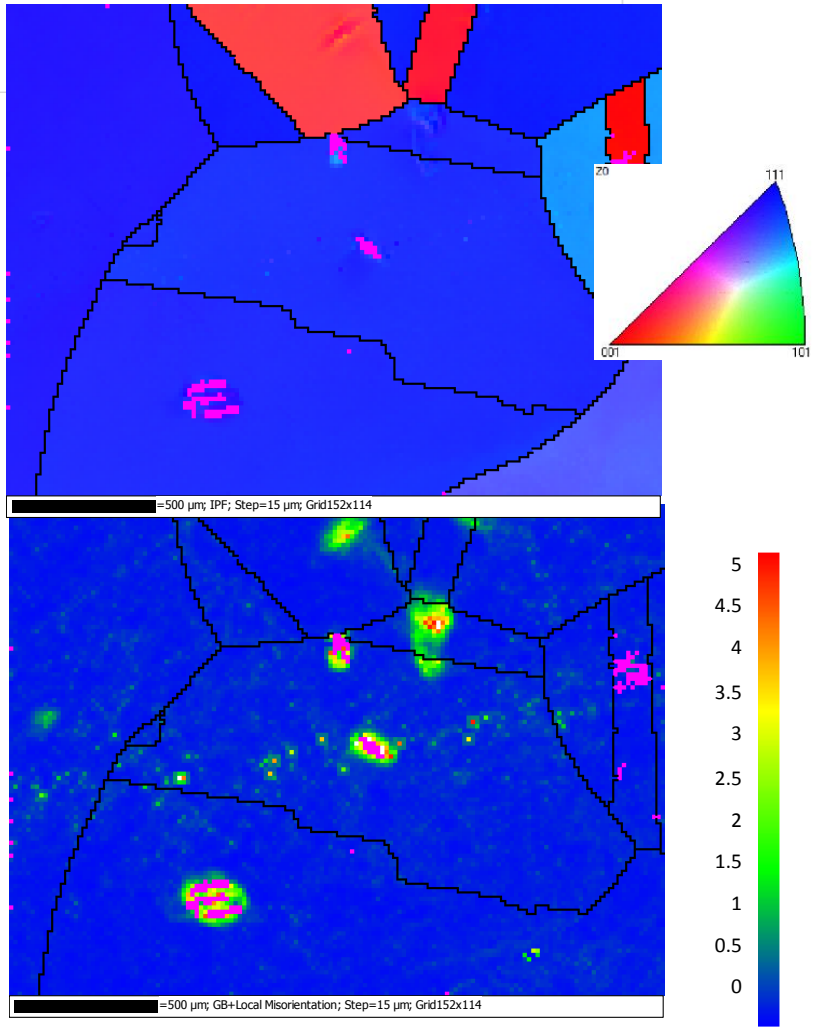


Ref (0% LT) Stepsize = 15 µm

- Not comparable with previous due higher step size

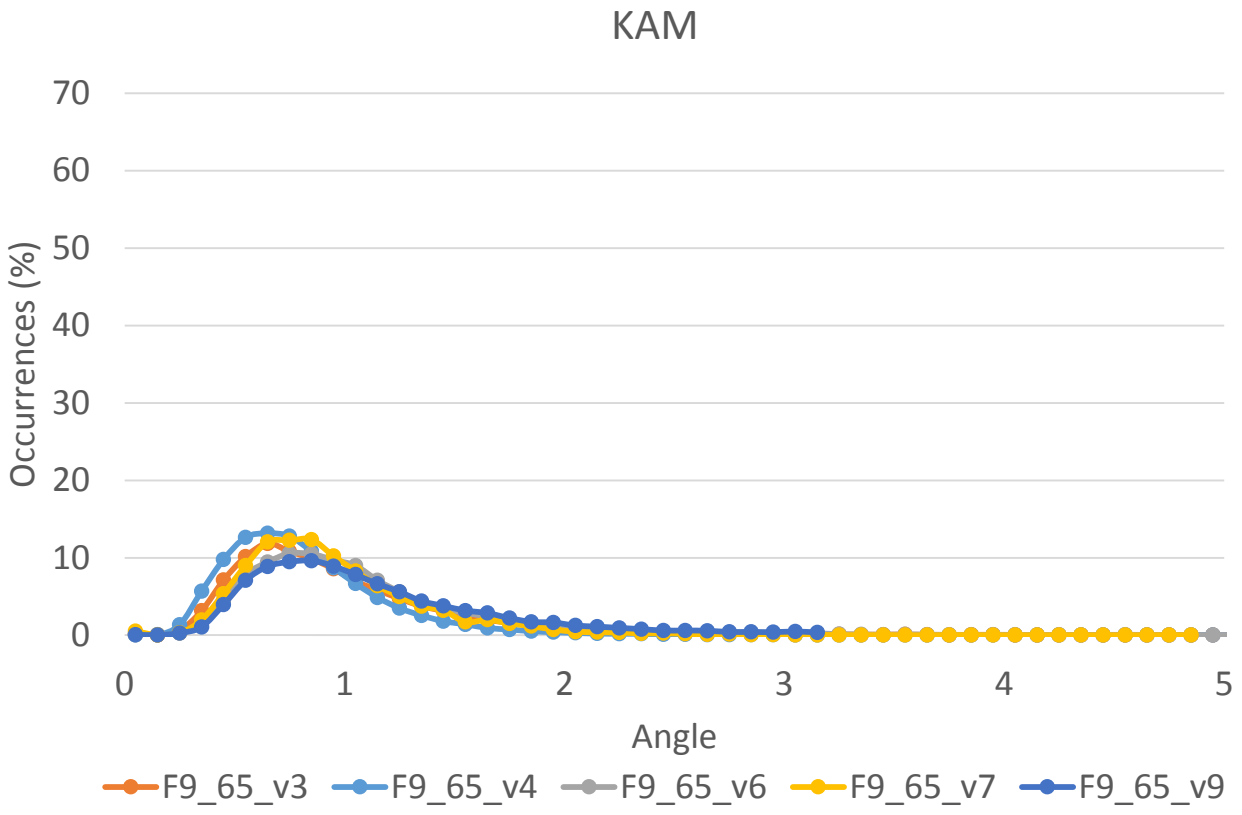


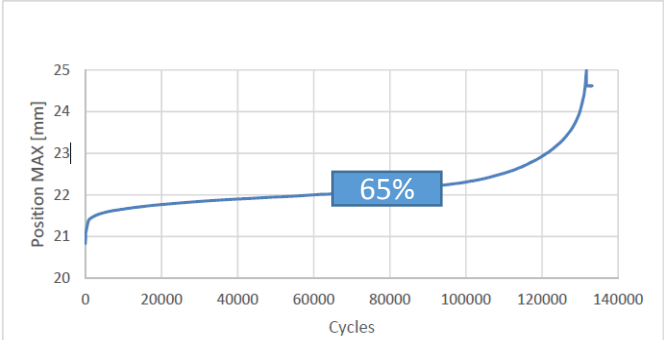


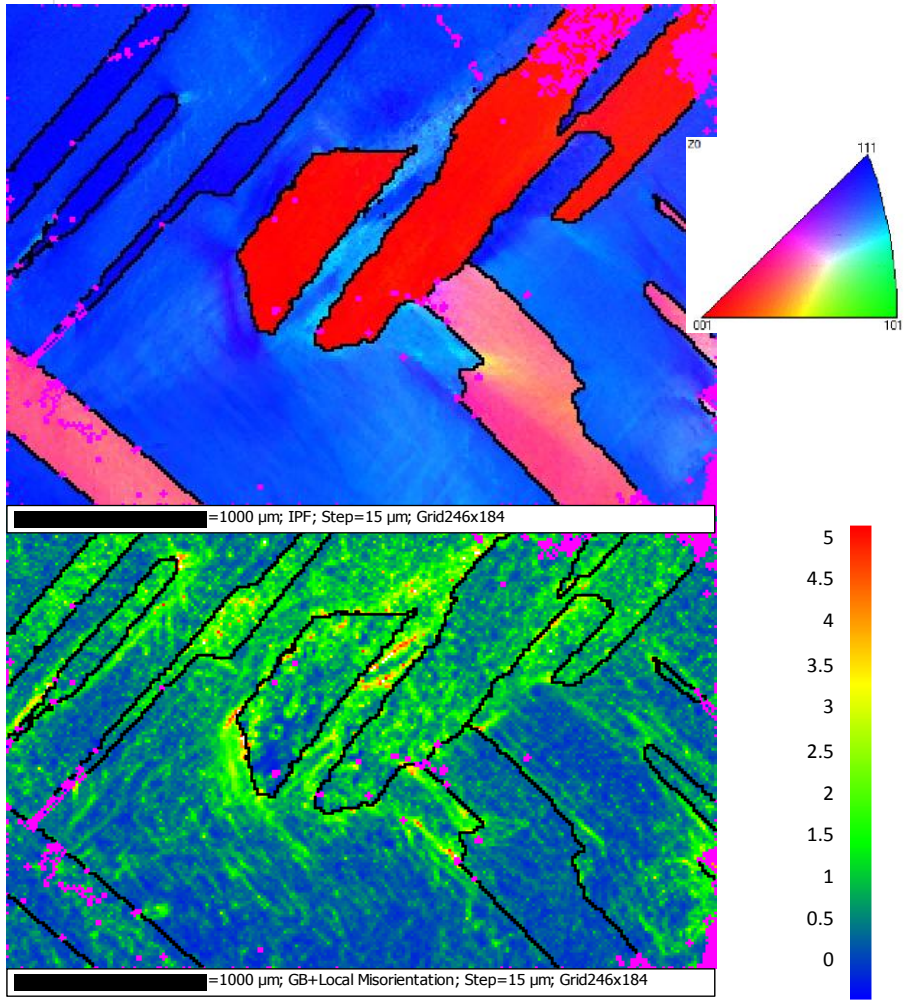


Fatigue (65% LT) 15µm stepsize

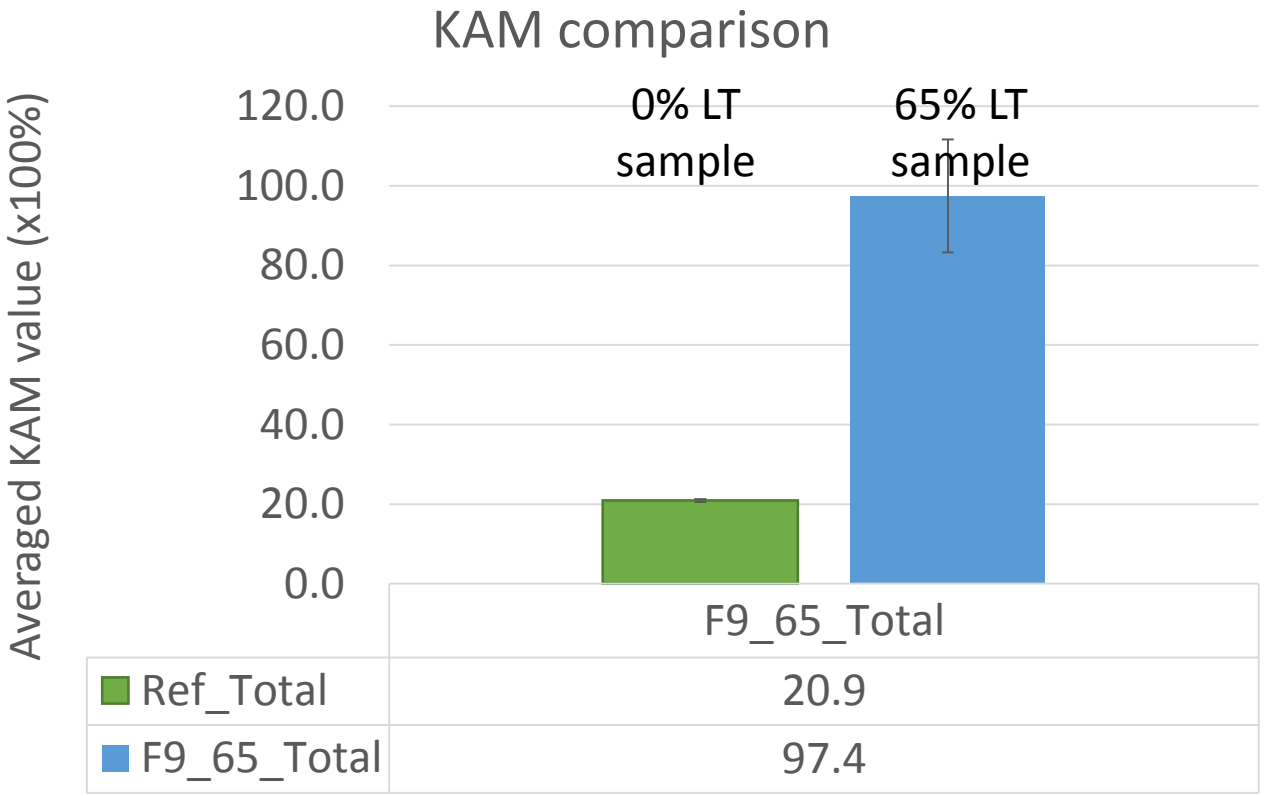
- Strong broadening of the distribution

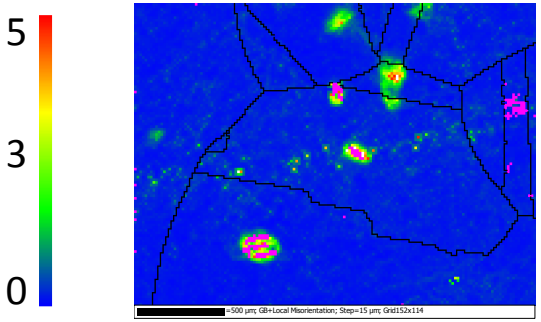


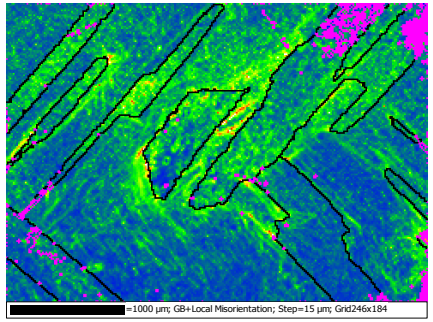




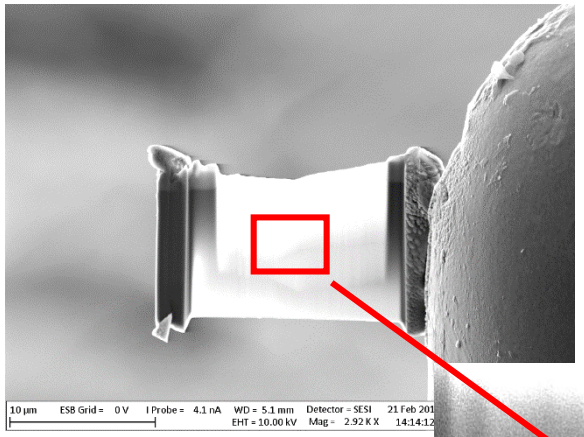
Comparison 15 µm stepsize



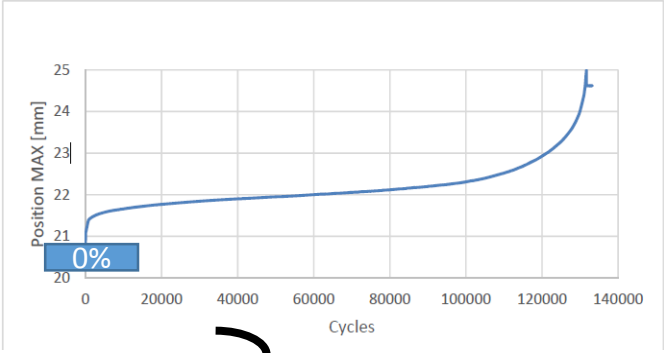




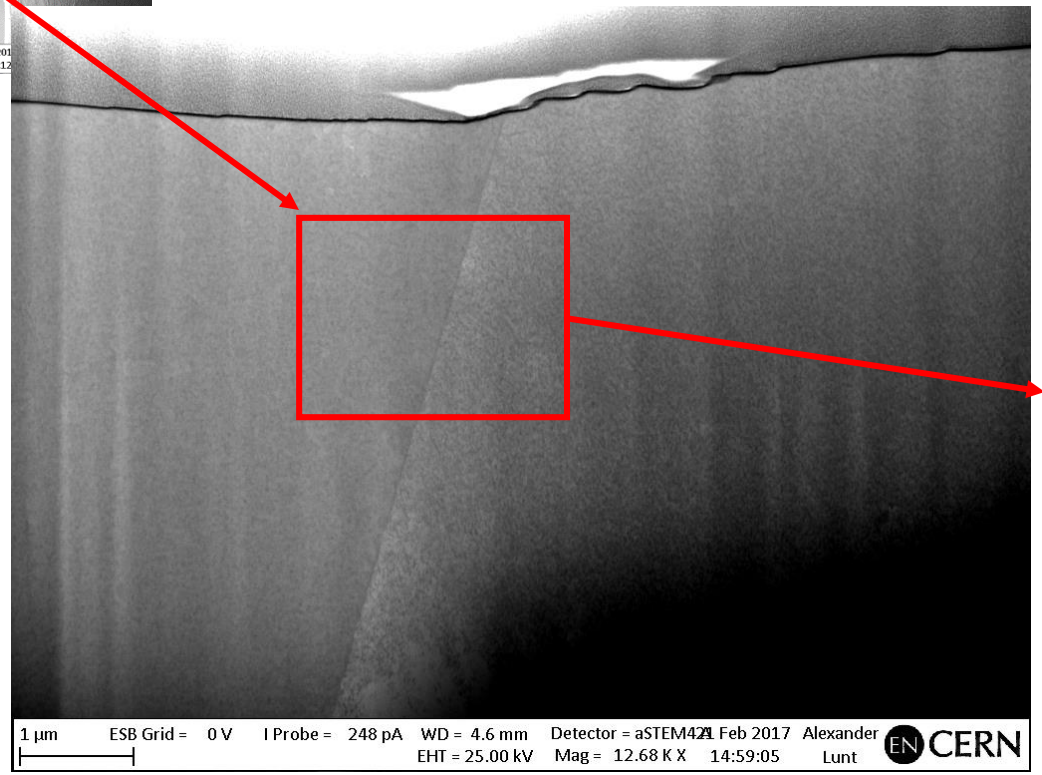
Reference (STEM)

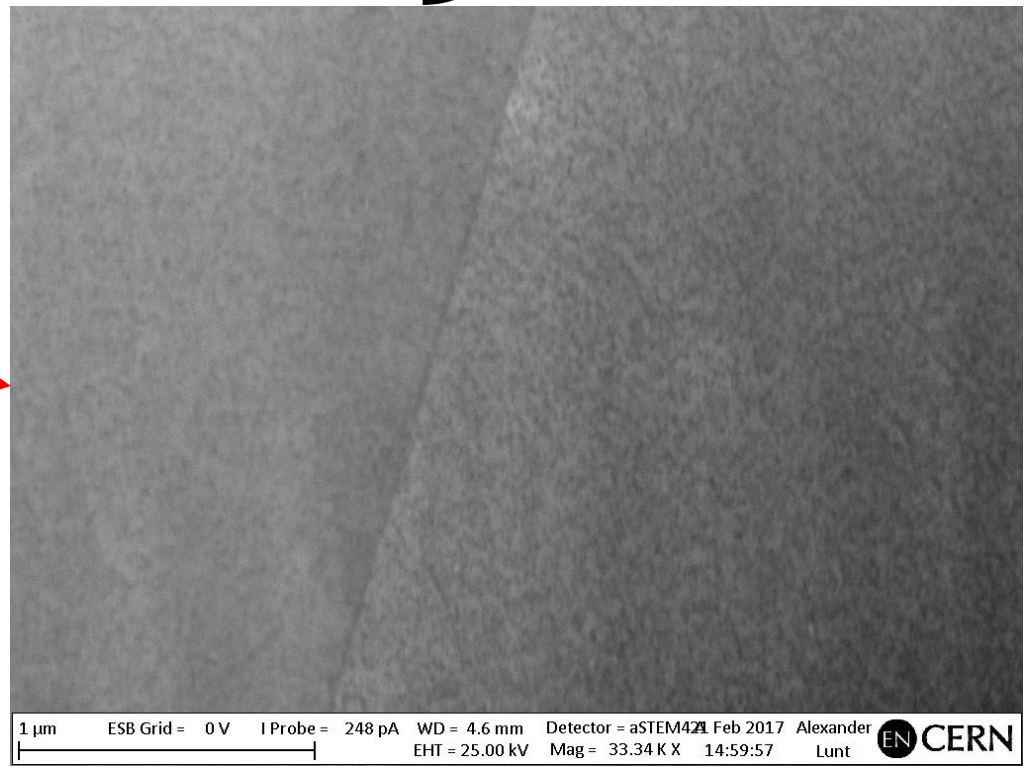


- Grain with different orientation
  - Homogeneous
  - Individual Dislocations?
  - Damage effect due to Ga beam preparation

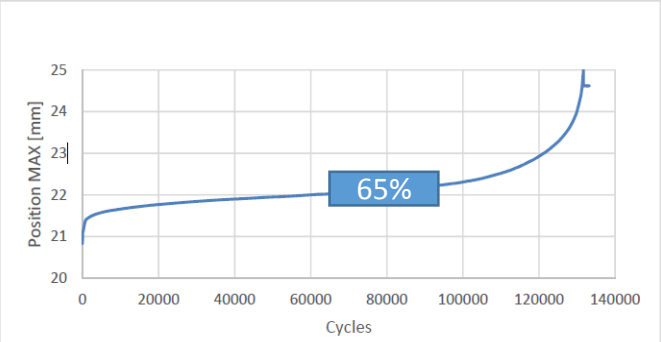


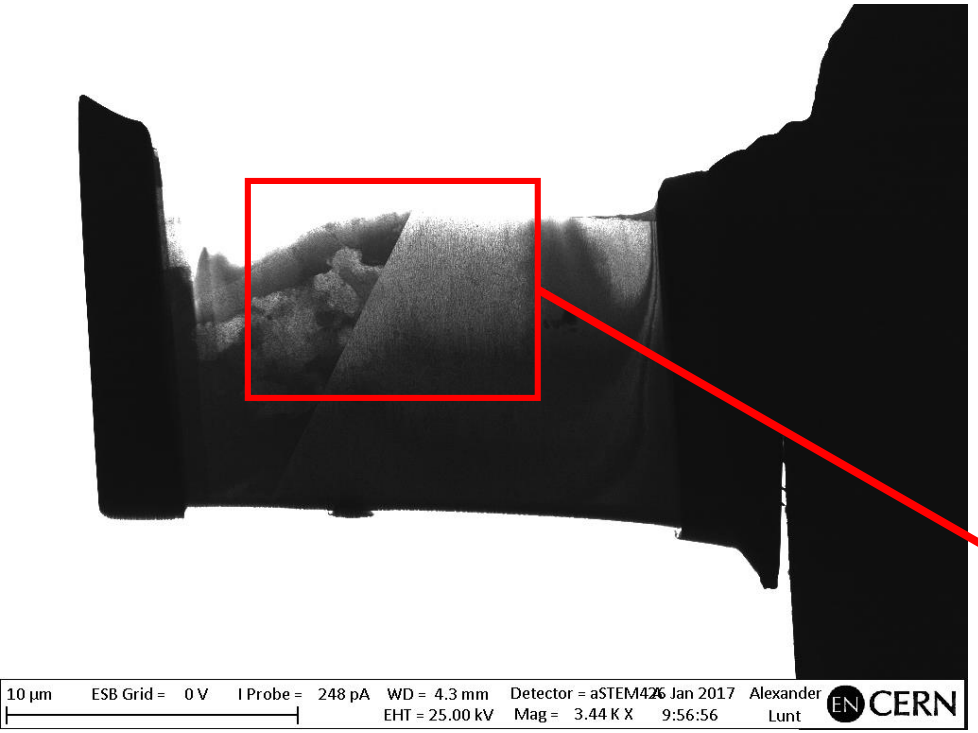
To be confirmed by TEM

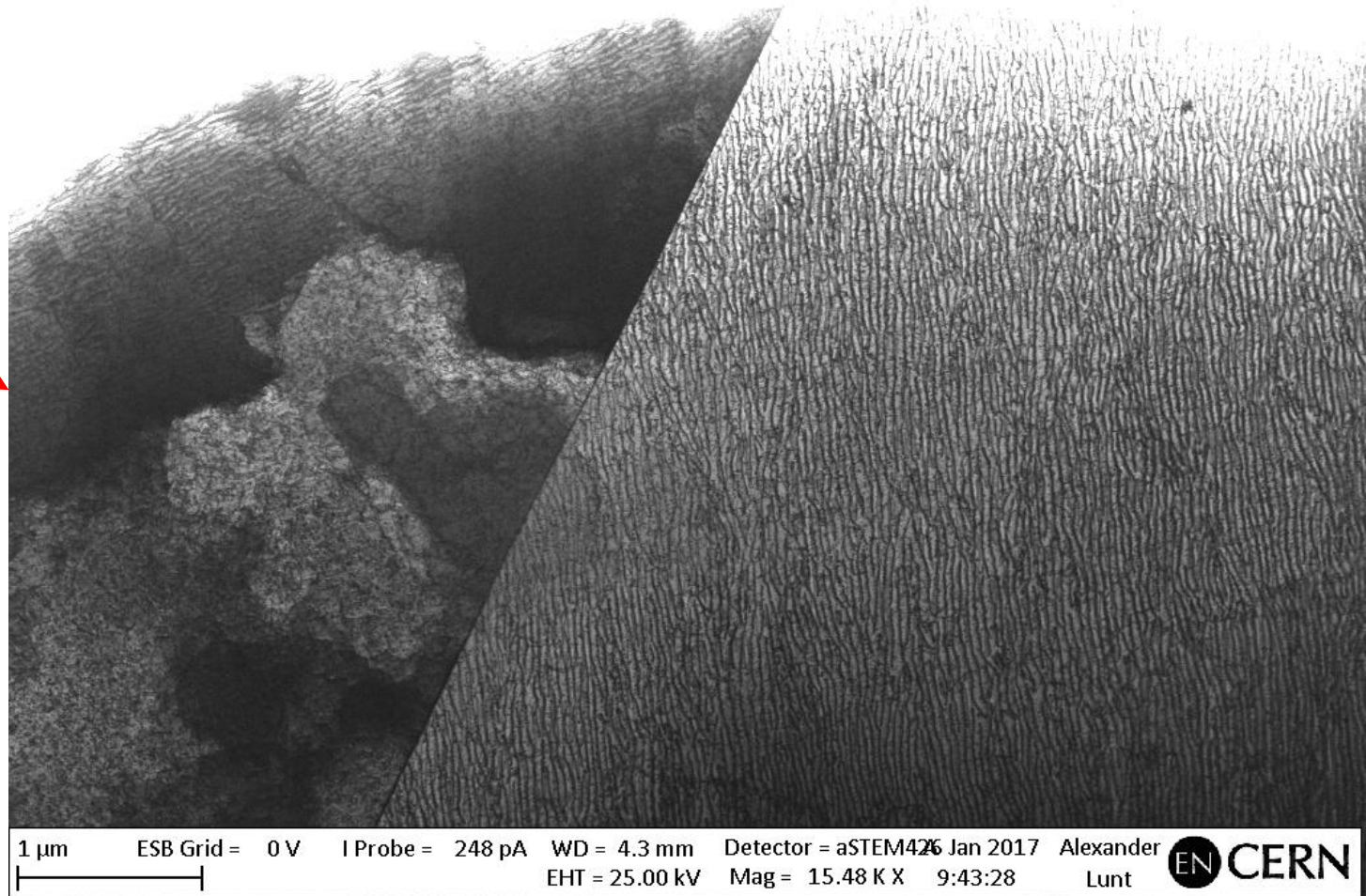




Fatigue (65% LT) 15µm stepsize

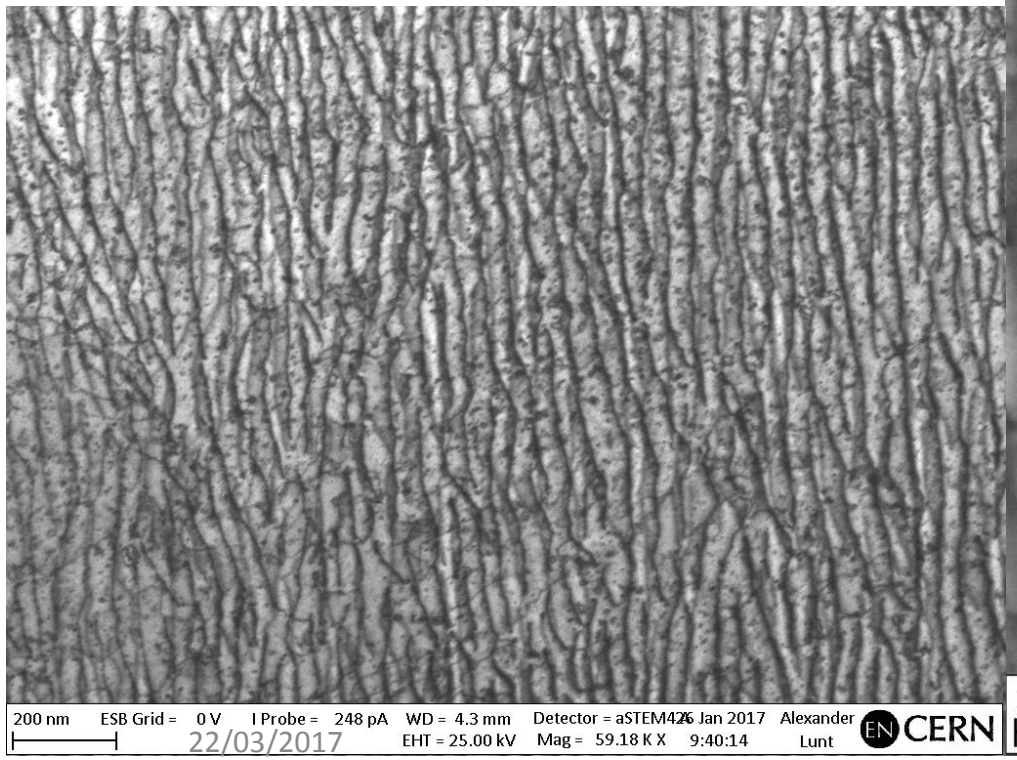


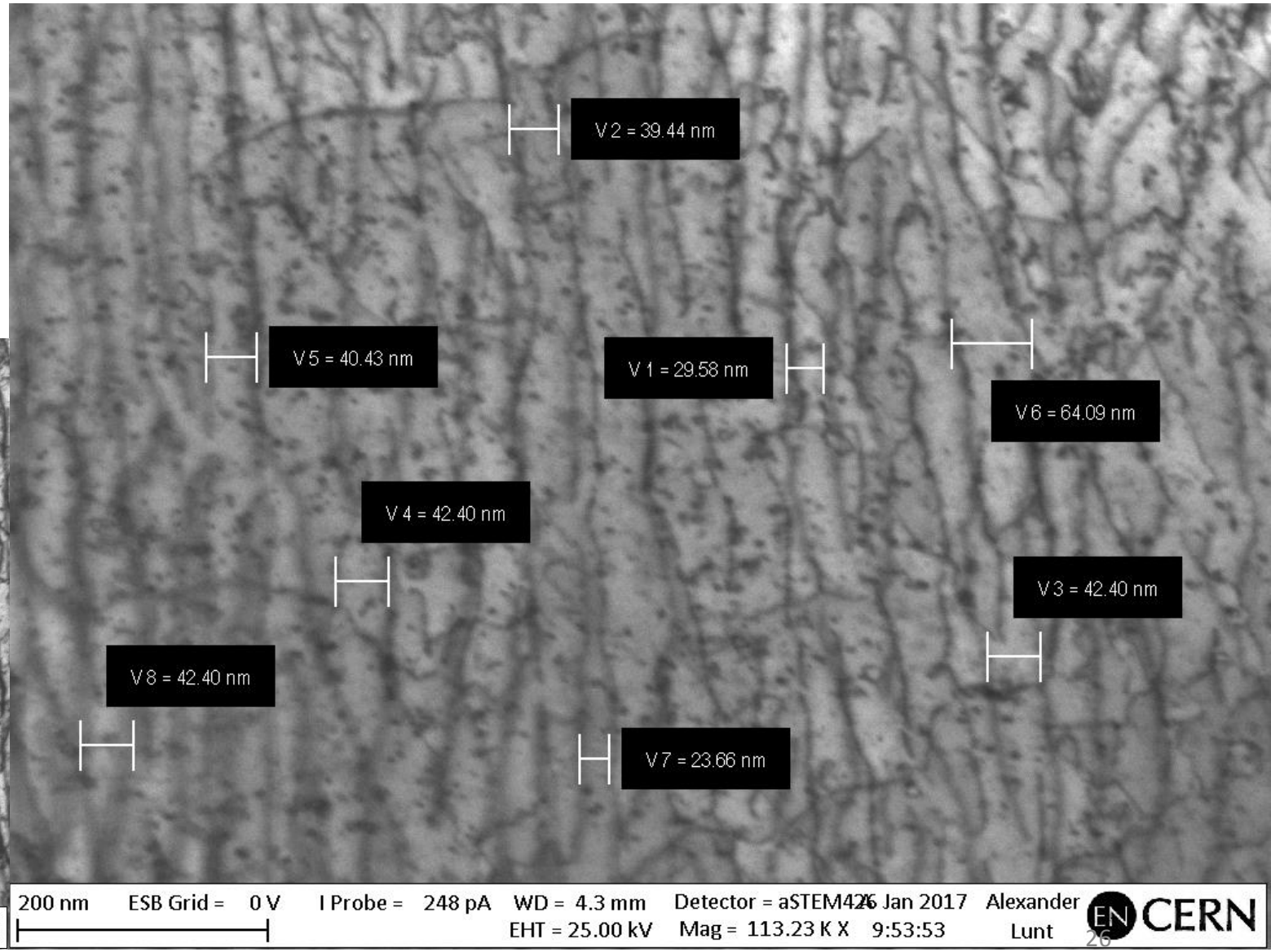




Fatigue (65% LT) 15µm stepsize

- Well structured in ladder type
- 20 – 60 nm



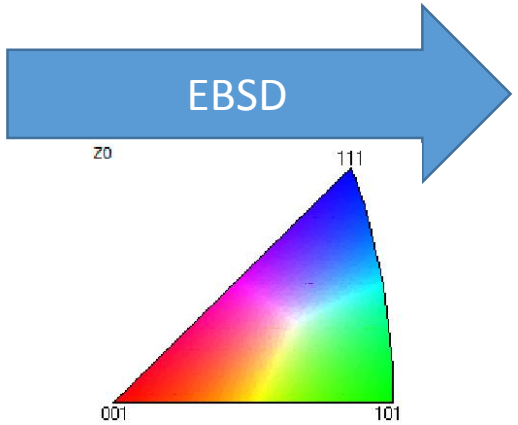


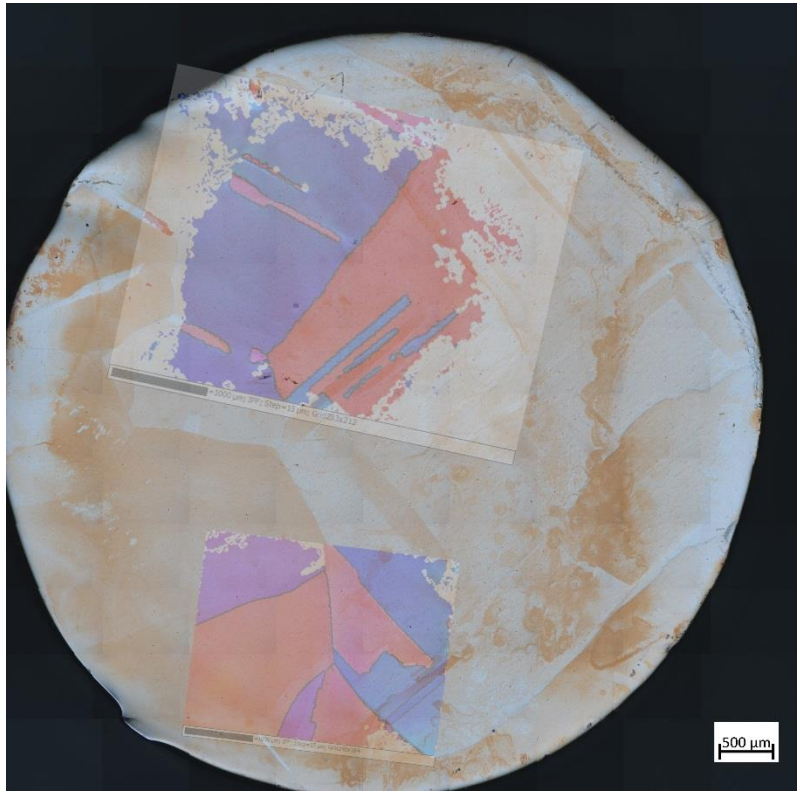
Outline

Introduction	Context		
	Aim of the study		
Protocol	Sampling	Heat treatment	
		Tensile test	
		Fatigue Test	
	Characterization method	EBSD (KAM)	
Results	Tensile test		
	Fatigue tested	0.2 µm stepsize	Reference
			Fatigue 83% LT
			Fatigue 100% LT
			Summary of results
		15 µm stepsize	Reference
			Fatigue 65% LT
			Summary of results
Perspective			
Conclusions			

Perspectives

- Those maps are important to select the area where the lamella (with SEM-FIB microscope) will be taken





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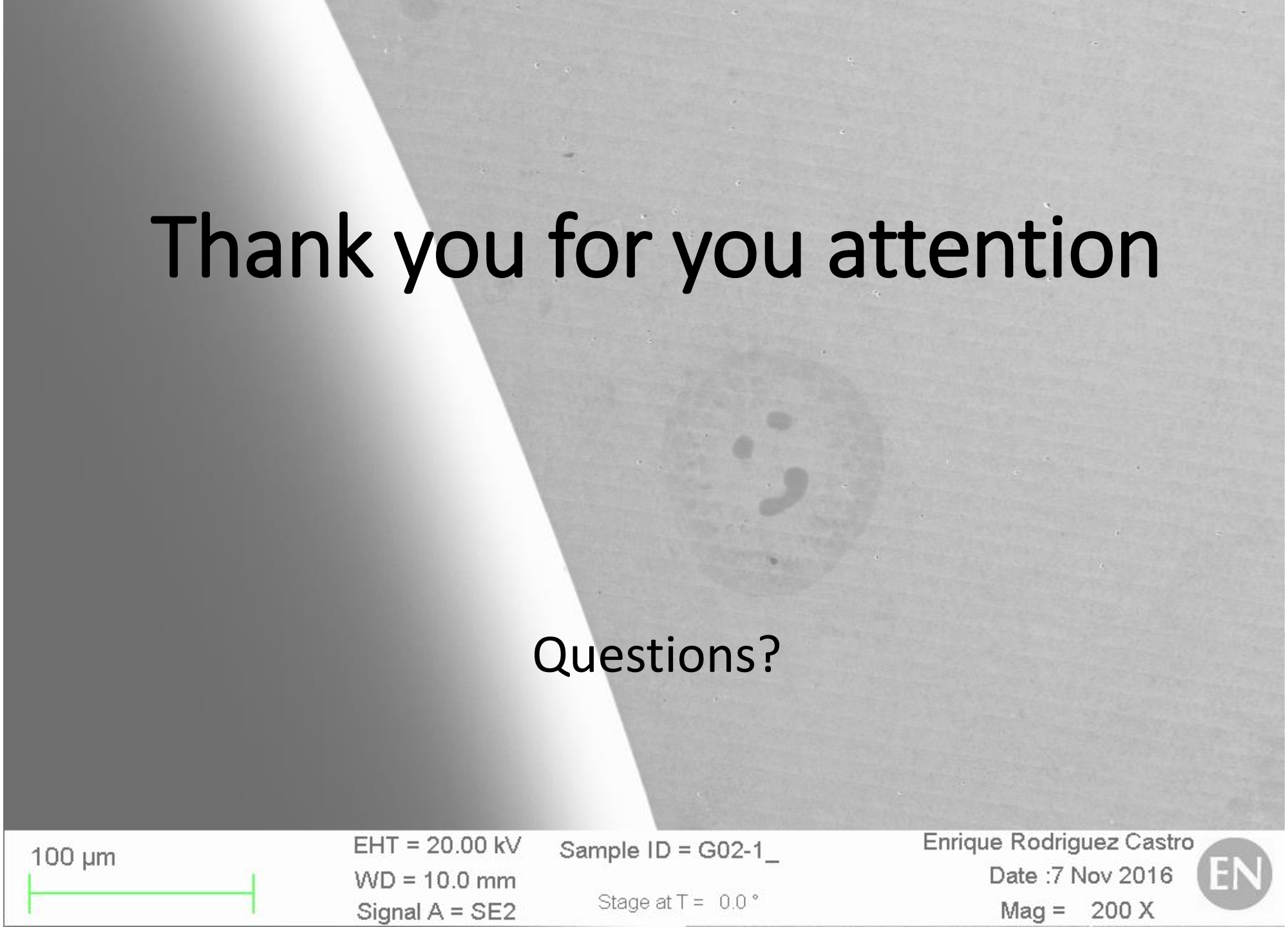
Conclussions

- EBSD has proved:
  - To be a good technique for quantification of ***dislocations arrangements over a large area***.
  - Different samples studied sample have higher dislocation density for increasing stress levels.
  - Statistics to be improved (currently only 3 stress levels and 2 lamellas)
- Further analysis with TEM
  - To confirm the observation done with STEM
  - To validate if observation of ***dislocations patterns*** through KAM maps (local mis-orientations) is possible → arrangement of dislocation patterns in bigger structures

Thank you for you attention

Questions?



Dislocation patterns in Cu-OFE by EBSD and their relation with BD

Advanced studies

Enrique RODRIGUEZ CASTRO
(CERN & University of Vigo)

Future

- FIB
  - In a well characterized area
  - Lamella
  - STEM

EBSD