Update on the post-irradiation examinations on high Z materials from HRMT-27 and HRMT-42 experiments

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RADIATE
Radiation Damage In Accelerator Target Environments
5th Collaboration Meeting

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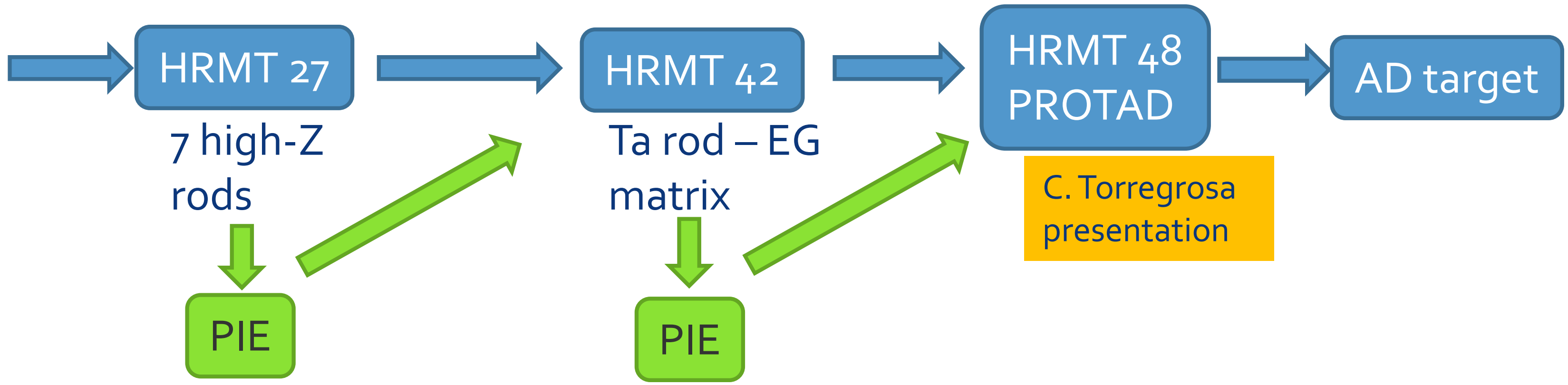
- HRMT-27
  - Tantalum
  - Iridium
- HRMT-42
  - Tantalum
- Bonus: W-TiC

Context

AD target consolidation



PIE: Post Irradiation Examinations

E. Fornasiere PhD work

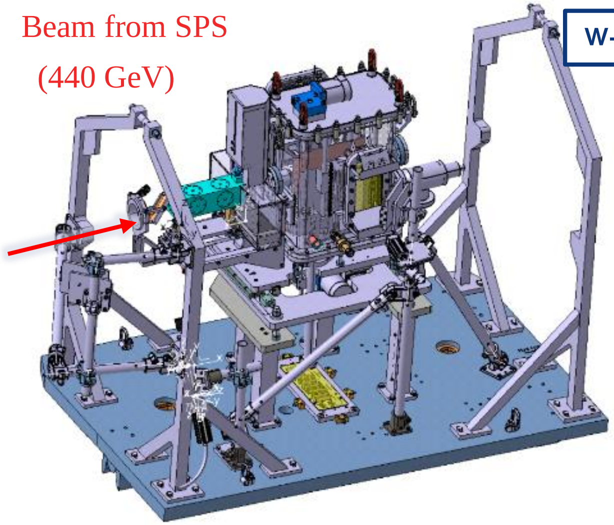
Non-destructive testing ⇨ **Sample handling** ⇨ **Destructive testing**

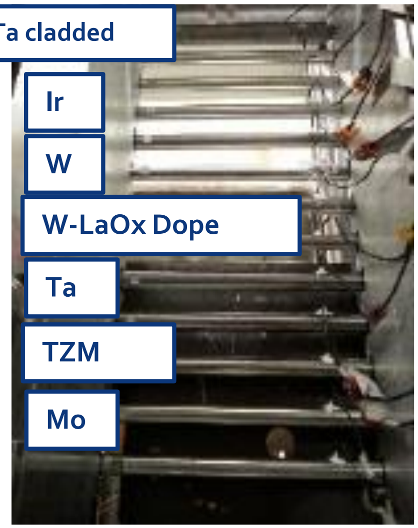
Non-destructive testing:
- Visual inspections
- Metrology
- Tomography (X-ray, neutron)
- ...

Sample handling:
- Transport
- Cutting
- Surface preparation

Destructive testing:
- Microstructure analysis
- Mechanical testing
- ....

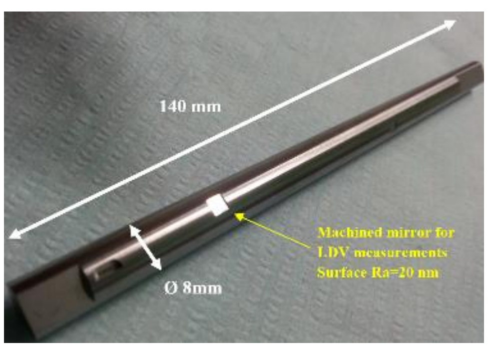
HRMT-27: The experiment (2015)

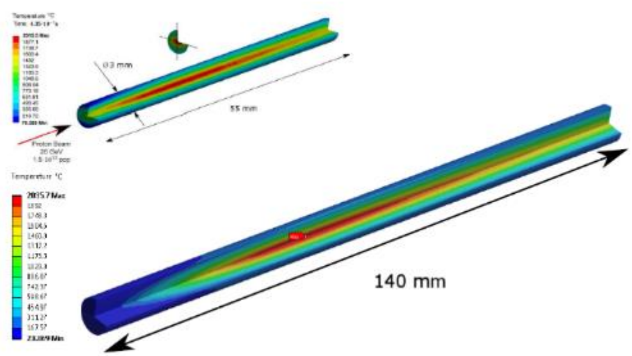




- 13 rods of high-Z materials impacted by 440 GeV/c beam
- Irradiation performed in a **ramped** way to obtain material response at intermediate state before reaching AD-Target conditions

- Targets geometry and beam parameters adapted to recreate AD target





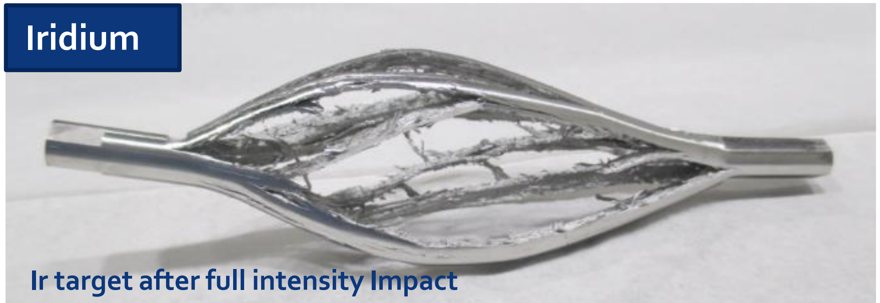
- Beam intensity ramped up to reach:

Material	Max ΔT (°C)	Max Tensile Pressure (GPa)
Ir	2200 °C	9 GPa
W	2000 °C	5.6 GPa
Mo/TZM	850 °C	1.3 GPa
Ta	1850 °C	4.5 GPa

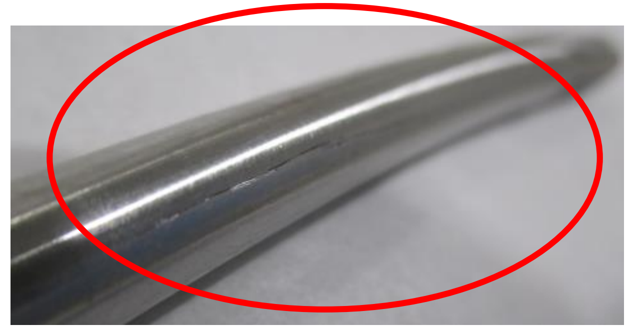
Courtesy of C. Torregrosa

HRMT-27: Targets after the experiment

Iridium



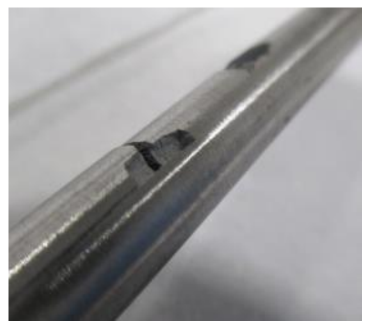
Ir target after full intensity Impact



Longitudinal cracks in **Iridium** at intermediate intensities.

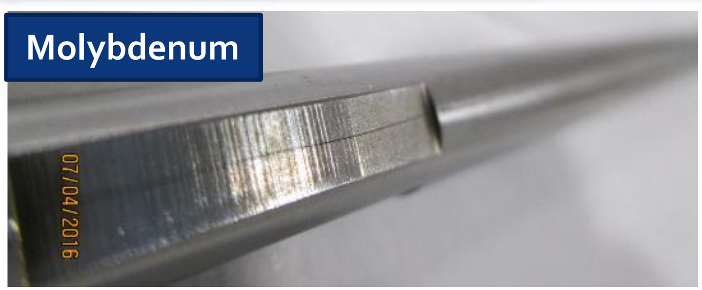
Tungsten

W after full intensity



- Tungsten targets present the larger amount of longitudinal cracks.
- Spall of fragments at the surface also took place in tungsten.

Molybdenum

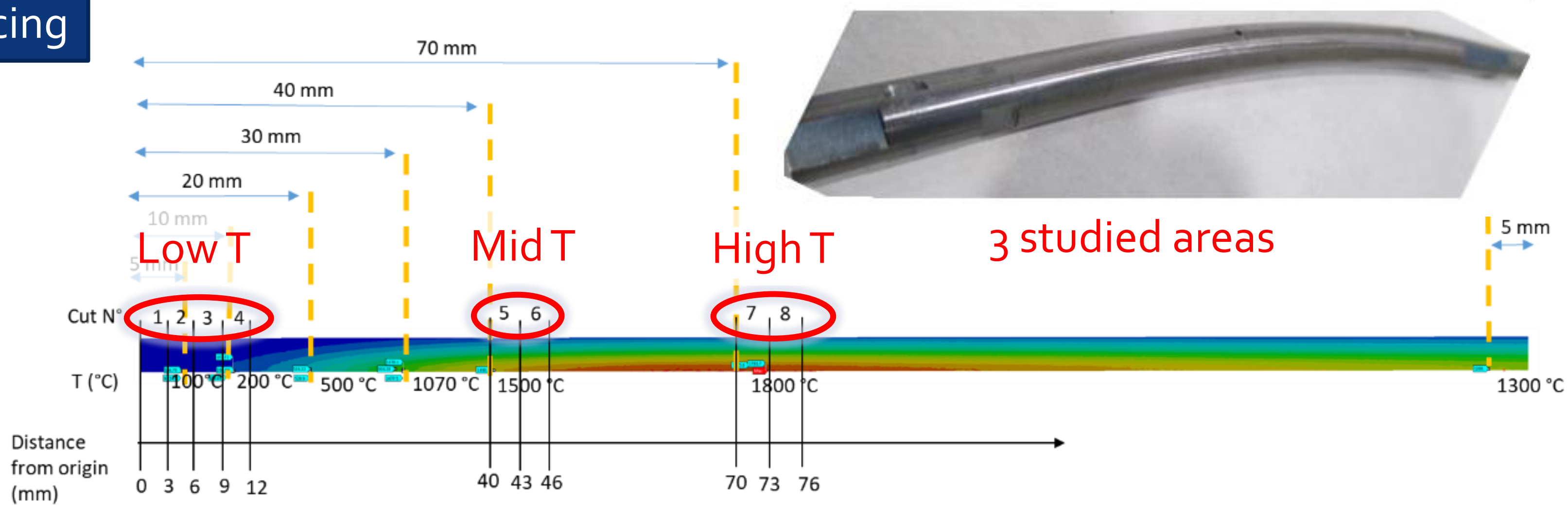


Tantalum

- No cracks were observed in Ta targets.

HRMT-27: Ta target

Slicing



Preparation

8 cross-sections

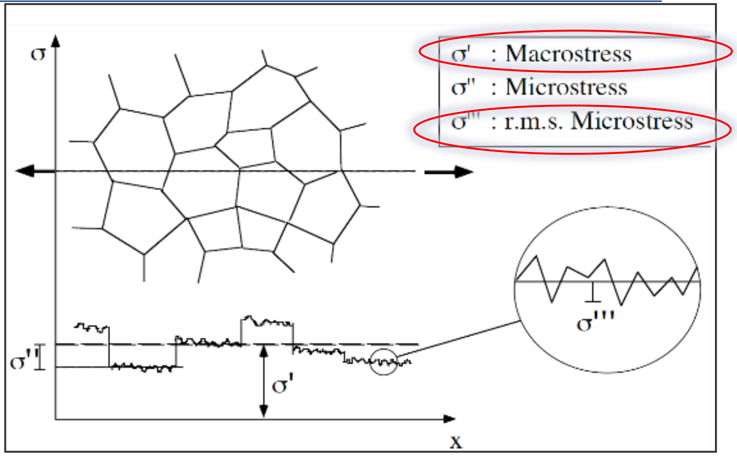
- Hot **mounting** in phenolic resin
- **Grinding** with grit papers (P500, P1200 and P2400)
- **Polishing** with diamond suspension (6 µm and 3 µm)
- Final polishing with **colloidal silica suspension** (0.025 µm) + 5% H_2O_2
- **Intermediate etching** in between each grinding and polishing step, with etching #66 from ASTM 407: sample immersion for 2' in 10 mL HF + 20 mL HNO_3 + 30 mL HCl
- **Removal** from the resin

Preparation procedure by S. Forsetlokken

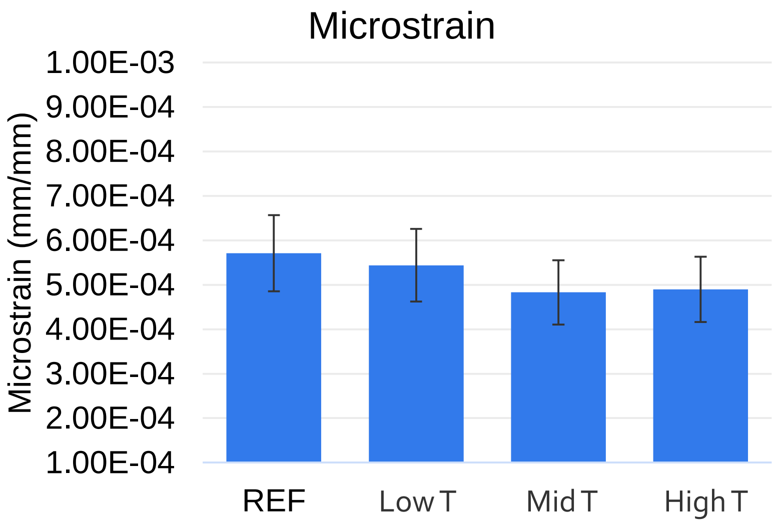
HRMT-27: Ta target

1. Microstructure quantitative analysis

- **X-ray diffraction**
- Microstructure analysis from peak profile analysis with Rietveld software (MAUD)
- Residual stress measurements in cross-sectional planes
- Samples representative of three areas: Ref, Low T, Mid T and high T
- Measurements probing entire cross-sections



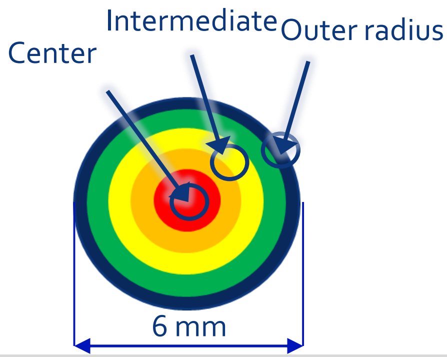
Microstructure analysis



Residual stress measurements

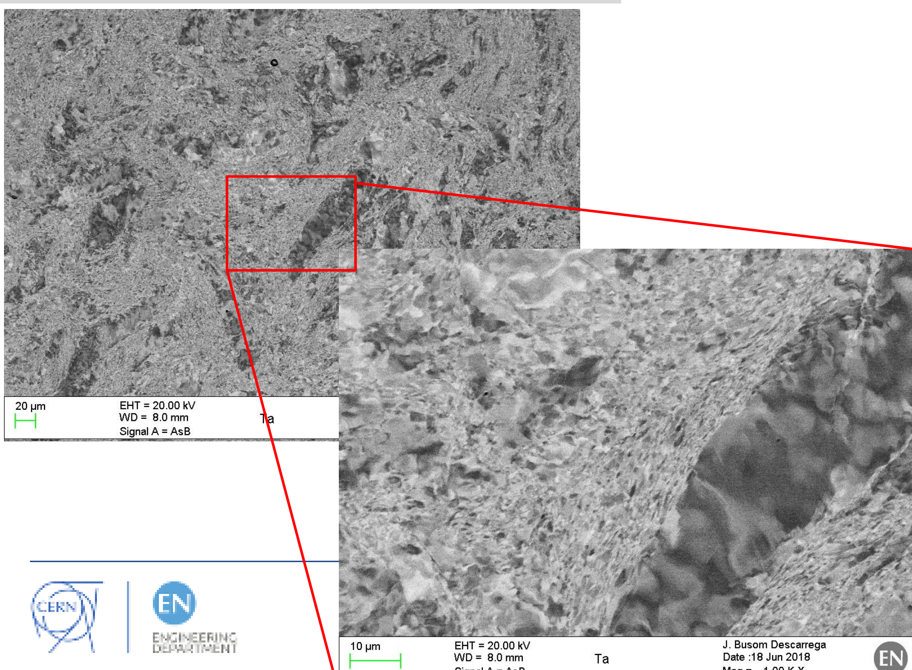
HRMT-27: Ta target

2. Microstructure imaging

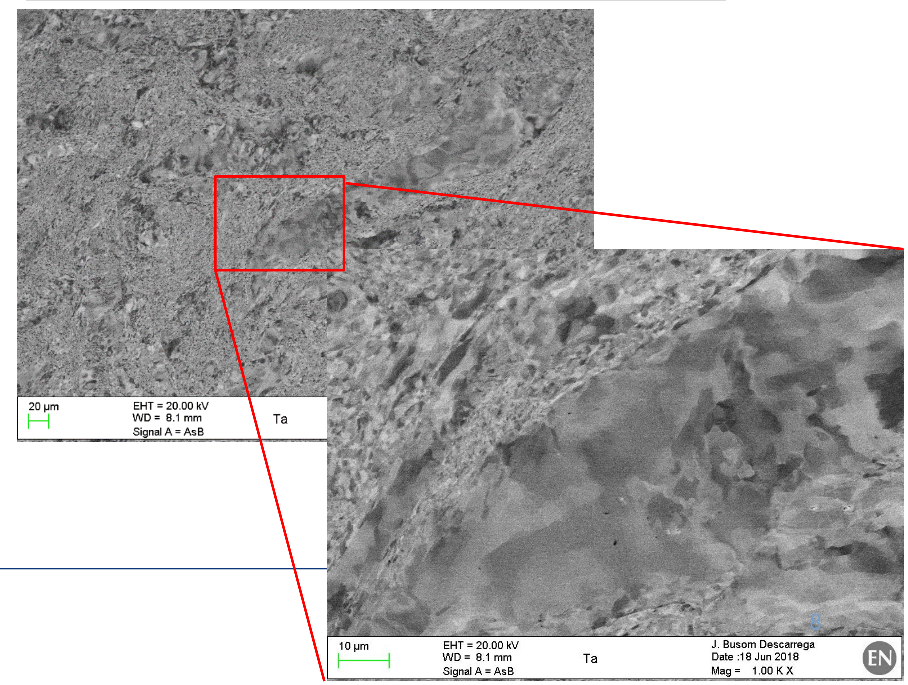


- Scanning Electron Microscope
- **Backscattered electron detector**
- Microstructure observable by channelling contrast
- Imaging in samples representative of the three areas: Ref, Low T, Mid T and high T
- And at three locations: Outer radius, intermediate and center
- >40 micrographs acquired

Reference sample at center

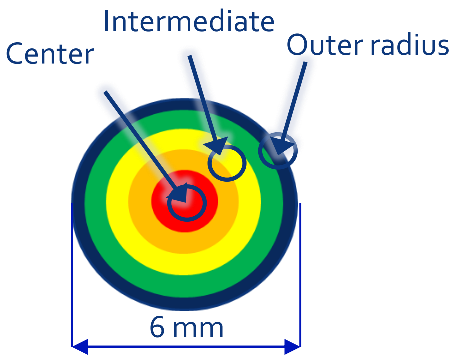


High T sample: Slice 7 center

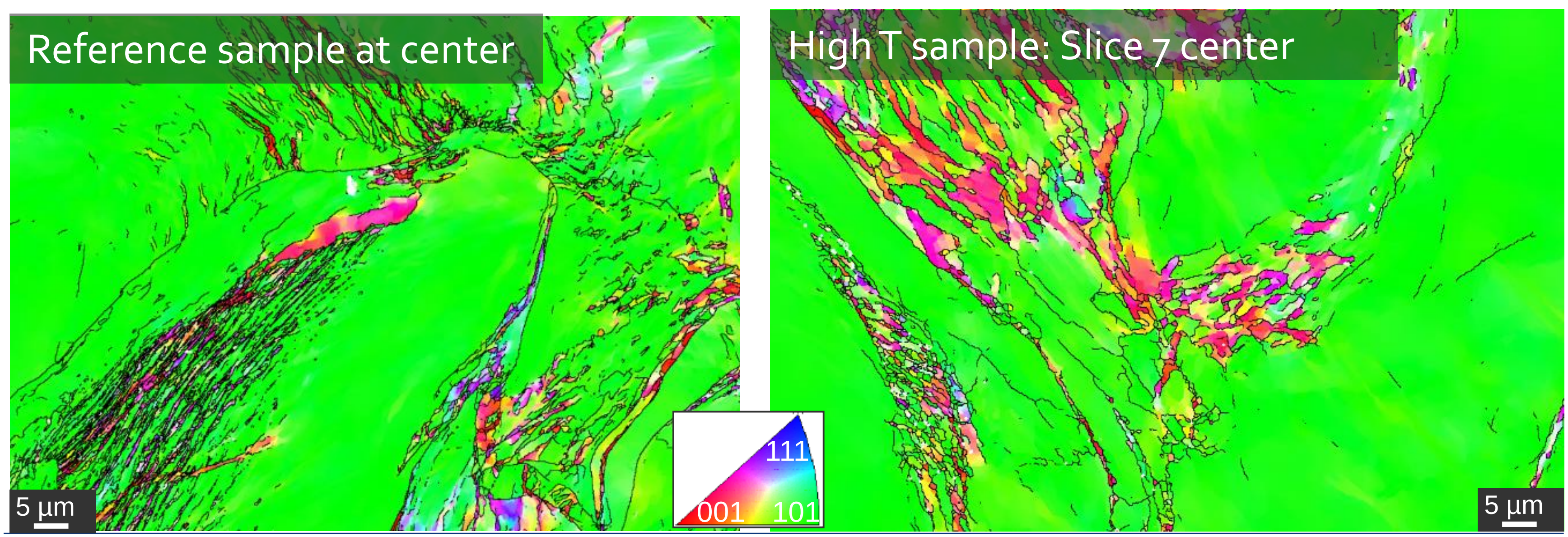


HRMT-27: Ta target

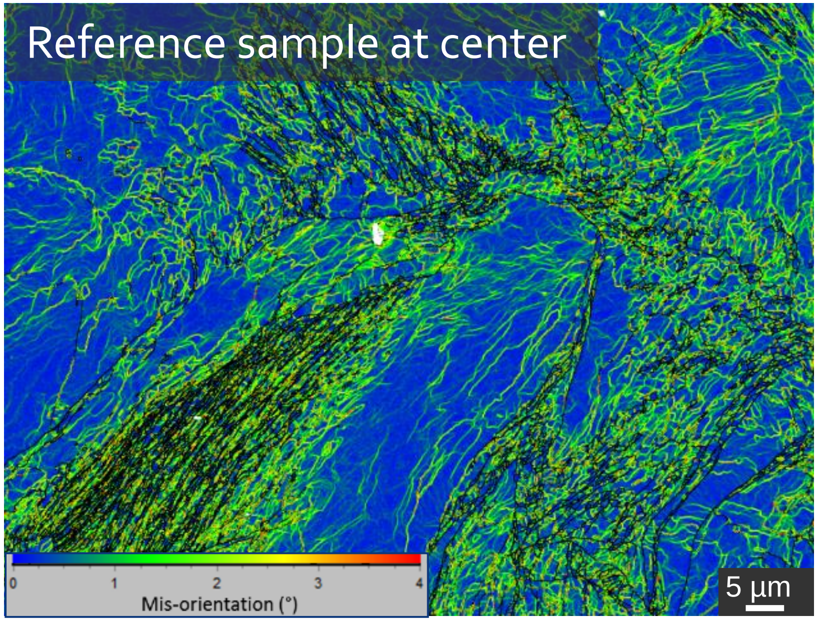
2. Microstructure analysis

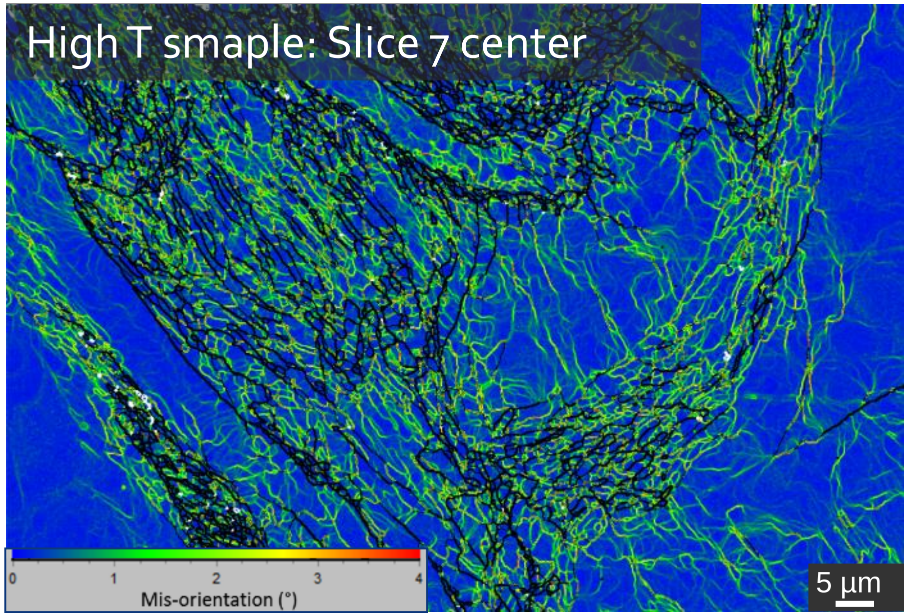


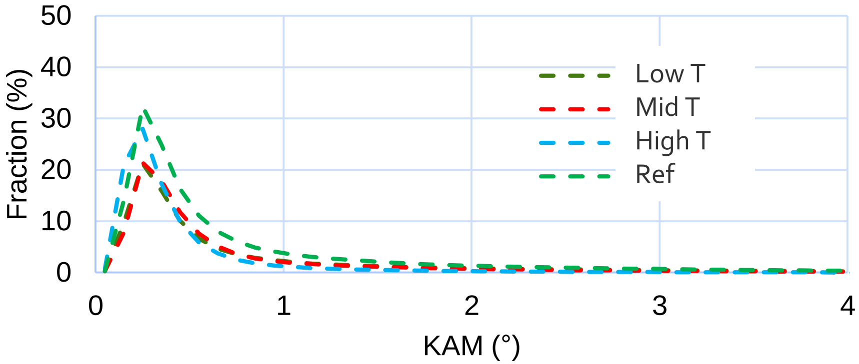
- Scanning Electron Microscope
- **Electron backscattered diffraction**
- Microstructure observable by inverse pole figure mapping
- Microstructure plastic deformation observable by misorientation mapping
- Samples representative of the three areas: Ref, Low T, Mid T and high T
- And at three locations: Outer radius, intermediate and center
- > 15 acquired EBSD mappings



HRMT-27: Ta target





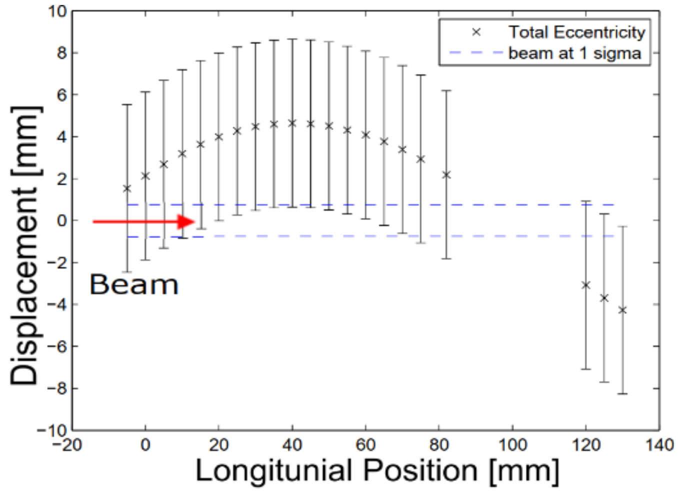


HRMT-27: Ta target

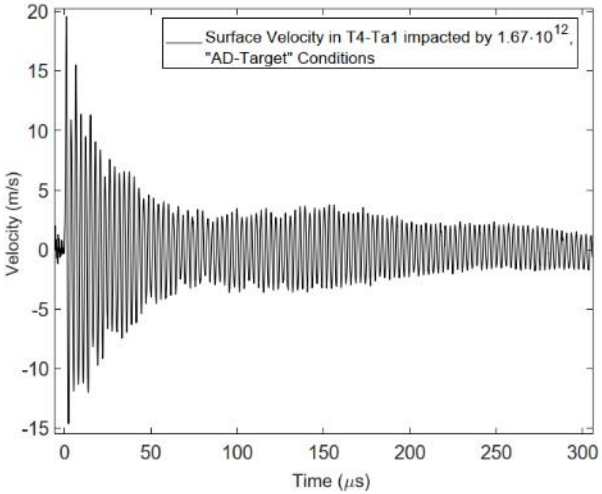
- No evidence of microstructural differences after beam impacts

- Target showed macroscopic bending

Target was certainly impacted at highest intensity



Surface speed indicated plastic deformation

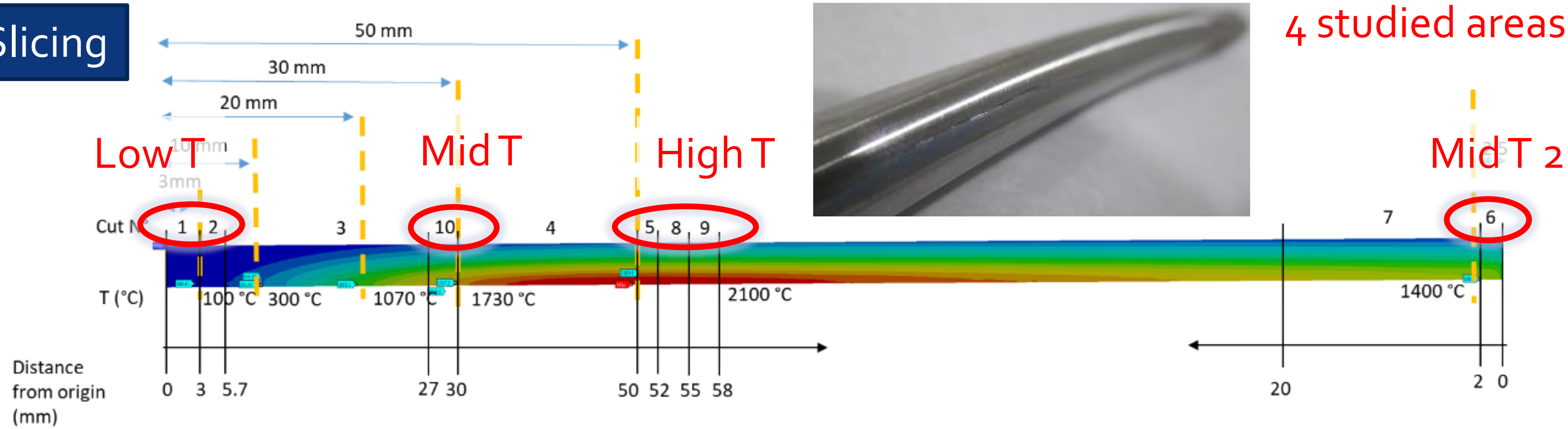


- Original microstructure already deformed due to forming

Courtesy of C. Torregrosa

HRMT-27: Ir target

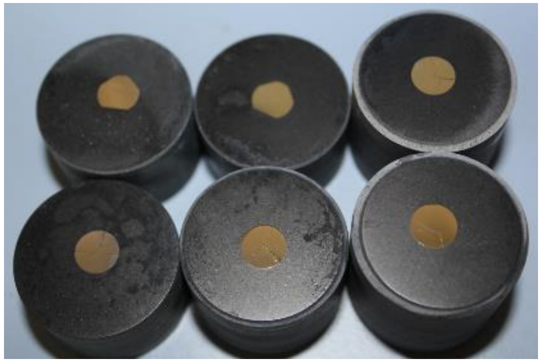
Slicing

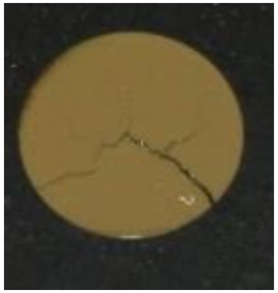


4 studied areas

Preparation

8 cross-sections





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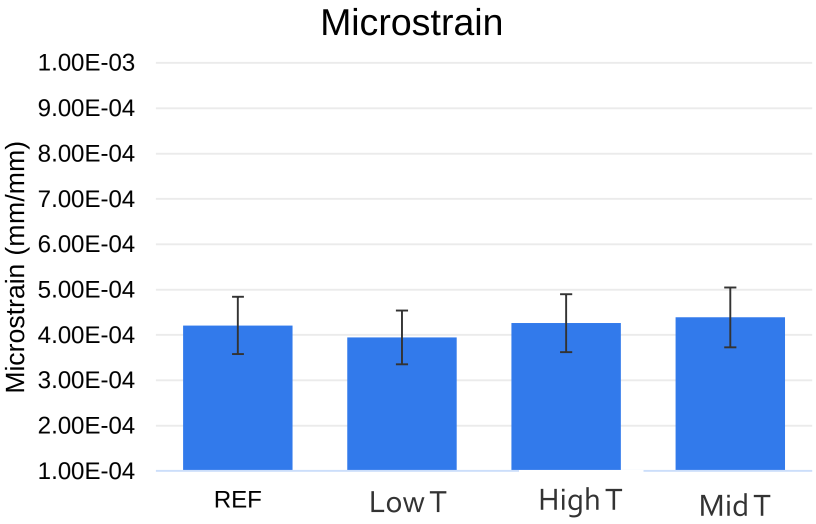
- **Polishing** with diamond suspension (6 µm and 3 µm)
- Final polishing with **colloidal silica suspension** (0.025 µm) + 5% H_2O_2
- **Removal** from the resin

HRMT-27: Ir target

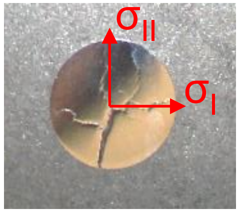
2. Microstructure quantitative analysis

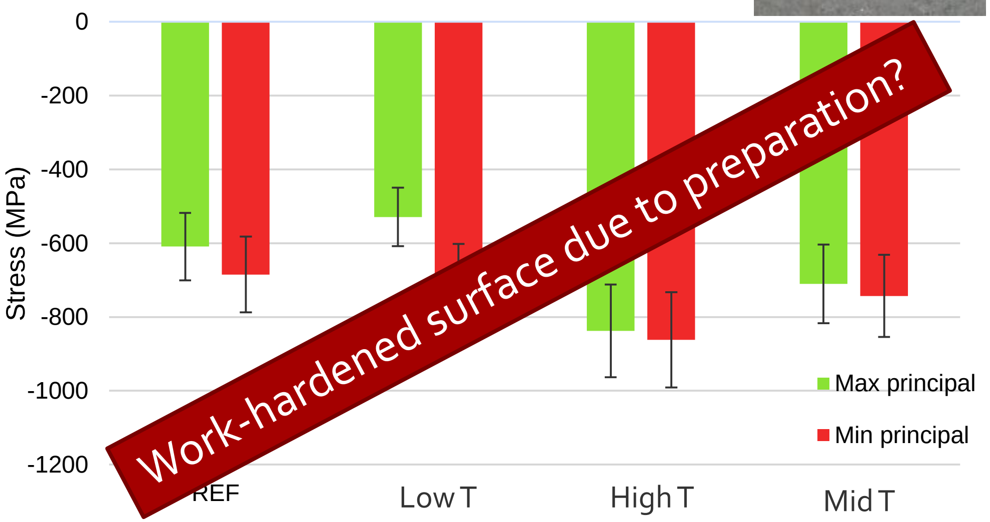
- X-ray diffraction
- Microstructure analysis from peak profile analysis with Rietveld software (MAUD)
- Residual stress measurements in cross-sectional planes
- Samples representative of the three areas: REF, Low T, Mid T and High T
- Measurements probing entire cross-sections

Microstructure analysis



Residual stress measurements



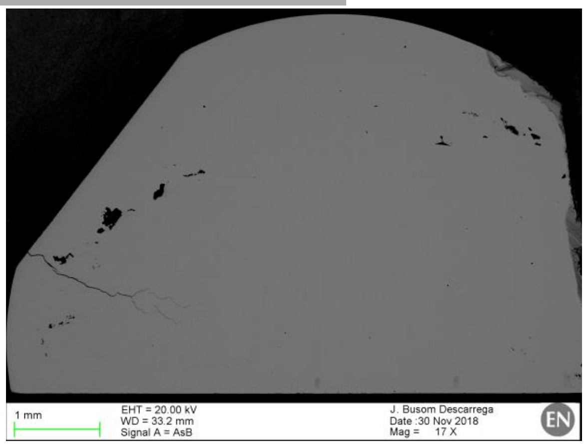


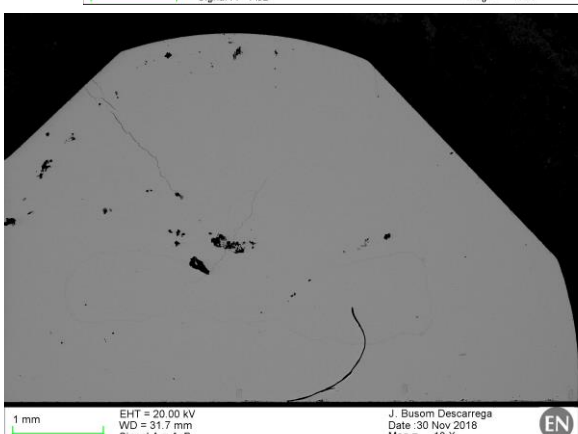
HRMT-27: Ir target

1. Fracture imaging

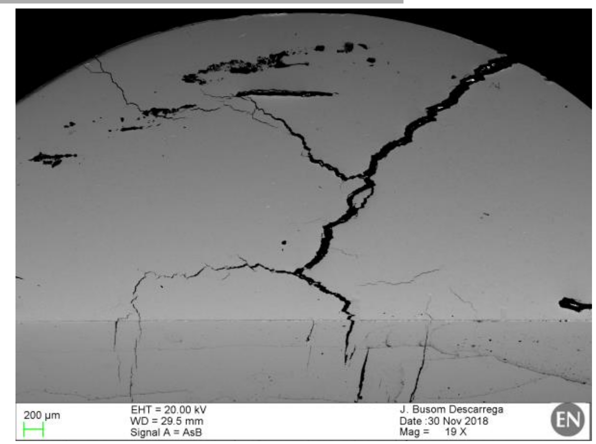
- Scanning Electron Microscope
- Secondary electron imaging

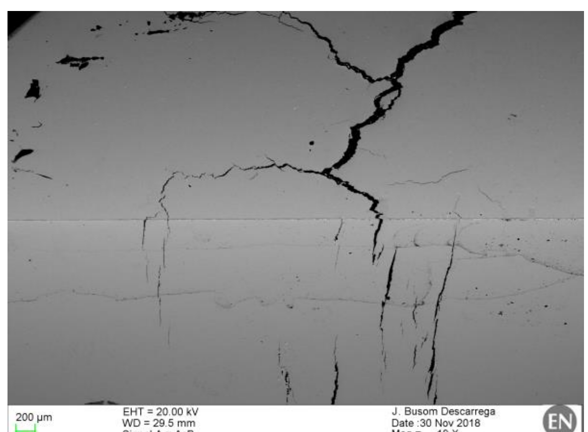
Extremity samples

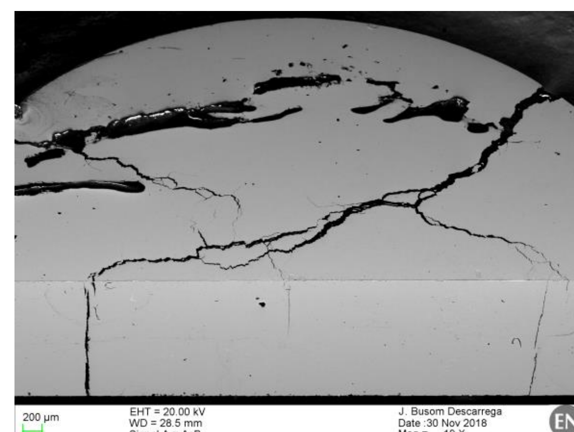




Most solicited region samples

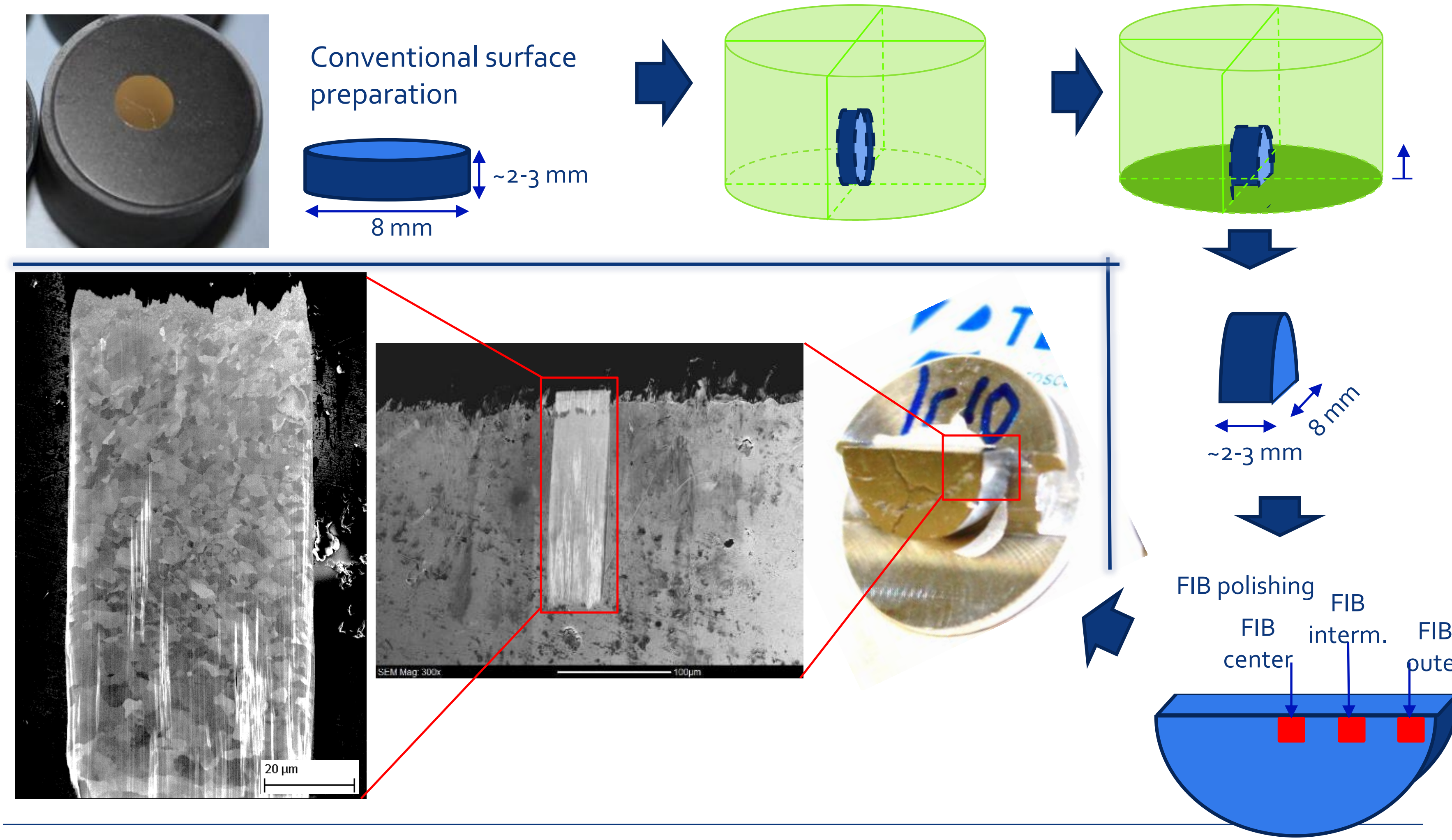






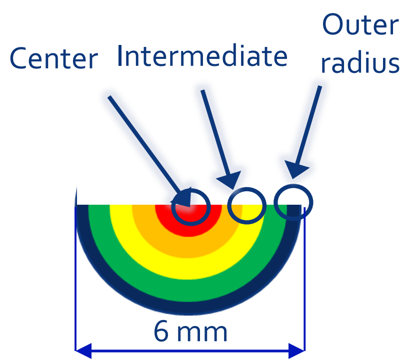
HRMT-27: Ir target

Preparation V2



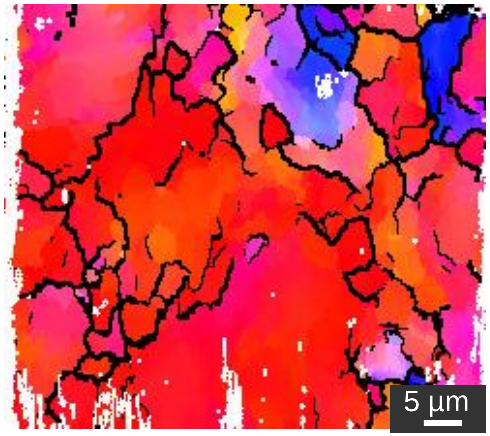
HRMT-27: Ir target

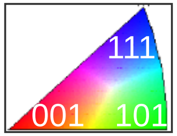
2. Microstructure analysis



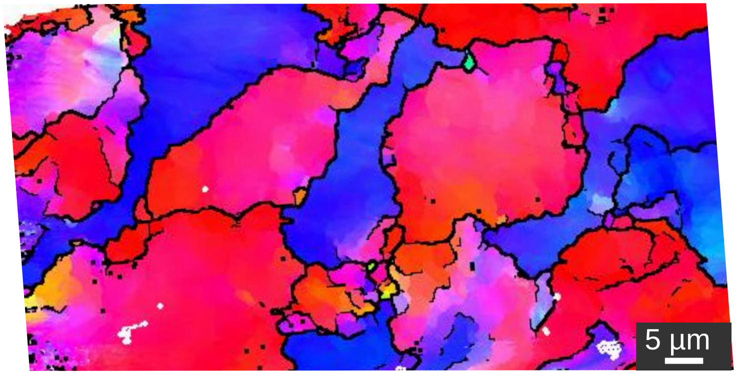
- Scanning Electron Microscope
- **Electron backscattered diffraction**
- Microstructure observable by inverse pole figure mapping
- Microstructure plastic deformation observable by misorientation mapping
- Microstructural damage quantifiable by KAM
- Samples: REF, #1, #2, #5, #6, #8, #9 and #10
- And at three locations: Outer radius, intermediate and center
- > 30 acquired EBSD mappings

Reference sample at center

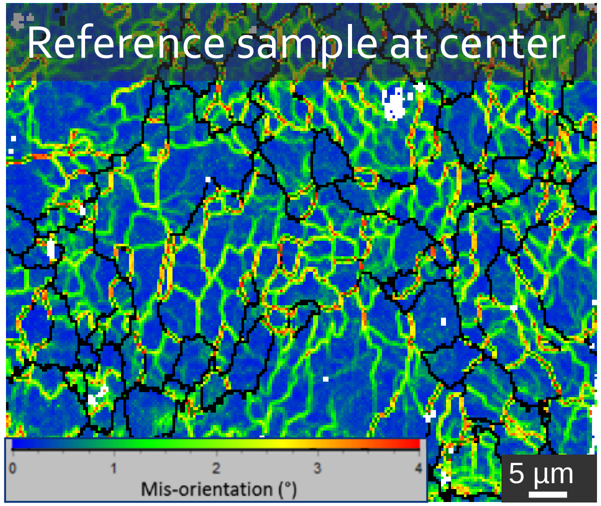


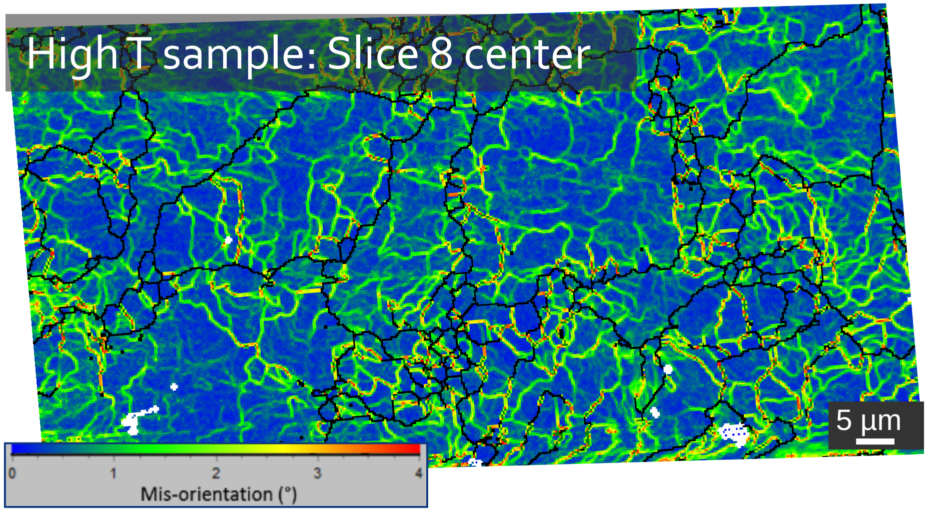


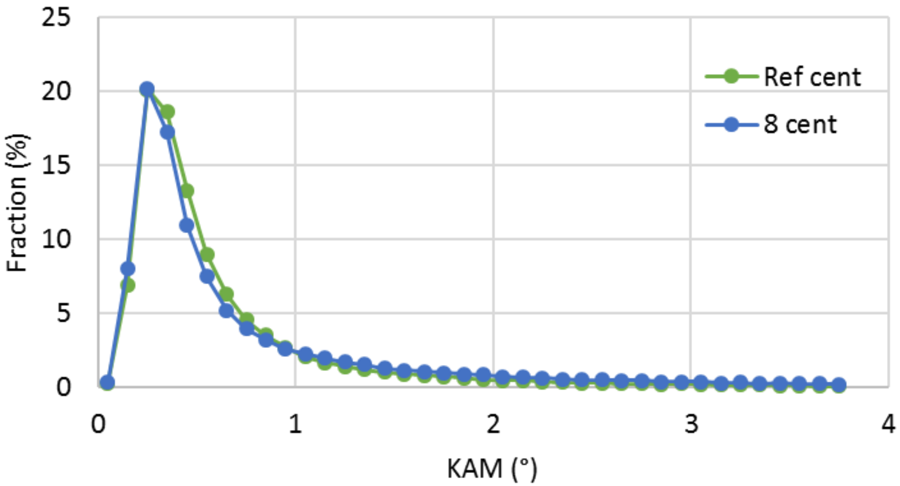
High T sample: Slice 8 center



HRMT-27: Ir target







HRMT-27: Ir target

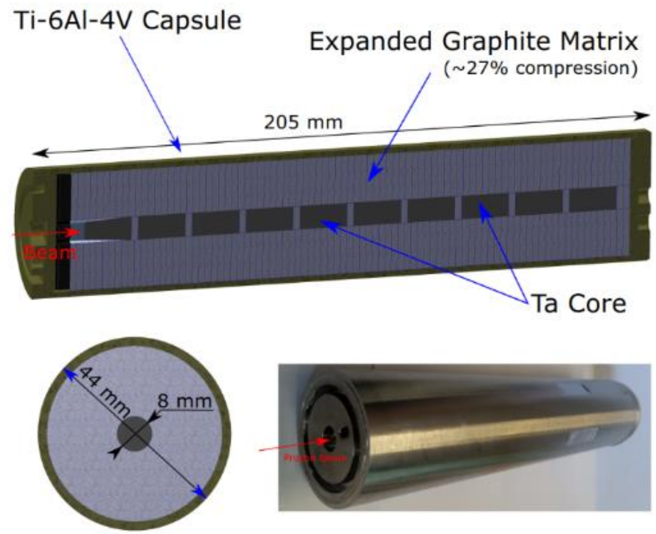
- No observable microstructure differences before and after thermal shock
- Iridium “brittle” response
- Cracking along rod axis
- Next steps: fractography

Fracture behavior at high strain rates in unirradiated Ir

Scapin, Martina, et al., *International Journal of Impact Engineering* 106 (2017): 191-201.

- Original microstructure already deformed due to forming
- Unknown detailed forming process

HRMT-42: Target setup



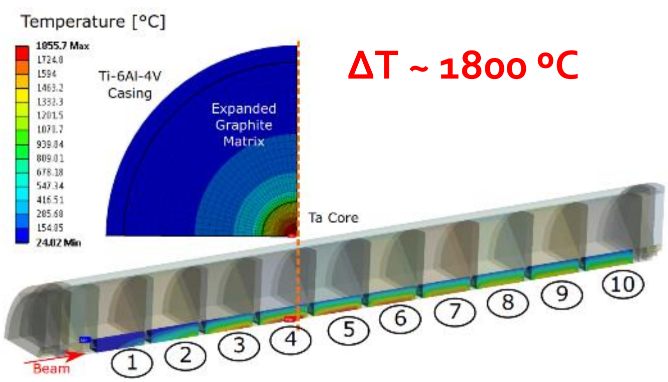
Scaled Prototype

- Core of 8 mm diameter Ta rods (un-annealed)
- Embedded in a matrix made of compressed layers of **Expanded Graphite (EG).**
- Encapsulated in Ti-6V-4Al e-beam welded container

Conditions reached during the Experiment

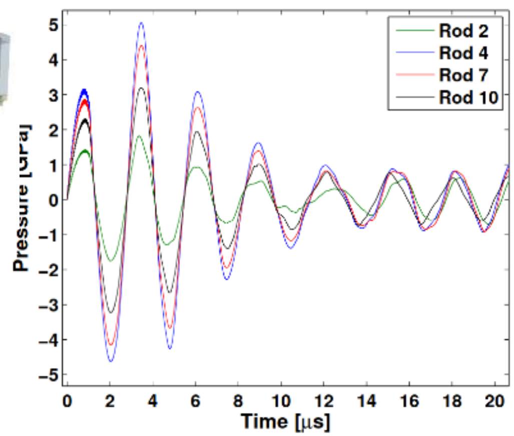
- Same beam parameters as in HRMT-27

#47 pulses were impacted (7·10[13] POT)



ΔT ~ 1800 °C

Max pressures reached:
~5 GPa (compression)
~4.6 GPa (tension)



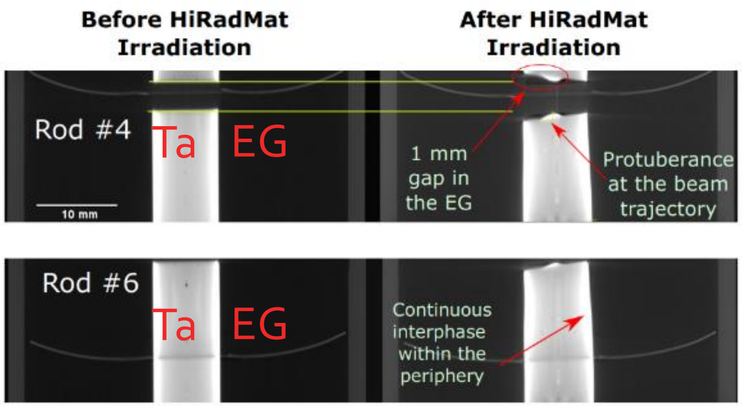
Accumulative heating

- Max temperature reached estimated in **2650 °C**
- Estimated accumulative time above 1600 °C = **14 s**
- Estimated accumulative time above 1000 °C = **5 mins**
- Estimated accumulative time above 800 °C = **15 mins**

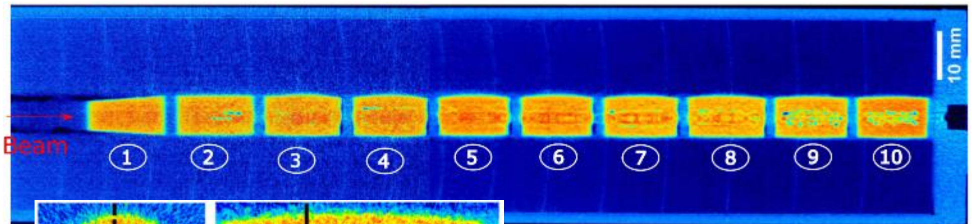
Courtesy of C. Torregrosa

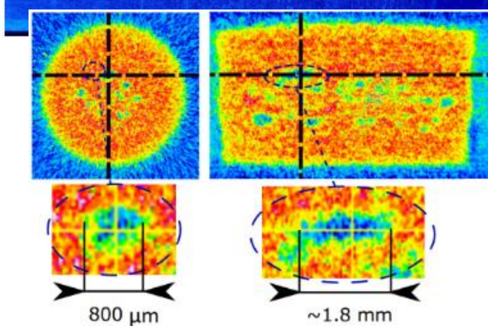
HRMT-42: Target PIE

X-ray tomography, ESRF (Grenoble)

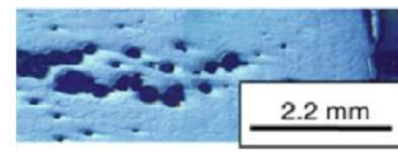


Neutron tomography Neutra-PSI (Zurich)





Spalling?



Gray, G. T. et al., (2010). *International Journal of Fracture*, *163*(1-2), 243-258.

Target Opening carried out at CERN

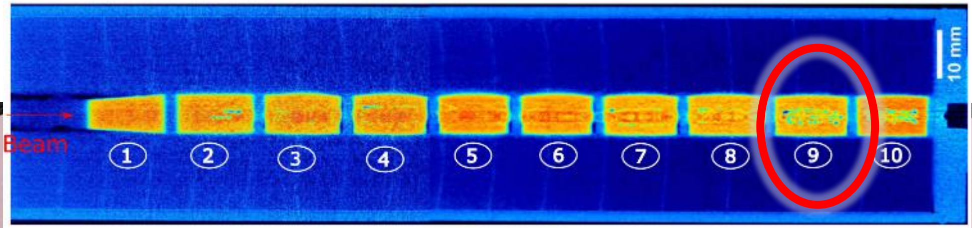
Metrology at CERN

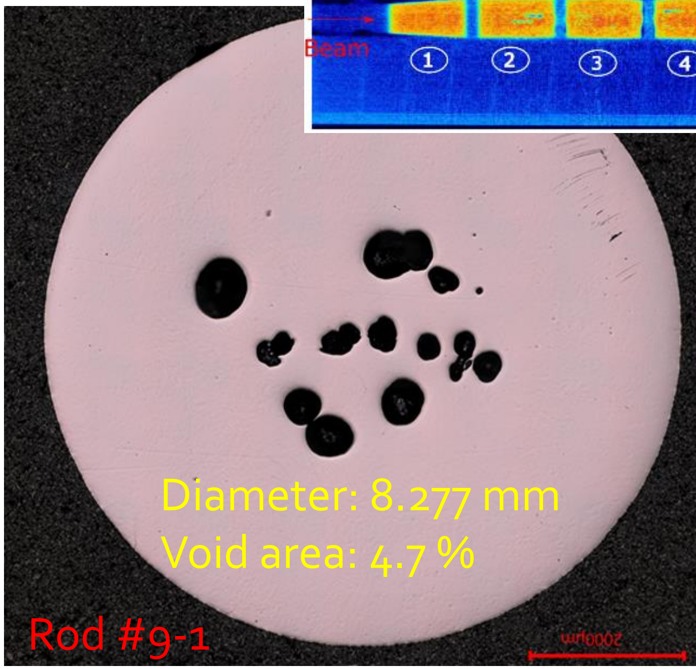
- Swelling
- Striates

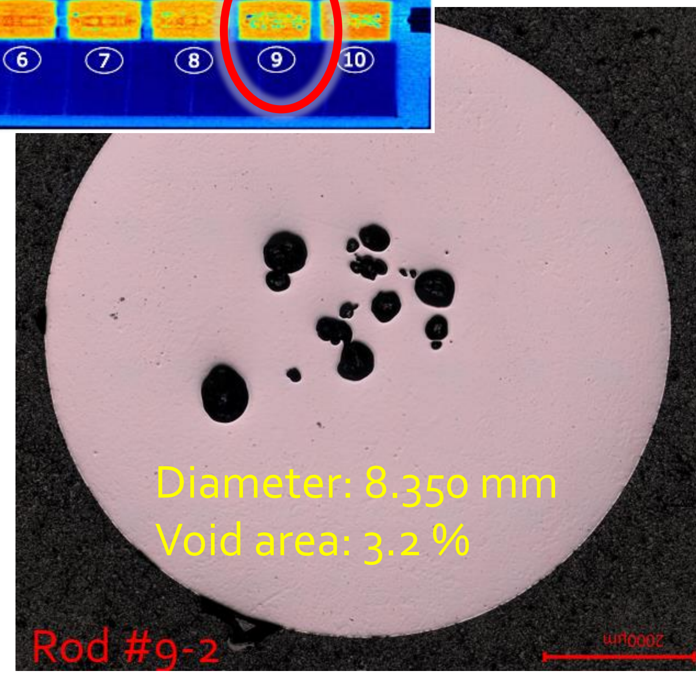
Courtesy of C. Torregrosa and E. Fornasiere

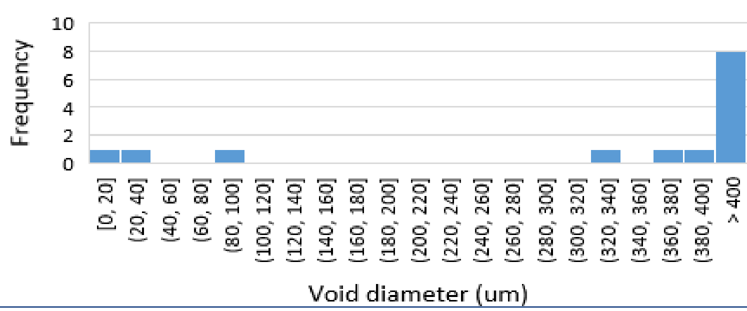
HRMT-42: Ta target

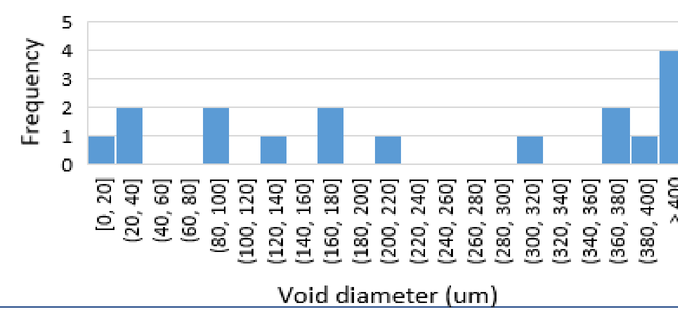
Optical microscopy





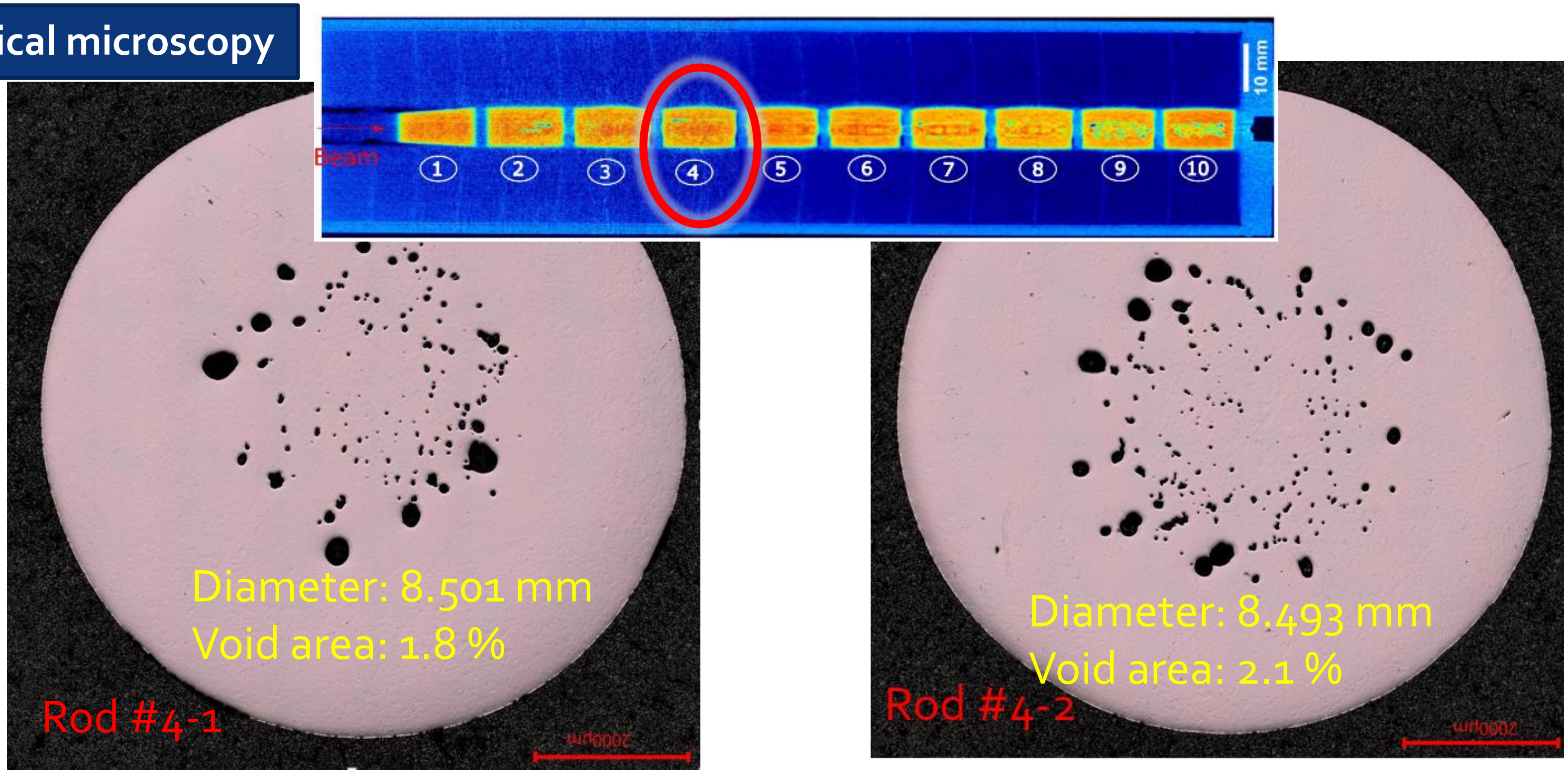


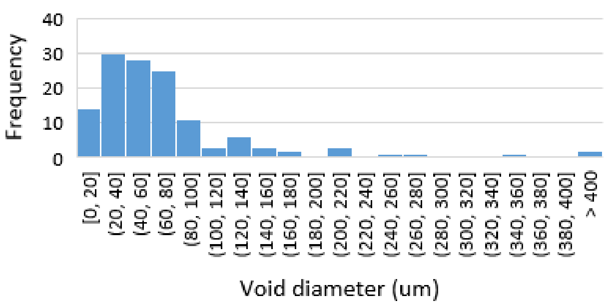


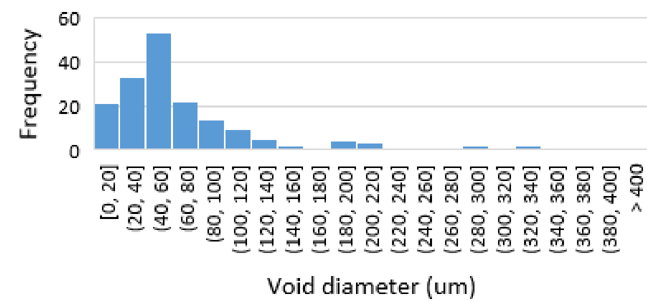


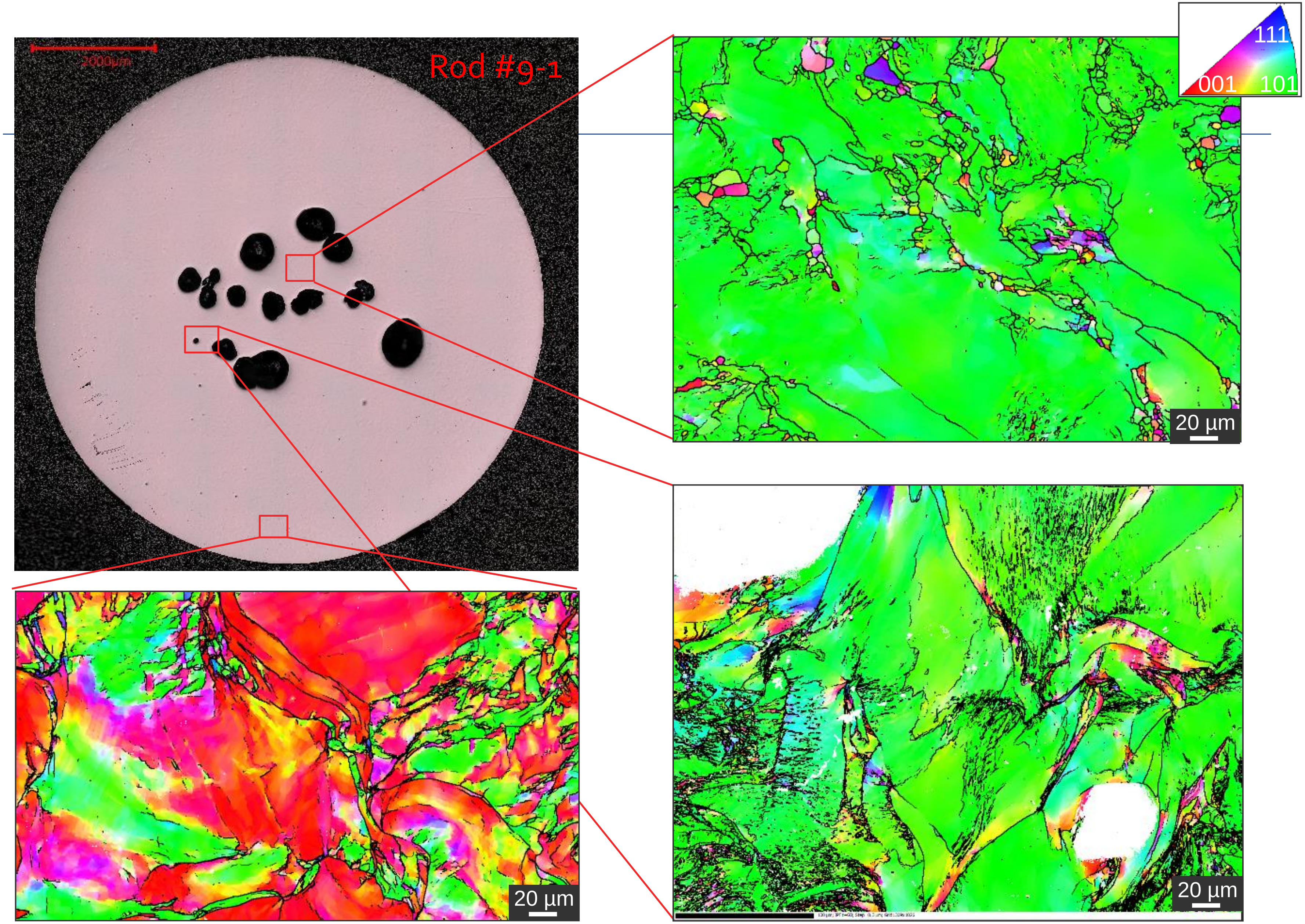
HRMT-42: Ta target

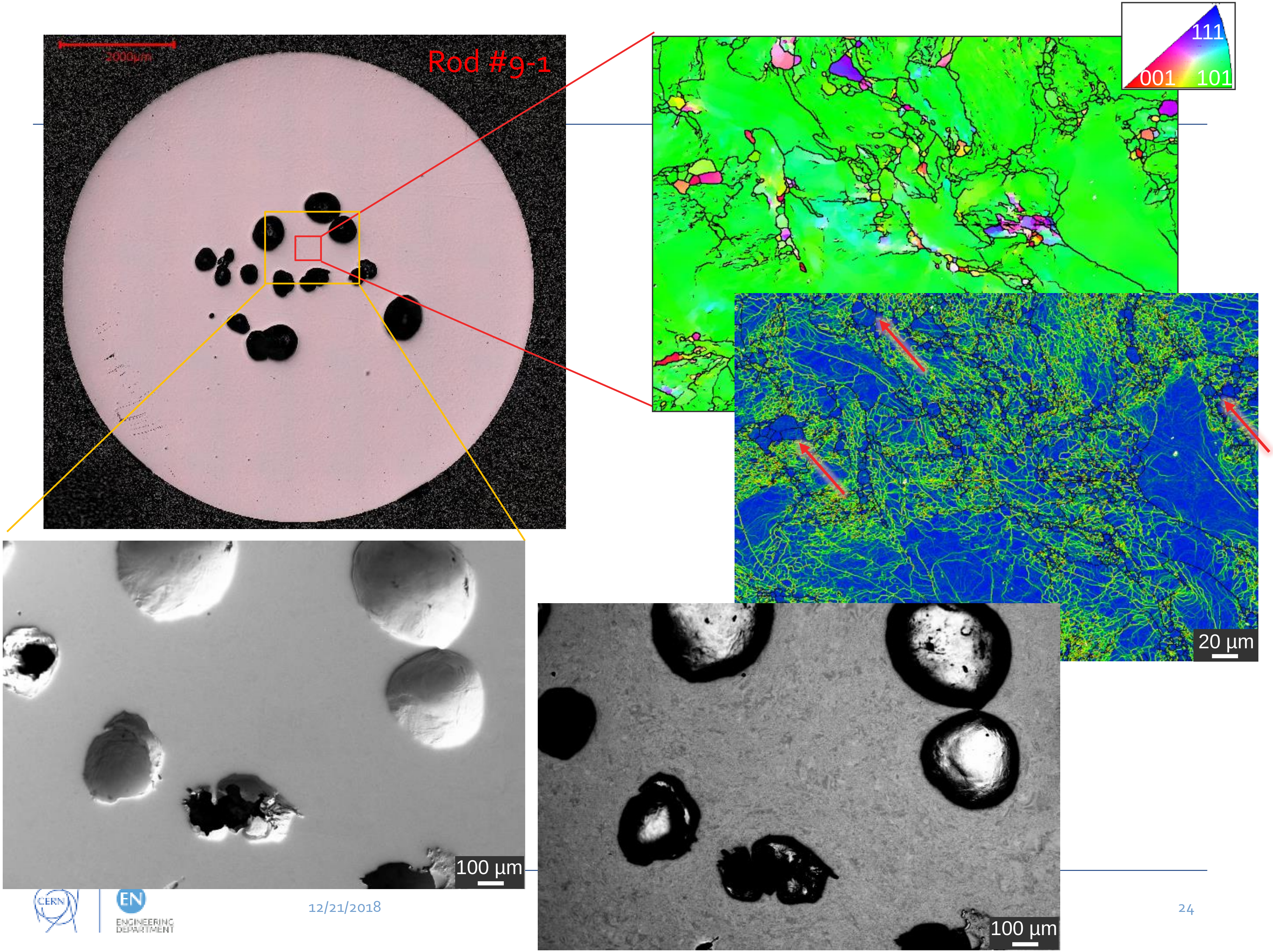
Optical microscopy

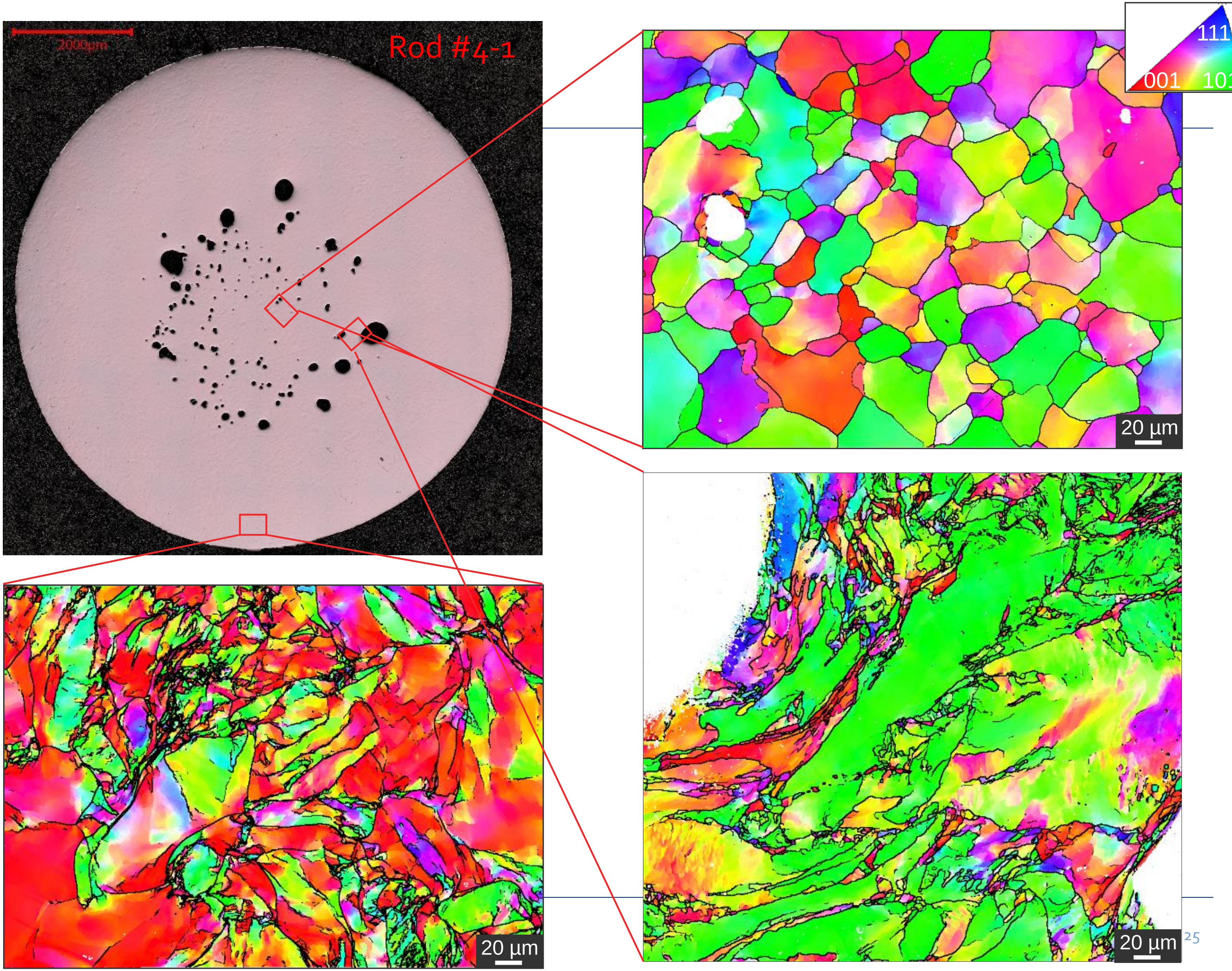


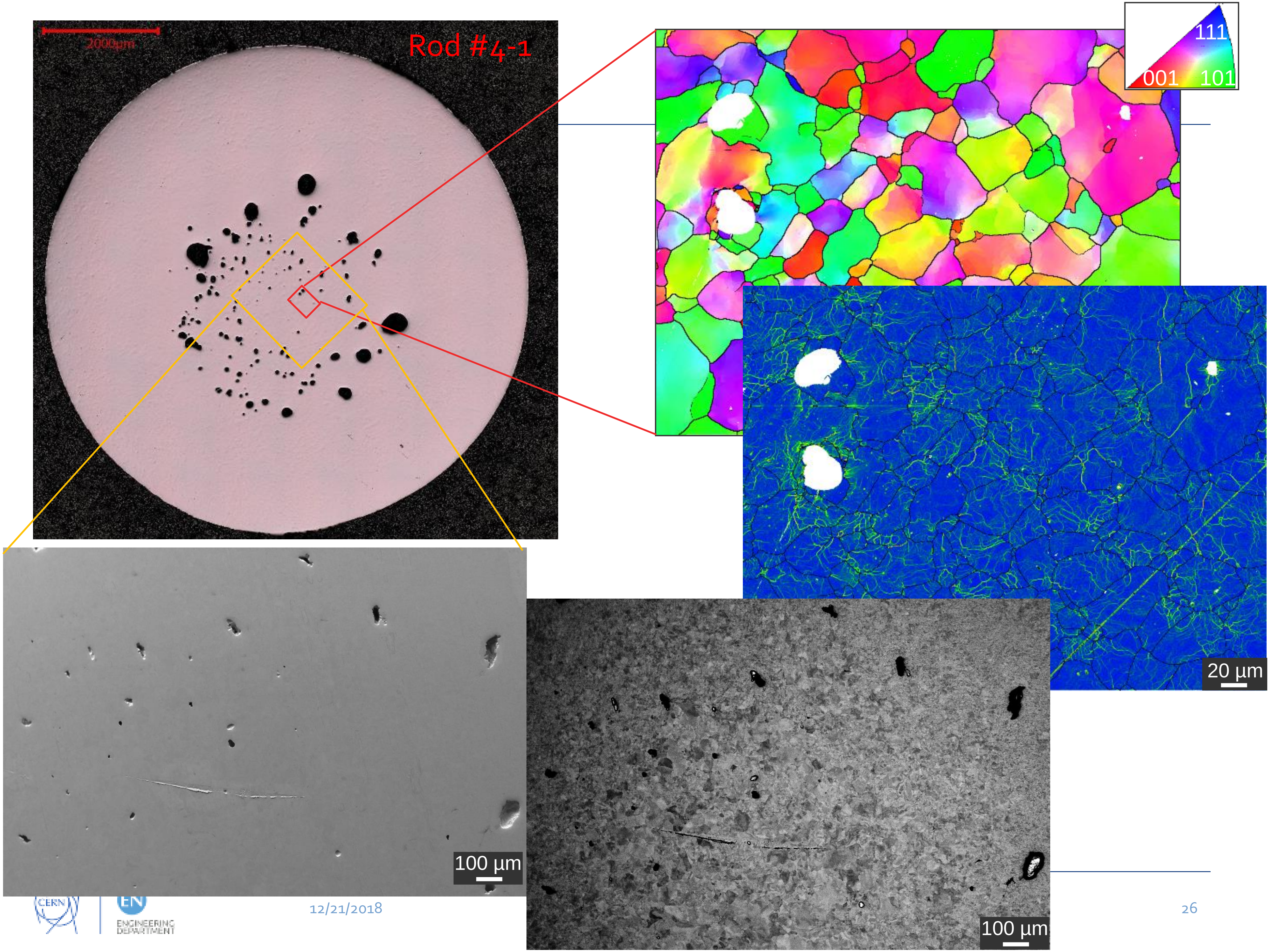





2000µm
Rod #9-1
111
001
101
20 µm
20 µm
20 µm

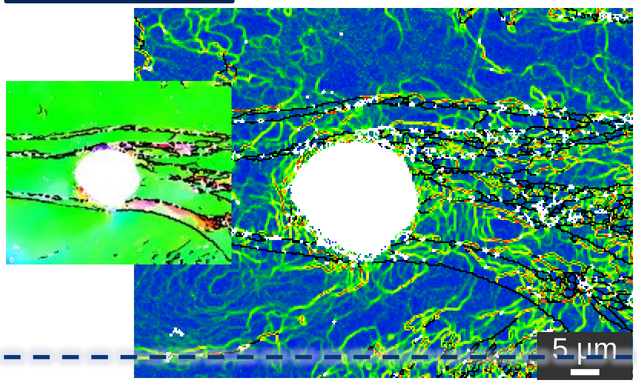

2000μm
Rod #9-1
111
001
101
20 μm
100 μm
100 μm


2000µm
Rod #4-1
111
001
101
20 µm
20 µm
20 µm

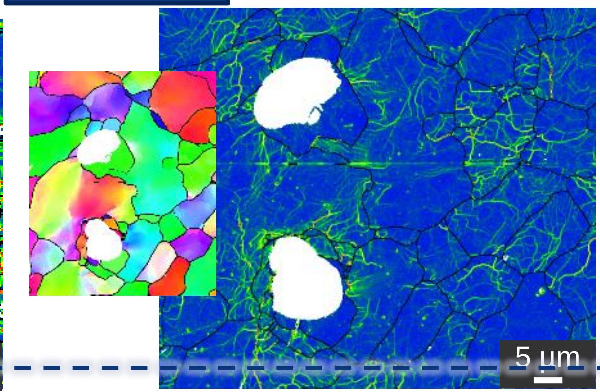

2000µm
Rod #4-1
111
001
101
20 µm
100 µm
100 µm

HRMT-42: Ta target

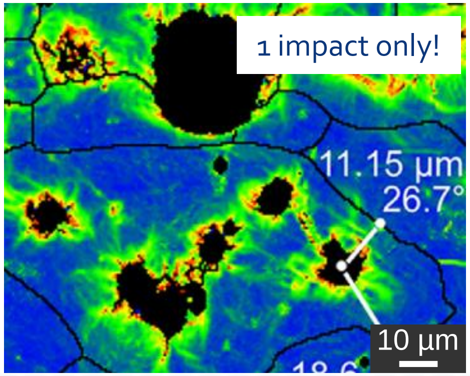
Rod #9-2



Rod #4-1

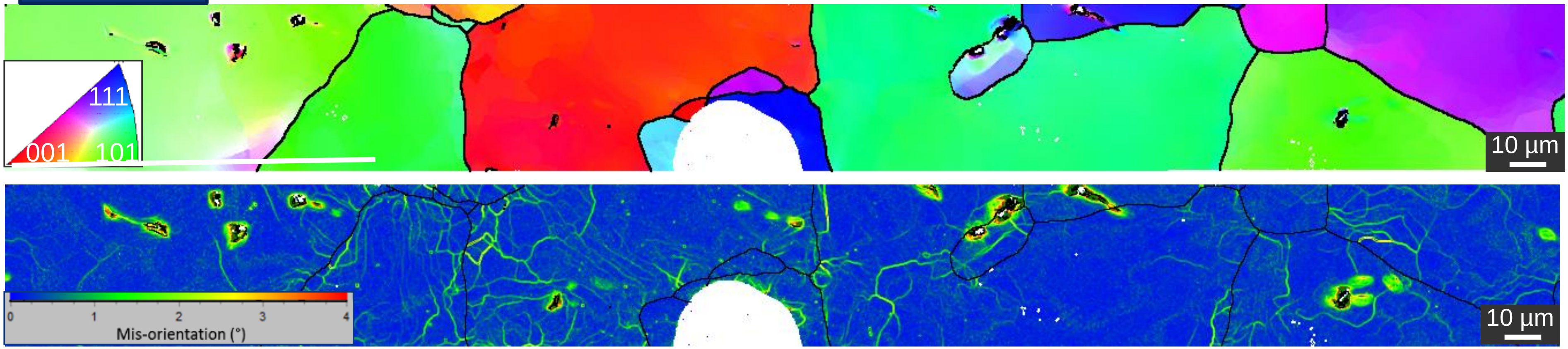


Ta spalling at RT



Cheng, M., et al. *Acta Materialia*148 (2018): 38-48.

Rod #4-2

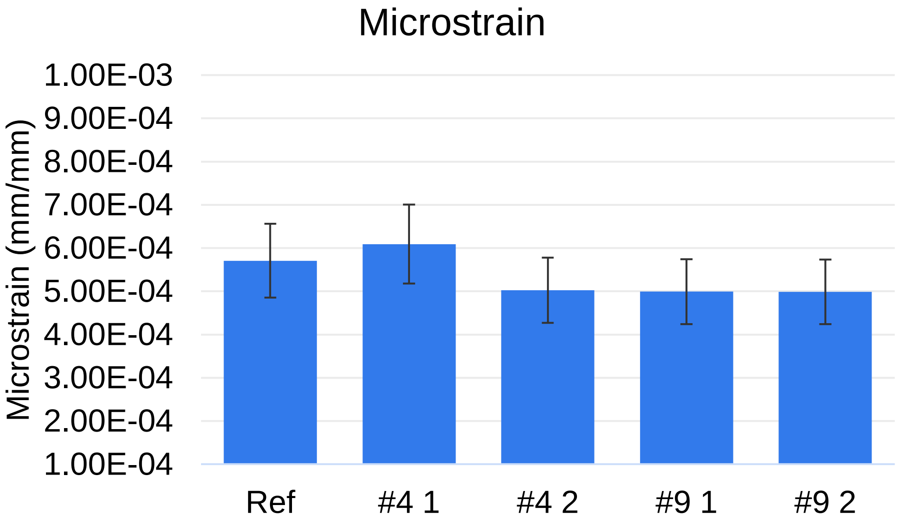


HRMT-42: Ta target

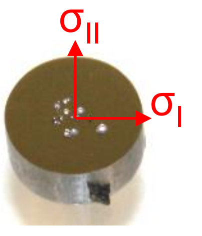
3. Microstructure quantitative analysis

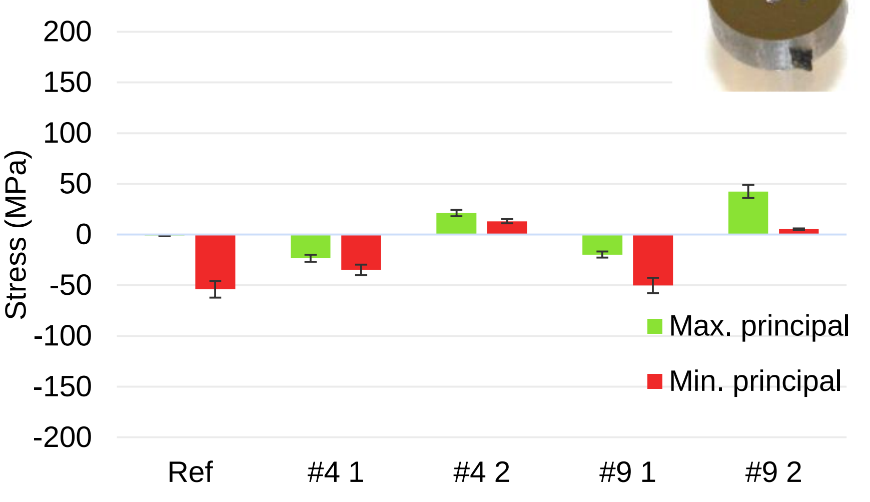
- X-ray diffraction
- Microstructure analysis from peak profile analysis with Rietveld software (MAUD)
- Residual stress measurements in cross-sectional planes
- All samples were measured
- Measurements probing entire cross-sections

Microstructure analysis



Residual stress measurements

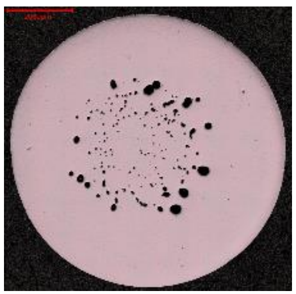


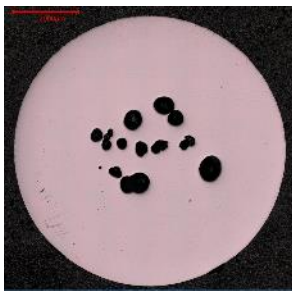


HRMT-42: Remarks

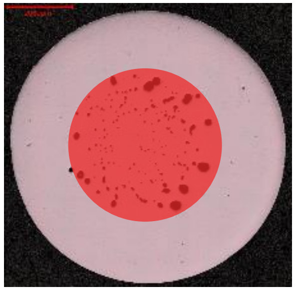
- Many questions to answer, which require simulations – PIE – literature precise correlation:

Void size and distribution



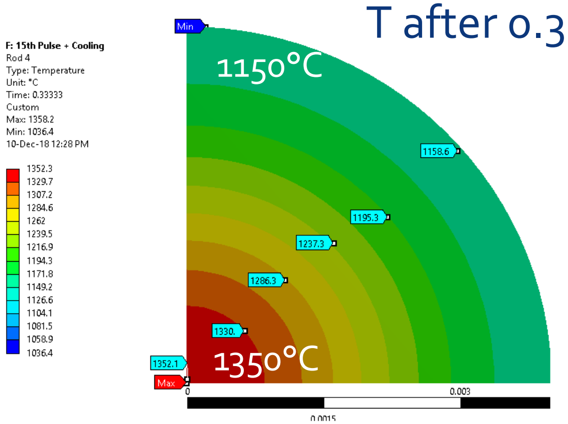


Void positioning



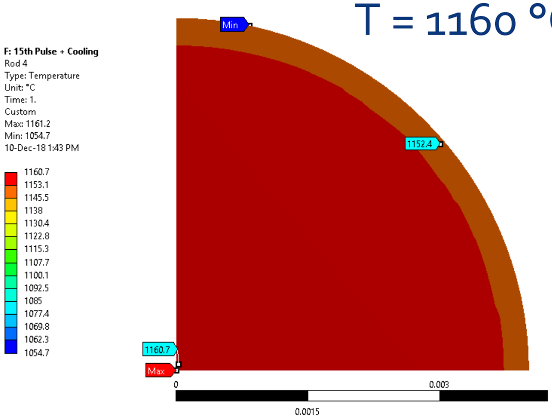
Recrystallization

T after 0.3 s



After 1 s

T = 1160 °C

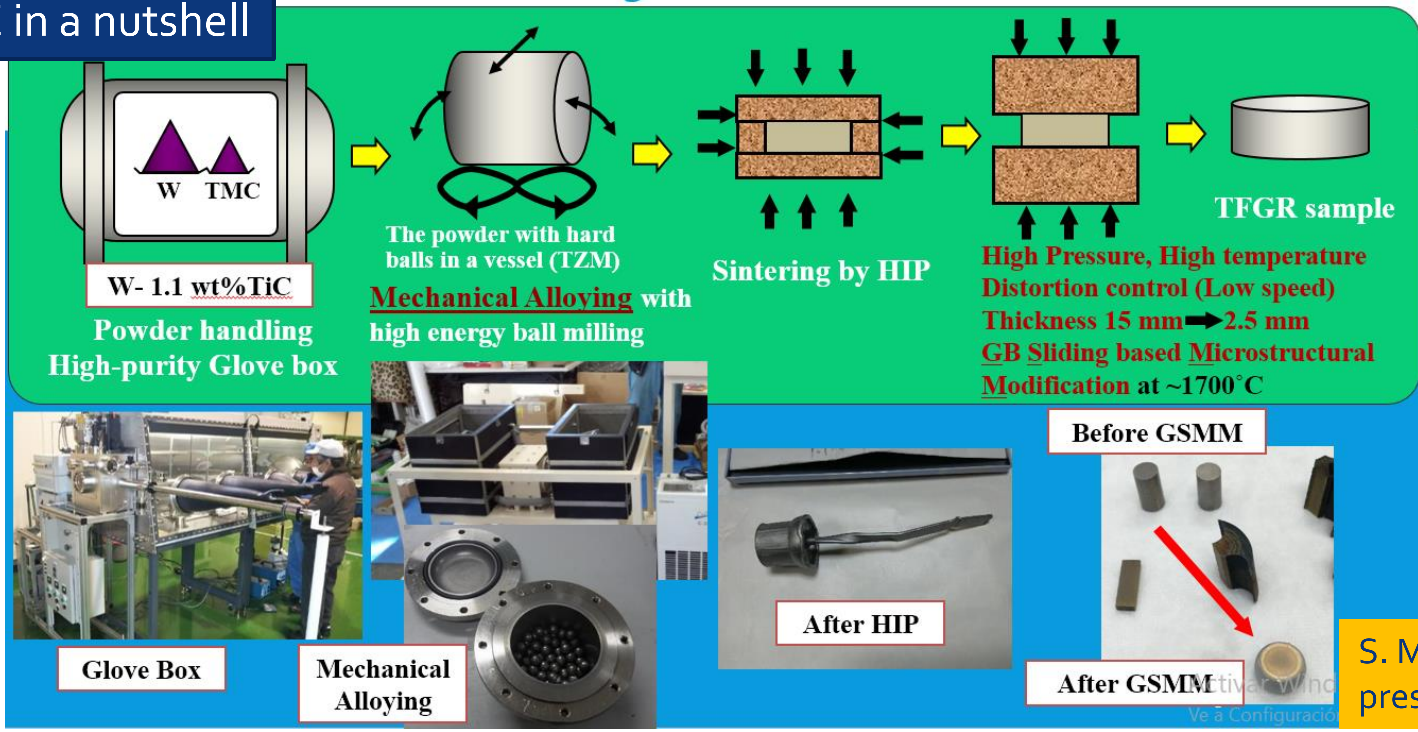


Conclusions

- Several irradiated rods from HRMT-27 and HRMT-42 have been studied
- Sample preparation procedures established and optimized
- Microstructural properties available at different scales
- Cracking and spalling phenomena directly observed
- Recrystallisation process observed in Tantalum
- Discussion on going to understand phenomena and application to AD target design

Bonus track: W-TiC for PROTAD

W-TiC in a nutshell

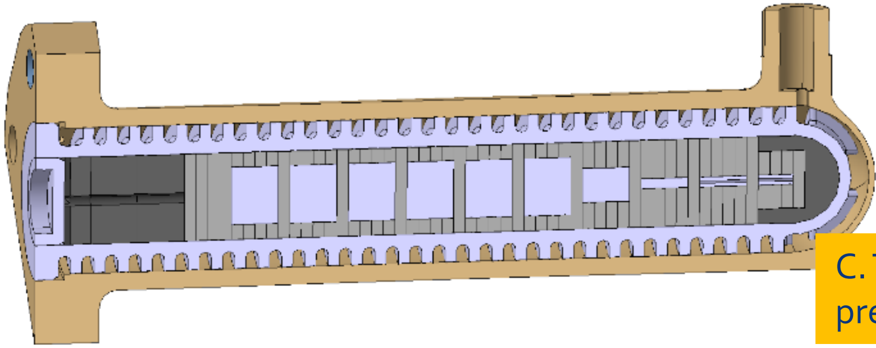


S. Makimura presentation

W-TiC in HiRadMat

PROTAD Target 5:

Core: Ø 10 mm Ta +
Ø 10 mm W + **W-1.1TiC** + Ø 10 mm Ir
+Ø 2 mm Ta tube
Matrix: Compressed EG

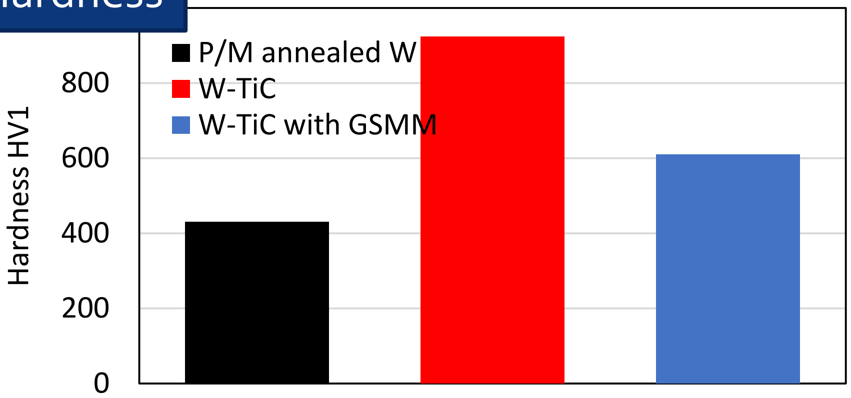


C. Torregrosa presentation

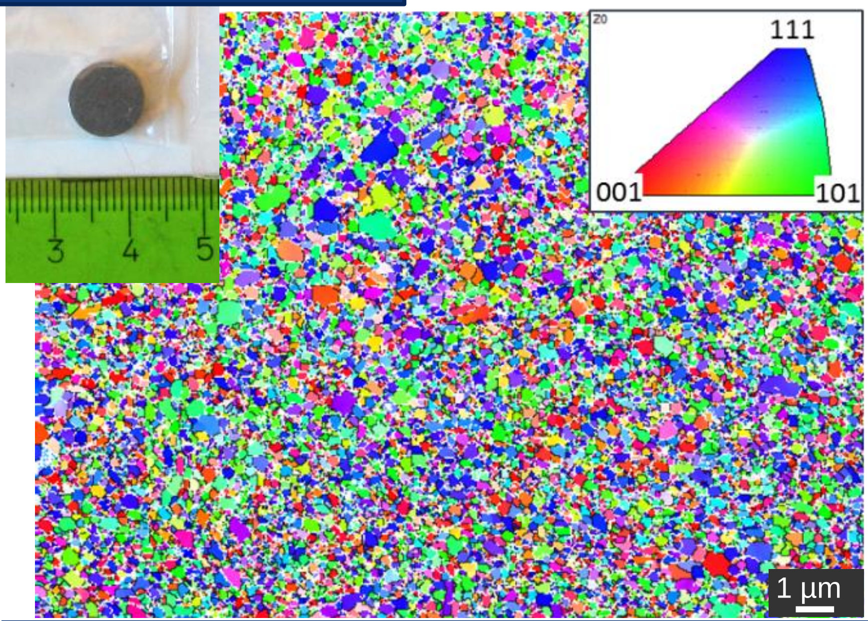
Bonus track: W-TiC for PROTAD

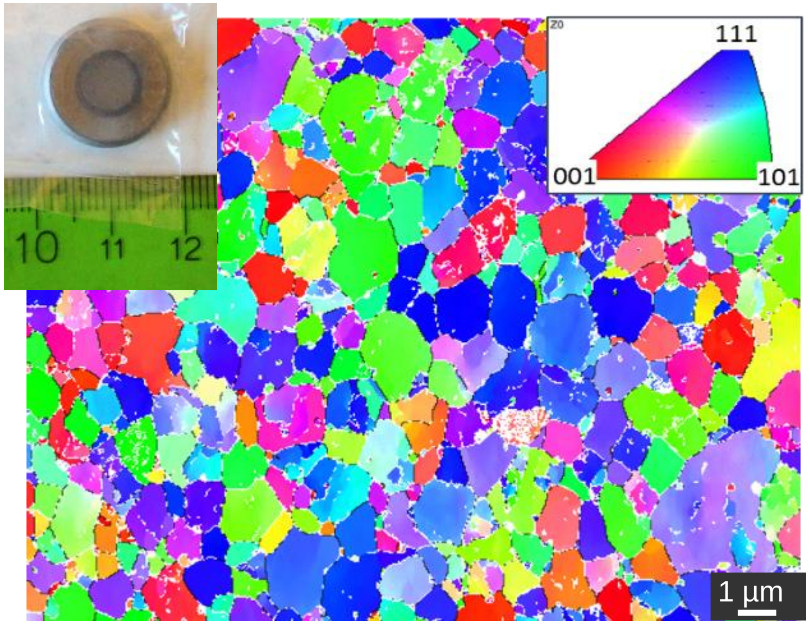
- Two spare W-TiC samples for microstructure characterization
- One sample after GSMM and one sample before GSMM

Hardness



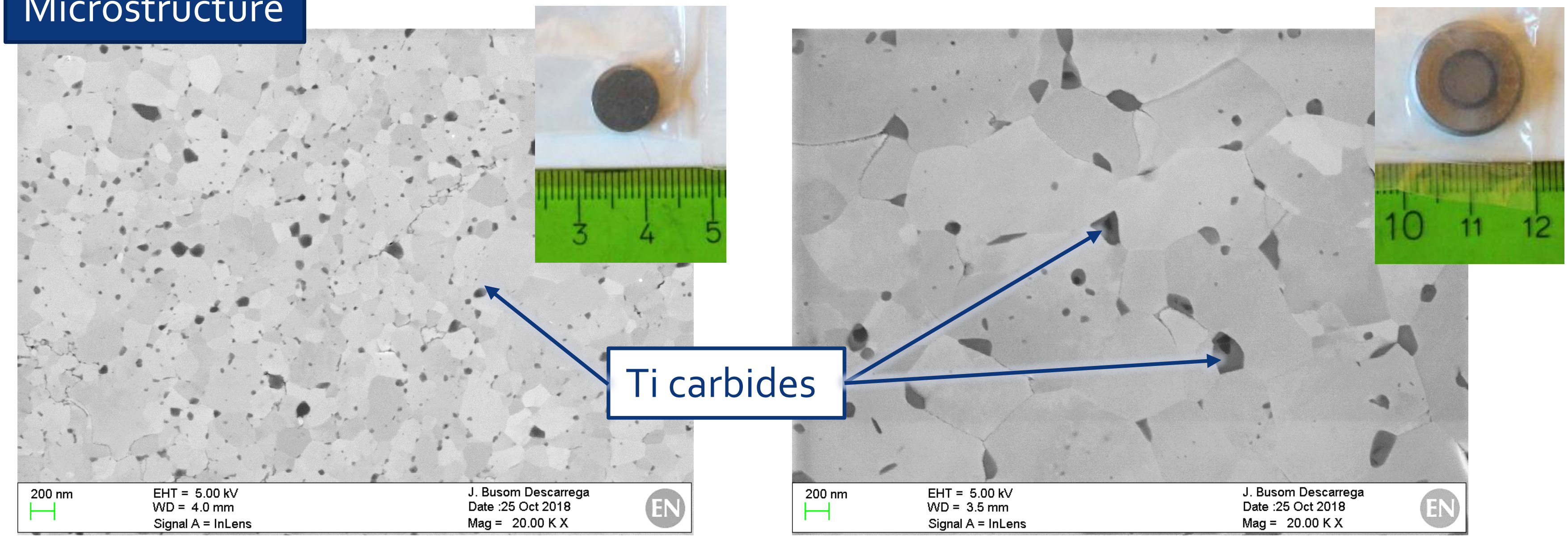
Microstructure- IPF



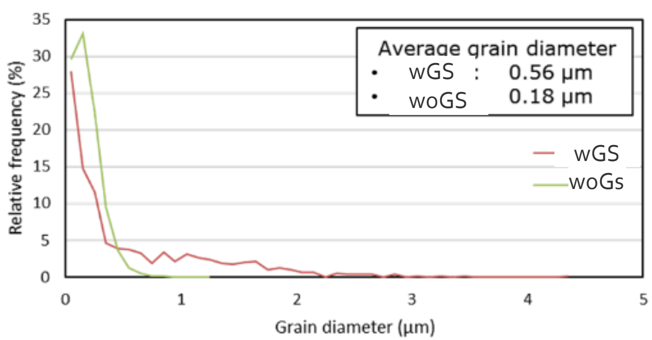


Bonus track: W-TiC for PROTAD

Microstructure



Grain size distribution



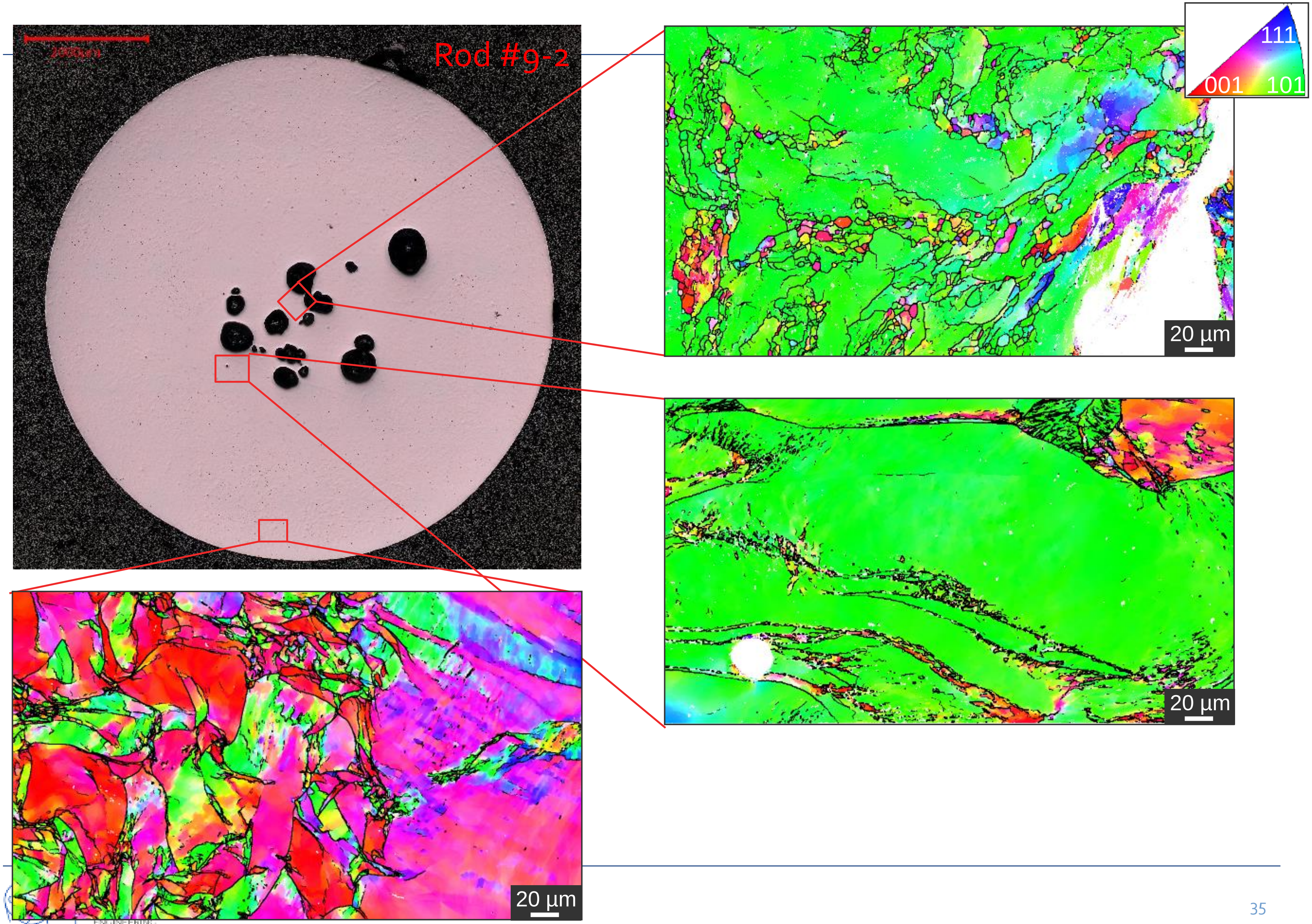
- Grain size increase with GSMM
- Coherent hardness decrease with GSMM
- Carbide growth with GSMM only at GBs
- Equiaxial and microstructure
- No grain preferential orientations

Thanks for your attention. Questions?

CERN

ENGINEERING DEPARTMENT

Spare slide: HRMT-42 Rod #9-2



Spare slide: HRMT-42 Rod #4-2