Evaluation of metallurgical risk factors in post-test, advanced 9%Cr creep strength enhanced ferritic (CSEF) steel

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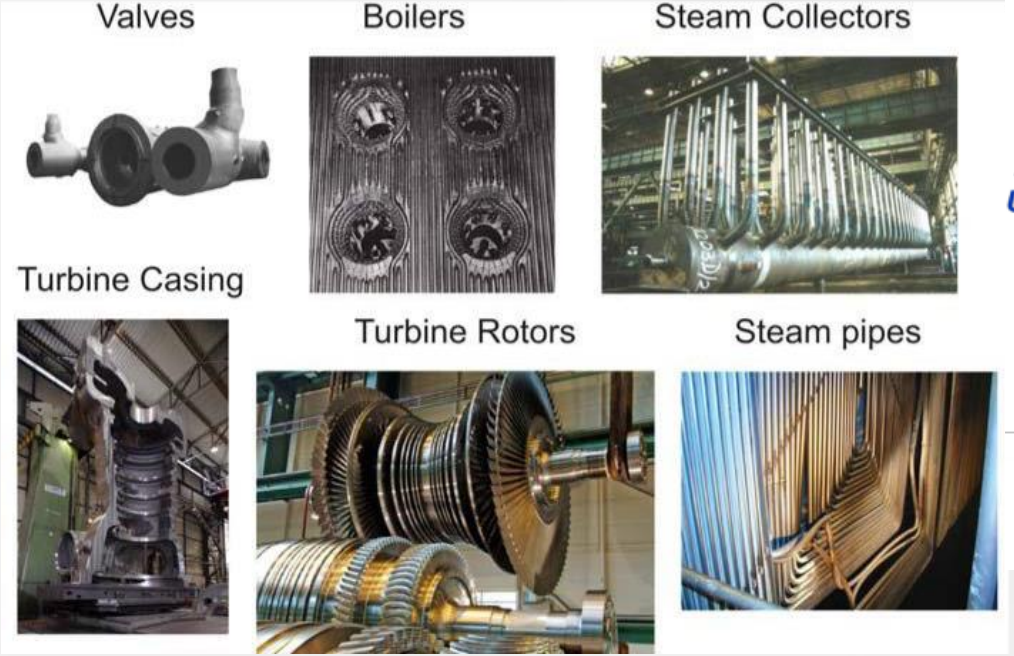
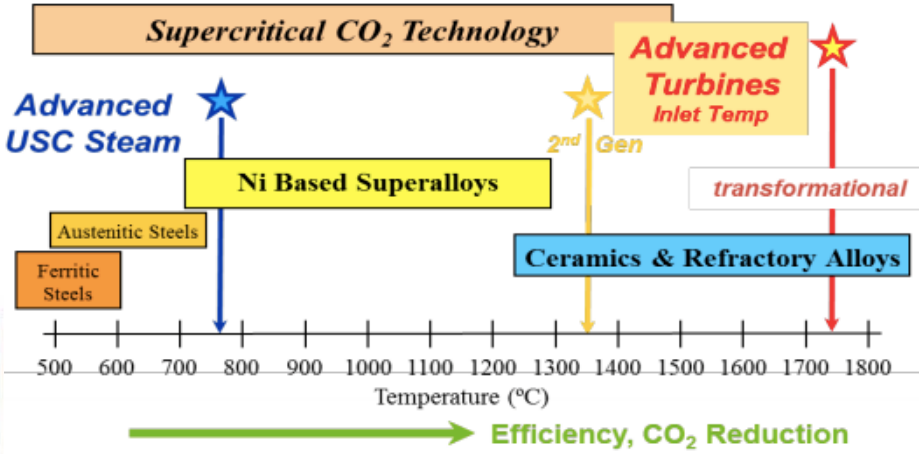
- Introduction
- Typical phases in 9Cr steels
- Ta enriched particles identification and quantification in a novel 9Cr steel (CPJ7)
- PFIB serial sectioning 3D reconstruction of microstructure of CPJ7 steel
- Conclusions and future work

Introduction



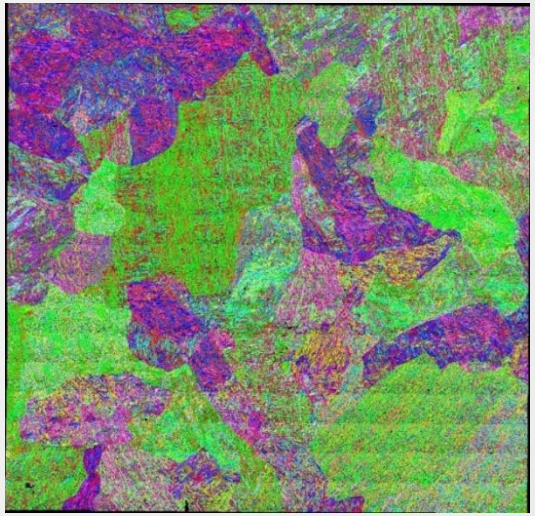
Applications of 9–12 wt. % Cr steels in fossil fuel power stations



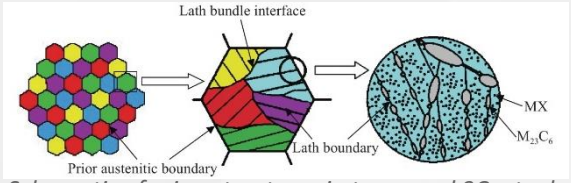
Evolution of power plant component steels as a function of temperature

F. Masuyama. "Development History and New Generation of Creep Strength Enhanced Ferritic Steels." *ETD Conference Fabrication and use of P91 Steel: International Industry and Plant Experience Conference.* New Castle, Australia, October 11-12, 2017.

Martensitic Steel Development and Microstructure

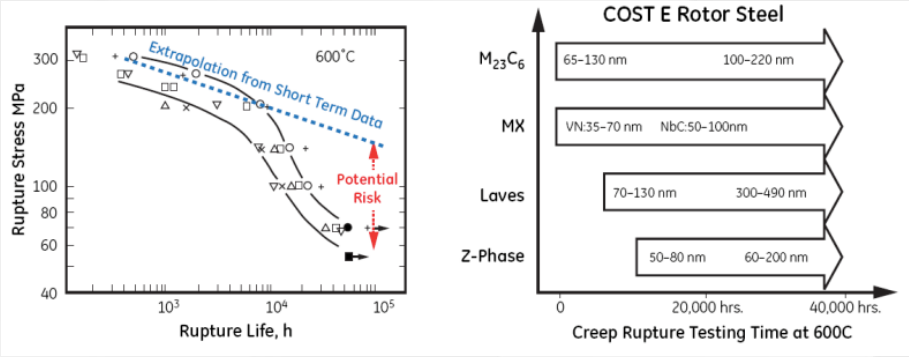


EBSD montage map of an as-cast 9Cr steel shows prior austenite grains and martensite laths



Schematic of microstructures in tempered 9Cr steels

- 1950s to date – Low alloy creep resisting steels
  - 2¼CrMo; CrMoV
    - Applications up to about 540 - 570°C (maximum)
- 1980s development – P91 or “Modified 9Cr-1Mo” steel
  - Applications generally up to about 580°C (or higher if at low stress)
- 1990 - 2000 – P92 steel
  - Applications – e.g., 600°C main steam, 620°C hot steam reheat



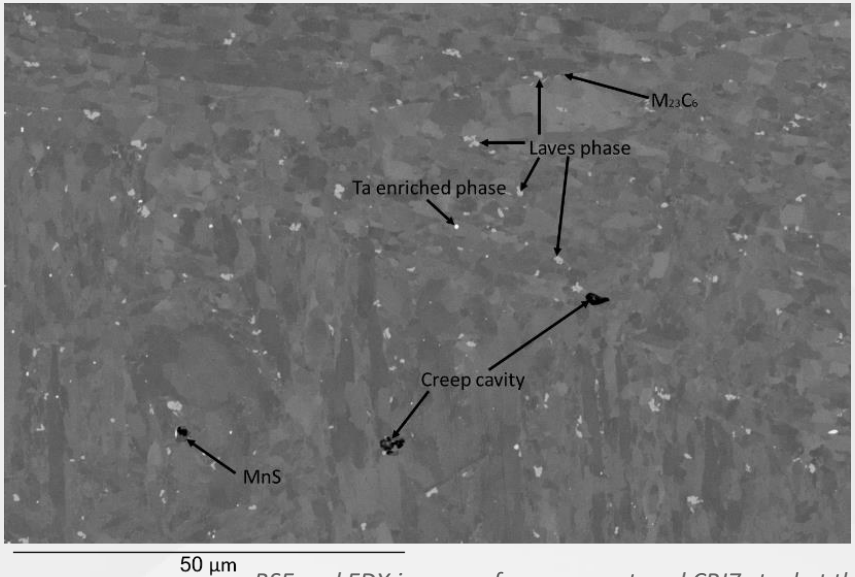
Z-phase, Laves phase, MX and/or $M_{23}C_6$, can cause an unexpected decrease in rupture stress as a function of time.

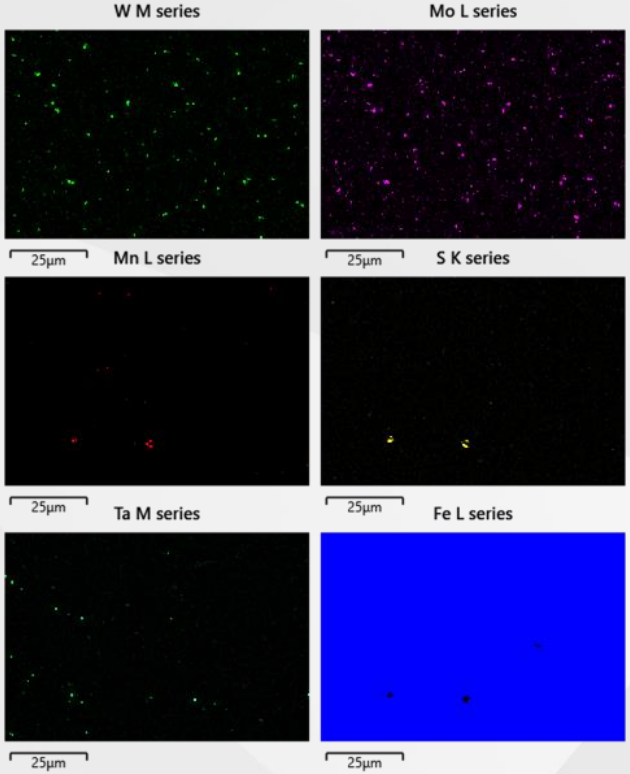
J.A. Hawk. “Ferritic-Martensitic 9% Cr Steels for Steam Turbine Applications.” *Parsons 2019, Cranfield University, UK, September 16-18, 2019*

Microstructure Overview of A Novel 9Cr Steel

	Cr	Mo	C	Mn	Si	Ni	V
Minimum	9.75	1.0	0.13	0.25	0.08	0.15	0.15
Maximum	10.25	1.5	0.17	0.50	0.15	0.30	0.25
	Nb	**N**	**W**	**Co**	**Ta**	**Cu**	**B**
Minimum	0.05	0.015	0.25	1.35	0.20	0.003	0.0070
Maximum	0.08	0.035	0.75	1.65	0.30	0.30	0.0110

Chemical composition of CPJ7 steel with Fe balanced in weight percentage (wt%)

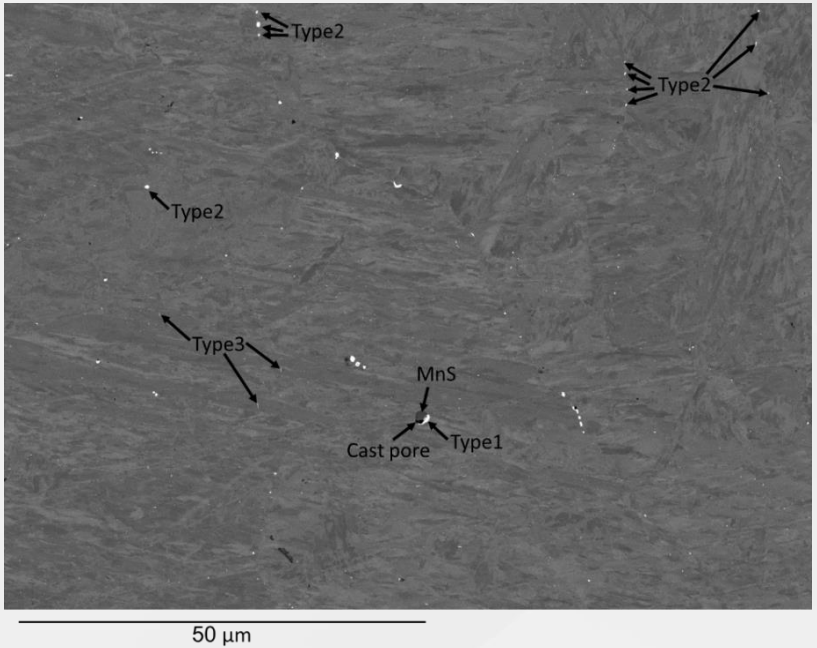




BSE and EDX images of a creep ruptured CPJ7 steel at the gauge section

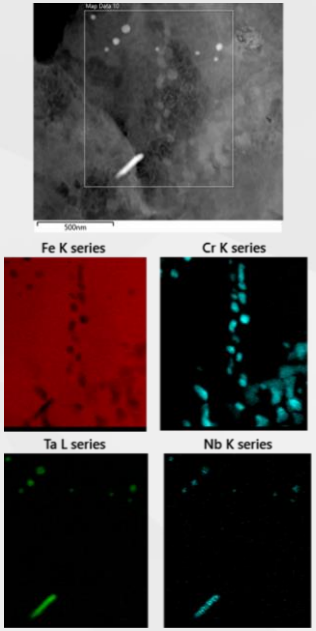
J.A. Hawk. "Ferritic-Martensitic 9% Cr Steels for Steam Turbine Applications." *Parsons 2019, Cranfield University, UK, September 16-18, 2019*

Ta-enriched Particles in CPJ7 Steel

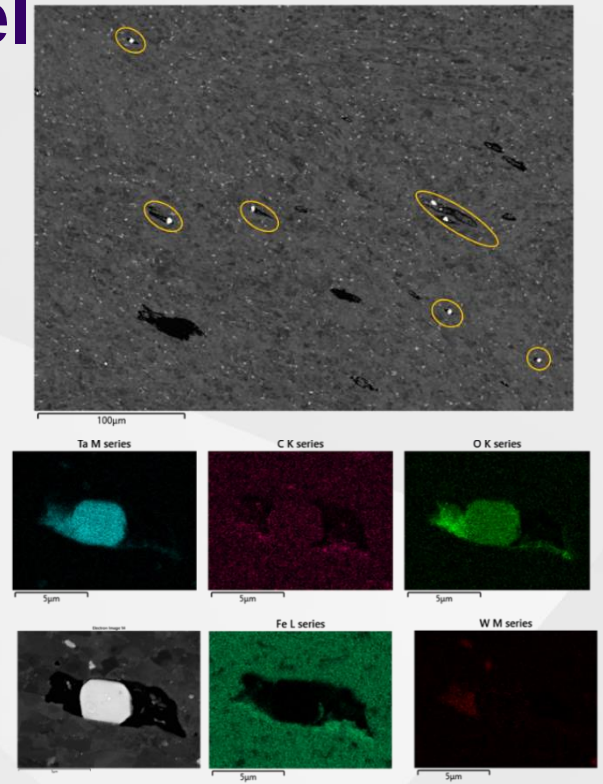


BSE-SEM micrograph shows Ta enriched particles in the as-receive material

- **Type 1: Ta_2O_5, ECD>1µm, associated with pores /MnS particles**
- **Type 2: TaC, ECD 0.1-1µm, along PAGs / in the matrix**
- **Type 3: TaC, rod shape, ~50nm in diameter 0.5-2µm in length**

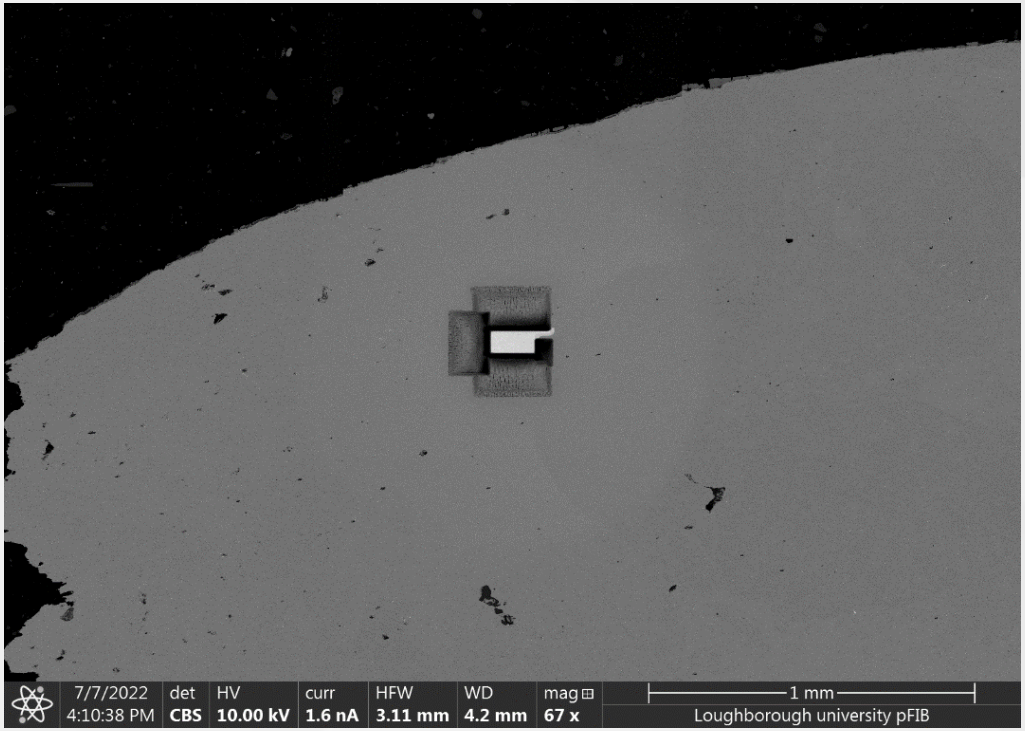


TEM high angle annular dark field (HAADF) image shows Type3 TaC particles of the as-cast CPJ7 sample

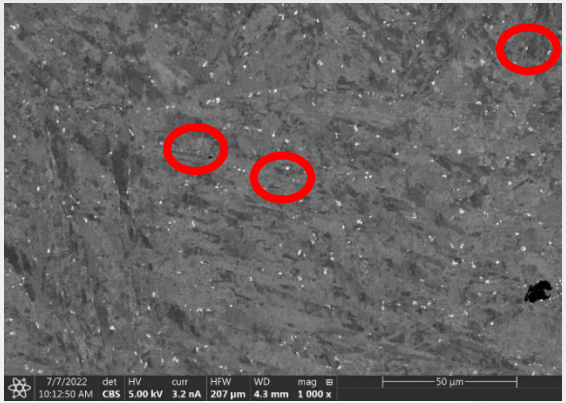


BSE-EDX maps shows Type1 Ta_2O_5 inclusions associating with creep damage in the creep CPJ7 sample at its gauge section closed to the fracture surface

PFIB Serial Sectioning 3D Reconstruction



Gauge section close to fracture of a creep ruptured CPJ7 steel



10kV

Creep damage under the polishing surface

30kV

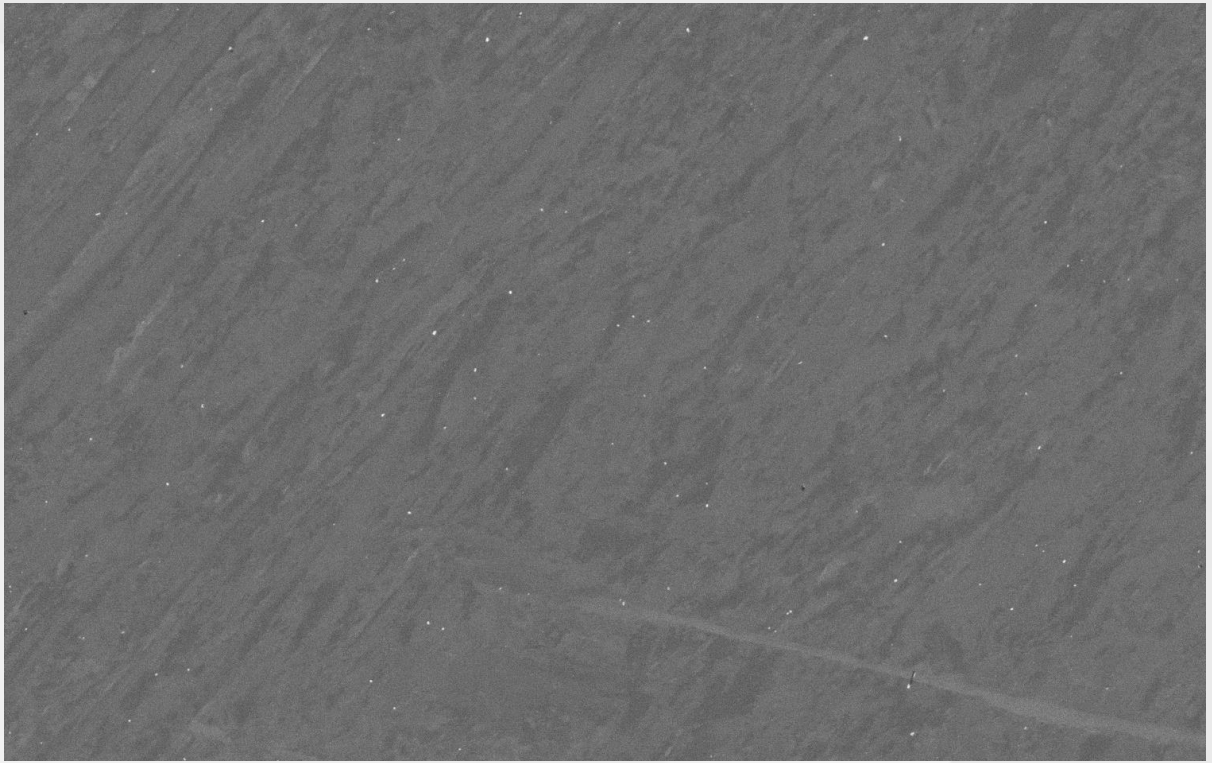
PFIB Serial Sectioning 3D Reconstruction

As-manufactured CPJ7 steel

100x50x50μm
BSE every 2 slices (dz=100nm)
500 slices in total

Accelerating Voltage=5kV
Probe Current=0.4nA
3072×2048 pixels
100×66.7 μm

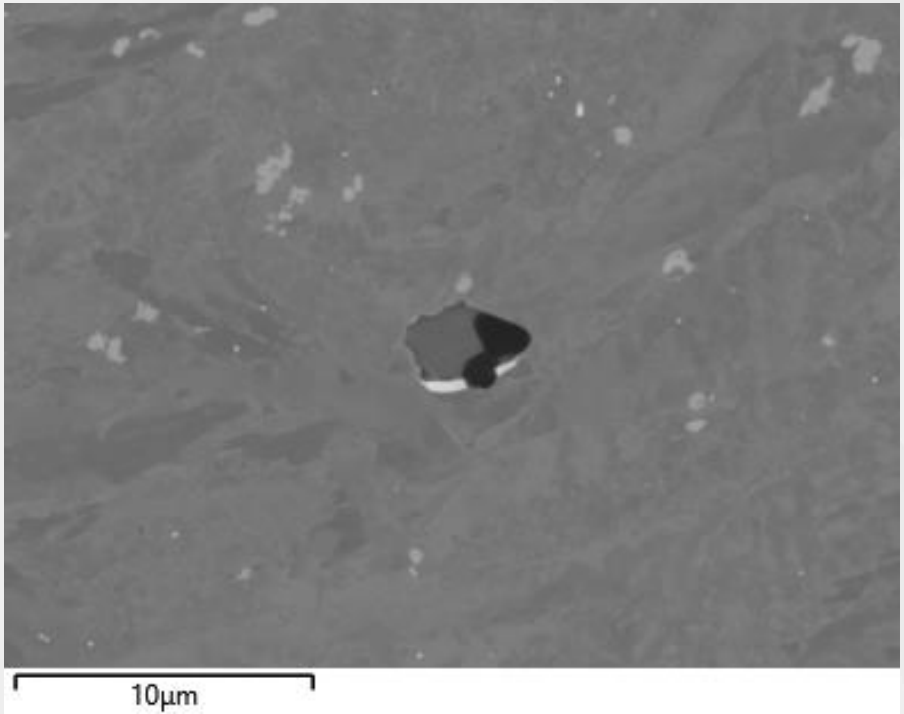
Sample preparation +
data collection time: ~72 hours



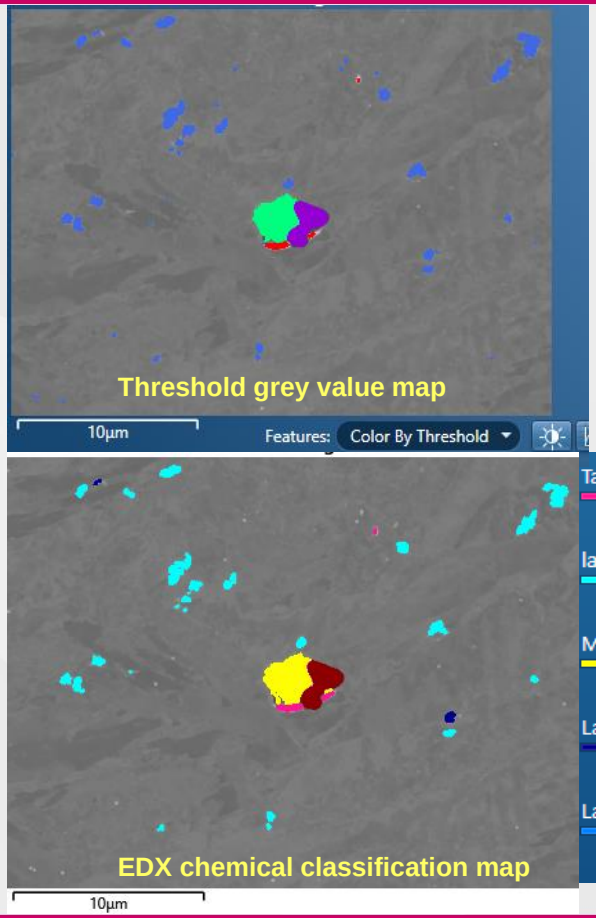
BSE slices of the as-manufactured CPJ7 steel sample

Precipitate Quantification

via Image Grey Value Thresholding



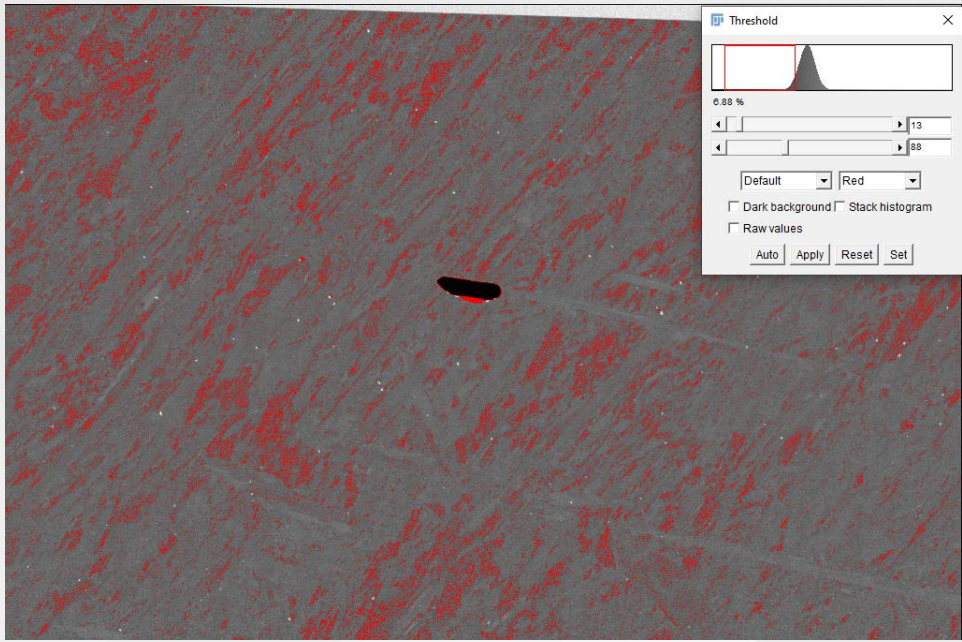
BSE-EDX FEATURE images of a creep tested CPJ7 steel sample at its head section



Precipitate Quantification

via Image Grey Value Thresholding: Troubleshooting

BSE micrograph of the as-receive CPJ7 steel sample



- **Strong channelling contrast in the martensite matrix**
- **Machine learning models must be applied**

3D Reconstruction by Machine Learning

As-manufactured CPJ7 steel

Phase segmentation results from machine learning

Background = matrix
Pink = TaC
Green = cast pores
Red = MnS

Developing model +
data analysis time: ~ 1 month

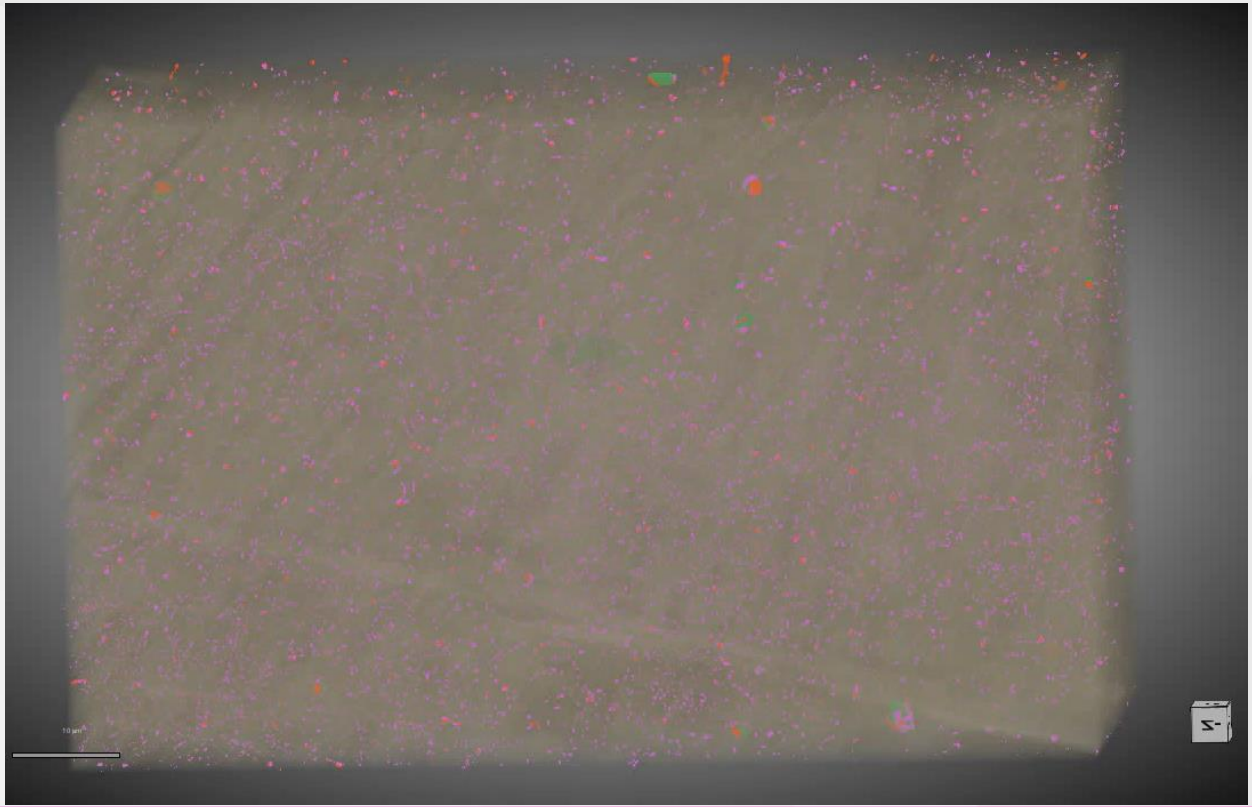
BSE slices of the as-manufactured CPJ7 steel sample after phase segmentation by machine learning

3D Reconstruction by Machine Learning

As-manufactured CPJ7 steel

3D visualisation of phase segmentation by deep learning

Yellow = matrix
Pink = TaC
Green = cast pores
Red =MnS



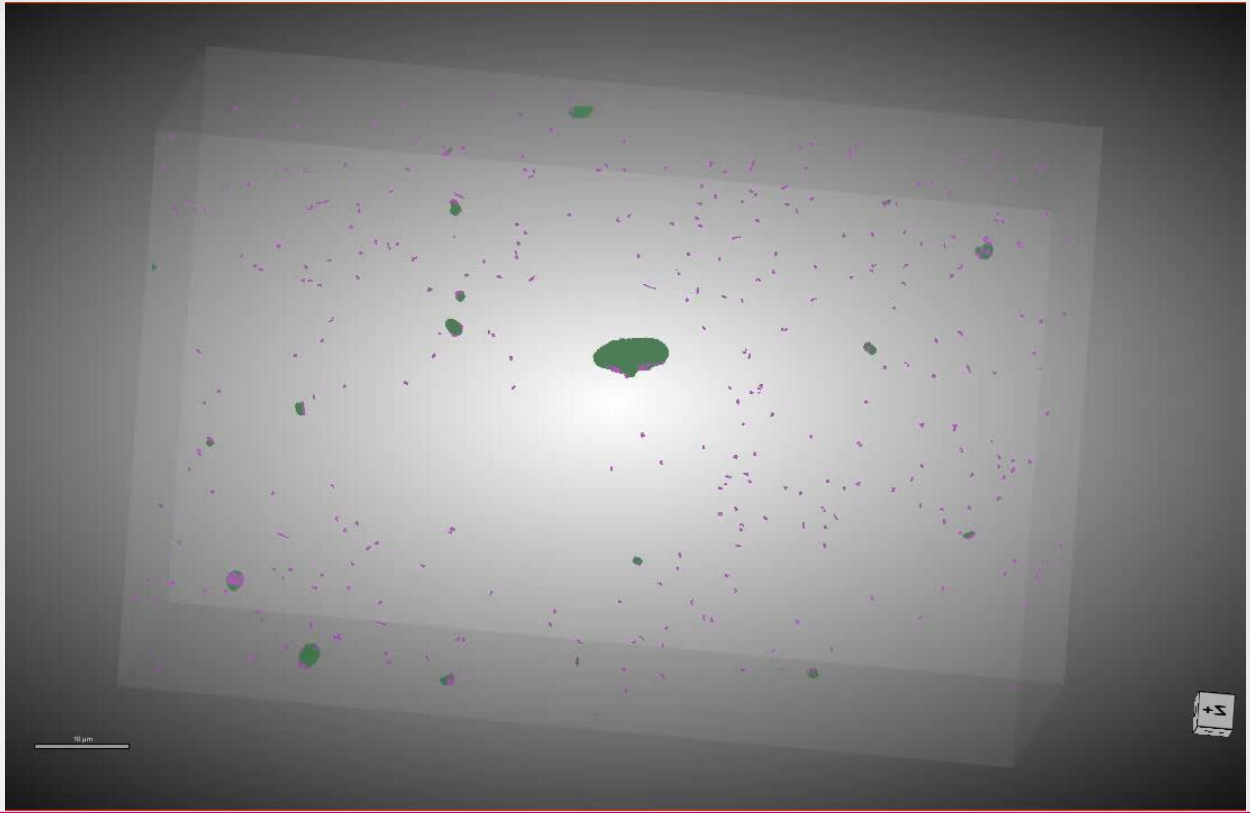
3D Reconstruction by Machine Learning

As-manufactured CPJ7 steel

3D visualisation machine learning result
Segmentation of Type1 Ta_2O_5 and Type2 TaC with cast pores

Pink = TaC
Green = cast pores

Ta enrich particles %volume=0.04%

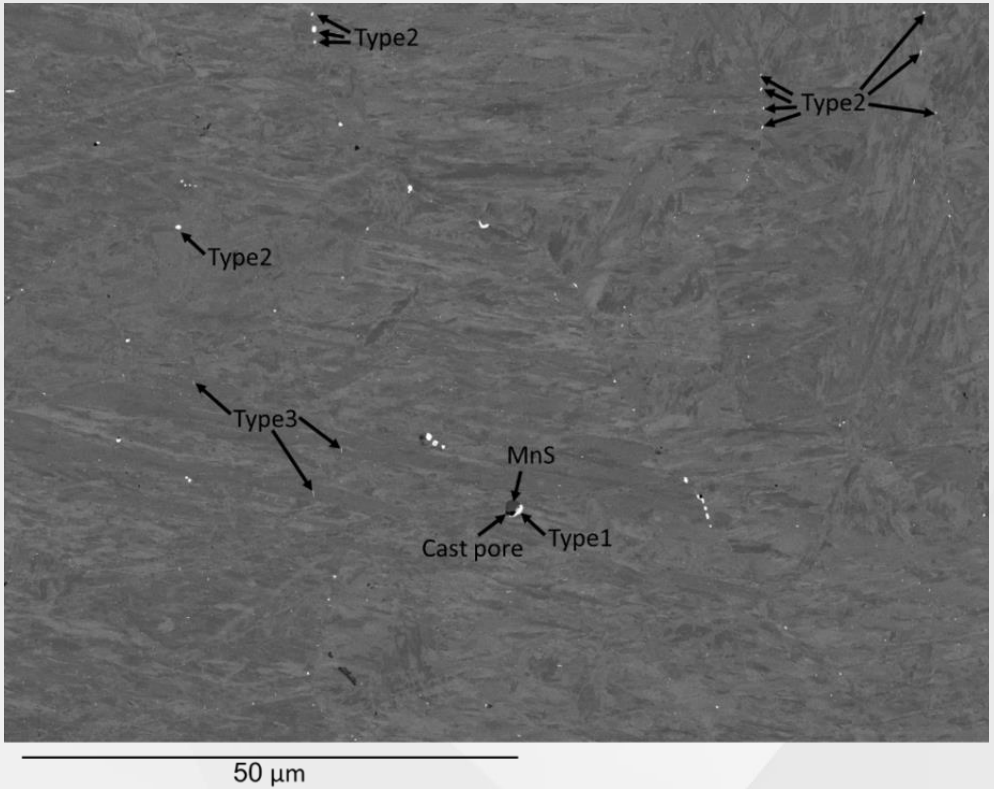


Quantification of Ta-enriched articles

- **Type 1: Ta_2O_5, ECD>1µm, associated with pores /MnS particles**
- **Type 2: TaC, ECD 0.1-1µm, along PAGs / in the matrix**
- **Type 3: TaC, rod shape, ~50nm in diameter 0.5-2µm in length**

2D area fraction vs. 3D volume fraction

method	% Ta enriched particles area/volume
EDX feature	0.05%
Helios PFIB BSE	0.07%
JEOL BSE	0.06%
Helios PFIB 3D reconstruction	0.04%



BSE-SEM micrograph shows Ta enriched particles in the as-receive material

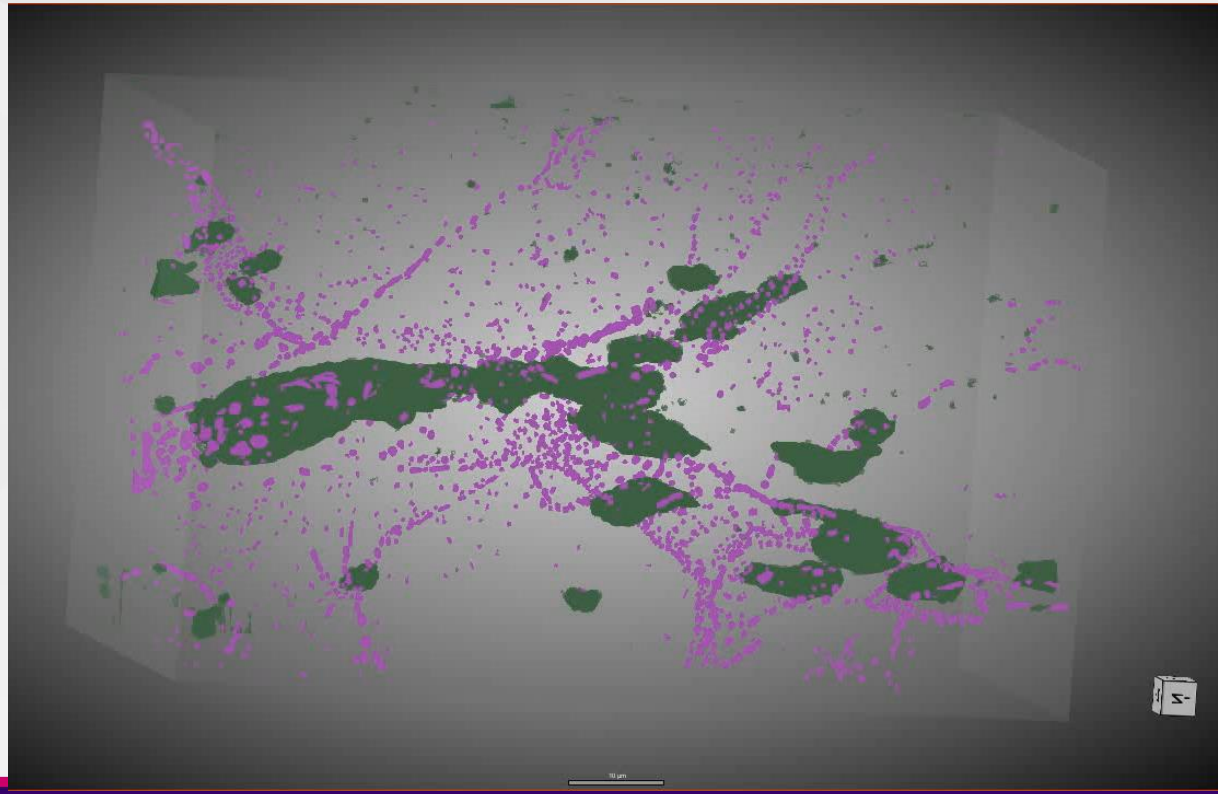
3D Reconstruction by Machine Learning

Gauge section close to fracture of a creep ruptured CPJ7 steel sample

Machine learning 3D visualisation
Segmentation of creep damages, Type1 Ta_2O_5 and Type2 TaC

Pink = TaC
Green = creep damage

Large TaC(>0.5µm) precipitates and Ta_2O_5 inclusions are likely to associate with creep damage and prior austenite grain boundaries



Conclusions and future work

- **Summary of conclusions:**
  - Laves phase, MnS inclusions, pores and Ta enriched inclusions were quantified.
  - 3D investigation confirmed a close association between Ta enriched particles and creep cavities in the creep ruptured material.
  - Relatively large Ta enriched phases can act as preferred sites for cavity nucleation.
  - **3D reconstruction of microstructures is critical to understand creep risk factors.**

- **Future work**
  - Further development of the 3D reconstruction model
  - Further quantification of the 3D reconstruction dataset

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Dr. Stuart Robertson

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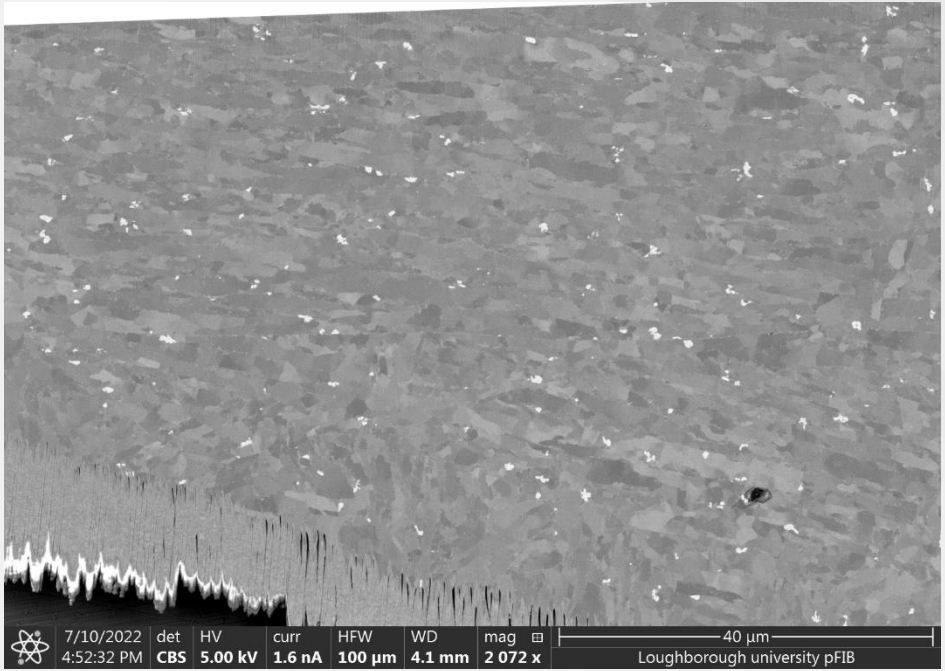
Thank you!

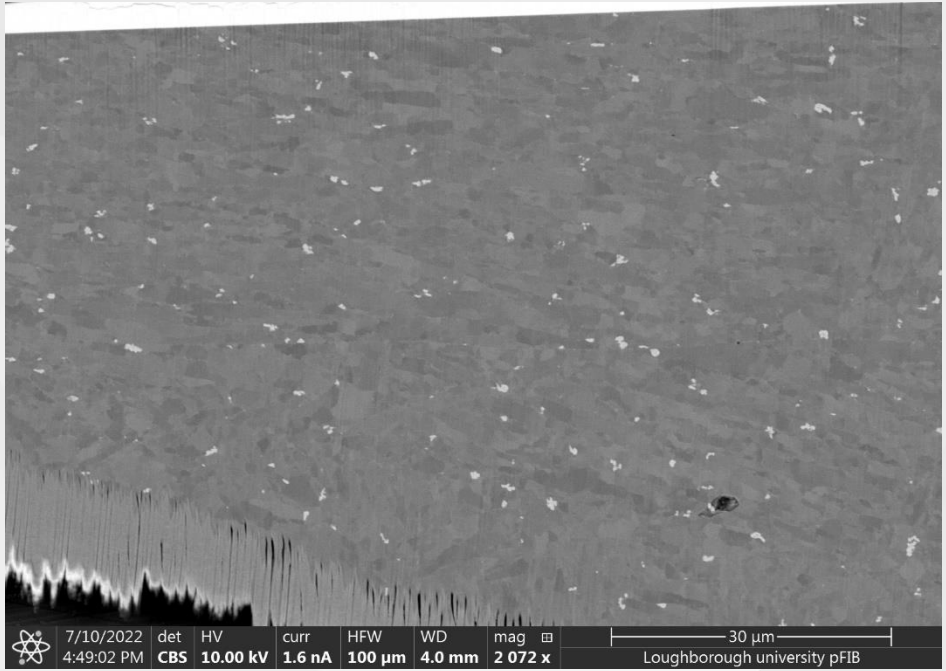
Any questions?

Detector test

Gauge section close to fracture of a creep ruptured 9Cr steel



5kV BSE



10kV BSE