

Characterisation of Thermally Aged Stainless Steels for Power Generation Applications

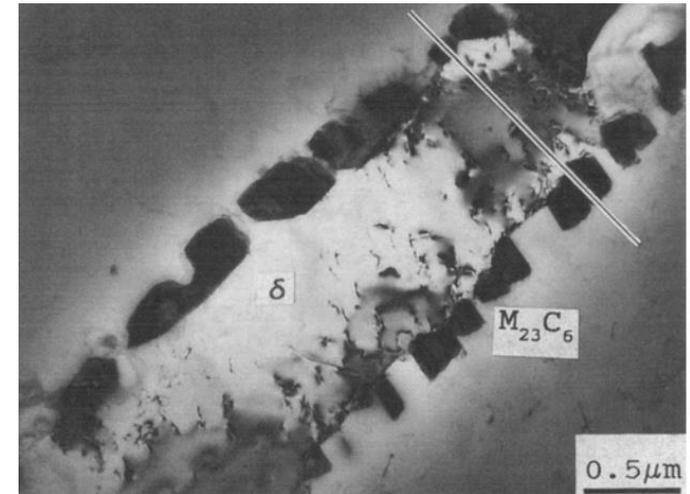
By Oscar Smith

Rebecca Higginson and Simon Hogg, Loughborough University
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- Introduction
- Background
- Experimental Method
- Initial Condition of Material
- Results
- Conclusion

Background

- Austenitic stainless steels can be subjected to high temperatures for extended periods of time in service. This can cause the formation of secondary phases.
- Sigma Phase and $M_{23}C_6$ can degrade the mechanical properties and corrosion resistance of the material.
- Regions of ferrite transform fastest.
- Previous work has relied heavily on TEM investigations to detect and characterise these phases.
- Work is needed to understand how the microstructure of these metals develop during time in service.



Tseng, C. C. Shen, Y et al, "Fracture and the formation of sigma phase, $M_{23}C_6$, and austenite from delta-ferrite in an AISI 304L stainless steel" *Metallurgical and Materials Transactions A*, Vol 25, 1994

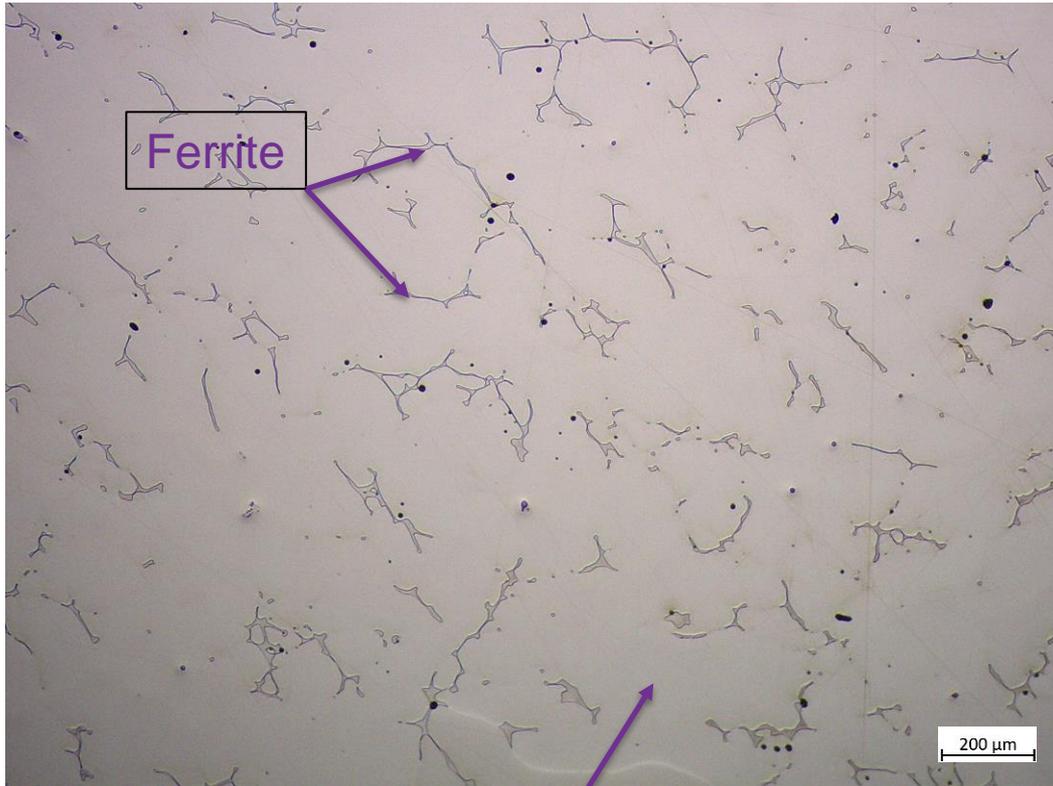
- Material: Cast 304L Stainless Steel:

Fe	Cr	Ni	C
70.0%	18.7%	8.86%	0.0136%

- Thermal aging of stainless steel components at 750°C and 650°C up to 10,000Hrs.
- Characterisation of the microstructure:
 - Optical Microscopy
 - Scanning Electron Microscopy - Electron Backscatter Diffraction (EBSD)
 - Transmission Electron Microscopy - Samples prepared using Focus Ion Beam (FIB)

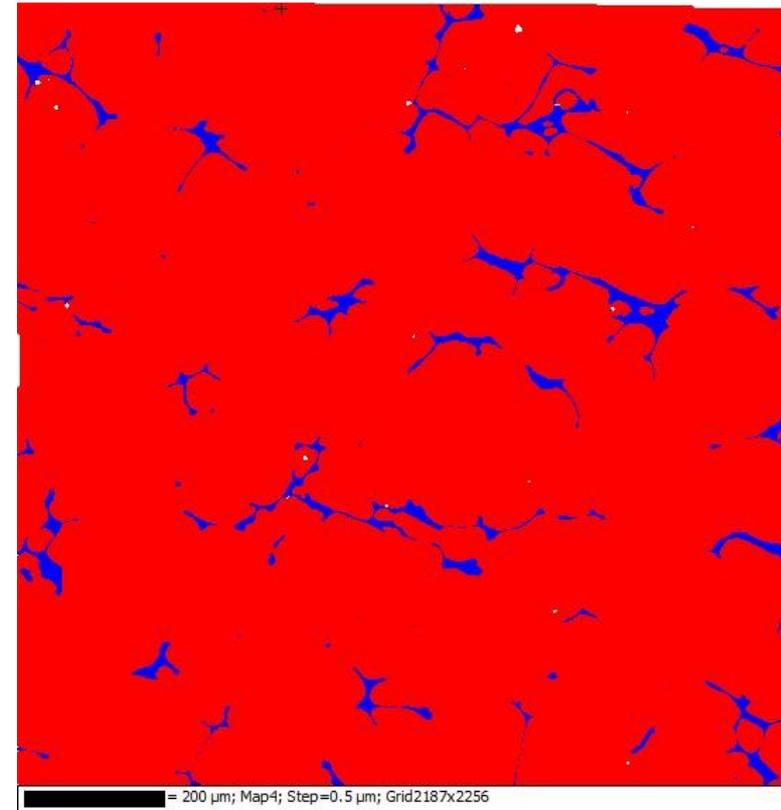
Initial Condition Of Material

Optical Image



Austenite matrix

EBSD Phase Map

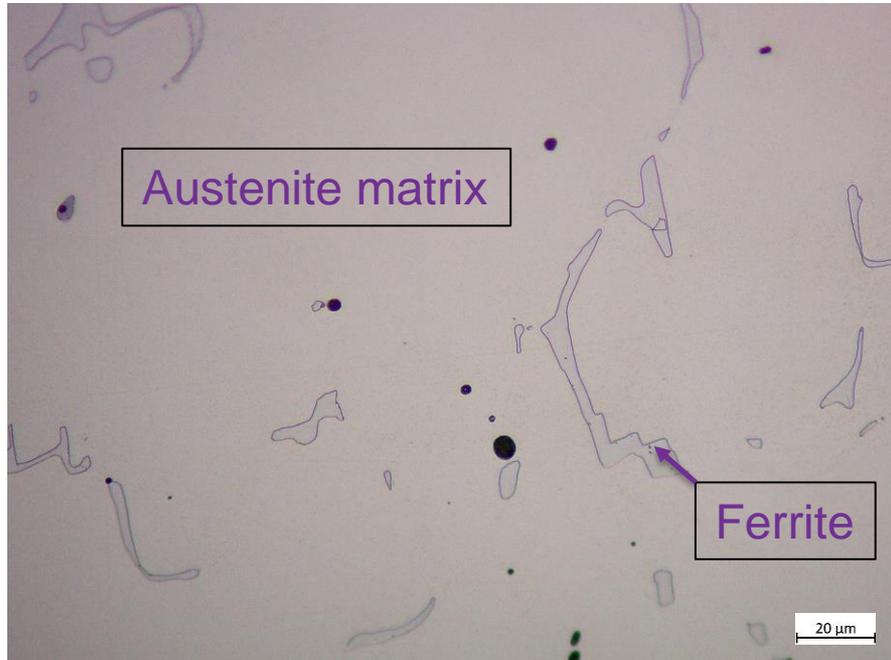


 Ferrite  Austenite

Ferrite	Austenite	Zero Solutions
2.03%	97.55%	0.42%

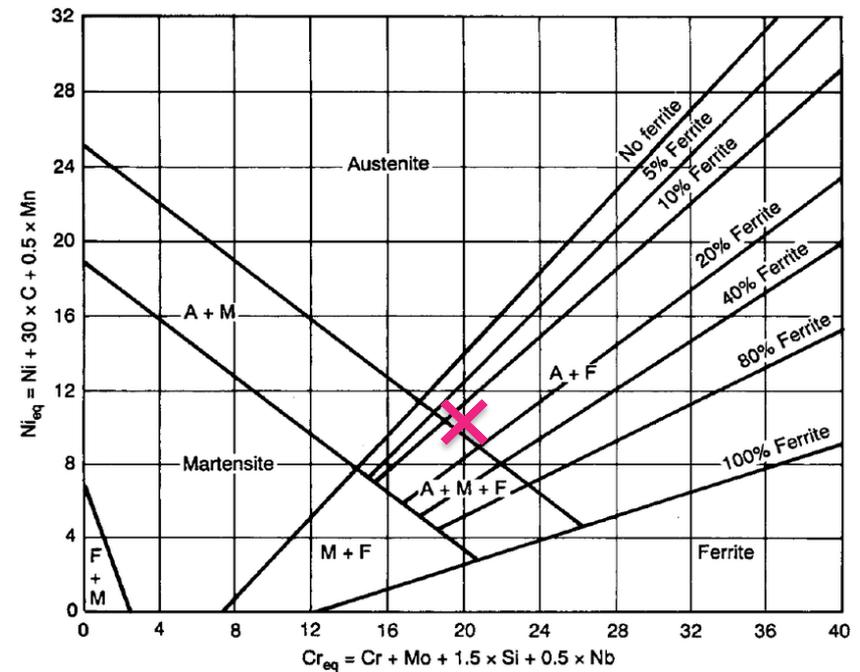
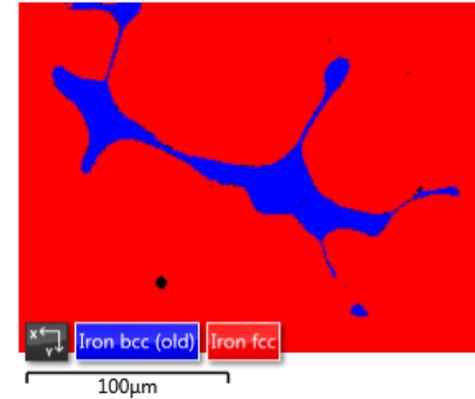
Initial Condition of Material

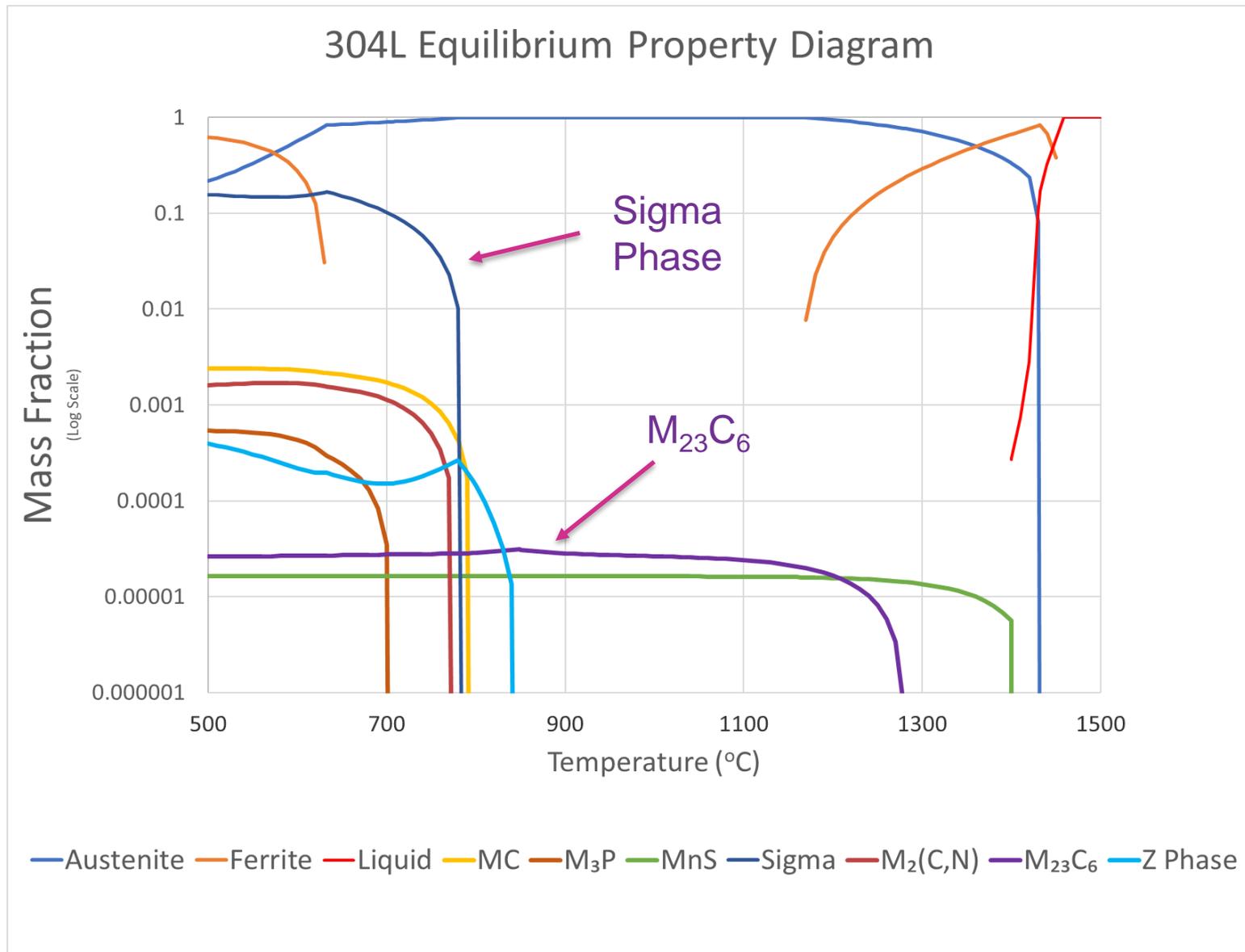
Optical Image



Equivalent:
Cr 20.23%
Ni 9.67%

Phase Color 46

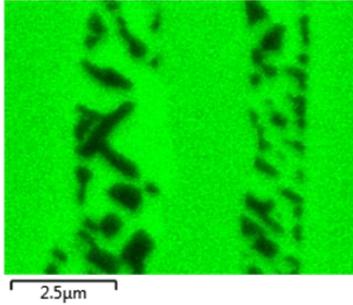




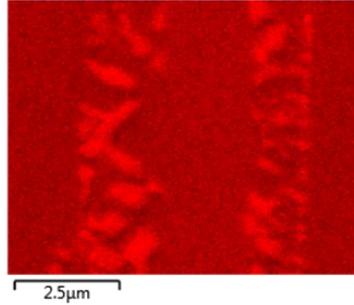
Investigation of Aged Material

750°C 200Hrs

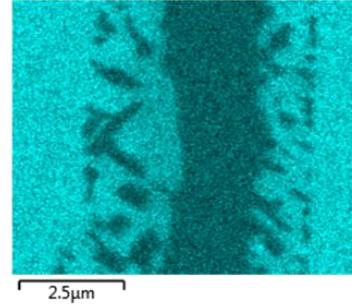
Fe L α 1,2



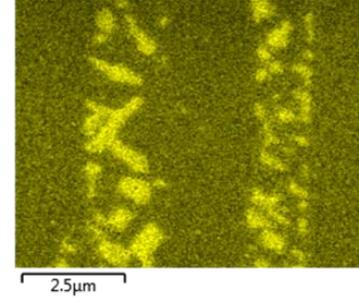
Cr L α 1,2



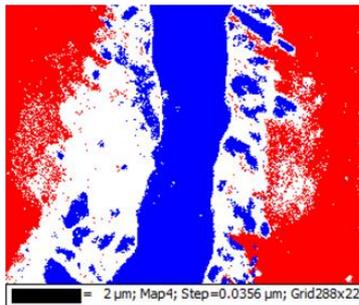
Ni L α 1,2



C K α 1,2

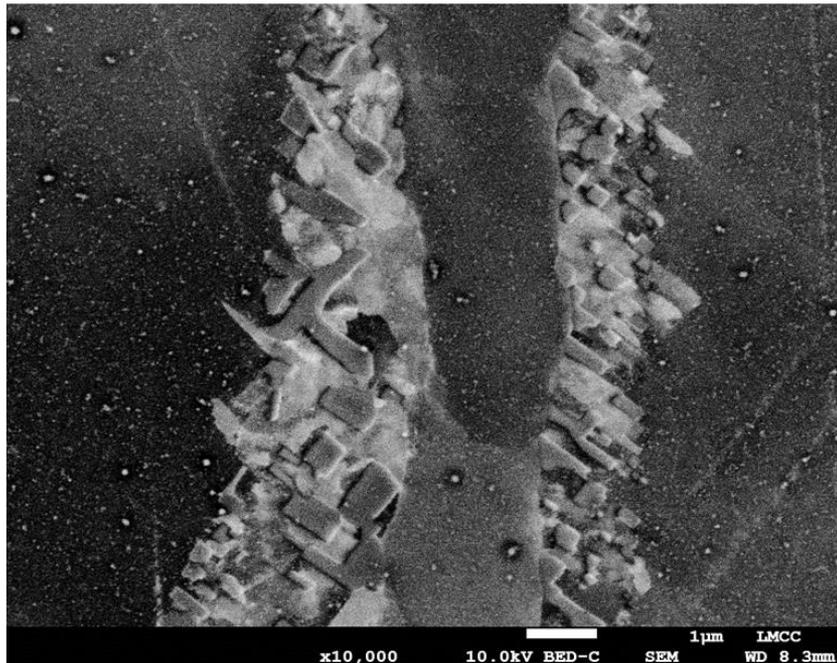
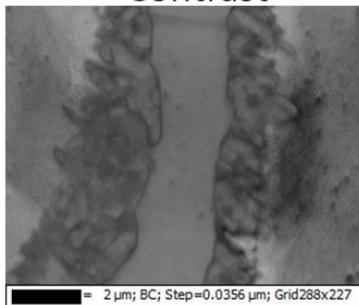


Phase Map



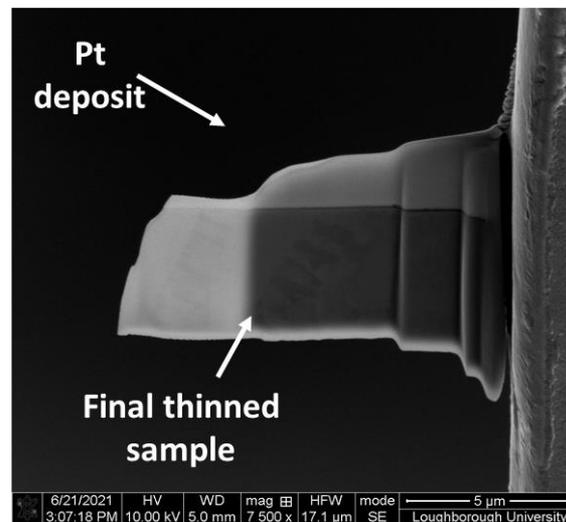
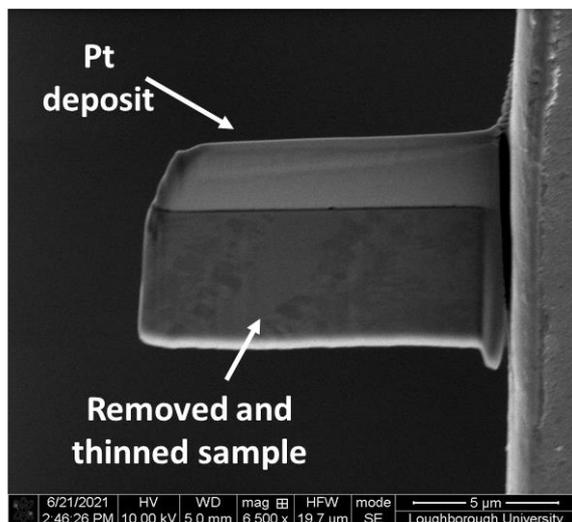
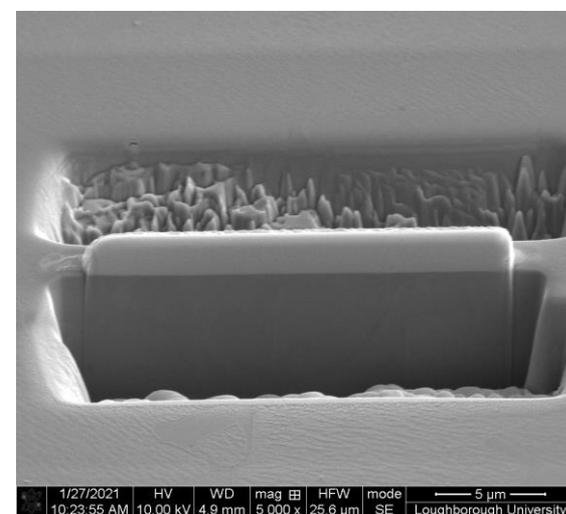
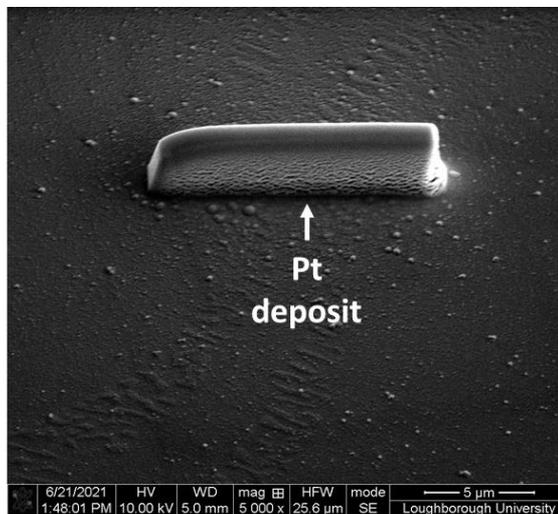
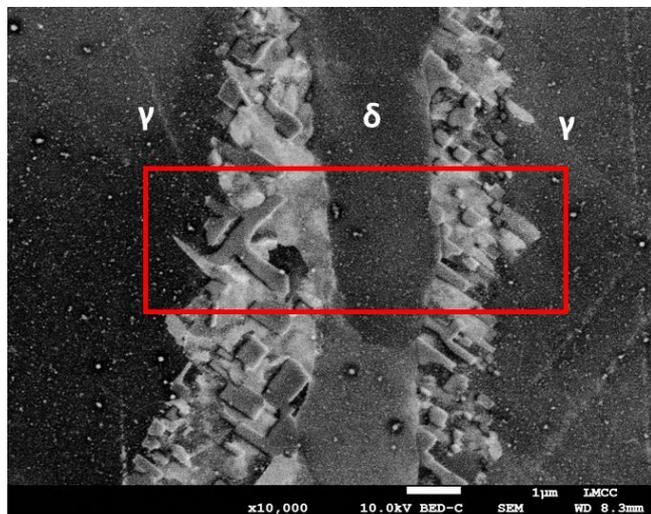
Band

Contrast

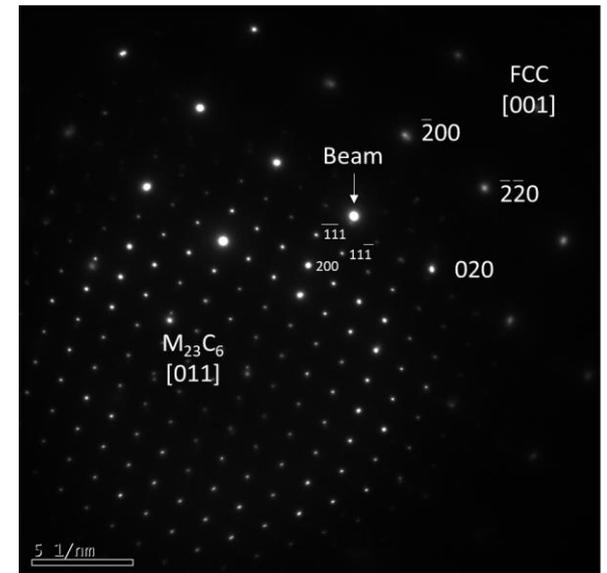
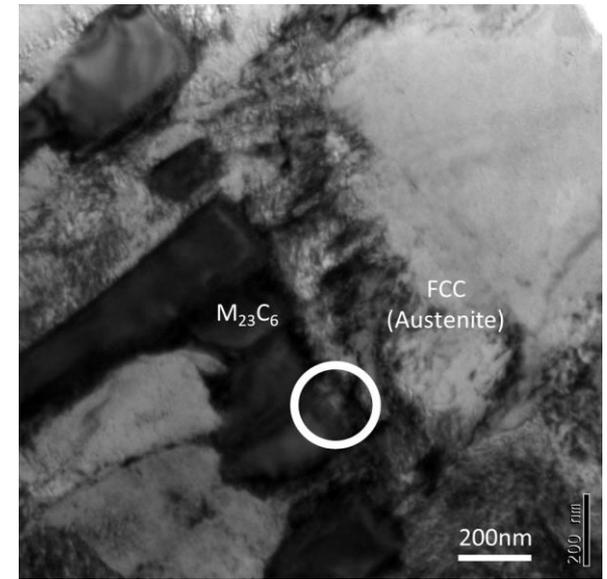
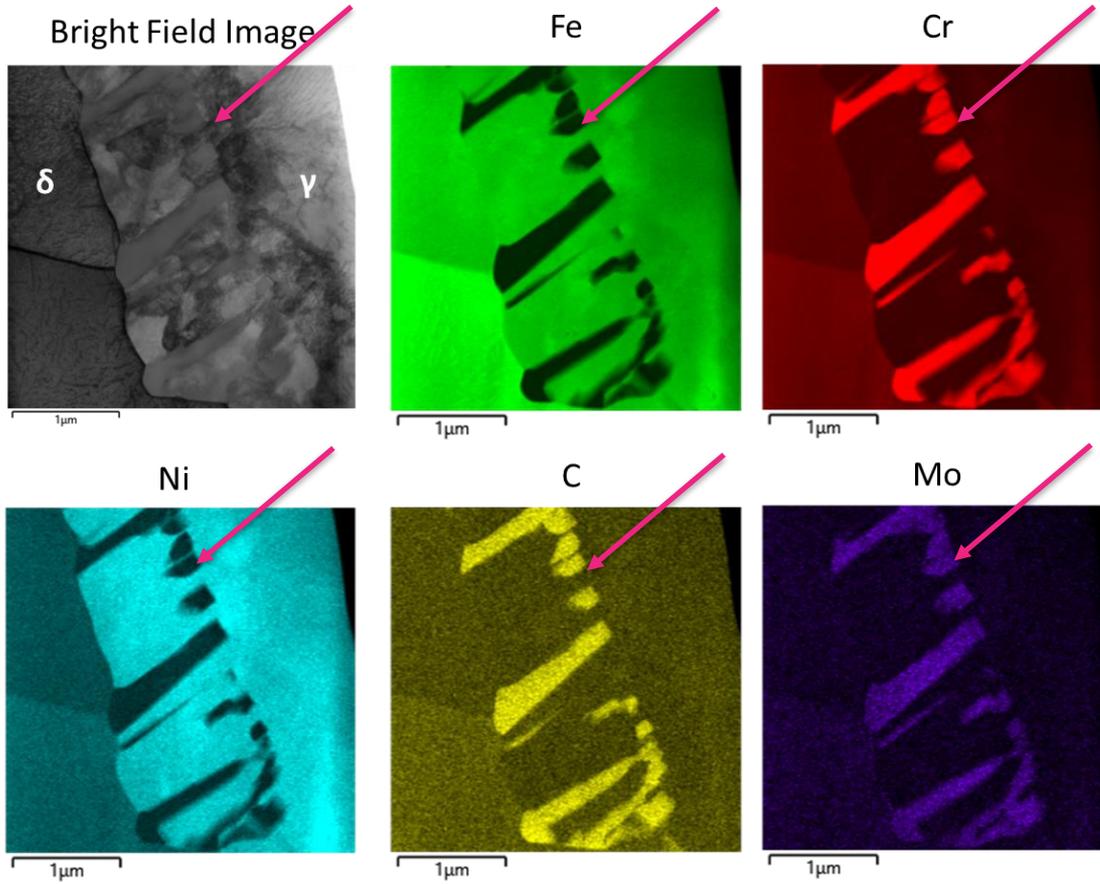


x10,000 10.0kV BED-C SEM 1μm IMCC WD 8.3mm

Sample prep for TEM using FIB

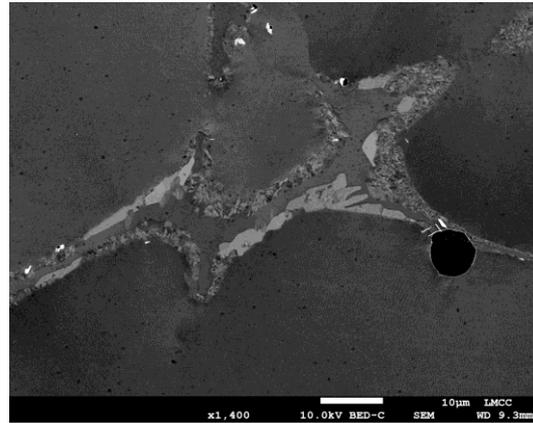


TEM Results

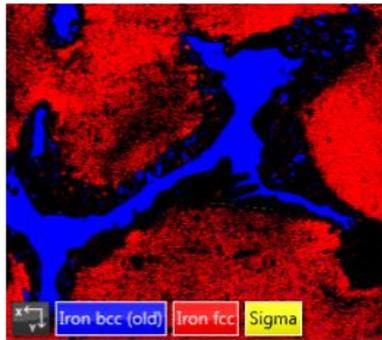


Updated EBSD CCD Detector (Close couple device)

650°C 500Hrs

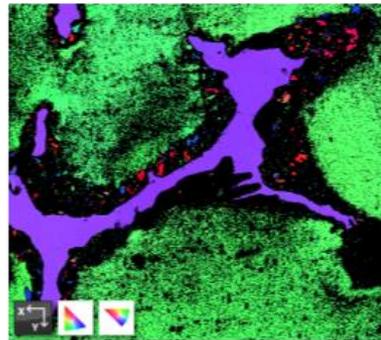


Phase Color 11



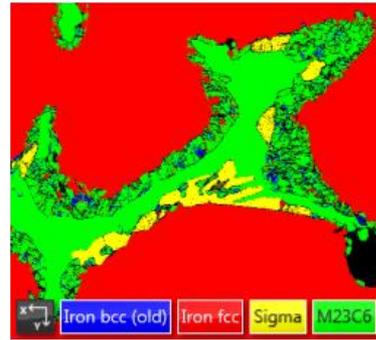
25µm Fe $\alpha_{1,2}$

IPF Z Color 11



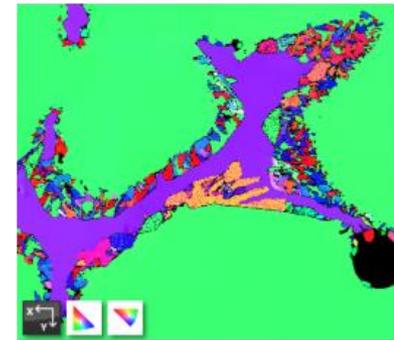
25µm Cr $\alpha_{1,2}$

Phase Color 10

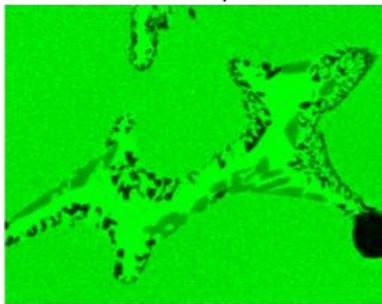


25µm Ni $\alpha_{1,2}$

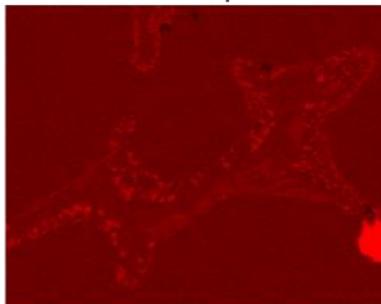
IPF Z Color 10



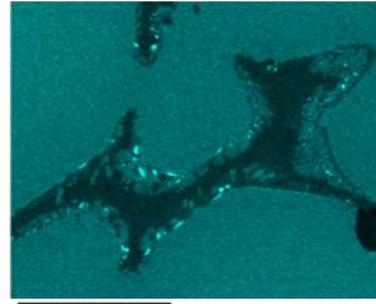
25µm C $\alpha_{1,2}$



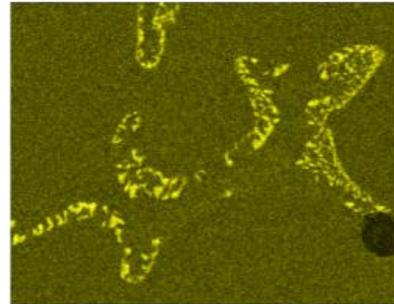
25µm



25µm



25µm

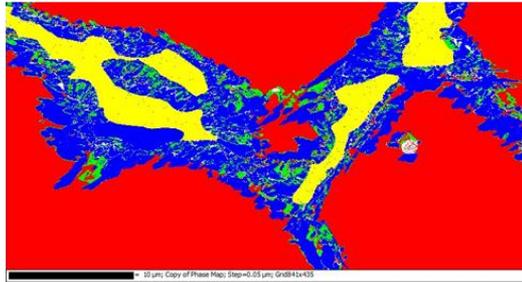


25µm

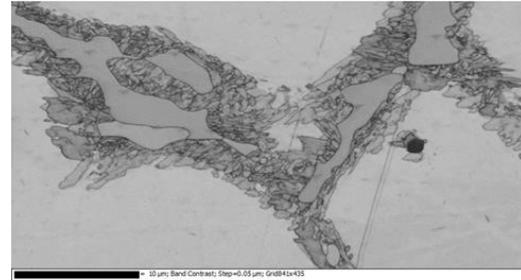
EBSD Data from “Symmetry” Detector Microscope

750°C 5000Hrs

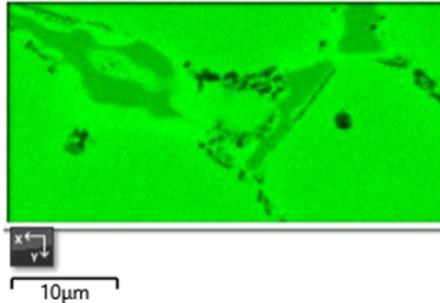
Phase Map



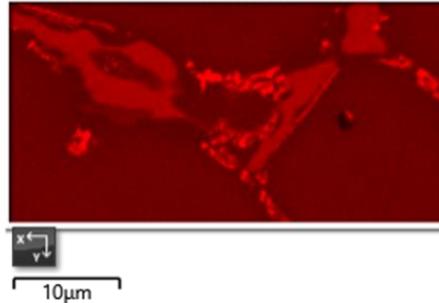
Band
Contrast



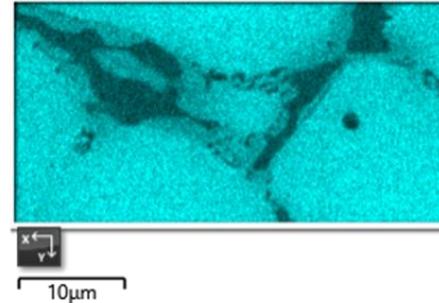
Fe



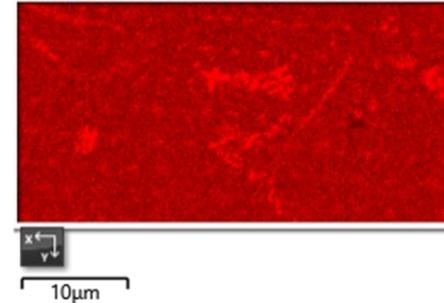
Cr



Ni



C

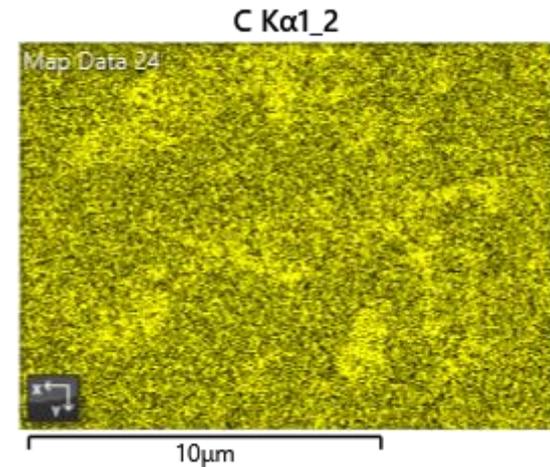
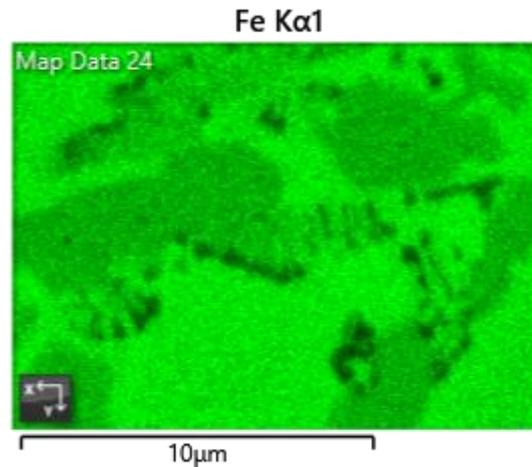
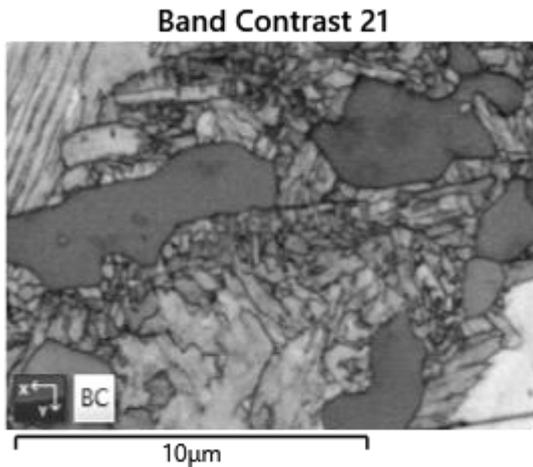
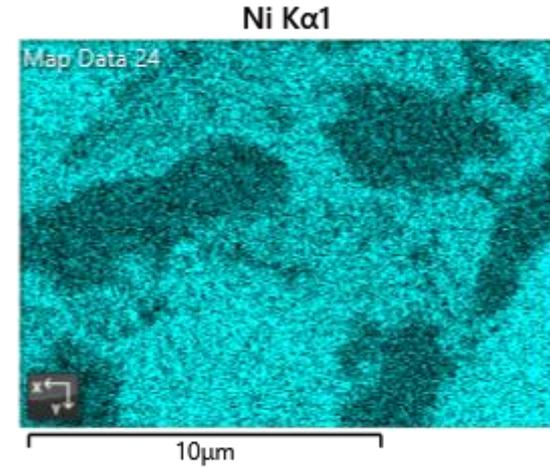
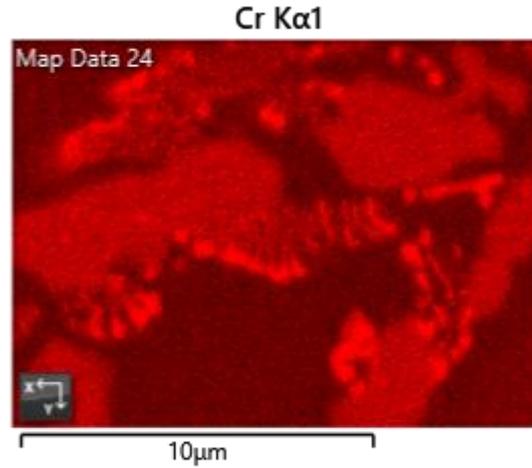
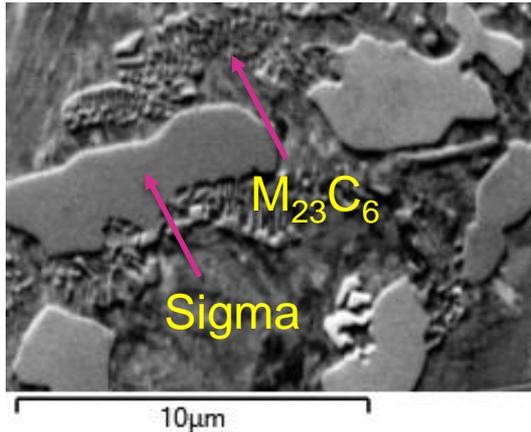


Results Achieved Using:

- Sensitivity Binning Mode
- Refined Accuracy Indexing Mode
- Centre Band Detection Mode

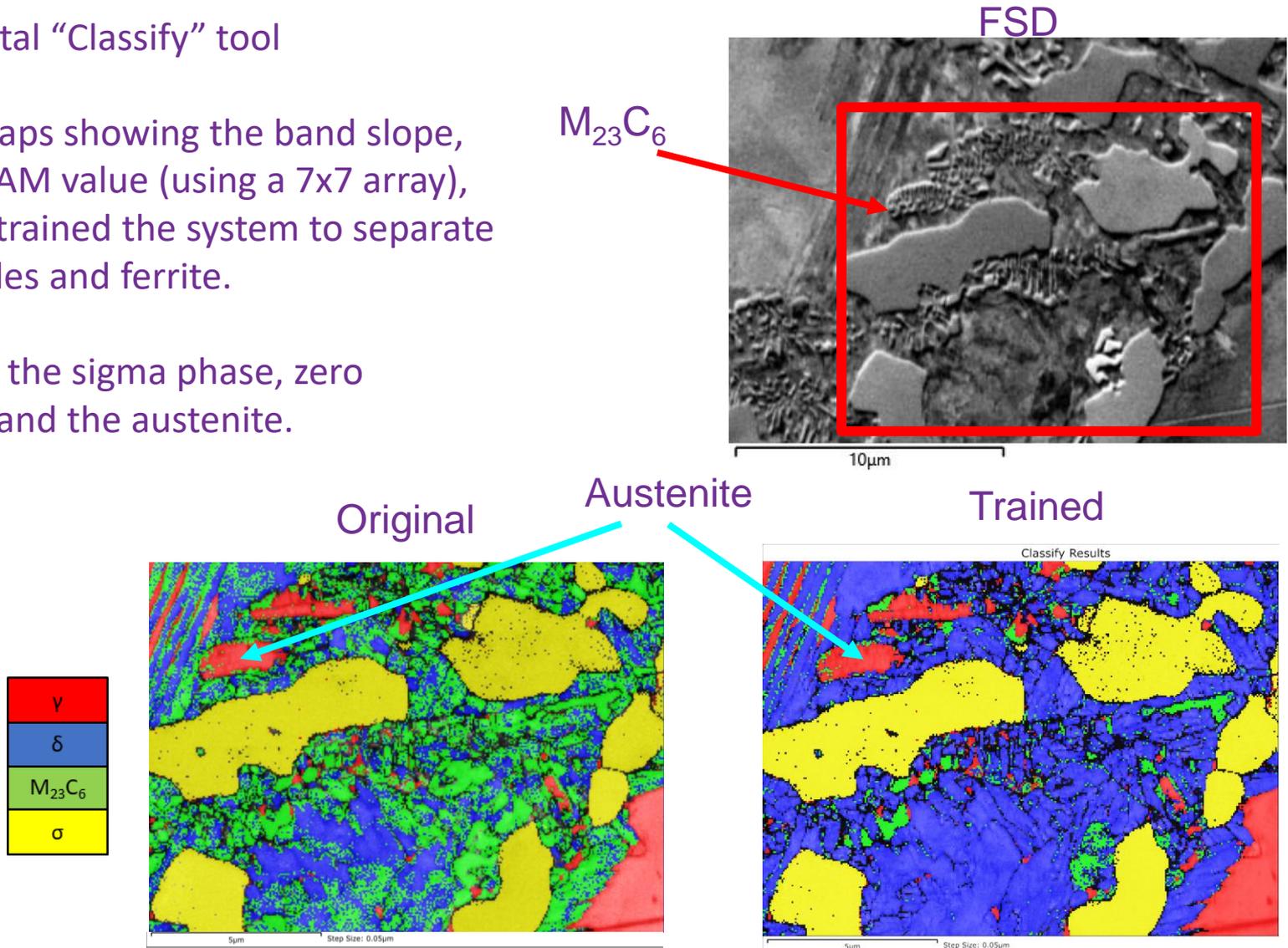
EDS for 750°C 5000Hrs Sample

Forward Scatter Diffraction



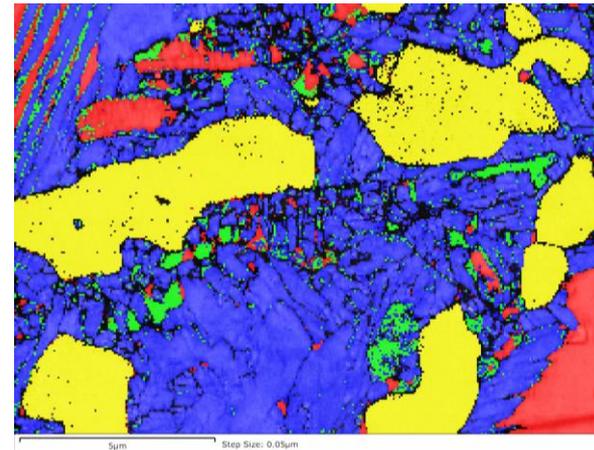
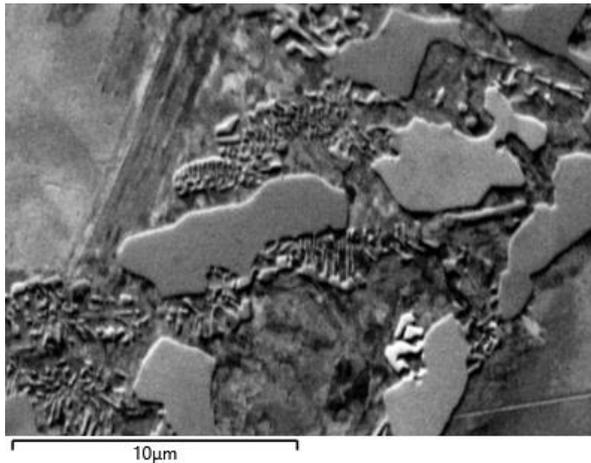
Processed Data Using Aztec Crystal

- AZtecCrystal “Classify” tool
- Plotted maps showing the band slope, and the KAM value (using a 7x7 array), and then trained the system to separate the carbides and ferrite.
- Protected the sigma phase, zero solutions and the austenite.



Conclusions

- Long term thermal aging has created more complex transformations than previously observed.



- Carbides were observed to form within the delta ferrite and not just at the interface.
- New grains of ferrite have also formed within the original delta ferrite.

Any Questions?