

HTC Project 2a

New corrosion resistant materials for challenging process environments – low oxygen activities and very high temperatures

Presented by LG. Johansson, T. Sand and M. Sattari

- ▶ *Project leader:* LG Johansson, Chalmers

- ▶ *Companies:*



Entech

- ▶ *Researchers in HTC :* LG Johansson, JE Svensson, C Geers, S. Bigdeli, M Halvarsson, M. Sattari, N Mortazavi (all Chalmers)
- ▶ *PhD student:* T. Sand (Chalmers)

Background

- ▶ Materials that can withstand process environments at very high temperatures without corroding are necessary in, e.g., steel production, in the thermal treatment of metals and ceramics, and in the manufacture of batteries and electronics
- ▶ New better materials that can withstand higher temperatures and more corrosive environments can lead to large gains in energy efficiency and also enable development of novel, energy efficient and environmentally friendly, processes and technologies

Goals

- ▶ Investigate the nitridation of high temperature alloys and the RE effect with the aim to formulate new strategies for developing alloys with superior ability to resist challenging environments and extreme temperatures
- ▶ **Materials:**
 - ▶ Alumina-forming alloys
 - ▶ Chromia-forming alloys

Exposures at 800-1100 °C in
95 % N₂, 5 % H₂, ppm-levels of O₂

Alloys tested

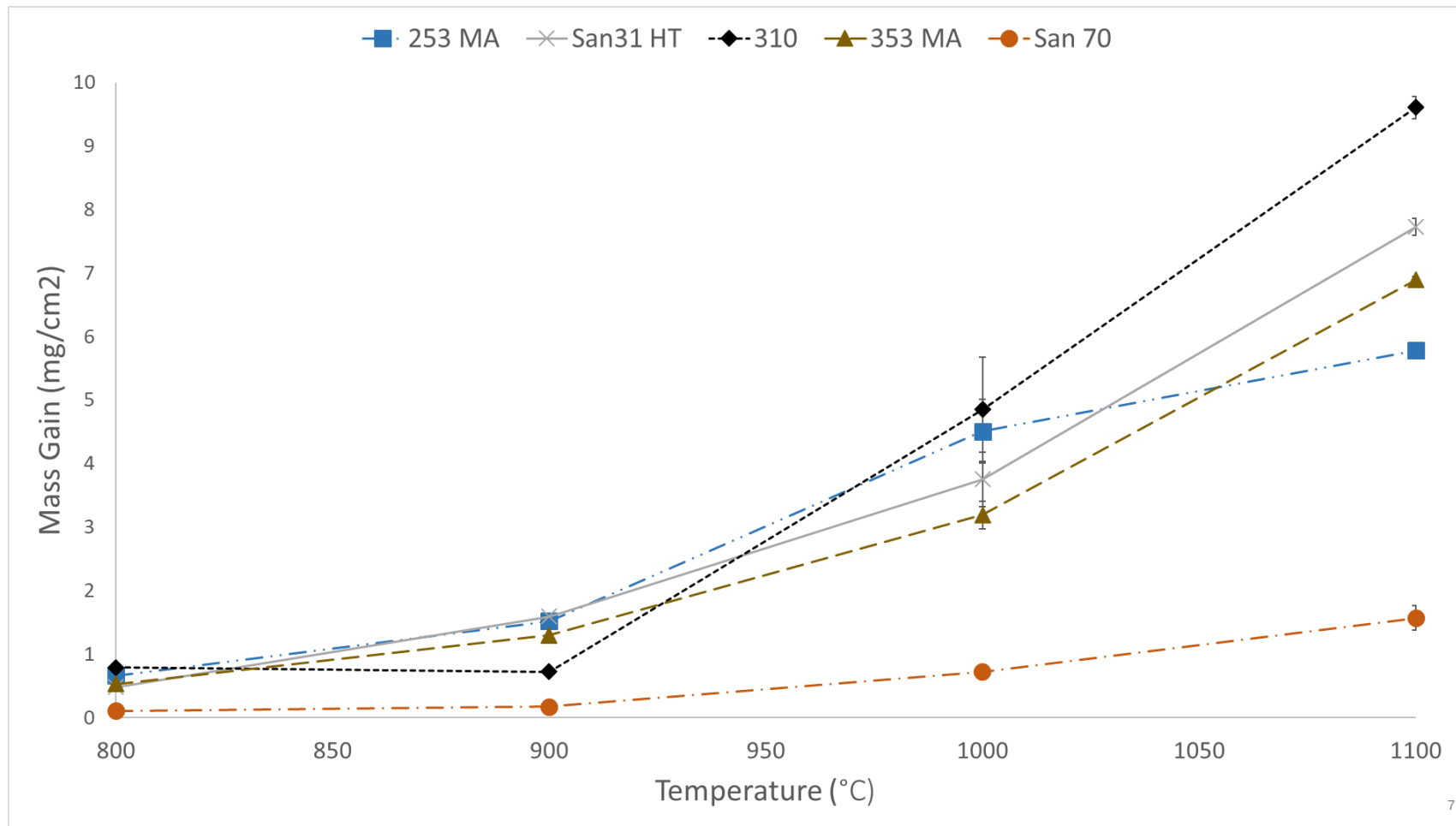
Grade	253MA	7RE10 (310H)	SANICRO 31HT	353MA	SANICRO 70
ELEM	V.VÄRDE	V.VÄRDE	V.VÄRDE	V.VÄRDE	V.VÄRDE
C	0.073	0.045	0.064	0.069	0.013
NI	10.80	20.16	30.31	34.91	Bal.
CR	20.95	24.31	20.26	25.65	16.73
SI	1.52	0.34	0.44	1.39	0.31
MN	0.67	1.24	0.50	1.68	0.19
N	0.156	0.038	0.012	0.172	0.006
CE	0.04			0.04	
AL			0.46		0.14
TI			0.51		0.33
FE	Bal.	Bal.	Bal.	Bal.	9.76

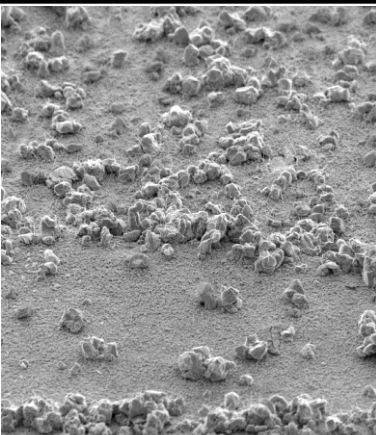
Nitridation resistance increases with increasing Ni and decreasing Cr

Expected increasing nitridation resistance

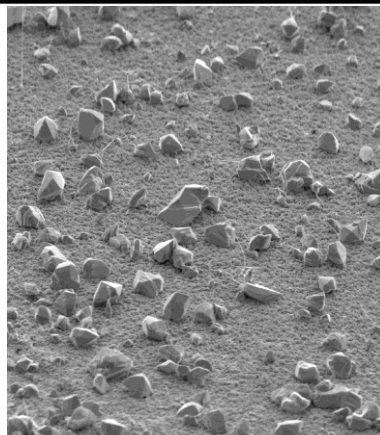


253MA 310H 353MA Sanicro 70
Sanicro 31HT

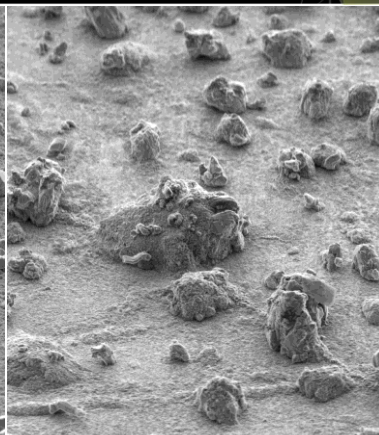




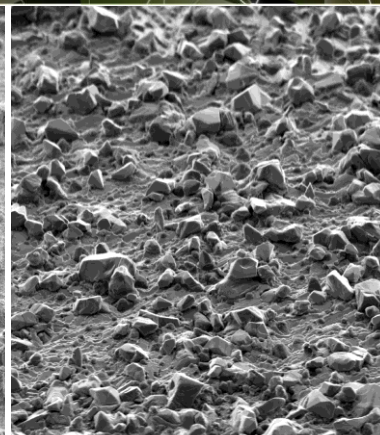
1 μ m
253MA



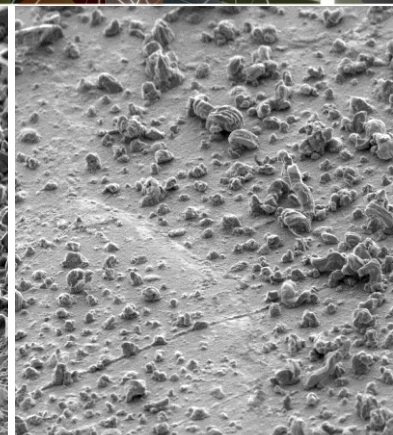
1 μ m
310H



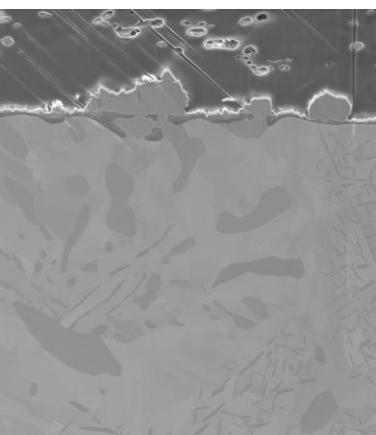
1 μ m
San31HT



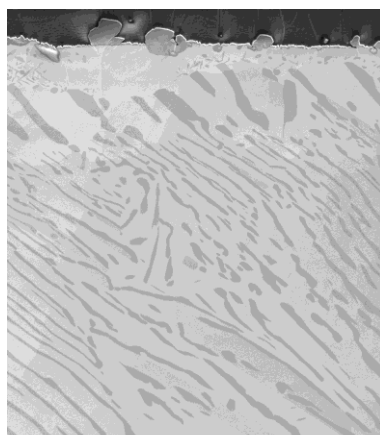
1 μ m
353MA



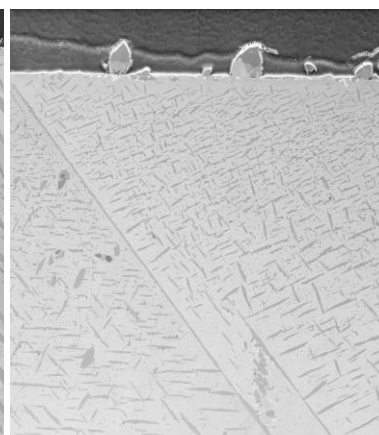
1 μ m
San70



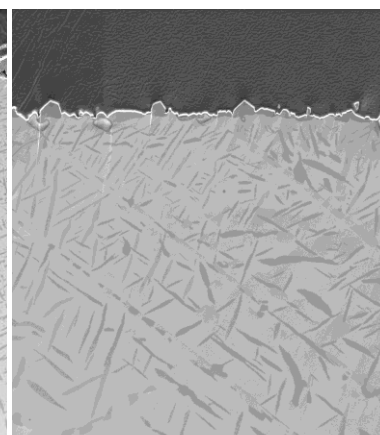
HT = 2.00 kV
VD = 2.0 mm
Signal A = InLens
Mag = 20.00 KX
Date : 24 Nov 201
Time : 12:29:47



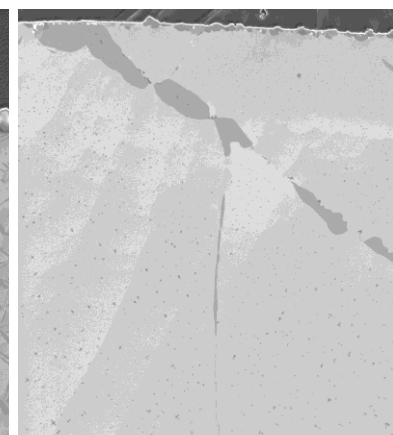
1 μ m
310H



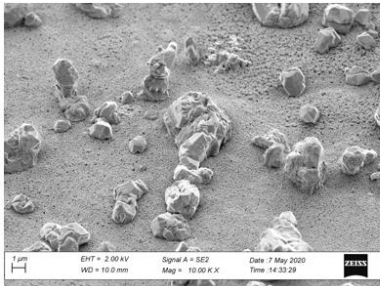
1 μ m
San31HT



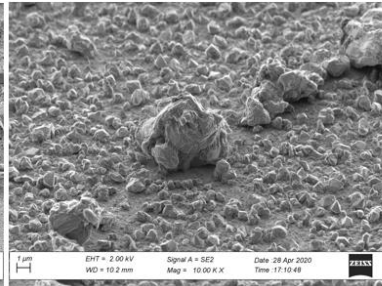
1 μ m
353MA



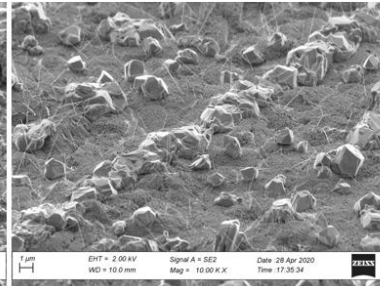
1 μ m
San70



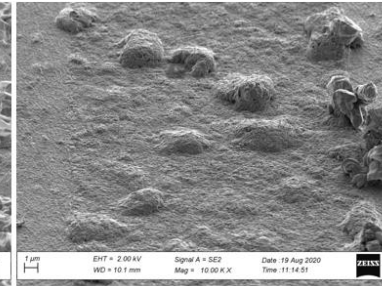
253MA



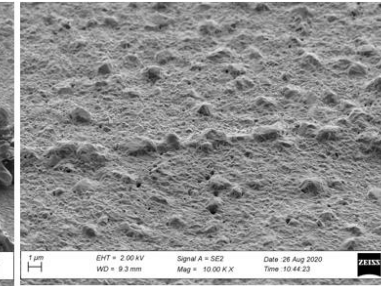
San31HT



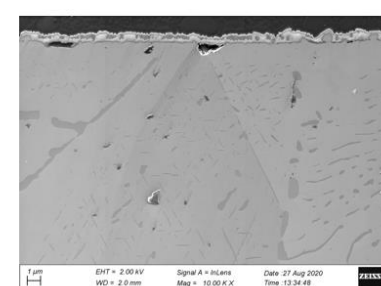
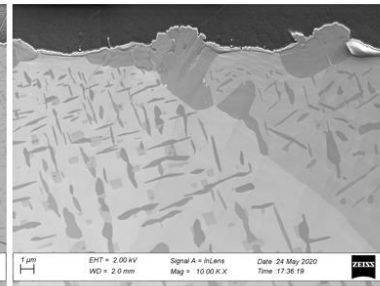
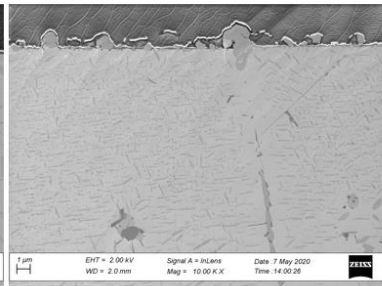
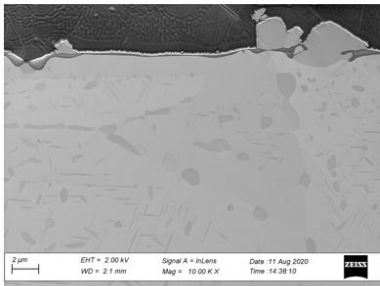
353MA



San70

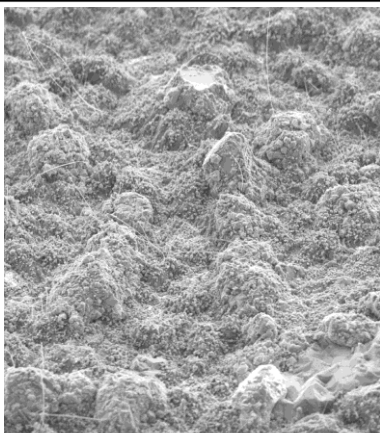


310H

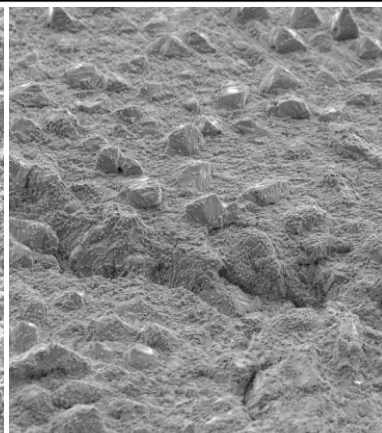


CHALMERS

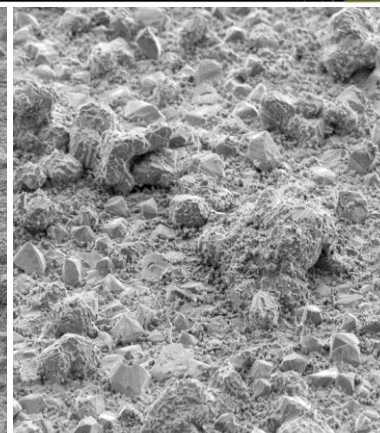
1000 °C



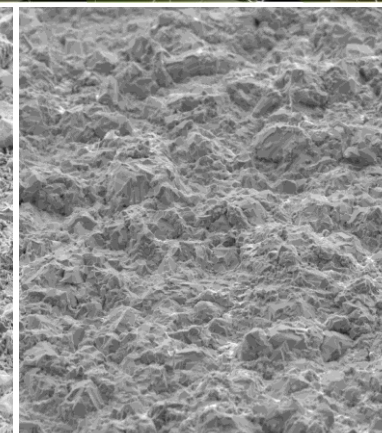
1 μm 253MA



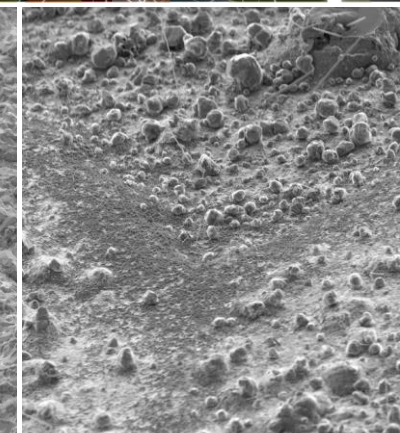
1 μm 310H



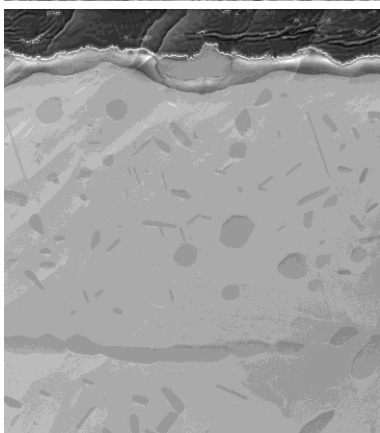
1 μm San31HT



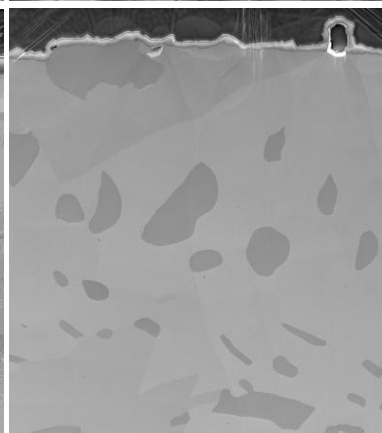
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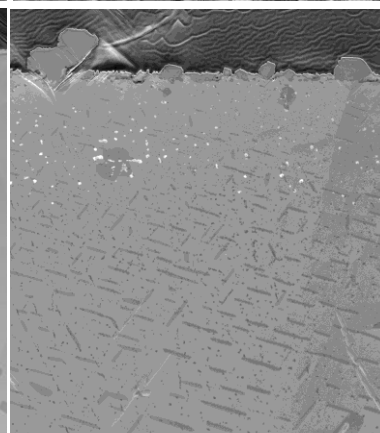
1 μm San70



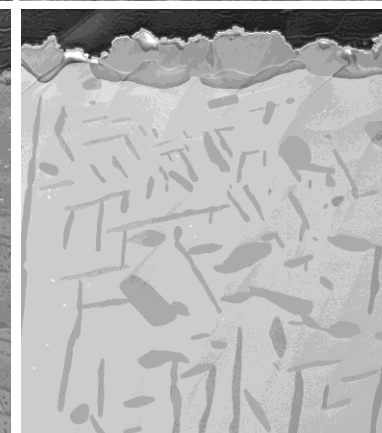
1 μm 253MA



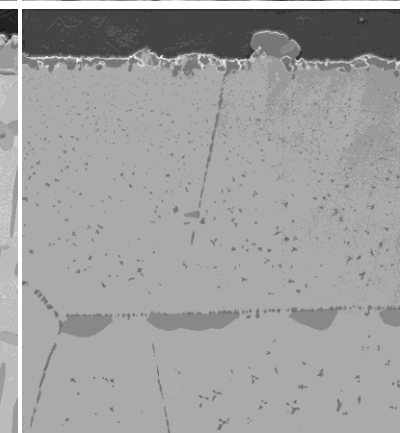
1 μm 310H



1 μm San31HT



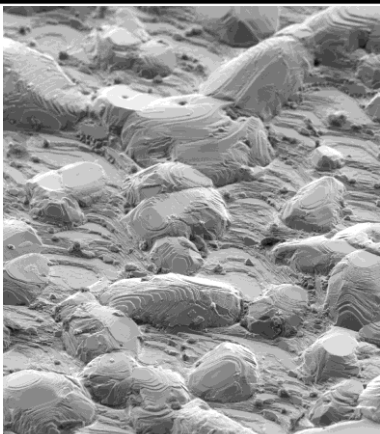
1 μm 353MA



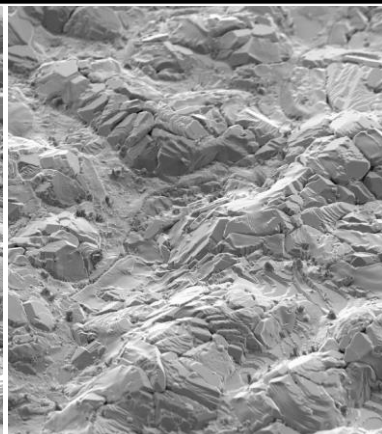
1 μm San70

CHALMERS

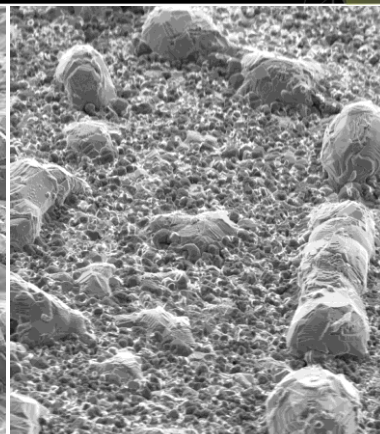
1100 °C



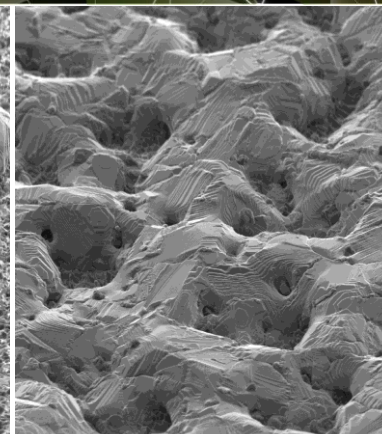
1 μm 253MA



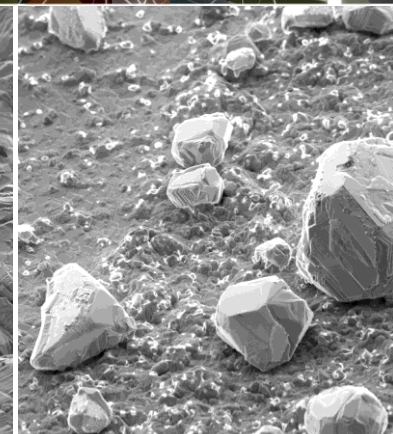
1 μm 310H



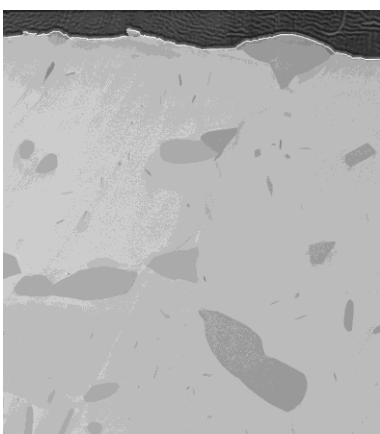
1 μm San31HT



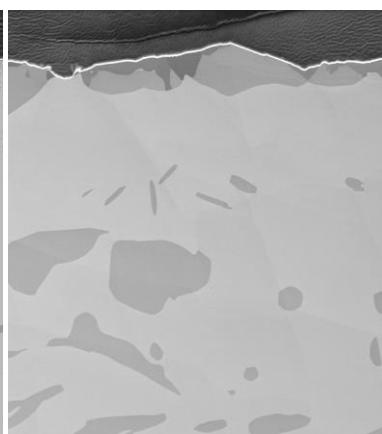
1 μm 353MA



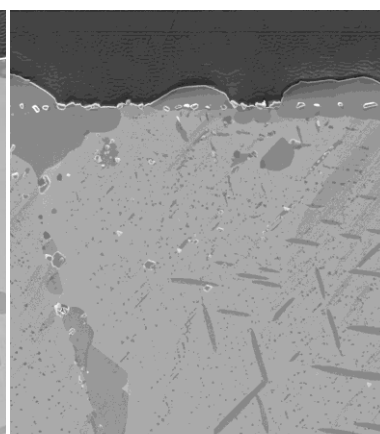
1 μm San70



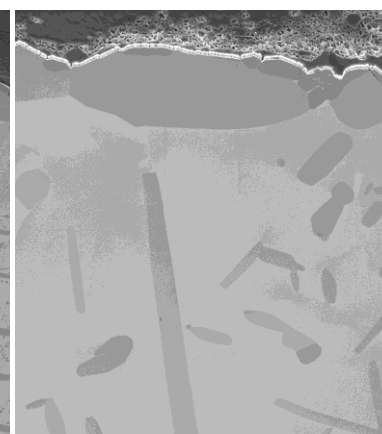
1 μm 253MA



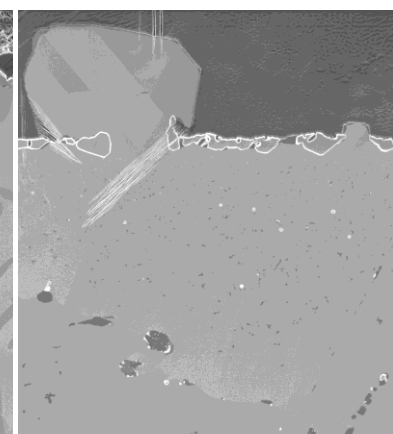
1 μm 310H



1 μm San31HT



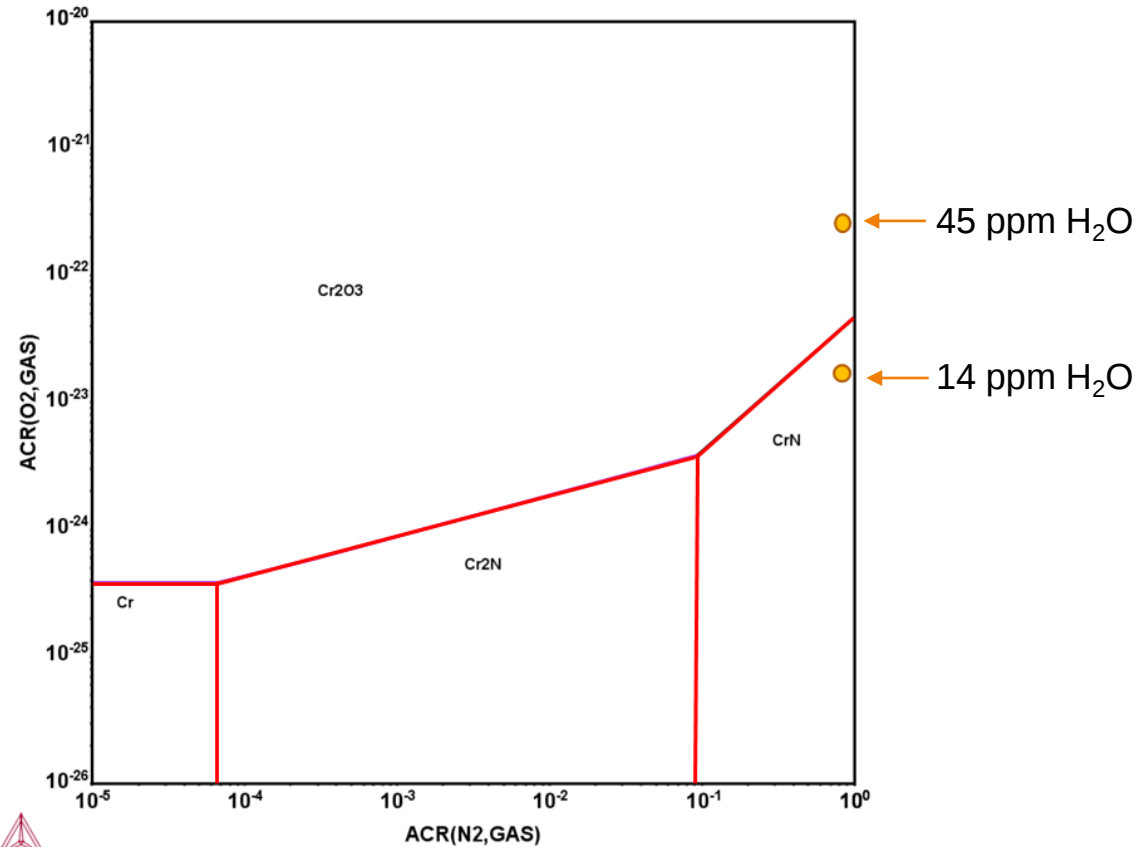
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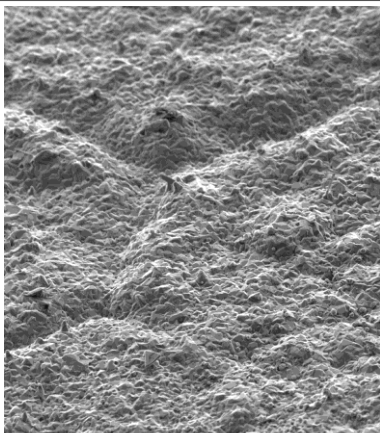


1 μm San70

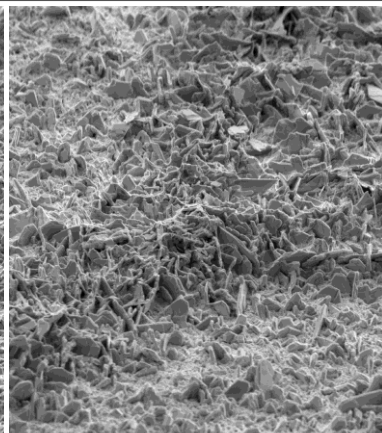
2020-02-27 11:48:09
TCFE10: CR, N, O
P=1E5, N=1, T=1173

Calculated by Sedigheh Bigdeli using TCFE10 database

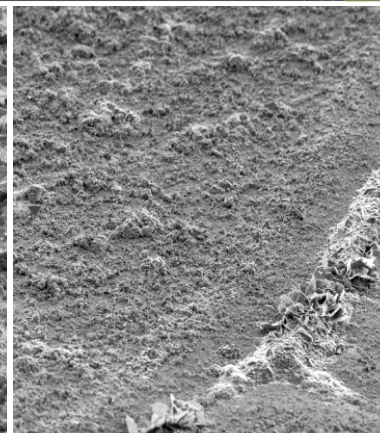




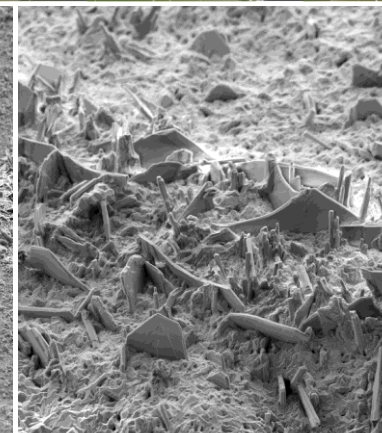
1 μ m 253MA



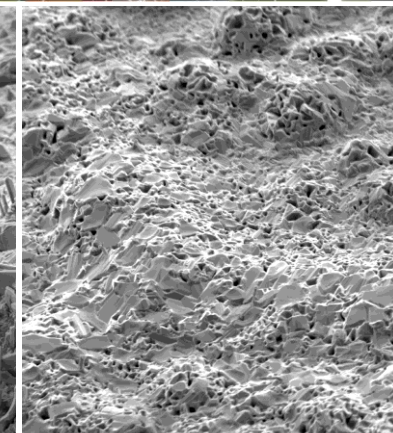
1 μ m 310H



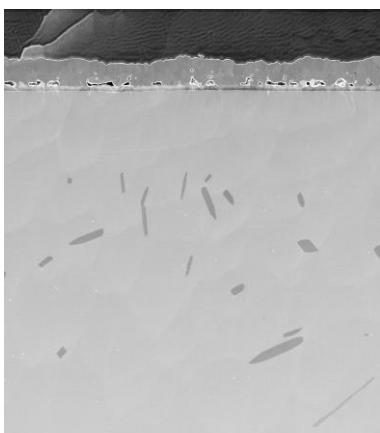
1 μ m San31HT



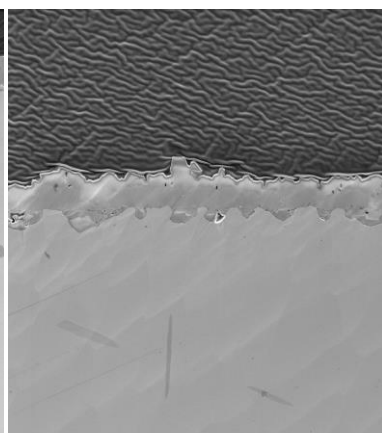
1 μ m 353MA



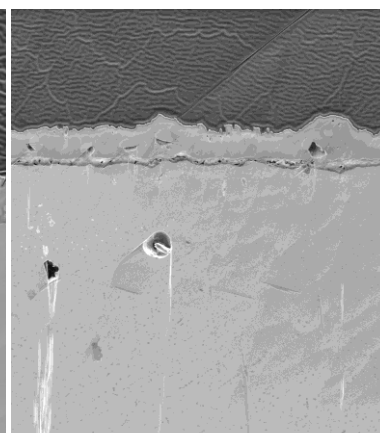
1 μ m San70



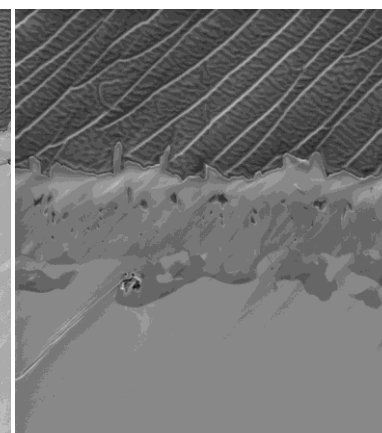
1 μ m 253MA



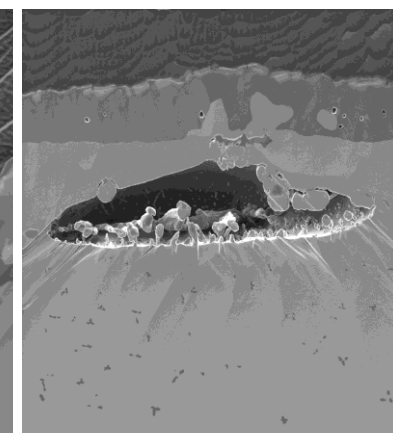
1 μ m 310H



1 μ m San31HT

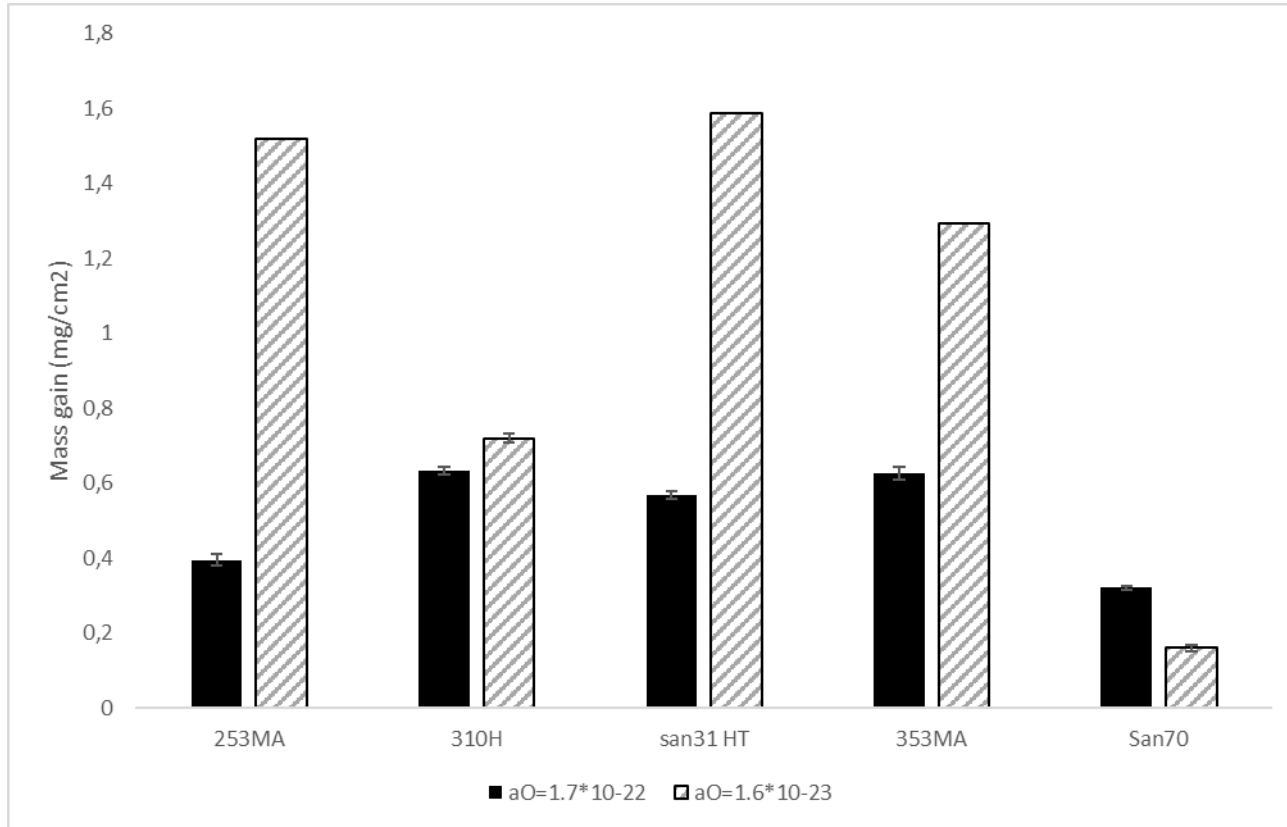


1 μ m 353MA



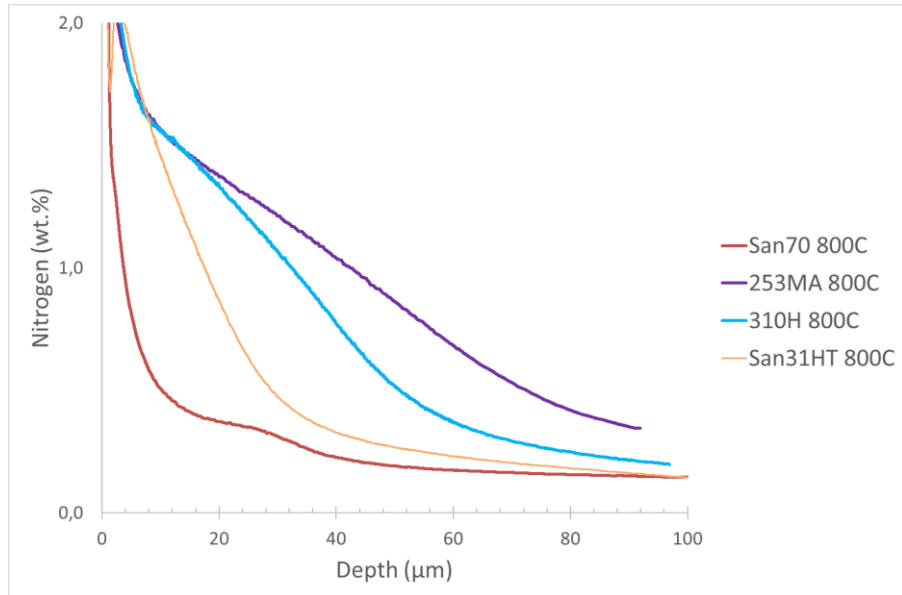
1 μ m San70

Mass gains after 168 h at 900 °C

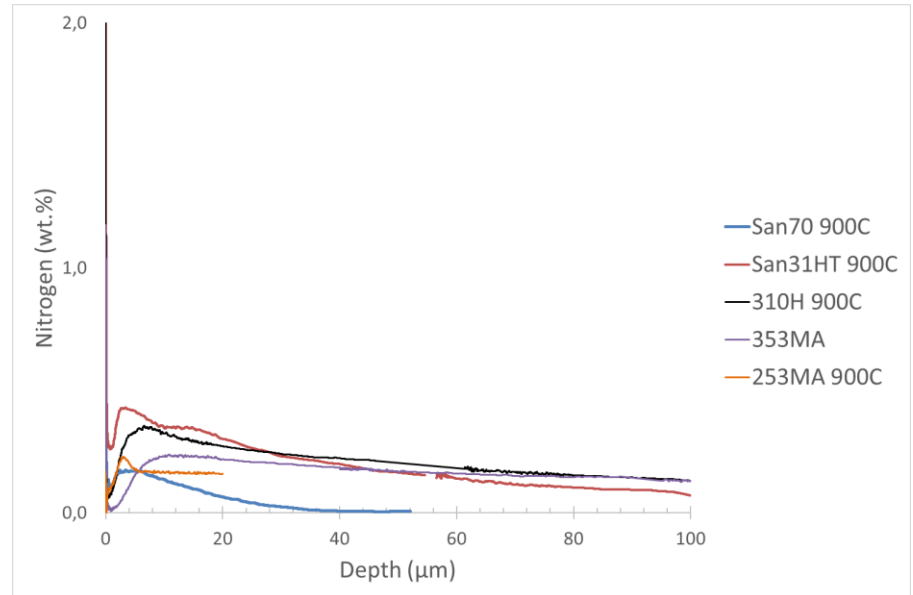


Nitrogen depth profiles by GD-OES

800 °C

 $\text{H}_2\text{O} = 14 \text{ ppm}$ 

900 °C

 $\text{H}_2\text{O} = 45 \text{ ppm}$ 

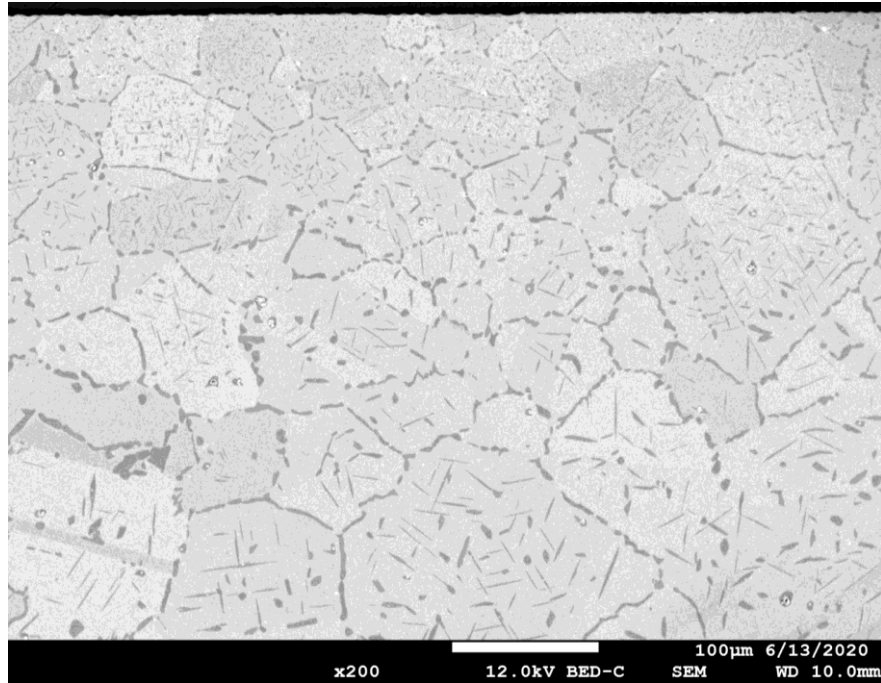
253 MA, 1100° C – outline

- SEM/EDX on BIB cross section
- FIB/SEM lift out from BIB cross section
- TKD on TEM foil
- STEM/EDX on TEM foil
- SEM/EDX on TEM foil

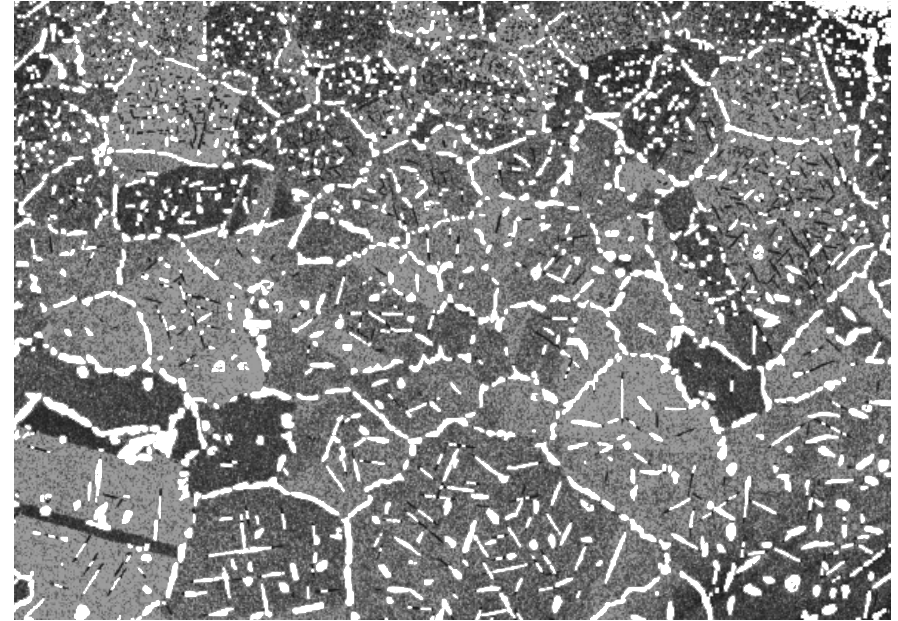
Exposure in 95% N₂, 5% H₂, ppm levels of O₂ for 168h

Cross-section SEM analysis

surface
↓ ↘

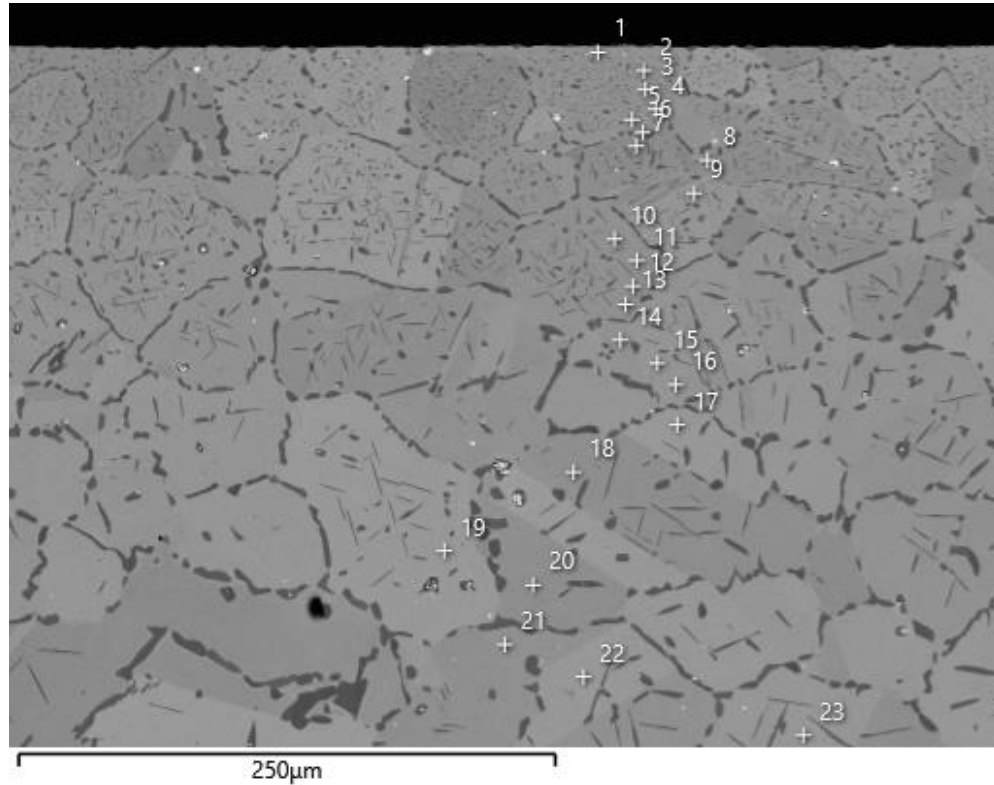
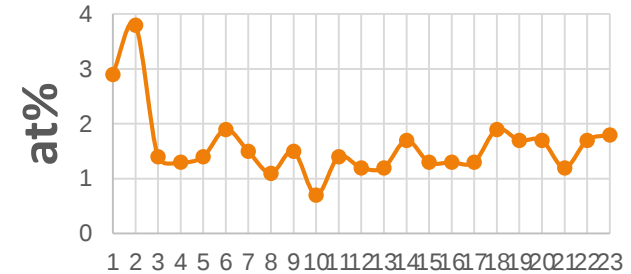
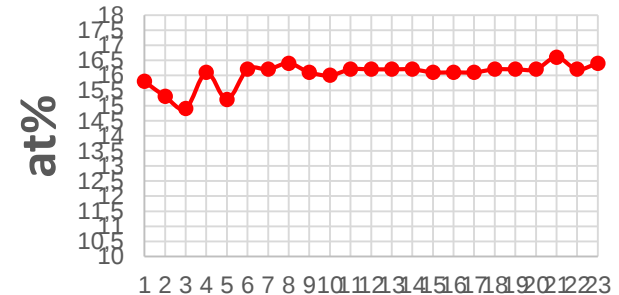


BSE image



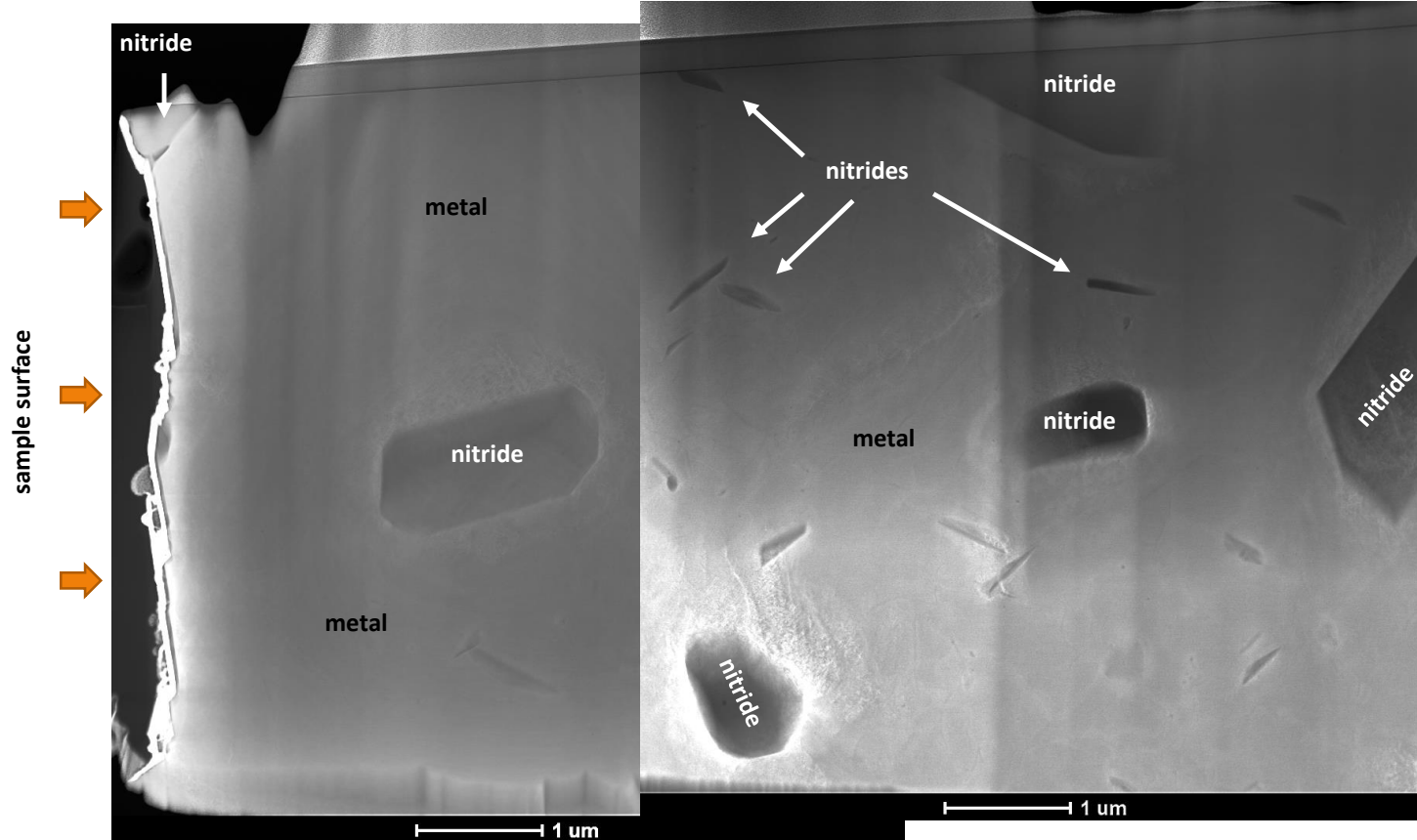
16 area-% nitrides

Cross-section SEM/EDX analysis

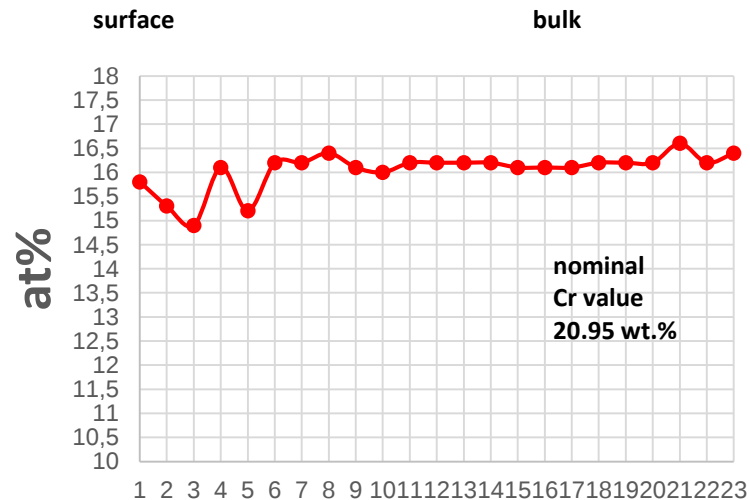
**N-253 MA-1100 C****Cr-253 MA-1100 C**

←
nominal
Cr value
20.95 wt.%

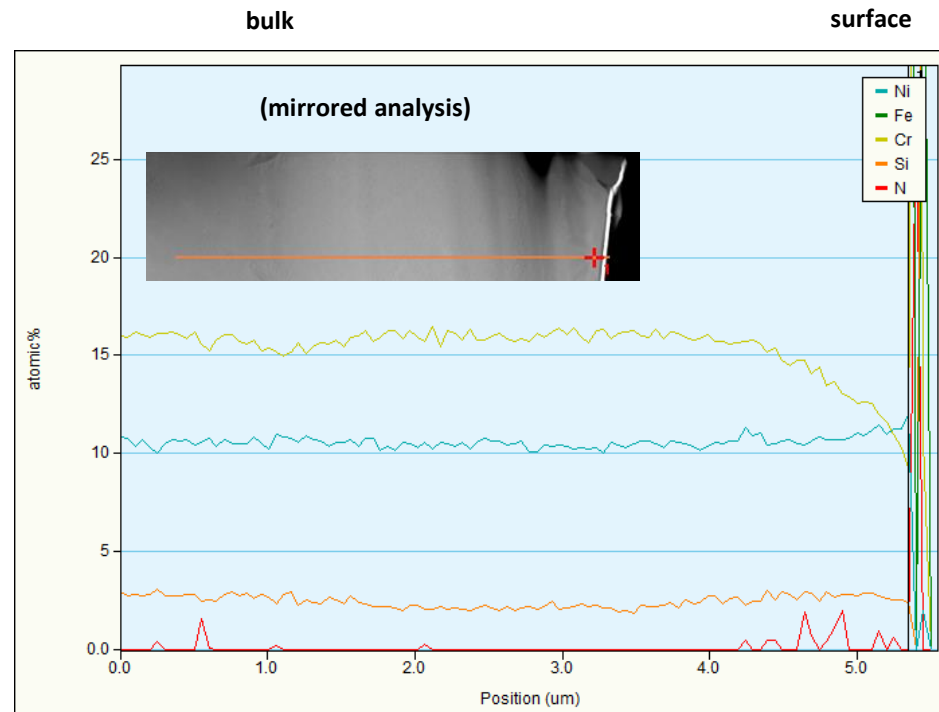
STEM HAADF from BIB cross-section



Depth profile



SEM/EDX

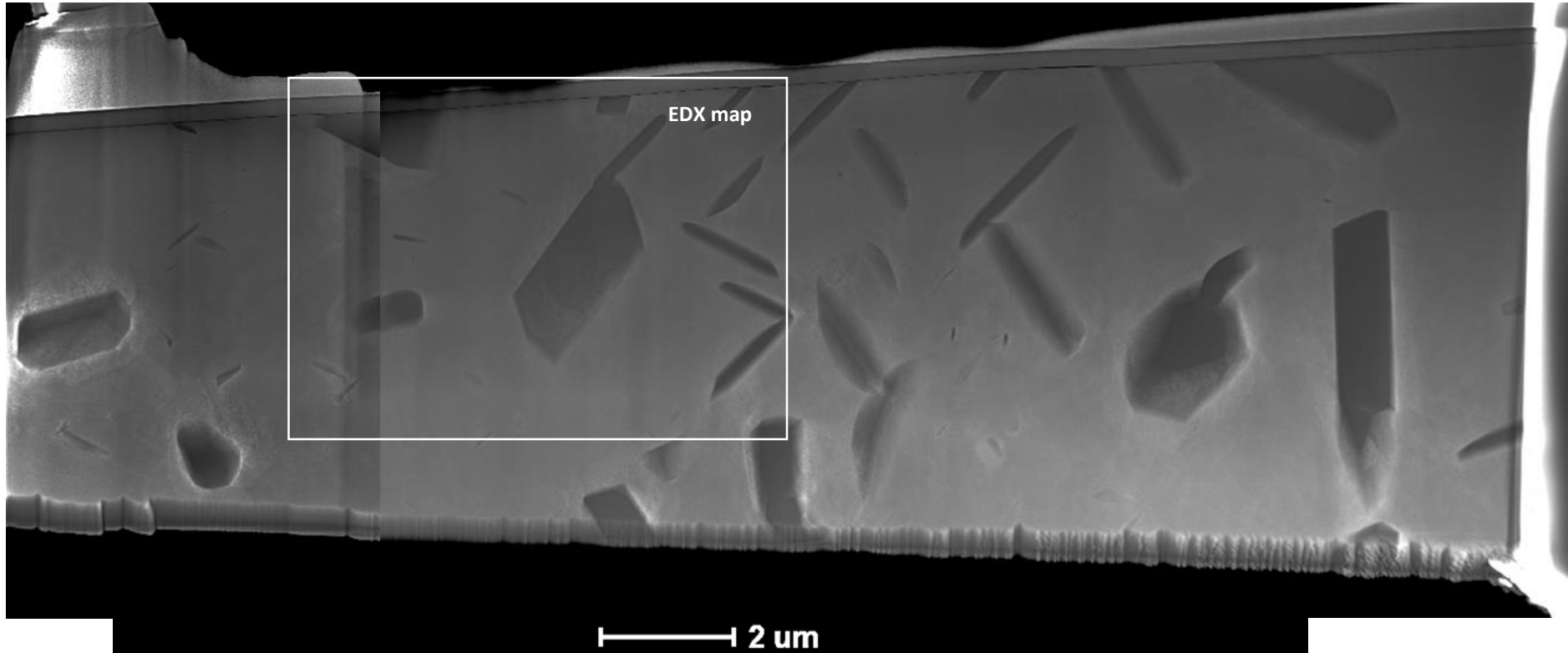


STEM/EDX

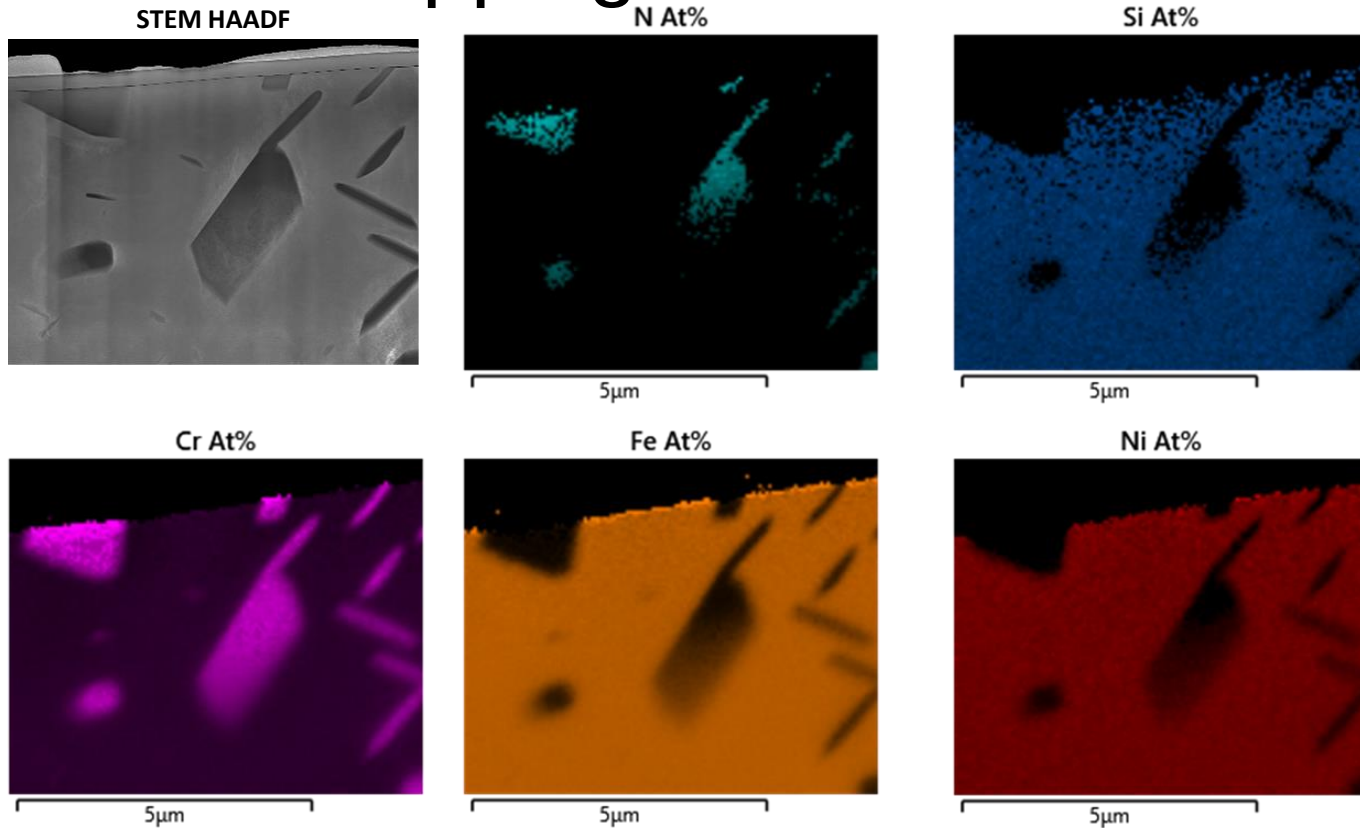
STEM HAADF overview

← surface outside
this micrograph

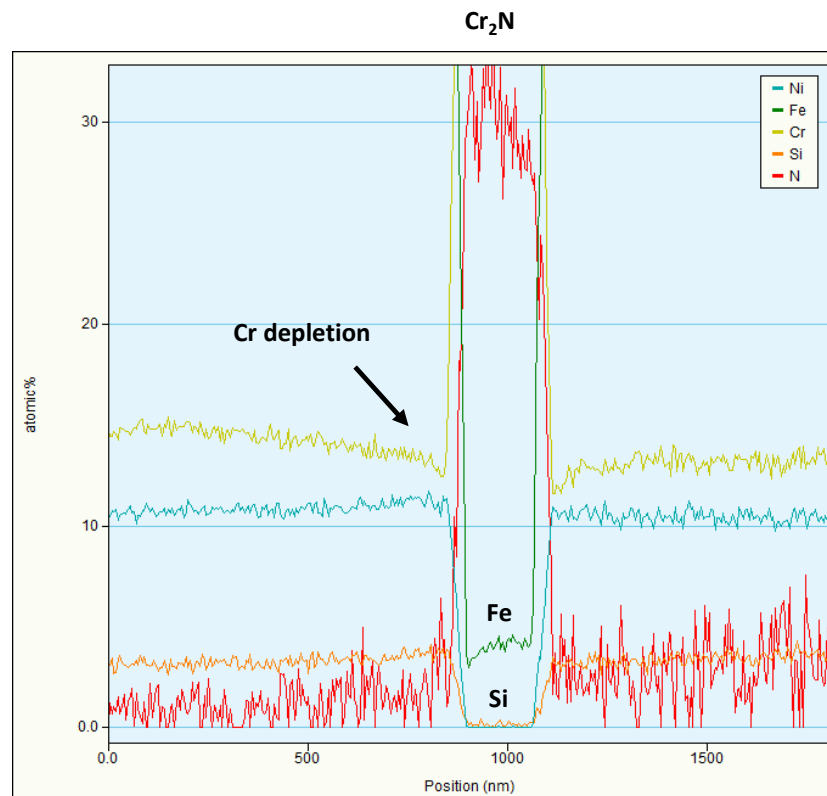
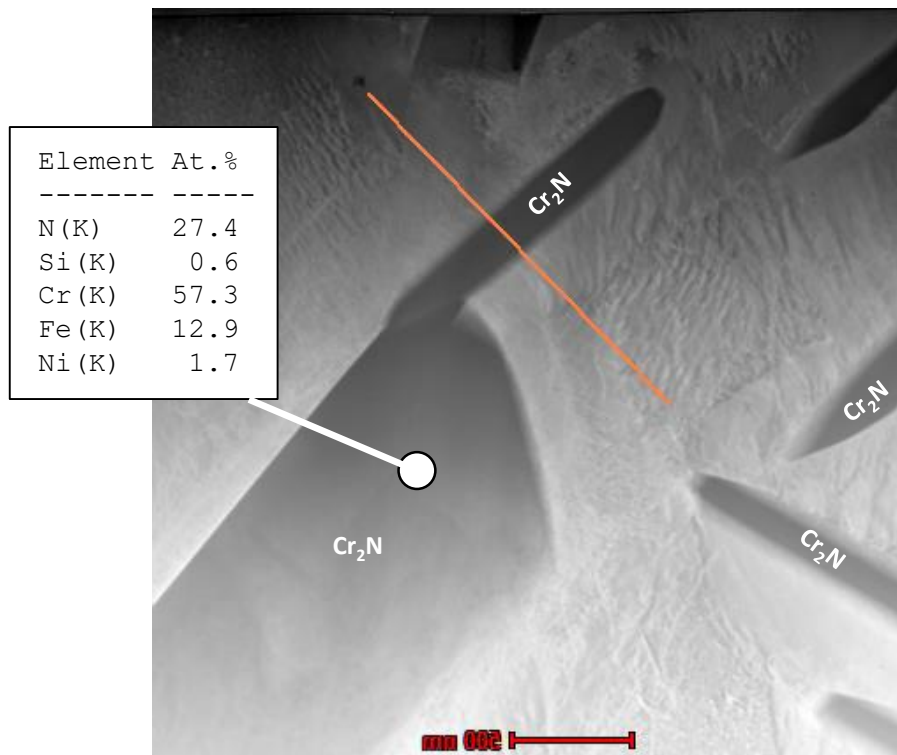
into the bulk
→



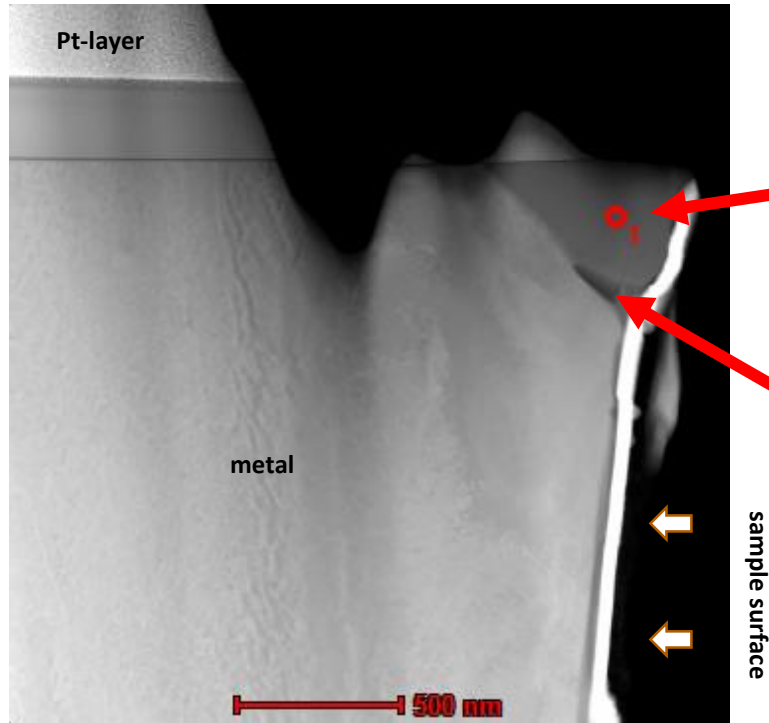
SEM/EDX mapping



EDX linescan profile



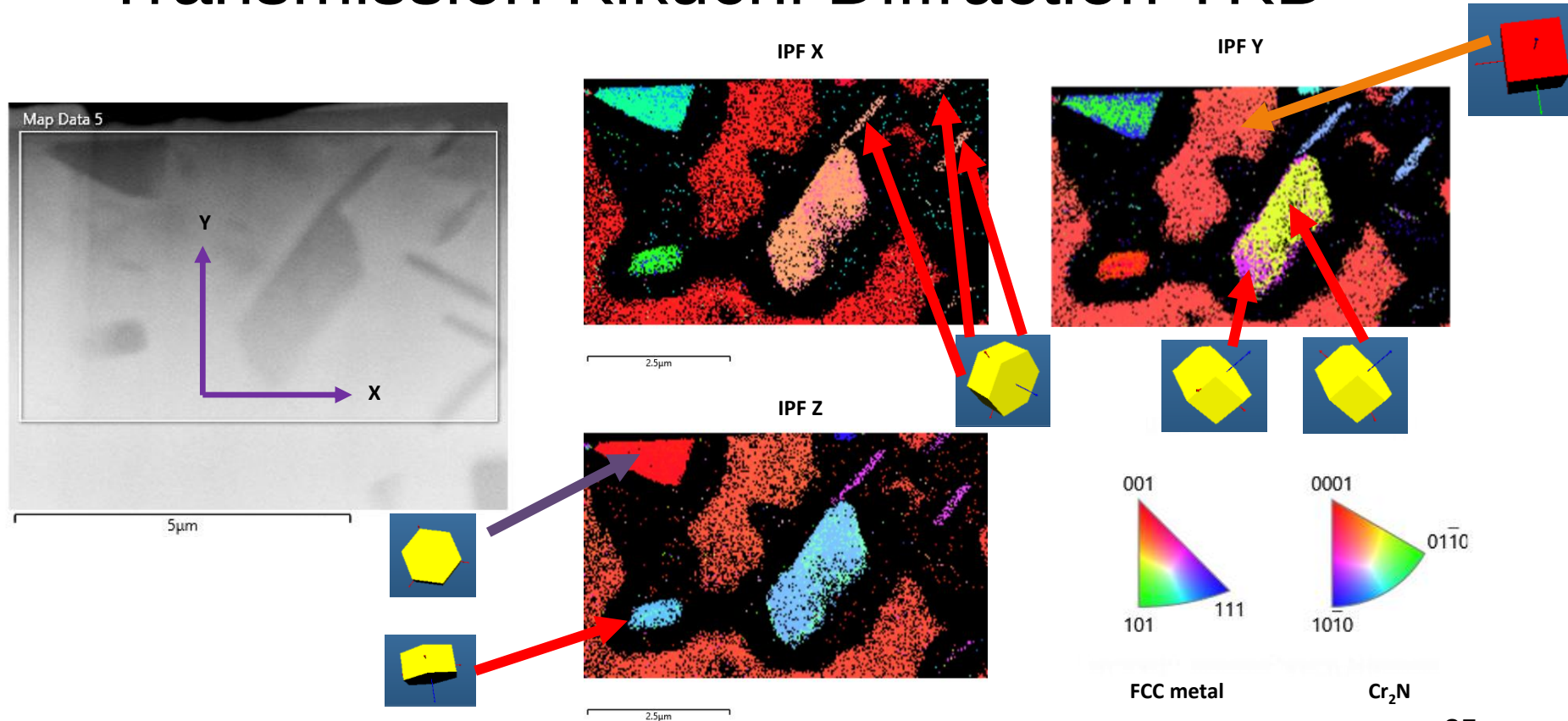
Surface features



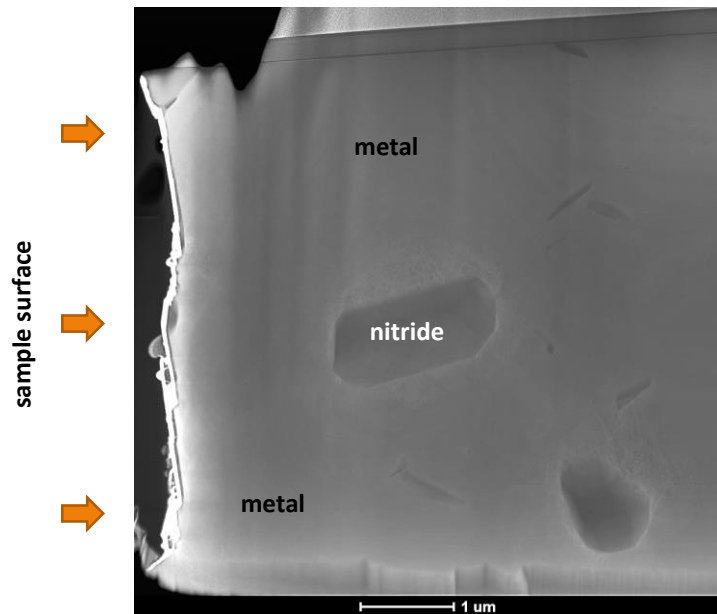
Element	Weight %	Atomic %
N (K)	18.34	45.58
Cr (K)	76.23	51.03
Fe (K)	5.42	3.37

Element	Weight %	Atomic %
N (K)	8.40	16.40
O (K)	23.16	39.56
Si (K)	18.22	17.73
Cr (K)	47.60	25.01
Fe (K)	2.60	1.27

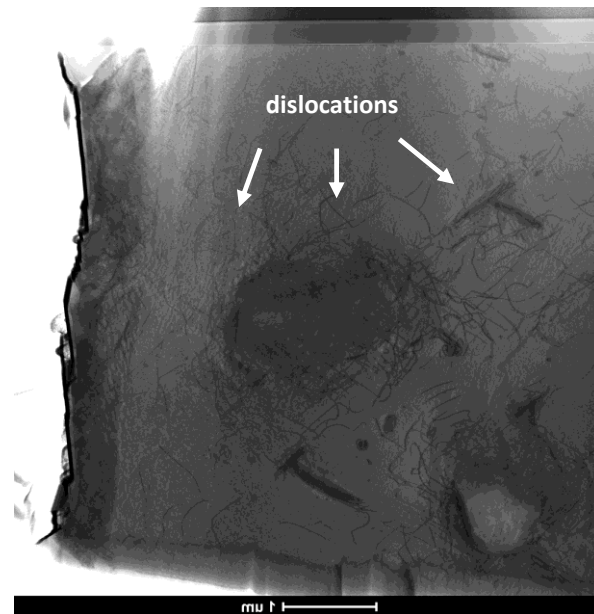
Transmission Kikuchi Diffraction TKD



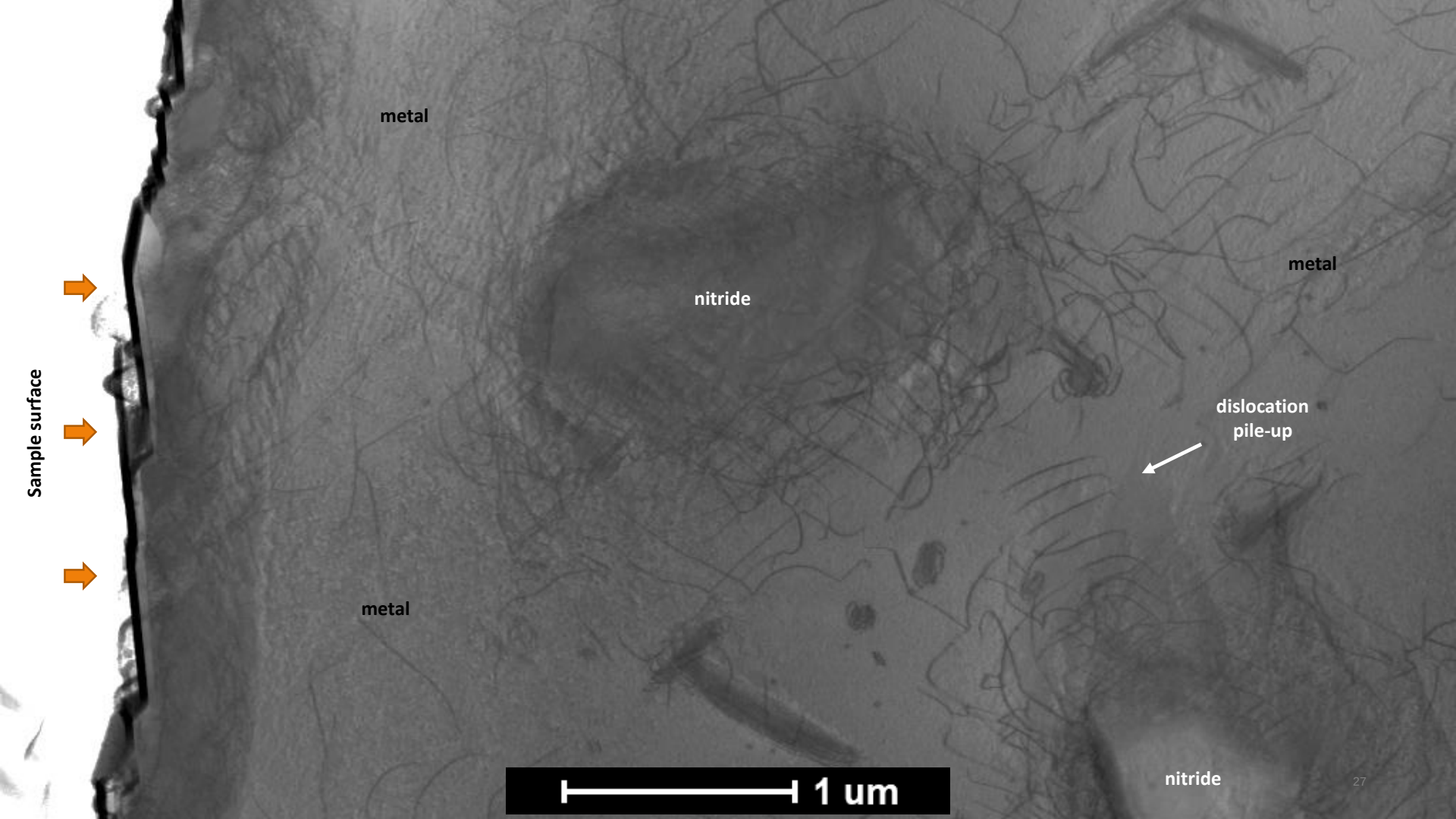
Defect formation



STEM HAADF



STEM BF



Sample surface

metal

nitride

metal

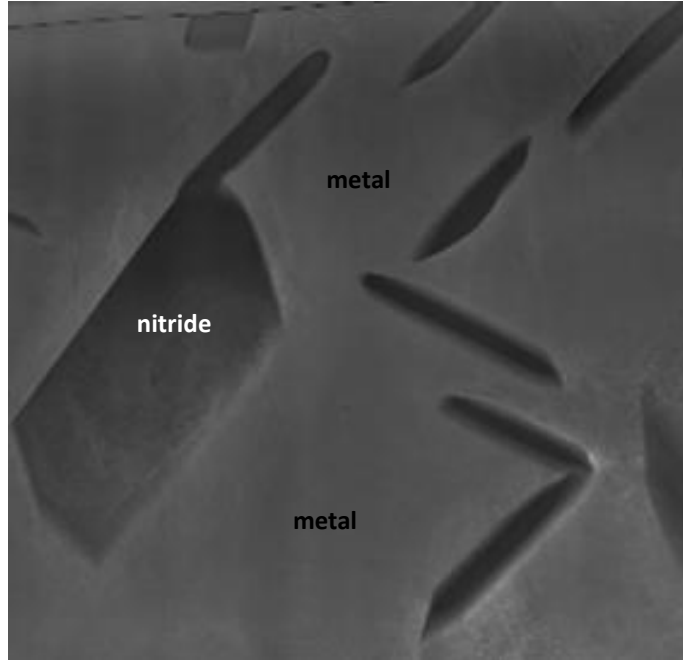
dislocation
pile-up

metal

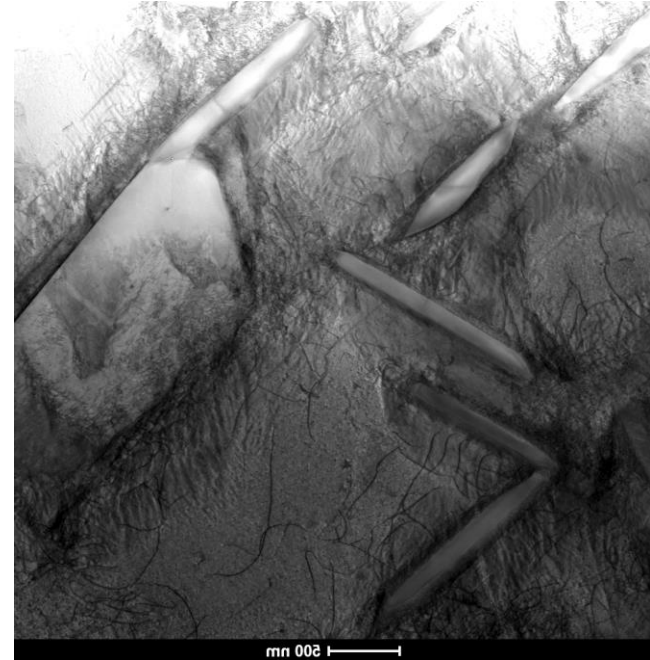
nitride

1 μm

Dislocation networks

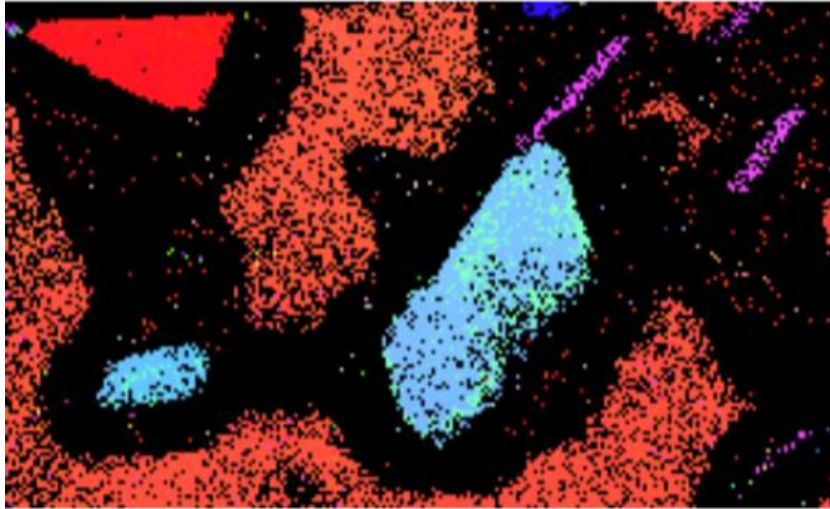


STEM HAADF

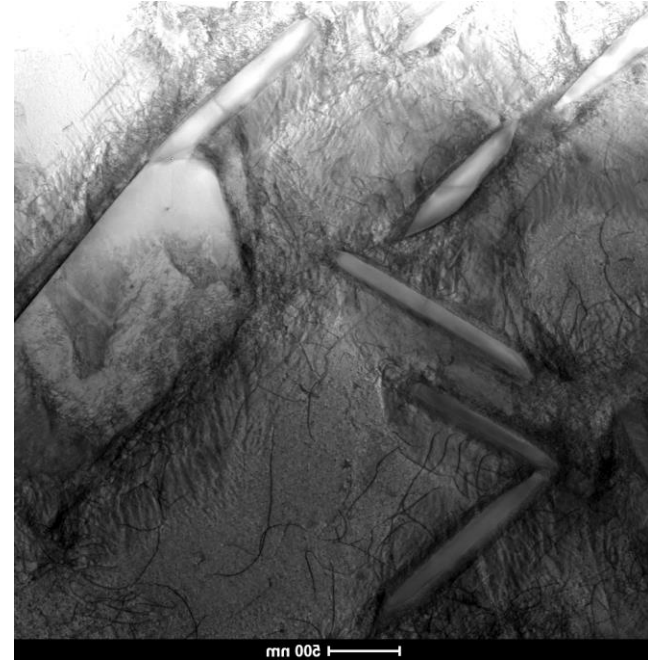


STEM BF

Defect strain – lower TKD indexing level

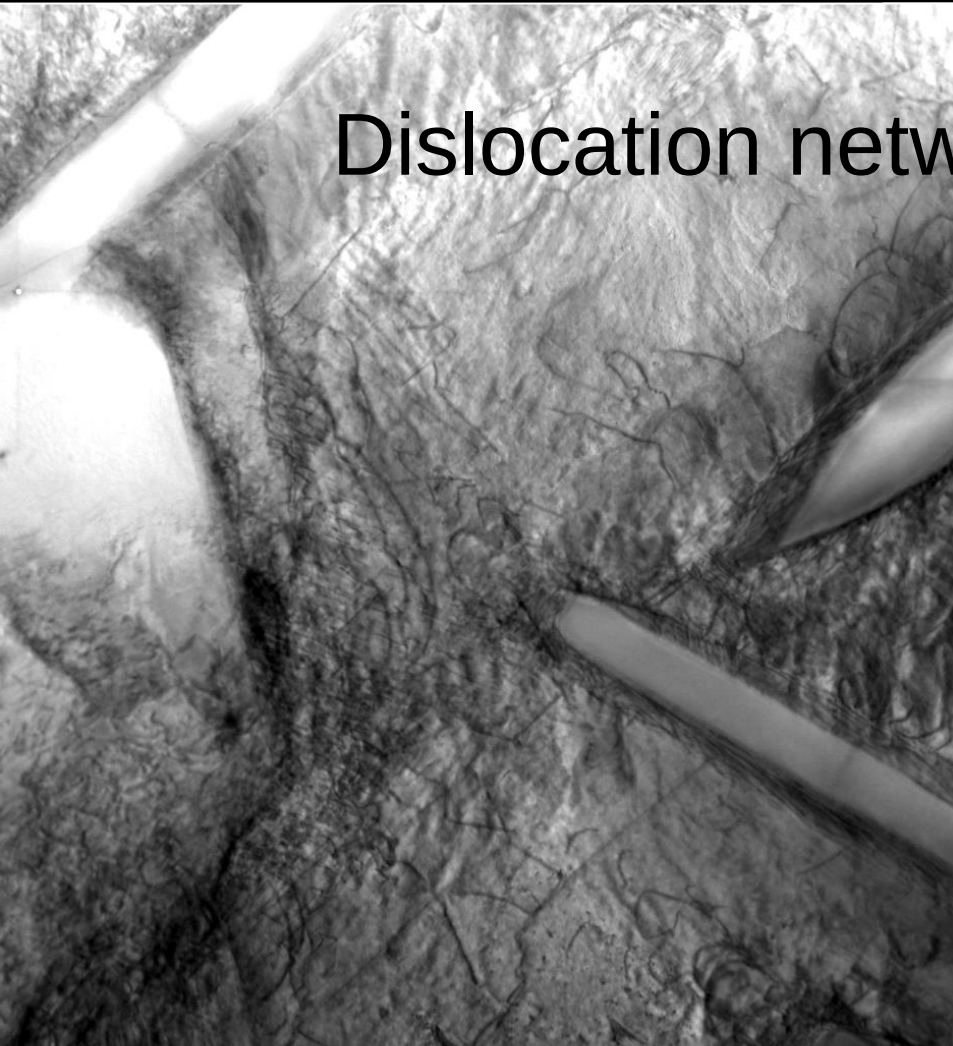


TKD map

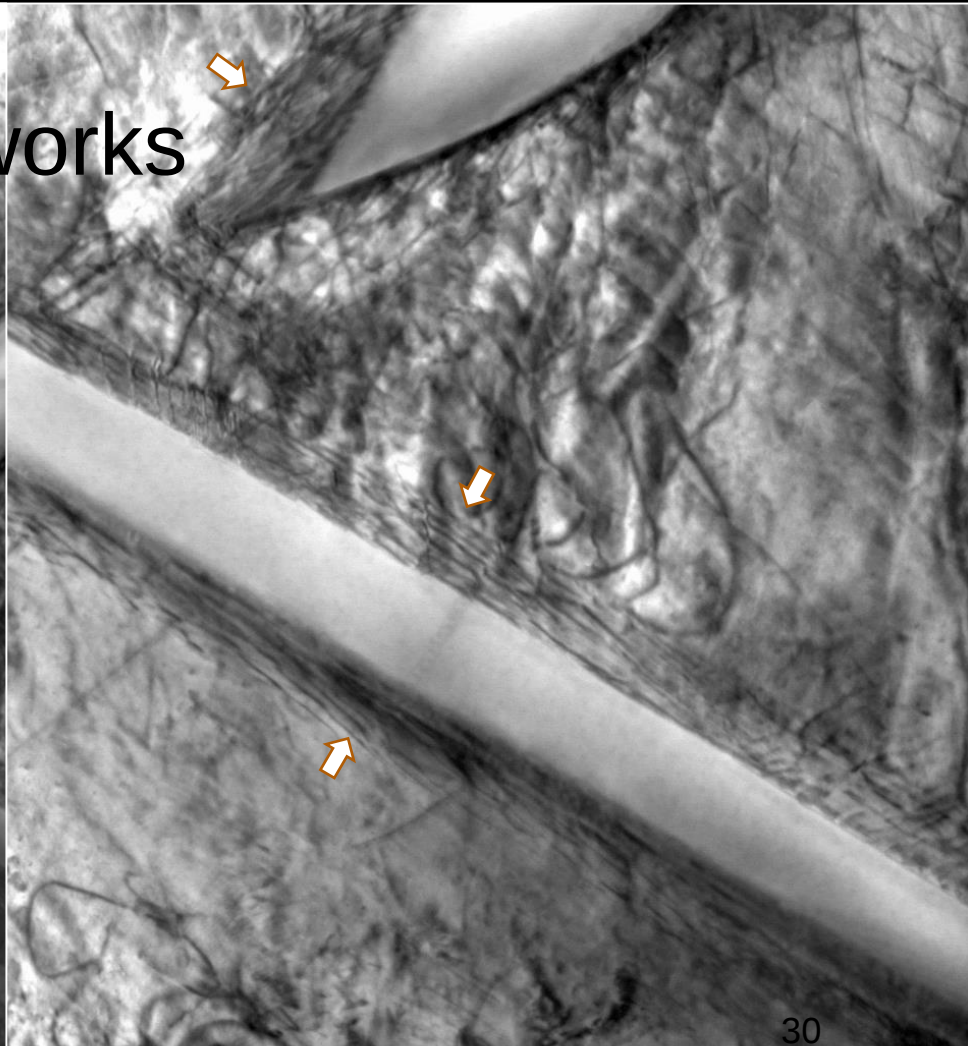


STEM BF

Dislocation networks



500 nm



500 nm