Preliminary Investigation of Solute Segregation to γ / α Interfaces

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Literature Review:

- Most of the literature on segregation is focussed on grain-boundaries.
- Some of the best data on segregation to grain boundaries in Fe-X and Fe-C-X comes from the work of Enomoto et al. [e.g. Met. Trans. 19A, 1807]

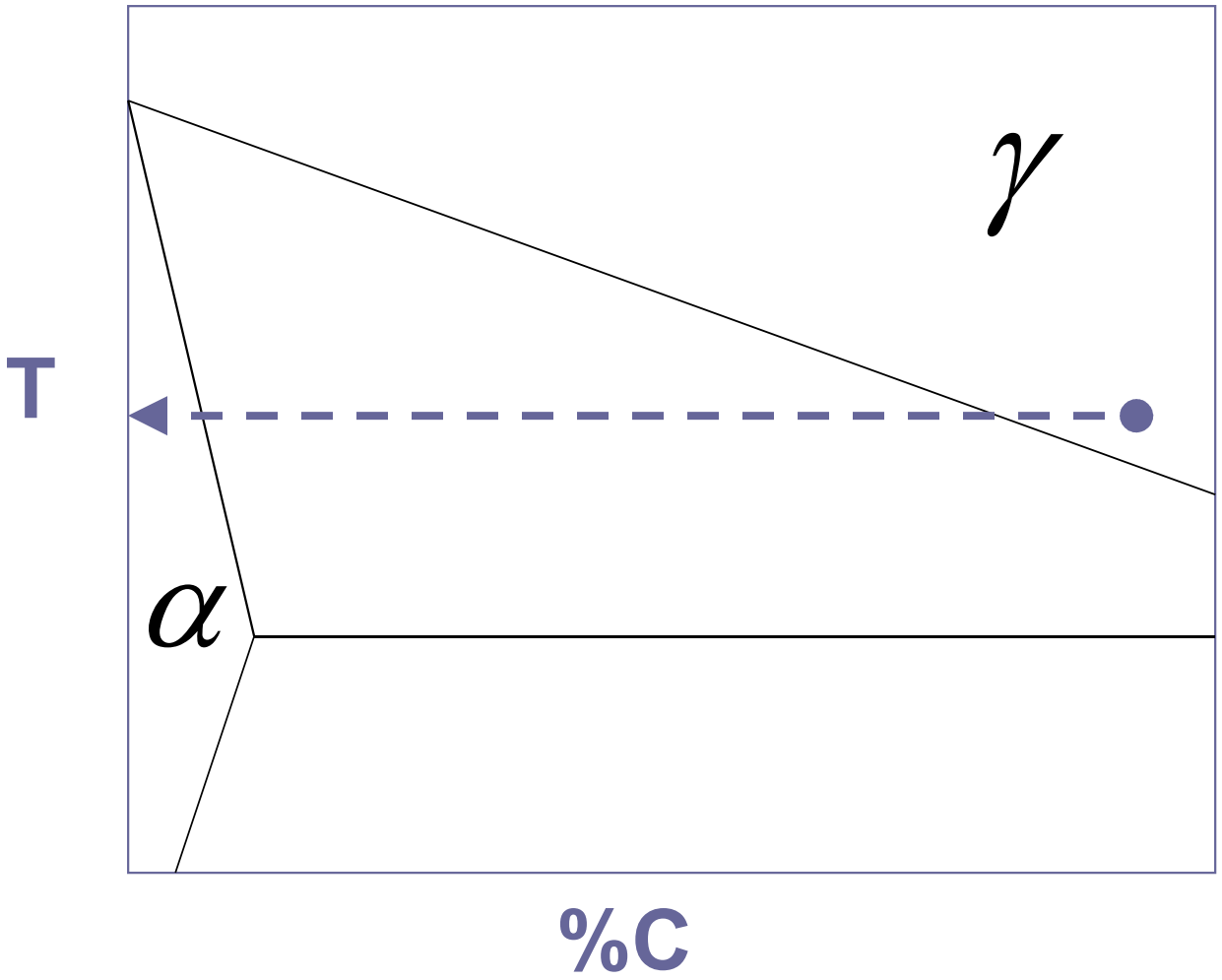
kJ/mole	**Austenite G.B.**	**Ferrite G.B.**
Mn	-8 +/- 3 (Enomoto et al.)	-8 +/- 4 [Kaufman]
Ni	Small (Enomoto et al.)	Small
Si	-23+/- 6 (Enomoto et al.)	-7 kJ/mole [Lejcek]
Mo	-15+/- 3 (Enomoto et al.)	-28 +/- 2 (Murayama & Smith)

- In recent years, an increasing number of studies have examined segregation to ferrite/austenite interfaces.
  - Enomoto et al. [MSEA 343, 151]
  - Guo et al., [Met. Trans. A, 37A, 1723]
  - Danoix and Goune [Alemi 2009].

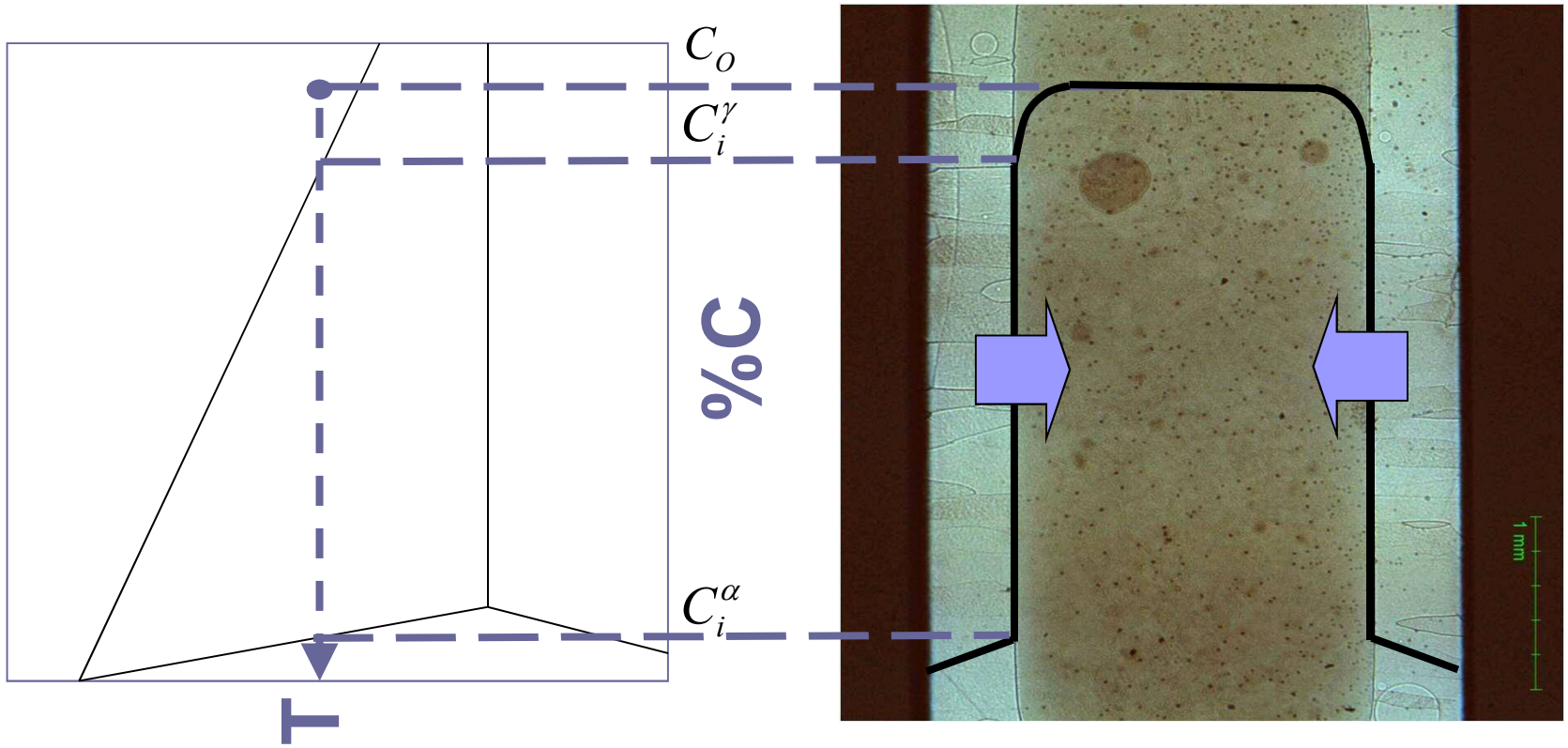
- One of the key findings of earlier work is that there is variation between the level of segregation measured at different boundaries.
  - Possibly due the structure of the boundary.
  - Possibly due to the velocity of the boundary.

Decarburization Method

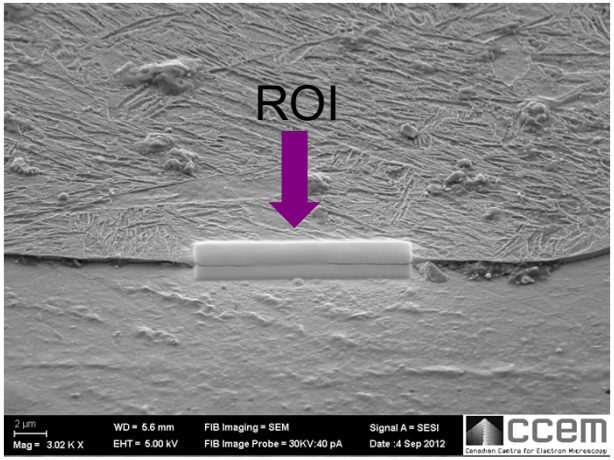
The Decarburization Method

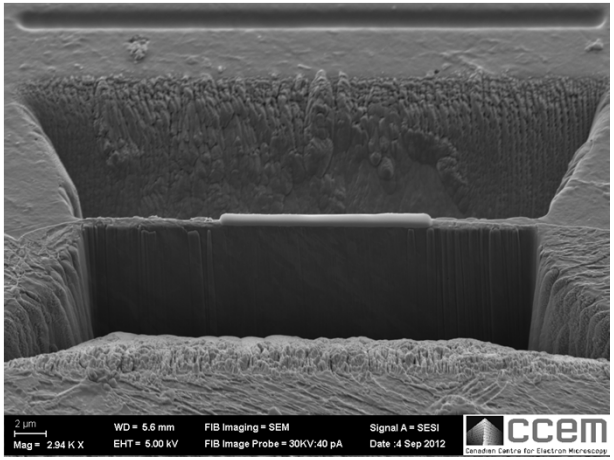


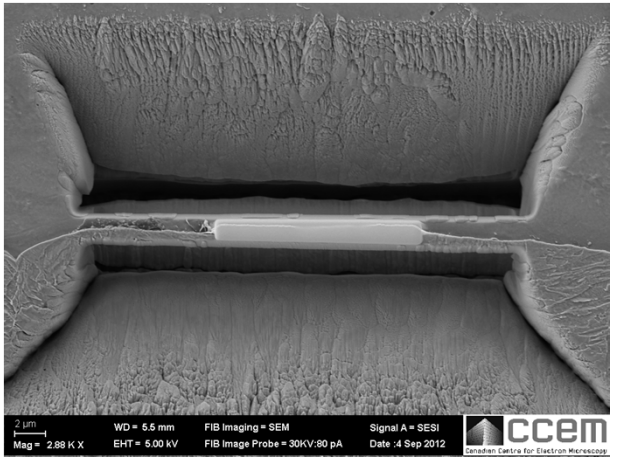
The Decarburization Method

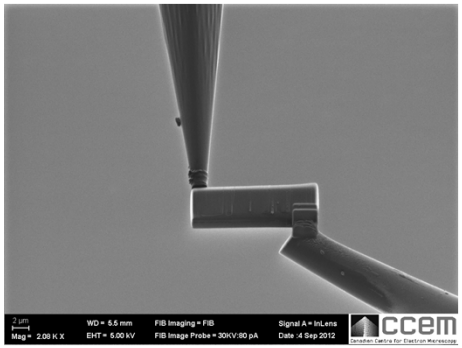


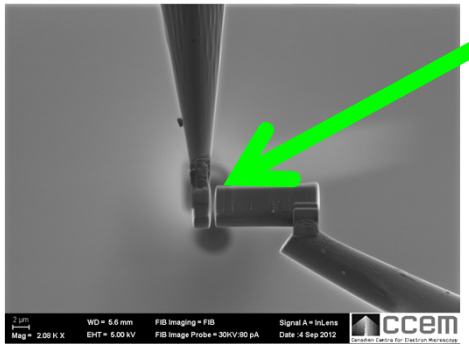
FIB specimen preparation – lift-out

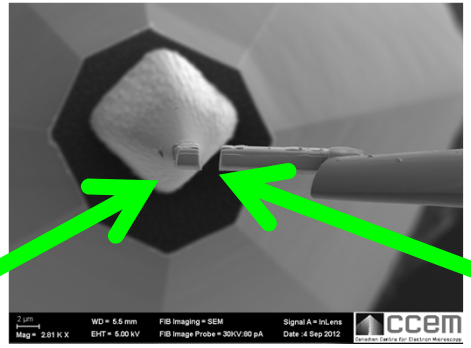


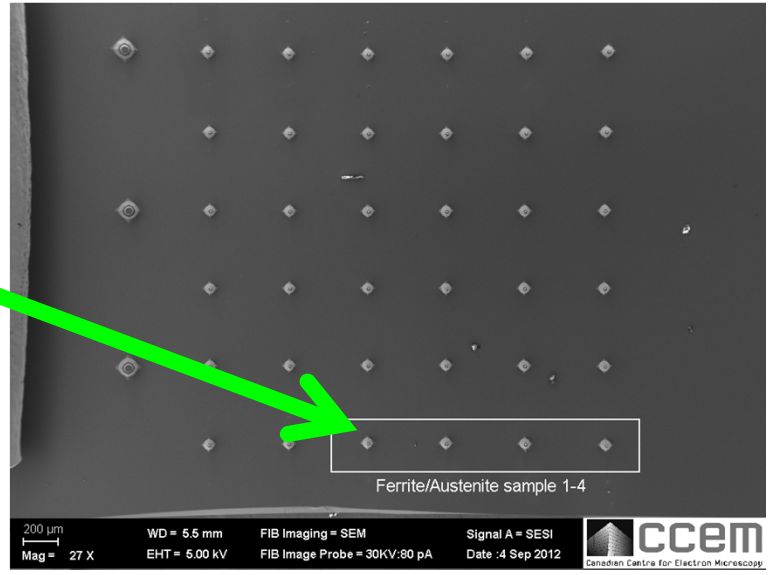




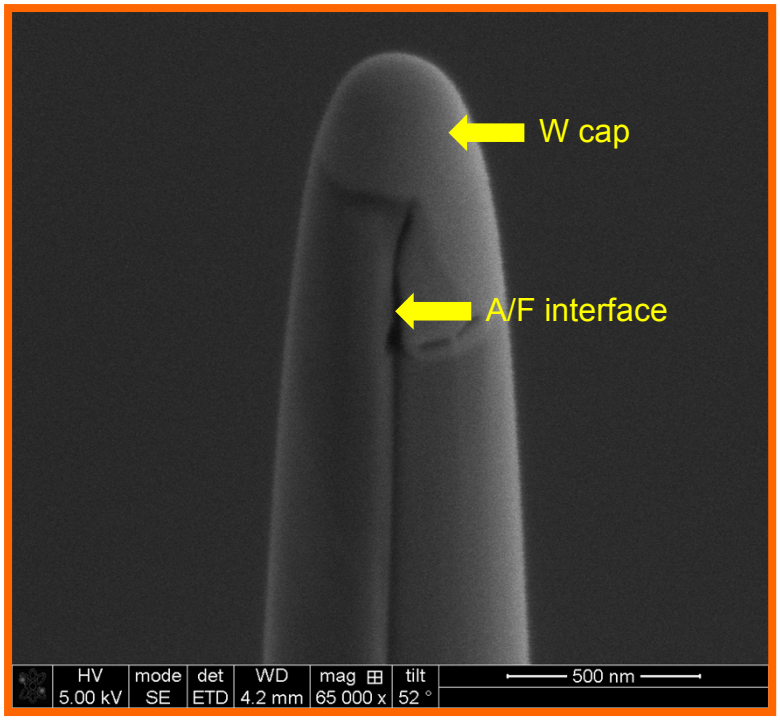


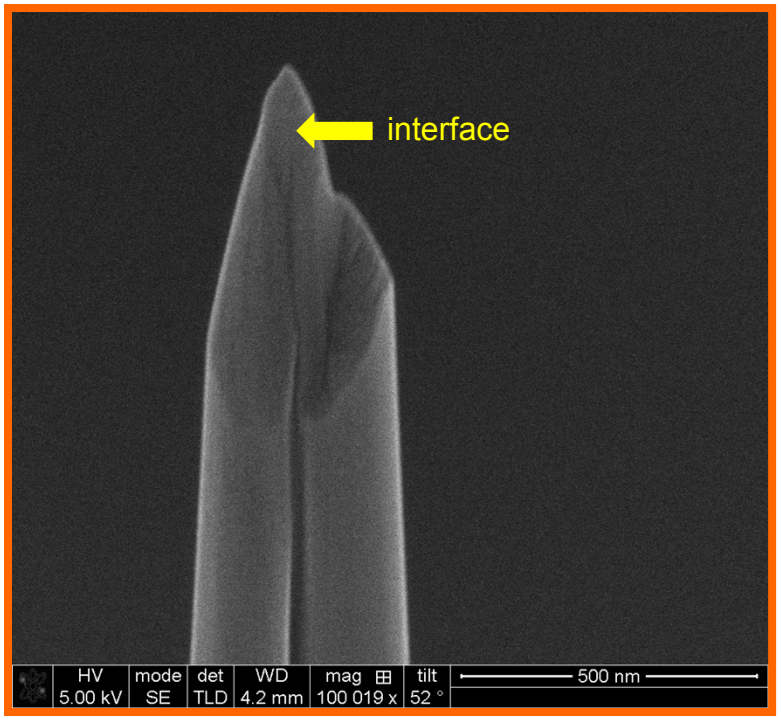






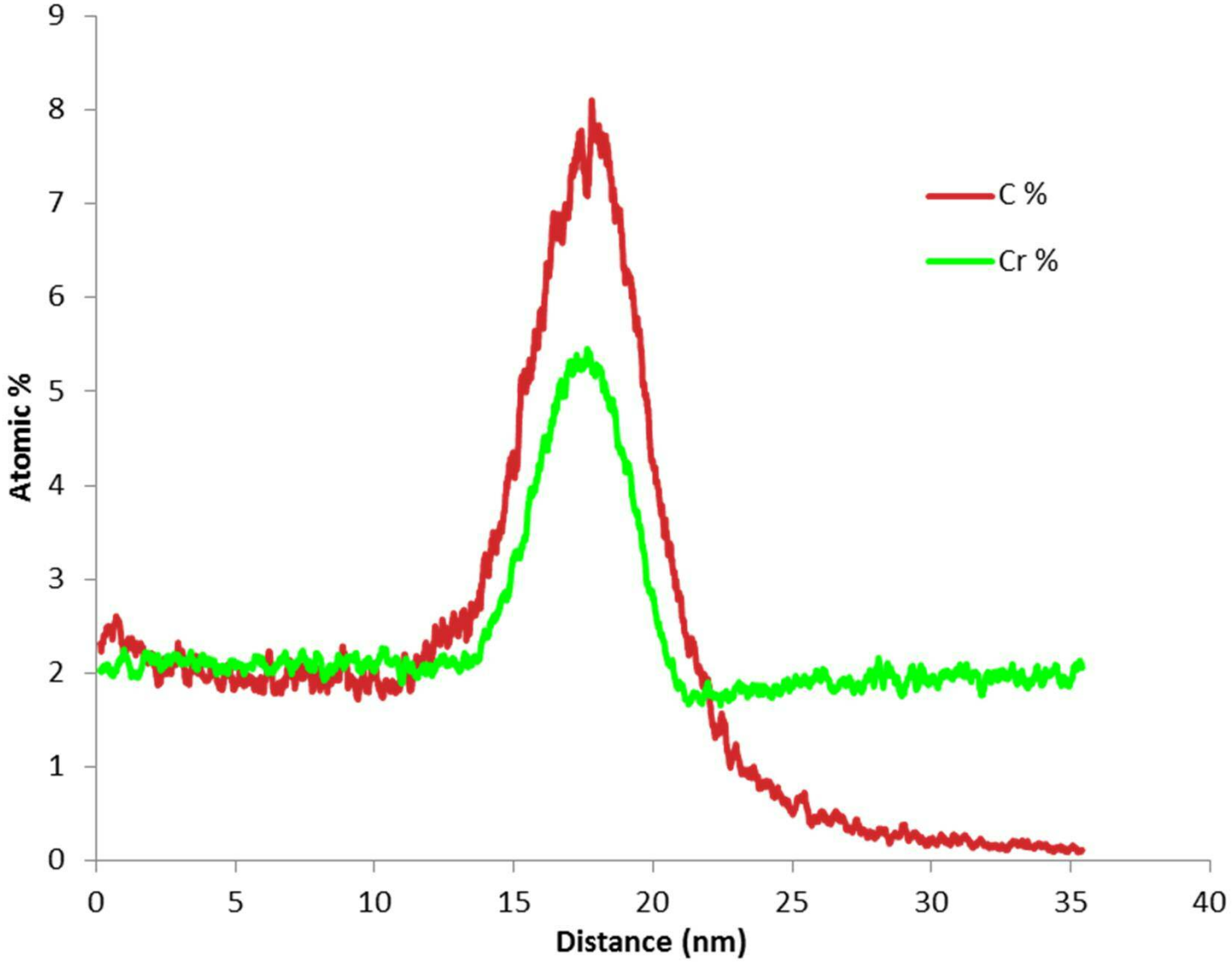
FIB specimen preparation – sharpening of atom probe specimens

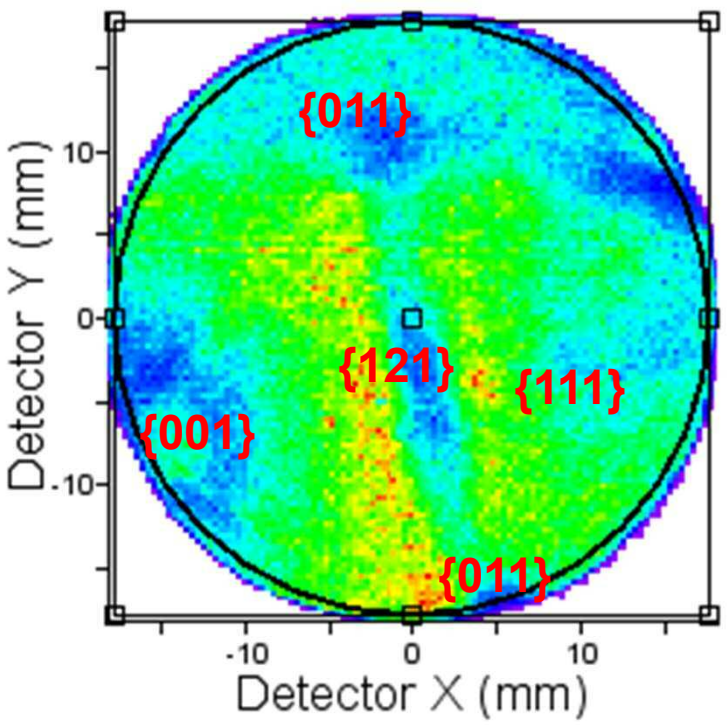




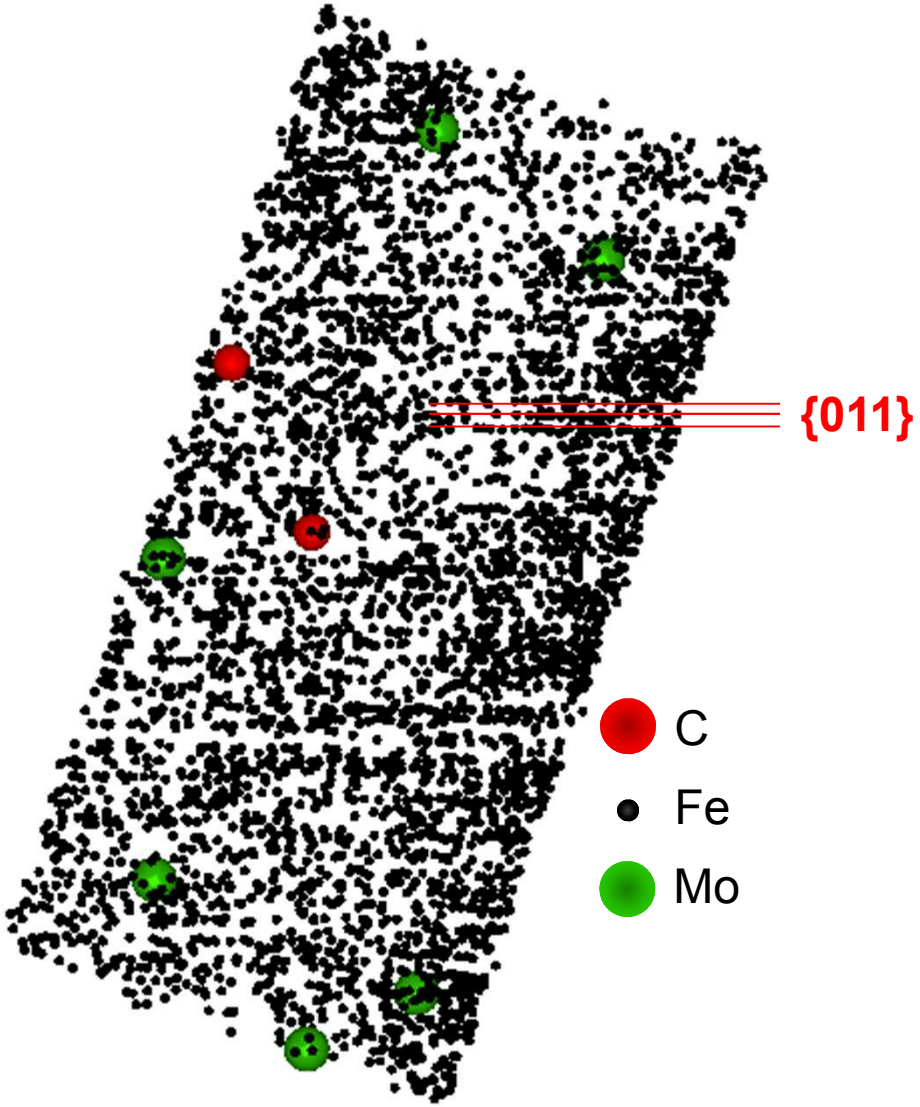
2. Preliminary Results

Fe-2%Cr-C: 806°C- 128min, *purged*.



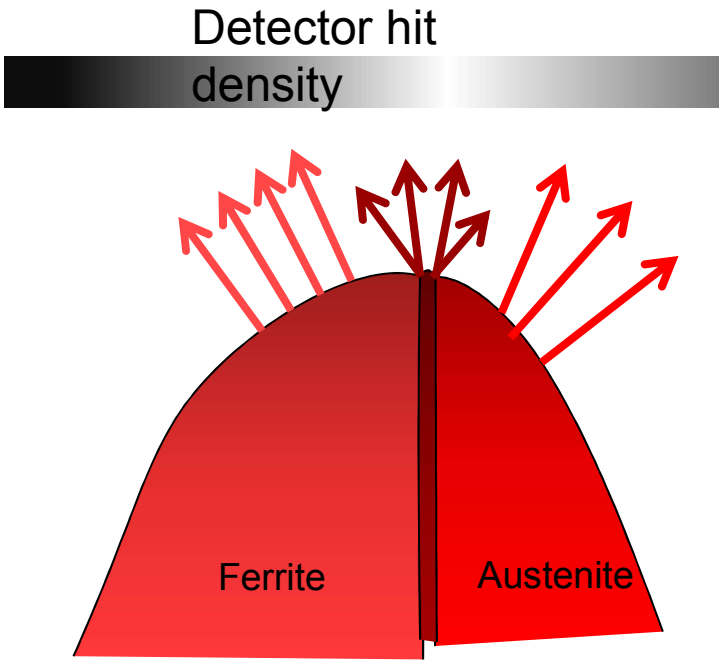


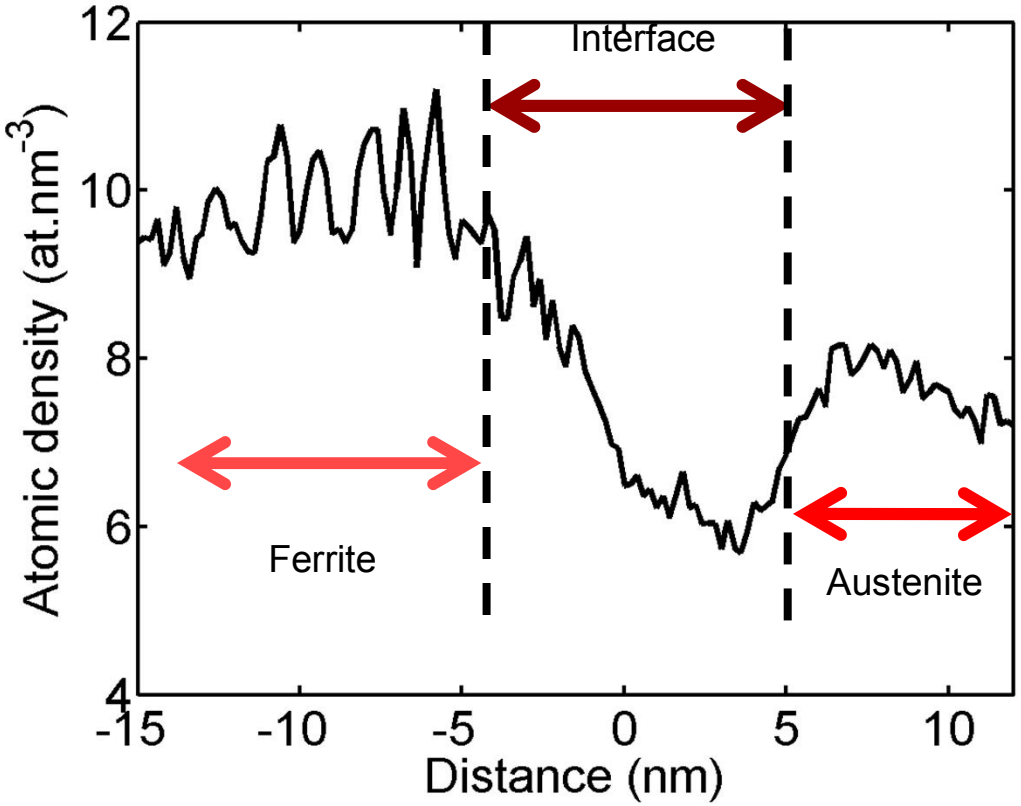
Detector histogram of the first 5M hits;
Clear pole figure from the BCC ferrite!
Atomic planes can be seen in the data
and used to calibrate the depth



Trajectory aberrations / spatial distortions

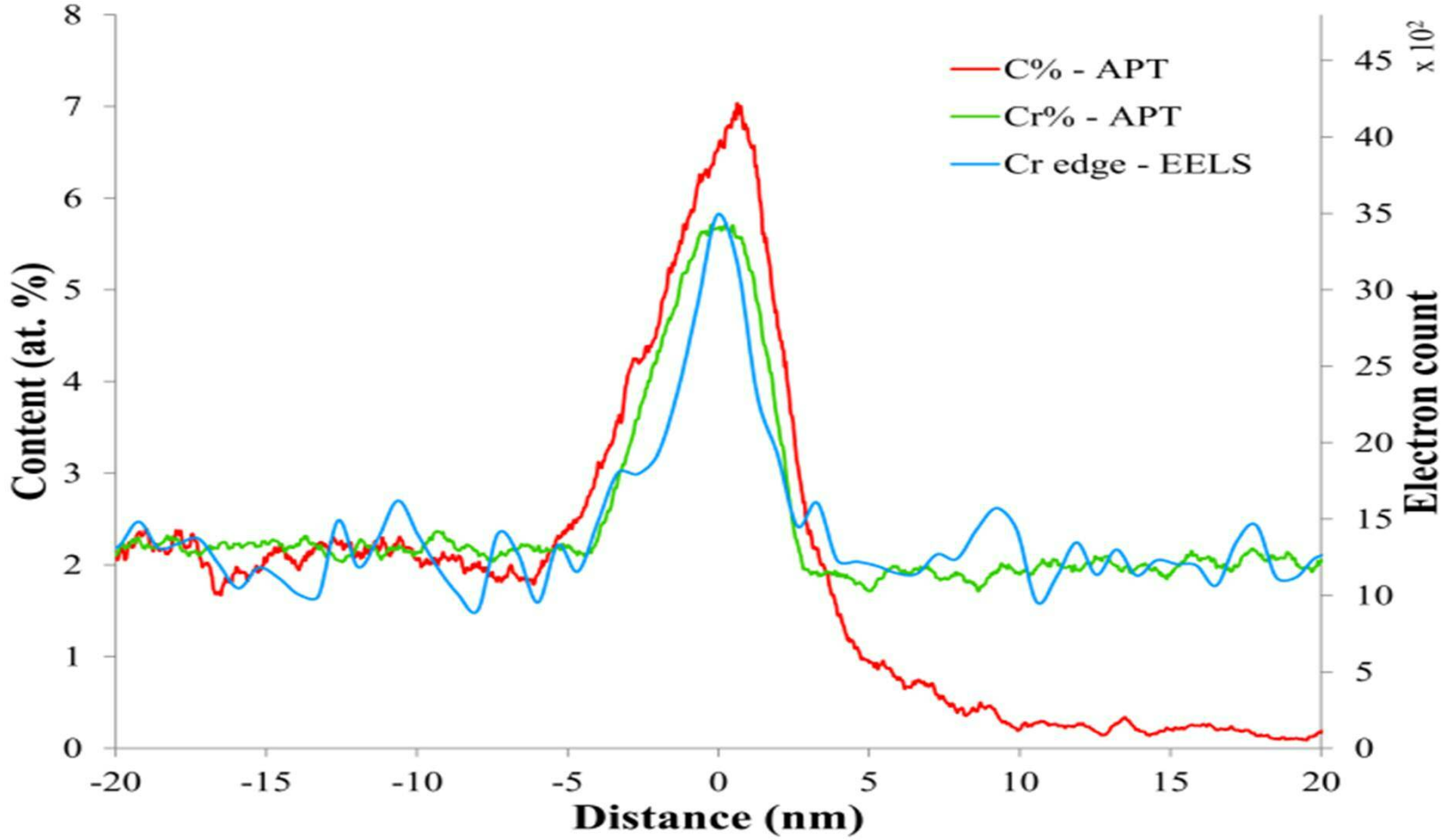
Since both the local composition and crystalline structure change from one side of the specimen to the other, the specimen develops different curvatures across its cross section, which translate into different magnifications and hence differences in the local atomic density within the reconstruction that can be tracked with a density profile.





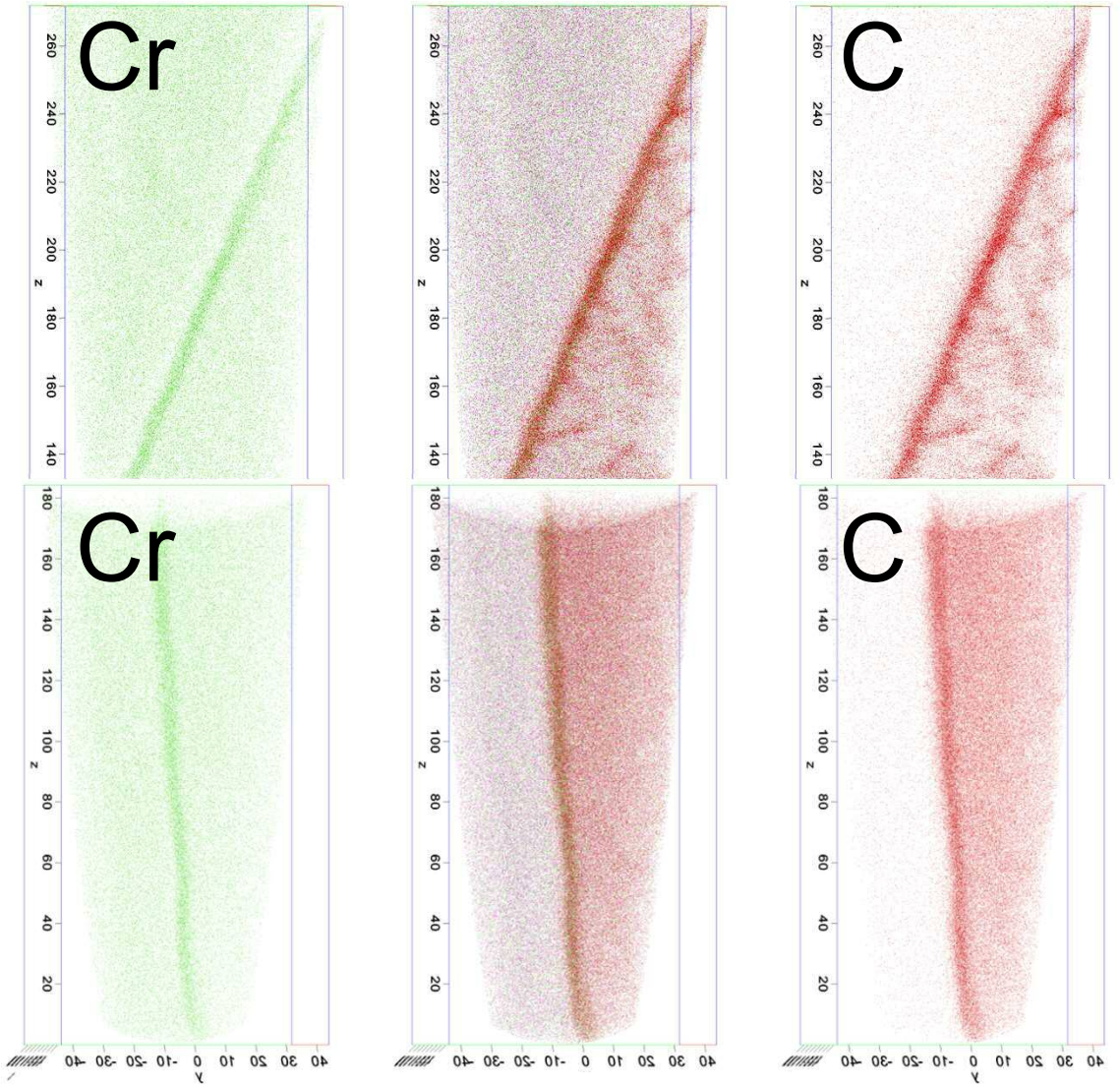
Gault et al. Ultramicroscopy, **111** 683-689 (2011) proposed a correction protocol for composition profiles to account for these variations. Subsequent profiles have been plotted using this correction, which allows for more accurate estimations of features size.

Fe-2%Cr-C: 806°C- 128min, *purged*.

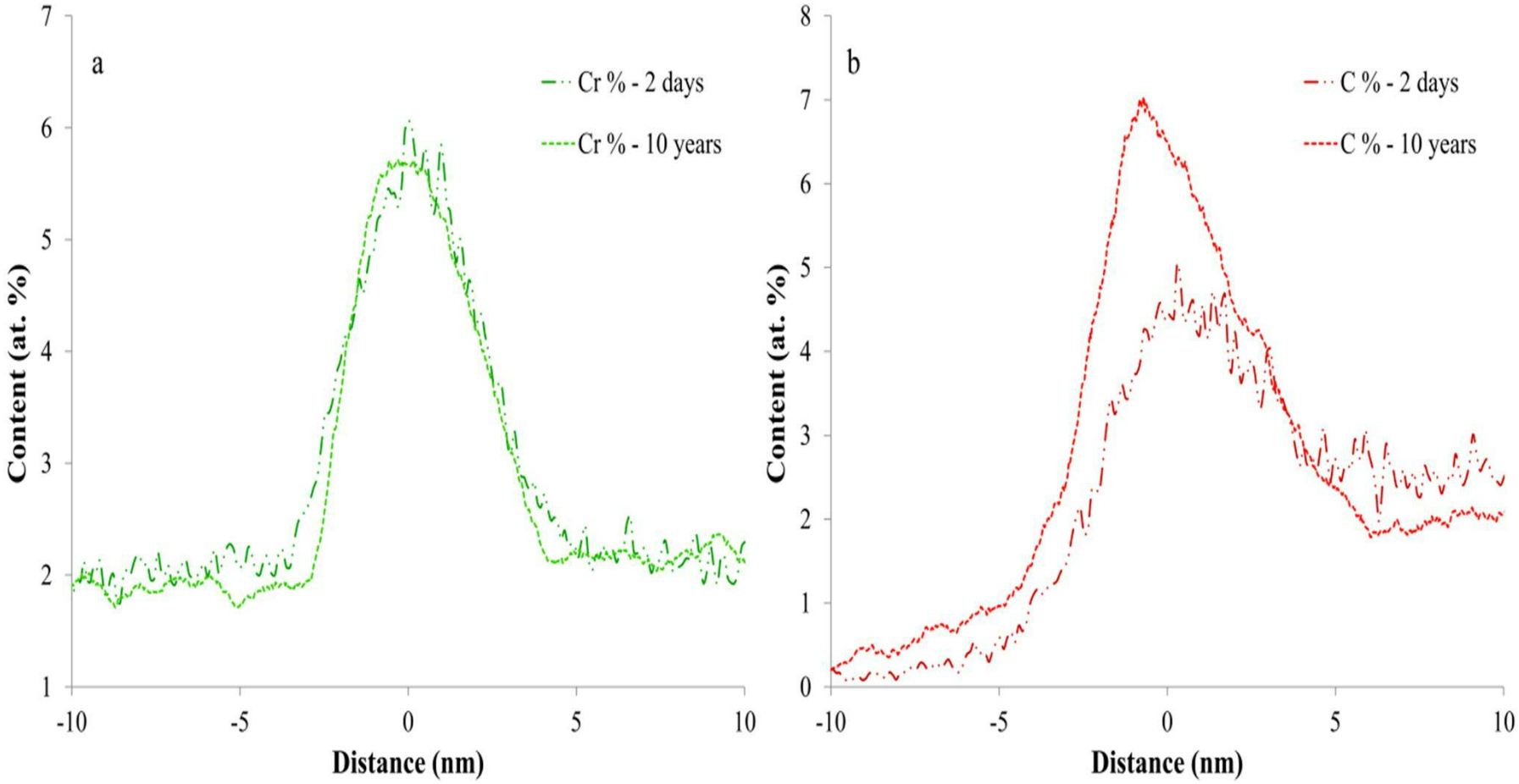


Composition profile recorded by 3D APT from the same decarburized Fe-Cr-C sample and superimposed EELS signal from the Cr $L_{2,3}$ ionization edges (575 - 608 eV). The shapes of Cr profiles from both techniques are closely similar.

Fe-2%Cr-C: 806°C- 128min, *purged*.

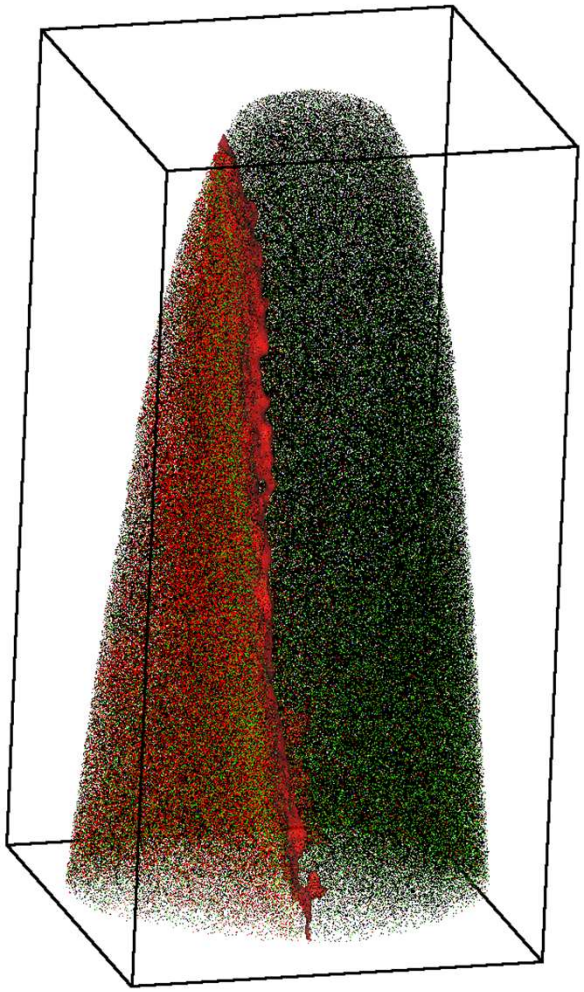


Fe-2%Cr-C: 806°C- 128min, *purged*.

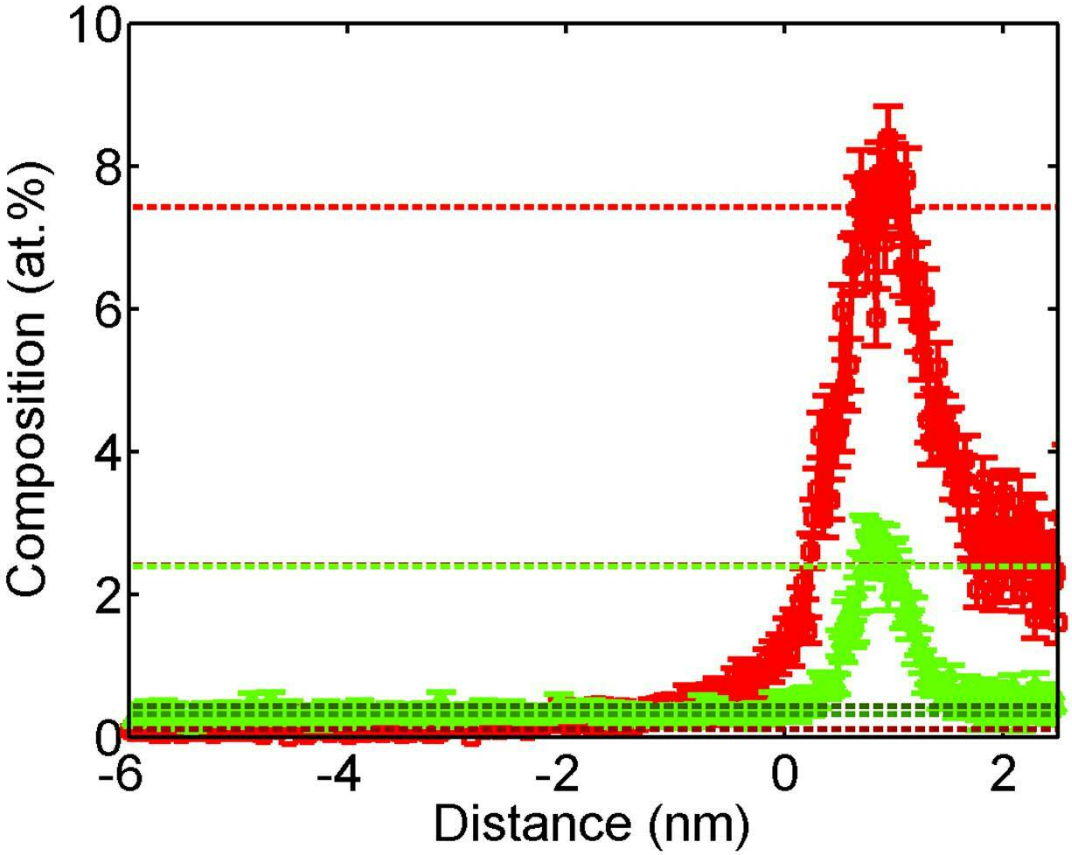


Comparison of concentration profiles calculated from APT data between a 2 day and a 10 year aging treatment for a) chromium and b) carbon.

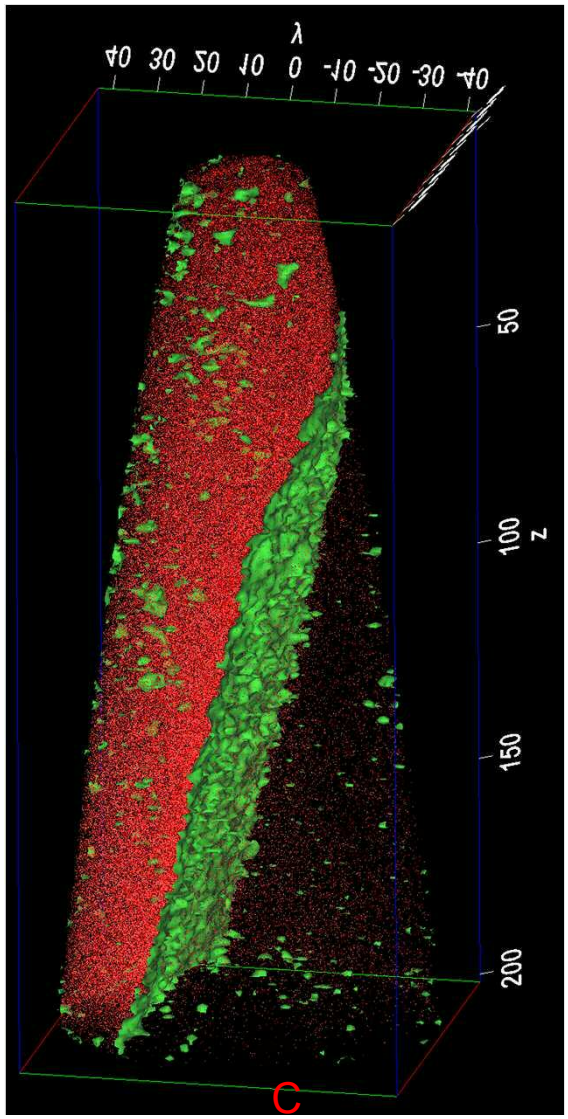
Fe-0.51%Mo-C: 806°C-128min, *purged*.



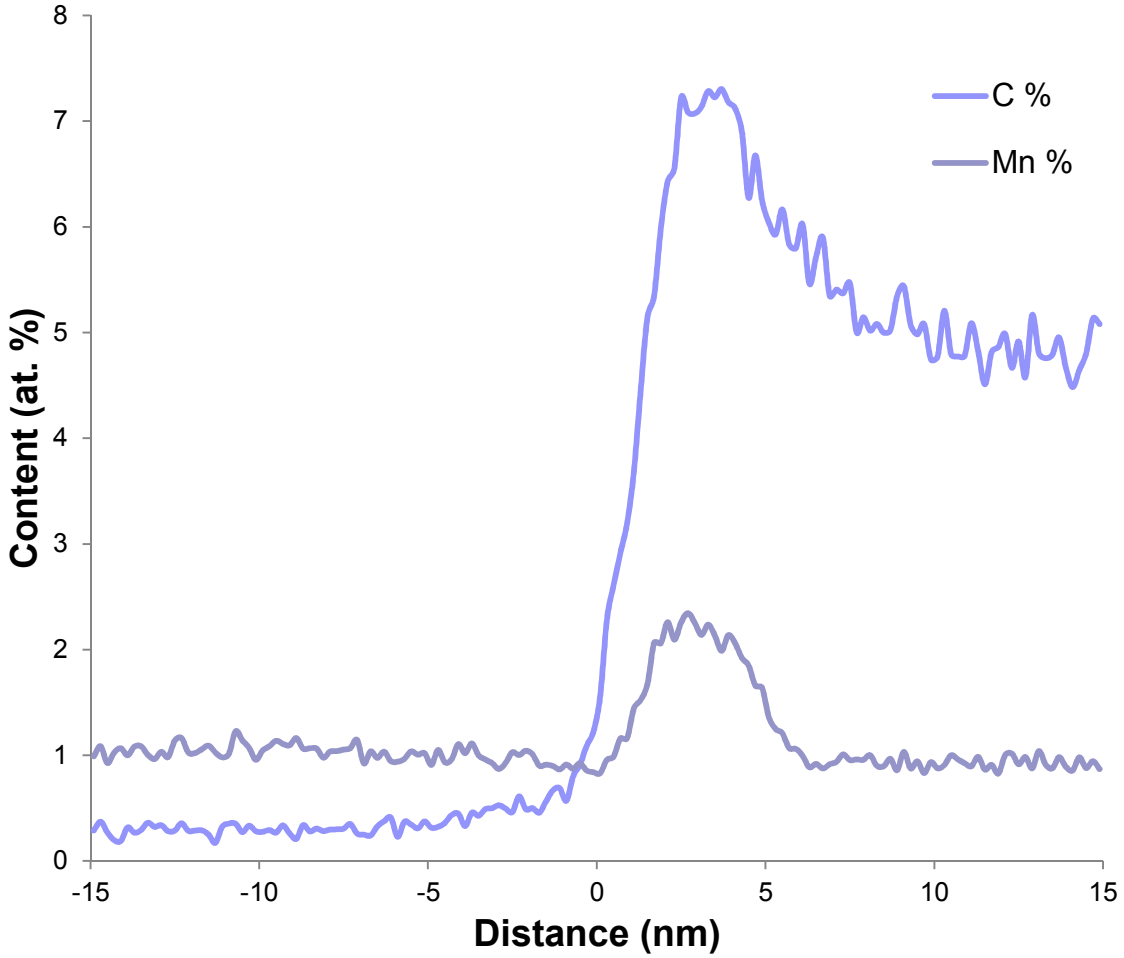
Isosurface @ 1.50 at. % C



Fe-1%Mn-C: 775°C-128min, *purged*.

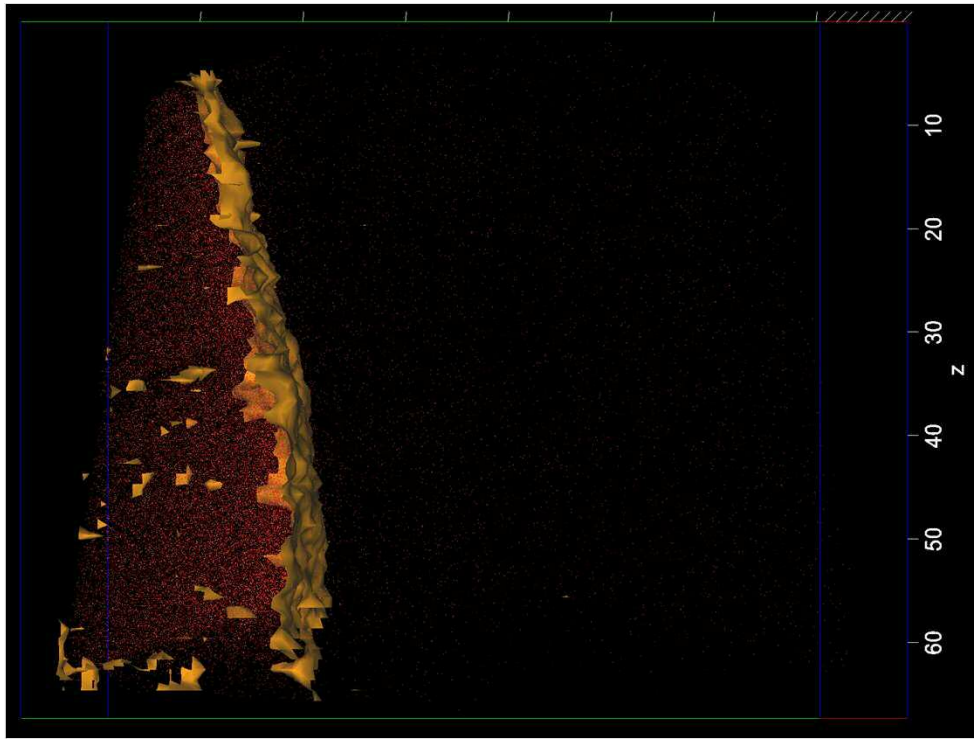


Isosurface @ 2.20 at. % C



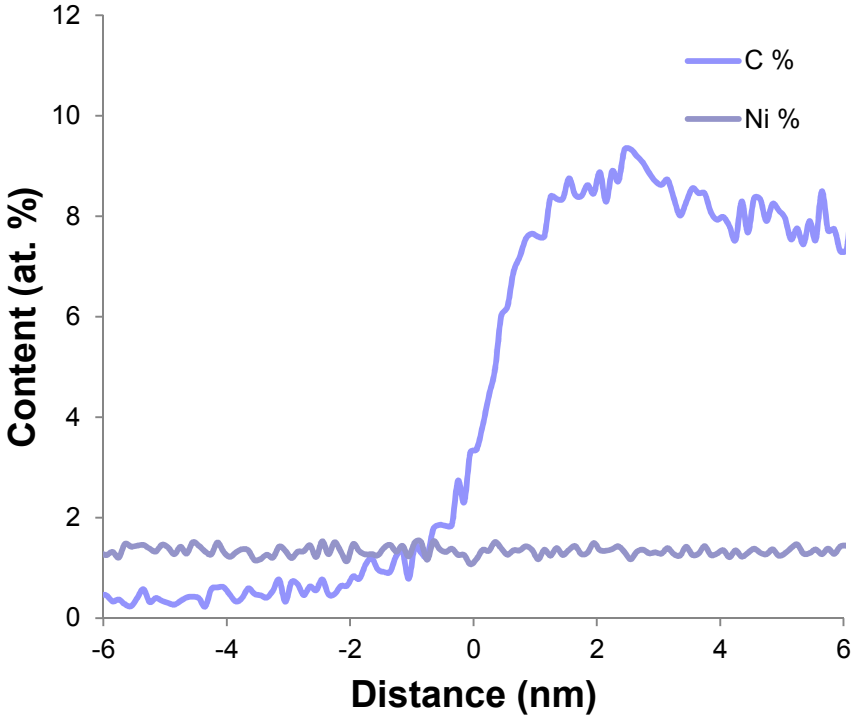
Fe-1%Ni-C: 775°C-16min, *purged*.

Partial voltage recon of ROI calibrated using (110) of α-Fe



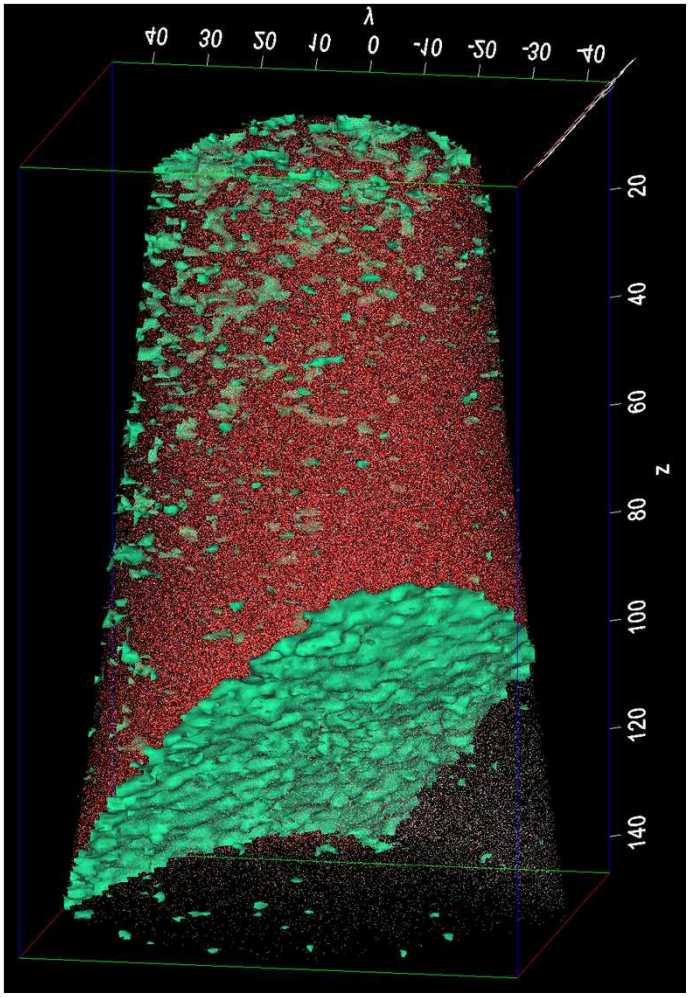
C

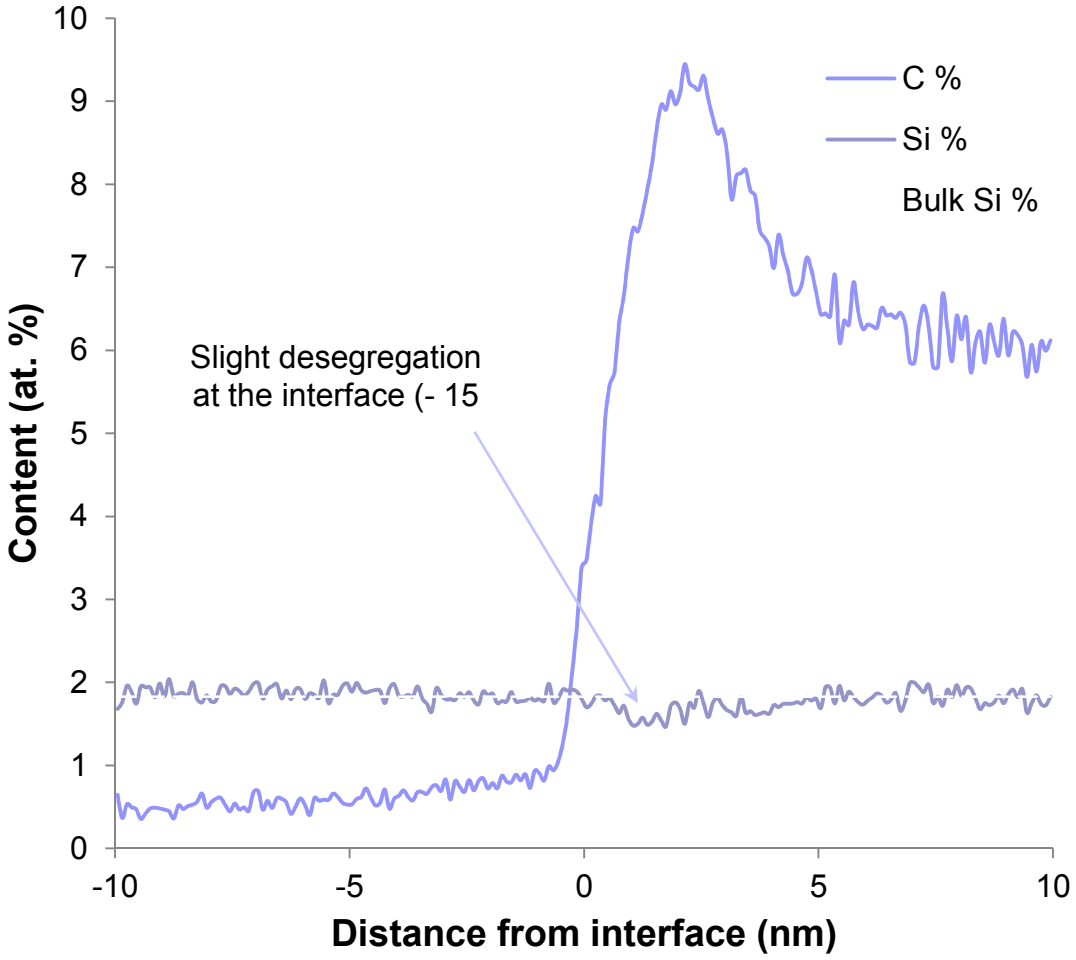
Isosurface @ 4.30 at. % C



Ni content is overestimated due to numerous overlap (Fe, FeH, C_5).

Fe-0.88%Si-C: 806°C-32min, *purged*.





Summary of Results:

Element (X)	**Si**	**Cr**	**Mn**	**Ni**	**Mo**
Bulk X content (at. %)	1.83 ±.09	2.19 ±.02	0.93 ±.06	1.12 ±.14	0.46 ±.15
Temperature (°C)	806	806	775	775	806
Interface at. % (avg.)	1.57 ±.10	5.66 ±.03	2.23 ±.08	1.17 ±.10	2.56 ±.22
Interface at.% (peak)	1.47	5.71	2.35	-	2.83
Seg. Ratio wrt avg.	0.86	2.6	2.4	1	5.6
Seg. Ratio wrt peak	0.80	2.6	2.5	1	6.2

New Insights:

- It would appear that segregation to the ferrite/austenite interface is similar to that observed at austenite grain boundaries (probably due to similar C content).
- Si results are difficult to explain, may be due to strong repulsion between C and Si.

New Insights:

Element (X)	**Si**	**Cr**	**Mn**	**Ni**	**Mo**
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Interface at.% (peak)	1.47	5.71	2.35	-	2.83
Seg. Ratio wrt avg.	0.86	2.6	2.4	1	5.6
Seg. Ratio wrt peak	0.80	2.6	2.5	1	6.2
Model Predictions	**1.9**	**1.8**	2.8	1.6	6

Modified binding Energies:

- Mo: -15 kJ/mole wrt midpoint (OK)
- Cr: -1.5 kJ/mole wrt midpoint (must be changed to -3 kJ/mole)
- Mn: -2.5 kJ/mole wrt midpoint (OK).
- Ni: +1.5 kJ/mole wrt midpoint (OK).
- Si: -10 kJ/mole wrt midpoint (must be changed to +8 kJ/mole)

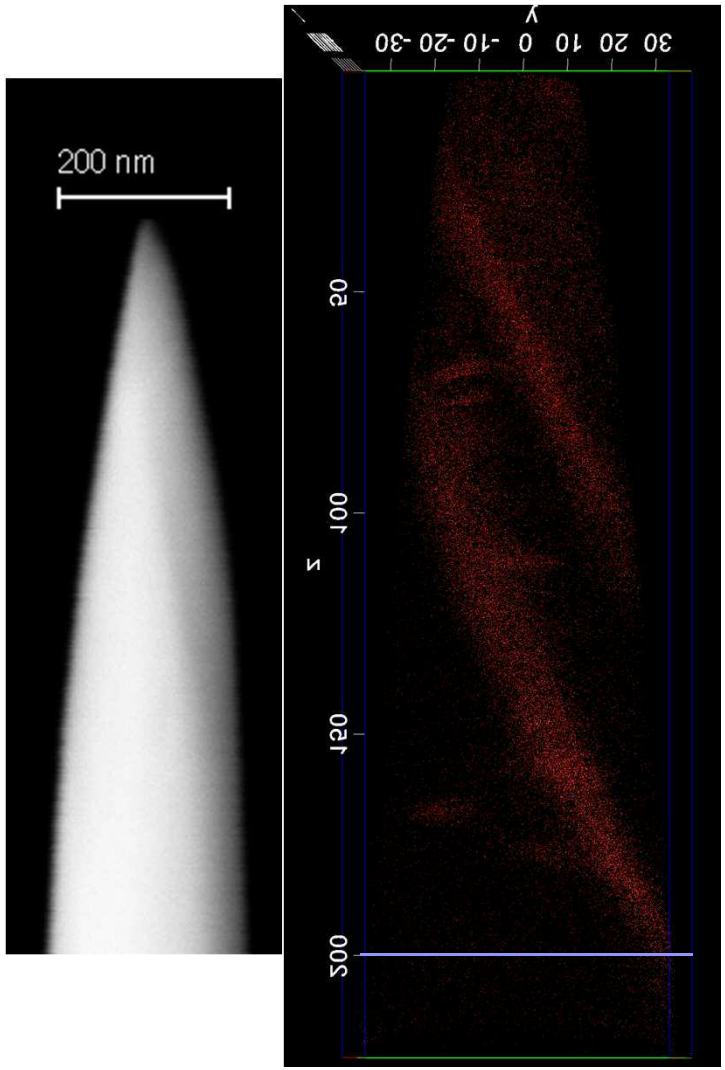
New Insights:

- If we look at the carbon distribution in tips we find:
  - Fe-C shows non-uniform distribution with some regions that could be carbides.
  - Si and Ni show non-uniform distribution of C.
  - Mn and Cr show uniform distribution for short times (few days) but non-uniform distribution after long times (10 yrs).
  - Mo shows uniform distribution even after 10 yrs at RT.
- These results may provide indirect data on the X-C interaction in martensite/ferrite.
- May provide ways of engineering C distributions in the future.

HAPPY RETIREMENT PROF. ENOMOTO

One more result :)

Unexpected results in Fe-Ni-C



C