Linear Complexions: Confined Chemical and Structural States at Dislocations in Metallic Alloys

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Atom Probe Tomography (APT)

R ~ 50 nm
T ~ 20–100 K

The specimen is the lens

Position sensitive detector

Ion sequence

\( X_1, Y_1, \text{ToF}_1 \)
\( X_2, Y_2, \text{ToF}_2 \)
\( X_3, Y_3, \text{ToF}_3 \)
\( X_4, Y_4, \text{ToF}_4 \)
\( X_5, Y_5, \text{ToF}_5 \)

Time of flight \( \rightarrow \) chemical identity
Position of hit \( \rightarrow \) X-Y coordinates
Evaporation sequence \( \rightarrow \) Z coordinate

3D point cloud data
Atom Probe Tomography (APT)

It's a point cloud

Can you get structure?

APT crystallography

Correlative APT&EM
Field desorption image

Detector Event Histogram

Specimen

Local electrode

shank angle

HV pulse ($V_p$)

Position Sensitive Detector

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Lattice plane reconstruction: APT crystallography

Fe$_3$Al (only Al displayed)

Atom probe crystallography:
80% of the ions
Chemistry and structure in 3D
LEAP 5000
Experimental setup for correlative TEM–APT probing

**Principle**

**FIB:** Tip is cut parallel to holder axis.

**TEM:** During tilt around holder axis tip always stays in focus range, whole sample in focus (!).

**APT:** Defined sample orientation in all instruments makes it possible to merge information.

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Correlative TEM-APT probe for 5D GB segregation analysis

BF-STEM micrograph of cold-drawn Fe-C

100 nm

e\textsuperscript{-} beam

Scanning nano beam diffraction

Nano beam diffraction

(00-1) (010)

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Correlative TEM-APT probe for 5D GB segregation analysis

- Carbon atoms
- 5 crystal. parameters
- N chemical species

Playing with martensite-to-austenite reversion: Fe-Mn

Cubic martensite

Austenite

\(\gamma(Fe, Mn)\)

\(\alpha\)
Segregation at dislocation lines (450°C/48h): 9 % Mn

9Mn-2Ni-0.15Al-1Ti-1Mo
Segregation at dislocation lines (450°C/6h): 9% Mn

Fe-9wt%Mn; 50% cold rolled, 450°C/6h

- Mn
- Fe

Mn 11 at% isosurface
Segregation at dislocation lines (450°C/6h): 9 % Mn

Fe-9wt%Mn; 50% cold rolled, 450°C/ 6h

- Mn
- Fe

Mn 11 at% isosurface
Correlated microscopy LAGB (50%CR+450°C/6h) Fe-9%Mn

Work in progress
Correlated microscopy LAGB (50%CR+450°C/6h) Fe-9%Mn

- Mn
- Fe
- Mn 11.2 at% isosurface

\[ \theta = \arcsin \frac{b}{d} \]

Mn09 50%CR 450C 6h
Correlated microscopy LAGB (50%CR+450°C/6h) Fe-9%Mn

Mn iso-surface 12 at%

Mn iso-surface 11.5 at%

Mn %
C %

0 5 10 15 20
Distance (nm)

Movie with an increasing iso-concentration value
Mn-concentration fluctuations along the dislocation line

Correlated microscopy LAGB (50%CR+450°C/6h) Fe-9%Mn
APT crystallography (50%CR+450°C/6h) Fe-9%Mn

Work in progress
Correlated microscopy LAGB (50%CR+450°C/6h) Fe-9%Mn

Correlated microscopy LAGB (50%CR+450°C/6h) Fe-9%Mn
Transformation at dislocations, Fe-9%Mn, stability, kinetics

Correlated microscopy: transformation at dislocation Fe-9%Mn

Dark-field TEM

Correlated microscopy: transformation at dislocation Fe-9%Mn

Small angle grain boundary

Edge dislocation

Screw dislocation

Edge dislocation

Atom Probe Tomography and Beyond:
Towards ‘Laplace Daemon‘: Atomic chemistry & structure in 3D, seeing chemo-mechanical mechanisms at lattice defects that were not visible before

Chemo-mechanics at dislocations & GBs: Multiple possible states / transitions: linear complexion, defect-assisted (heterogeneous) nucleation; defect-assisted pseudo-spinodal