Joint Nanoscale Structural and Chemical Characterization by Correlative Atom Probe Tomography and Transmission Electron Microscopy <u>M. Herbig</u>



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Correlative Microscopy



Short fatigue cracks in titanium









Diffraction Contrast Tomography (DCT)

st Phase Contrast T) Tomography (PCT)



DCT + PCT

Atom Probe Tomography

Postdoc - Group leader





Transmission Electron Microscopy



Research Group: Materials Science of Mechanical Contacts





Principle of Atom Probe Tomography (APT)





Strengths of APT





pretation "simple" and robust

APT excellent for measuring chemistry at nm scale of

- 1D defects (segregation at dislocations)
- 2D defects (segregation to grain / phase boundaries, stacking faults)
- 3D bulk objects (precipitates, thin films, grains)

Comparison: Chemical analysis in TEM and APT



→ Quantitative chemical analysis on nm scale is usually done easier or more accurately by APT than by TEM

Atom Probe Tomography – Specimen Size





Sample Preparation by Electropolishing



- > Wire cutting \rightarrow rough polishing \rightarrow fine polishing \rightarrow check with SEM or OM
- > Non-site-specific
- Electrolytes are materials-specific
- Requires conductive material
- Needs practice
- Cheap



APT Sample Prepartion by FIB-Liftout



Cold-drawn Pearlitic Steel Wire





Characterization of GB Segregation

Crystallographic parameters for GB description

- 2 x rotation axis
- 1 x rotation angle
- 2 x GB normal
- 5 macroscopic parameters



GB segregation

- Depends on all cryst. parameters
- Often involves light elements
- Happens in < 2 nm distance from interface
- Requires accurate near-atomic scale joint crystallographic plus chemical information

Why Combine TEM and APT?

1µm



Crystallographic analysis is often challenging, sometimes even impossible for APT



Correlative TEM / APT

- Combines the best of both worlds
- Access to answers of many fundamental materials science questions
- (Grain orientations)
- (Orientation relationships)
- Dislocations

dislocations

- Stacking faults
- Grain / phase boundaries

Slip bands

- Crystallographic phase identification
- Atomically resolved defect structure
- Strain gradients

Here, TEM is usually better suited

Setup for Correlative TEM / APT









Correlative TEM / APT





M. Herbig et al., Physical Review Letters 112, 126103 (2014)

Carbon Excess over Grain Disorientation in Ferrite





Segregation to Dislocations





Segregation to Dislocations in Fe-9Mn





Deformation Mechanism of Metallic Glasses



S. Balachandran, J. Orava, M. Köhler, A.J. Breen, I. Kaban, D. Raabe, M. Herbig, Scripta Materialia (2019)



True 1:1 correlation: HR-STEM on APT needle (faceted Σ9 in Si)



C.H. Liebscher et. al, Phys Rev Lett 121(1) (2018).





True 1:1 correlation: APT vs. HR-STEM (faceted Σ9 in Si)







Thank you for your attention!