Integrating Atom Probe Tomography and Transmission Electron Microscopy into a Single Instrument

Eric Van Cappellen; Joachim Mayer; Rafal E. Dunin-Borkowski; Joseph Bunton; Frank de Jong and Hugo van Leeuwen.

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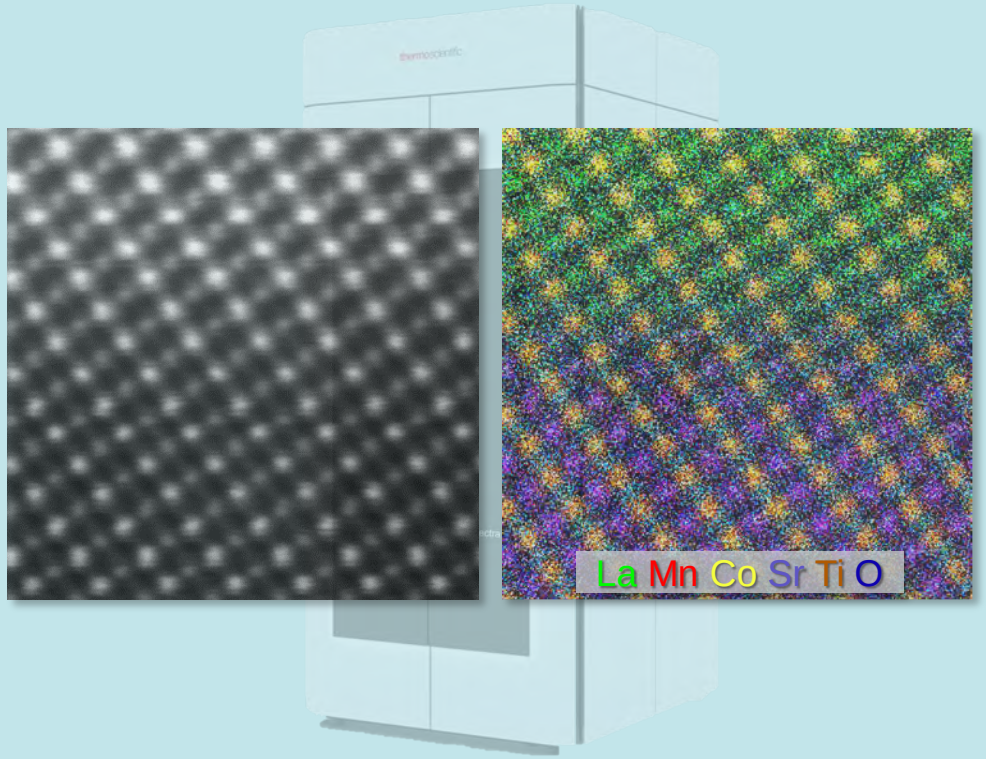
The world leader in serving science

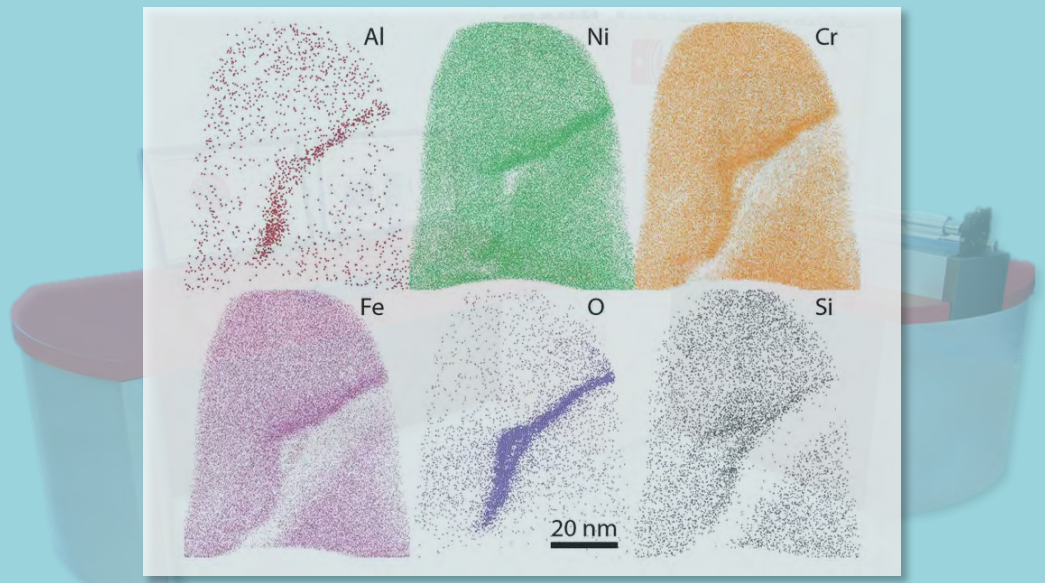
Motivation

Combining two powerful materials characterization techniques

(Scanning) Transmission Electron Microscopy



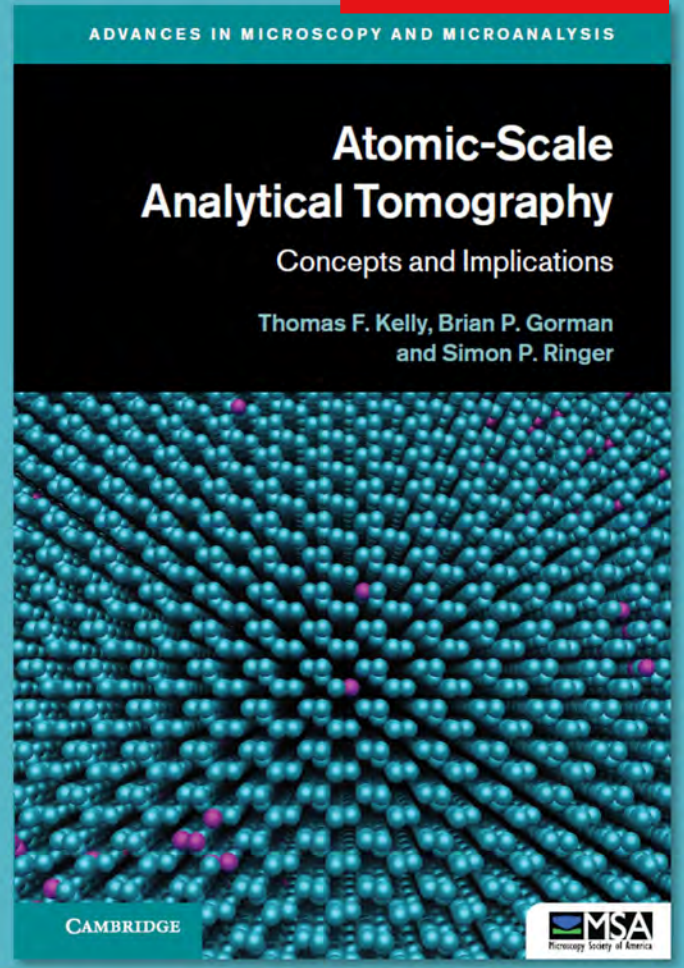
Atom Probe Tomography



Motivation

Atomic-Scale Analytical Tomography (ASAT)

- Integrating an atom probe microscope into a transmission electron microscope

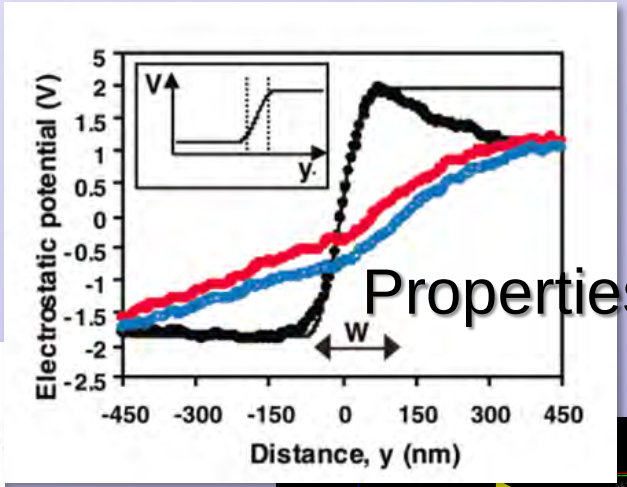


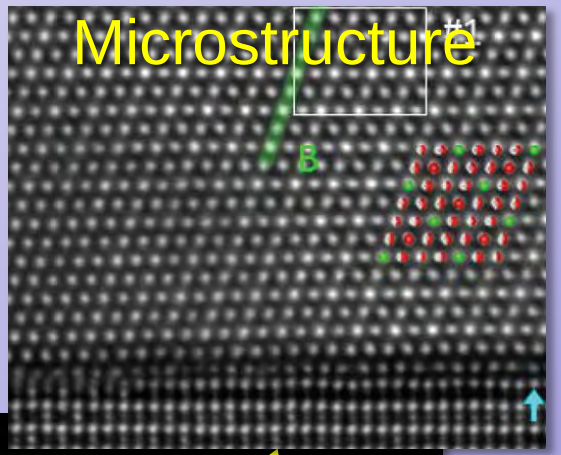
"The largest effort towards ASAT is a recently funded program at the Ernst Ruska Center in Germany called Project Tomo. This project will integrate an atom probe into the objective lens of an aberration-corrected TEM"

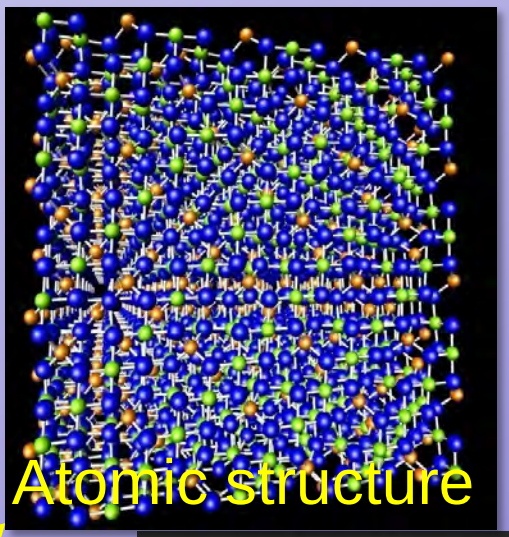
Motivation: ASAT

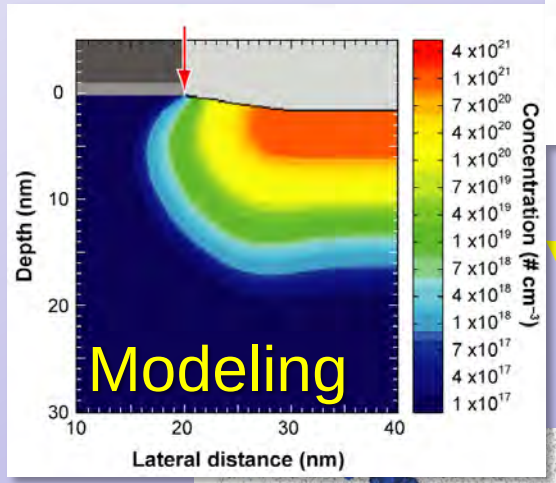
Courtesy: Tom Kelly

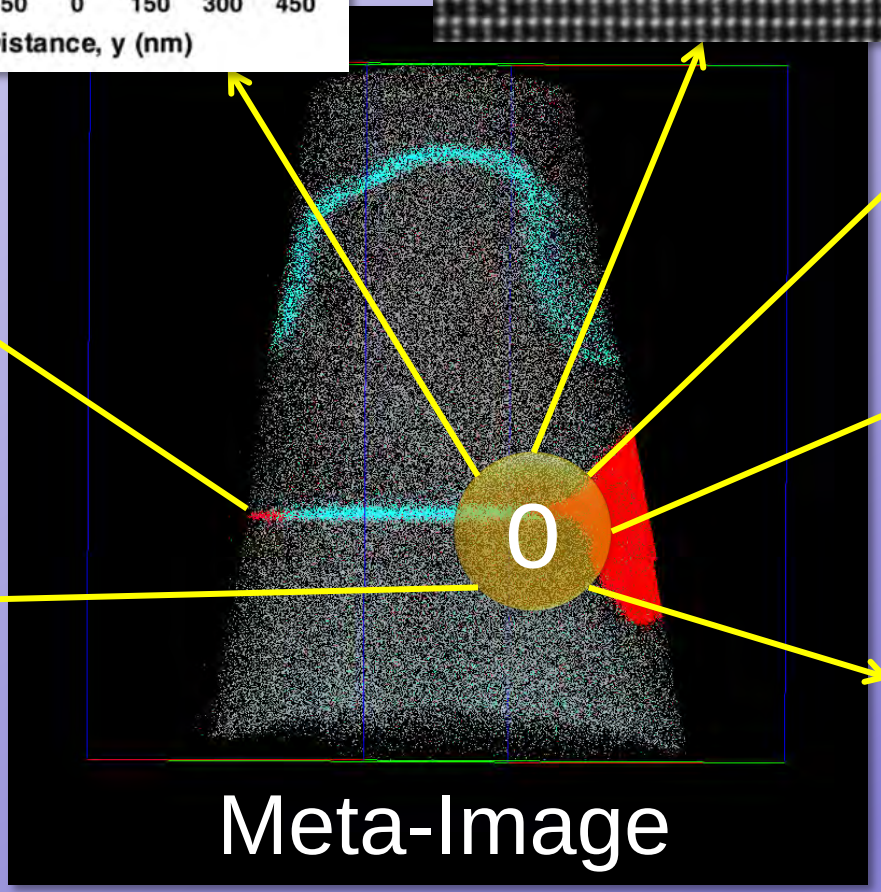
- Wishlist

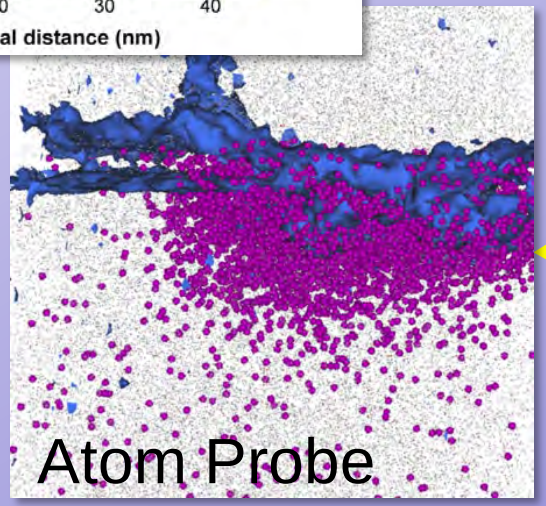


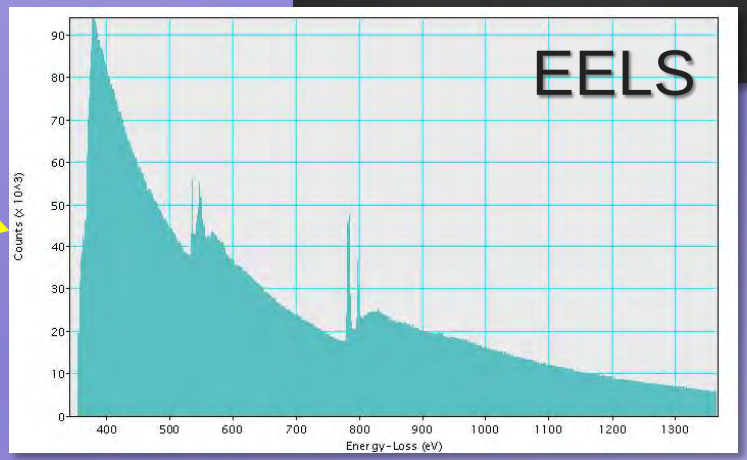






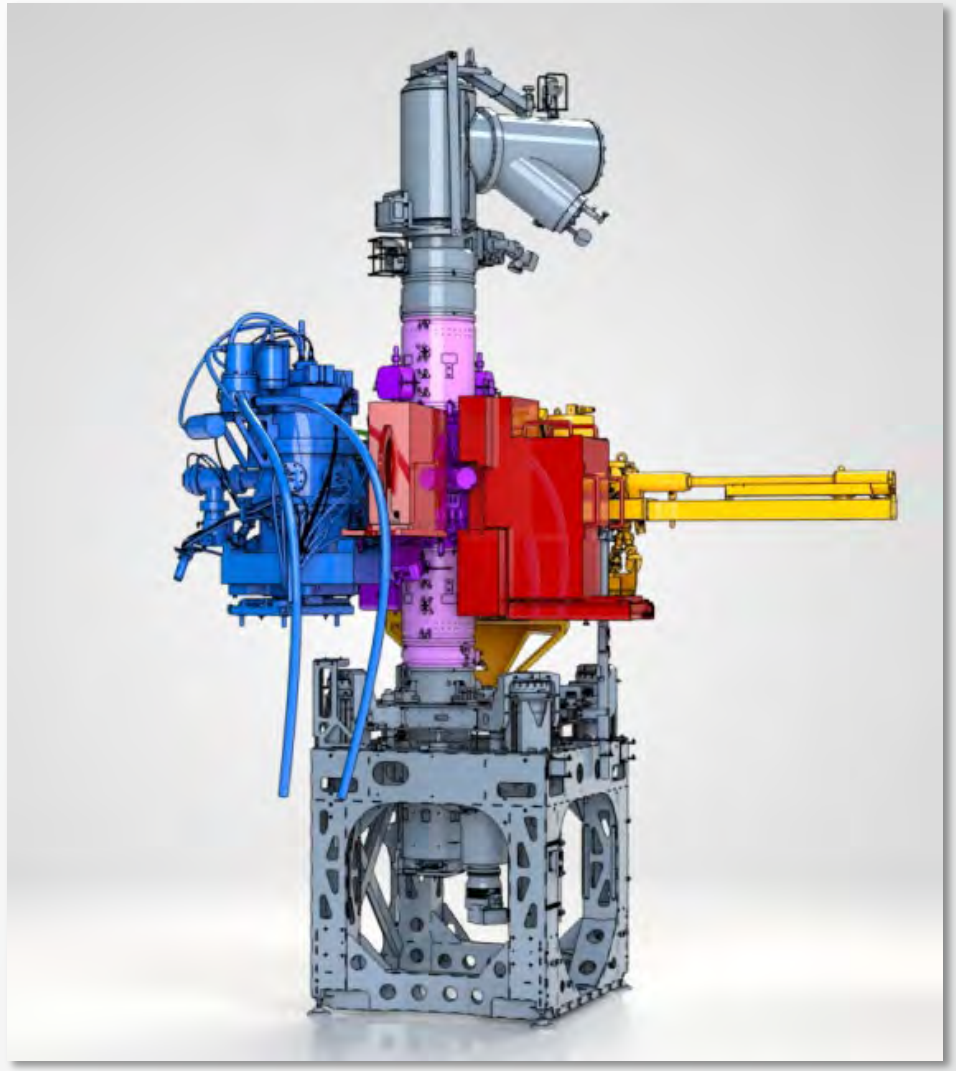






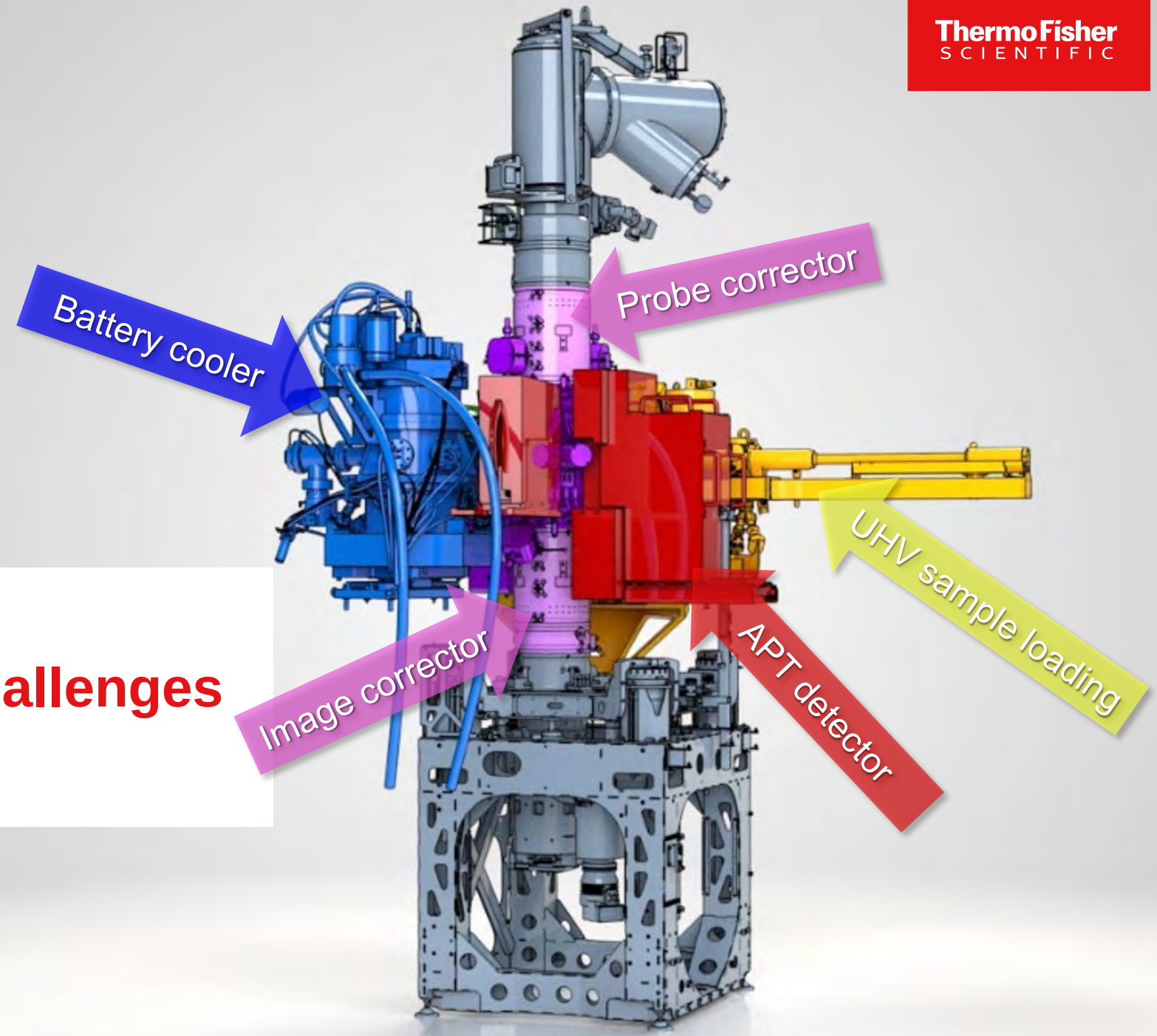
Outline

1. Introduction & design challenges
2. The Octagon
3. Ultra High Vacuum
4. Vibration-free cryo cooling
5. Workflows
6. First experimental results
7. Closing remarks



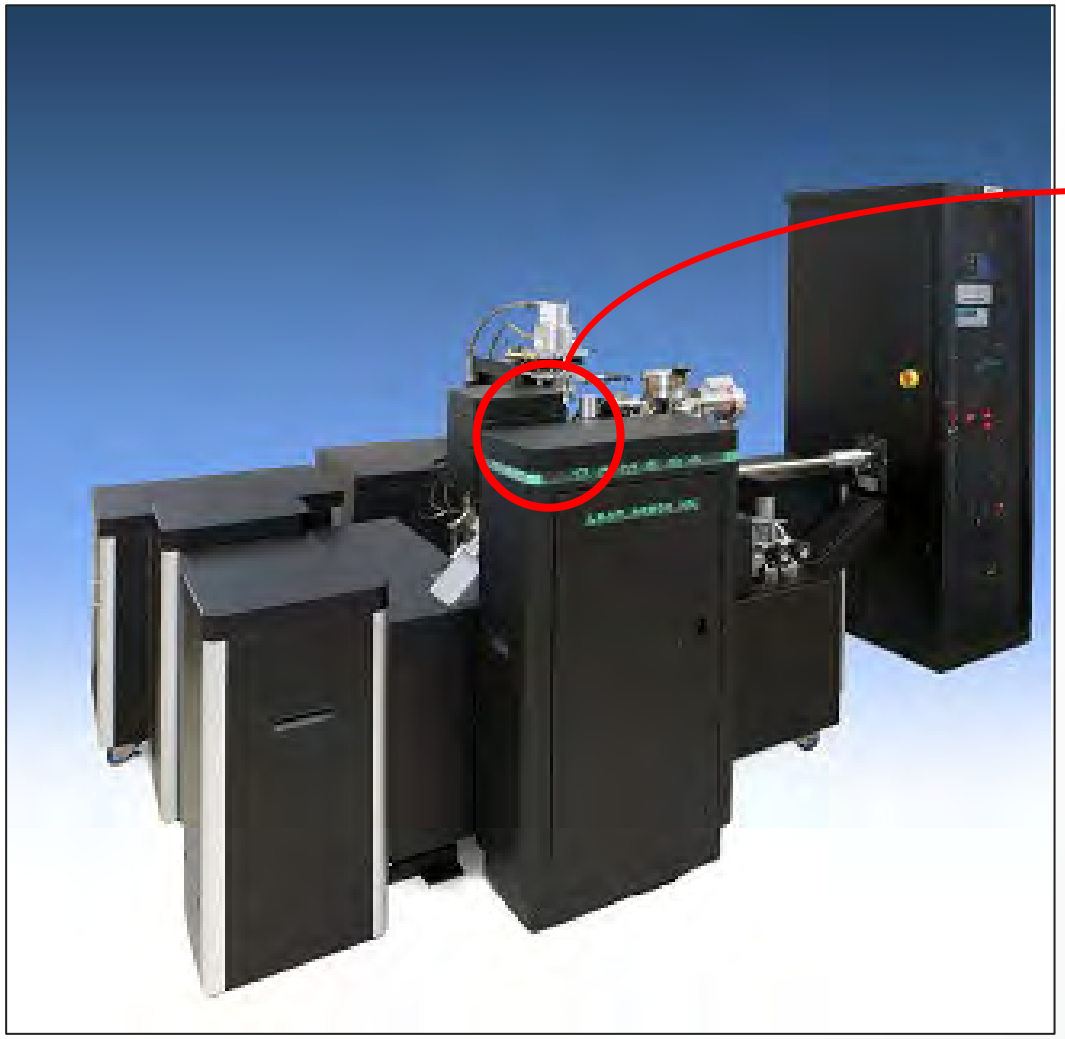
Introduction & design challenges

Integrating an APT into HR-(S)TEM



Introduction: Concept

Integrating an Atom Probe Microscope into a high-end (S)TEM



XFEG mono

Biprism (in C2)
Biprism (in C3)
Condenser system

SCOR II

Valve

Loader
Laser
AP detector
Objective; pole pieces+ UHV+APT
Cryo UHV stage

Valve

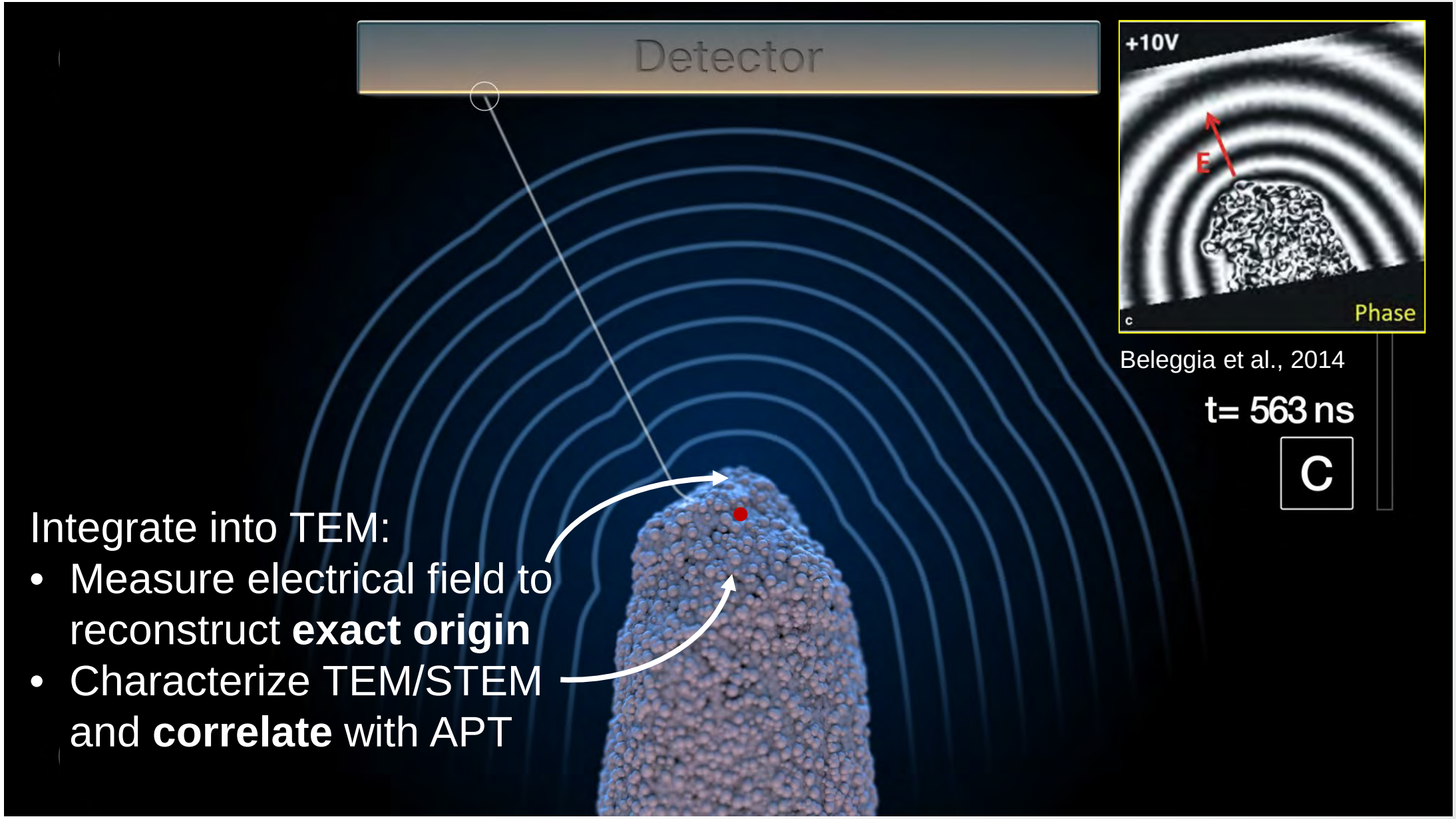
CETCOR PRIME

Biprism
Biprism (Holo-X)

Biprism (SA)
HAADF
BF/DF
EMPAD
Projection system
Ceta 2

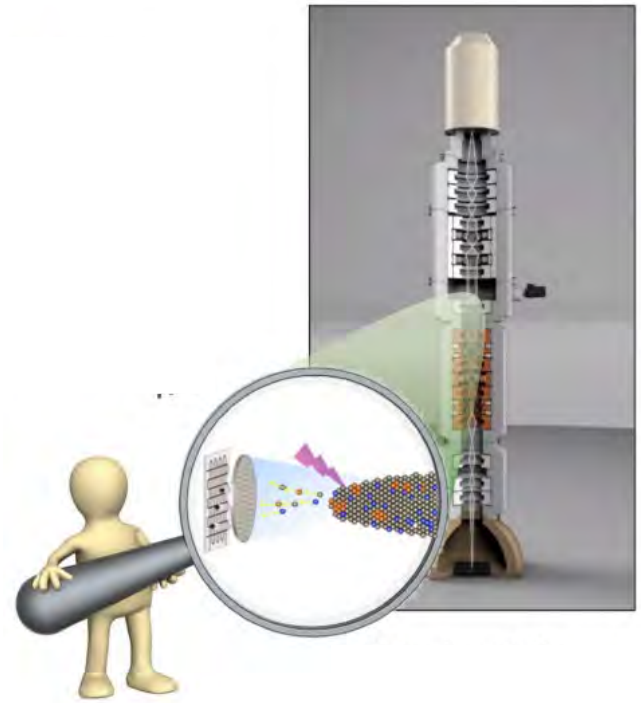
Filter (EFTEM+EELS)

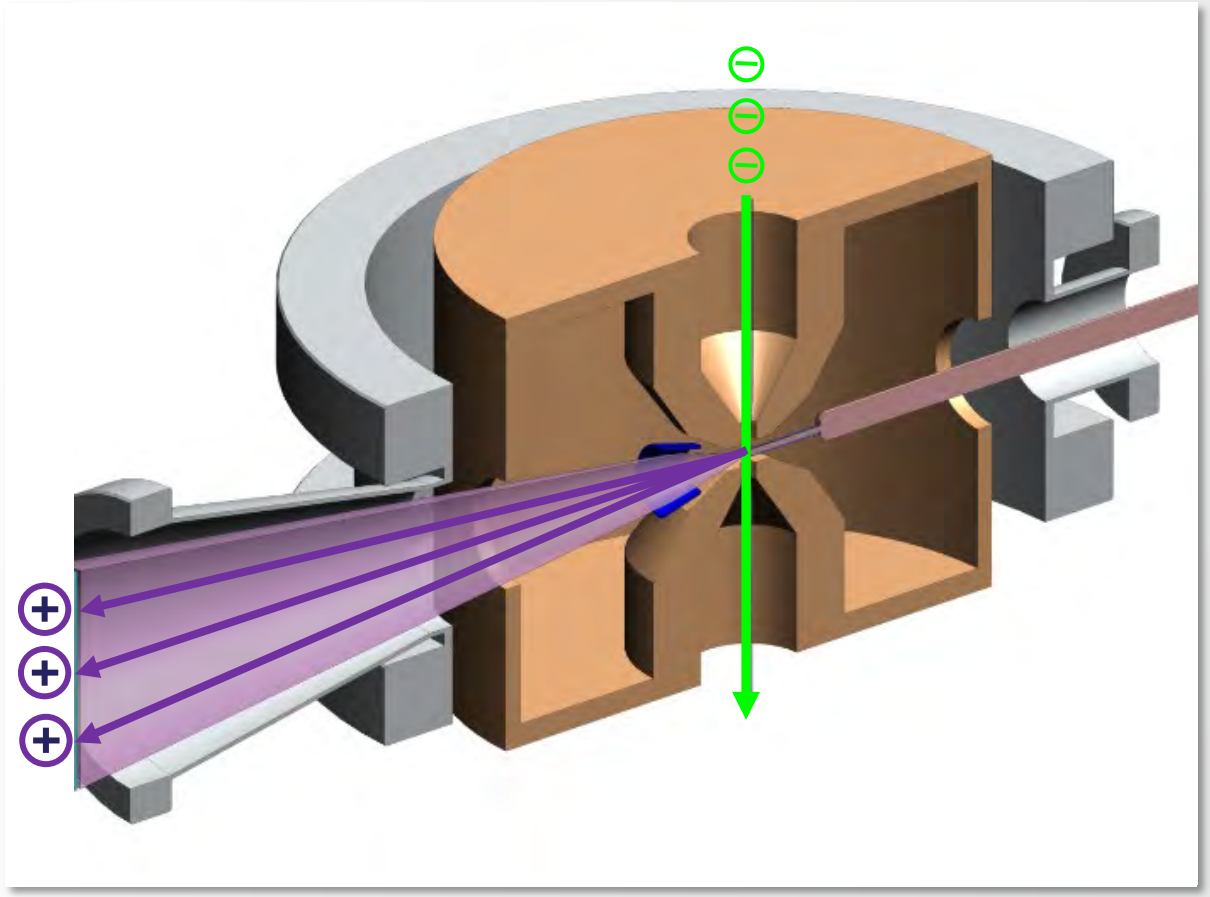
Introduction: APT – Evaporation principle



Design challenges: concept

Tomo instrument design



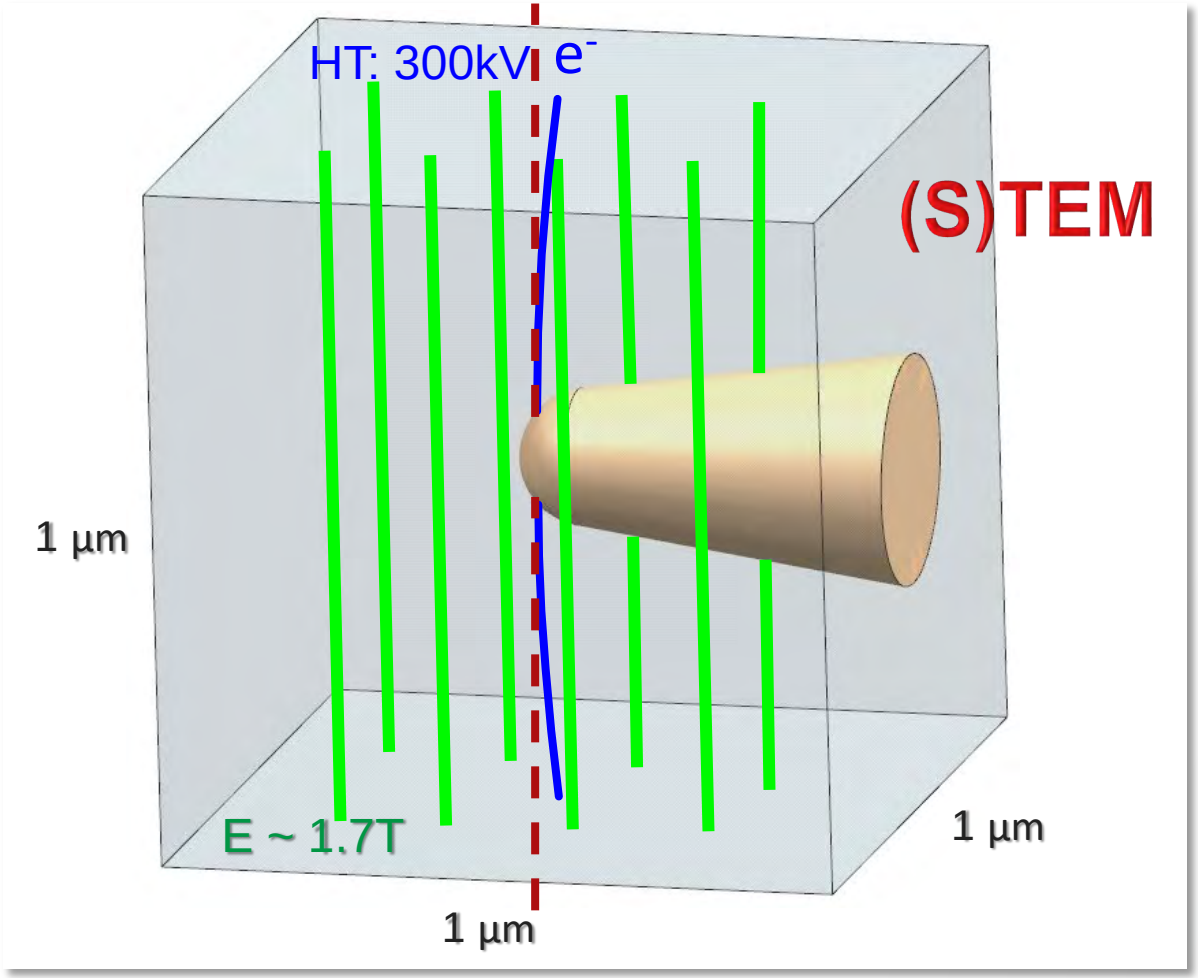


- Consecutive operation: fast switching between:
  - APT mode: **B**-field free
  - (S)TEM mode: **E**-field free

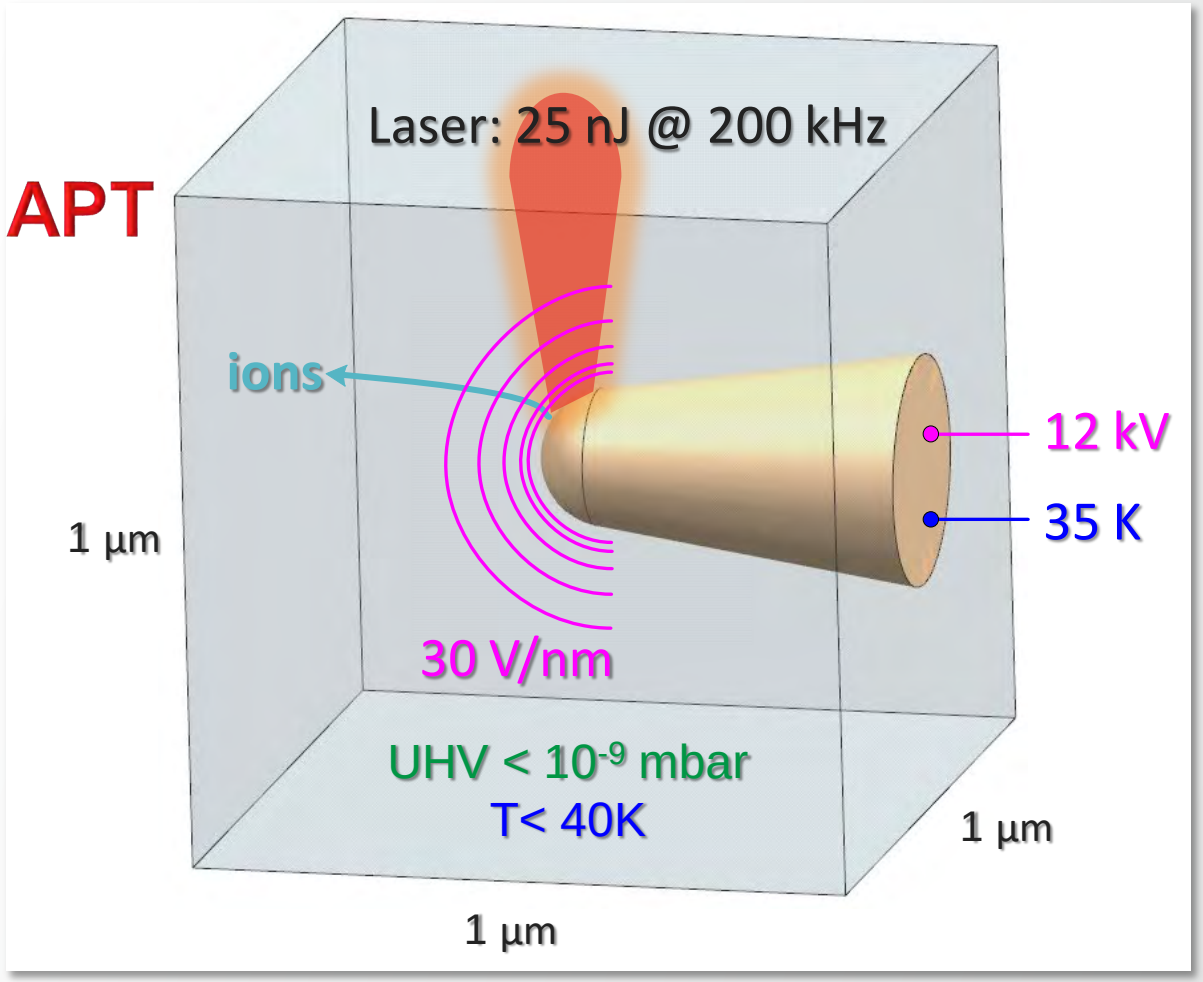
Design challenges: divergent experimental conditions

Principal instrument requirements in (S)TEM and APT mode

- Very thin sample
- Sample stability: < 10pm (0.1 – 5000 Hz)

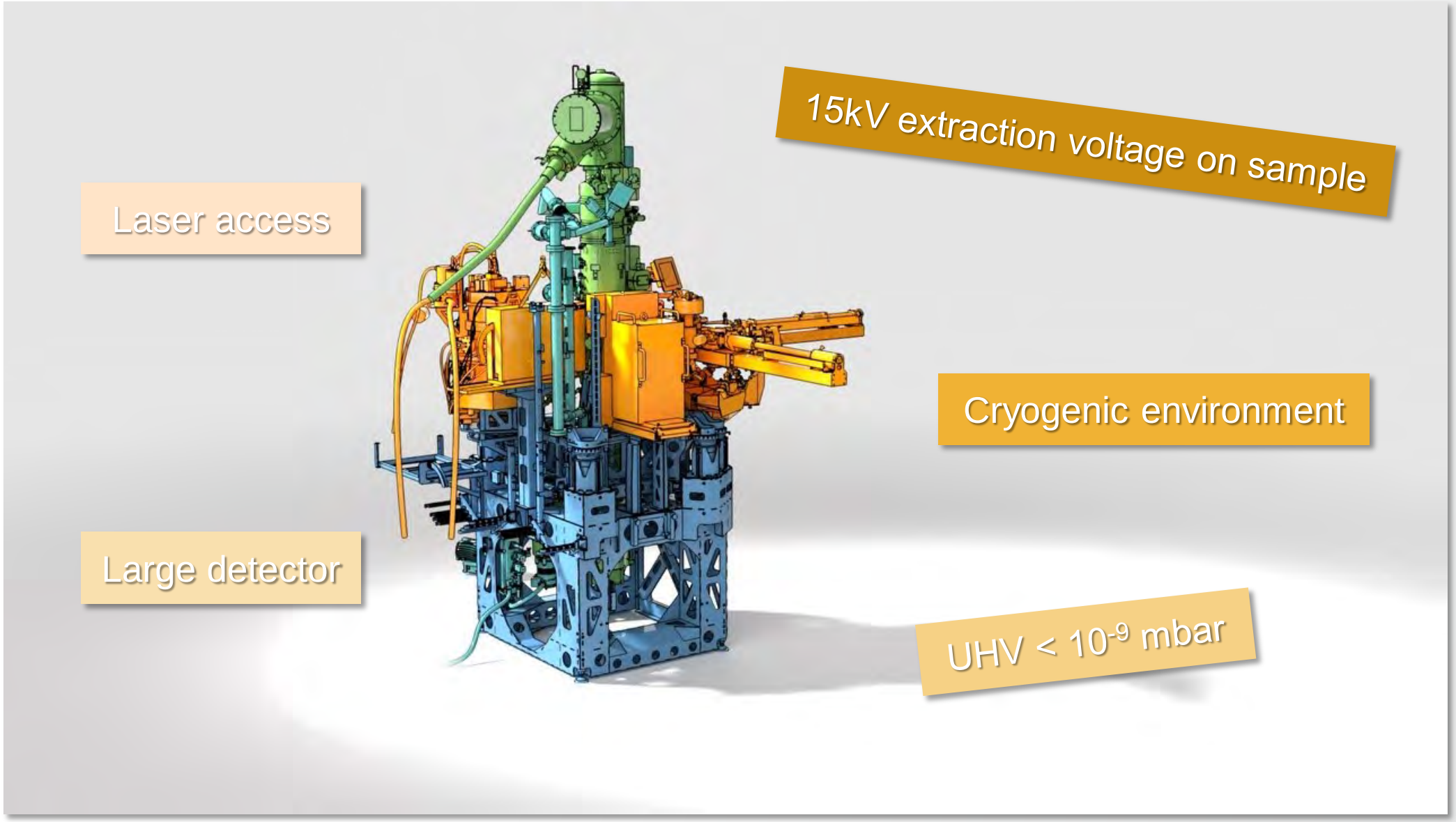


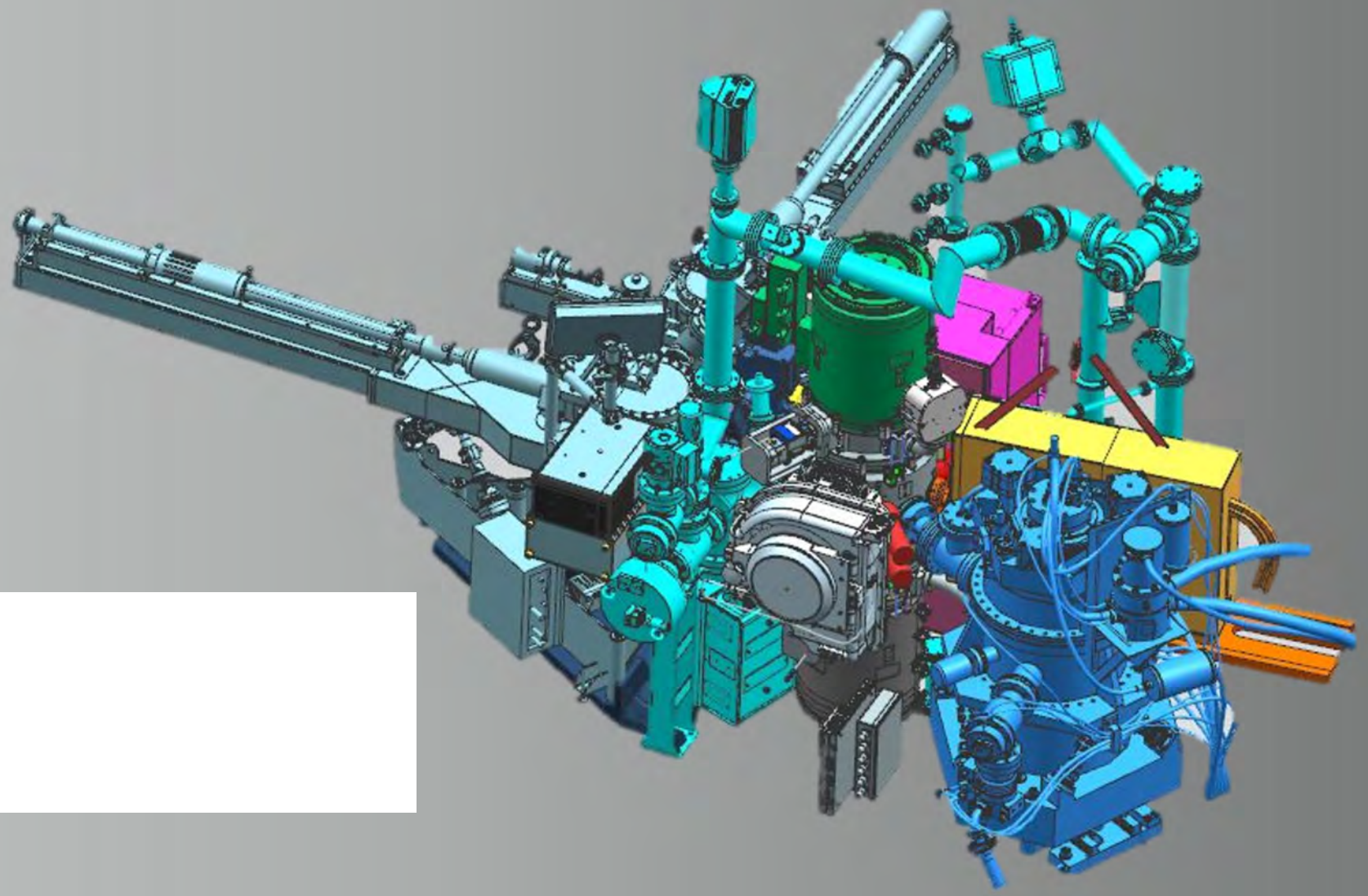
- Needle sample
- Minimize surface diffusion and ion loss.



Design challenges: APT experimental conditions

Extreme requirements:



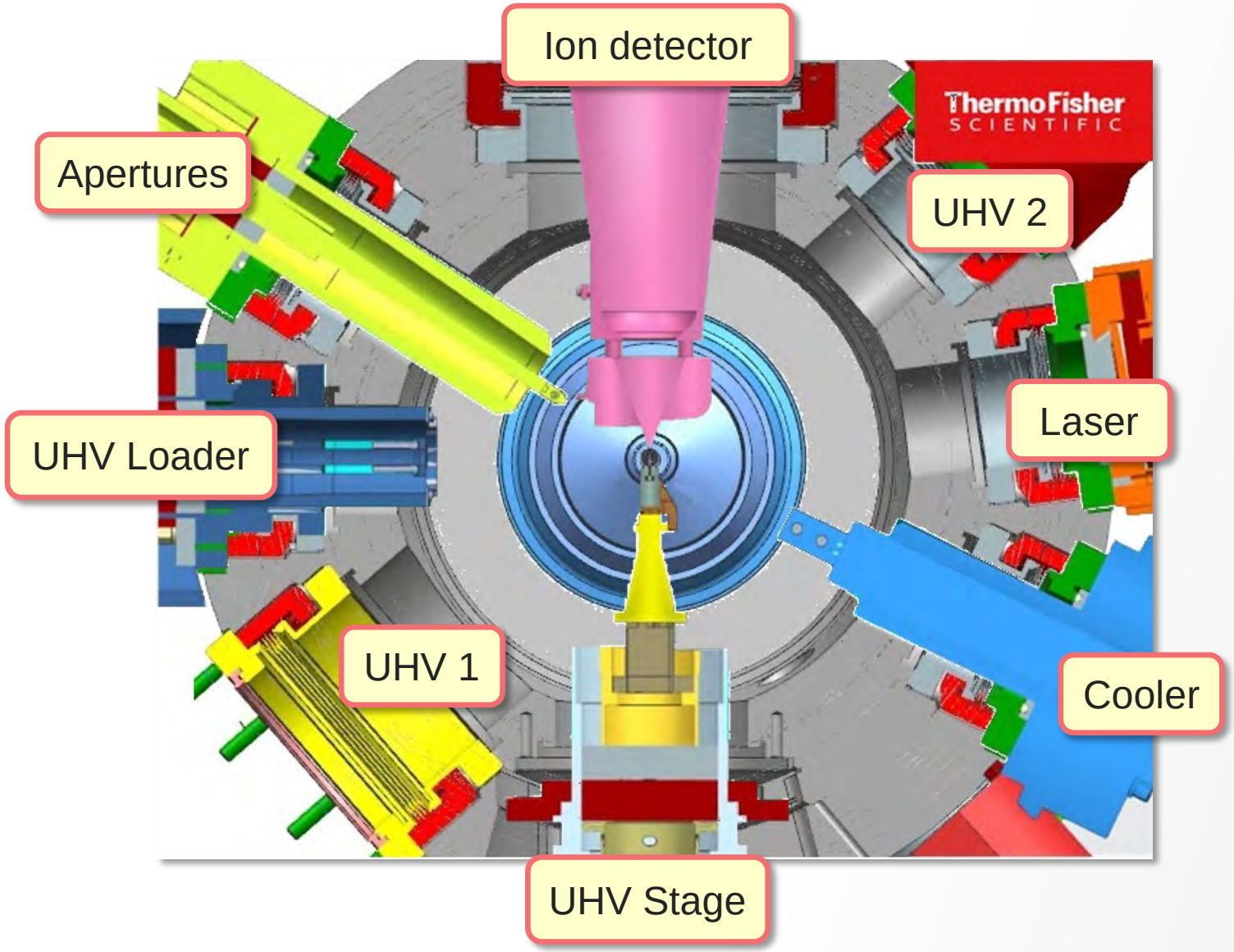


The Octagon

The Heart of the system

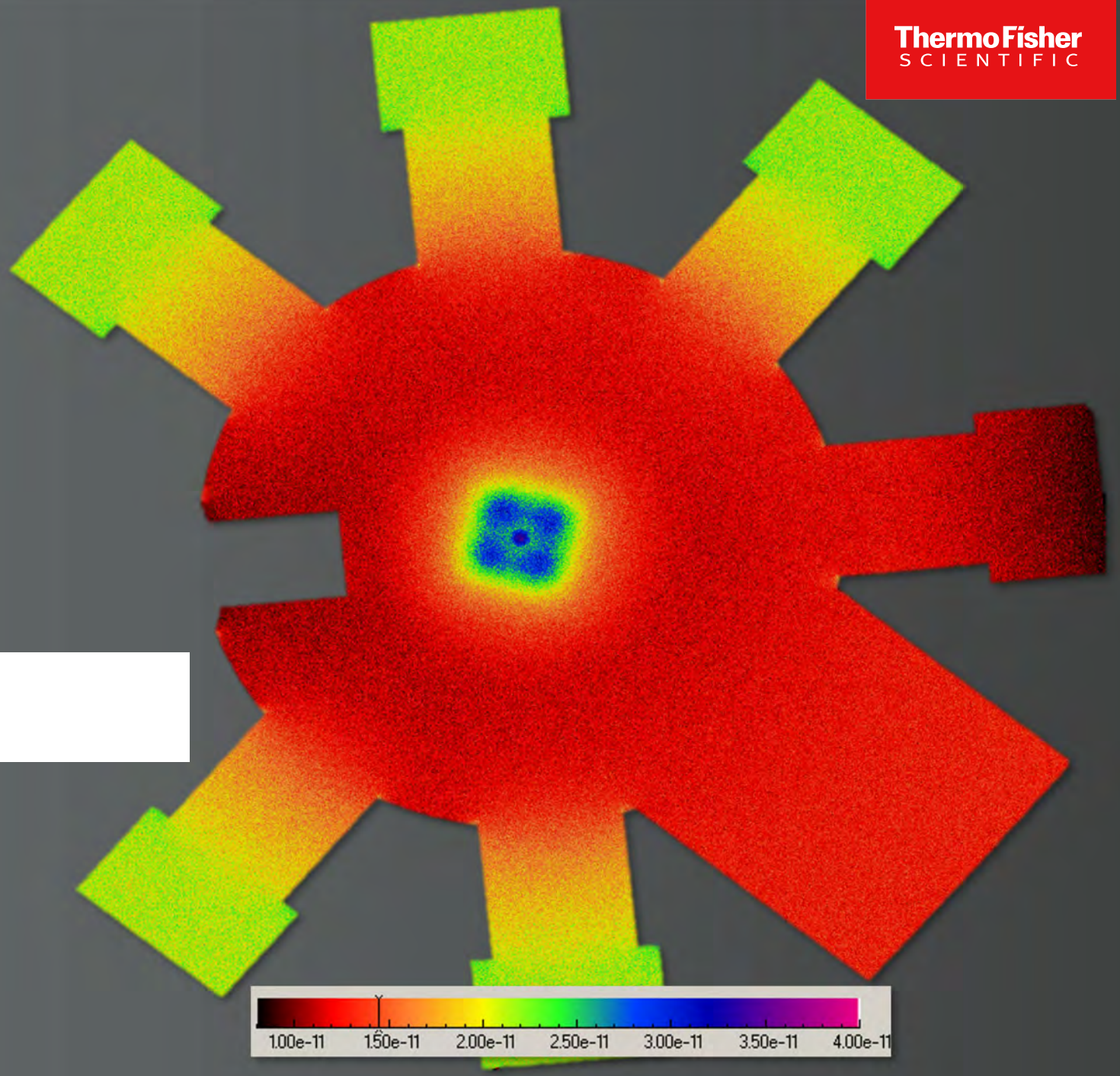
The Octagon

Sample area



- Laser
- Ion Detector
- Specimen chamber
  - Ultra High Vacuum
  - Optics
- Stage
- Loader
- Cooler
- … and many more:
  - Elongated correctors
  - Holography
  - Tomography
  - 4x electrical contacts
  - EELS
  - Scripting access
- Coincidence

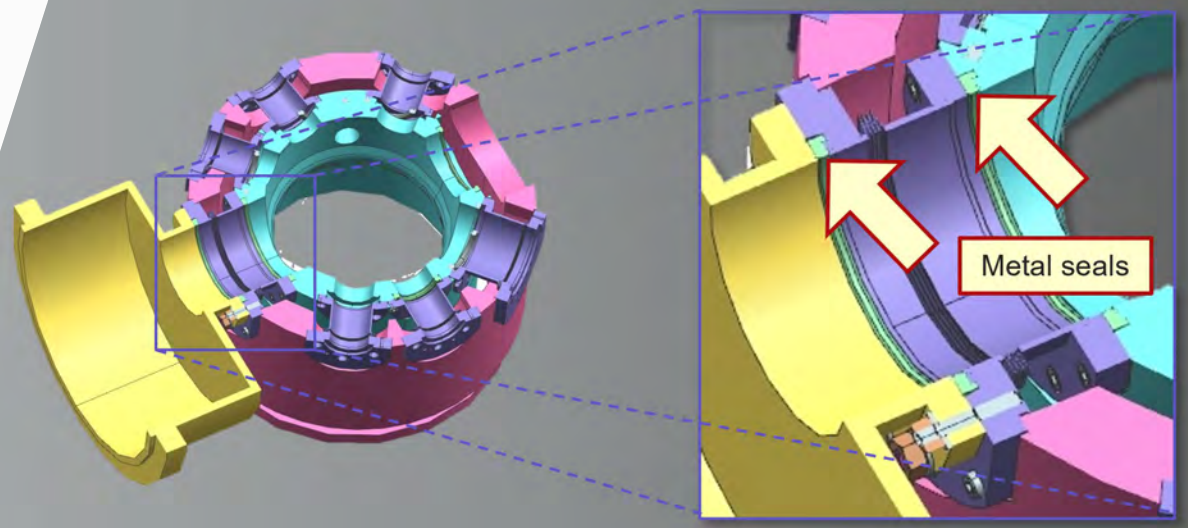
Ultra High Vacuum



Ultra High Vacuum

Achieving UHV

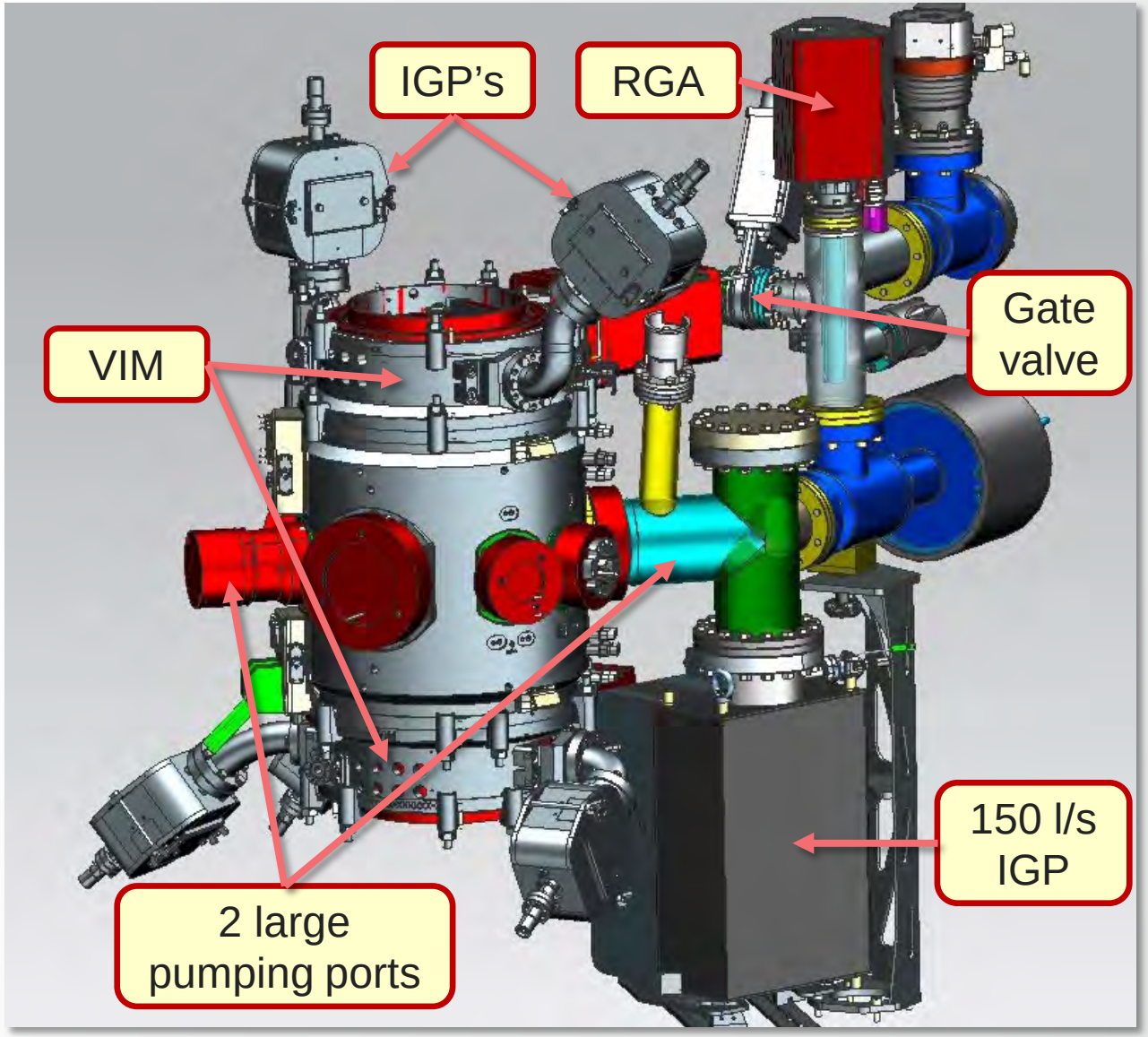
- Large pumps and pumping ports ☑
- Differential pumping ☑
- Bakeable ☑
- Metal seals ☑
- Rest Gas Analyzer for diagnostics ☑
- UHV sample loader ☑
- UHV compatible stage ☑



Ultra High Vacuum

Achieving UHV

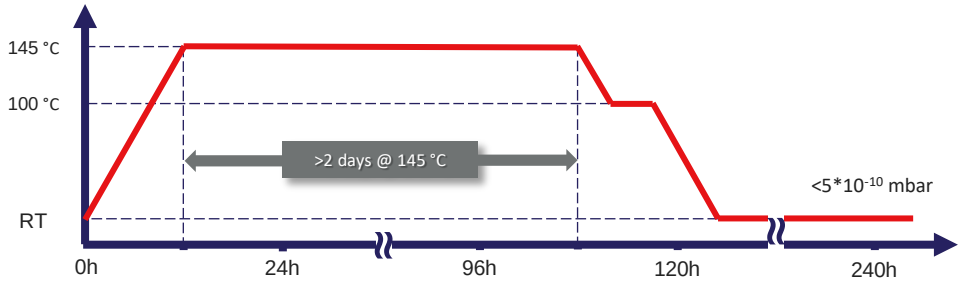
- Metal seals in all ports
- Heaters are build-in the Octagon:
  - Liner tube + stage + loader
- Active cooling for:
  - Lens coils
  - Stage drive train components
- Differential vacuum
  - Pressure limiting apertures in liner tube
  - Differential pumping by separate IGPs
- Vacuum Interface Modules (VIM)
  - Gate valves to preserve UHV (e.g., during service)



Ultra High Vacuum

Bakeable at 145 °C

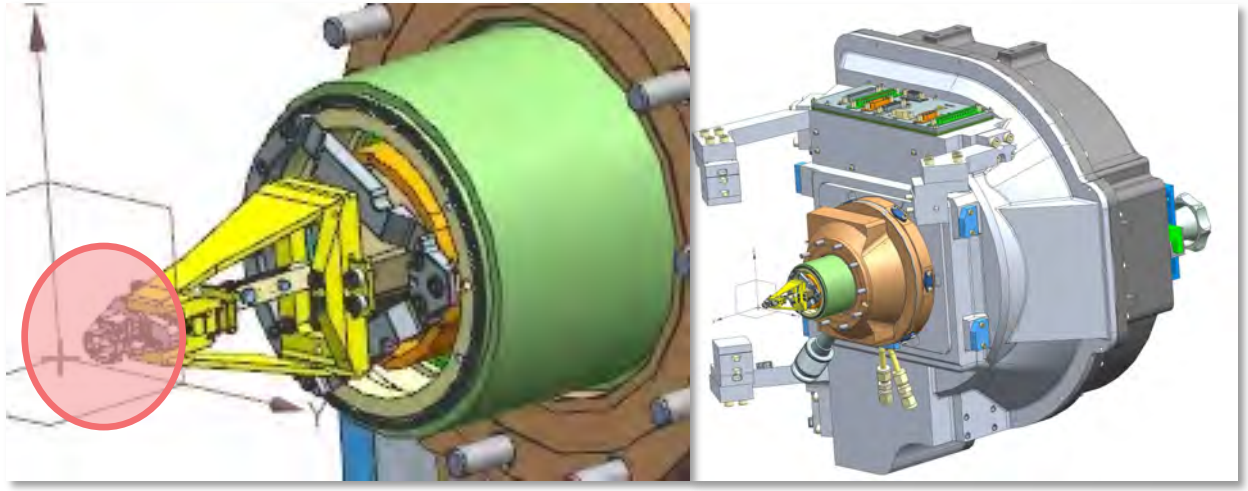
- Specimen chamber and Loader enclosed by tent
- Duration: ~ 1 week

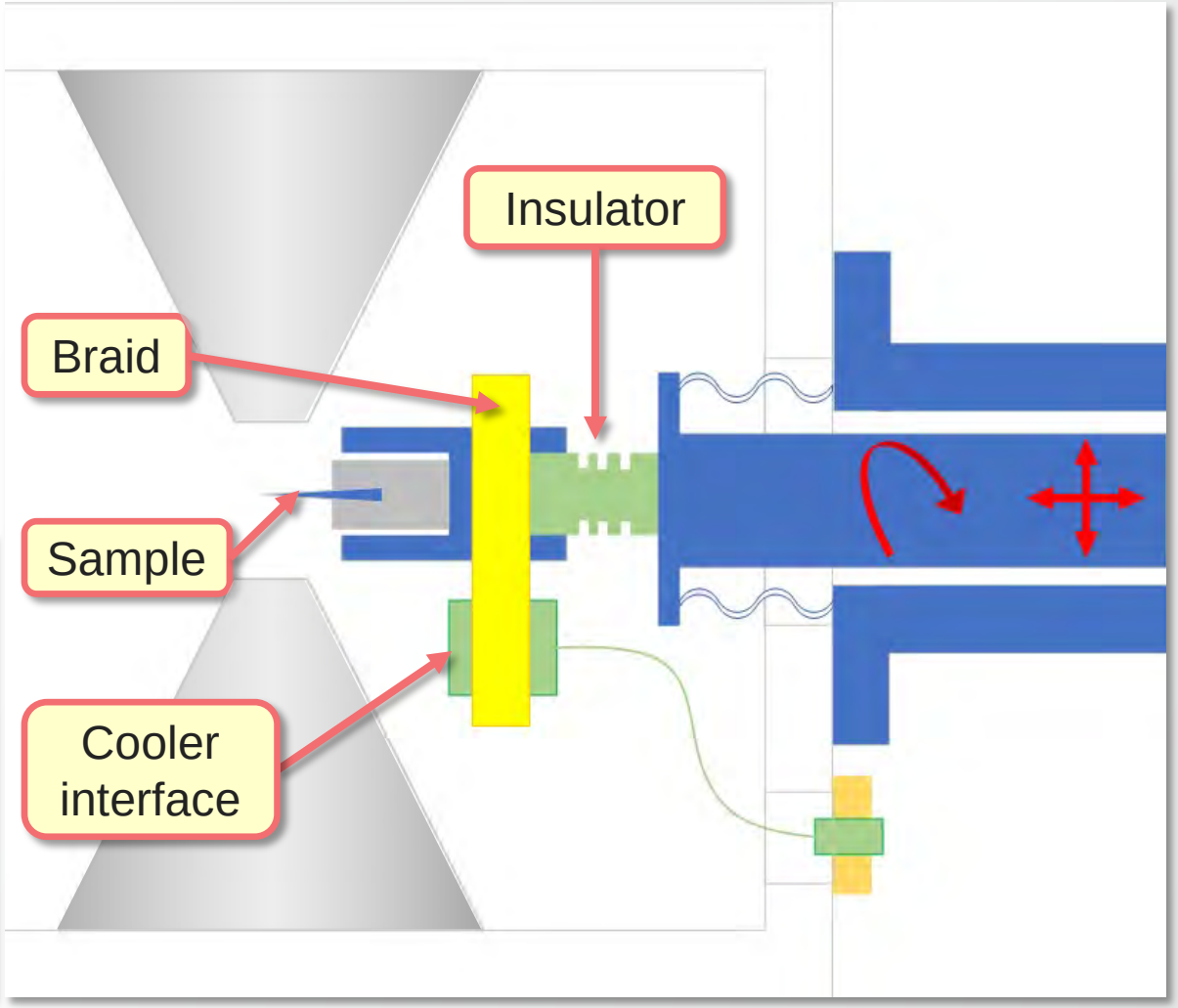


Ultra High Vacuum

UHV compatible stage

- UHV compatibility aspects:
  - Minimum moving components in vacuum
  - Cartridge loading from UHV Storage Chamber
  - Integrated heaters for bakeout
- -90º to +90º alpha tilt: full range tomography
- Compliant coupling towards Cryo cooler

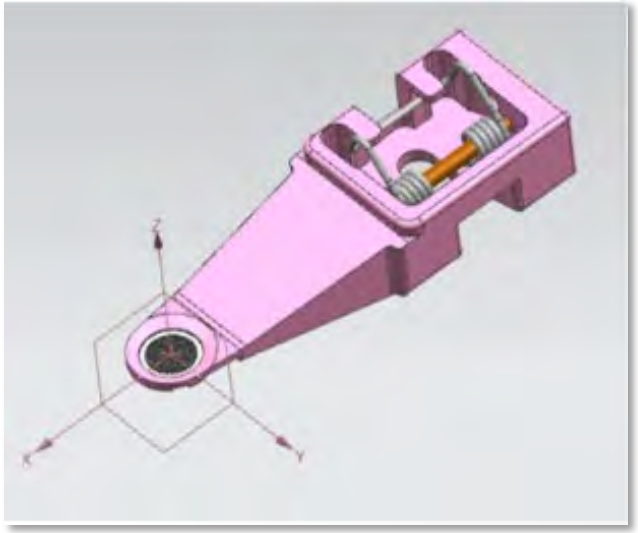


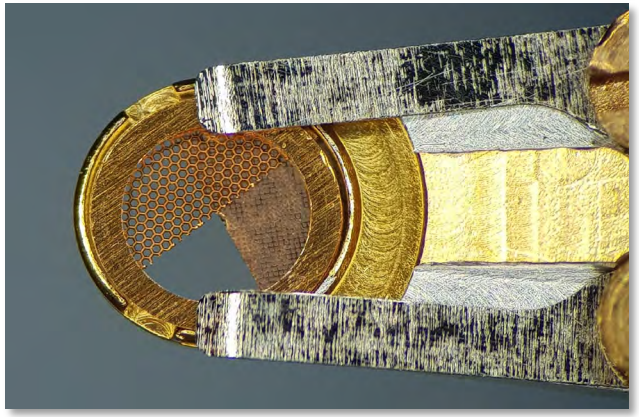


Ultra High Vacuum

Cartridge variants for different sample types

3mm grid cartridge



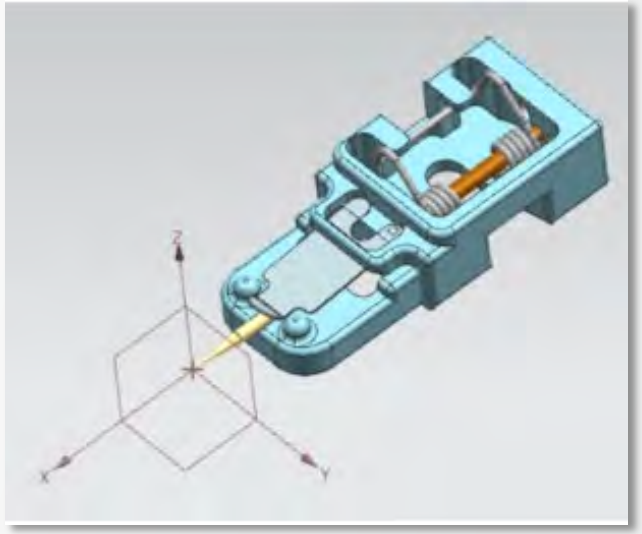


± 90° Alpha tilt

± 6° Beta tilt

(exceptionally up to ± 10°)

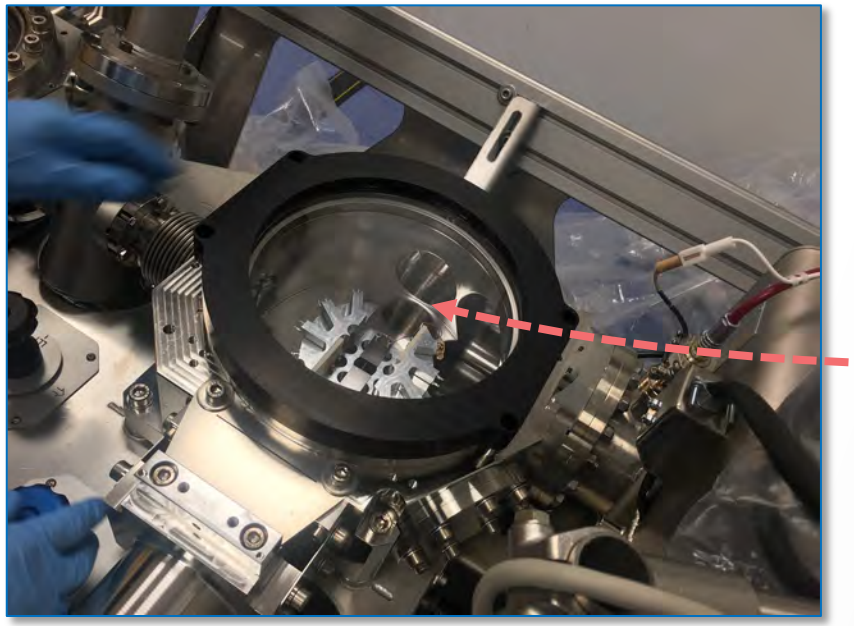
APT cartridge

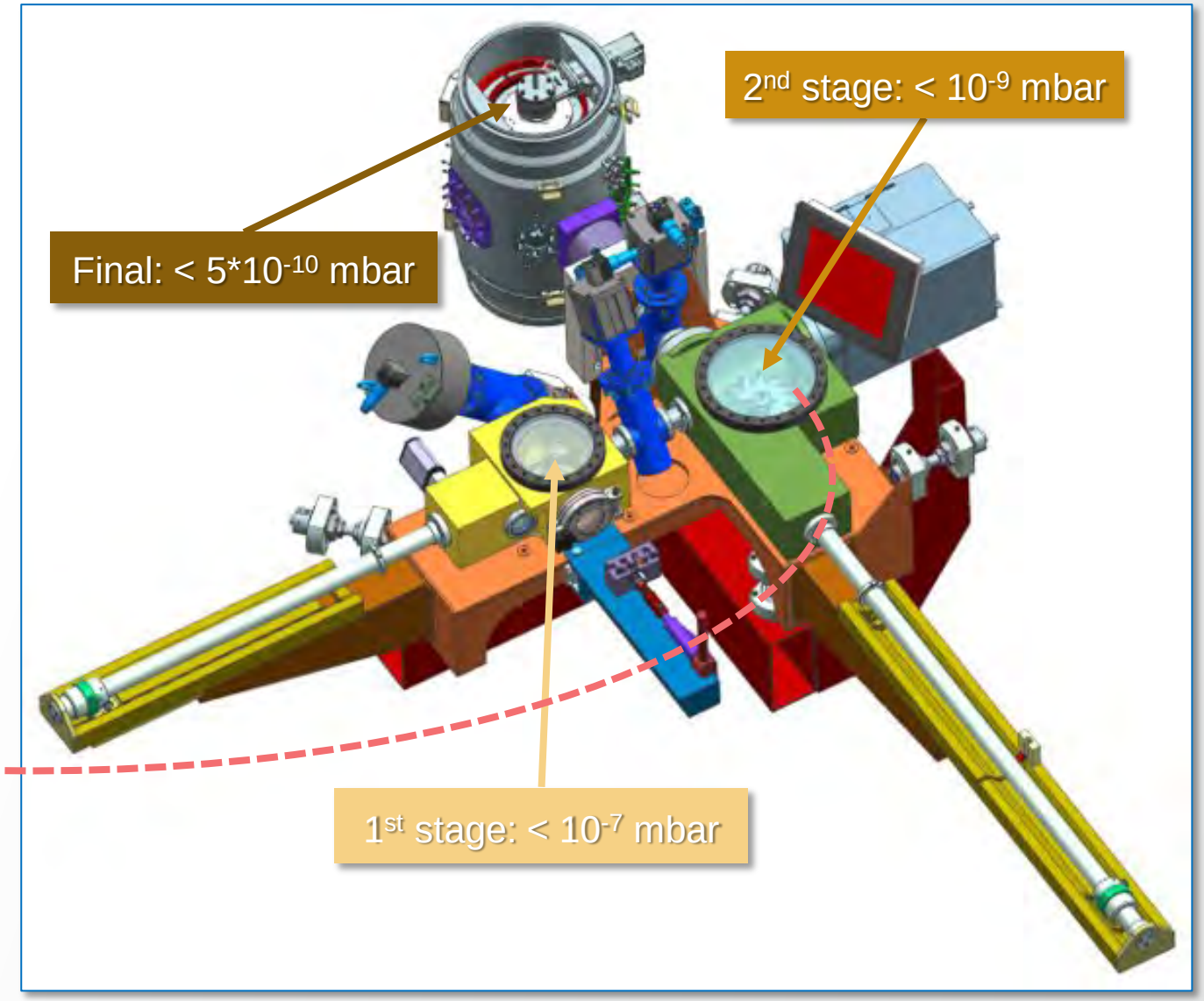


Ultra High Vacuum

Sample loading

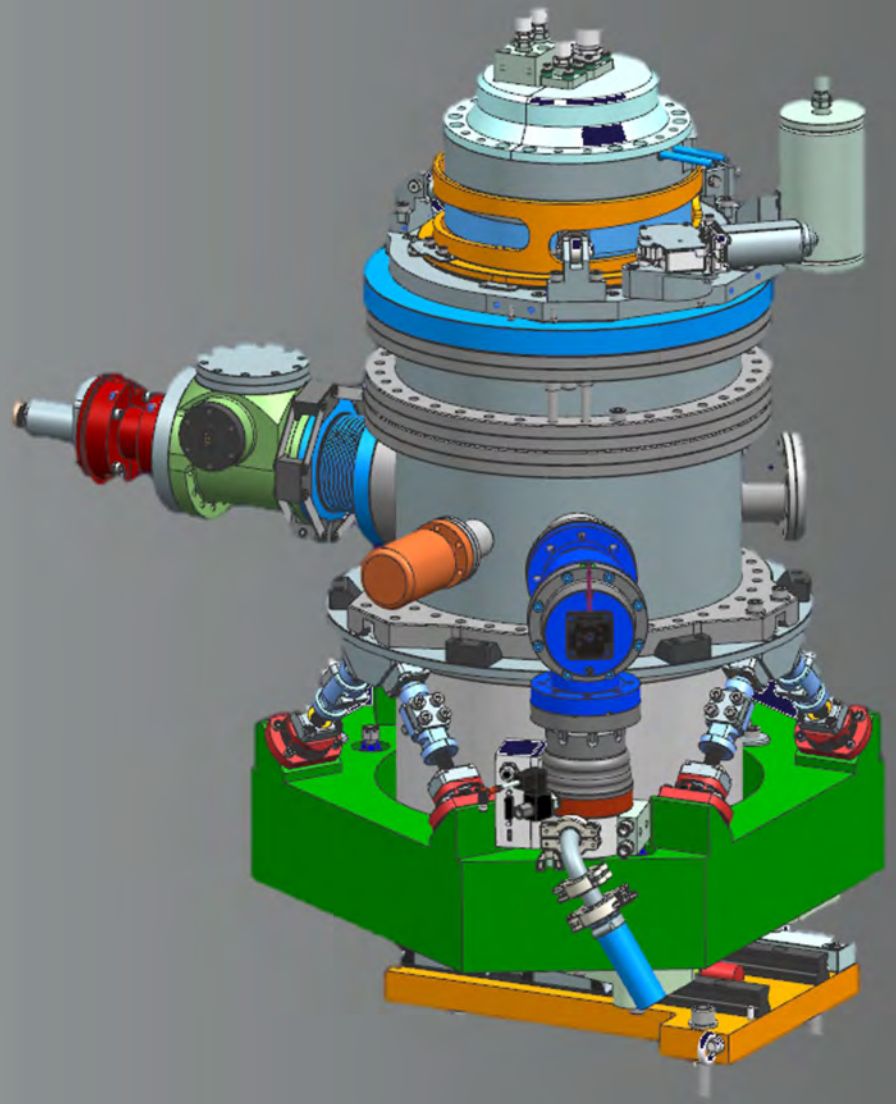
- Two-stage sample loading process
  - Load lock
  - Storage chamber
  - ~ 30 min pumping per stage
- Storage chamber prepared for direct UHV connection





Vibration-free cryo cooling

The battery cooler

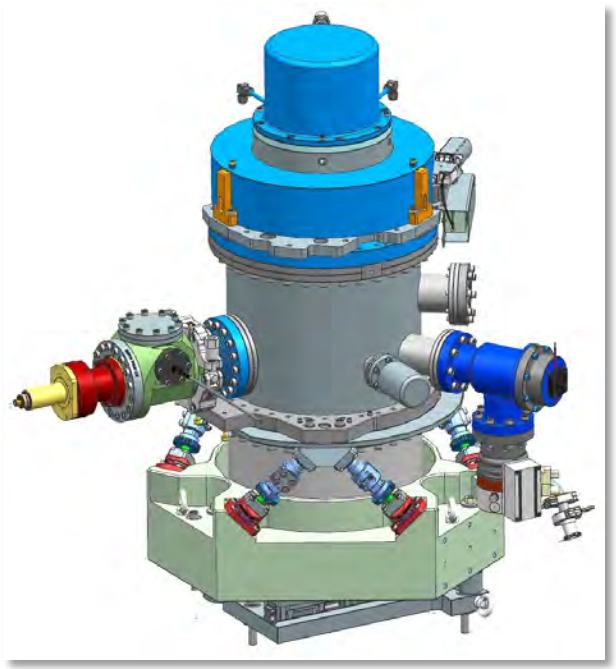


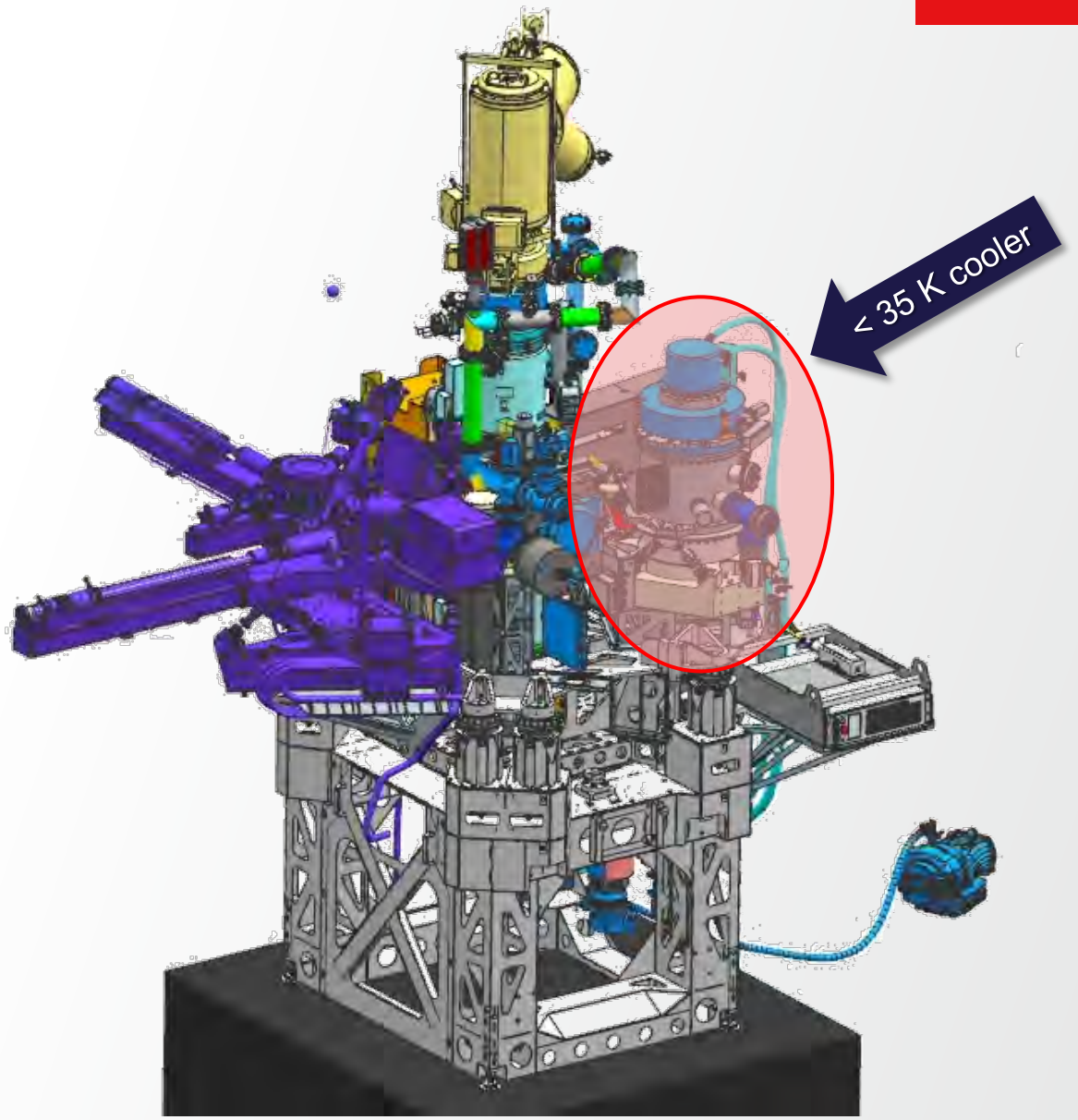
The battery cooler

Vibration-free *adjustable* cooling

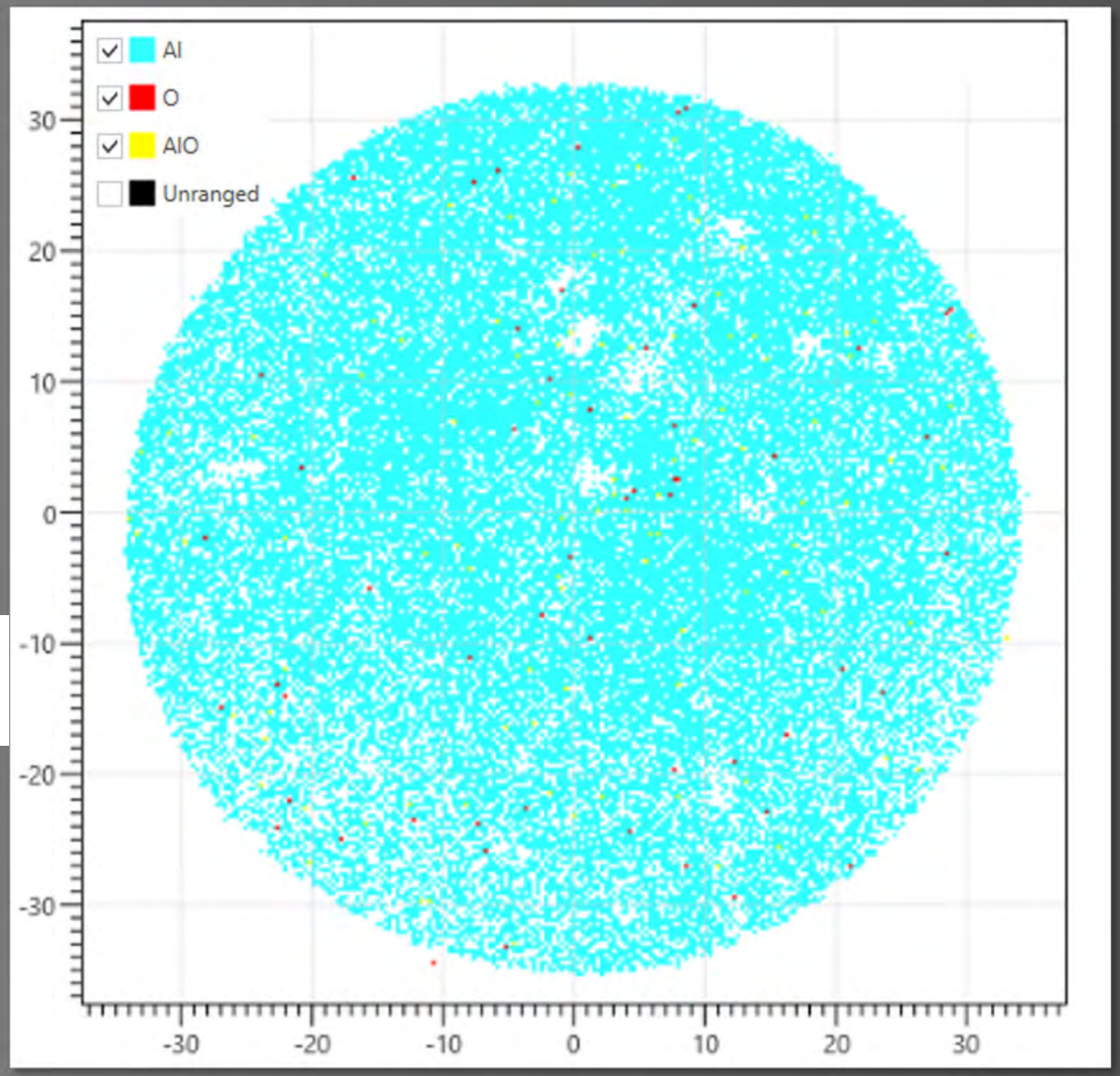
Cooler module

- < 35 K – 100 K
- Vibration free





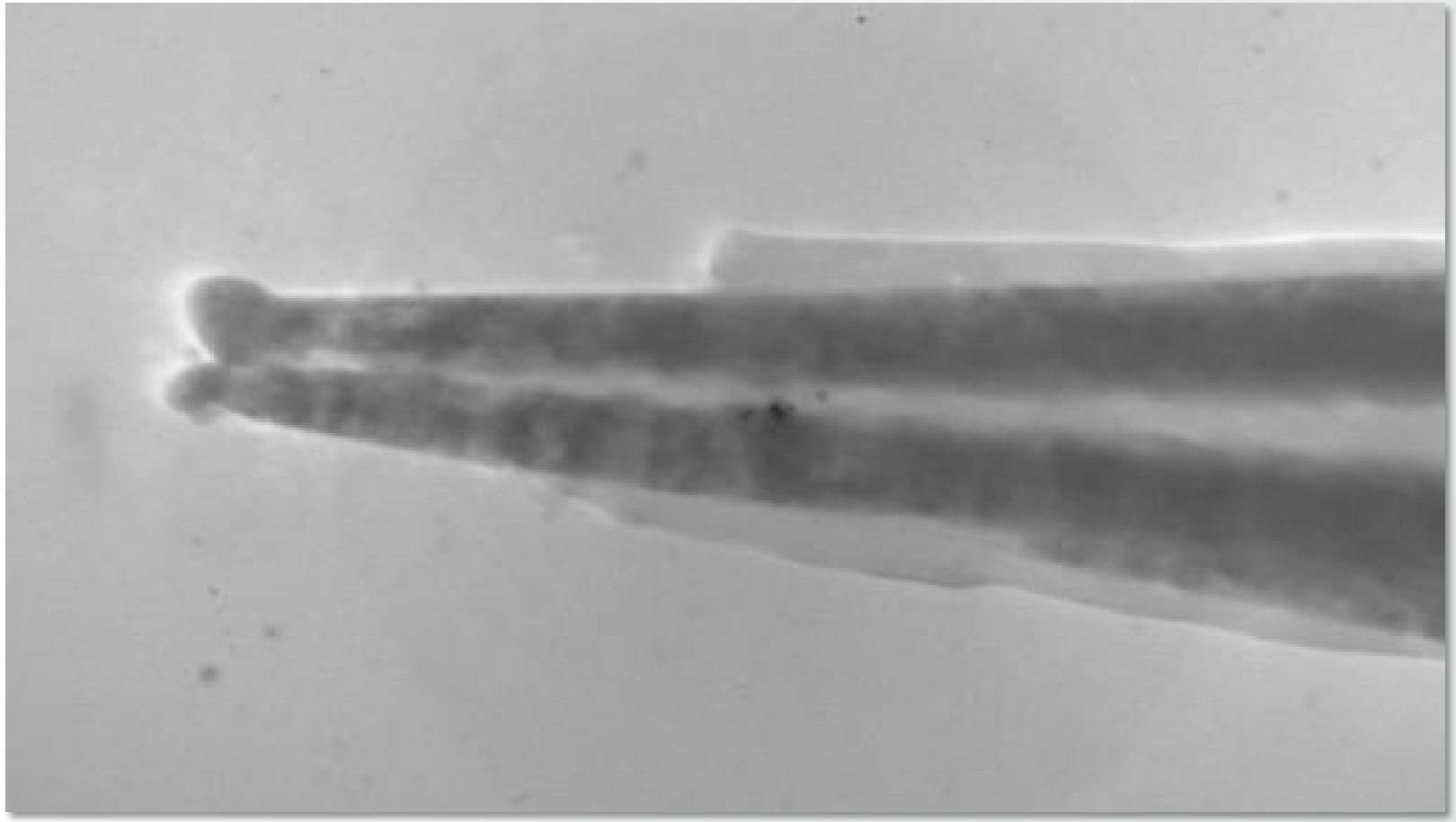
First experimental results



APT & TEM data on NiCr multilayer

Sample and data provided by Cameca

- Standard Reference Material from NIST: SRM 2135c
- Consisting of Ni- and Cr-multilayers
  - Cr evaporates at 27 V/nm
  - Ni evaporates at 35 V/nm



APT & TEM data on NiCr multilayer

APT data acquisition

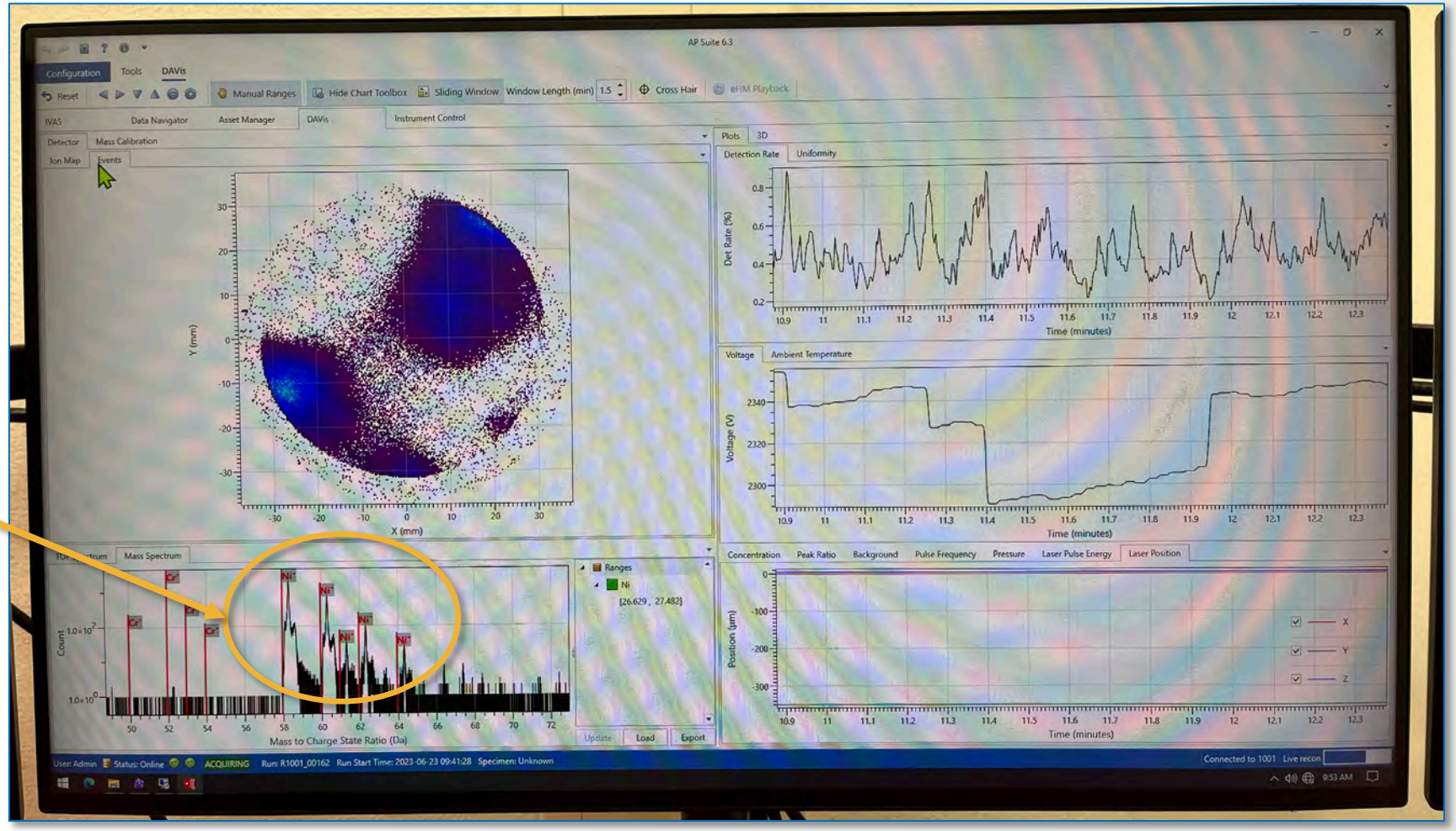
- Reasonable acquisition for about 26 min
- Expected elements were manually entered
  - Auto-calibration attempts were unsuccessful, elements shown are from a previous data run
- Spectral data looks good

APT & TEM data on NiCr multilayer

APT data acquisition

- Run 2 adds 600,000 ions to the 400,000 ions of run 1 for a total of **1 million ions**

Nickel shows
5 stable isotopes:
58, 60, 61, 62, 64

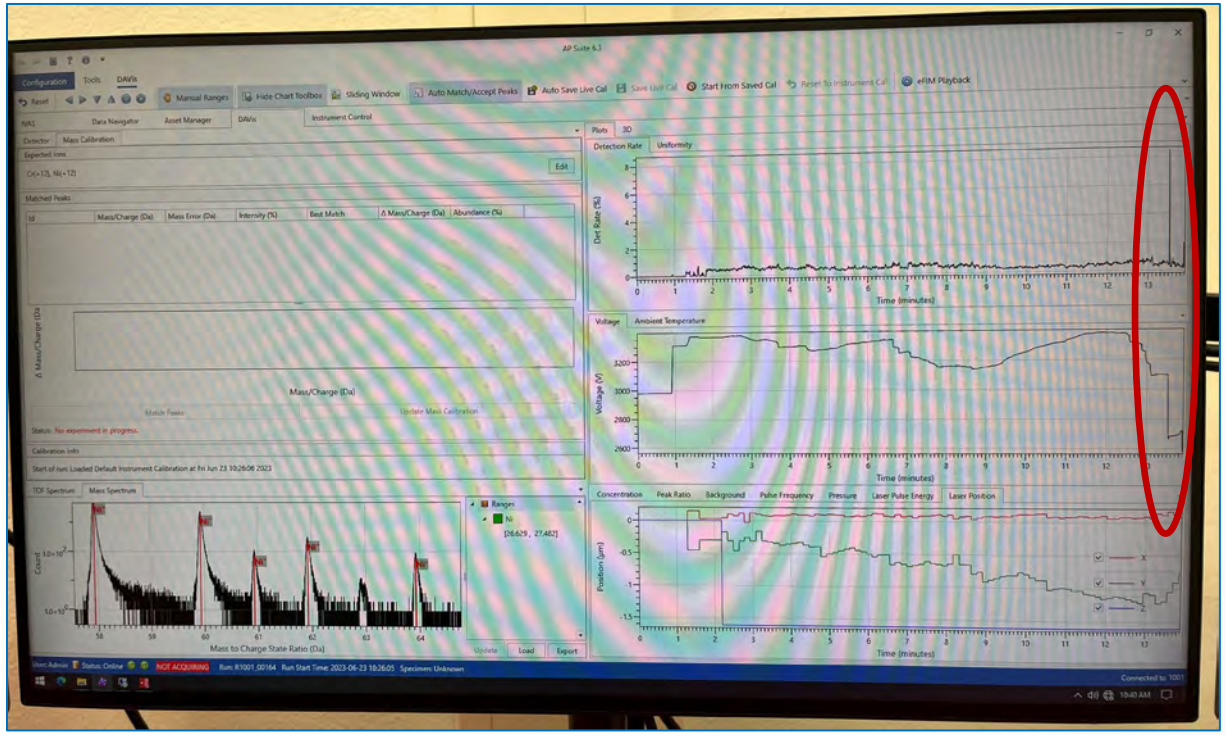


NiCr1-Exp2-IMG7887

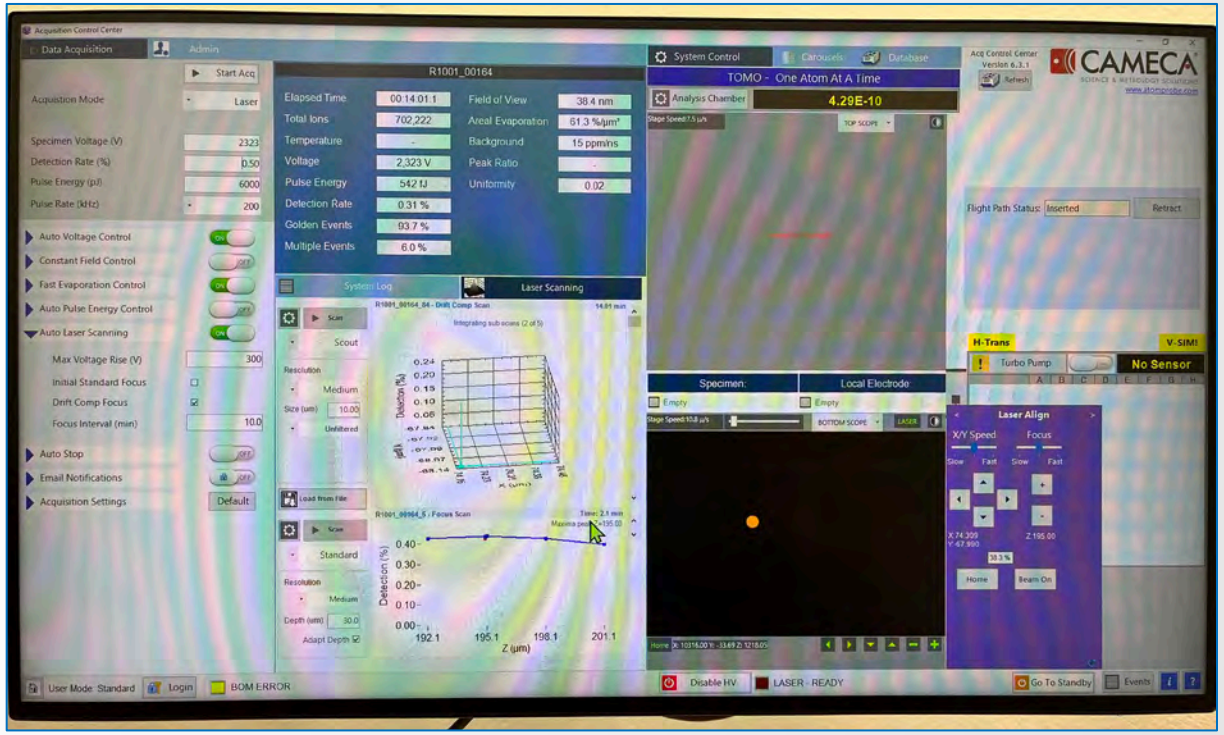
APT & TEM data on NiCr multilayer

APT data acquisition

- Run 4 adds 700,000 ions for a total of **2.7 million ions**
- Fracture event happens 13.5 minutes into run 4



NiCr1-Exp2-IMG7897

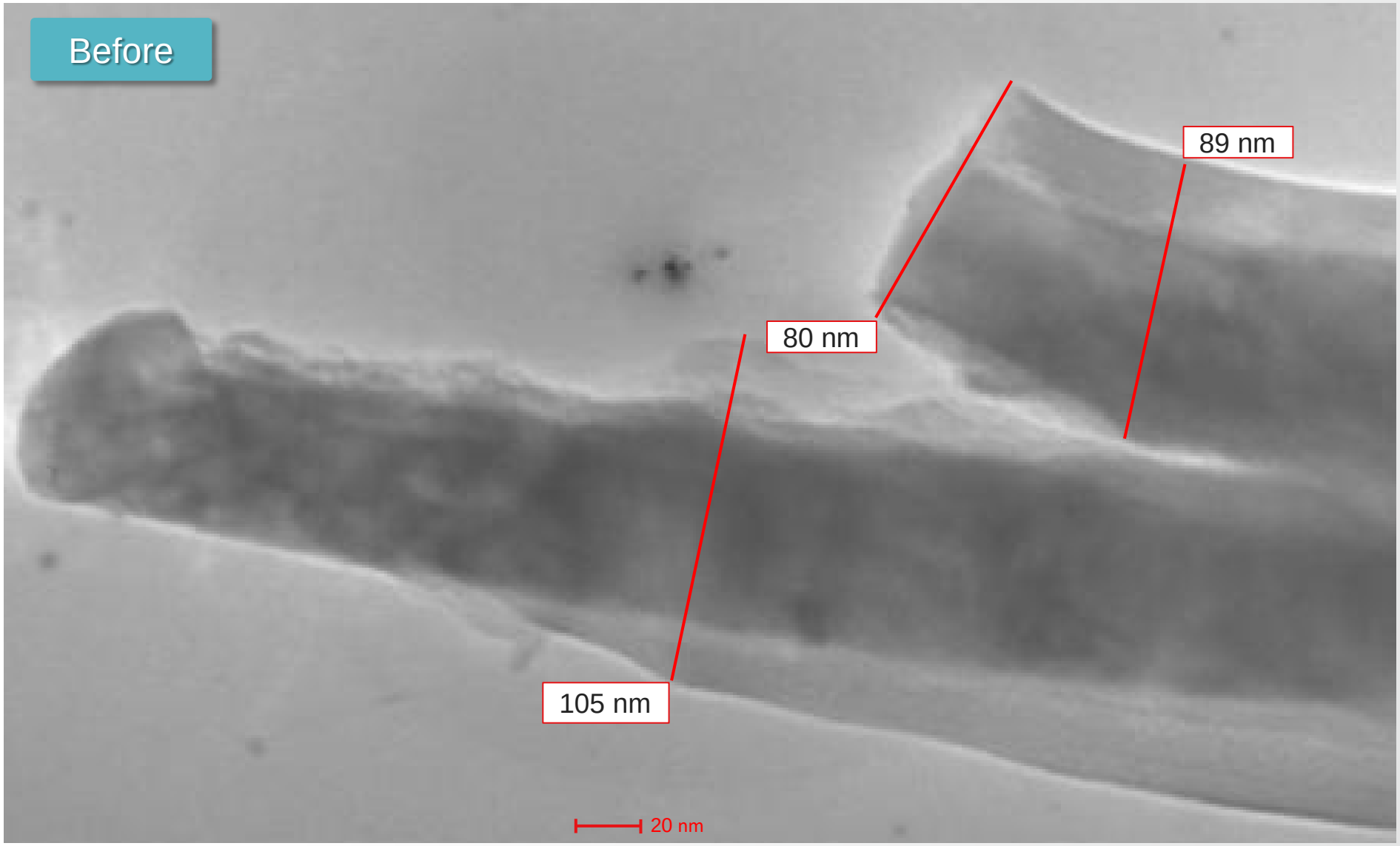


NiCr1-Exp2-IMG7896

APT & TEM data on NiCr multilayer

TEM imaging ***before*** and ***after*** run 4:

- Run 4: **2.7M ions**
- Fracture event at 13.5 min into run 4



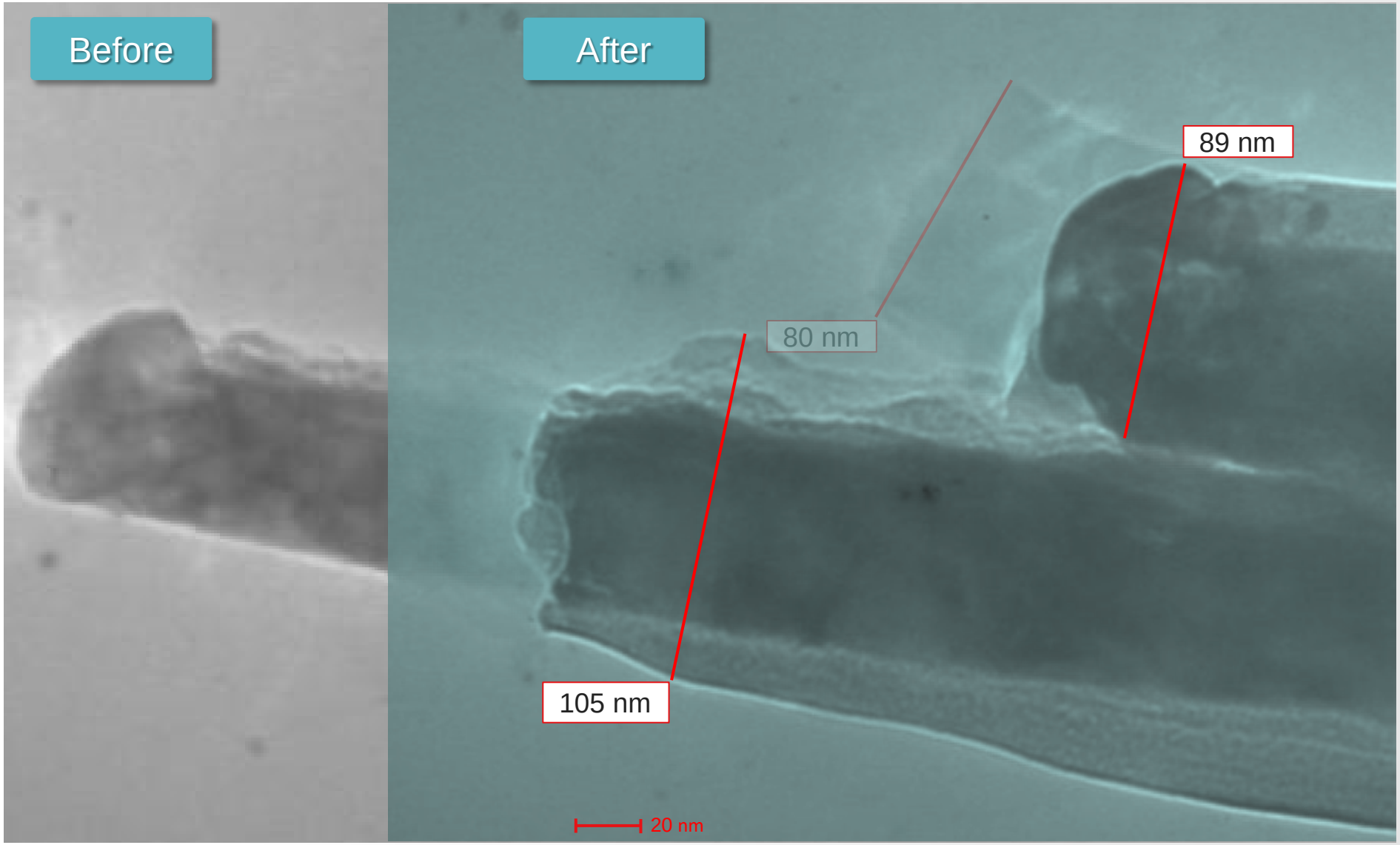
NiCr1-Exp2-15.41.16 SA 46 k

NiCr1-Exp2-17.43.17 SA 74 k

APT & TEM data on NiCr multilayer

TEM imaging ***before*** and ***after*** run 4:

- Run 4: **2.7M ions**
- Fracture event at 13.5 min into run 4



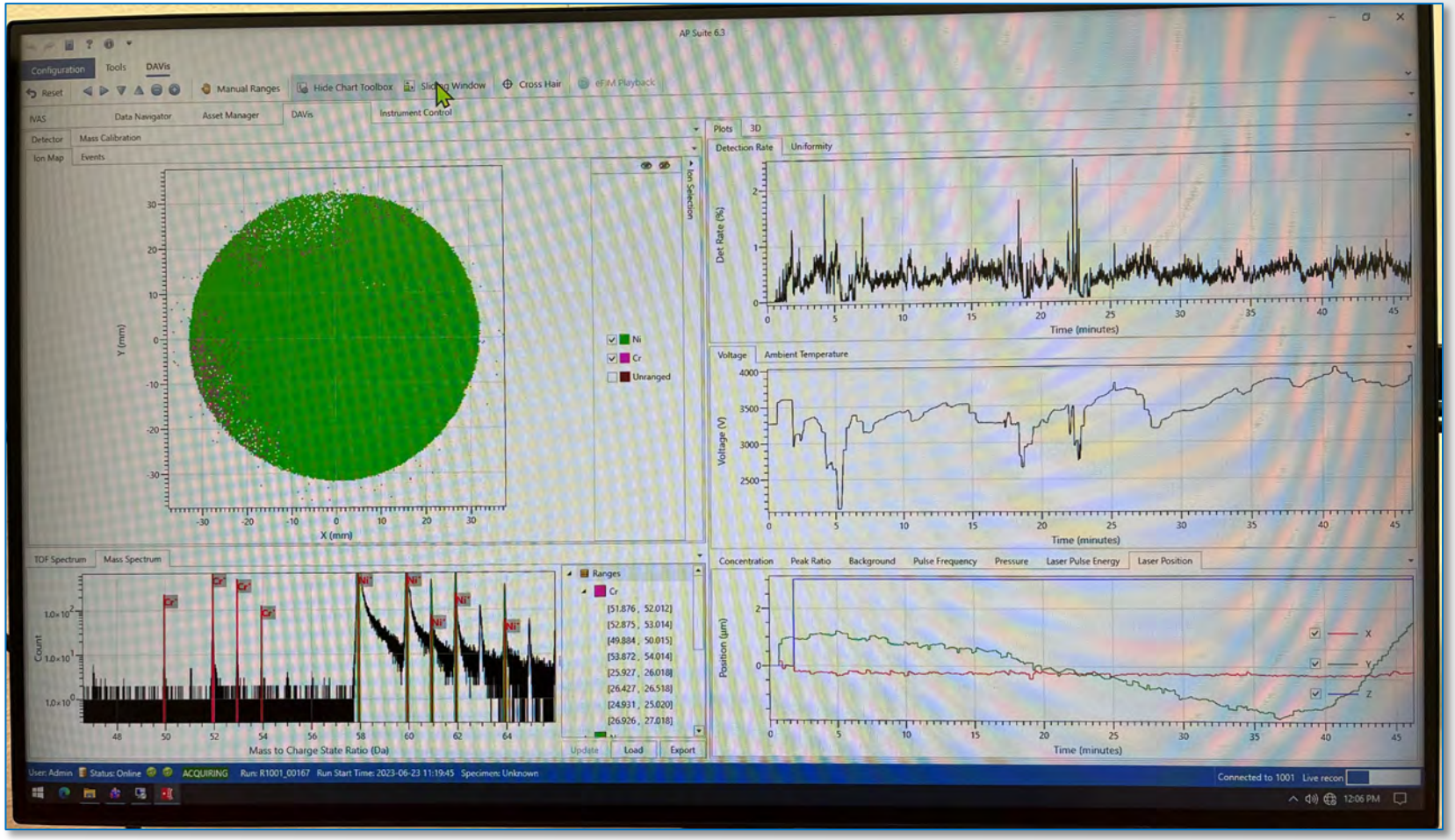
NiCr1-Exp2-15.41.16 SA 46 k

NiCr1-Exp2-17.43.17 SA 74 k

APT & TEM data on NiCr multilayer

APT data acquisition

- Run 7 adds another 2,300,000 ions for a total of **6.2 M ions**
- Full run: 46:32 min
- Near instantaneous lock of laser positioning

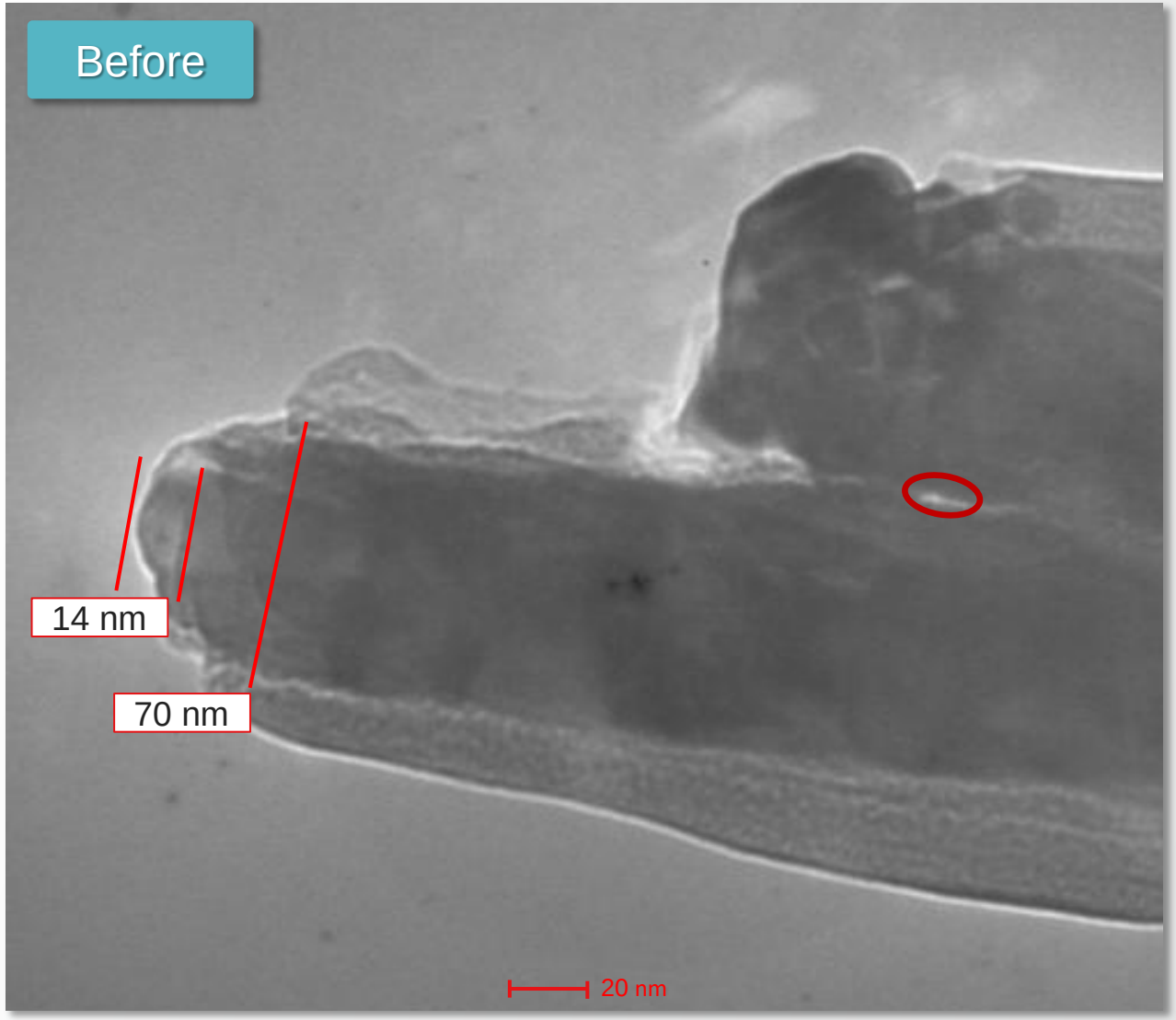


NiCr1-Exp2-IMG_7906

APT & TEM data on NiCr multilayer

TEM imaging ***before*** and ***after*** run 7:

- Run 7 adds another 2,300,000 ions for a total of **6.2 M ions**
- Full run: 46:32 min
- Needle recedes 14 nm

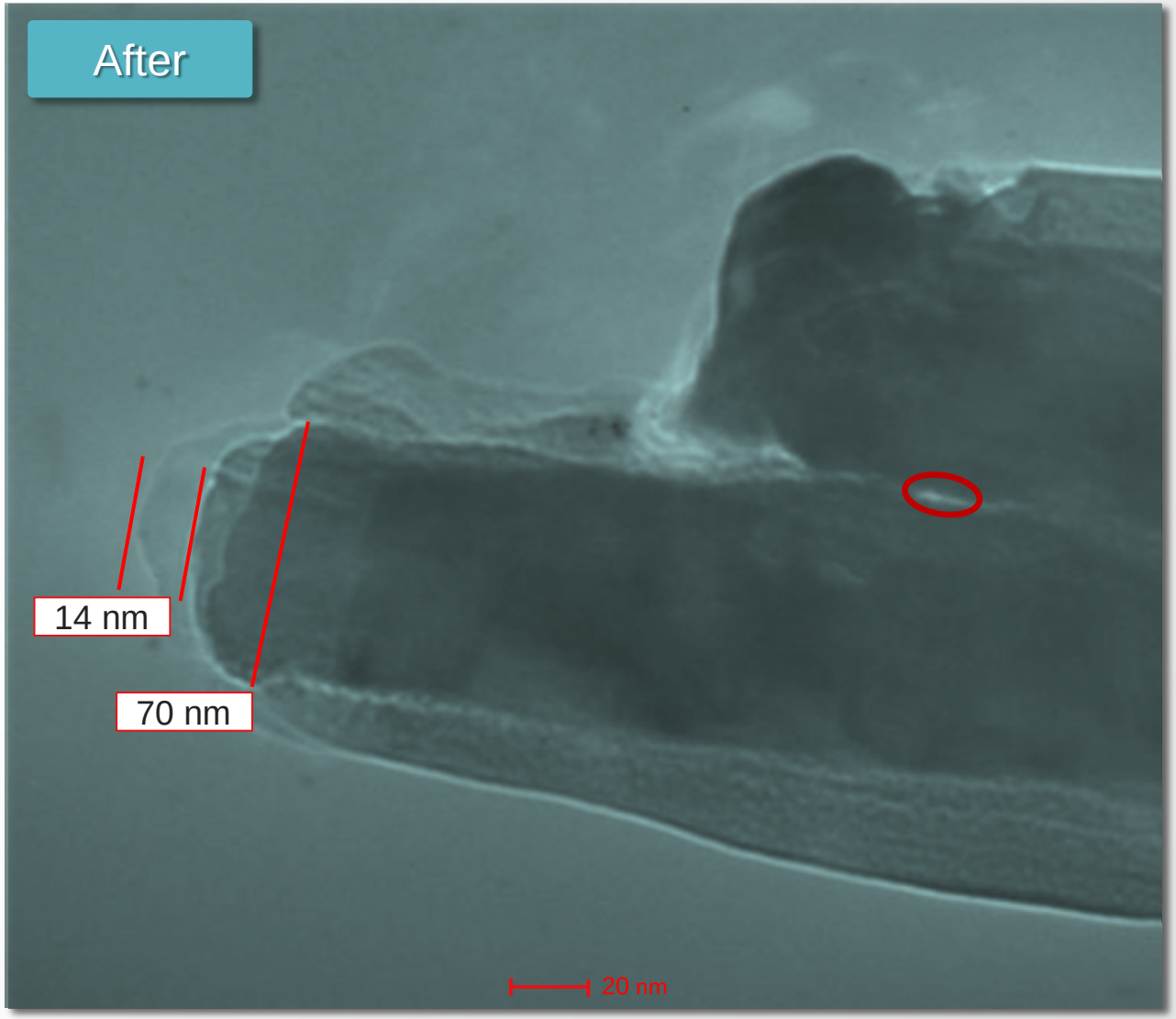


NiCr1-Exp2-18.20.22 SA 74 k
NiCr1-Exp2-19.09.40 SA 74 k

APT & TEM data on NiCr multilayer

TEM imaging ***before*** and ***after*** run 7:

- Run 7 adds another 2,300,000 ions for a total of **6.2 M ions**
- Full run: 46:32 min
- Needle recedes 14 nm

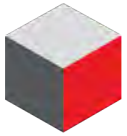


NiCr1-Exp2-18.20.22 SA 74 k
NiCr1-Exp2-19.09.40 SA 74 k

Closing remarks

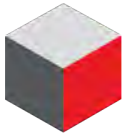
Closing remarks:

Dedicated UHV vibration-free cryogenic (S)TEM + APT

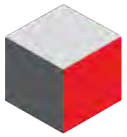


Semiconductors:
3D-visualization of dopants

Energy materials:
Reactive elements in a pristine state, chemical reactions in controlled conditions



Quantum materials:
Experimental stimuli at stable conditions and adjustable, low temperatures



Surface analysis:
Chemistry in ultra-clean conditions, long experimentation windows

Thank you