Modern In Situ TEM Ion Irradiations with Real-Time AI-Powered Data Analysis

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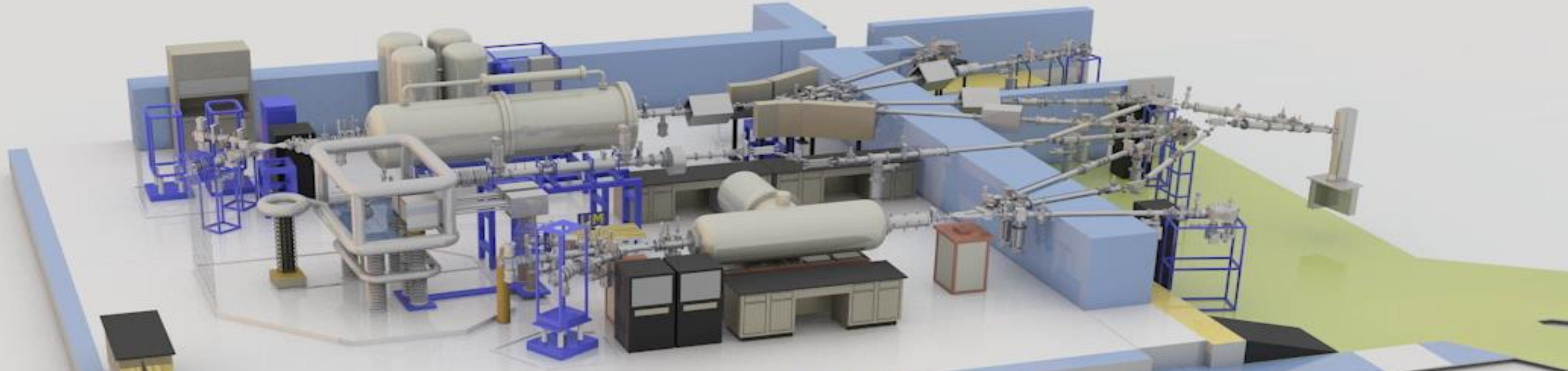
MICHIGAN ENGINEERING

MICHIGAN ION BEAM LABORATORY

MIBL: Michigan Ion Beam Laboratory



Zhijie (George) Jiao
MIBL Manager

Kevin G. Field
MIBL Director

In 2022, ~192 irradiations were conducted by 113 researchers working on 45 projects and using ~5,713 hr of beam time.

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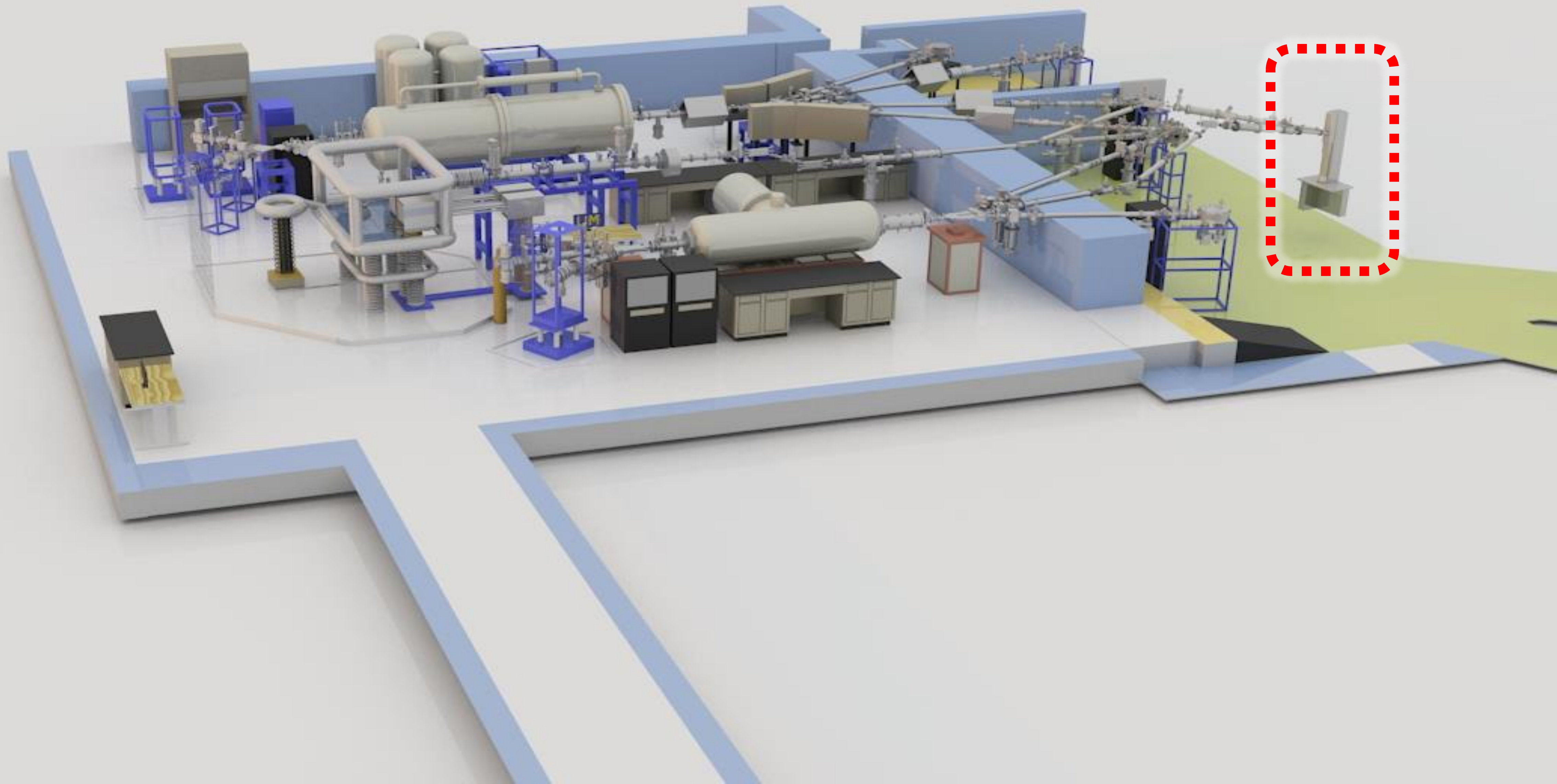
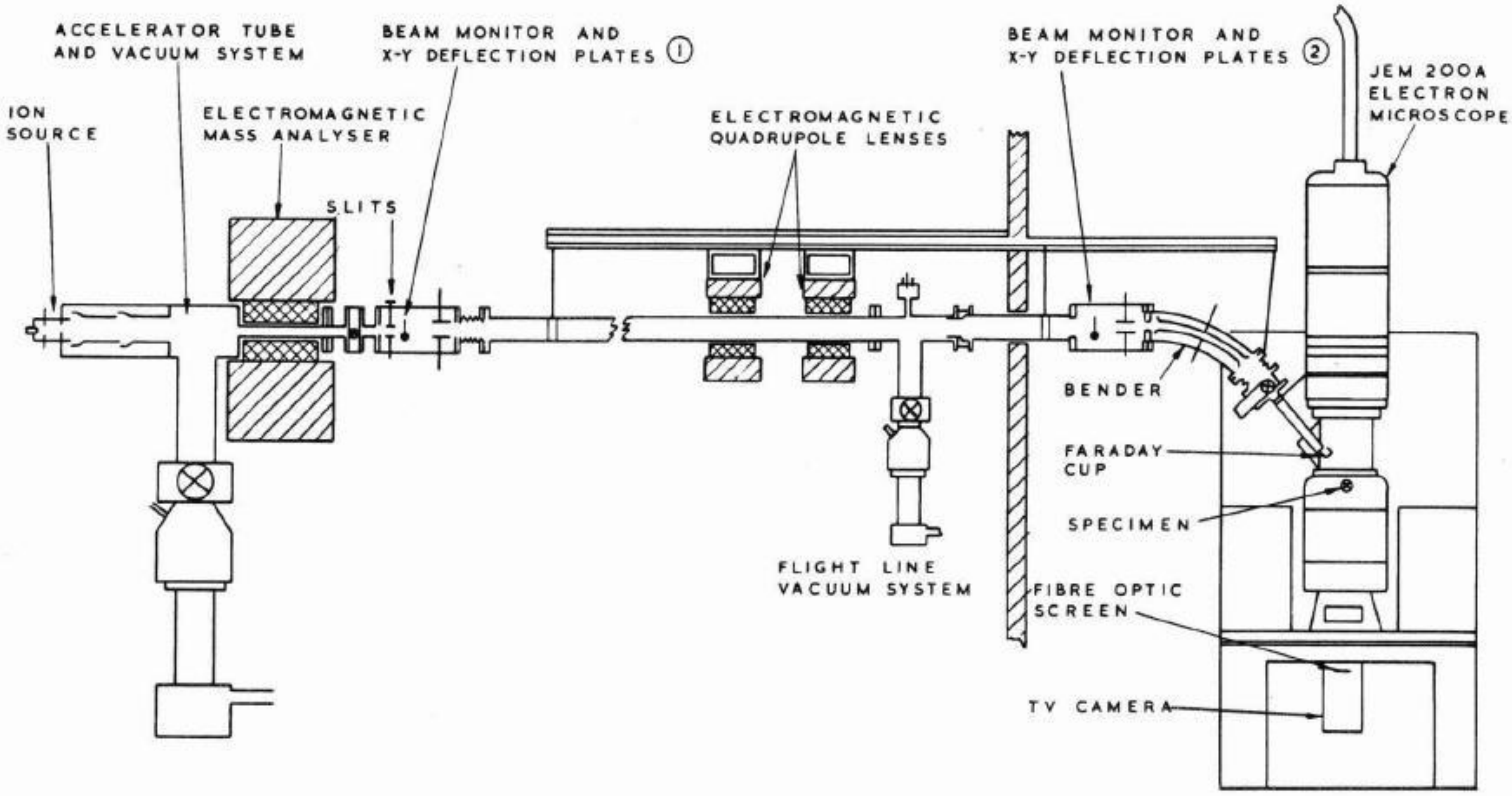


Cut-through of most "modern" in situ TEM ion irradiation configurations



Rad. Eff. **22** (1974) p163

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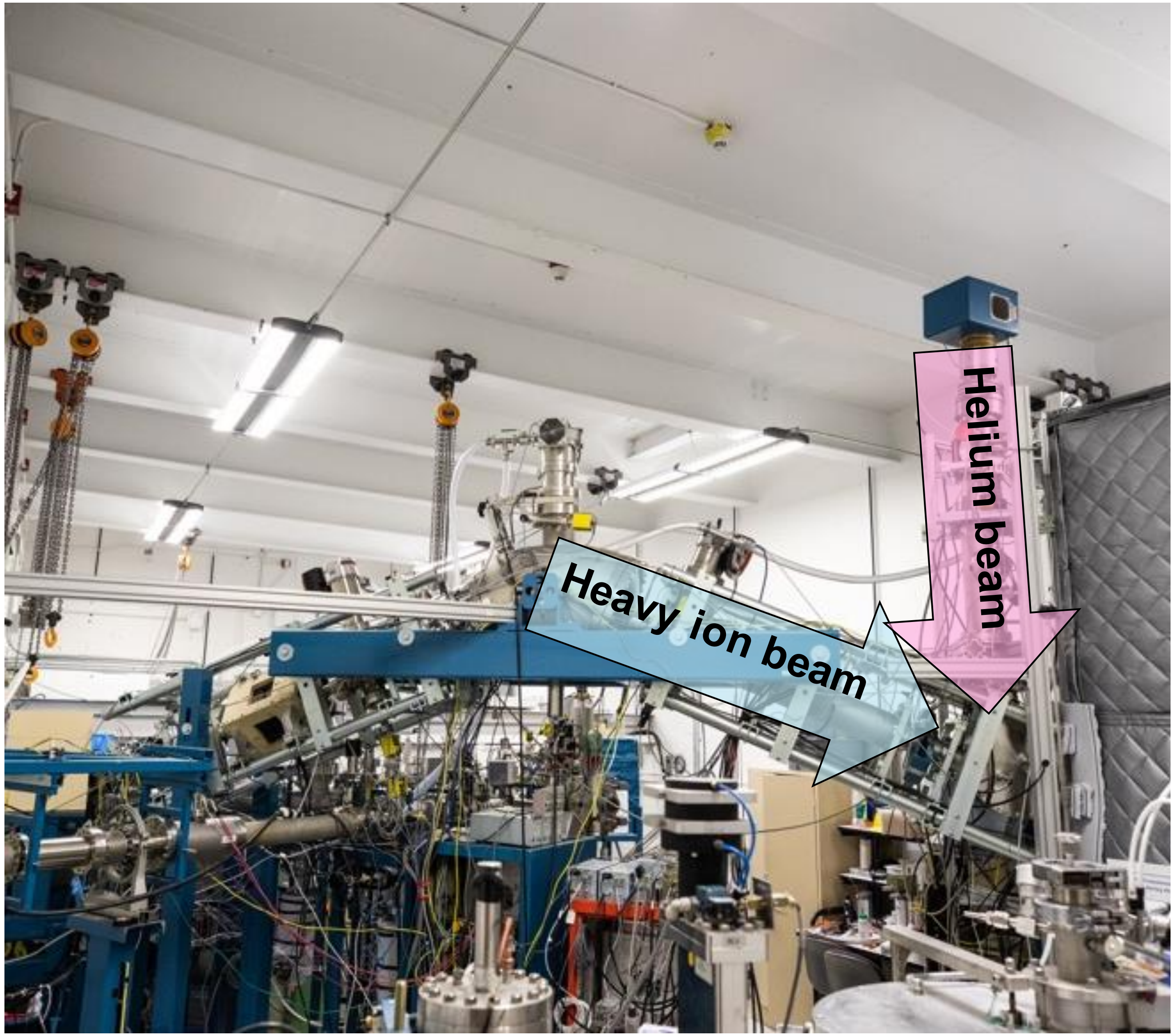
MiTEM is capable of in-situ single and dual beam TEM ion irradiations



Ion Beam Systems --- Two beam lines linked to:

- A 400kV NEC accelerator equipped with a Danfysik Model 921 ion source can provide a wide range of ions (H^+ to Au) ions with energy 20 keV ~1.6 MeV
- An Alphatross NEC ion source can deliver H and He ions in the energy range 5~30 keV

- Dual beam irradiation capability
- Remote control

Slide courtesy of K. Sun

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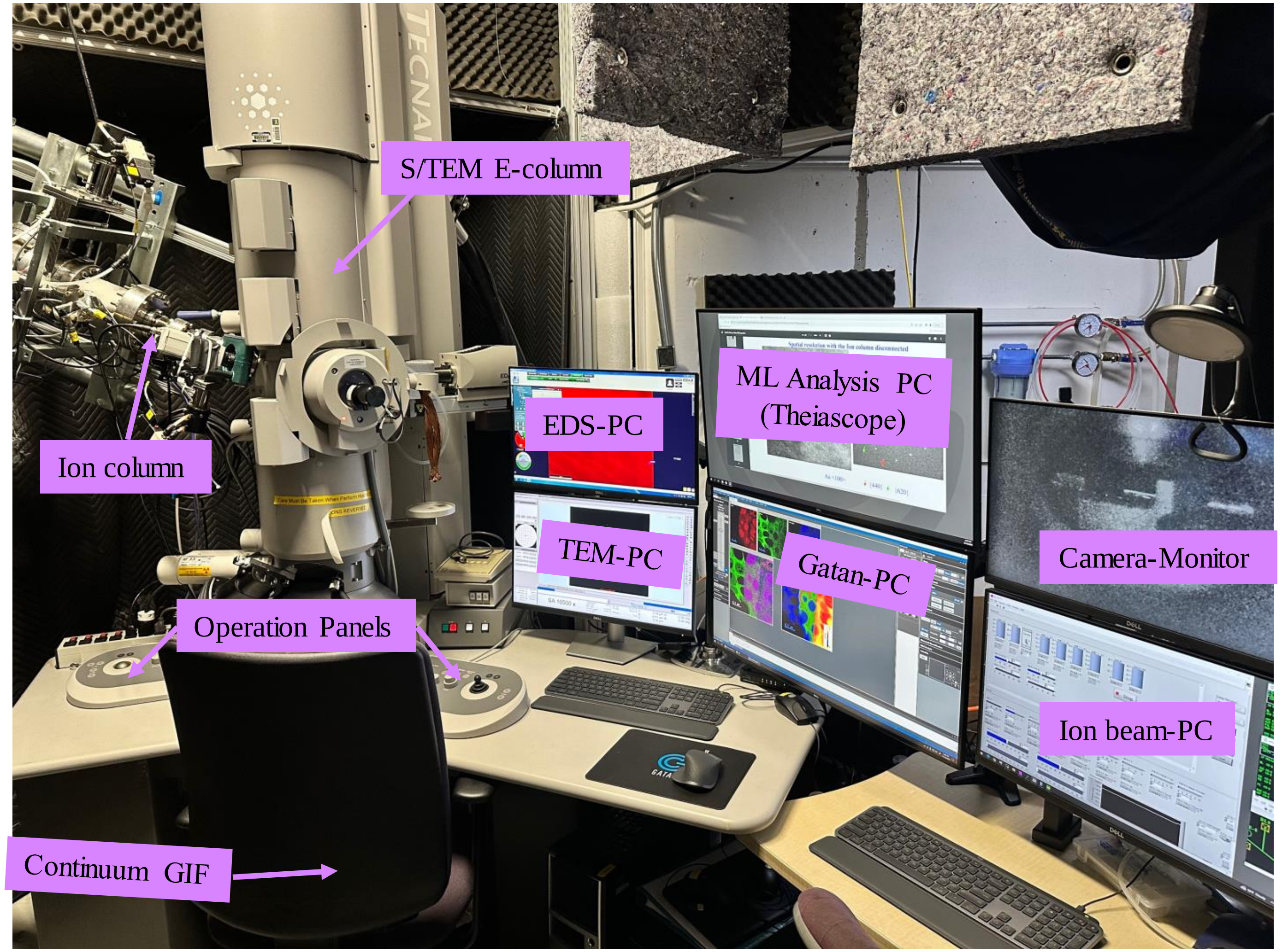
MiTEM is unique in the US as it is AI/ML enabled with in-situ S/TEM capabilities including EELS/EFTEM



TEM Specs---TF30 TEM/**STEM**

- **Gun**: Thermo-FEG, HT: 100, 200 and 300keV
- **Operation modes**: CTEM: 0.1 nm lattice resolution and STEM: 0.34 nm point-to-point resolution
- **Cameras**: 2Kx2K UltraScan Pre-GIF CCD and 2Kx2K GIF CCD
- **Detectors**: HAADF, ADF and BF
- **EDS**: EDAX Appolo 30mm^2 SDD
- **GIF**: Gatan Continuum ER GIF---Dual EELS and STEEM SI and EFTEM SI
- **Holders**: TF low dose DT ($\alpha \sim \pm 70°, \beta \sim \pm 30°$), DENS Wildfire DT and Modified Gatan 952 DT ($\alpha \sim \pm 30°, \beta \sim \pm 25°$) Heating holders and two home made Faraday Cap holders and a modified Gatan 952 DT heating holder.
- **Remote control**
- **AI/ML Enabled**

Slide courtesy of K. Sun

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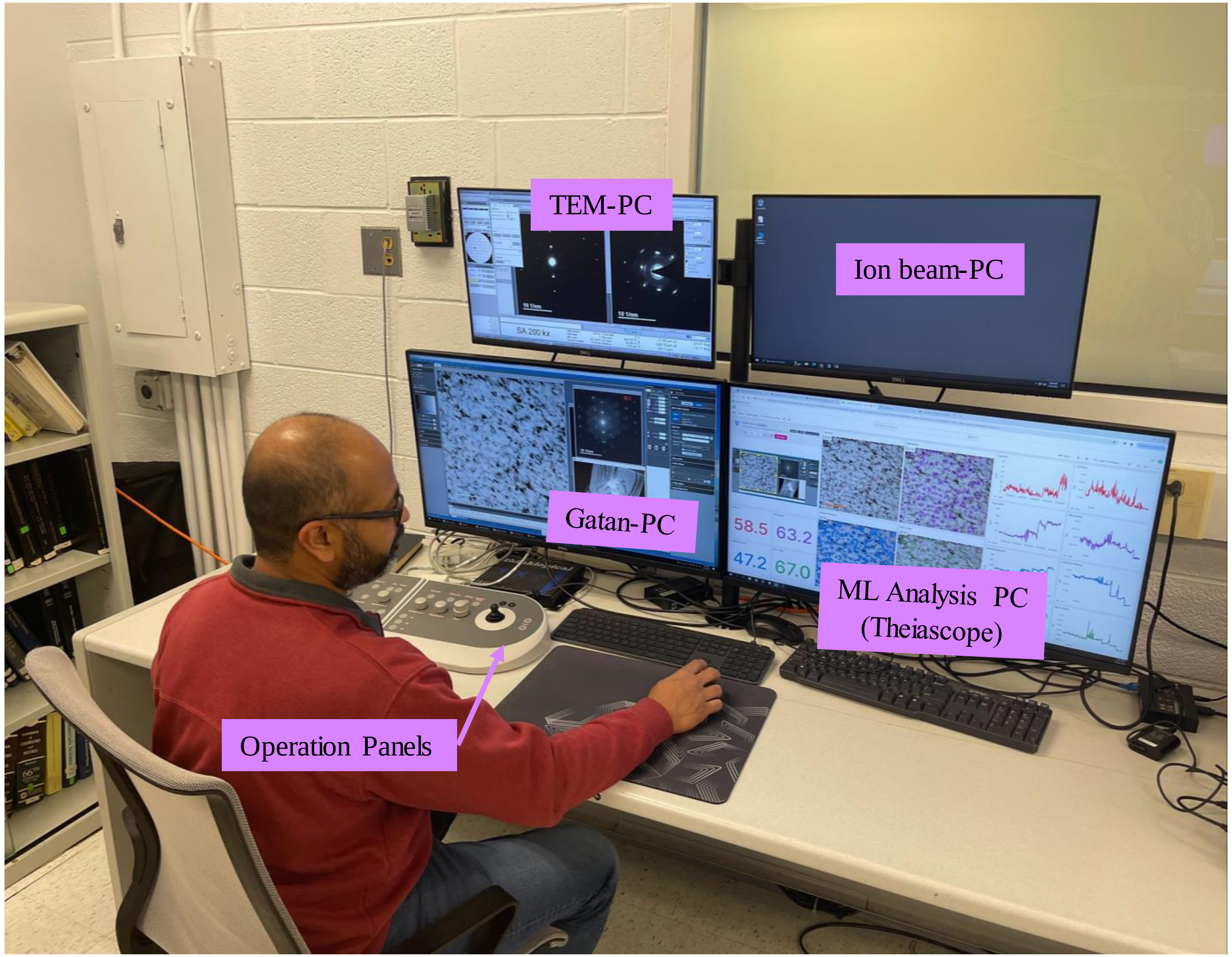

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- **Remote control**
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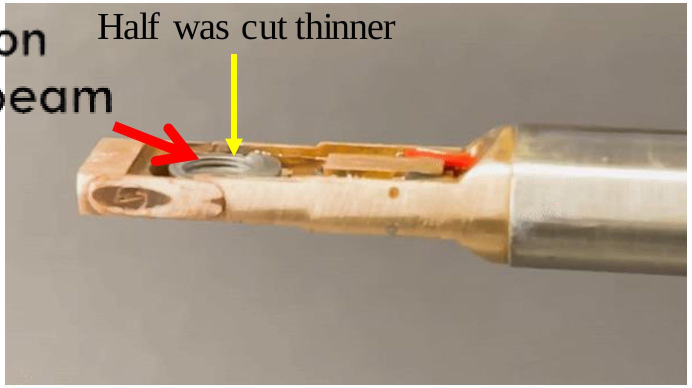
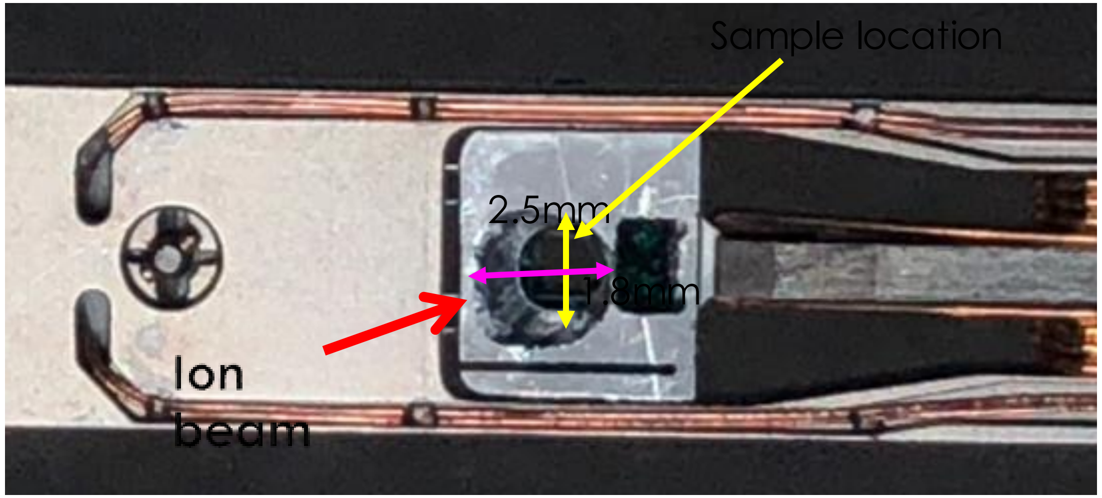
MiTEM users have access to both ***traditional heating holders*** and a ***MEMS based heating*** holder to cover a wide range of irradiation temperatures and rates

A modified Gatan 952 Double-tilt Heating Holder



- Temperature ~1000°C; Good for <500 °C
- Tilt angle range: $\alpha = \pm 30°;\ \beta = \pm 25°$

A modified DENSsolutions Double-tilt Heating Holder

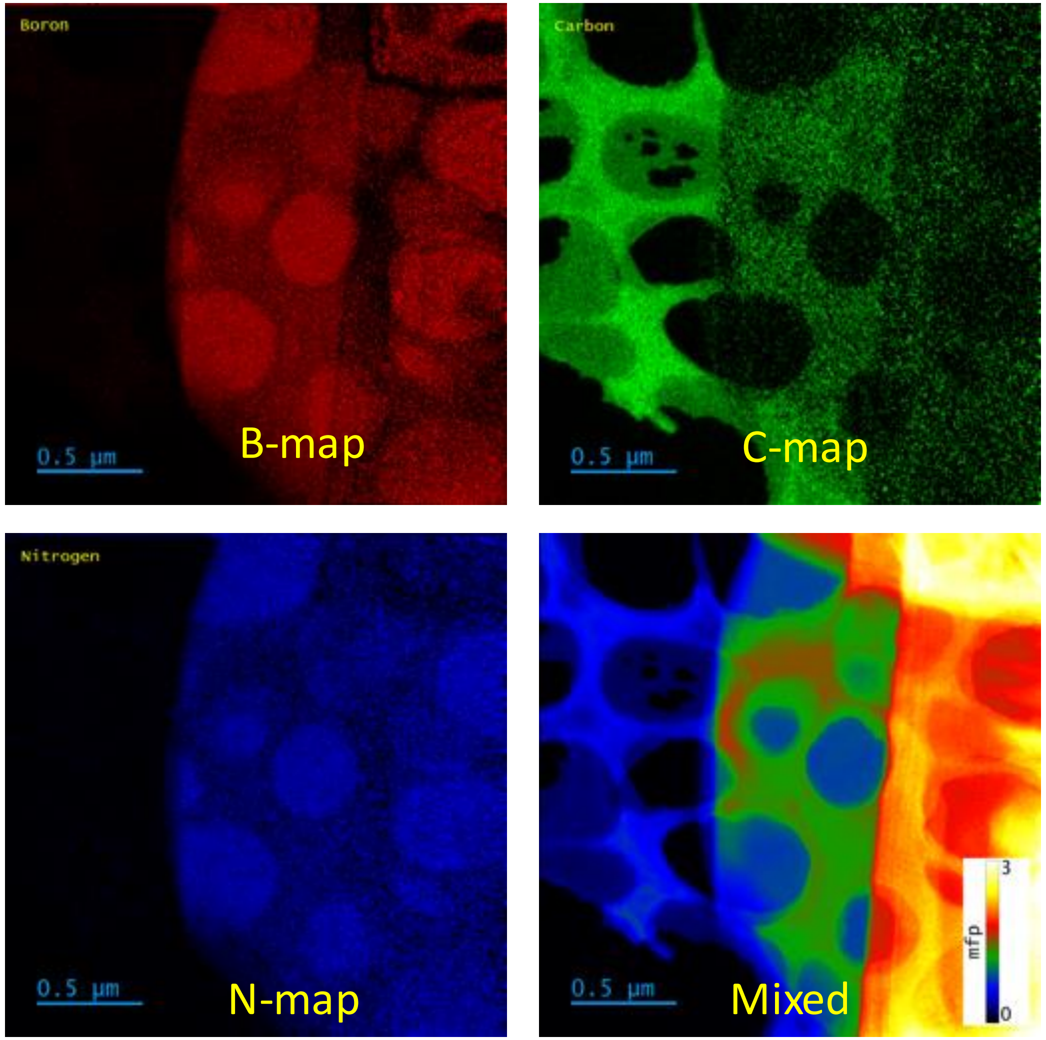


Ion beam-side of the DENS holder

- Temperature ~1300°C, Good for > 450 °C
- Tilt angle range: $\alpha = \pm 30°;\ \beta = \pm 25°$

Slide courtesy of K. Sun

MiTEM users have access to Gatan Continuum Imaging Filter (GIF) to perform high resolution ***chemical imaging*** when performing ***in-situ TEM ion irradiations***

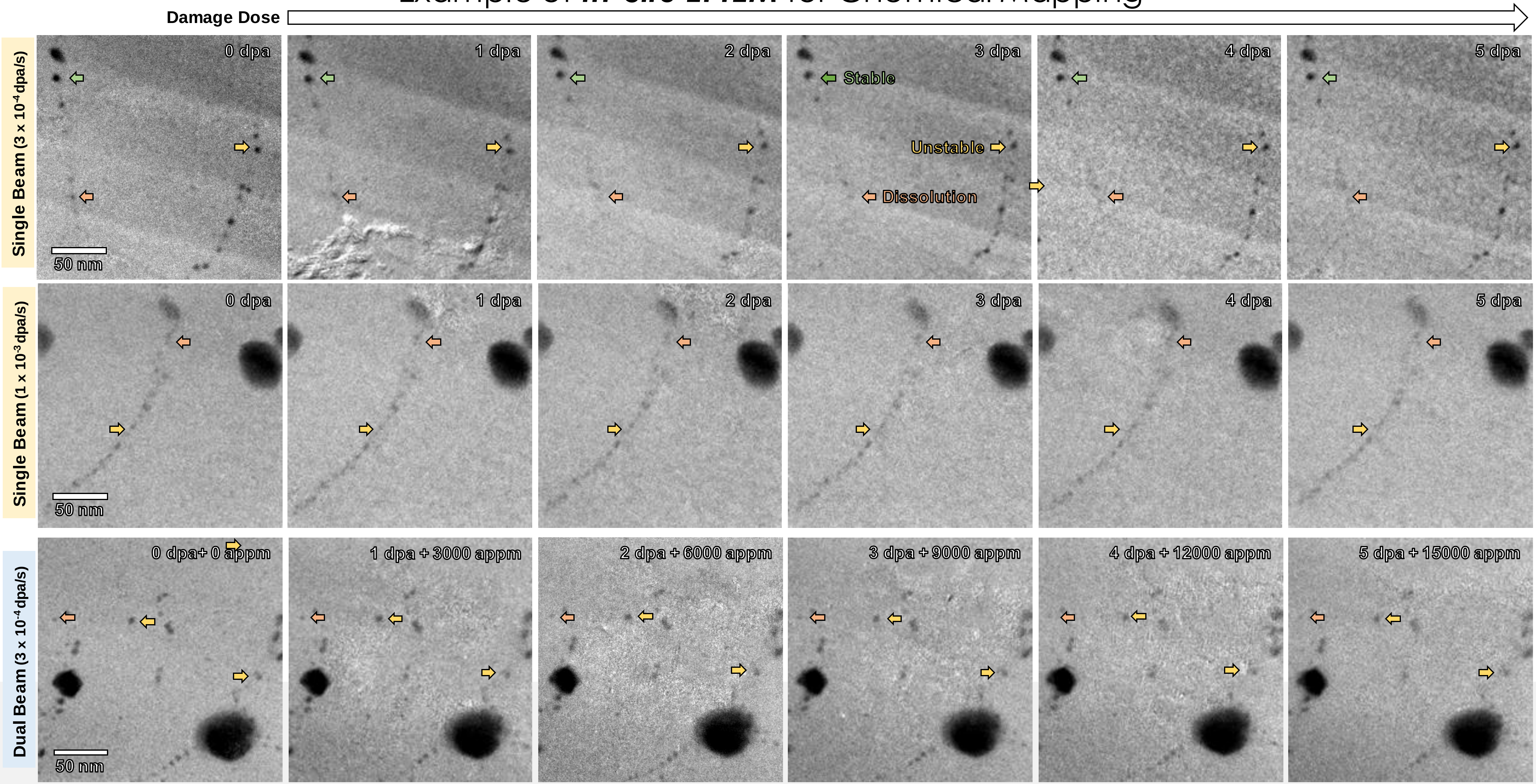


Gatan Continuum ER GIF:

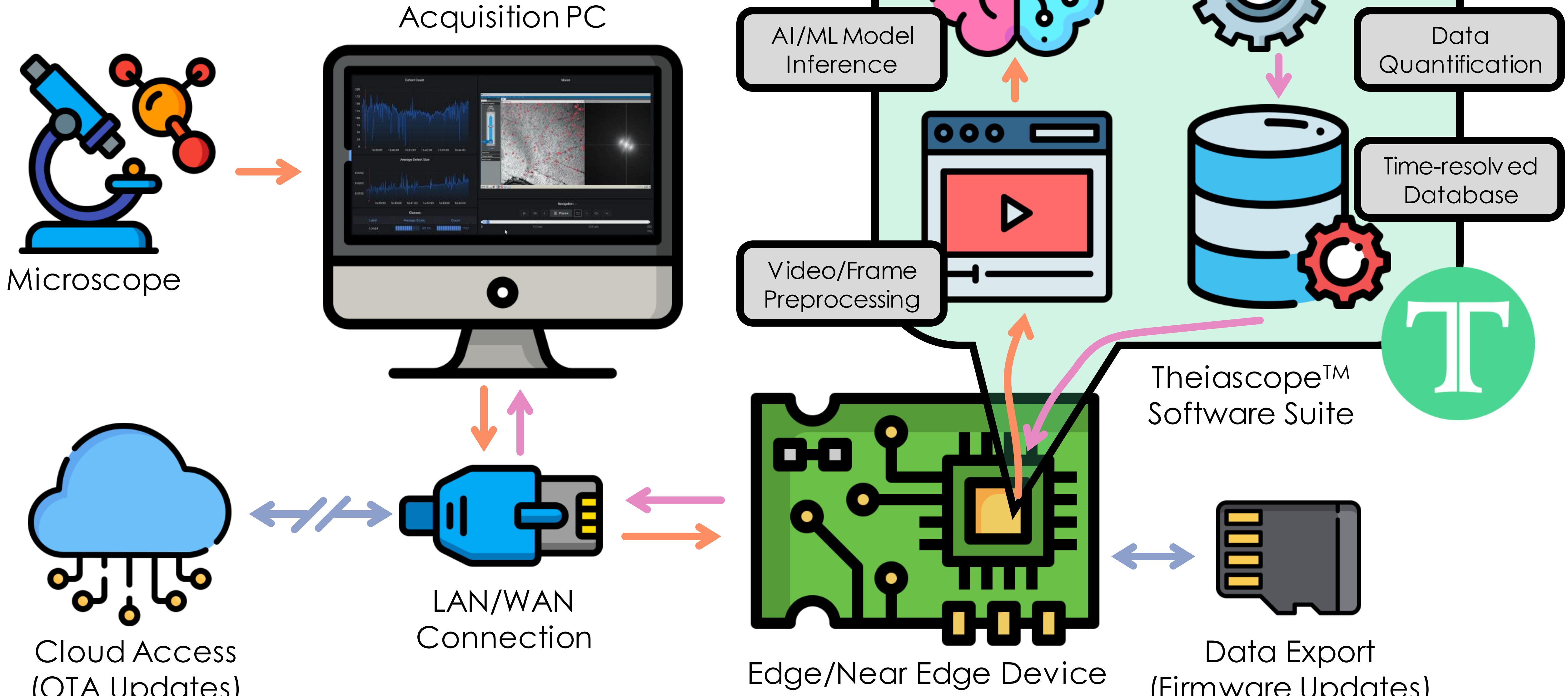
- EF- BF, HREM and Electron diffraction patterns
- EELS, EFTEM and EFTEM SI
- STEM SI combined with the attached ADF detector

Slide courtesy of K. Sun

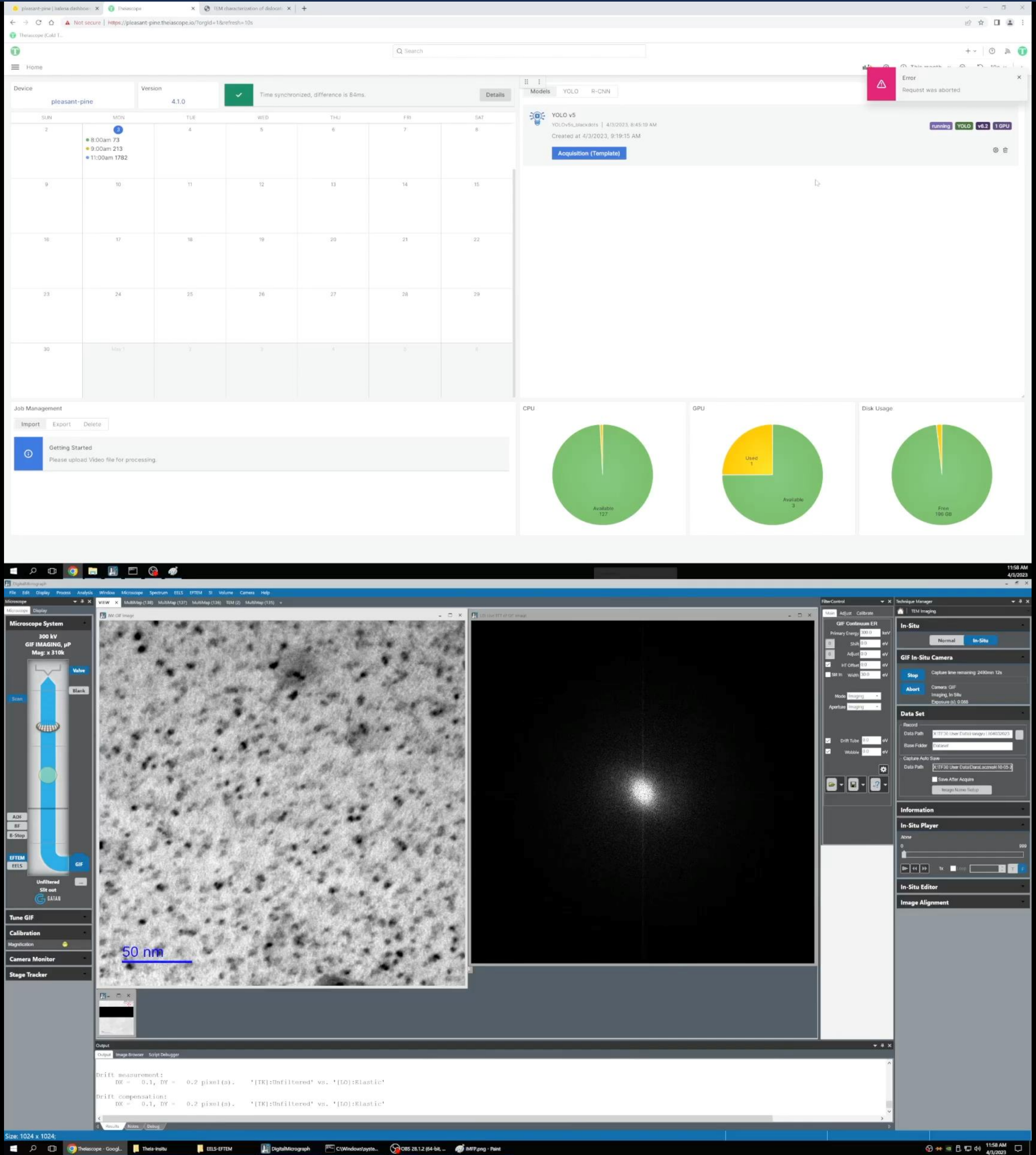
Microstructure Evolution of Nano-oxides in an ODS alloy – Example of ***In-Situ EFTEM*** for Chemical Mapping



Schematic of a real-time ML computing deployment architecture: Theiascope™



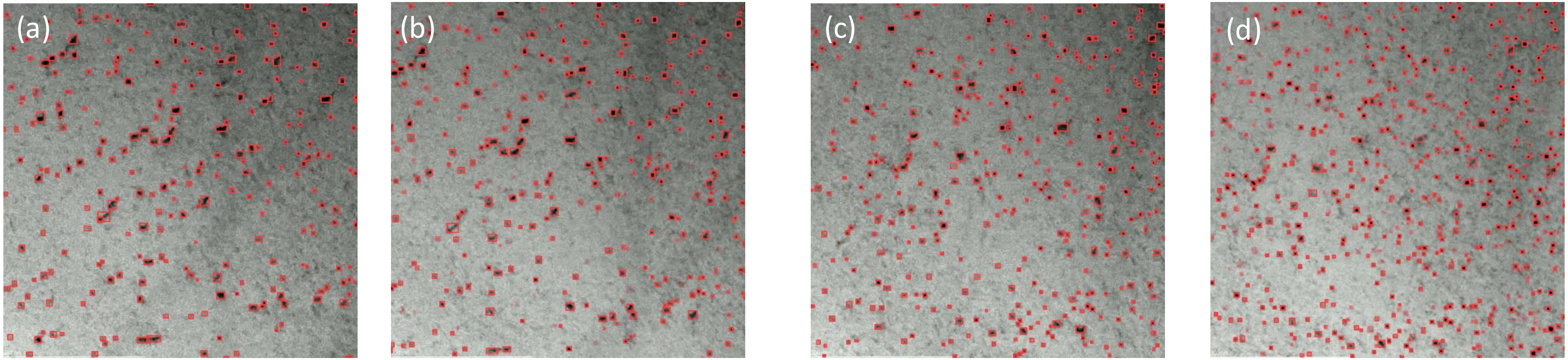
*C.R. Field & K.G. Field, U.S. Patent Application No. 17/718,805



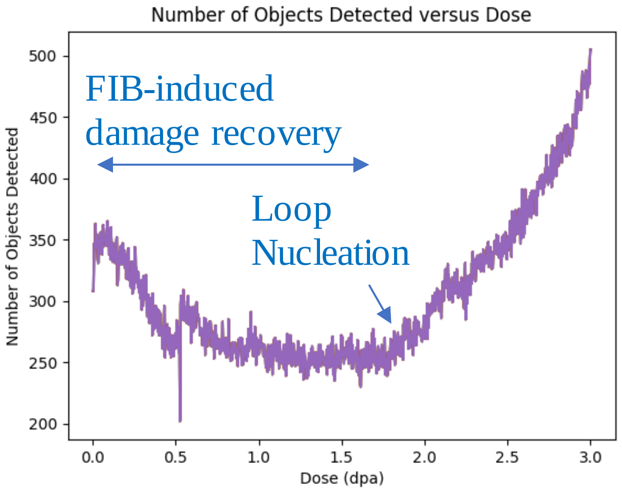
In-situ TEM irradiation of a FeCrAl alloy in MIBL-(MC)²

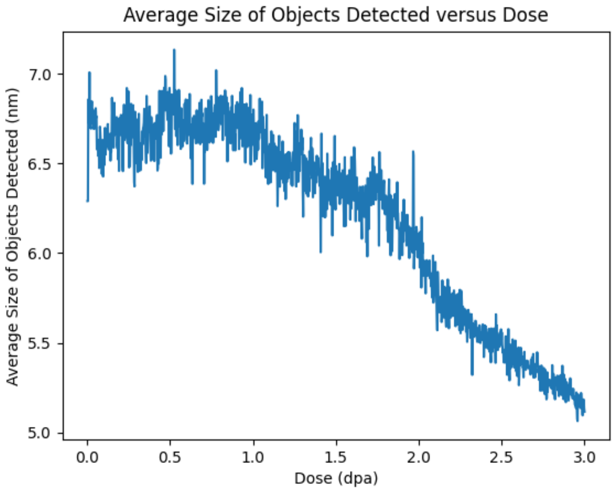
Results courtesy of K. Sun and H. Li (UM)

Early Dislocation Loop Formation in FeCrAl Alloys



2-Beam BF images showing the elimination of FIB damage then generation of radiation induced defect clusters in a model FeCrAl alloy irradiated by 1.2 MeV Kr ions at 320 °C with a damage level of (a) 0, (b) 1, (c) 2, and (d) 3 dpa, respectively.





Determination of critical dose to visible dislocation loop nucleation in the model FeCrAl using ML-based automated analysis

More details on Tues. at 10:25 am in Blue Spring I!

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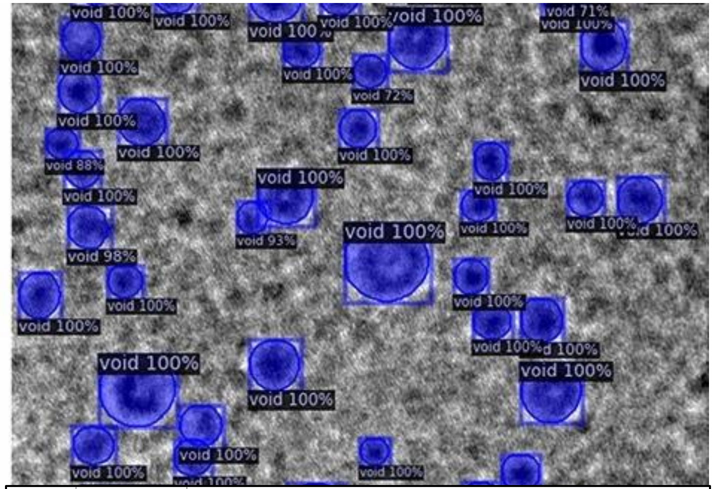
BUT! What model should I use for my experiment?!



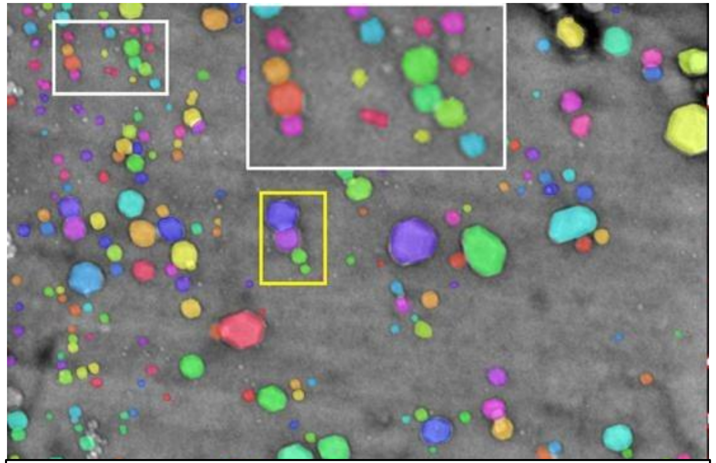
Article | Open access | Published: 30 March 2023

Materials swelling revealed through automated semantic segmentation of cavities in electron microscopy images

Ryan Jacobs, Priyam Patki, Matthew J. Lynch, Steven Chen, Dane Morgan & Kevin G. Field

Scientific Reports **13**, Article number: 5178 (2023) | Cite this article

1285 Accesses | **4** Citations | Metrics



Full Length Article

A deep learning model for automatic analysis of cavities in irradiated materials

Qinyun Chen [a], Chaohui Zheng [b], Yue Cui [b], Yan-Ru Lin [c], Steven J. Zinkle [a c]

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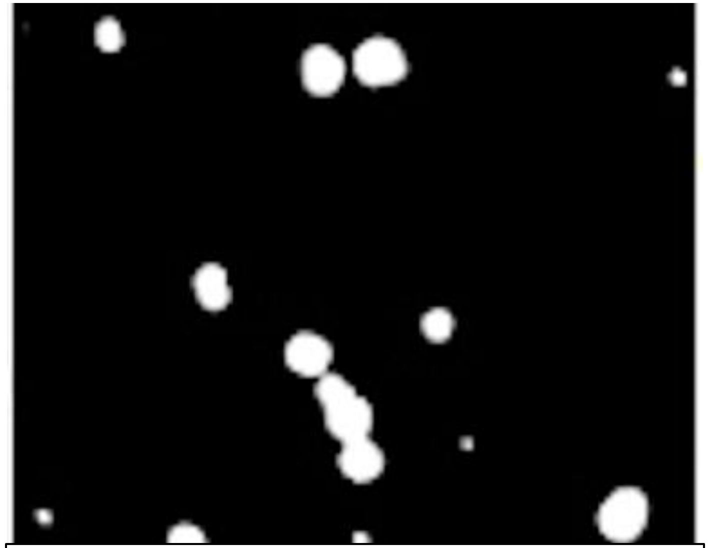

Article | Open access | Published: 04 September 2019

Deep Learning for Semantic Segmentation of Defects in Advanced STEM Images of Steels

Graham Roberts, Simon Y. Haile, Rajat Sainju, Danny J. Edwards, Brian Hutchinson & Yuanyuan Zhu

Scientific Reports **9**, Article number: 12744 (2019) | Cite this article

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A wide range of different ML techniques being developed for the same features

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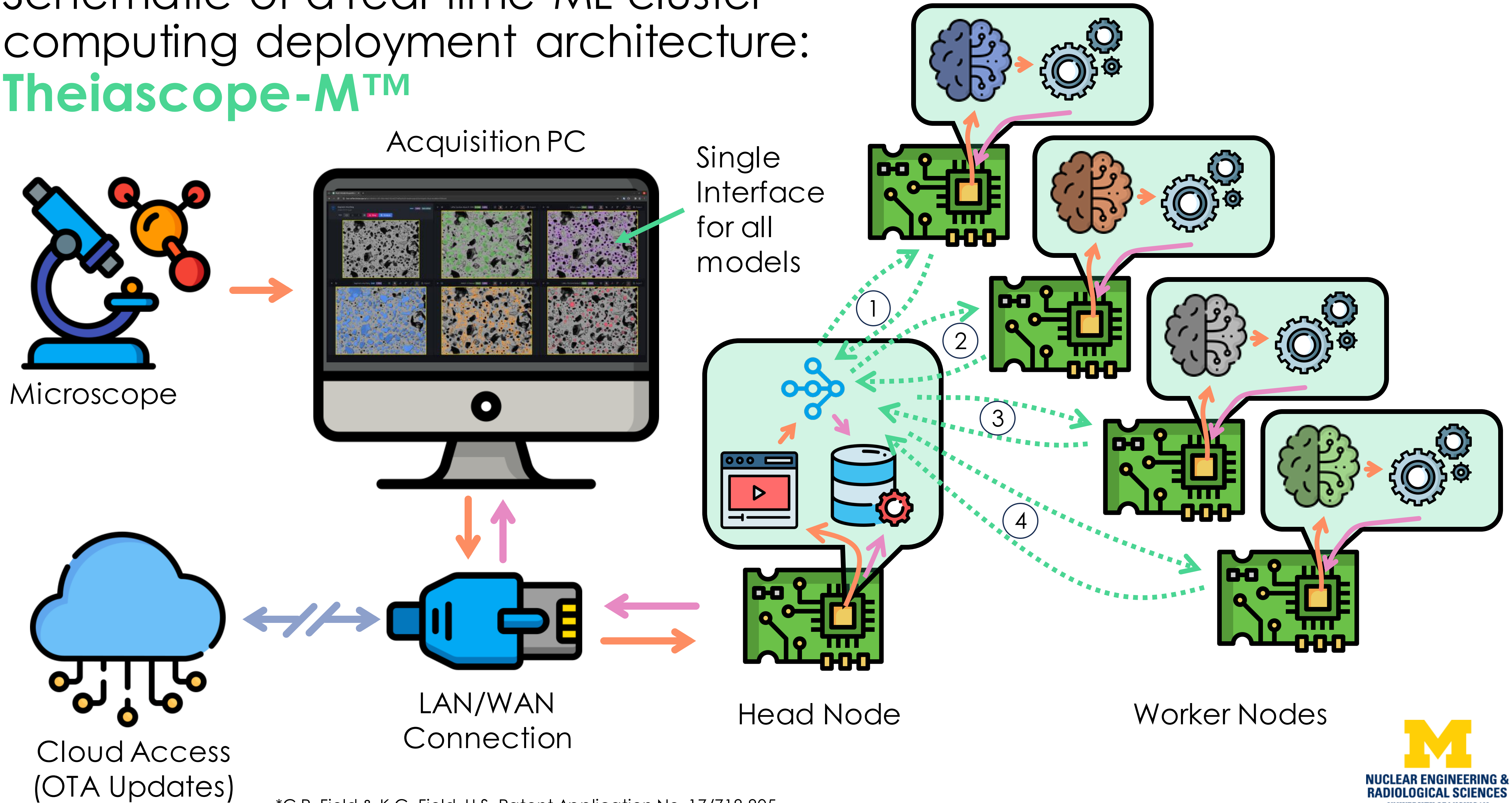
Just run ***all*** the models!

Schematic of a real-time ML cluster computing deployment architecture: Theiascope-M™



*C.R. Field & K.G. Field, U.S. Patent Application No. 17/718,805

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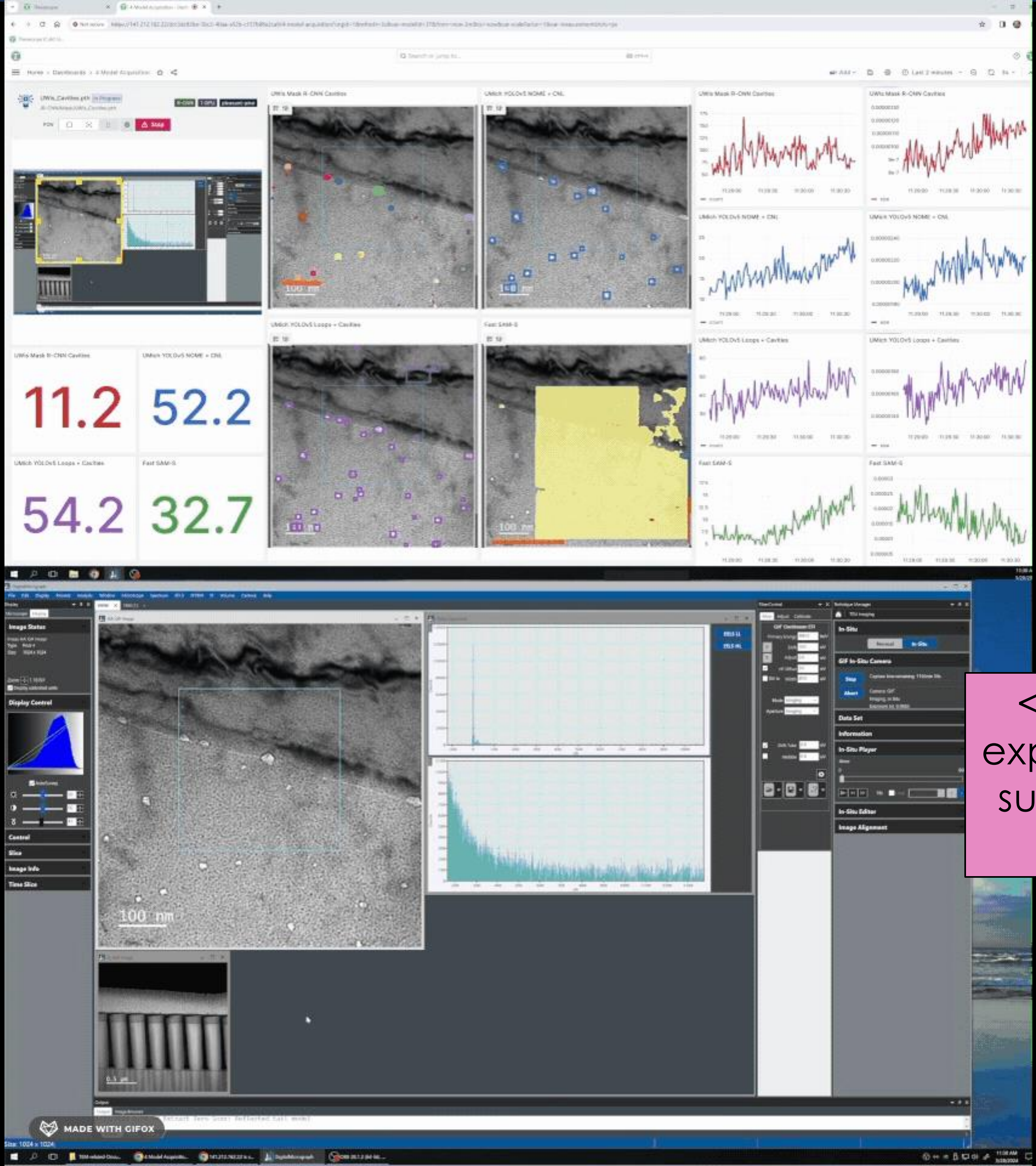


<1 hr from experiment to summary for paper!

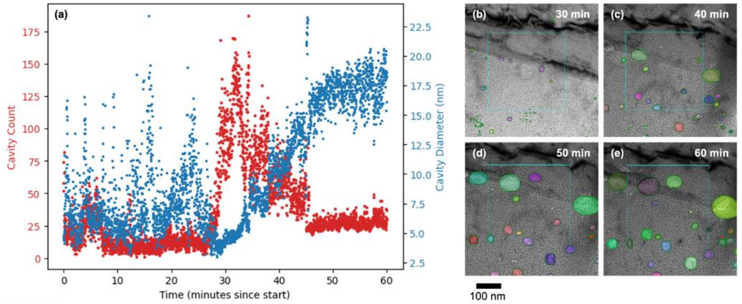


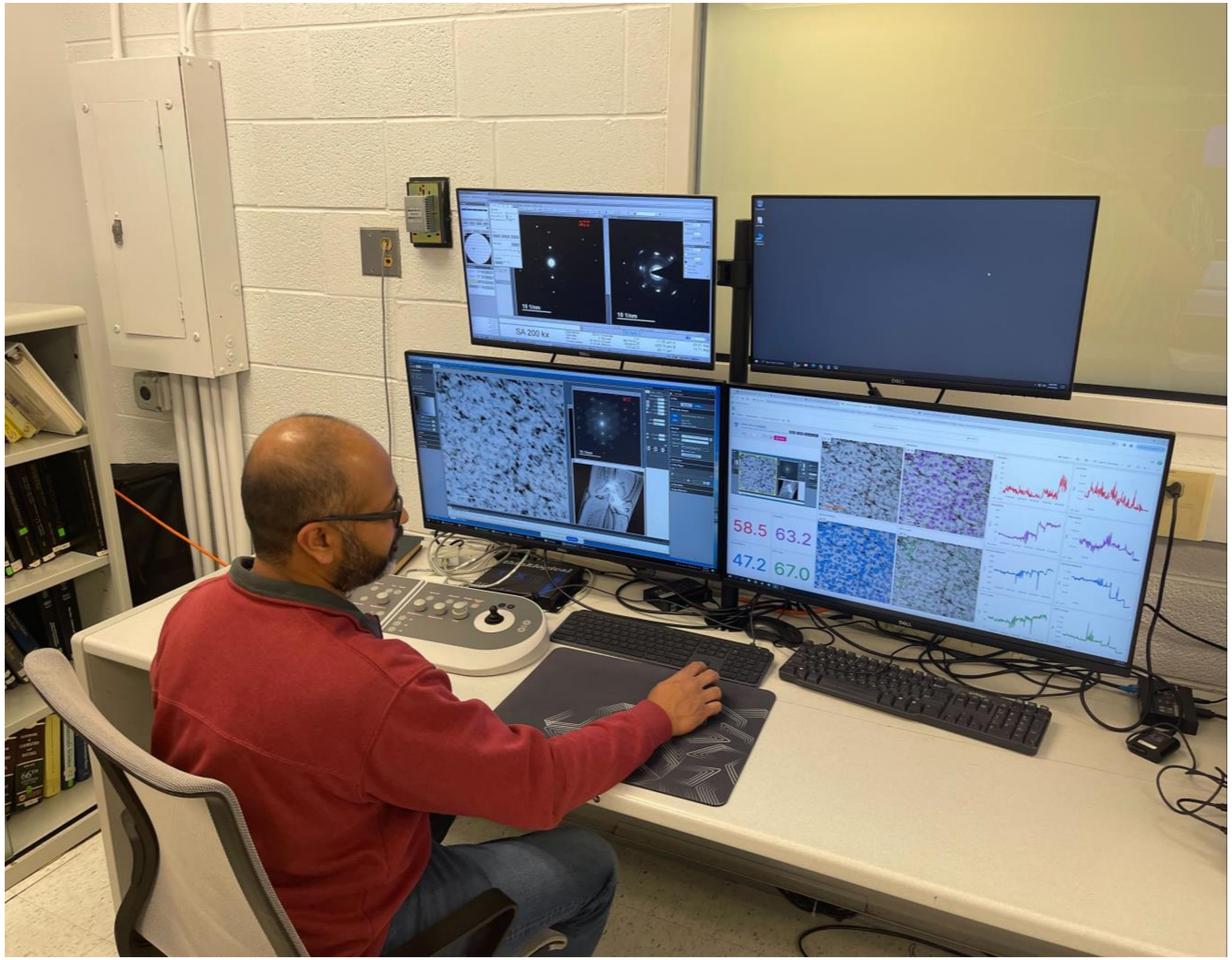
Fig X: Microstructural evolution of a 100 appm He pre-implanted 316L sample during a 1 hour in-situ TEM anneal at 500°C showing the evolution of (a) average cavity diameter – in blue, (b) cavity count – in red, and corresponding (b-e) micrographs during the anneal where the colored polygons show the real-time cavity detections using a machine learning model [ref].

Over the past six months, the University of Michigan has performed ex-situ Helium implantations to 100, 1000, and 2000 appm Helium at 100°C into target materials including 316L stainless steels (for all conditions) and Inconel X-750 (for the 2000 appm Helium condition only). All irradiations were performed using 3.42 MeV He^{2+} through a rotating 6.20 µm Al foil to promote a uniform appm Helium concentration profile across the entire implantation depth which exceeds 1 µm. All irradiations maintained a 2σ temperature deviation less than 5°C. Preliminary transmission electron microscopy (TEM) investigation of the overall microstructures in the 100 appm Helium condition showed no observable cavities in the implanted regime for the as-implanted condition of 316L. With increasing implantation to 1000 appm Helium, small (<5 nm) cavities were detected in the implanted region using bright field TEM imaging in the under focused condition for the 316L material. Characterization of the 2000 appm Helium condition are on-going and will be reported later.

In-situ TEM annealing of the 100 appm Helium condition of the 316L sample has been performed with preliminary results shown in Fig. X. The TEM lamella was isothermally annealed at 500°C using a Gatan 652 holder for 1 hour on a Thermo Fisher Tecnai G^2 F30 TWIN TEM and continuously imaged in the underfocused BF-TEM condition at 1 frame per second (fps). During annealing+imaging, a Theiascope-X™ (Version 6.3.0) running the Jacobs et al. [1] machine learning model for cavity detection and quantification was run in real-time, with the quantitative results shown in Fig. Xa and segmentation overlays shown in Fig Xb-e. The results show that cavities nucleation or growth above the detection limit of the TEM did not occur for the first 27 minutes of the in-situ TEM annealing experiment. The initial scatter in Fig Xa is the result of the ML model having low-confidence predictions – additional, post processing of the data will remove these erroneous results. After 27 minutes, cavity formation was observed intra- and inter- to the grain boundaries (Fig. Xa,b) with significant increase in the number of cavities and cavity size occurring through 50 minutes into the in-situ TEM anneal (Fig. Xa,c-d). Past 50 minutes, no significant new cavity formation was observed or quantified via the Theiascope-X™ and the material transitioned to a steady state coarsening regime (Fig. Xa,e). Results also showed that intragranular cavities coarsened more significantly compared to intergranular cavities suggesting a strong impact of grain boundary characteristics on the cavity nucleation and growth kinetics in the 316L implanted sample. Further analysis is on-going with additional in-situ TEM annealing studies planned for the 1000 and 2000 appm Helium implanted samples.

Take aways

- MIBL & DOE-NE/NSUF have placed significant investments to form MiTEM, a modern, state-of-the-art in-situ TEM ion irradiation facility
- Key differentiators for MiTEM:
  - Low cost- and hassle-free access for users
  - Remote operation simplifies user experiences
  - S/TEM capable microscope means both TEM and STEM-based experiments can be performed
  - Multiple stages enable a wide range of temperatures and heating/cooling rates
  - Gatan Imaging Filtering enables in-situ chemical mapping (GSI-21-25126)
  - Theiascope-X™ enables the most advanced real-time quantification system for nuclear materials



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Thanks!

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