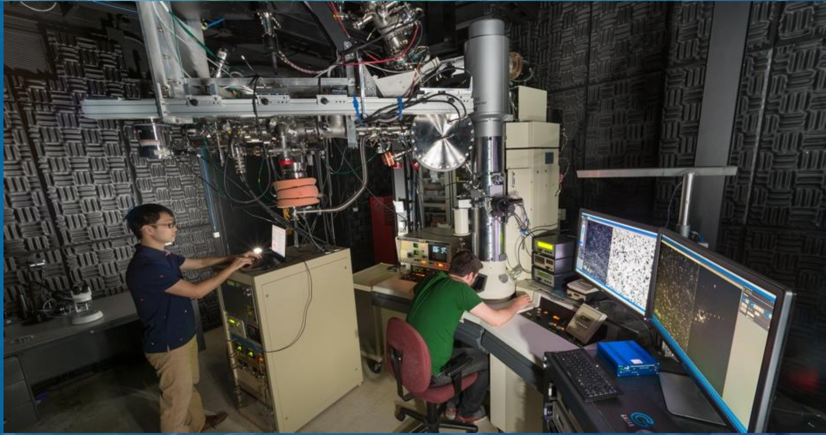
8/26/2025 NSUF TEM DATA MANAGEMENT WORKSHOP

# IN-SITU TEM DATA CHALLENGES AND OPPORTUNITIES



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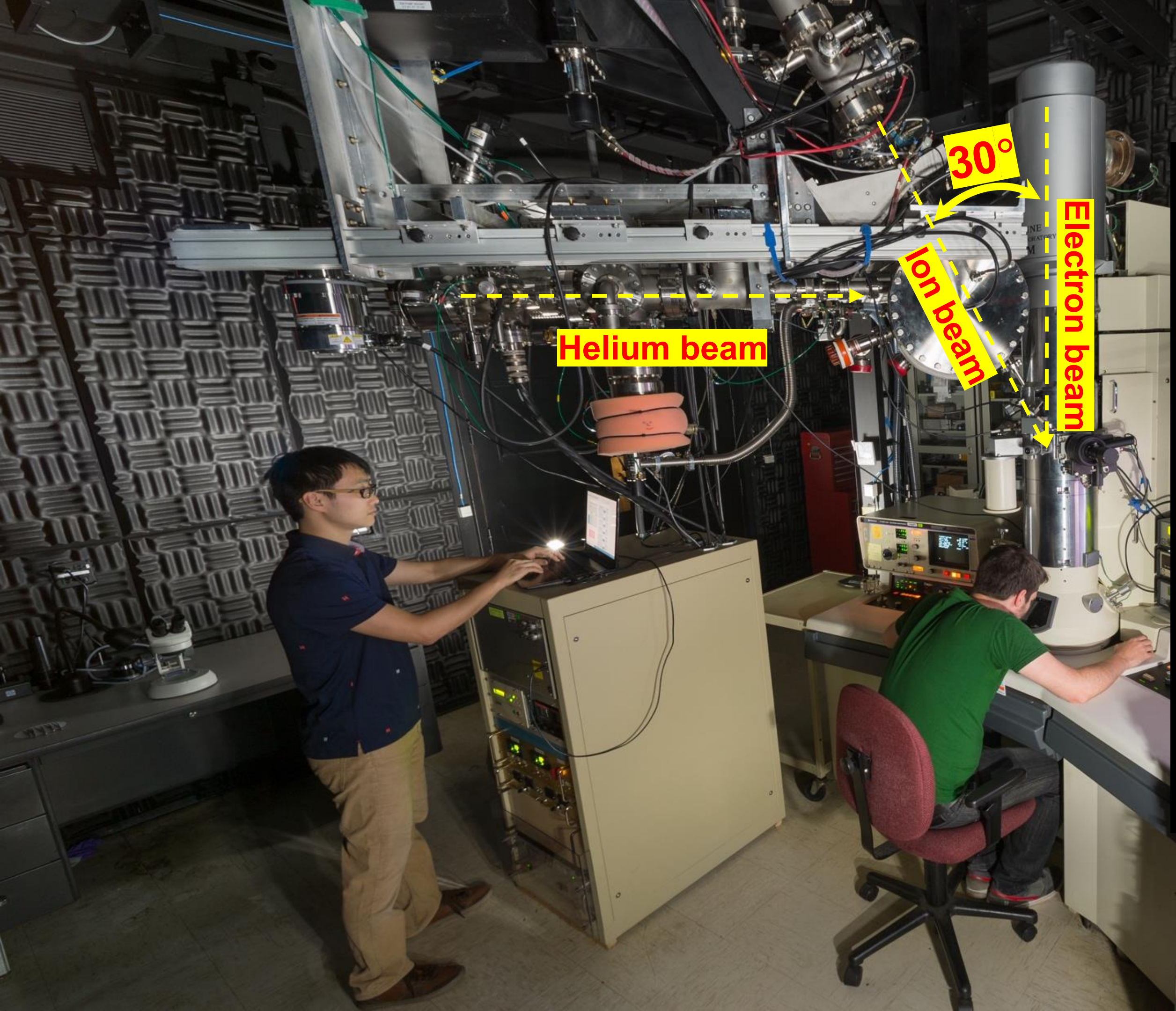
U.S. DEPARTMENT OF ENERGY

Argonne National Laboratory is a U.S. Department of Energy laboratory managed by UChicago Argonne, LLC.

Argonne NATIONAL LABORATORY | Intermediate Voltage Electron Microscope

# H-9000NAR TEM




- Ion and electron beam angle, 30° allowing for real-time imaging
- Dual-beam irradiation capability
- $LaB_6$ filament, high current, high brightness, excellent for real-time imaging
- Electron energies, 100-300 keV,
- Minimum Spot Size: 25 nm
- Point resolution: 0.25 nm at 300 keV
- Gatan OneView camera enabling Video recording up to 313 fps
- Vacuum: $5x10^{-8}$ to $4x10^{-7}$ torr
- Bruker Xflash 6160 EDS detector

# *IN SITU* ION IRRADIATION/IMPLANTATION

## High energy Ion Irradiation

| | |
|---|---|
| **Ion Energy/Types** | • Implanter: 50 keV – 1 MeV, H, inert gases, elements Al to Au<br>• Tandem: 2.5 MeV Ni |
| **Ion Flux** | $10^8 – 10^{12}$ ions/cm$^2$/s ($10^{-7}$– $10^{-3}$ dpa/s) |
| **Beam size** | Uniform beam of 1.5 mm ∅ on sample center |
| **Dosimetry** | Real-time dosimetry (Faraday cup in TEM column) |

## Helium ion source

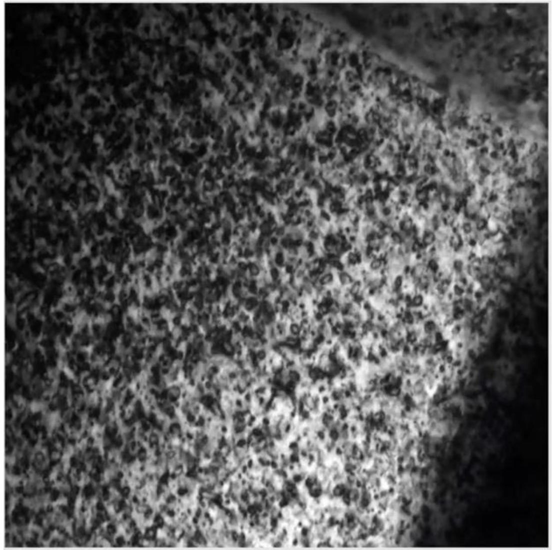
| | |
|---|---|
| **Ion Energy/Types** | 10-20 keV He |
| **Ion Flux** | $10^{11} – 10^{13}$ ions/cm$^2$/s |
| **Beam size** | Uniform beam of 1 mm ∅ on sample center |
| **Dosimetry** | Faraday cup in TEM column measured before and after irradiation |

The two-floor design ensures a low-angle between ion and electron beams for real-time imaging, and mitigation of mechanical vibration from the ion beamline.

# TEM holders for *in situ* TEM

| | |
|---|---|
| ***In situ* heating (20 to 1100°C)** | - High temperature (20-800°C, ±3°C), double tilt (±40°, ±20°)<br>- High temperature (20-1100°C), single tilt (±40°) |
| ***In situ* cooling (20 to 300 K)** | - Low temperature (20-300°K), double tilt (±40°, ±20°) |
| ***In situ* straining (100 K to 400°C)** | - Straining, high temperature (20-400°C), single tilt (±40°)<br>- Straining, low temperature (100-400°K), single tilt (±40°)<br>- PI95, Room temperature, Single tilt (±40°) |
| ***In situ* corrosion** | - Environmental Cell TEM Stage:<br>▪ Environmental chamber (20-300°C)<br>▪ TEM stage (20 - 700°C) |
| **Tilt-rotation (tri-axis)** | - 3D tomography |

UN under *in-situ* ion irradiation at 1000°C



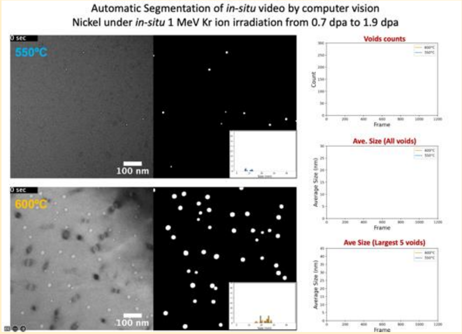
Kosmidou et al. Acta. Mater. 288 (2025)
RTE 23-1868

# IN-SITU TEM AND AI/ML FOR NUCLEAR MATERIAL RESEARCH

## A Mutually-reinforced scheme to accelerate discovery

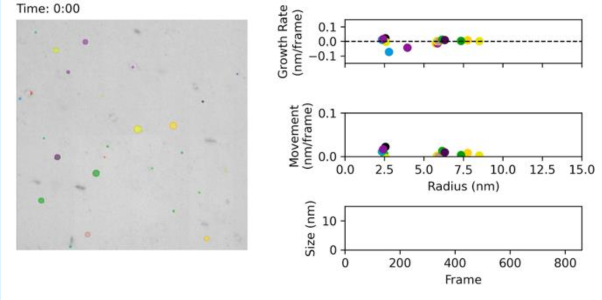
### Rapid Data Acquisition

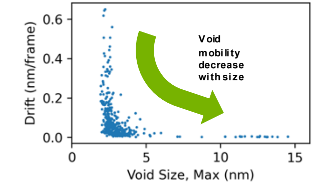
300+ TEM images per second

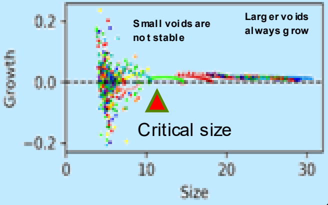



### Unique Data

that is not available with regular PIE

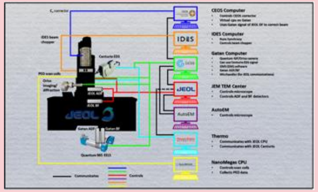


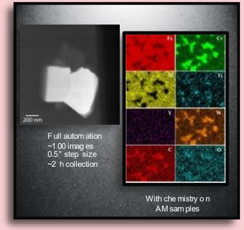




### Automatic 3D Reconstruction

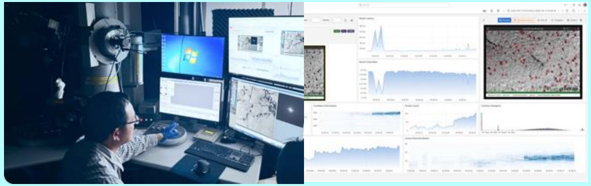
(PNNL, Olszta et al.)






### Real-Time analysis

Decision making on-the-fly



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# IN-SITU TEM CAN HELP NRDS SHAPE FUTURE NUCLEAR MATERIAL RESEARCH

## A Database, A Platform and A GPT

- Manage of TEM image and image annotations
- Train and maintain CV models for defect analysis of TEM images
- Maintain a web-based platform to analyze the NRDS images with those CV models
- Produce synthetic TEM images - *Show me the TEM image of 316 SS irradiated at 3.7 dpa at 513°C*
- Train and maintain regression models on CV analyzed data. Predict material property – *What is the dislocation loop density in 316 SS irradiated at 3.7 dpa at 513°C?*

# TEM DATA SPECIFICATIONS IN IVEM

**Image specs** (Gatan OneView Camera)

- Resolution: 4K, 2K, 1K, 512
- File size: (32-bit, 64 MB for 4K image)
- File format: DM3, DM4 (Gatan proprietary format of TIFF that include microscope metadata)

**TEM image Data types**

- Bright field image
- Dark field image
- Diffraction pattern
- EDS spectrum

**Videos**

- Frame rate: 313 fps (maximum)
- File size: 60 GB for typical 1 hour video (~4 dpa for $10^{-3}$ dpa/s) with 1K resolution and 1 fps.
- Recording:
  1. DigitalMicrograph - each frame is saved as one dm3 file
  2. ANL system with data bar (screen recording – 15 fps)

**Challenges**

Taking long video in high resolution is difficult due to hardware limitation

# IMPORTANT METADATA FOR TEM IMAGES IN IVEM

## Associated information for each TEM image (that could be useful for the ML endeavor at NRDS)

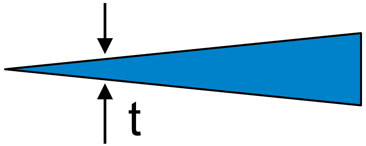
- **Sample**
  - Material (multi-phase)
  - Foil thickness
  - Sample preparation method (e.g., FIB, electropolishing)
  - Geometry (e.g., disc, lamella, strip)
- **Irradiation/implantation condition**
  - Ion type
  - Ion Energy
  - Ion flux
  - Ion fluence
  - Temperature
  - Electron
  - History
  - Accelerator
  - Ion source
  - Ion charge state
  - Rastered/defocused beam
- **Image condition**
  - Magnification – pixel size
  - Defocus
  - Crystal orientation of the image
  - g-vector and diffraction contrast condition
  - Image type (eg. BF or DF)
  - Sample tilt
  - STEM/TEM
- **Other conditions**
  - Load-displacement
  - Corrosion (e.g., liquid cell, gas)

### Challenges

- How to save those information along with the images in NRDS database?
- How to label ROI to link images with different magnification/imaging condition?
- Not practical to log all those info in the filename

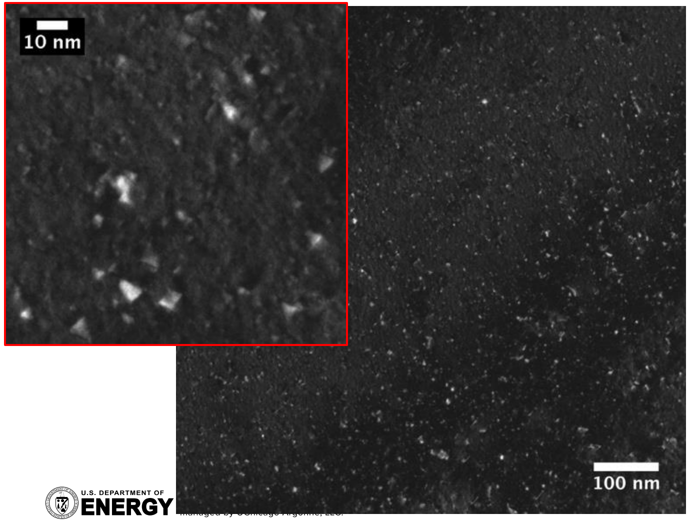
# FOIL SURFACE EFFECTS AND LOOP DENSITY MEASUREMENT

High mobility of point defects and defect clusters at high temperature results in denuded zone  close to the foil surface of TEM specimen.

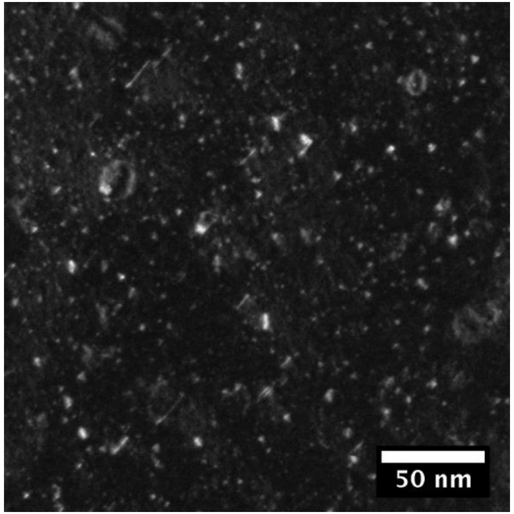



**$Al_{0.3}$CoCrFeNi 500C 1 dpa**

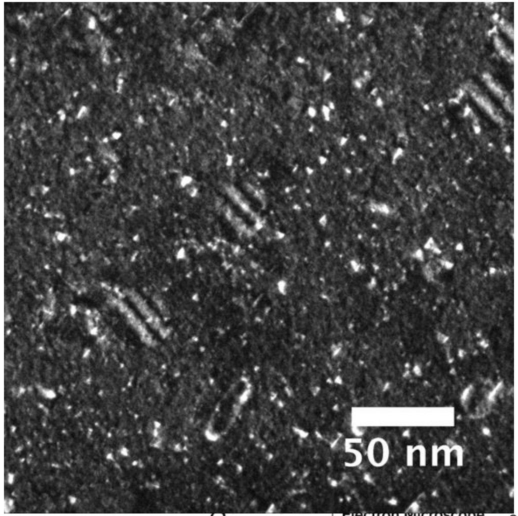
**t=84 nm SFT**




**t=103 nm SFT+loop**

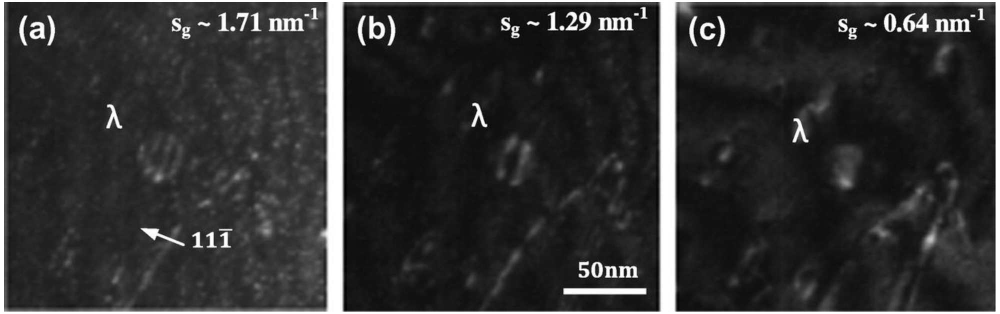


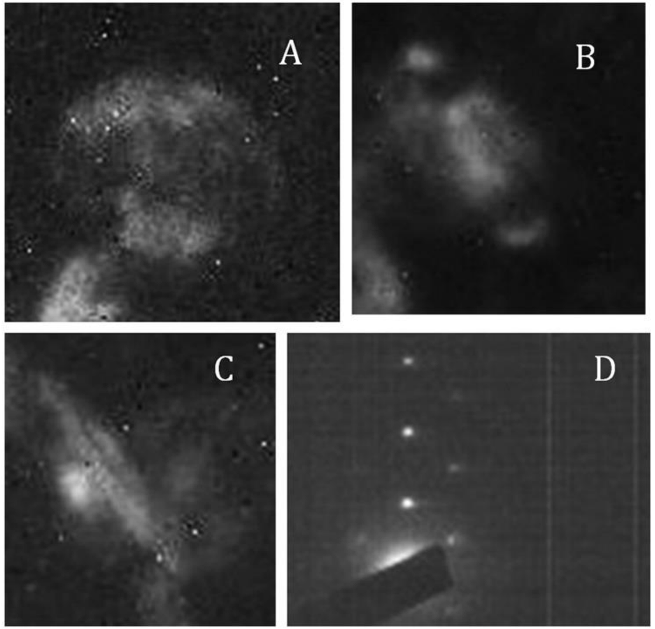

**t=154 nm SFT+loop**



# EFFECT OF SAMPLE ORIENTATION ON THE DEFECT IMAGES

- The shape and the contrast of the image of irradiation-induced defects is sensitive to the imaging condition (crystal orientation etc.)
- The computer vision models need to be able to recognize all those variants.
- (Calcuated diffractrion pattern for crystal orientation)



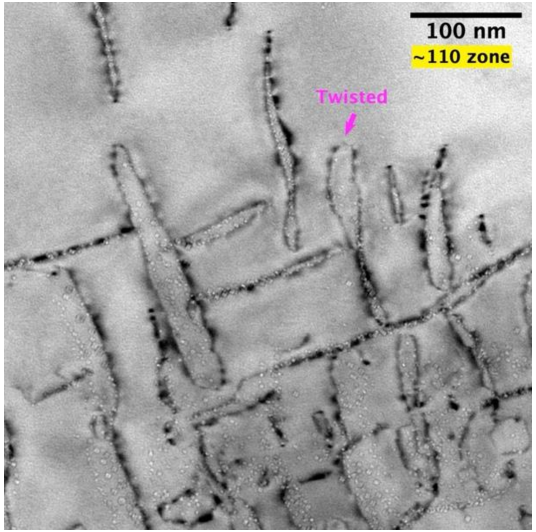



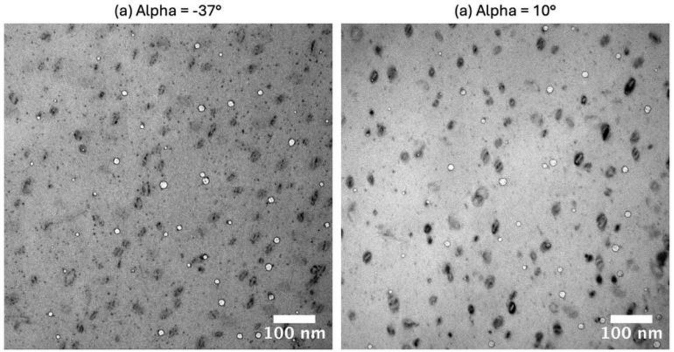

*Chen, Wei-Ying, et al. "Characterization of dislocation loops in CeO2 irradiated with high energy Krypton and Xenon." Philosophical Magazine 93.36 (2013): 4569-4581.*

# Tilting information of TEM images can be used to perform 3D tomography

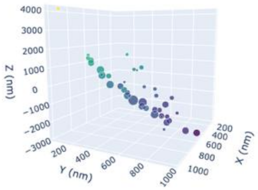
- Spatial relationship between defects/objects in 3D
- Enable foil surface effect studies

Ni after *in-situ* He ion implantation at 500°C




$$Z = \frac{\Delta d}{2 \times \sin\left(\frac{\theta}{2}\right)}$$




*Chen, Ward, Mei (ANL, AMMT)*

# IN-SITU VIDEO DATA IN IVEM

**Challenges** specific for in-situ video data:

- Extremely large number of images (1k-10k) each video where each image corresponds to different dose and temperature, stress condition, and likely dose rate.
- Operator changing imaging conditions (e.g. magnification, ROI, foil thickness) during video recording.
- Sample tilting during irradiation changing the diffraction contrast and focus.
- Current camera not suitable for long diffraction pattern recording

**Best practice** to obtain good video data:

- Lower ion flux if possible
- Give holder time to stabilize from thermal drift
- Work on areas with relatively thicker foil thickness

# DATA STORAGE AND TRANSFER IN IVEM

**Storage**

- Gatan camera computer – user data kept locally for around 1 year
- Box – mostly for images
- Storage cluster in ANL – mostly for video files

**Transfer**

- (Fresh) Hard drive – during user visit
- Box – most convenient for data transfer of small batch size.
- Storage cluster in ANL – use Globus for transferring data

**Challenge**

- Transfer large video files (several hundreds GB to TB each project) to users outside ANL.

# RECOMMENDATIONS ON FILENAME CONVENTION AND WORKFLOW

- In DigitalMicrograph, use the Global info – session info – save numbered
- Use serial number
- Manual saving filename (the filename by auto-save won't contain specific and irradiation condition).

*My current filename convention:*

316H-1MeVKr-600C-1dpa_300kV _20kX _0402-df-over-5um-200g-011z-g5g-a1.dm3

Material | Irradiation condition | Microscope info | Serial | Imaging condition | ROI

Automatically generated with Global info – save numbered | Manual input

**Challenge**

- Need better way to save meta data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