SAND2018-4466C

Symposium: Advancements in In-situ Electron Microscopy Characterization II

Irradiation of Nanoporous Gold and Niobium – Effects on Mechanical Properties

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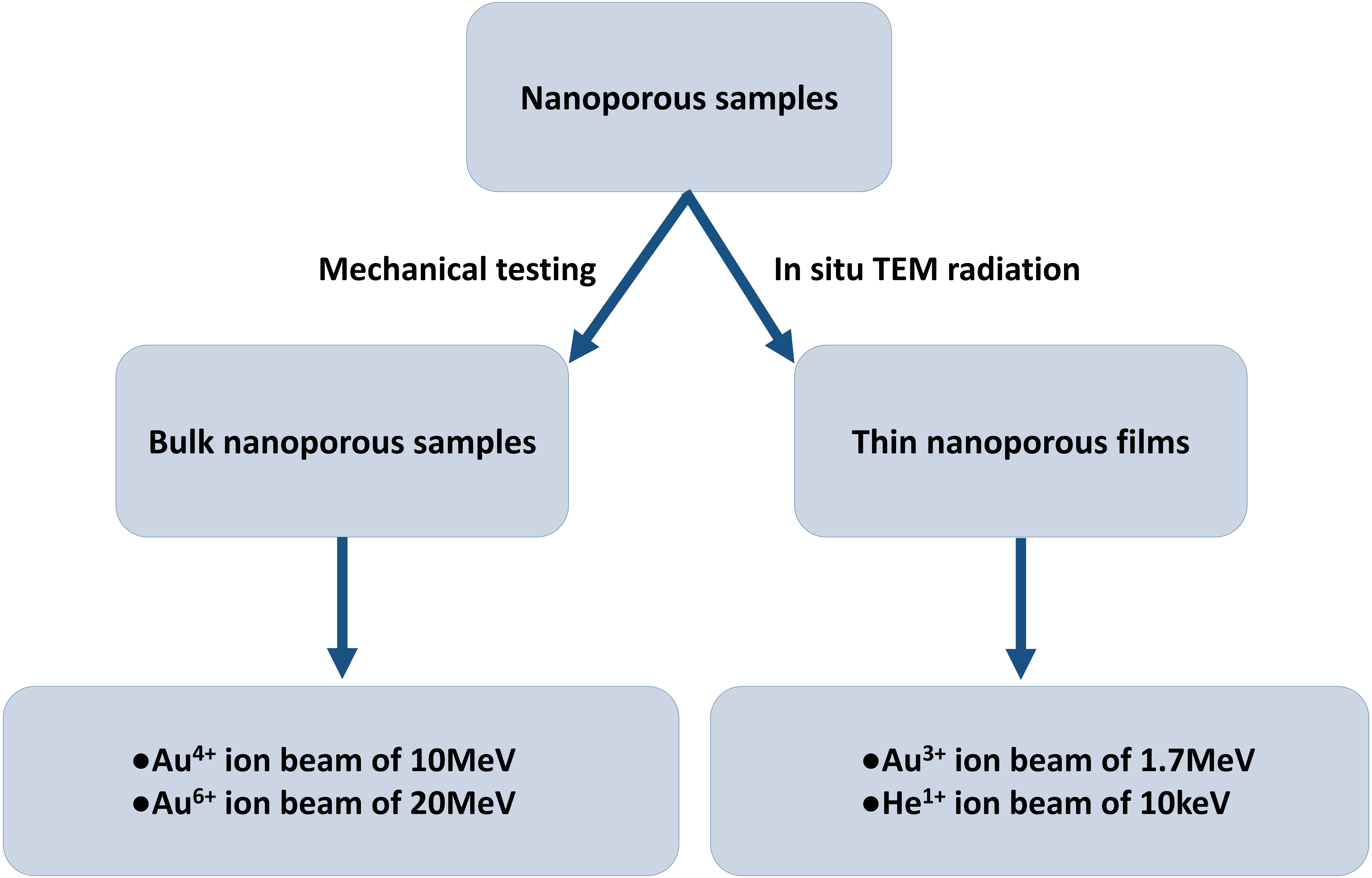

Nanoporous samples
Mechanical testing
In situ TEM radiation
Bulk nanoporous samples
Thin nanoporous films
●Au^{4+} ion beam of 10MeV
●Au^{6+} ion beam of 20MeV
●Au^{3+} ion beam of 1.7MeV
●He^{1+} ion beam of 10keV

Preparation of bulk nanoporous Au samples

Arc melting (66.6 at% Ag & 33.4 at% Au) → Cold working (rolling) to reduce thickness (~17.5%) → Heat treatment at 900°C for 90h → Grinding (600 & 800 CAMI grit designation) → Polishing (1μm & 0.05μm alumina) →

Dealloying process:

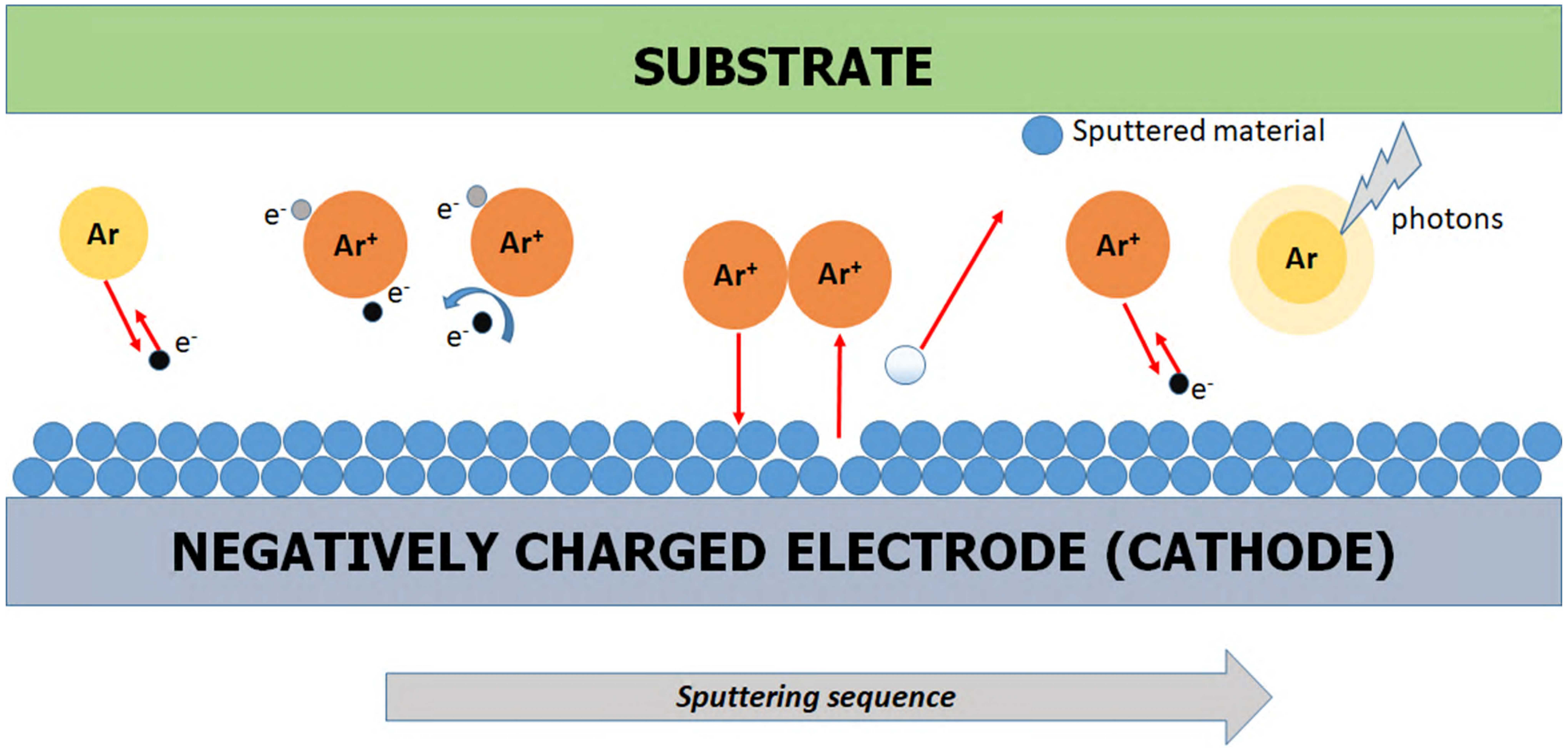
1. free dealloying in 1HNO3:2H2O for 2h
2. electrochemical dealloying for 72h
3. free dealloying in concentrated HNO3 for 48h

Cross section image on SEM defining porosity depth of bulk np Au sample

Preparation of thin nanoporous Au samples

1. Magnetron Sputtering (Polycrystalline np Au)

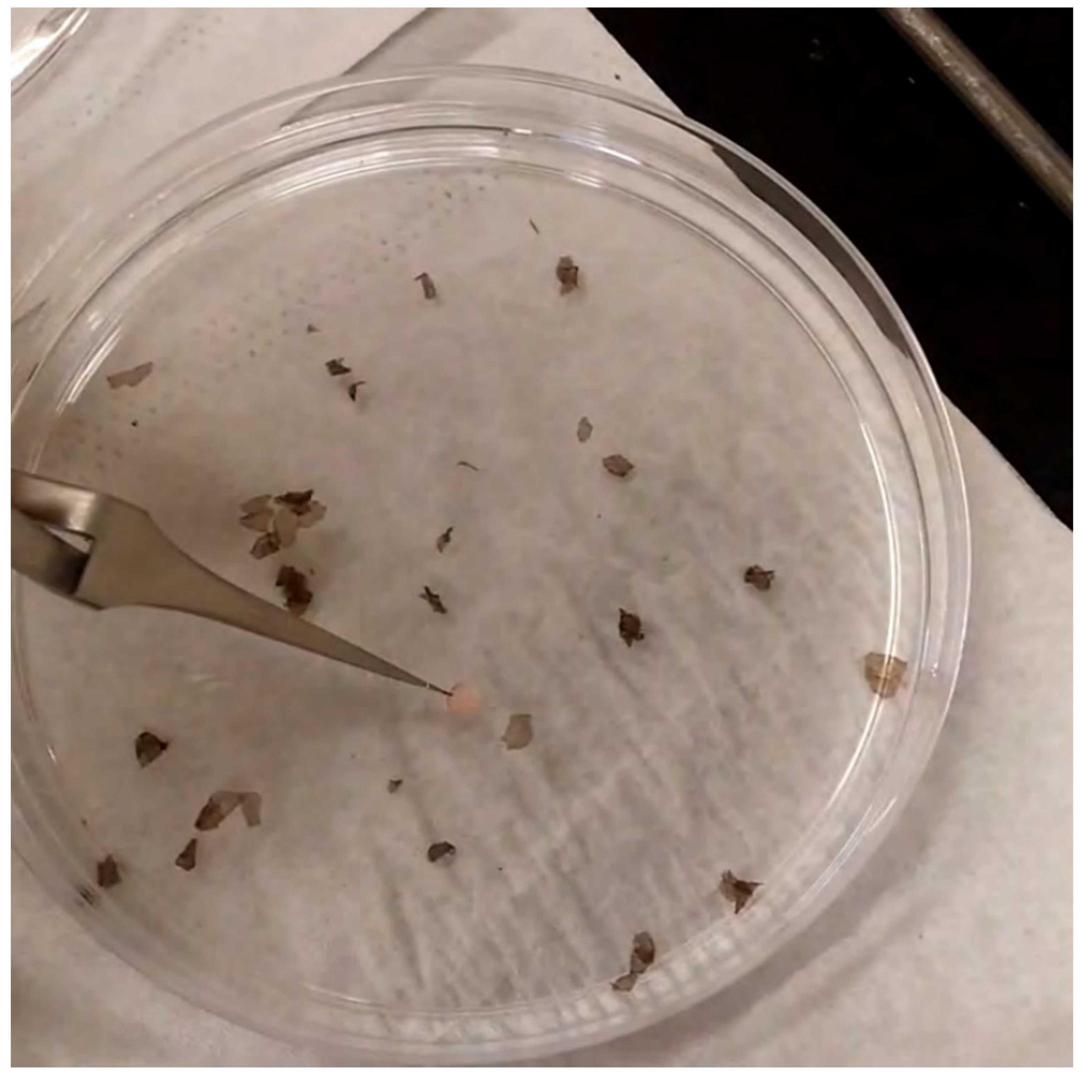
- Interlayer of Mg (160nm thick applied with 50W DC power supply for 8’ at 2.5mtorr)
- Thin layer of Au/Ag (less than 40nm) with 50W DC for Au target and 97W DC for Ag target for 1’ at 2.5mtorr
- Free dealloying in $3HNO_3:2H_2O$ for 17h
- Floated thin film scooped and cleaned with deionized H_2O for 24h ready to be attached on Cu grid



2. Gold/Silver leaves (Monocrystalline np Au)

- 35 at% Au and 65 at% Ag leaves were rolled to reduce thickness
- Free dealloying in concentrated HNO_3 for 2h
- Cleaned in deionized H_2O for 24h

Dealloying Au/Ag leaves in concentrated HNO_3

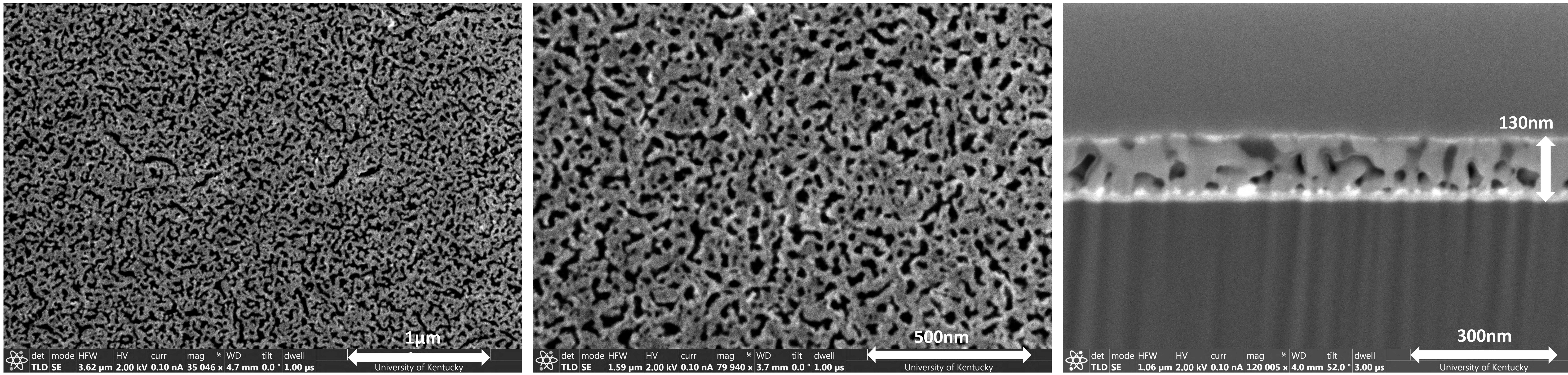


Thin films of np Au in deionized H_2O prepared on Cu grids

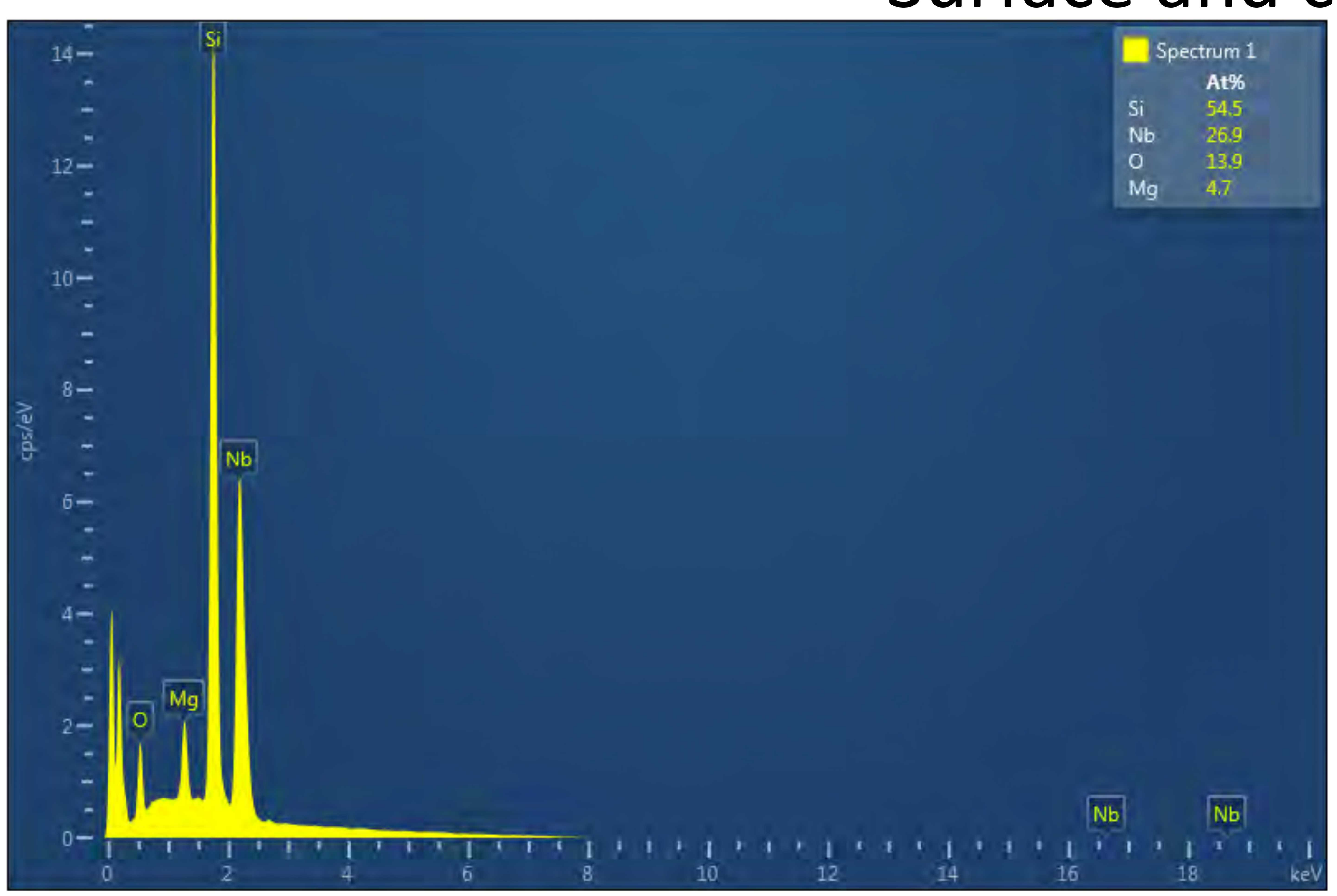
Preparation of thin nanoporous Nb samples

Magnetron Sputtering

- Interlayer of Ta (50W DC power supply for 3’ at 2.5mtorr)
- Thin layer of Nb/Mg gradient with 70W DC for Nb target and 80W DC for Mg target for 15’ at 2.5mtorr
- Thermal dealloying at 600^{o}C for 2h



Surface and cross section of nanoporous Nb

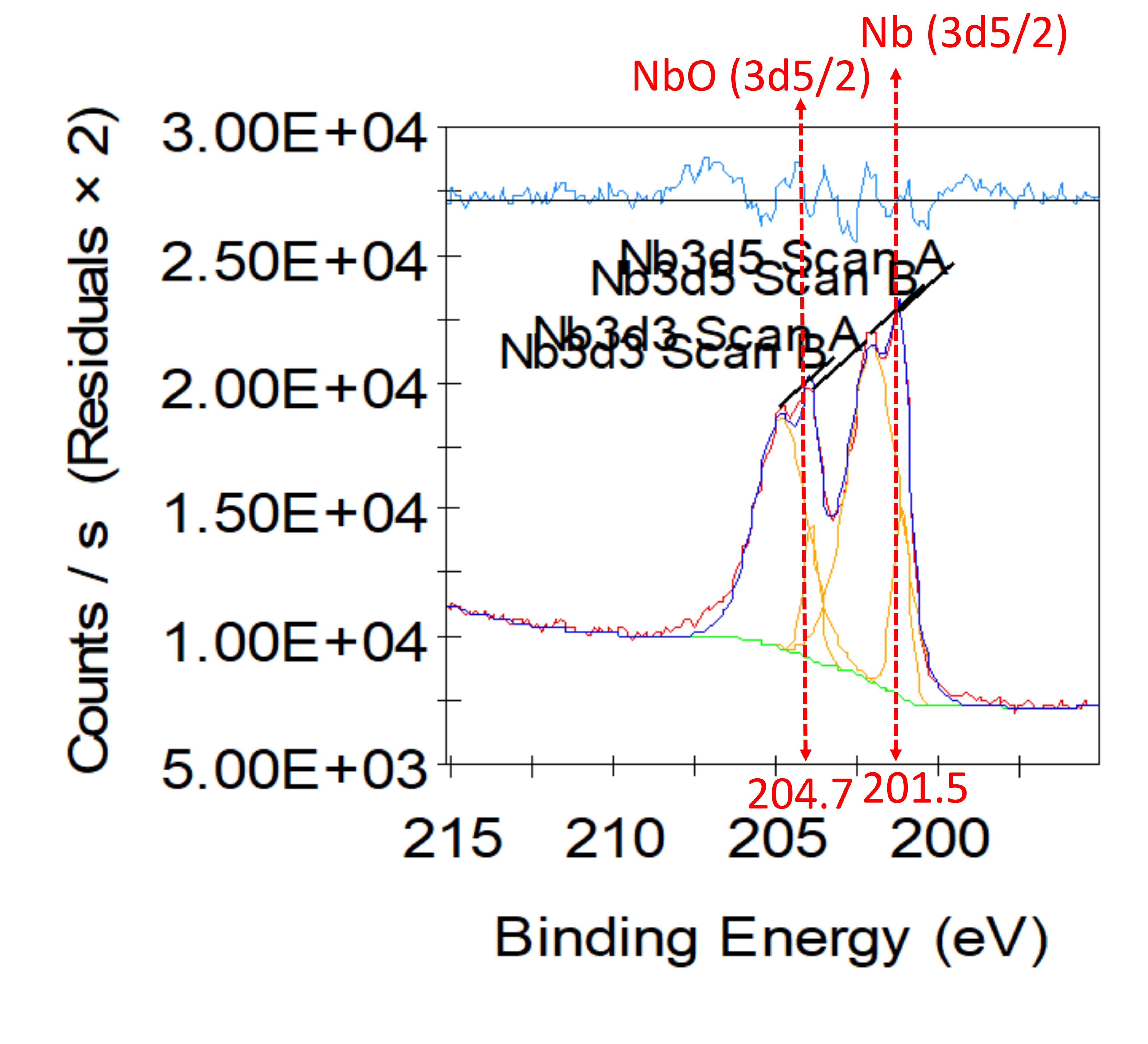


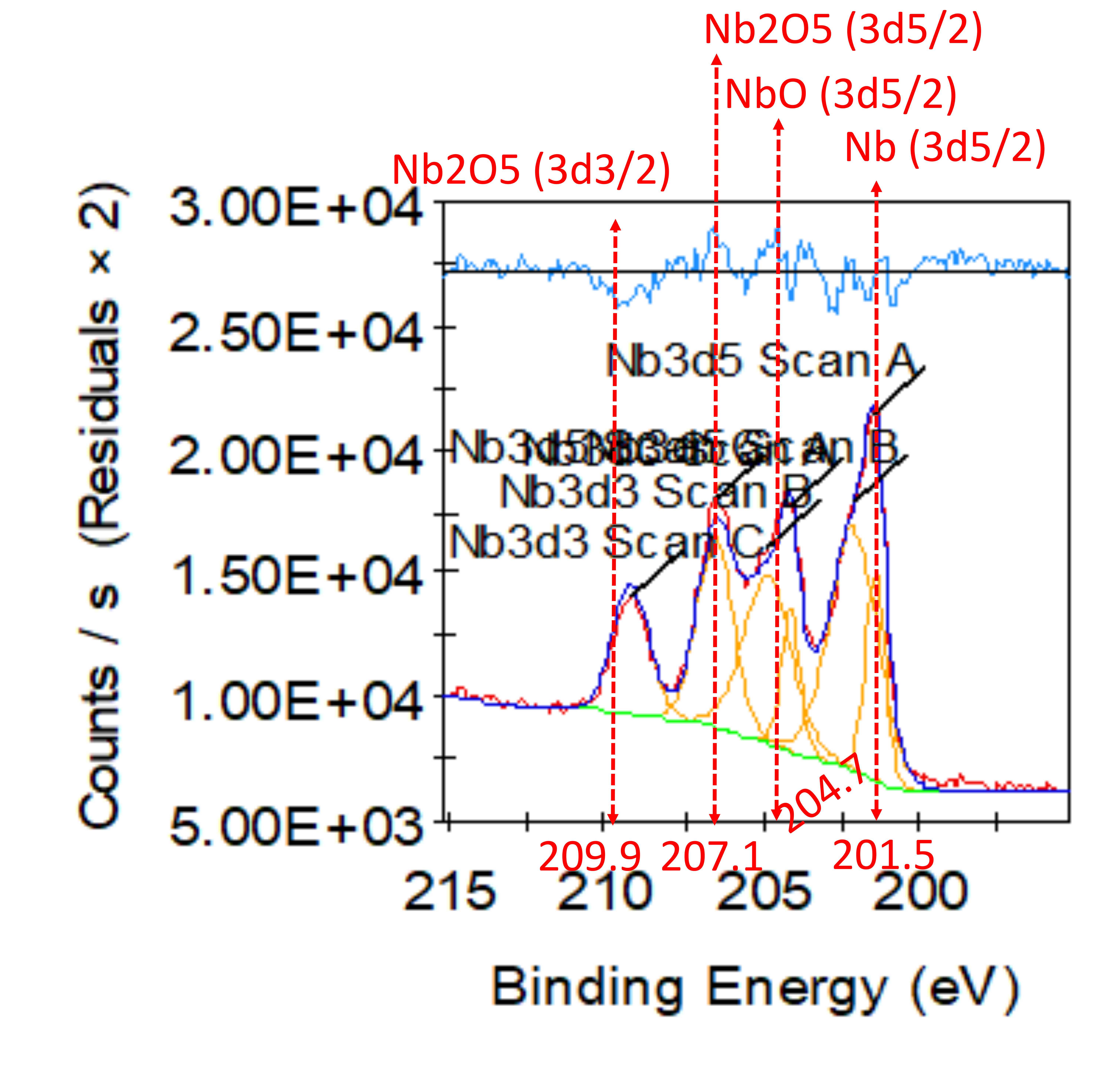
	At%
Si	54.5
Nb	26.9
O	13.9
Mg	4.7

EDS results of nanoporous Nb

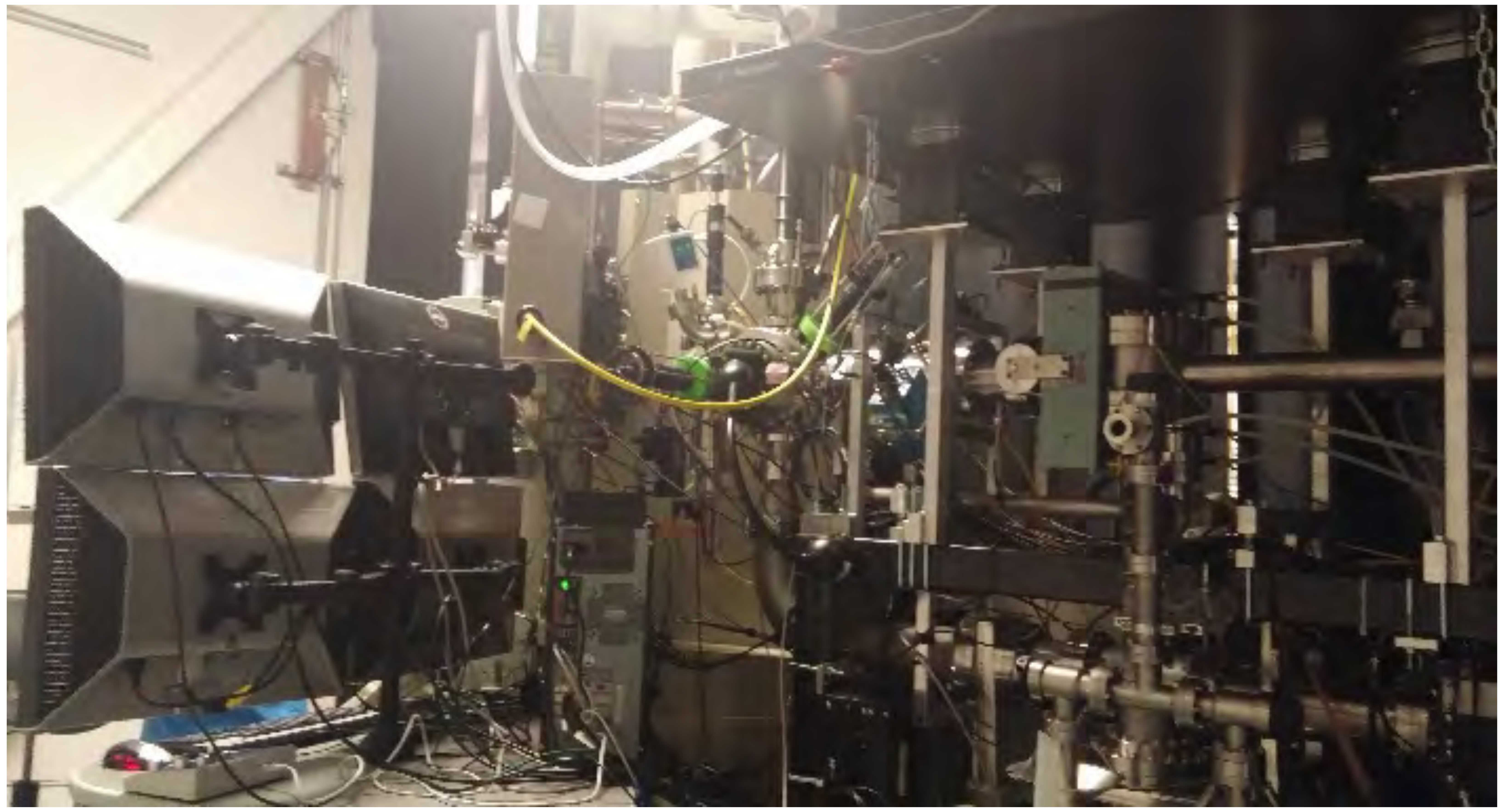
Preparation of thin nanoporous Nb samples

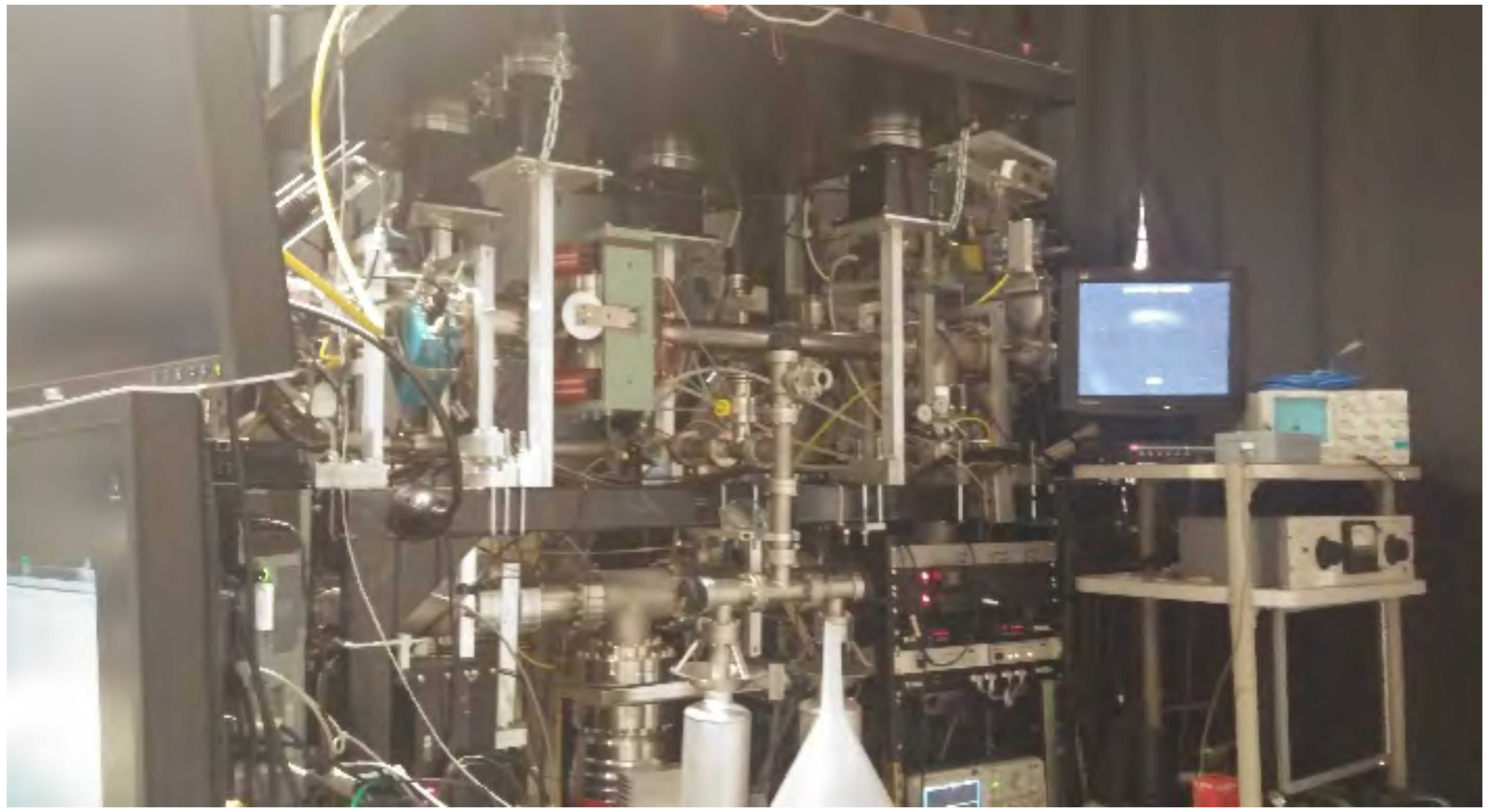
XPS results for Nb3d Scan:



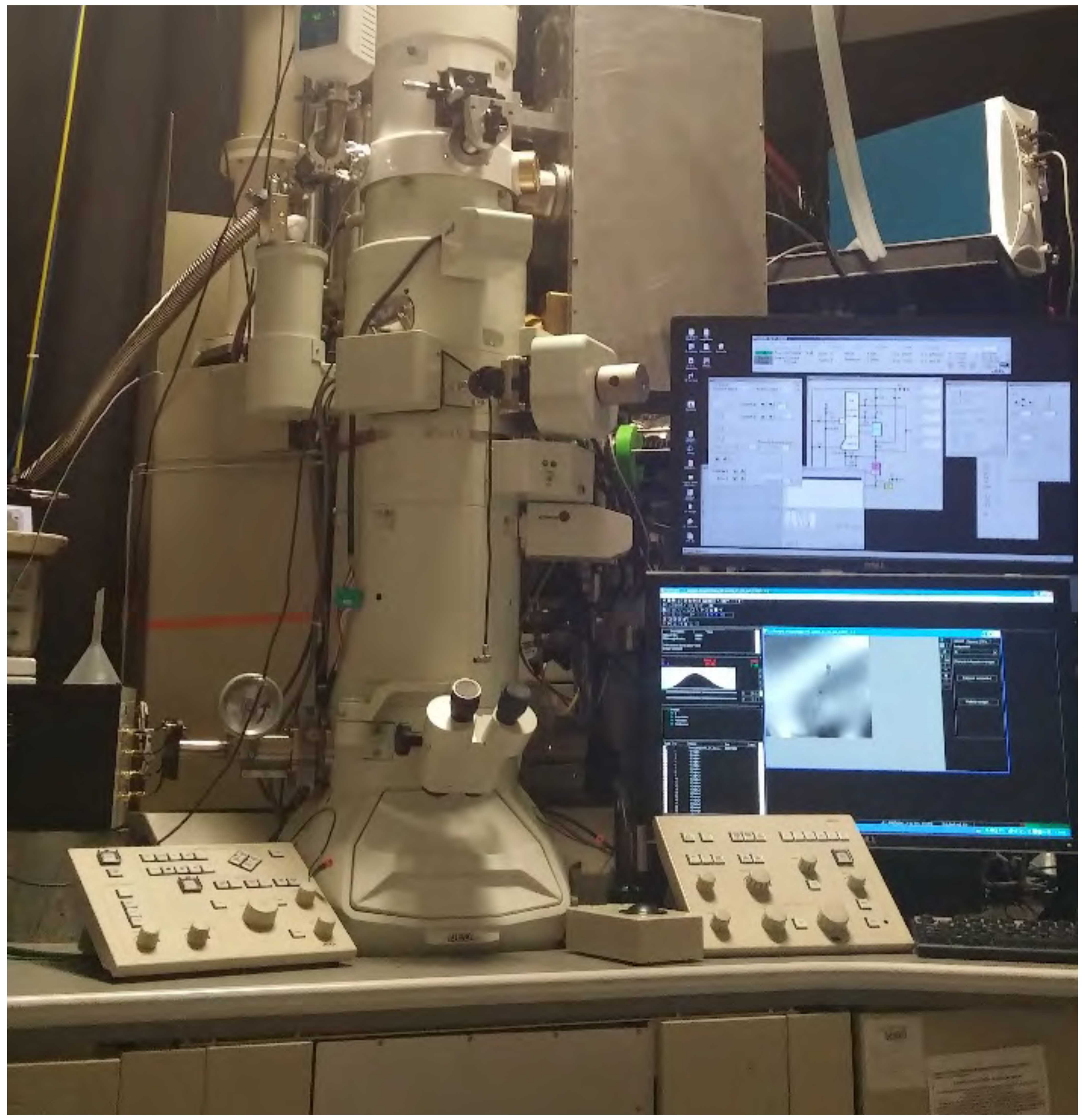


Irradiation with in situ Transmission Electron Microscopy Characterization





TEM JEOL JEM-2100 in Sandia National Laboratories



Applied ion beams:

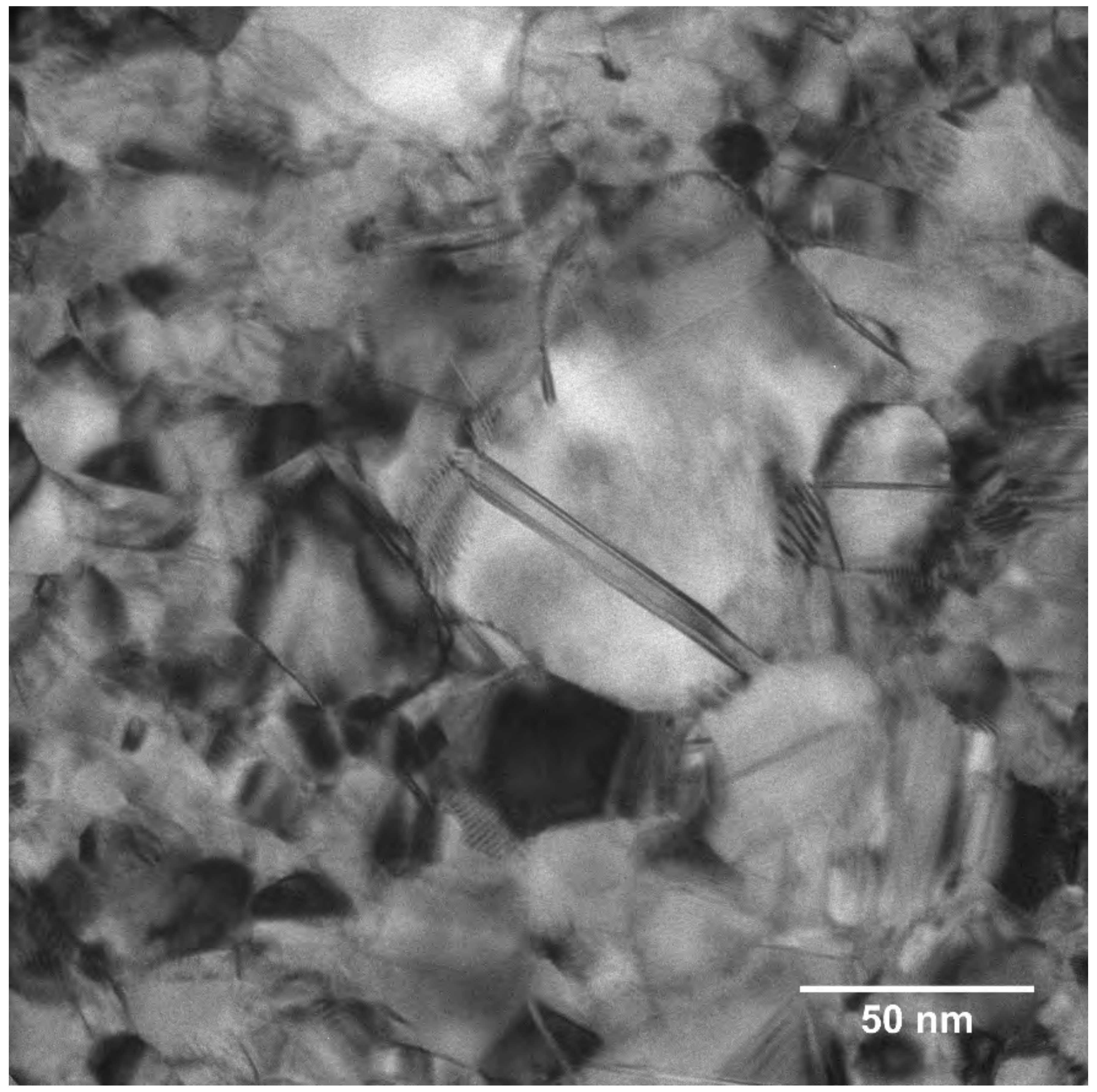
- 10 keV He^{1+} ion beam implantation
- 1.7MeV Au^{3+} ion beam was produced by accelerating ions from a source of negative ions by cesium sputtering (SNICS) through a 6MV EN Tandem accelerator

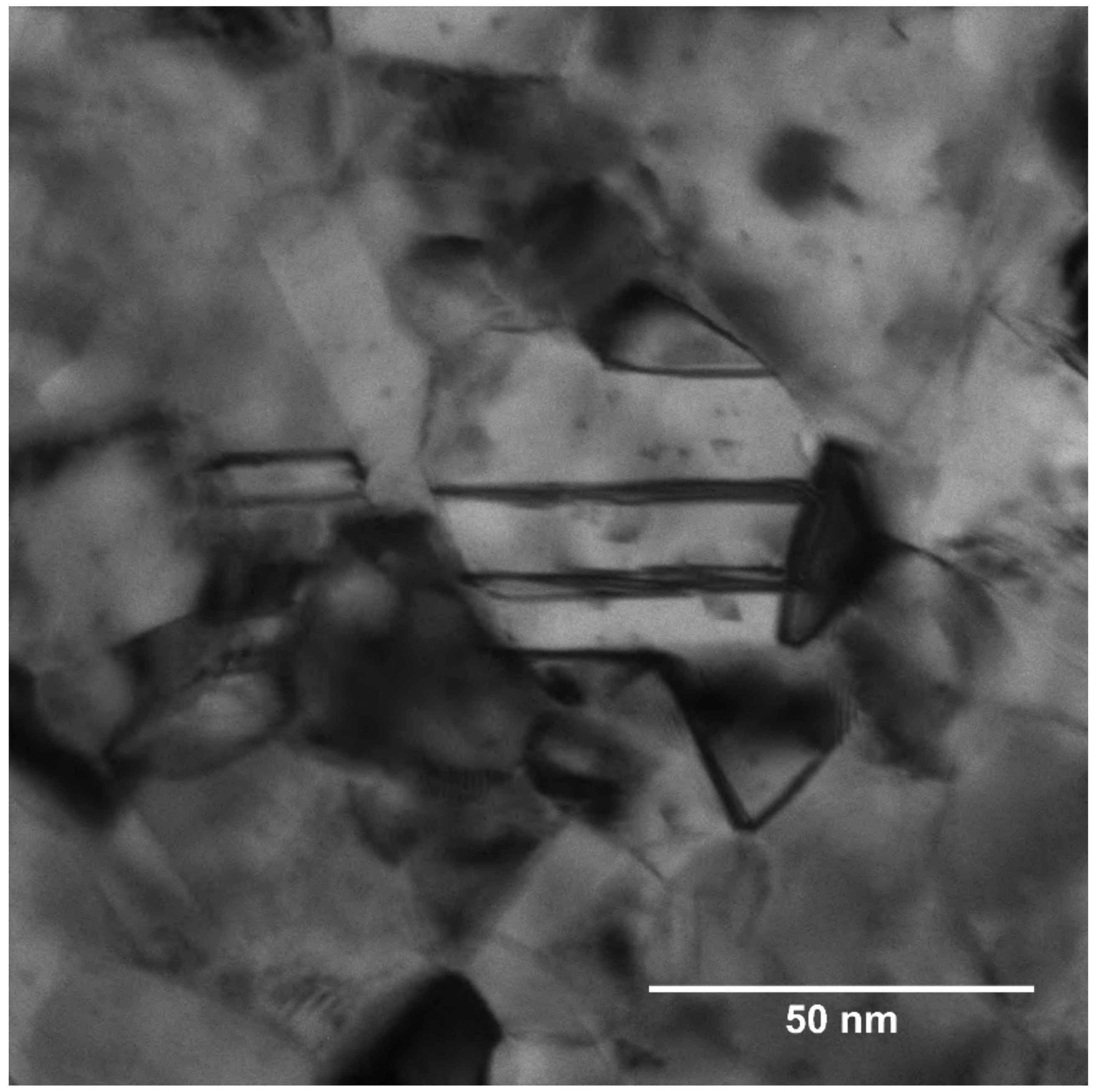
Thin film nanoporous Au →

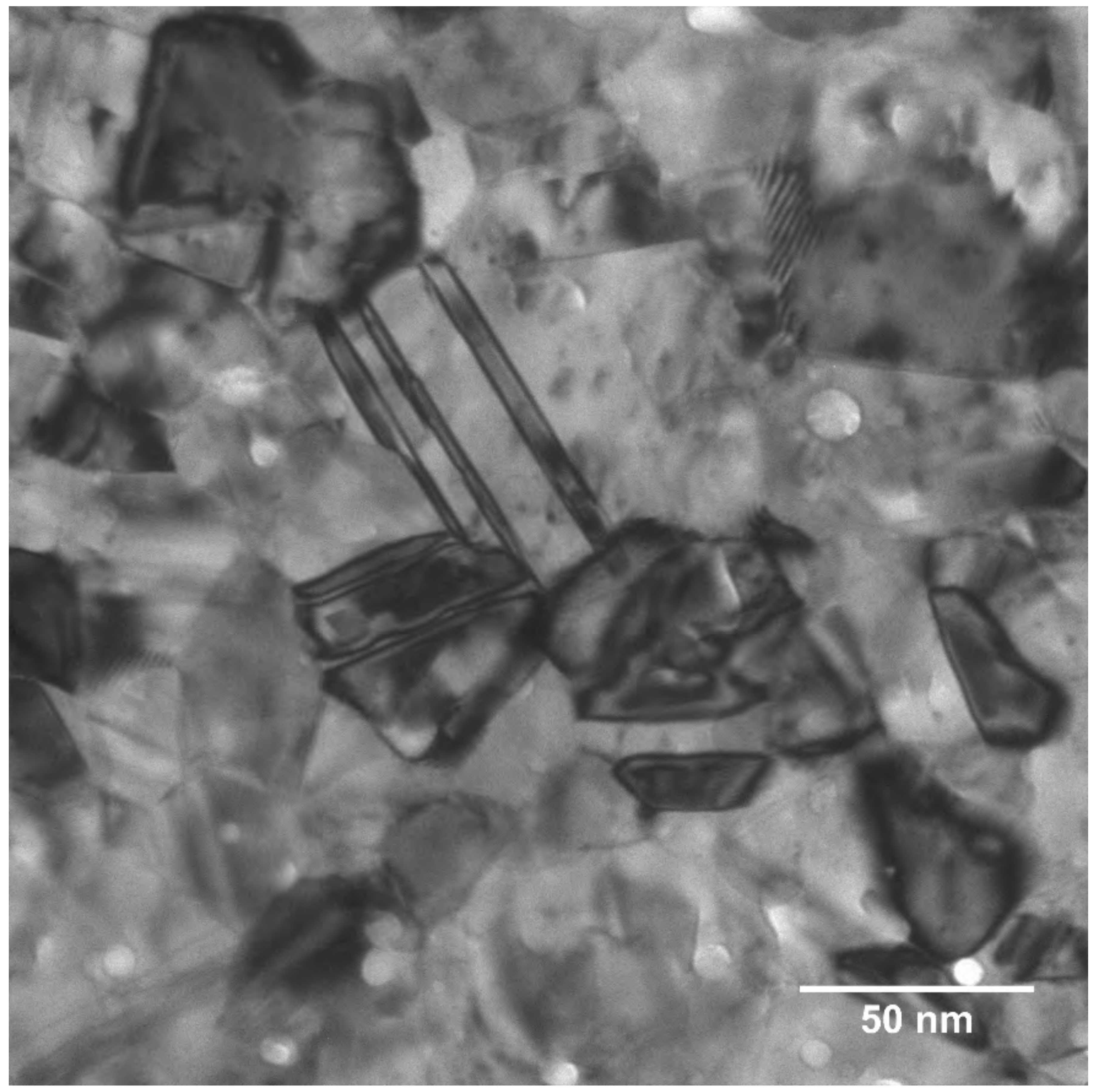
i. 1.7MeV Au^{3+}
ii. 10keV He^{1+}
iii. 10keV He^{1+} and 1.7MeV Au^{3+} simultaneously

Irradiation with in situ Transmission Electron Microscopy Characterization

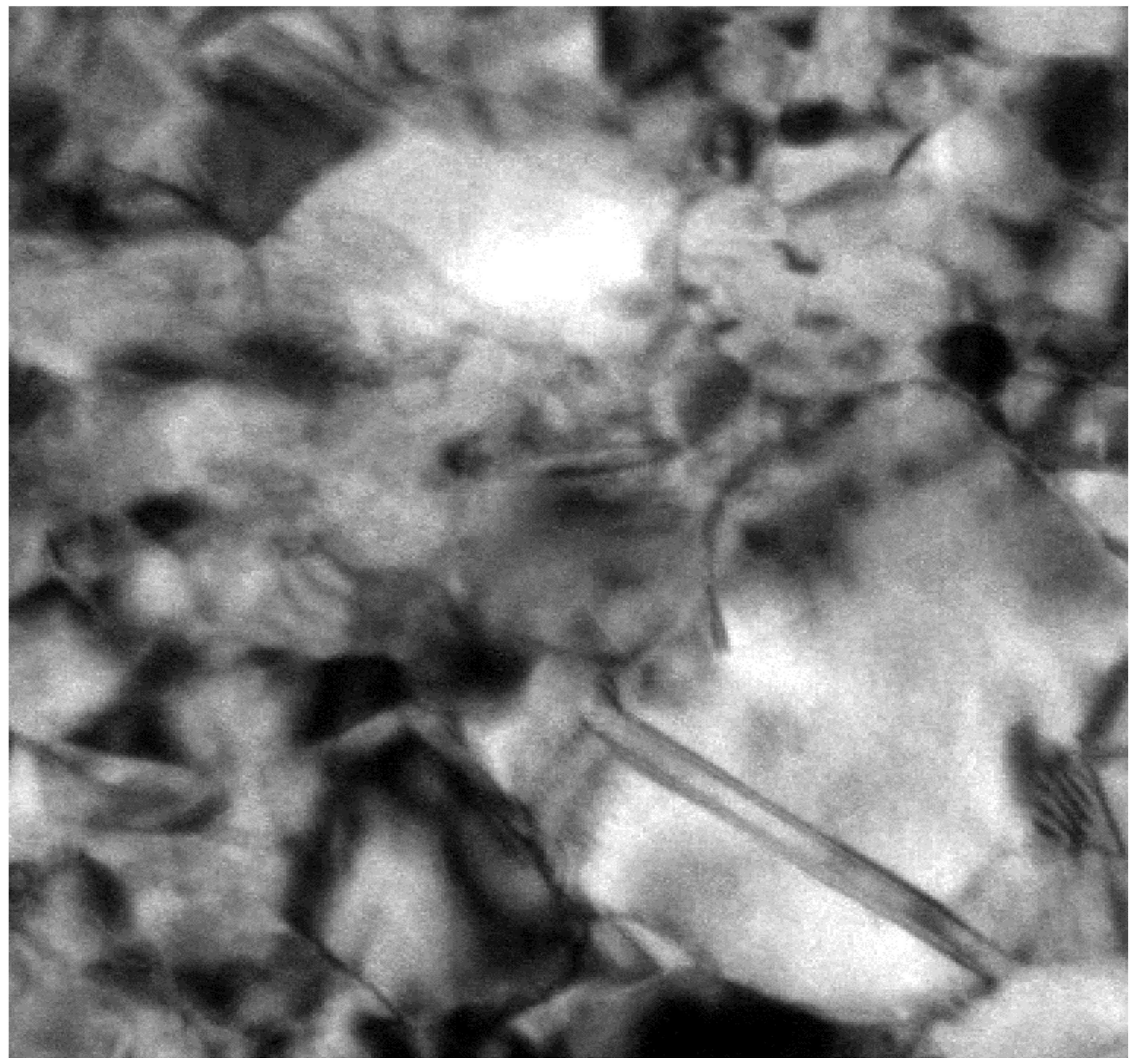
Thin film *nanocrystalline* Au → ‣ 1.7MeV Au^{3+} for 20minutes



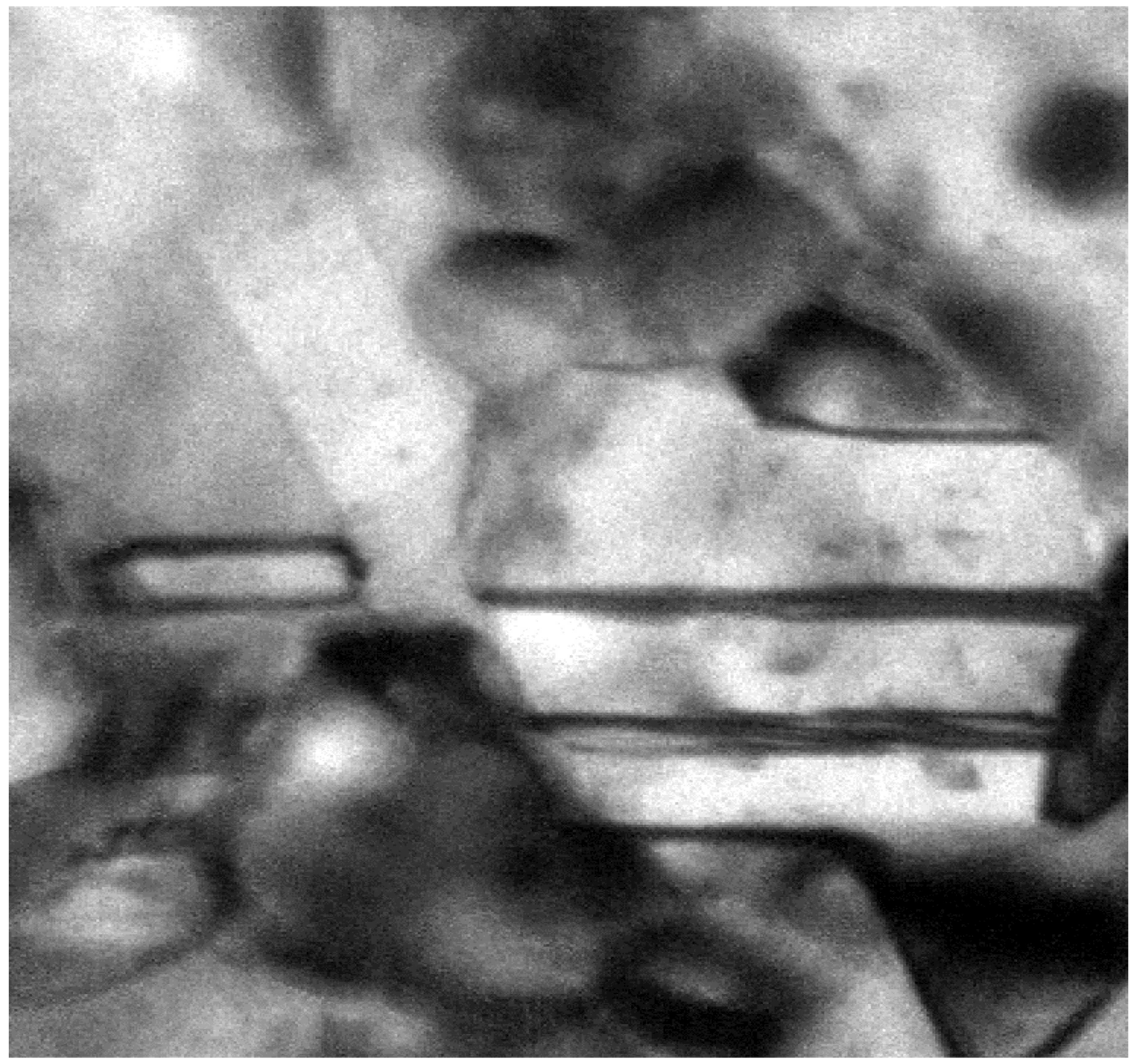




TEM image of nc Au i) before radiation, ii) after 10’ and iii) after 20’ of 1.7MeV Au^{3+} ion beam exposure



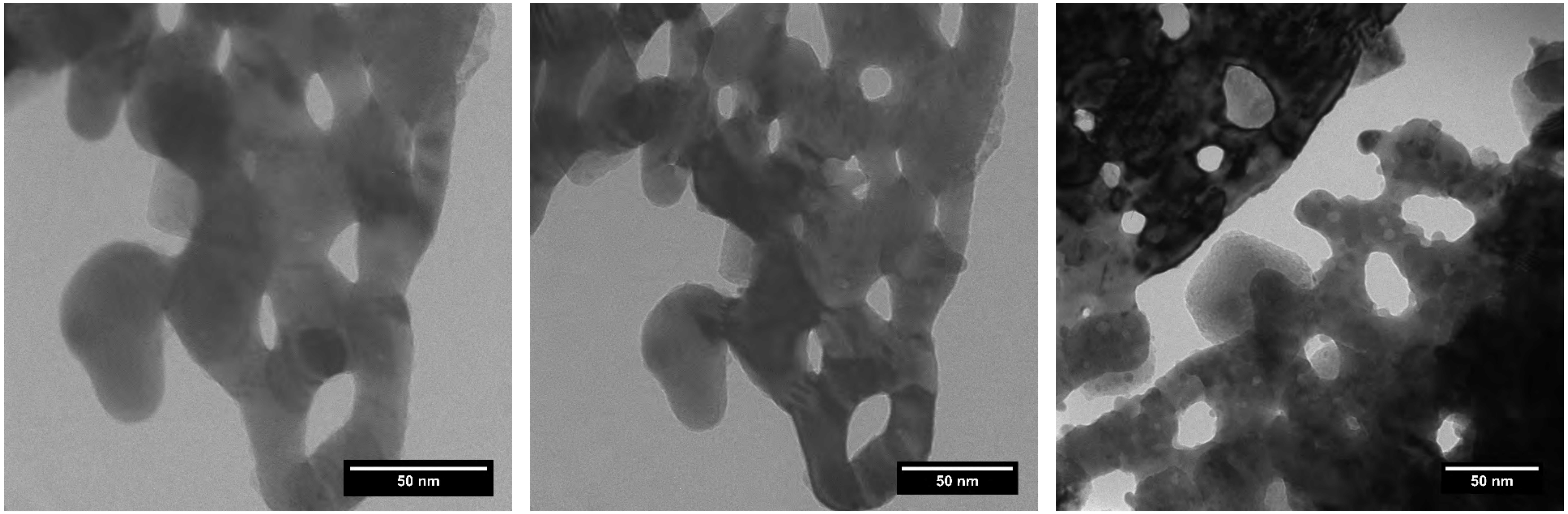
TEM video of nc Au for first 5’ of 1.7MeV Au^{3+} ion beam exposure



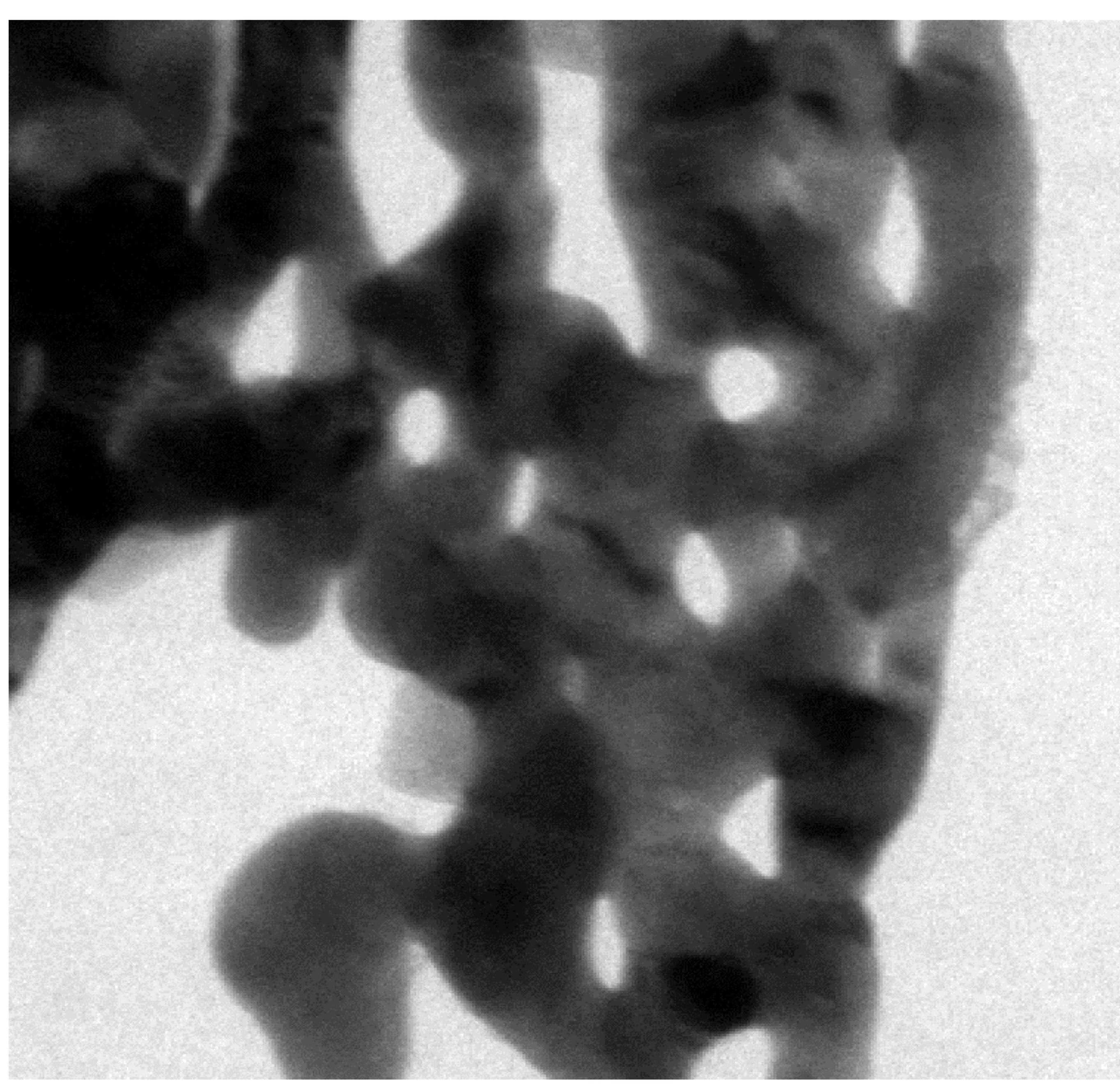
TEM video of nc Au from 10’-15’ of 1.7MeV Au^{3+} ion beam exposure

Irradiation with in situ Transmission Electron Microscopy Characterization

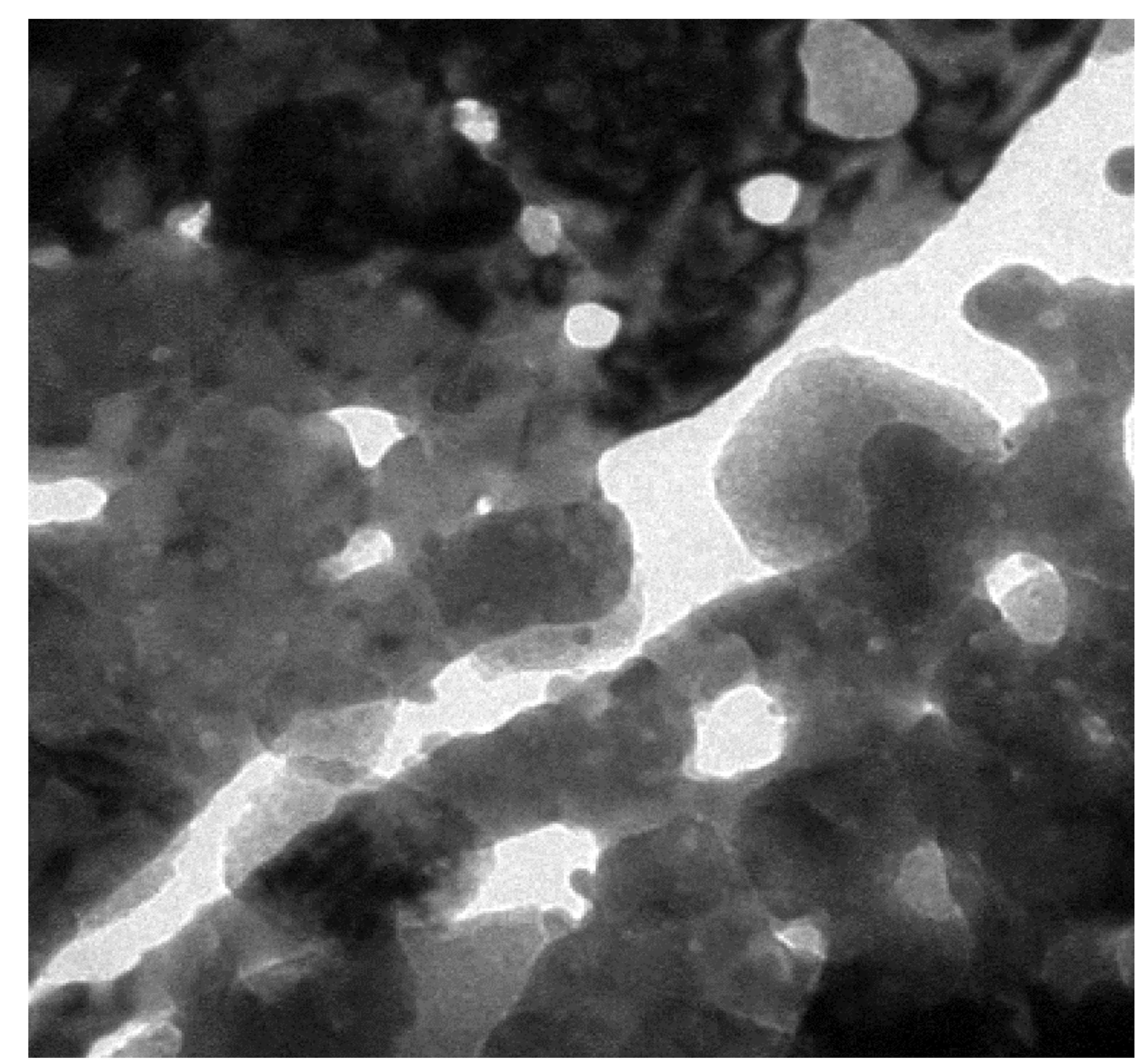
Thin film nanoporous Au → i. 1.7MeV Au^{3+} for 20minutes



TEM image of np Au i) before radiation, ii) after 10’ and iii) after 20’ of 1.7MeV Au^{3+} ion beam exposure



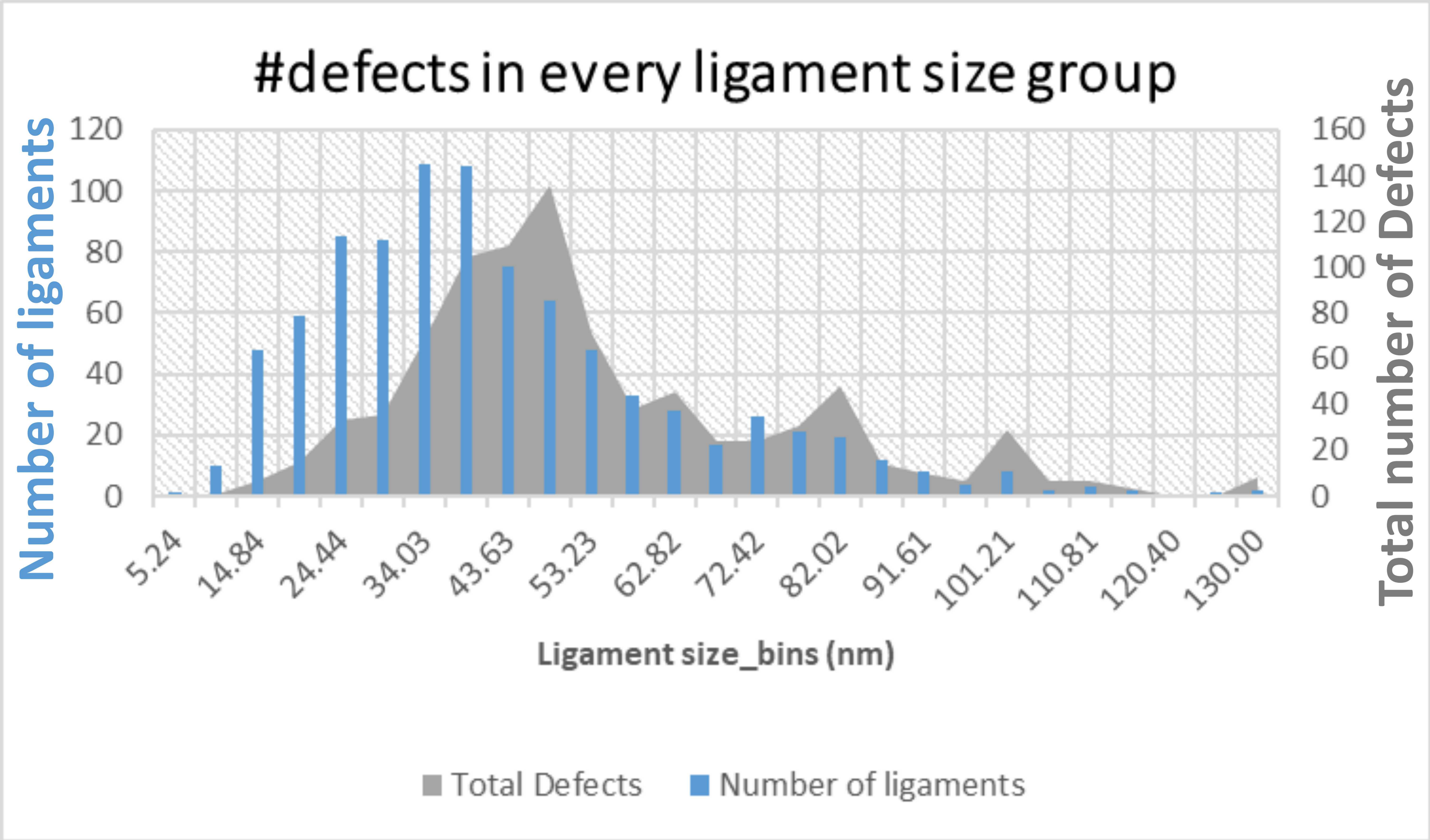
TEM video of np Au for first 10’ of 1.7MeV Au^{3+} ion beam exposure

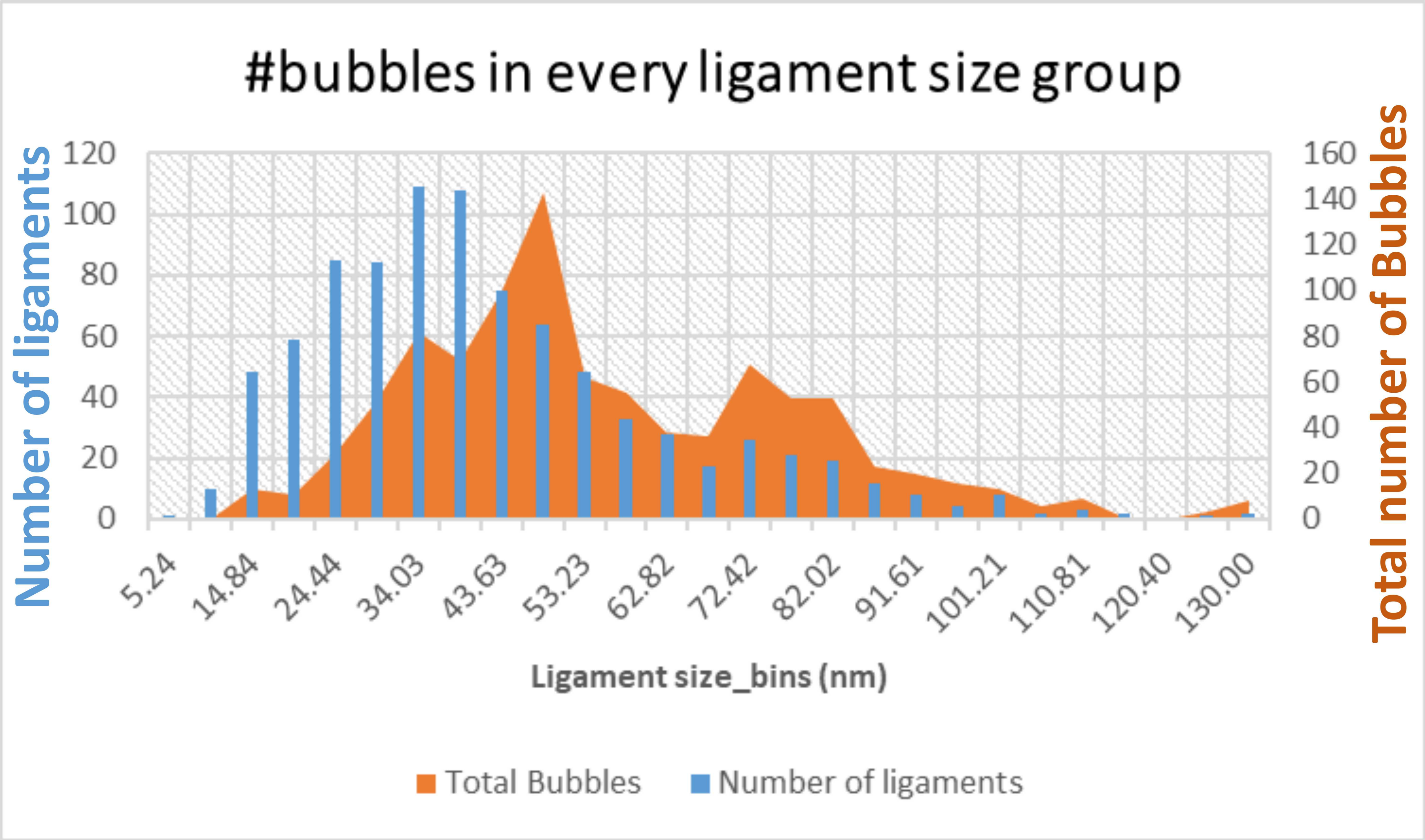


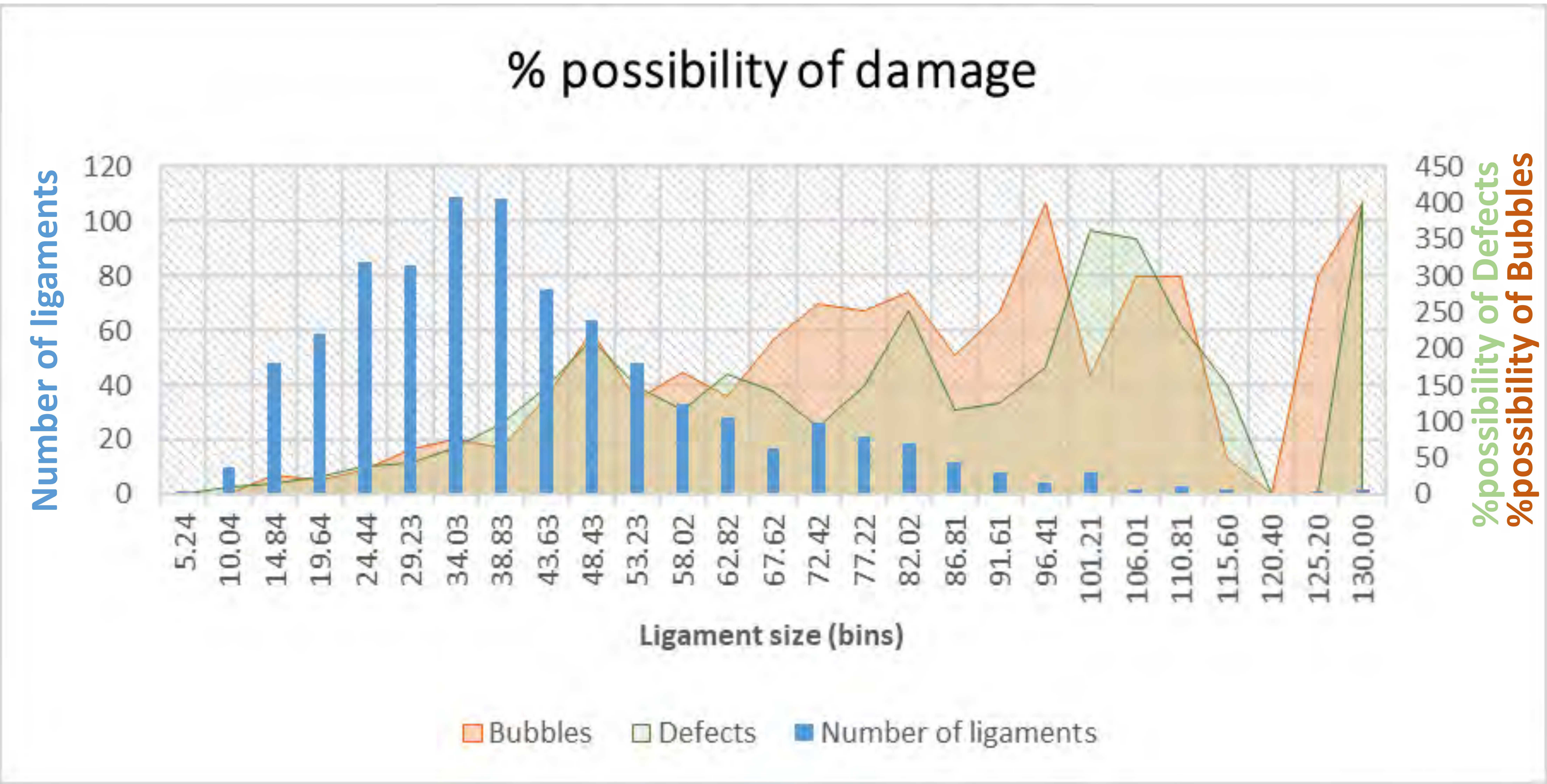
TEM video of np Au from 10’-20’ of 1.7MeV Au^{3+} ion beam exposure

Irradiation with in situ Transmission Electron Microscopy Characterization

Thin film nanoporous Au → ii. 1.7MeV Au^{3+} for 20minutes



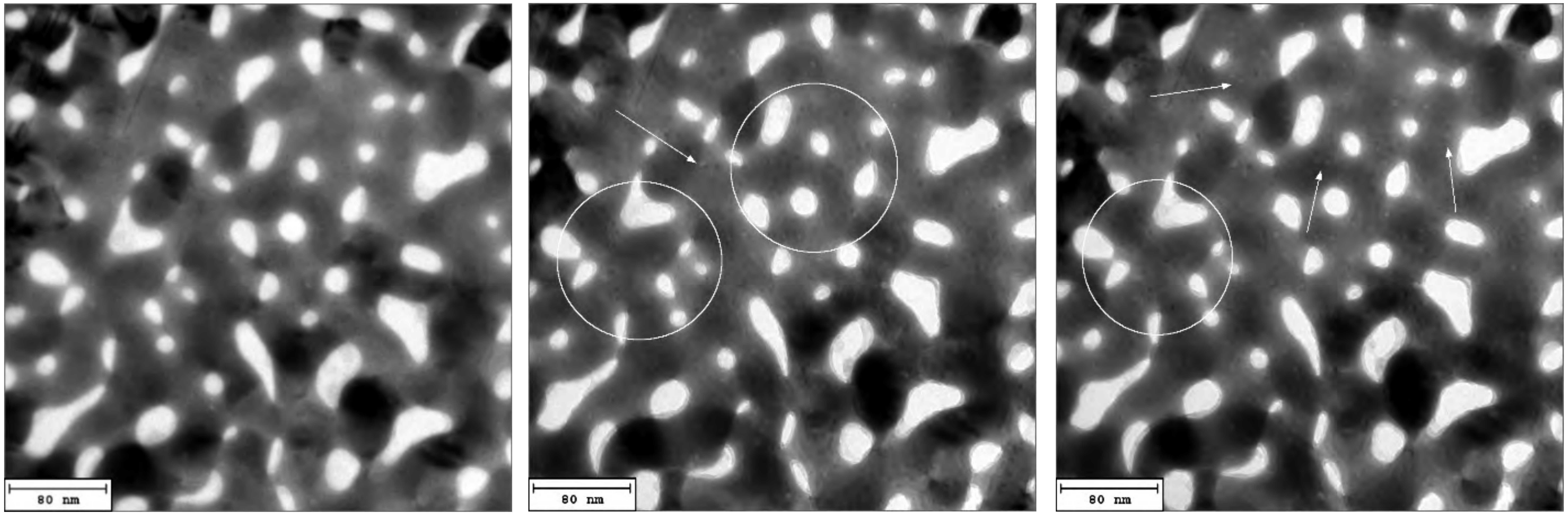




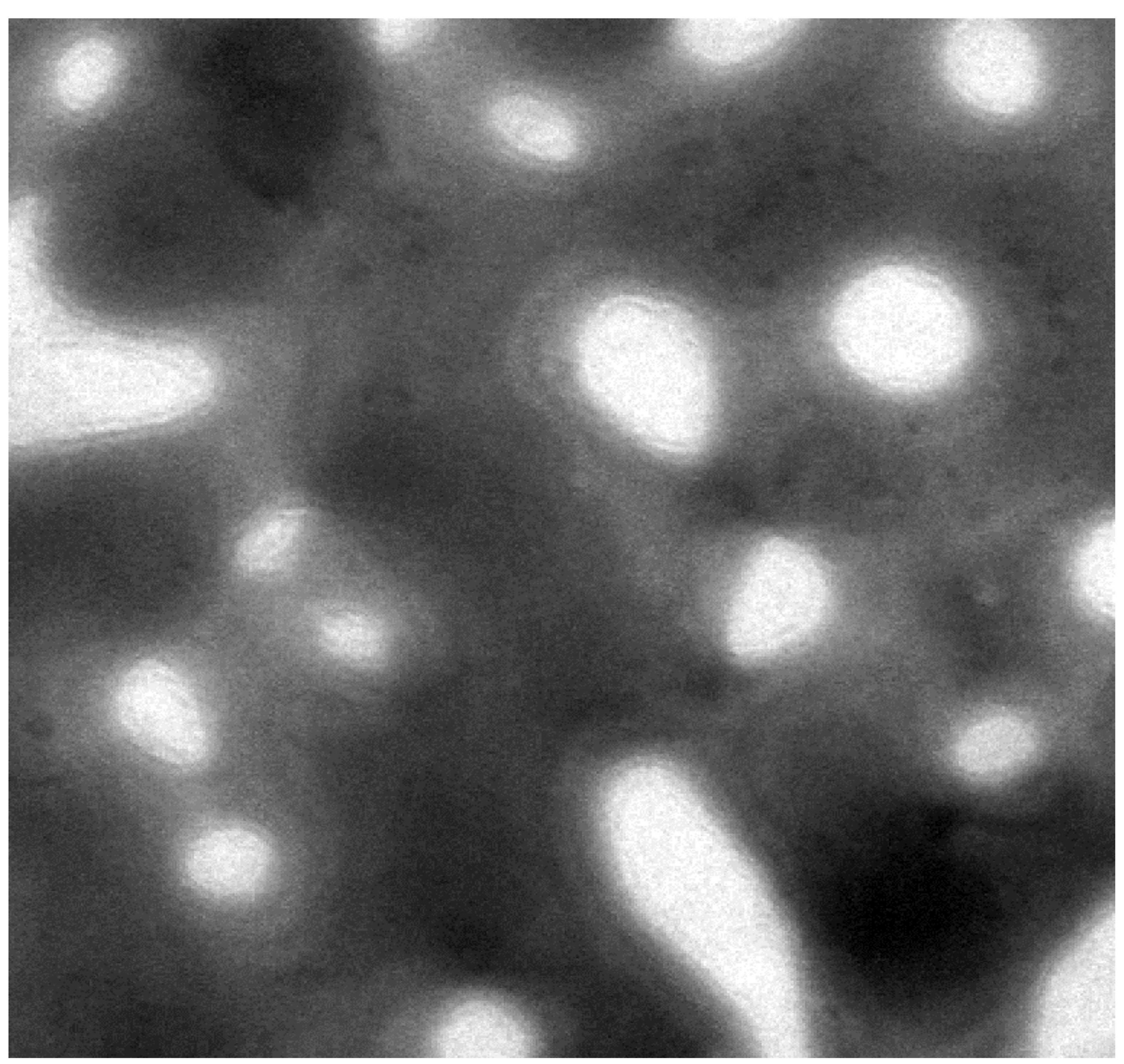
$$\%\text{possibility of damage} = \frac{\text{Total number of damage}}{\text{Number of ligaments}} * 100$$

Irradiation with in situ Transmission Electron Microscopy Characterization

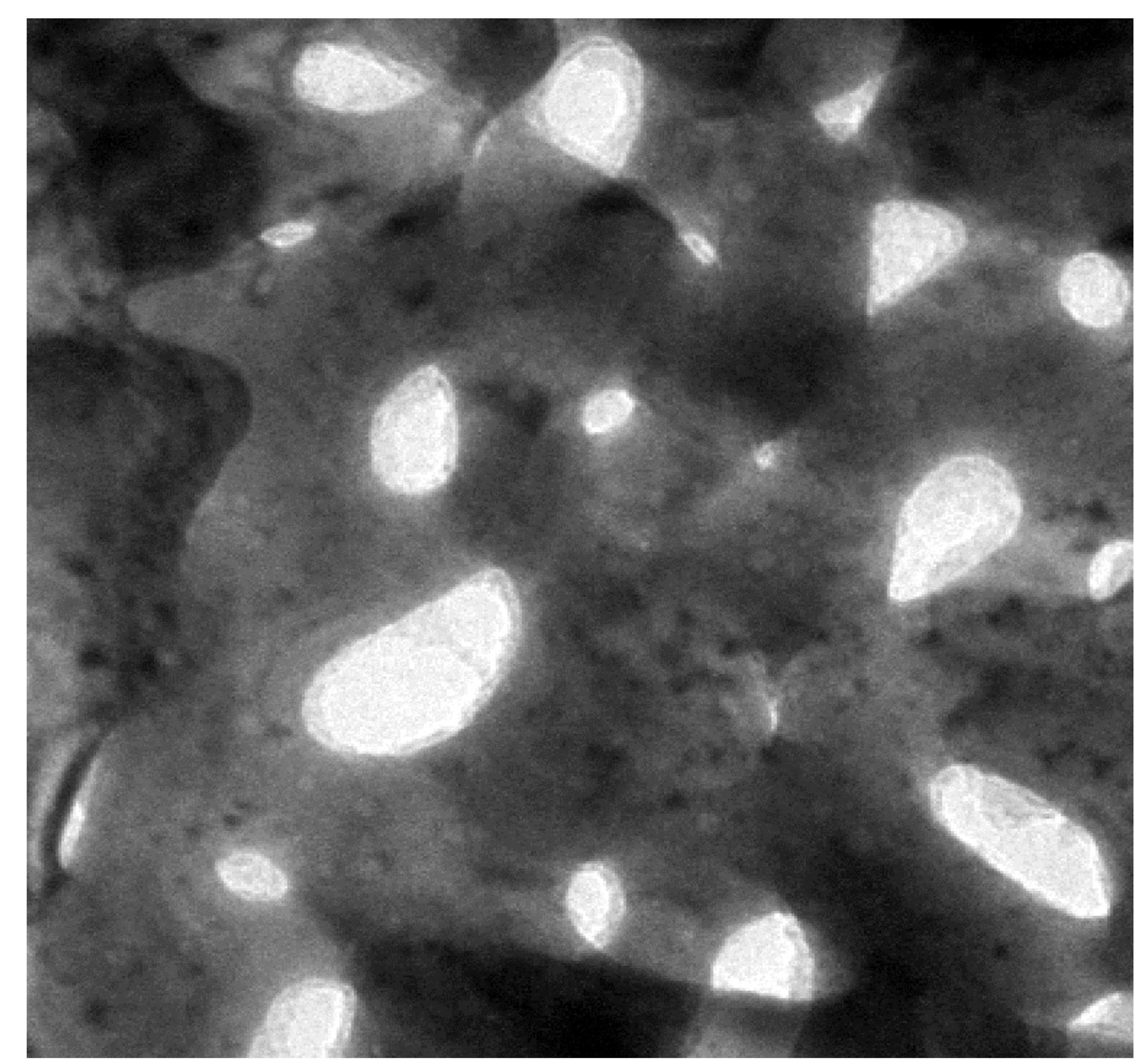
Thin film nanoporous Au → ii. 10keV He^{1+} for 20minutes



TEM image of np Au i) before radiation, ii) after 10’ and iii) after 20’ of 10keV He^{1+} ion beam exposure



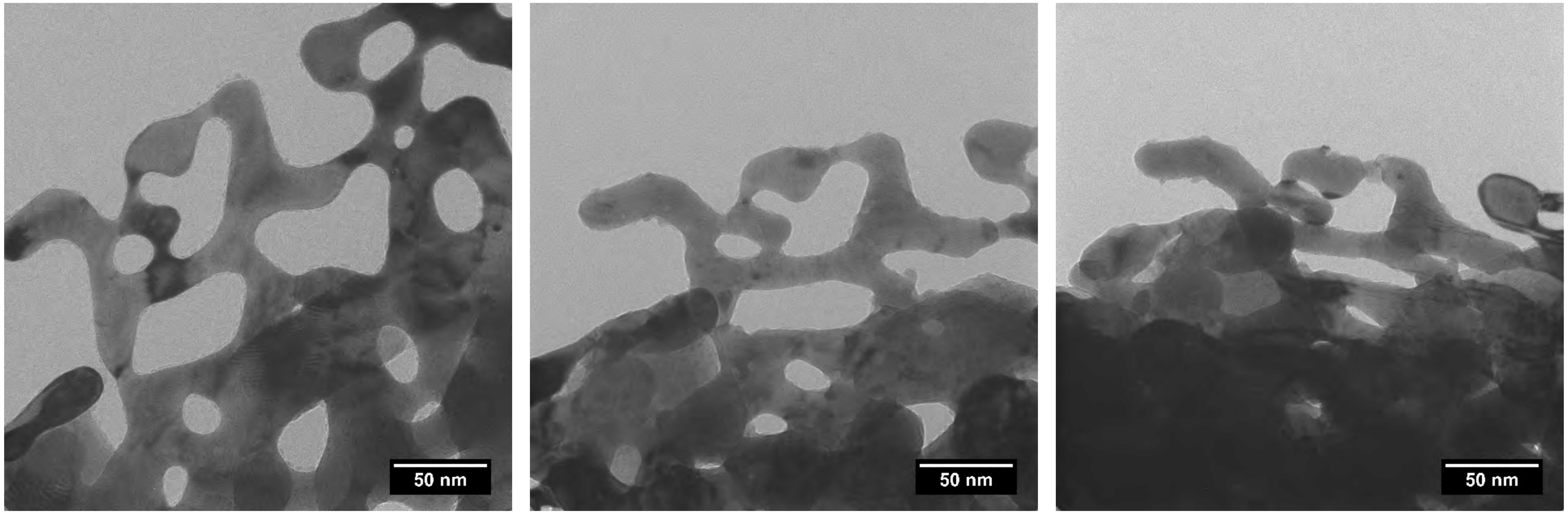
TEM video of np Au for first 5’ of 10keV He^{1+} ion beam exposure



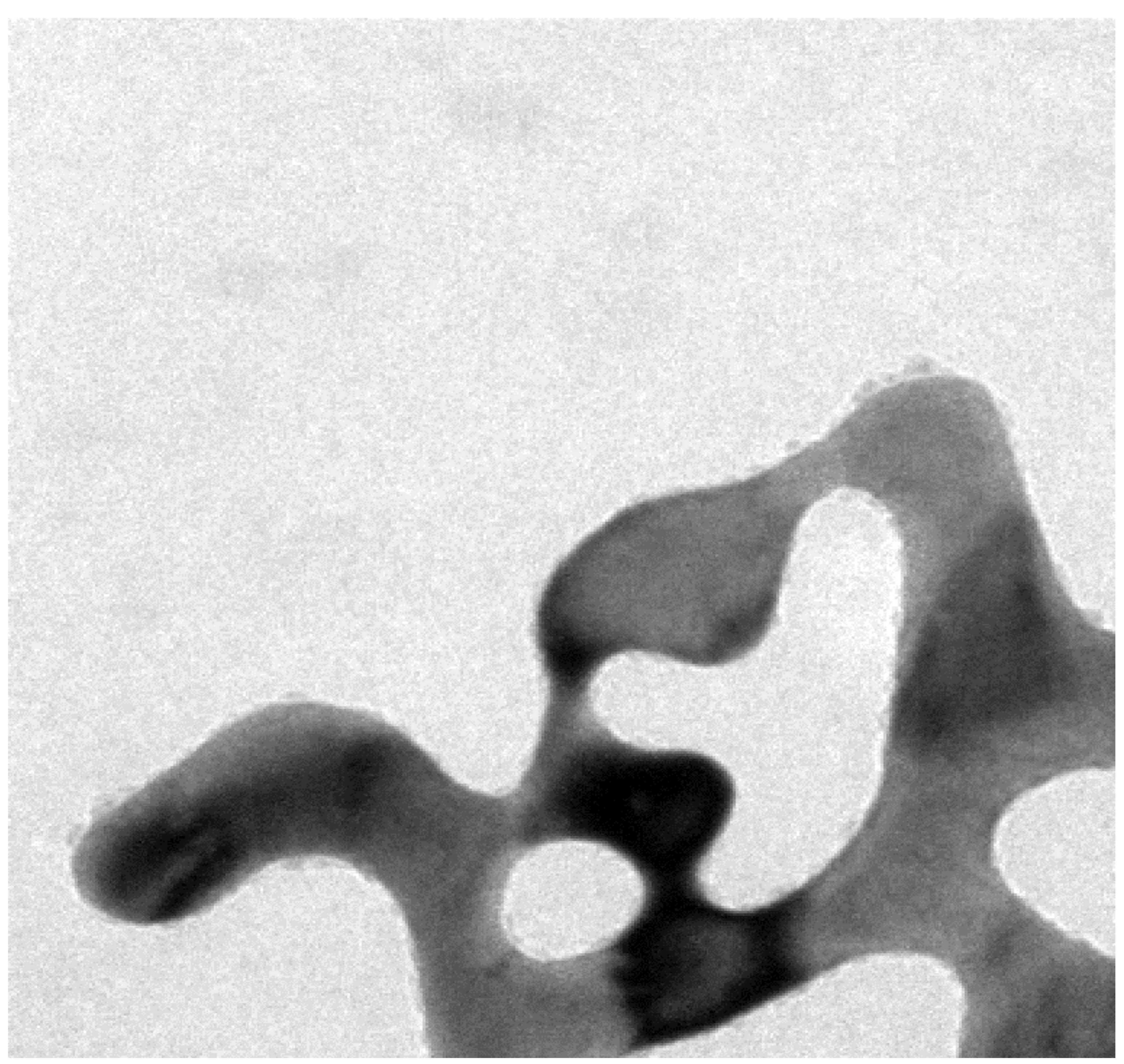
TEM video of np Au from 10’-20’ of 10keV He^{1+} ion beam exposure

Irradiation with in situ Transmission Electron Microscopy Characterization

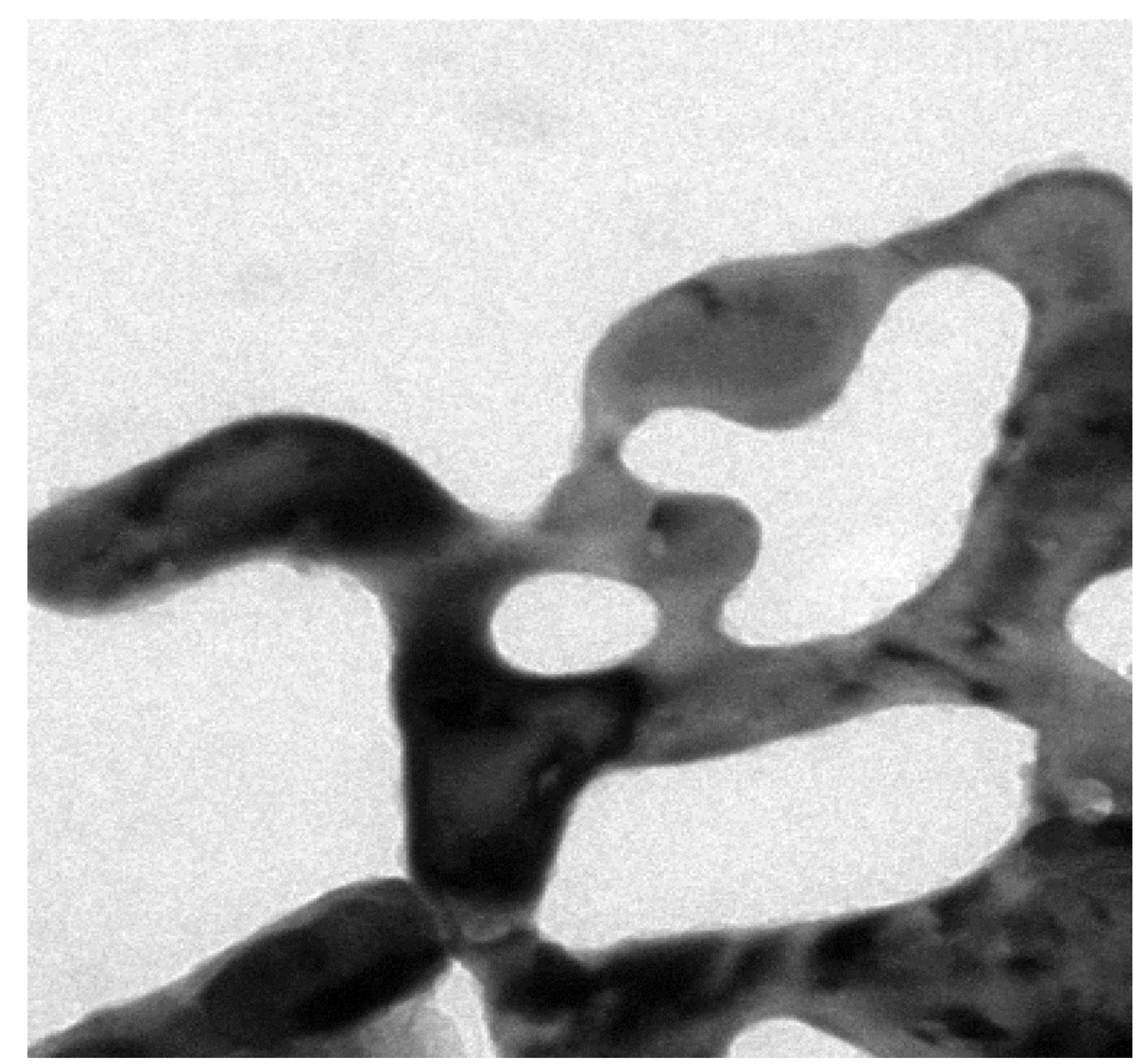
Thin film nanoporous Au iii. 10keV He^{1+} and 1.7MeV Au^{3+} for 30minutes



TEM image of np Au i) before, ii) after 15’ and iii) after 30’ of 10keV He^{1+} and 1.7MeV Au^{3+} ion beam exposure



TEM video of np Au for first 5’ of 1.7MeV Au^{3+} and 10keV He^{1+} ion beams exposure

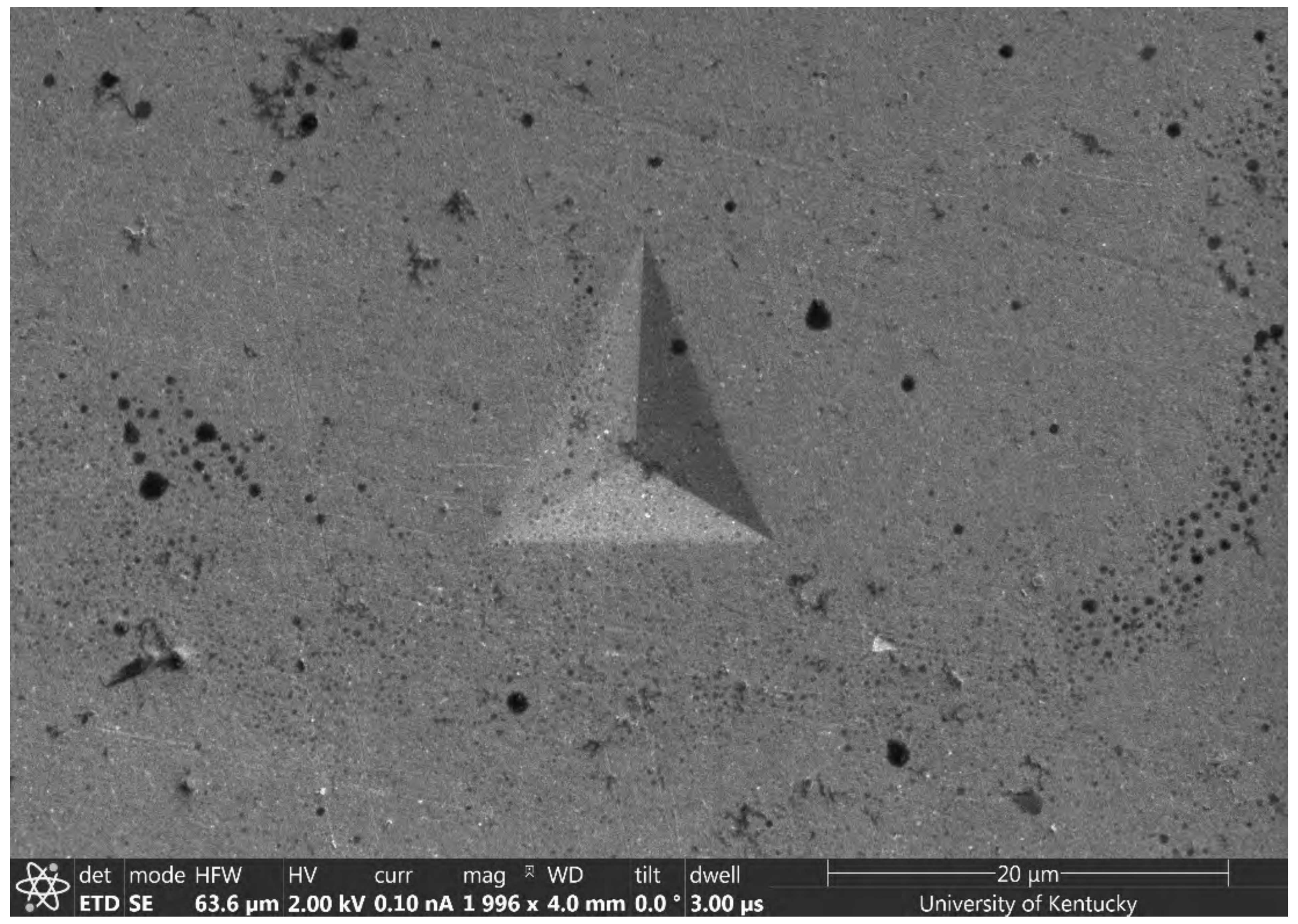


TEM video of np Au from 10’-15’ of 1.7MeV Au^{3+} and 10keV He^{1+} ion beams exposure

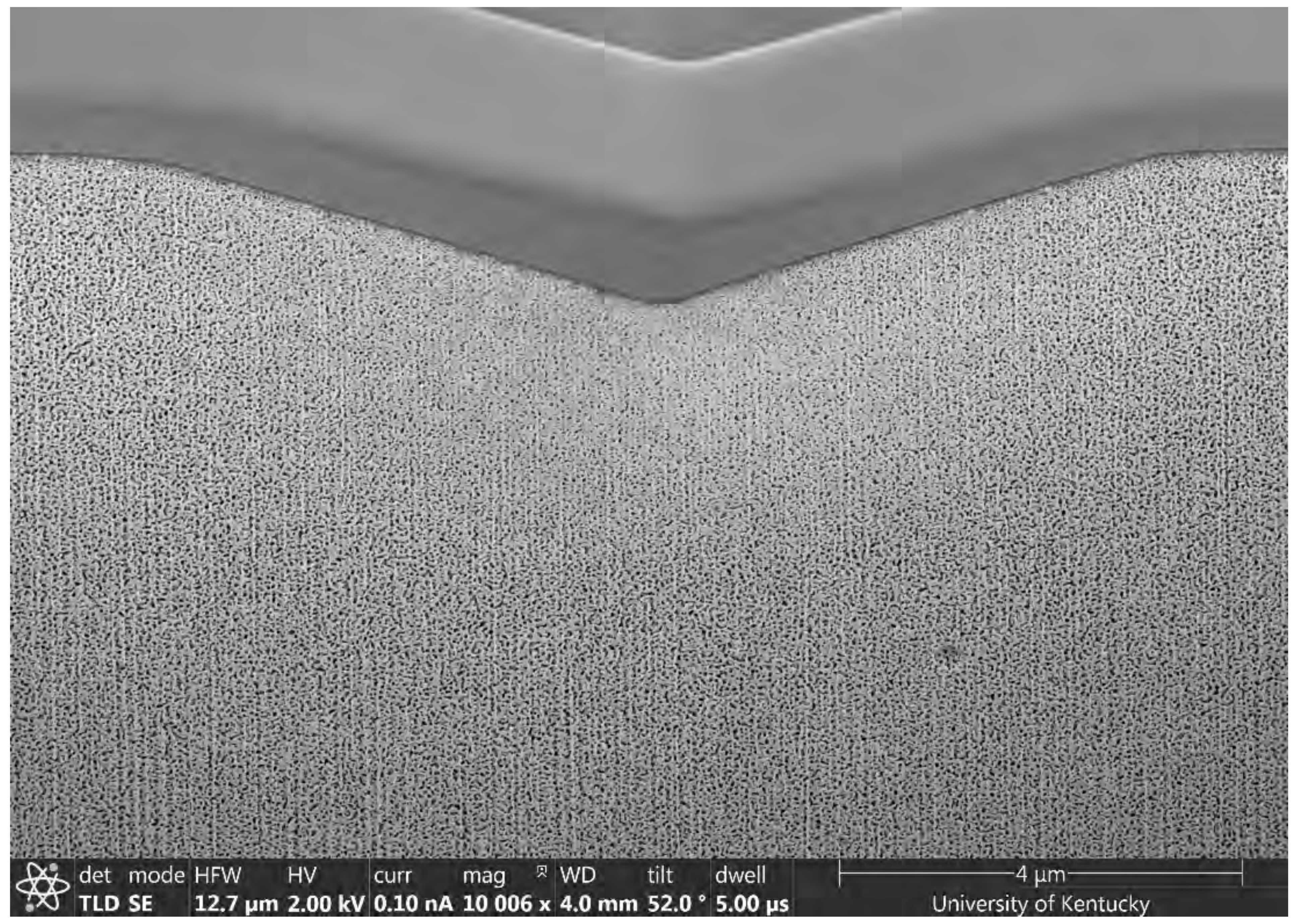
Material characterization with nanoindentation

Radiation conditions of each bulk np Au sample

A/A	Ion Beam	Energy of ion beam (MeV)	Ion dose (ions/cm^2)
#1	Au^{4+}	10	10^{10}
#2	Au^{4+}	10	10^{12}
#3	Au^{6+}	20	10^{10}



Nanoindented surface of np Au



Nanoindented cross section of np Au

Hardness versus depth for bulk np Au samples

Conclusion

‣Nanocrystalline samples exhibit greater radiation damage than nanoporous structures.

‣Ligaments of lower width had higher possibility not to appear any radiation damage compared to larger ones.

‣Longer exposure as well as greater ion dose should be applied on bulk nanoporous Au samples in order to identify the hardness response to radiation.

Thank you for your attention