

In-Situ Studies of Nanoporous Metals During Irradiation and Deformation

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During Irradiation and Deformation*



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SPRING MEETING & EXHIBIT

April 2-6, 2018 | Phoenix, Arizona

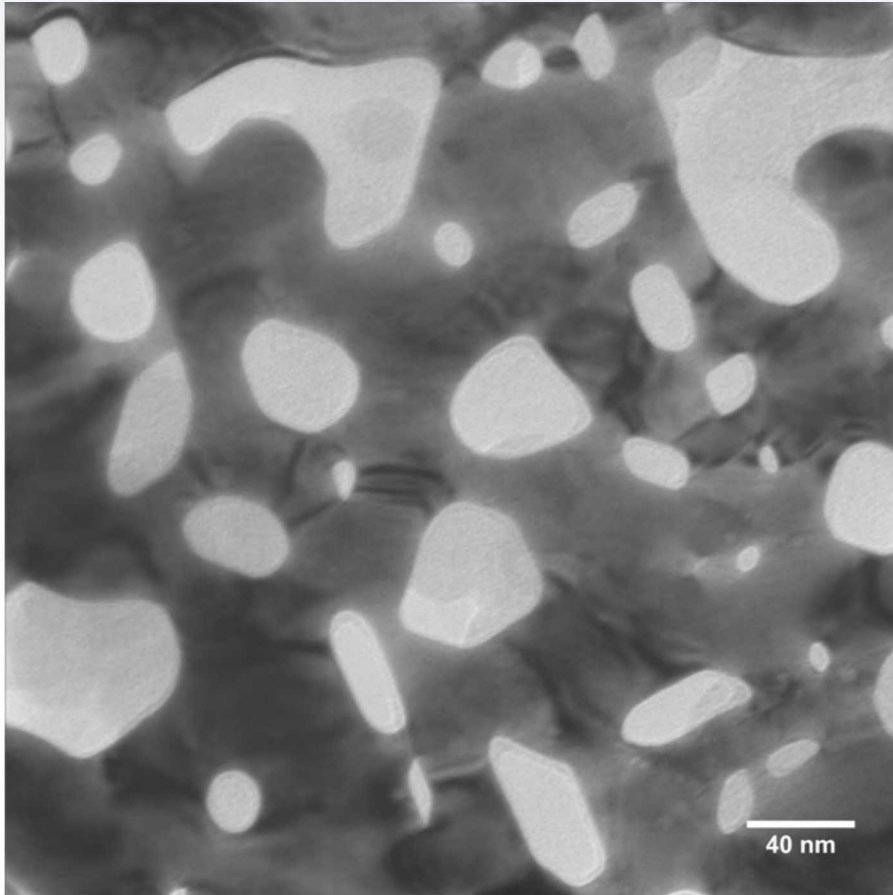
Preparation of thin nanoporous Au samples

Gold/Silver leaves (Monocrystalline np Au)

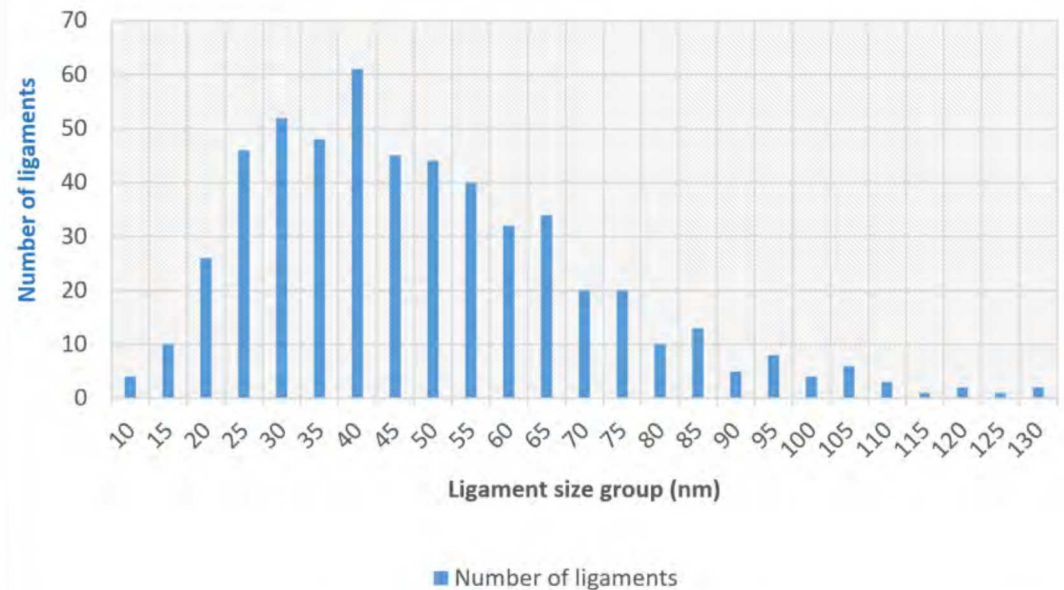
- 35 at% Au and 65 at% Ag leaves (Giusto Manetti Battiloro[®]) were rolled to reduce thickness
- Free dealloying in concentrated HNO_3 for 2h
- Cleaned in deionized H_2O for 24h



Dealloying Au:Ag leaves in concentrated HNO_3

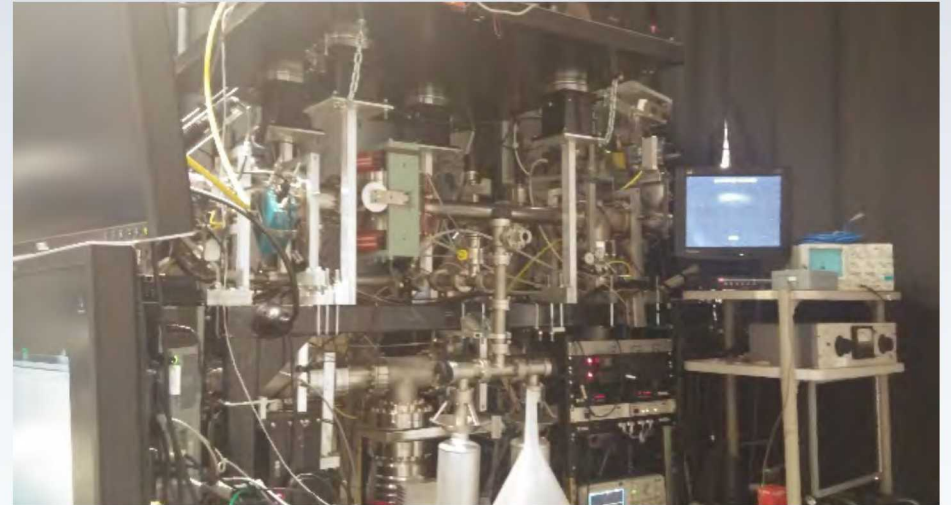
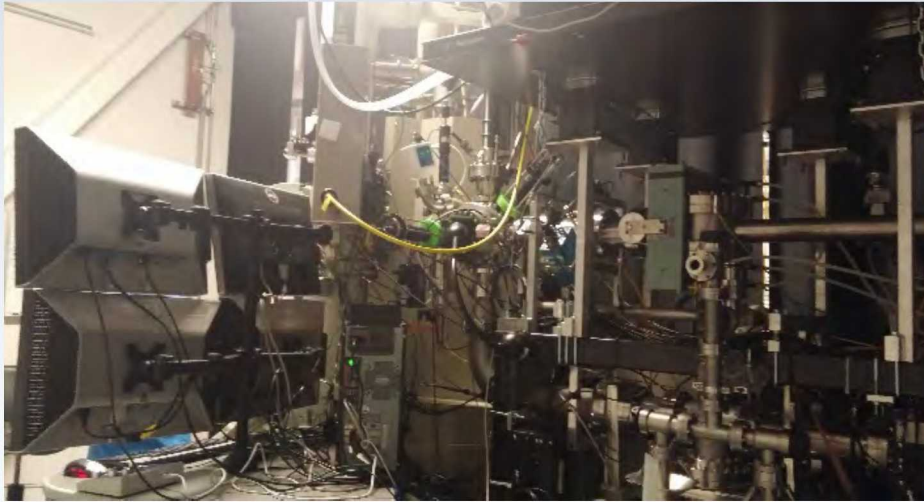


TEM micrograph of as-prepared nanoporous Au after dealloying process with an average ligament size in the order of 40nm



Average ligament size of nanoporous Au is 40nm

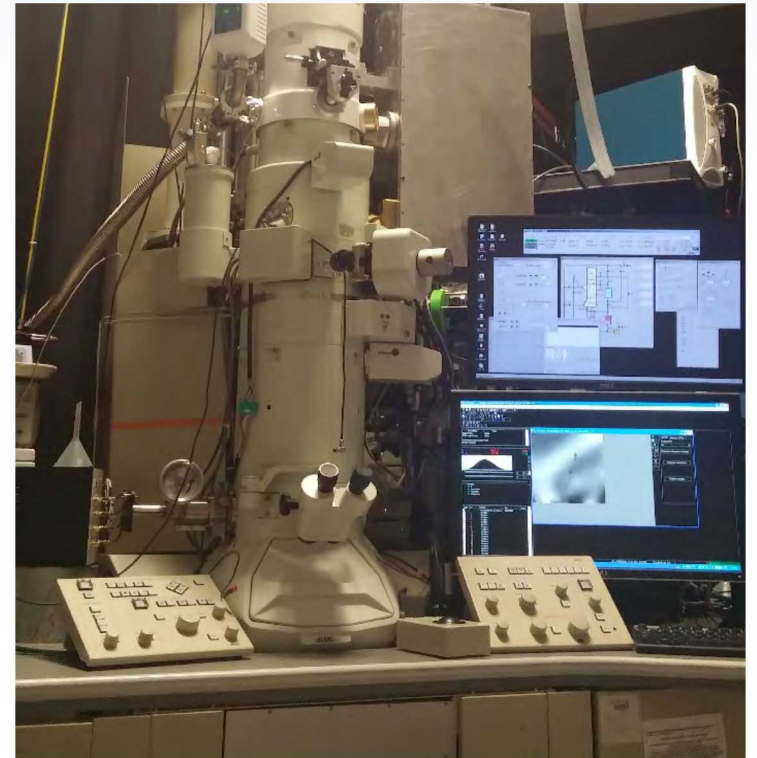
In situ Transmission Electron Microscopy Irradiation Center in SNL



TEM JEOL JEM-2100 in Sandia National Laboratories

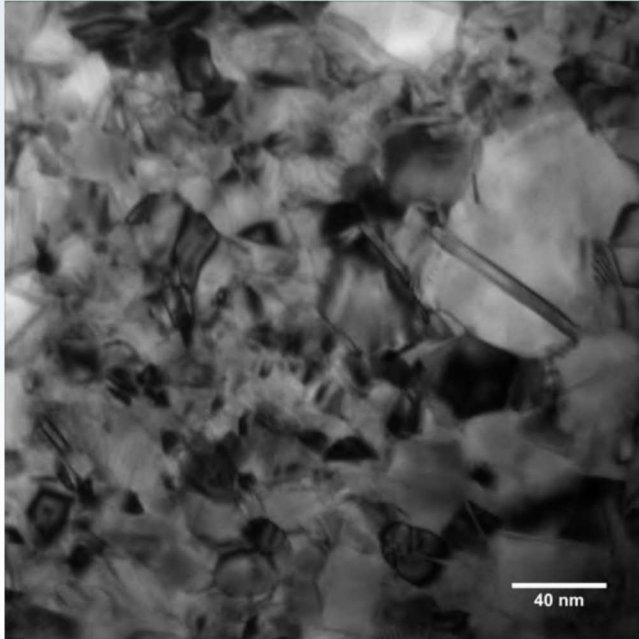
Applied ion beams:

- ▶ 10 keV He^{1+} ion beam implantation was produced by a Colutron G-1 (0.5-10 kV)
- ▶ 1.7 MeV Au^{3+} ion beam was produced by accelerating ions from a source of negative ions by cesium sputtering (SNICS) through a 0.8-6MV HVE EN Tandem accelerator

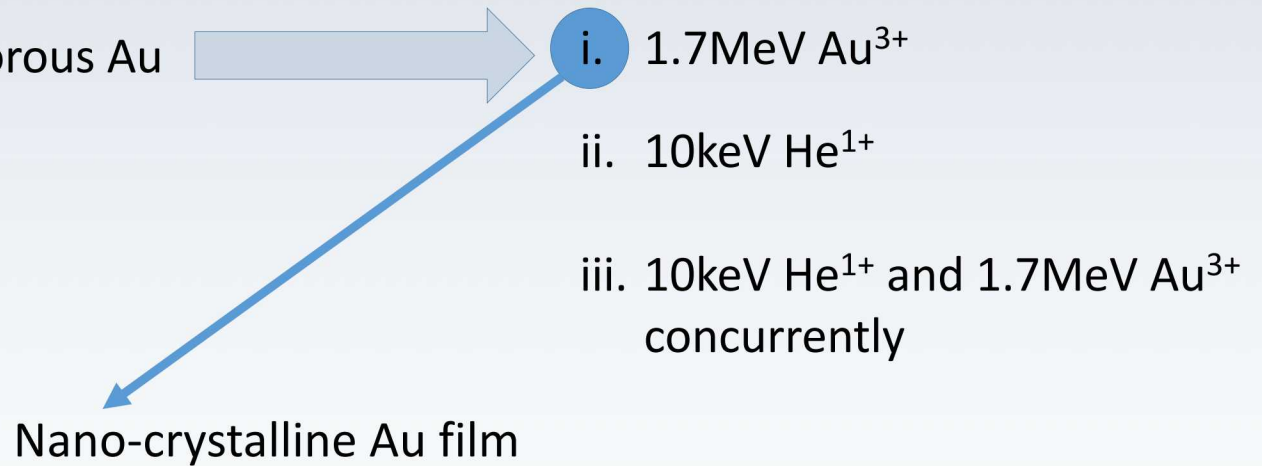


Irradiation with in situ Transmission Electron Microscopy Characterization

Thin film nanoporous Au

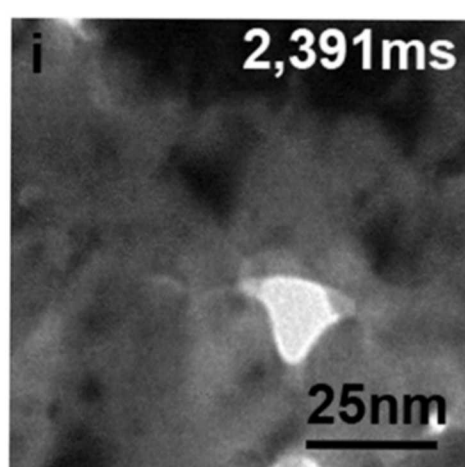
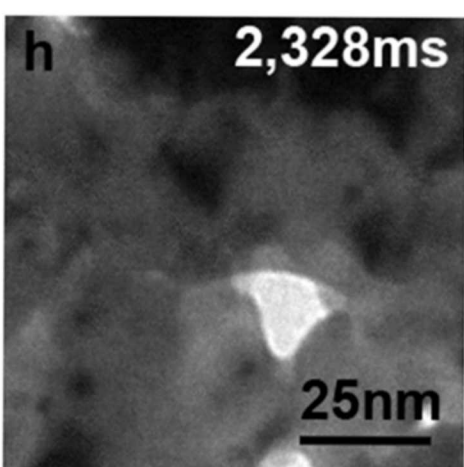
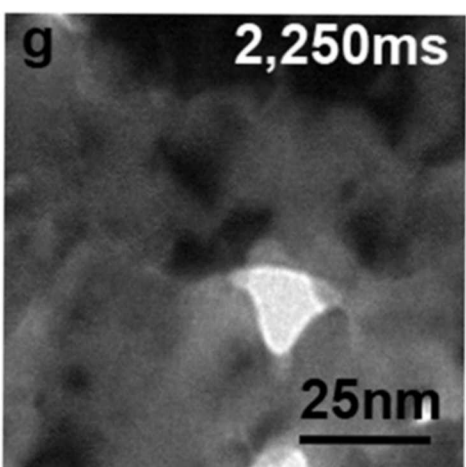
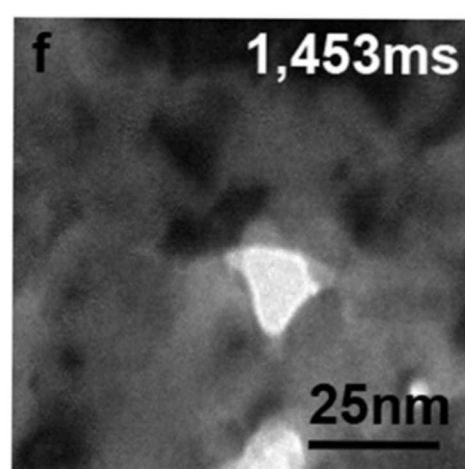
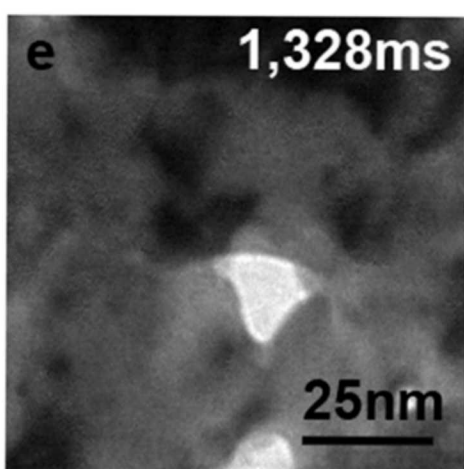
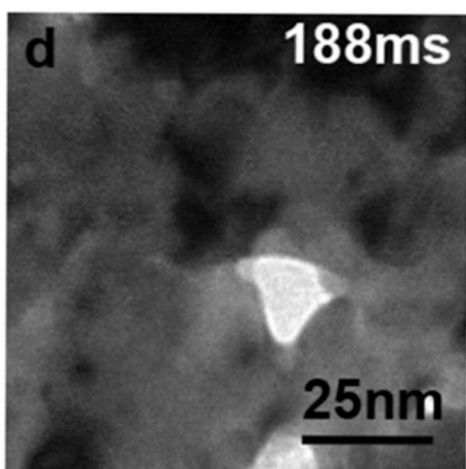
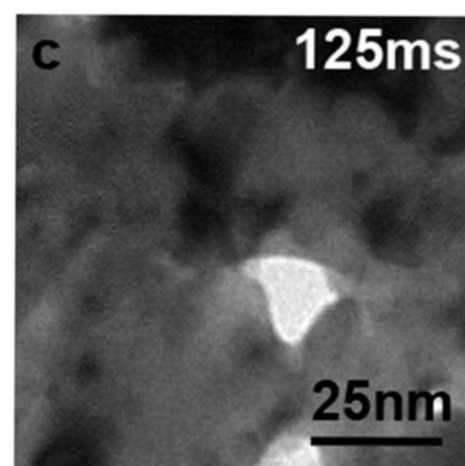
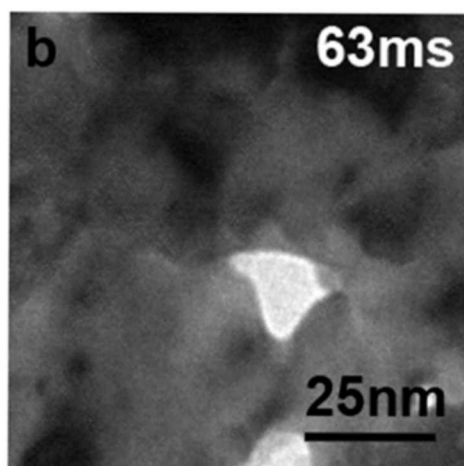
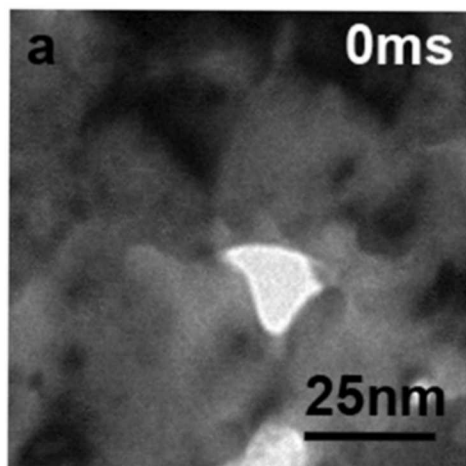


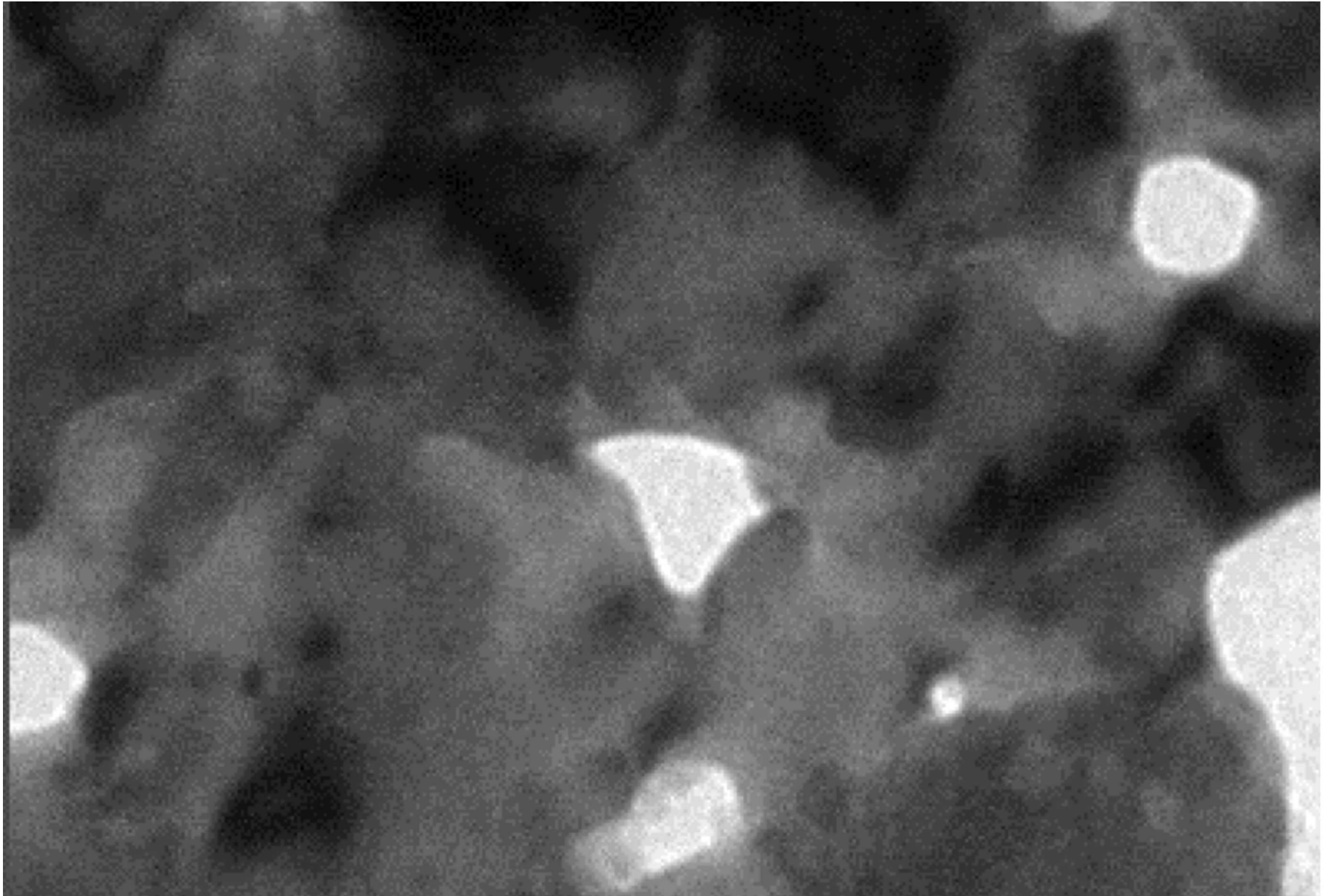
TEM micrograph of as-prepared nc Au



Conditions of in situ irradiation of nanoporous Au

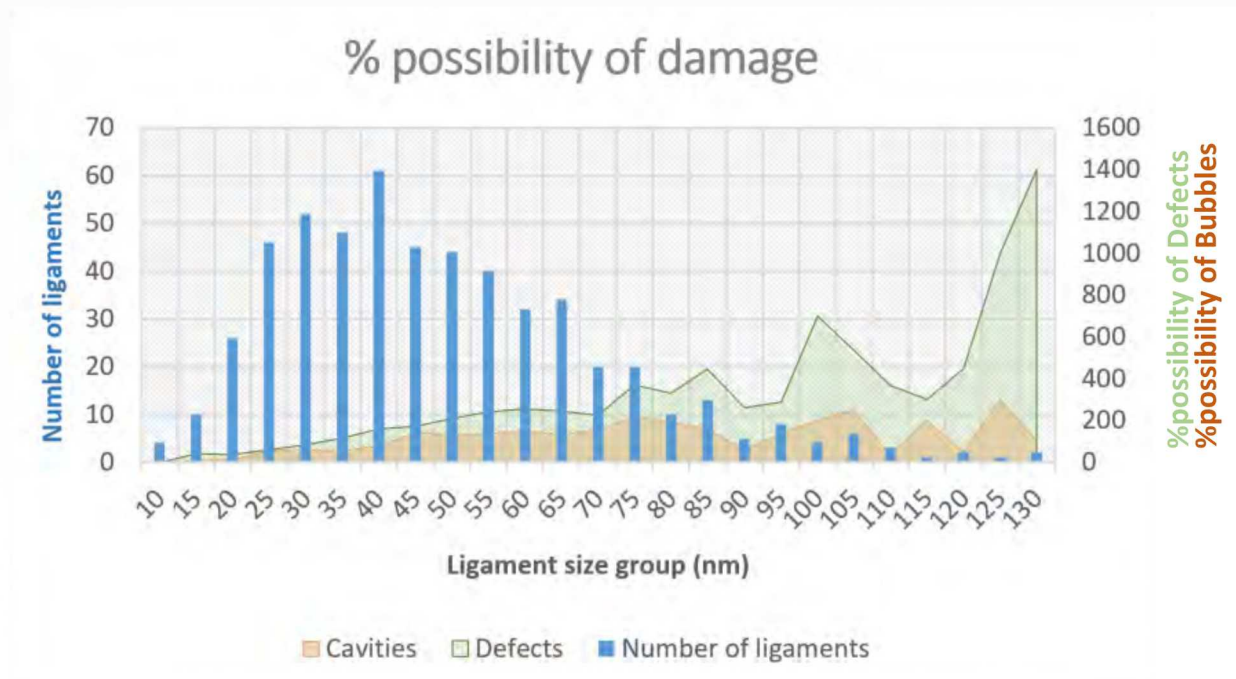
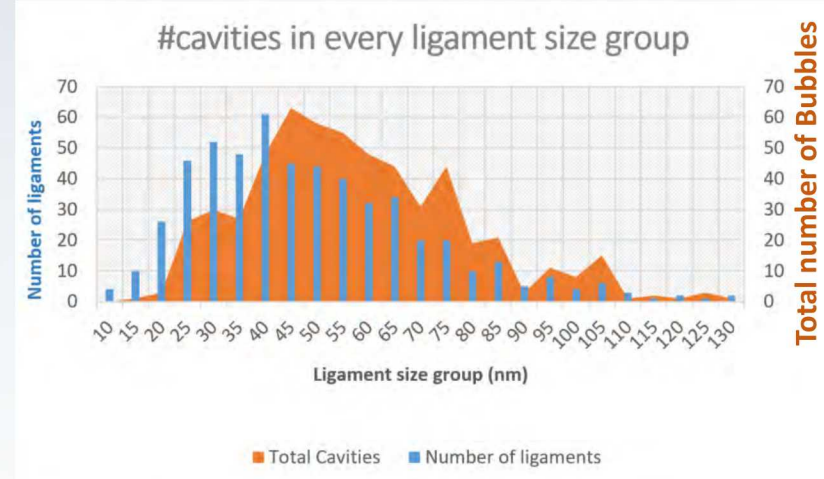
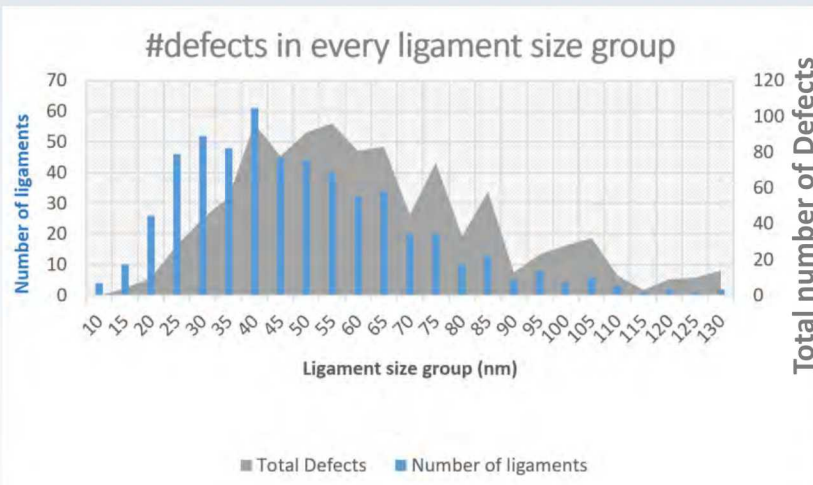
	Ion Energy	Flux (ions / (cm ² ·s))	Fluence (ions/cm ²)
He ⁺ implantation	10keV	4.13 x10 ¹⁴	4.96x10 ¹⁷
Au ³⁺ ion irradiation	1.7MeV	9.53x10 ¹¹	1.14x10 ¹⁵



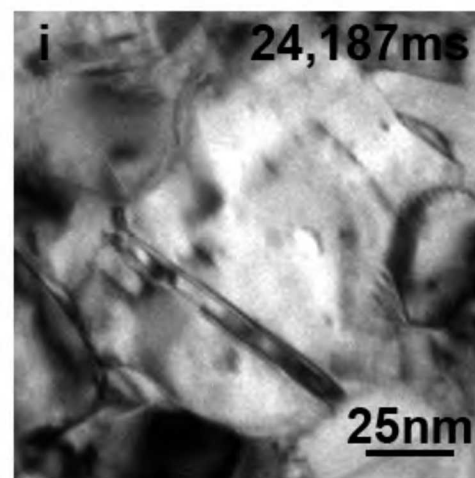
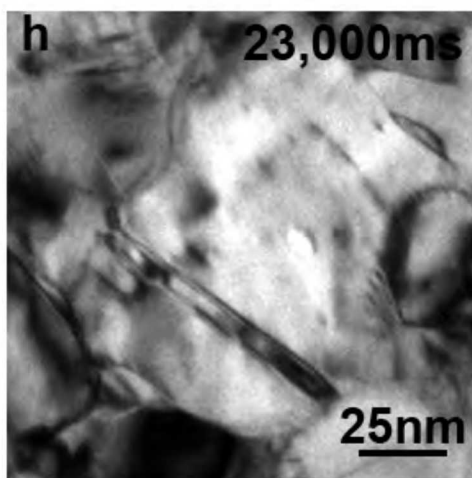
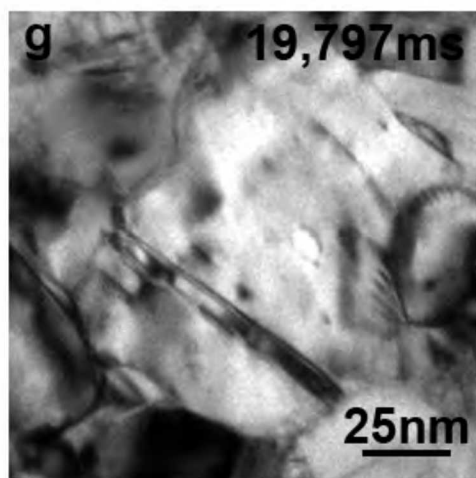
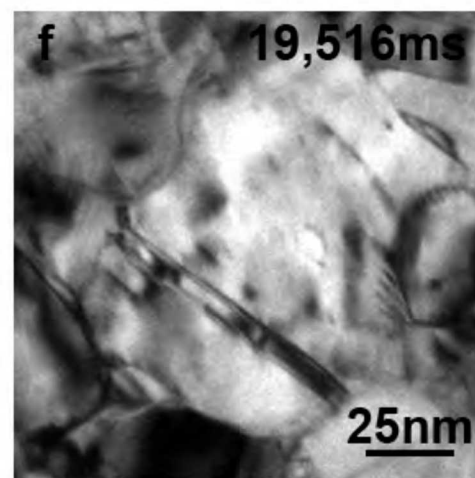
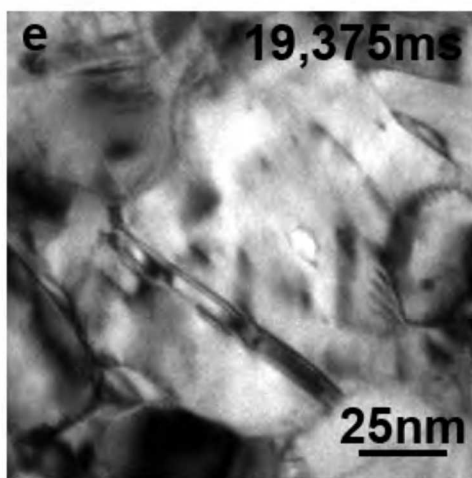
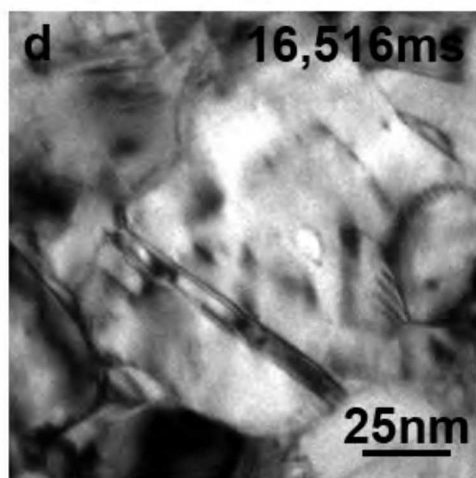
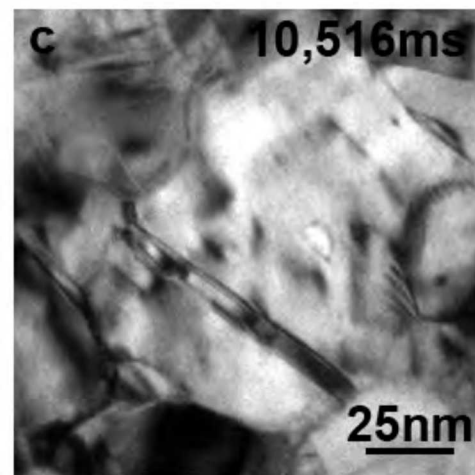
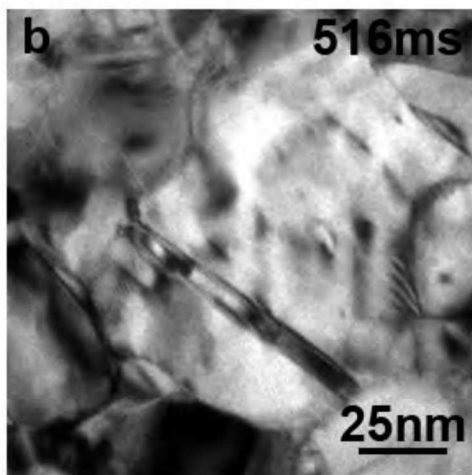
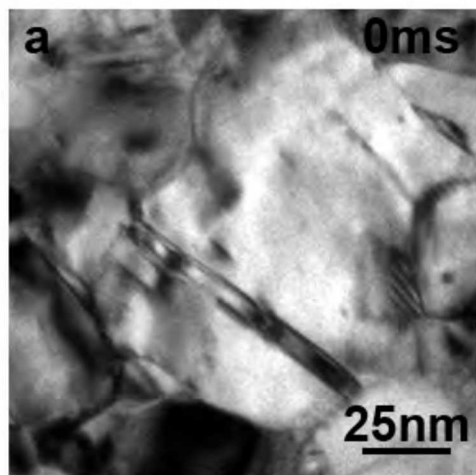


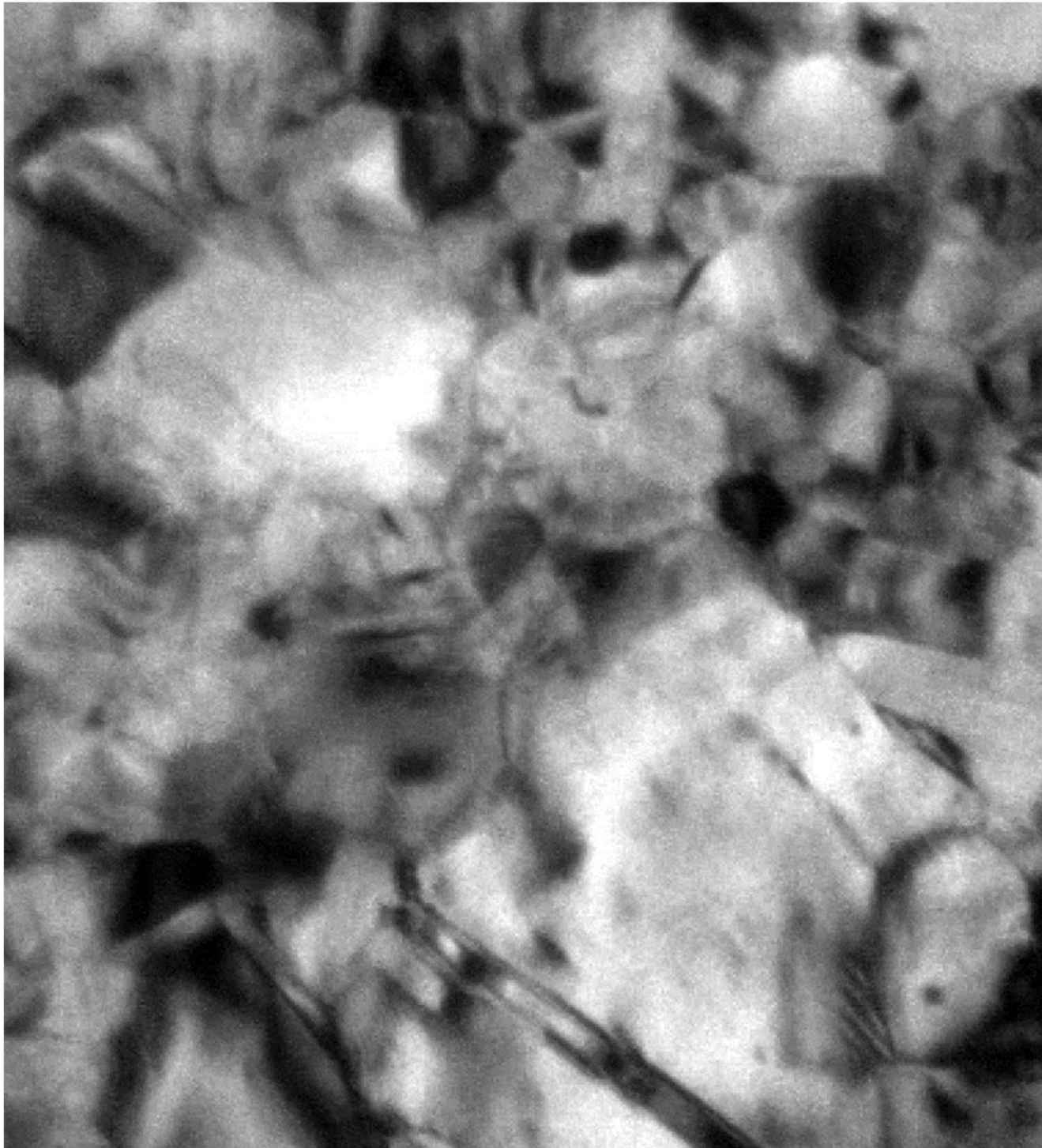
Irradiation with in situ Transmission Electron Microscopy Characterization

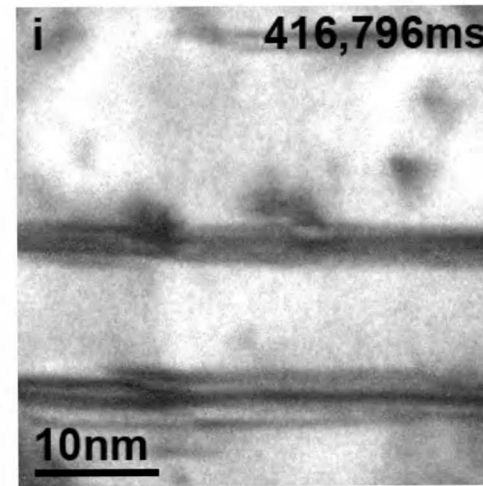
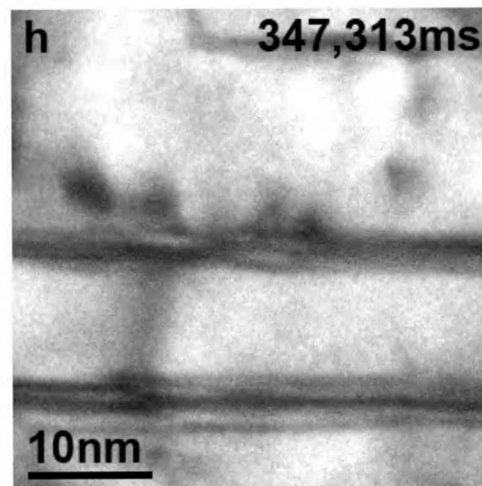
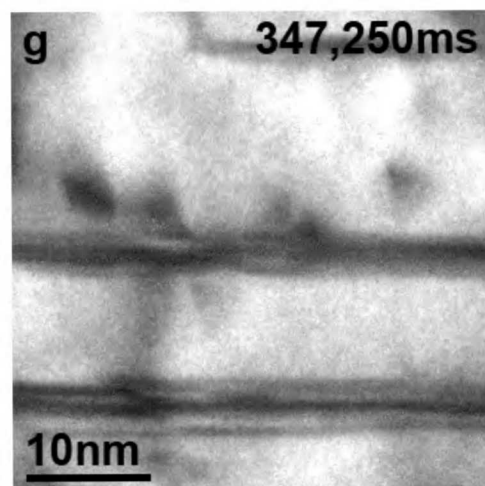
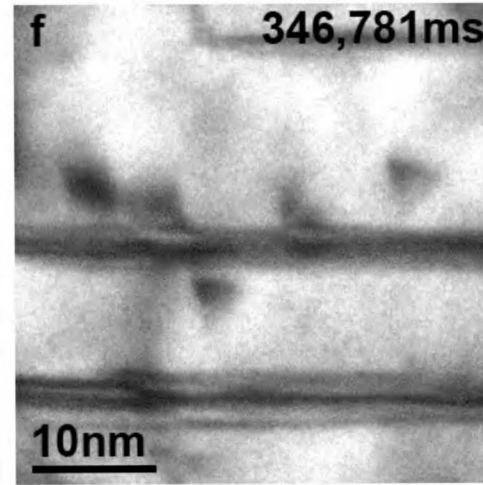
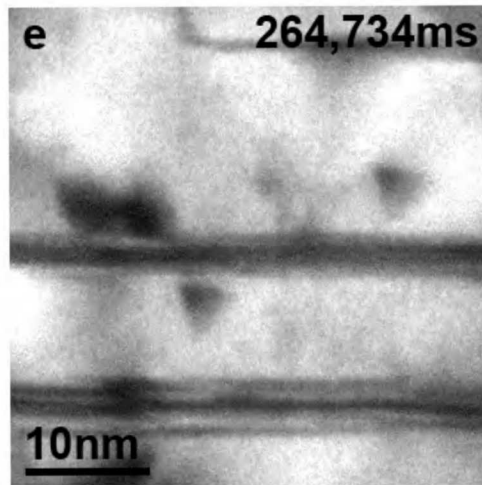
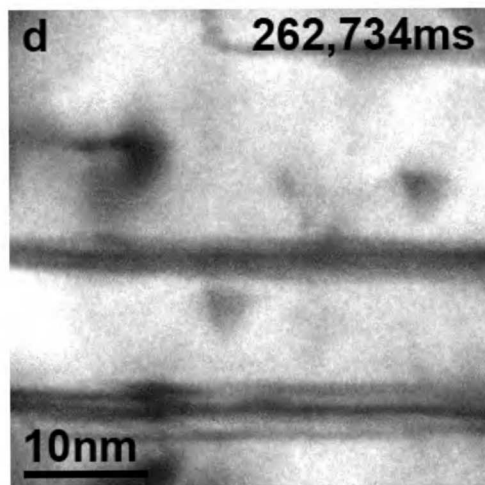
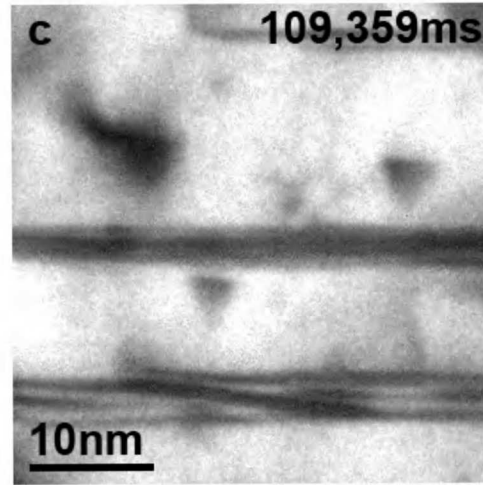
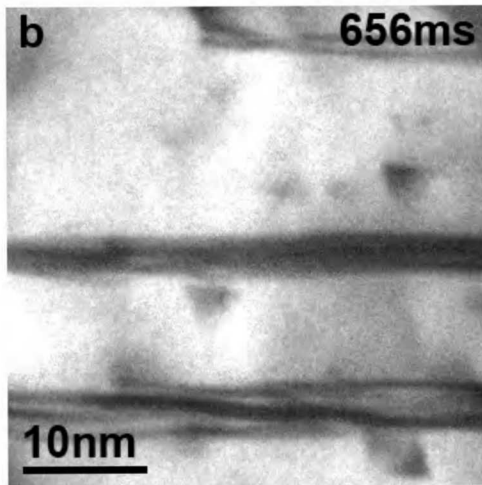
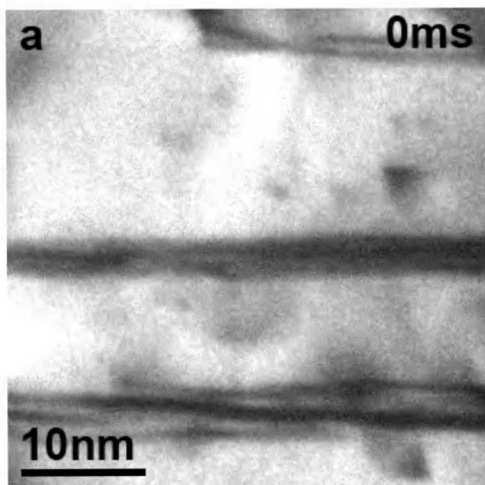
Thin film nanoporous Au \longrightarrow ii. 1.7MeV Au³⁺ for 20minutes



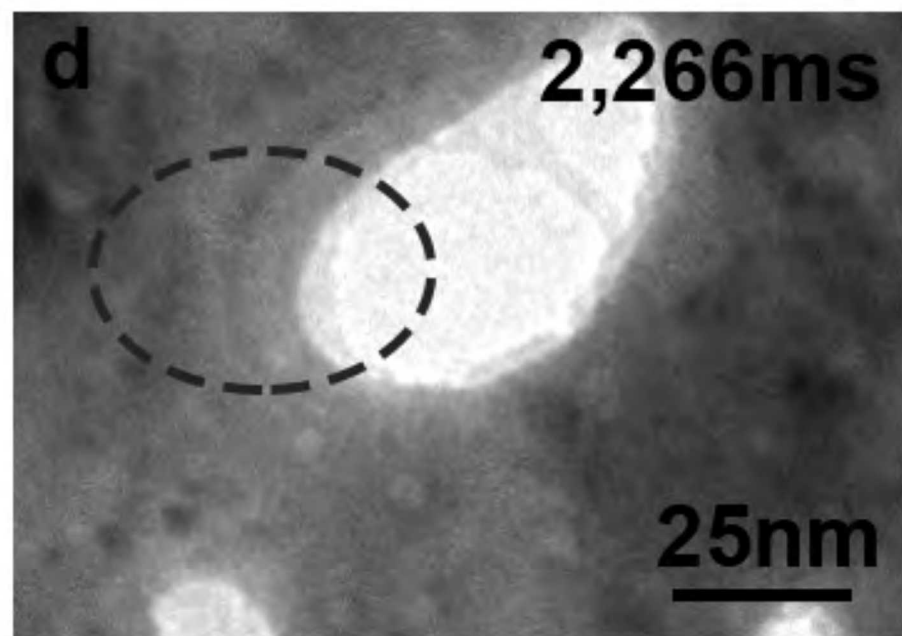
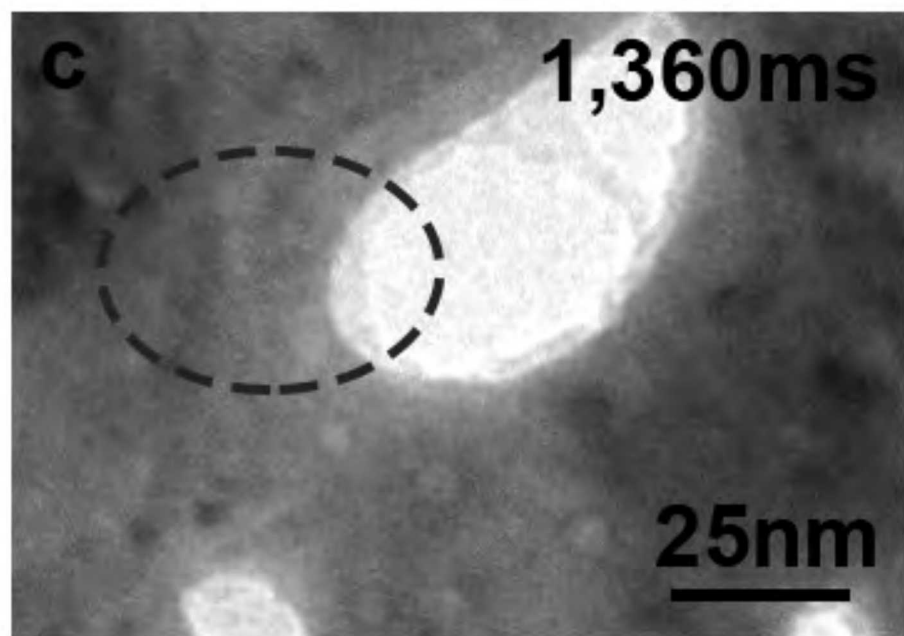
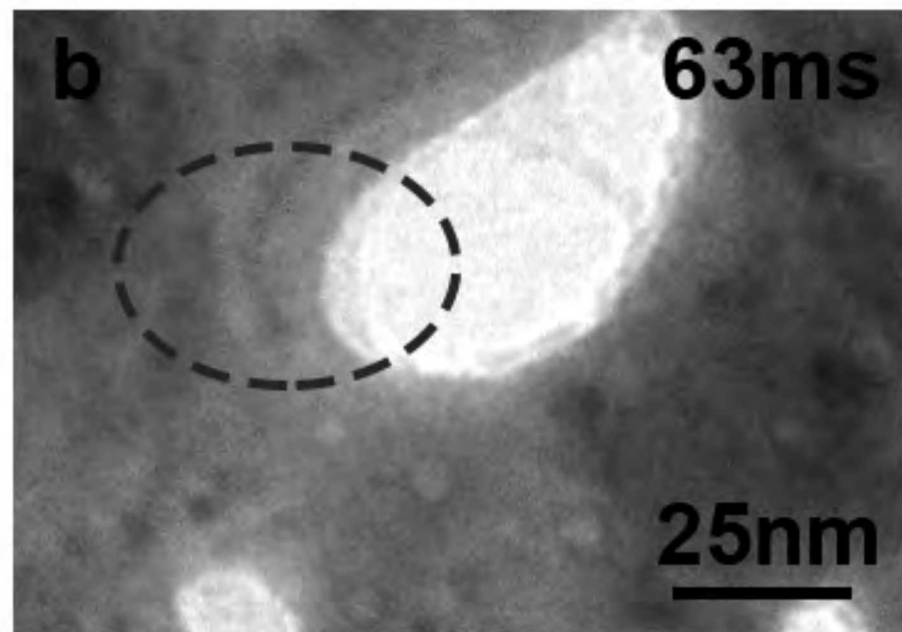
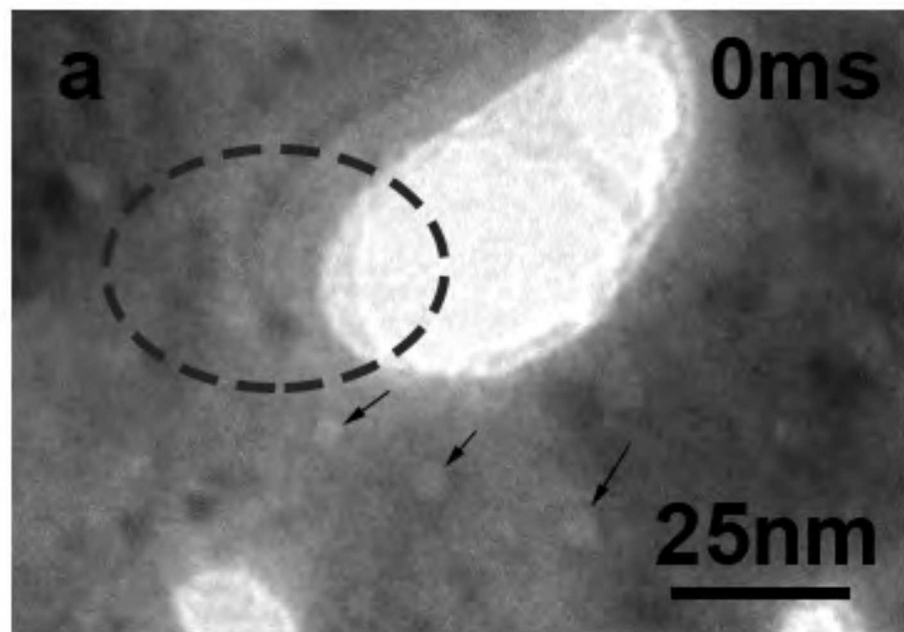
$$\% \text{possibility of damage} = \frac{\text{Total number of damage}}{\text{Number of ligaments}} * 100$$

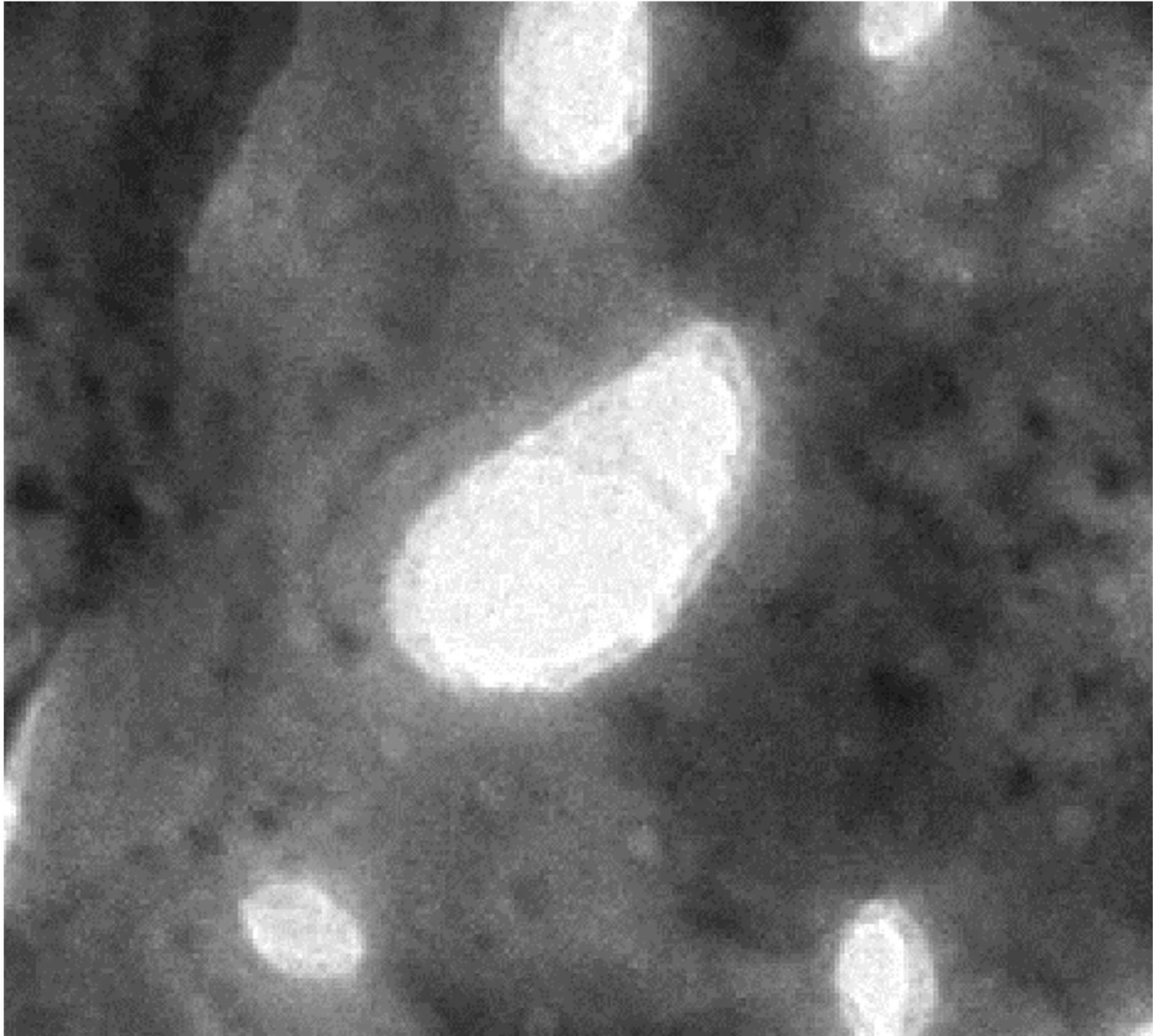






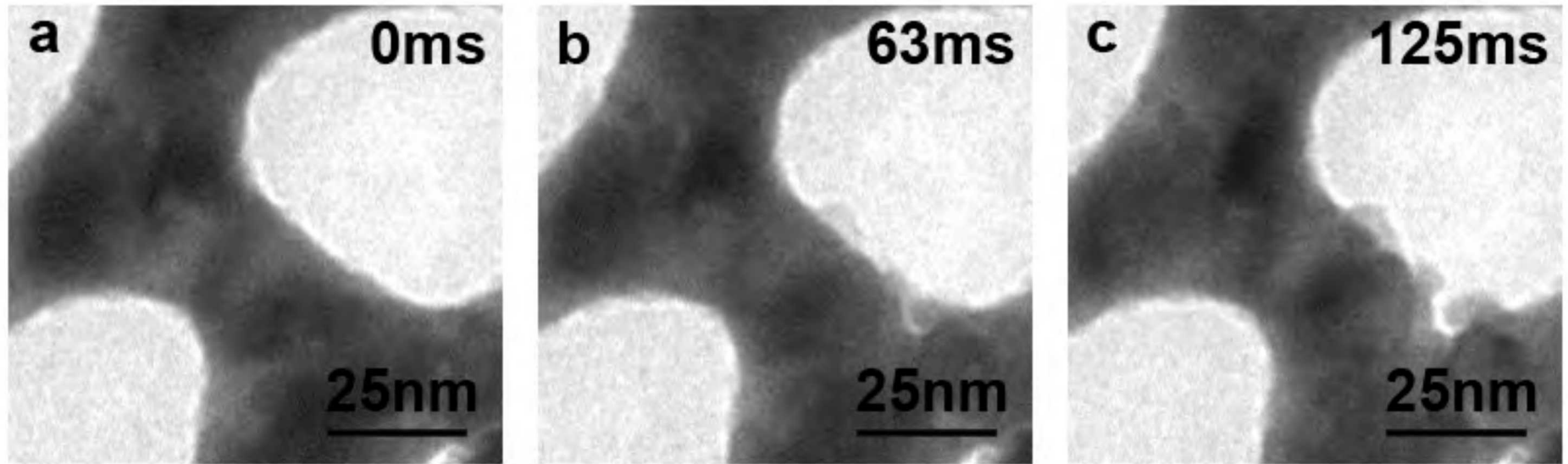




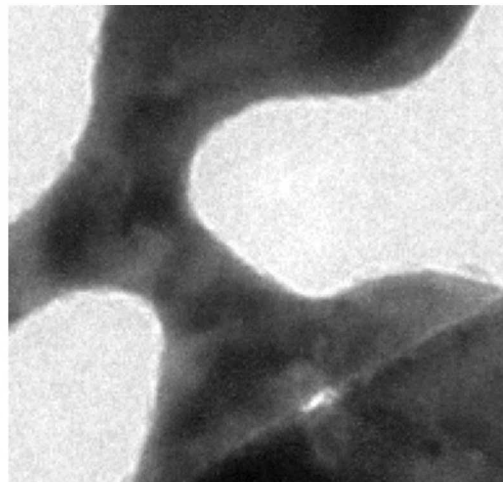


Irradiation with in situ Transmission Electron Microscopy Characterization

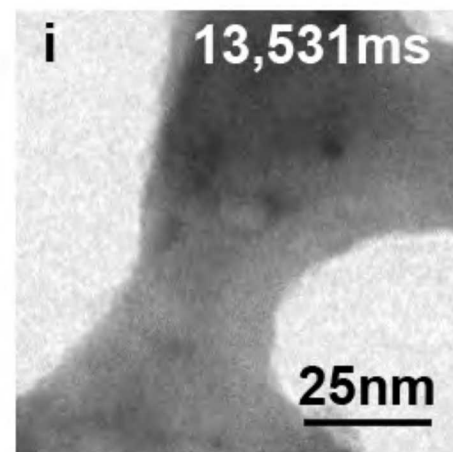
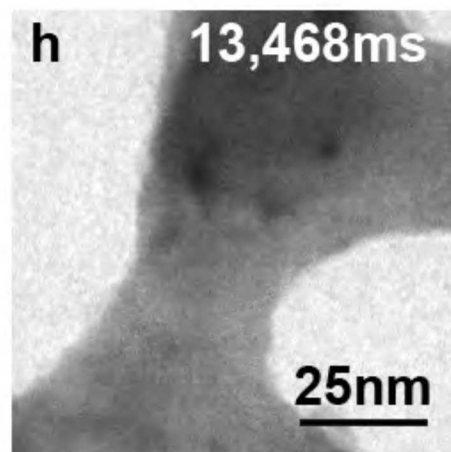
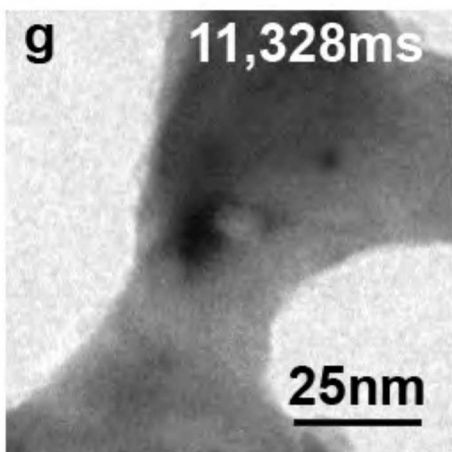
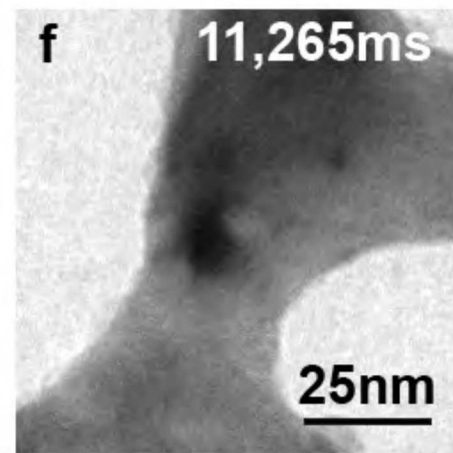
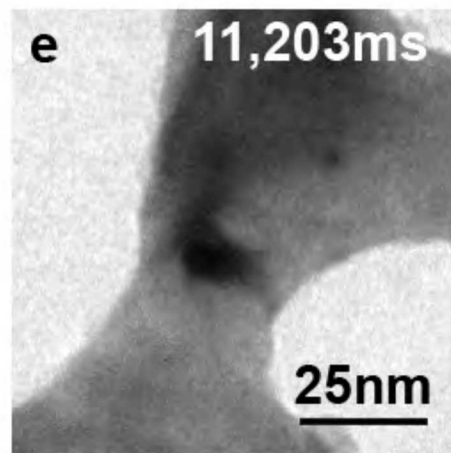
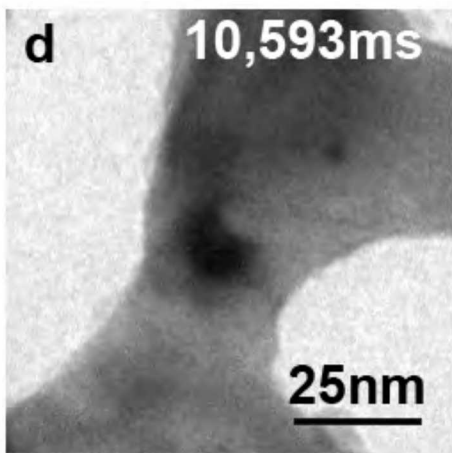
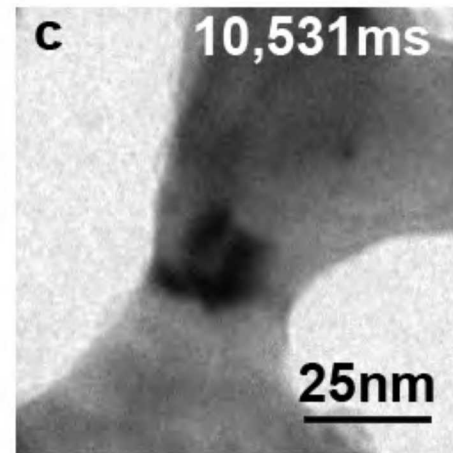
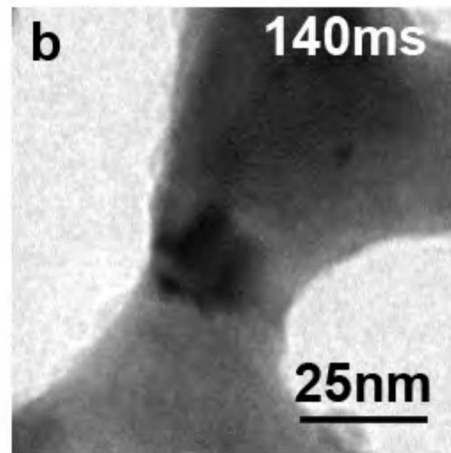
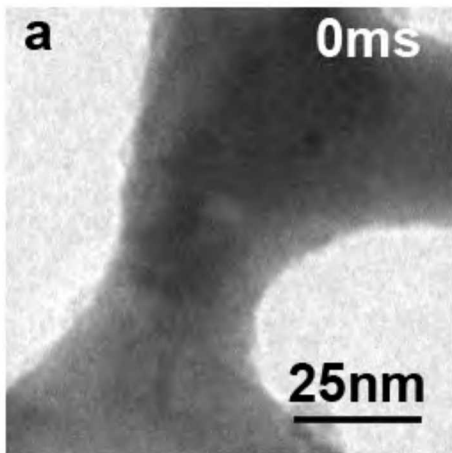
Thin film nanoporous Au \longrightarrow iii. 10keV He¹⁺ and 1.7MeV Au³⁺ for 30minutes

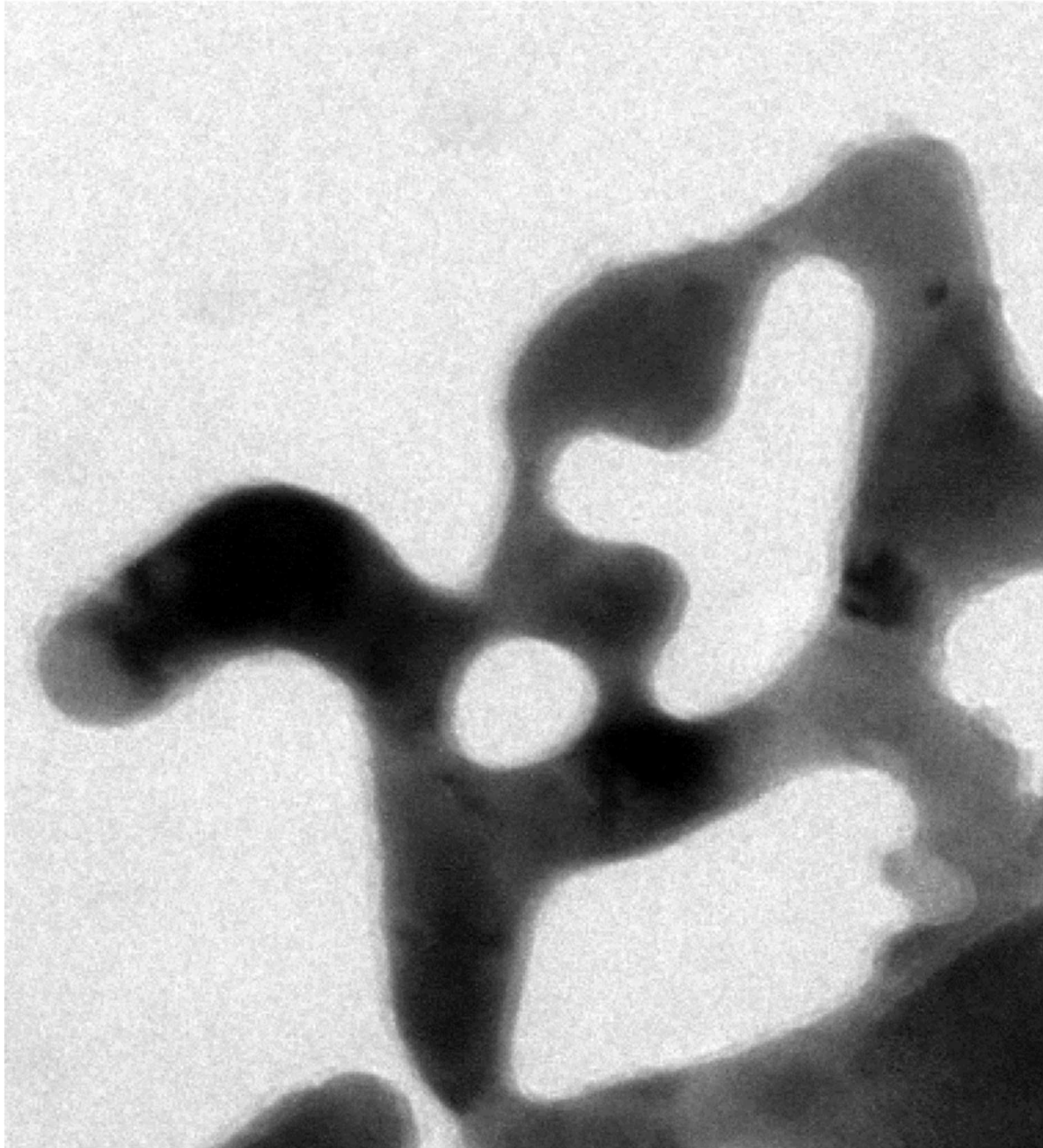


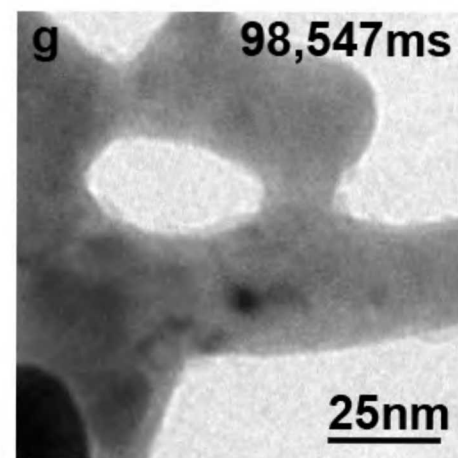
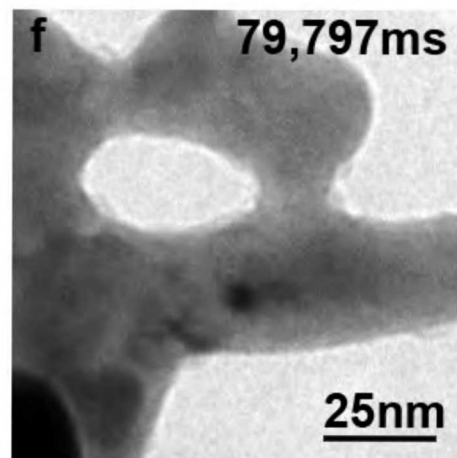
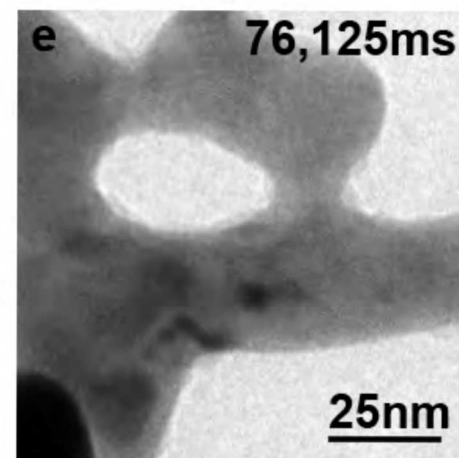
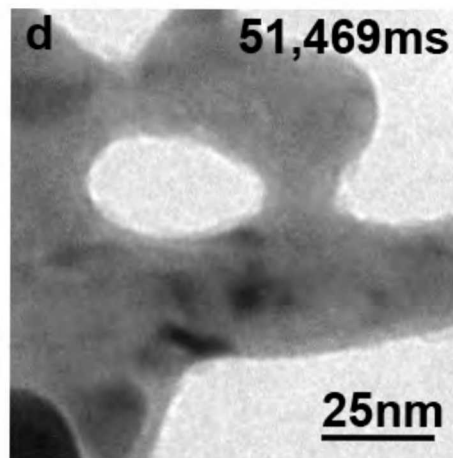
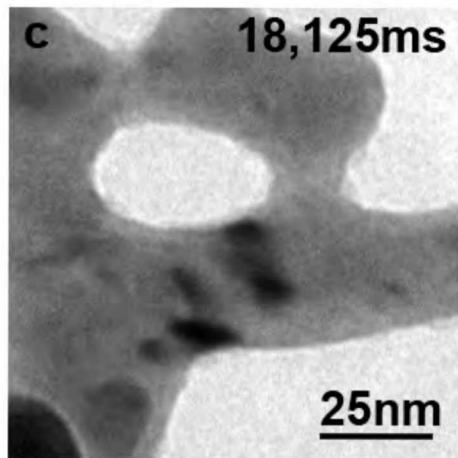
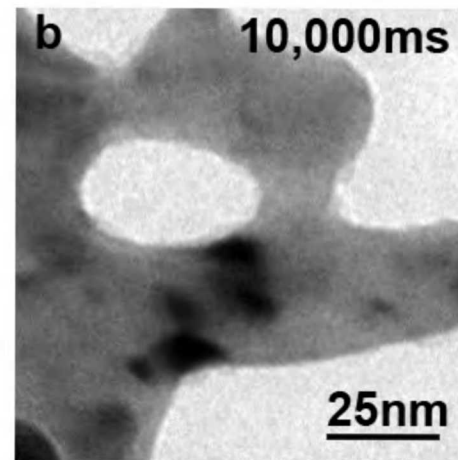
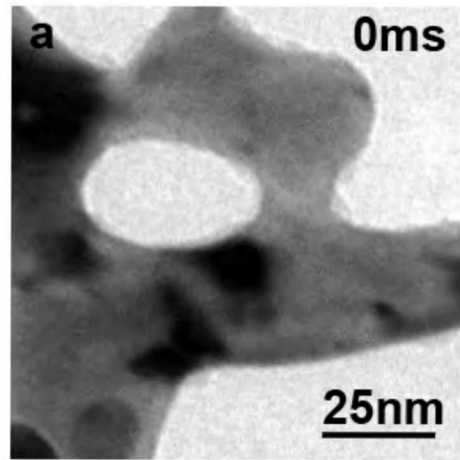
TEM micrographs demonstrate sputtering effect close to a node in 125m (current fluence $9.14 \cdot 10^{13}$ ions/cm²)



TEM video of nanoporous Au with recording time 1minute and 35.969seconds up to 1minute and 36.094seconds











Conclusion and discussion

- Summary:

1. Preparation of nanoporous Au
2. In situ TEM radiation on thin films of nanoporous Au with 10keV He⁺ and 1.7MeV Au³⁺ individually and concurrently

- Results:

1. Light ions with low energy and high dose rate ($4.13 \cdot 10^{14}$ ions·cm⁻²·sec⁻¹) result in:
 - i. doping effect (stable He bubbles)
 - ii. pore sink effect
2. Self-ion irradiation with high energy and high dose rate ($9.53 \cdot 10^{11}$ ions·cm⁻²·sec⁻¹) result in:
 - ▶ pore sink effect (nanoporous Au)  defect clusters: 2.391'' (relative required time)
 - ▶ twin boundary crossing effect (nanocrystalline Au)  defect clusters: 23.671''
SFT: 3' and 46.594''
3. Concurrent He⁺ ion implantation and Au³⁺ ion irradiation result in:
 - i. pore sink effect
 - ii. He bubble sink effect
 - iii. sputtering effect

- Further Research:

1. Ion irradiation on nanoporous Au with light and self ions in sequence (and vice versa)
2. Ion irradiation on nanoporous Nb (vary ion species, energies, ion doses and applied temperature)
3. Neutron irradiation on nanoporous Nb

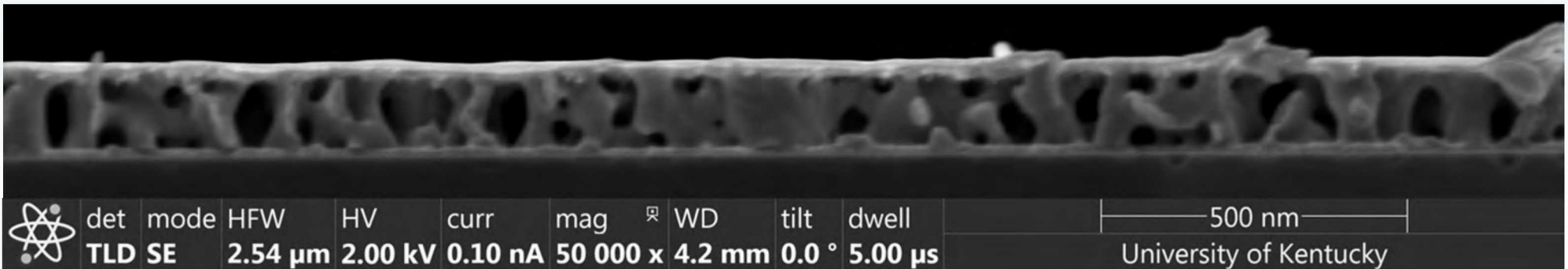
Thermal dealloying technique

Sputtering of refractory metal with Mg

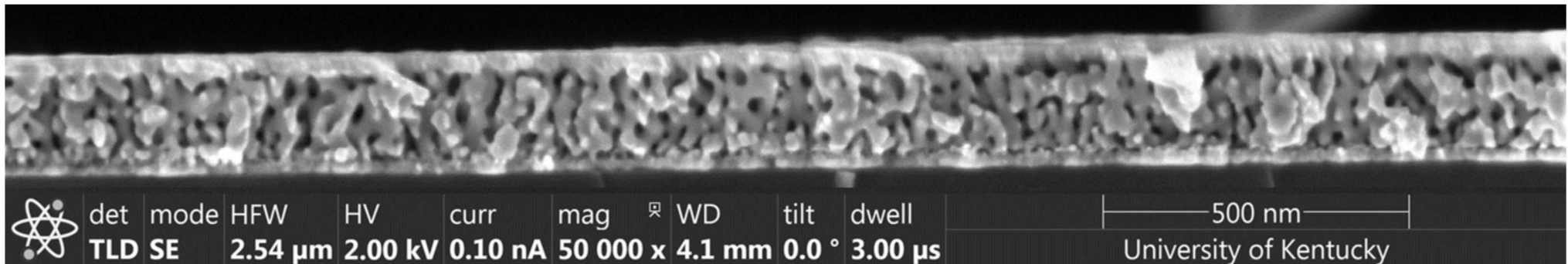
Thermal dealloying at 600°C for 2 hours

Nanoporous structure of refractory metal

Nanoporous Niobium:

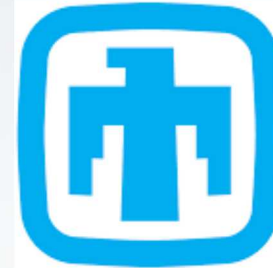


Nanoporous Tantalum:





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National
Laboratories**

This work was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science.

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