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Cavity Formation During *In Situ* He^{+} Ion Implantation and Heating

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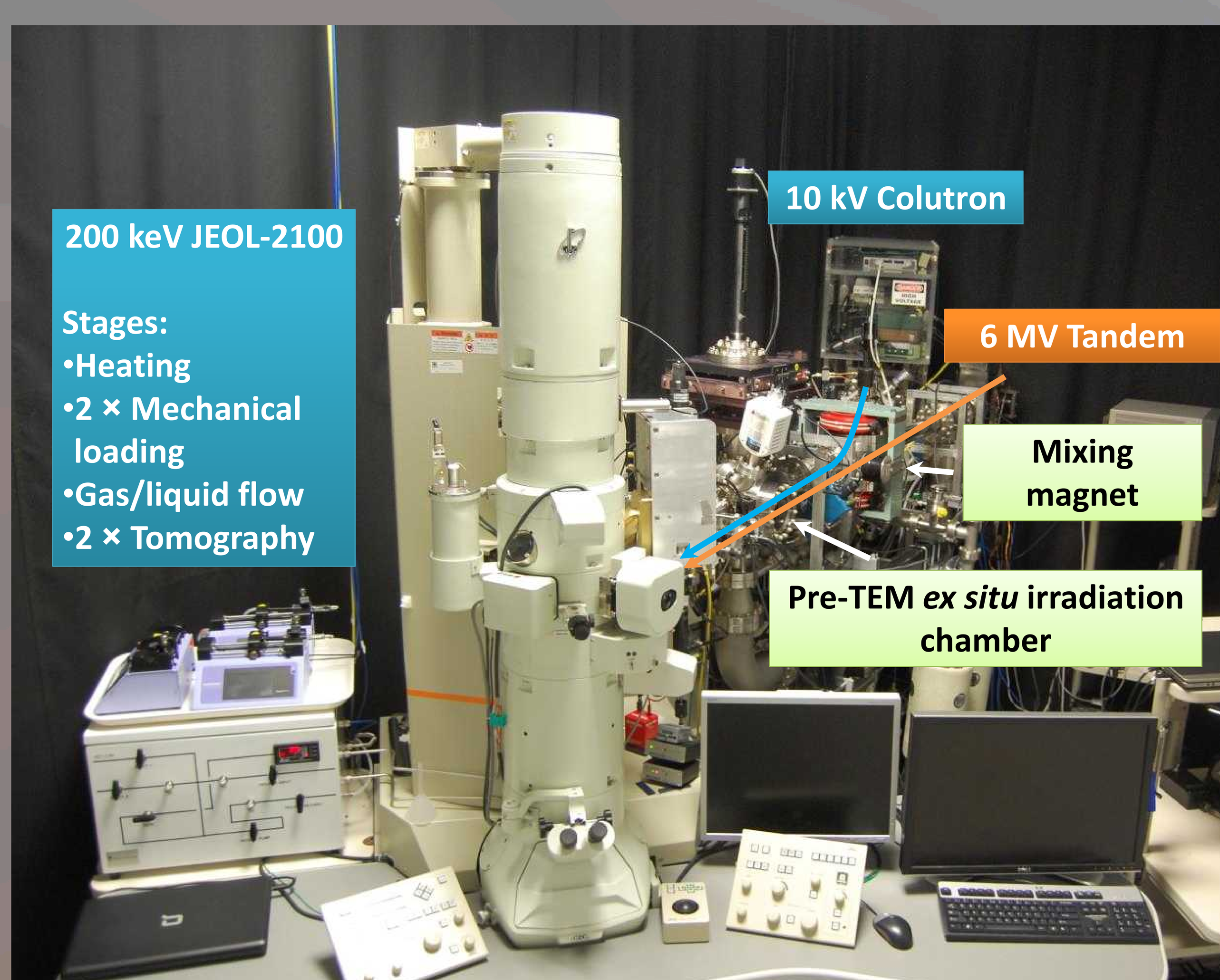
U.S. DEPARTMENT OF ENERGY

Motivation

- Metal hydrides are interesting materials for solid state hydrogen isotope storage
- Tritium decay produces ^{3}He, which may form bubbles within the storage material
- Ion implantation provides a means to study He accumulation effects more quickly

The Microscope



I³TEM Facility

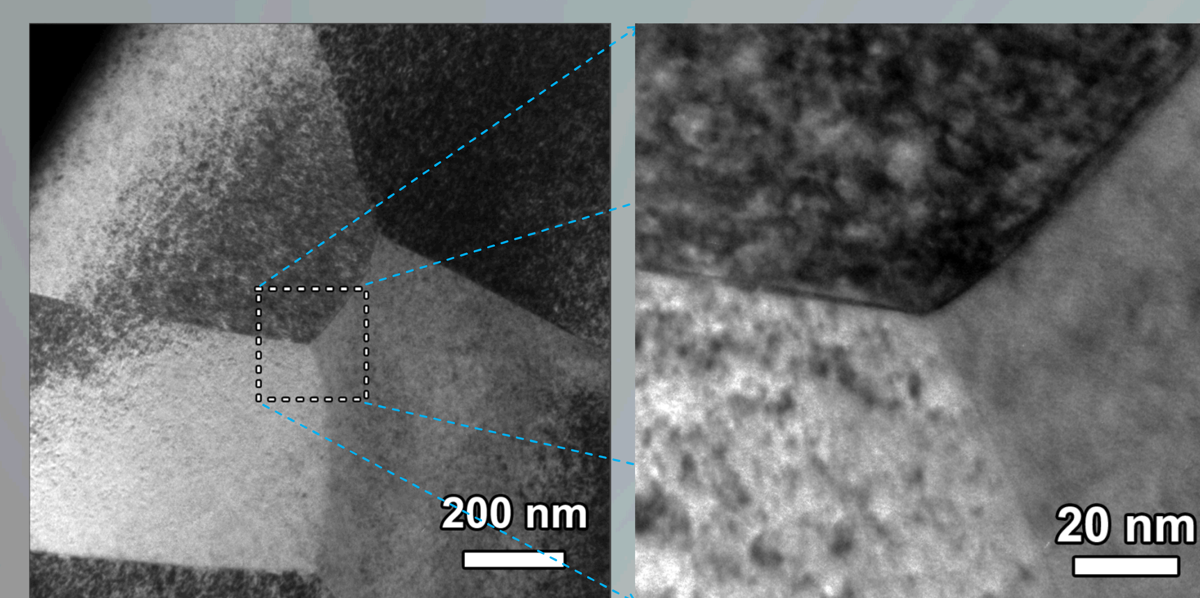
- TEM equipped with two attached ion accelerators and a variety of *in situ* sample manipulation capabilities

Goals and Approach

Study the differences in bubble/cavity formation induced by He in Er and ErD_2

- *In situ* He^{+} implantation and annealing in the TEM

Before: ErD_2



FIB lift-outs from bulk material (ErD_2 pictured)

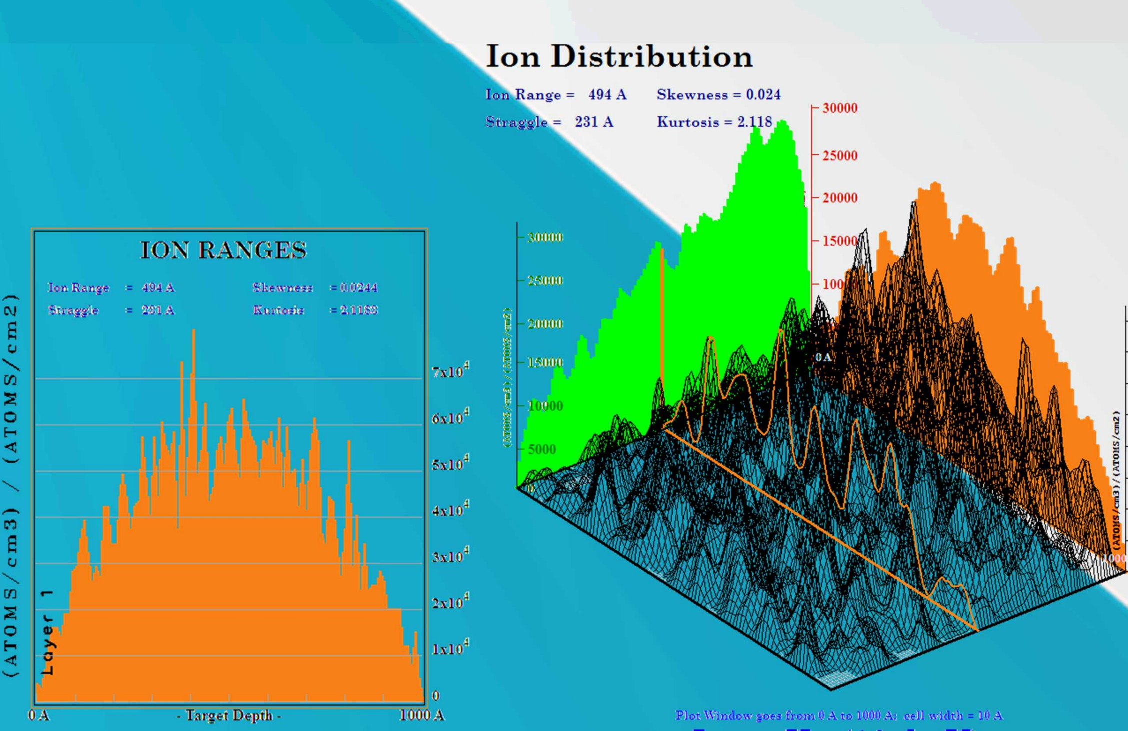
- Samples on α-carbon/Cu grids
- Triple junction selected for observation

He^{+} Implantation: ErD_2

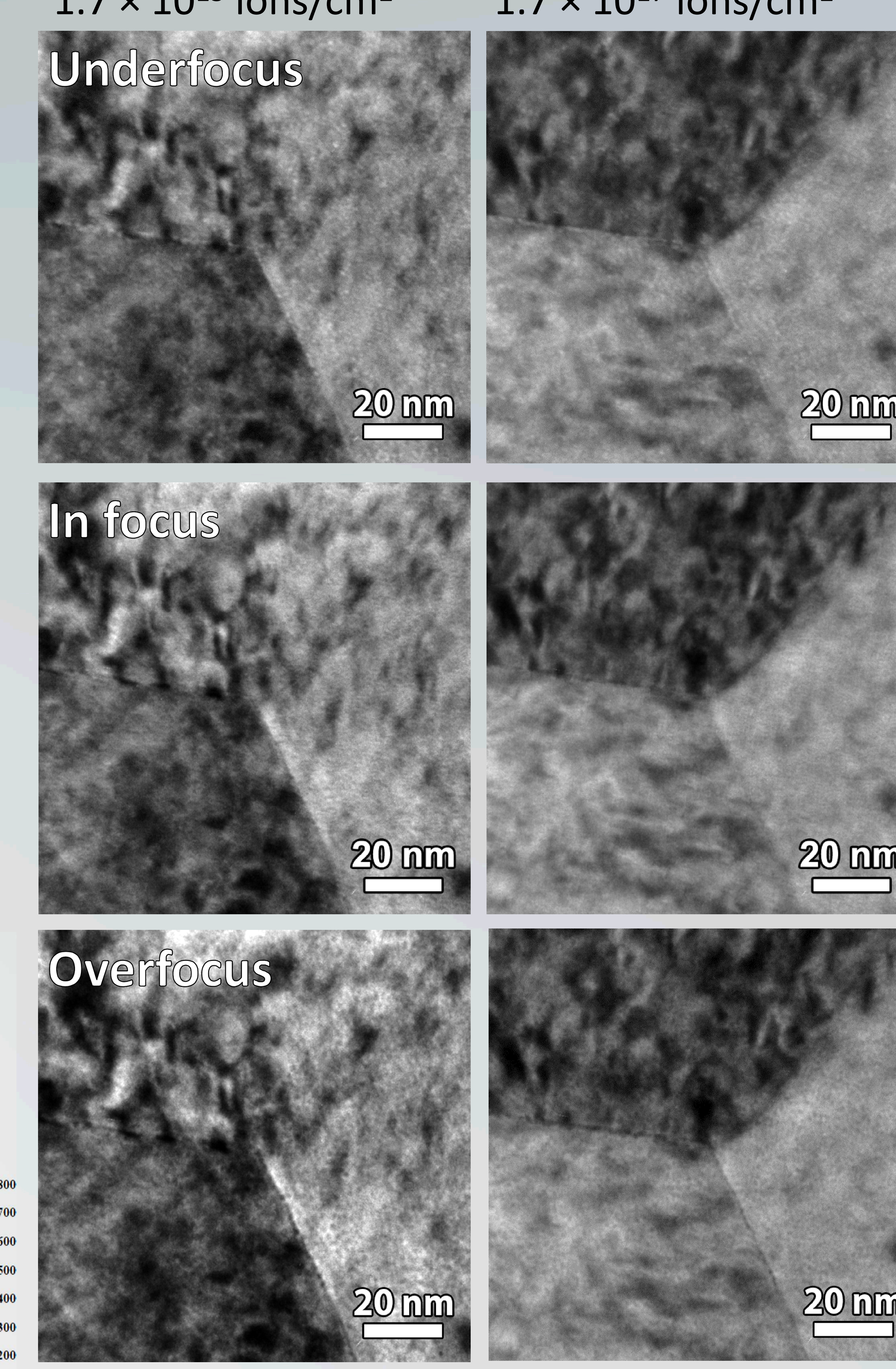
Simulation and *in situ* experiments

TRIM Simulation

- 10 keV He ions into 100 nm Er layer
  - 49% backscattered
  - 11% transmitted
  - 40% remain in sample
- Significant vacancy production
  - 26 vacancies/ion

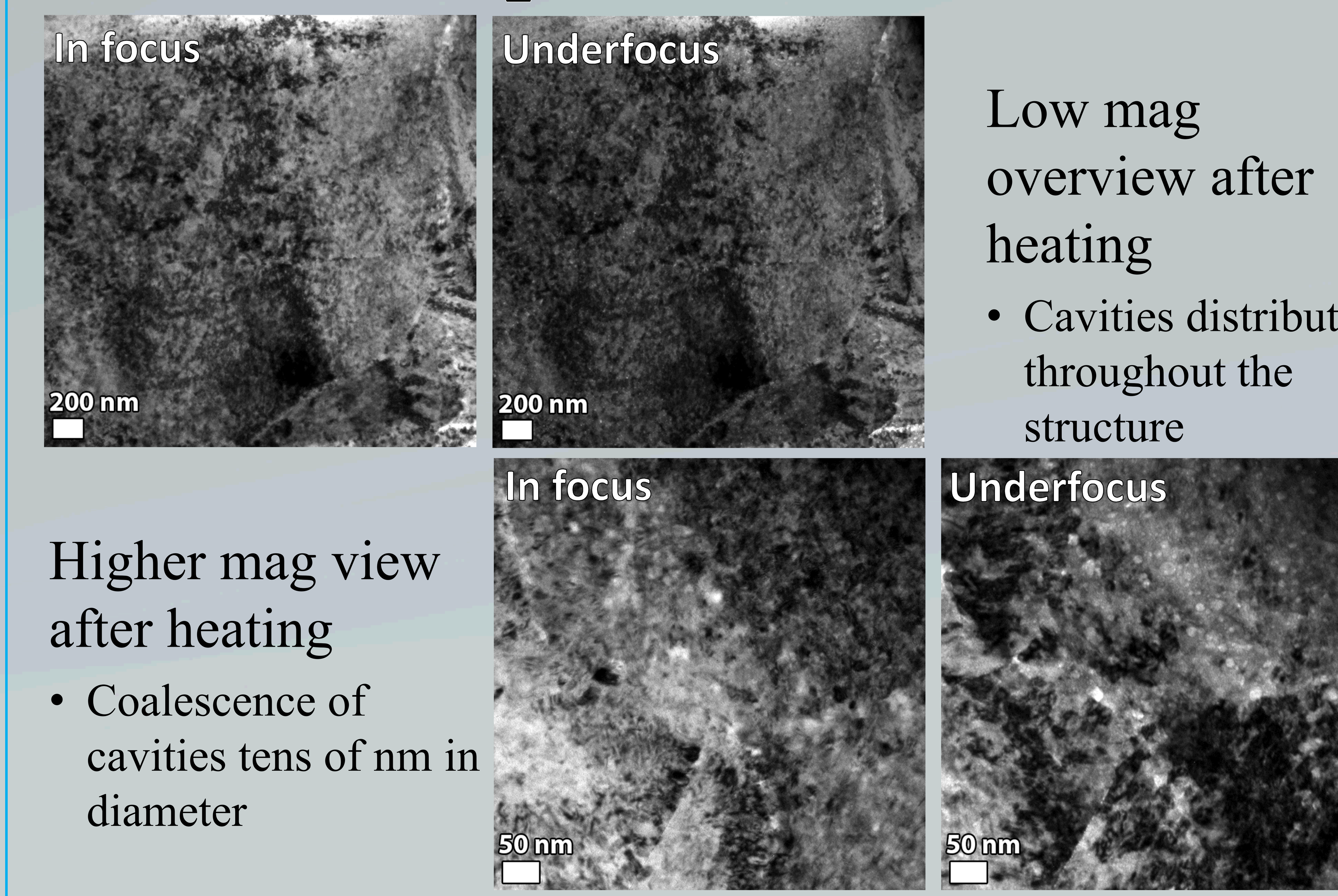


Simulated ion distributions plotted in 2D and 3D



- Through-focus imaging reveals small cavities (vacancy or He-filled)
- Cavities appeared rapidly
- Little growth over time

Heating: ErD_2 *In situ* annealing to 785 °C



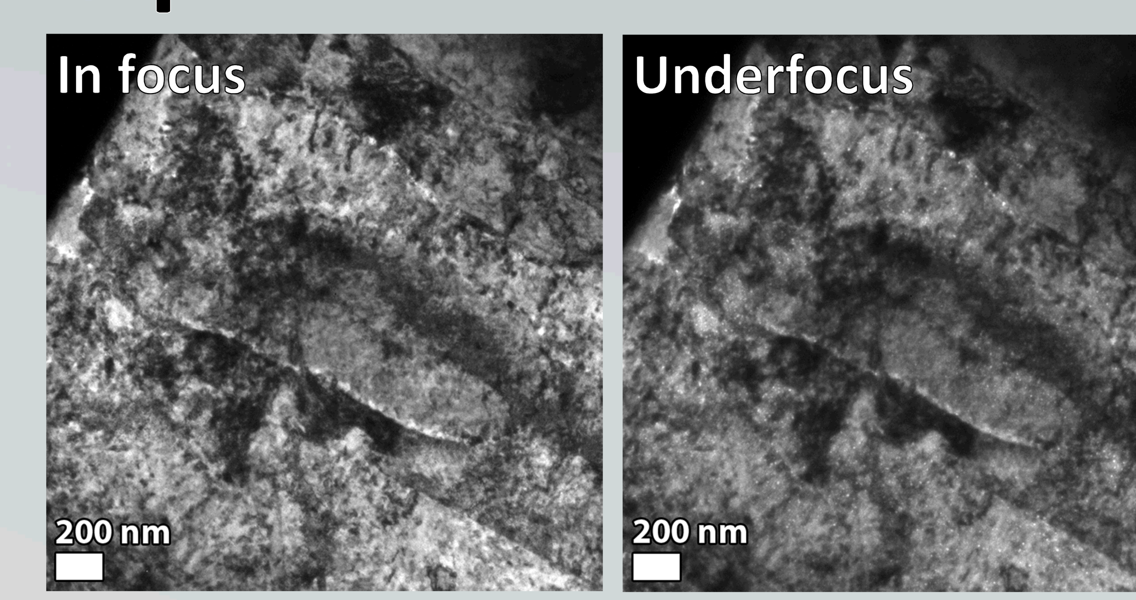
Low mag overview after heating

- Cavities distributed throughout the structure

Higher mag view after heating

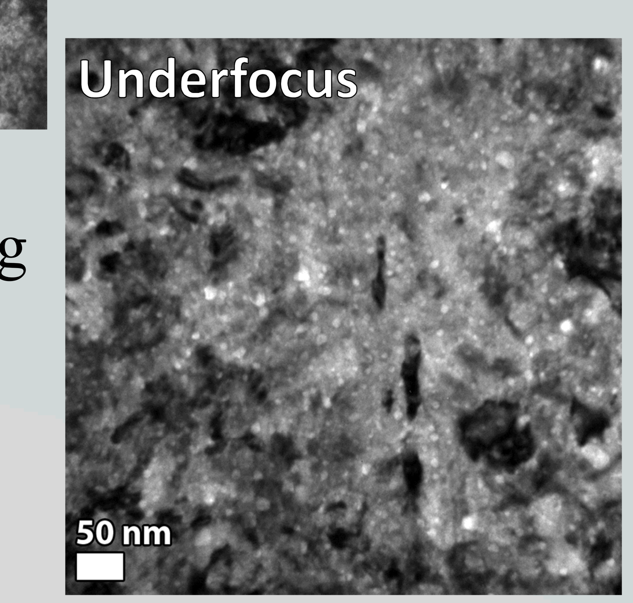
- Coalescence of cavities tens of nm in diameter

Implantation and Heating: Er



Low mag overview after heating

- Fine bubbles throughout the structure



Higher mag view after heating

- Typical cavity size smaller than in ErD_2

Conclusions

- Cavity nucleation was induced using *in situ* ion irradiation
- Cavity coarsening was observed during subsequent *in situ* heating experiments
- Smaller cavity size in Er

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