SAND2018-12895PE

Comparison of Helium Aging in Ion Implanted and Tritium Loaded Metals

PRESENTED BY

Caitlin Taylor

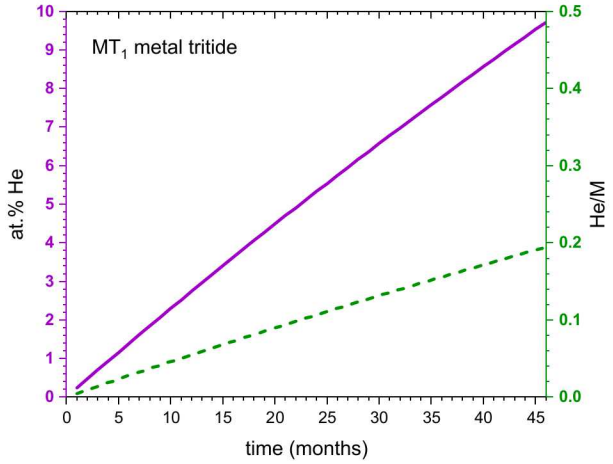
SNL-NM: Brittany Muntifering, Khalid Hattar

SNL-CA: Josh Sugar, Dave Robinson, Noelle Catarineu, Warren York

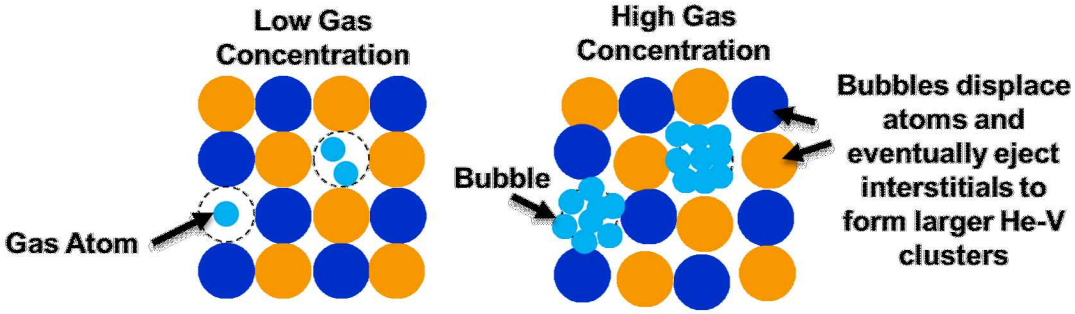
Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia LLC, a wholly owned subsidiary of Honeywell International Inc. for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

Solid-State Tritium Storage Materials undergo Helium Accumulation

- Typically metal hydrides
- Tritium β-decays with a half-life of 12.3 years, releasing ^{3}He into the lattice.

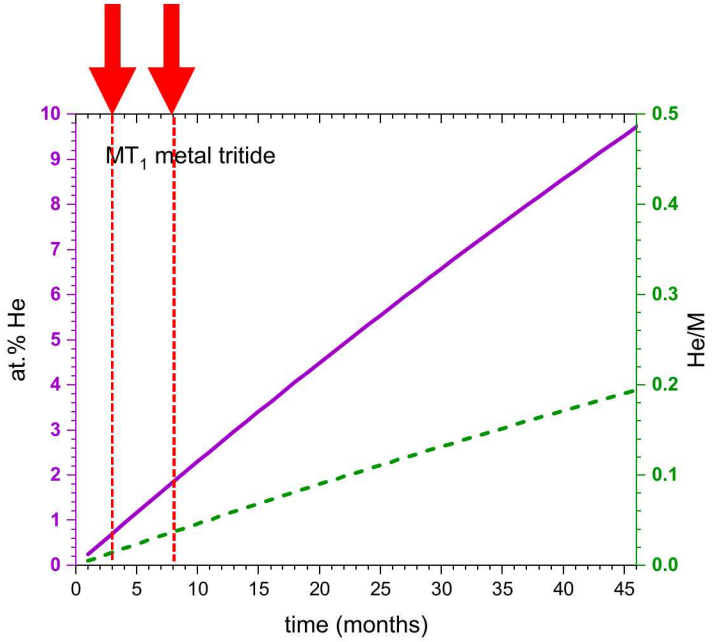


- He accumulates into bubbles in the lattice

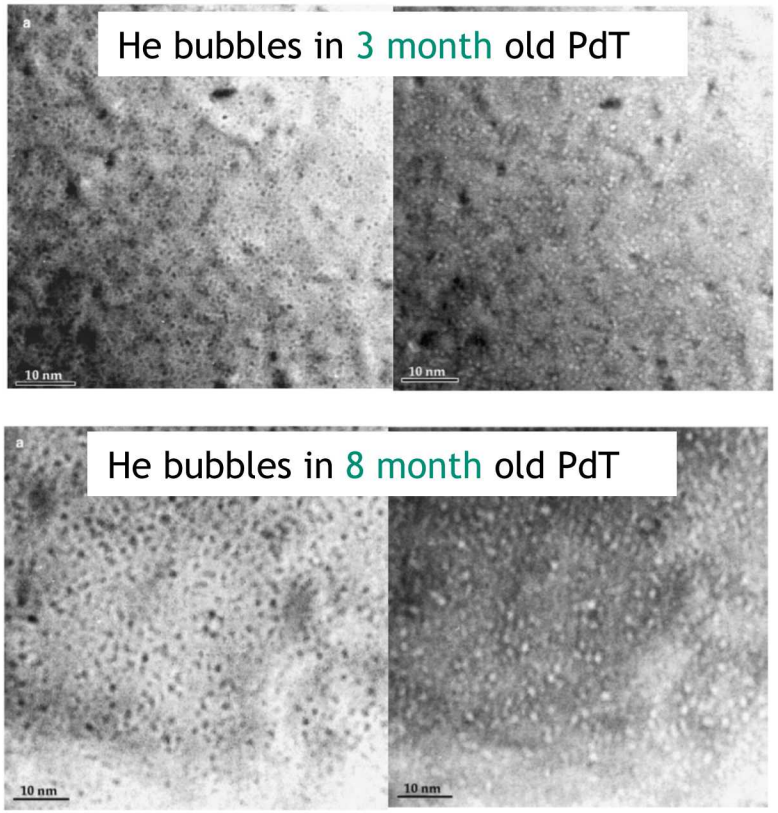


- Knowledge of bubble nucleation, growth, and He release mechanisms are required for predictive aging models. Most of these mechanisms are not well understood.

Helium Bubble Formation Occurs Rapidly in Tritides

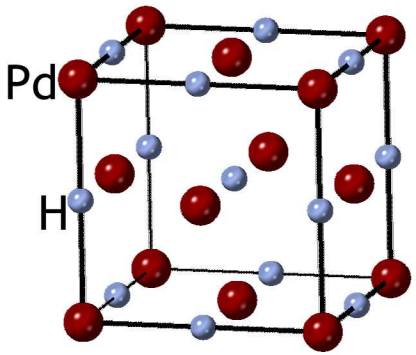


Fabre et al, JNM 342 (2005) 101-107



- Hydrogen resides in the octahedral sites in PdH, causing a slight lattice expansion

PdH is not fully stoichiometric. Hydrides close to $PdH_{0.6}$.

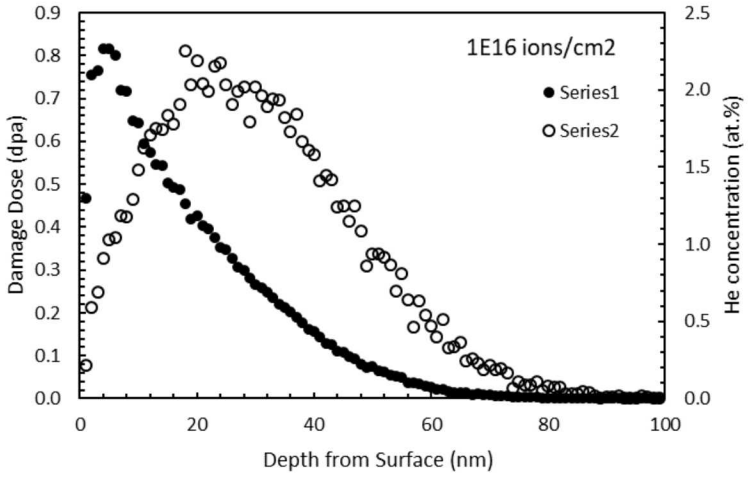


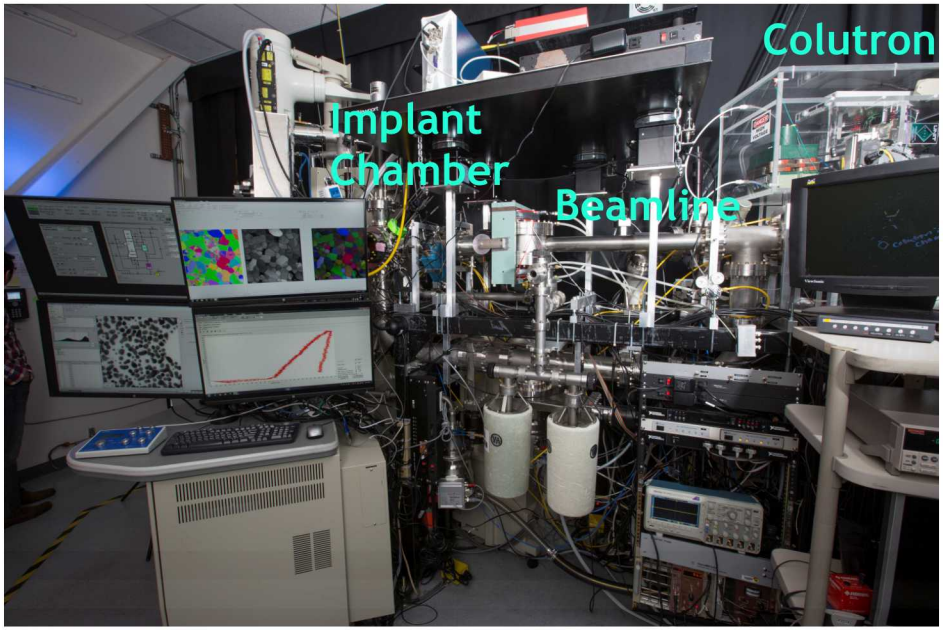
- Tritium β-decays to ^3He, which likely either remains in the octahedral site, or moves to the tetrahedral site

Different Microstructures are Expected from He Accumulation from to ^{3}H Decay vs. He Implantation into Metals

- Helium bubble formation in palladium was compared between ion implanted and tritium aged material
- Tritium β-decay induces no displacement damage; the ^{3}He is released with a maximum energy of 3.4 eV and the β-particle has a maximum energy of 18.6 keV. → Takes months to reach a considerable He concentration
- Helium implantation results in displacement damage except at very low energies:
  - <280 eV for Pd → Takes hours to reach a considerable He concentration
- All samples were annealed in-situ to accelerate bubble growth

SRIM profile for He in Pd





Helium Implantation and Thermal Aging of Palladium

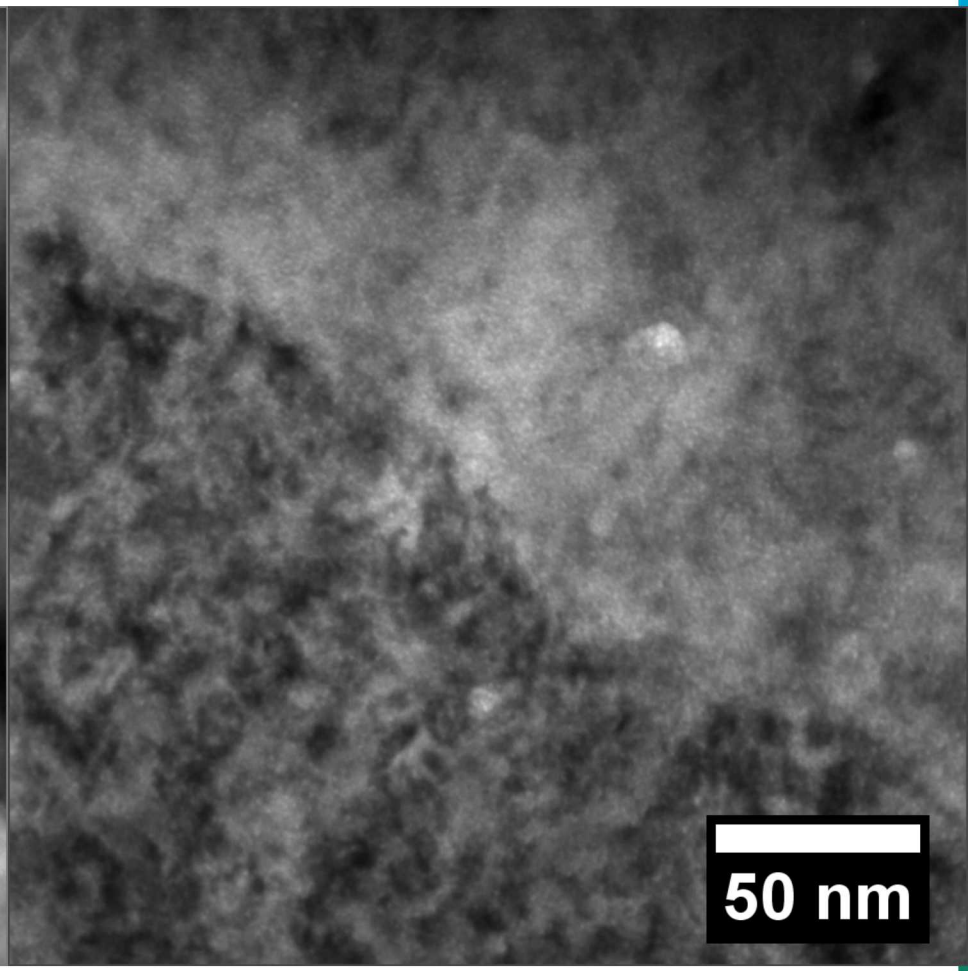
- Helium was implanted at three temperatures: 25, 250, and 400ºC
- Different microstructures were observed depending on defect mobility
- Each implanted sample was annealed in-situ to accelerate bubble growth

Helium implantation into palladium resulted in temperature dependent microstructures

25ºC, $T/T_m = 0.16$

Before He implantation

After He implantation to ~6.9×10^{16} He/cm^2

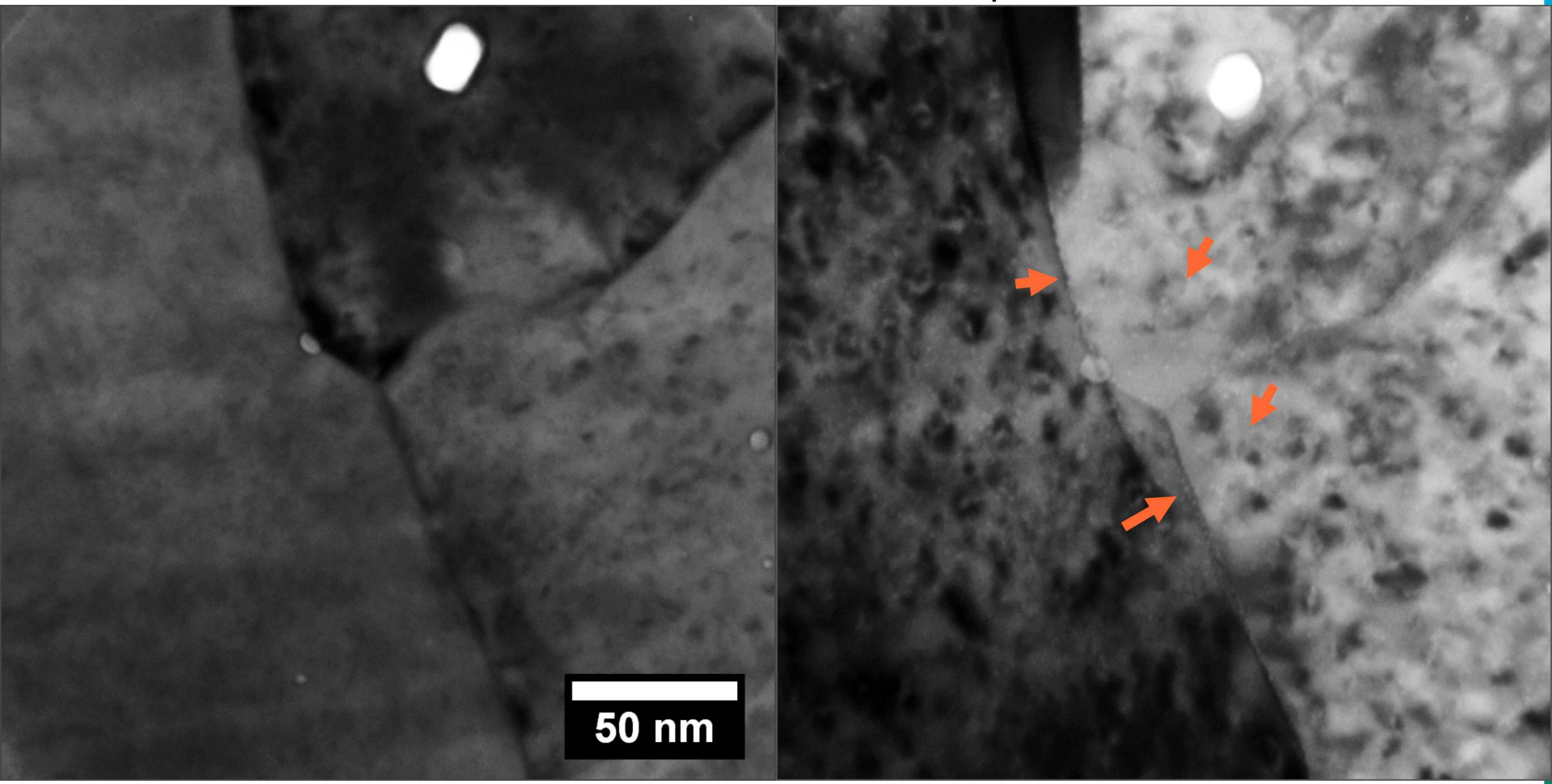


- Indicates very little He diffusion before trapping in lattice
- 5.6 dpa, 14 at.% He

Helium implantation into palladium resulted in temperature dependent microstructures

250ºC, $T/T_m = 0.29$

Pristine Material | After He implantation to ~3.3×10^{18} He/cm^2



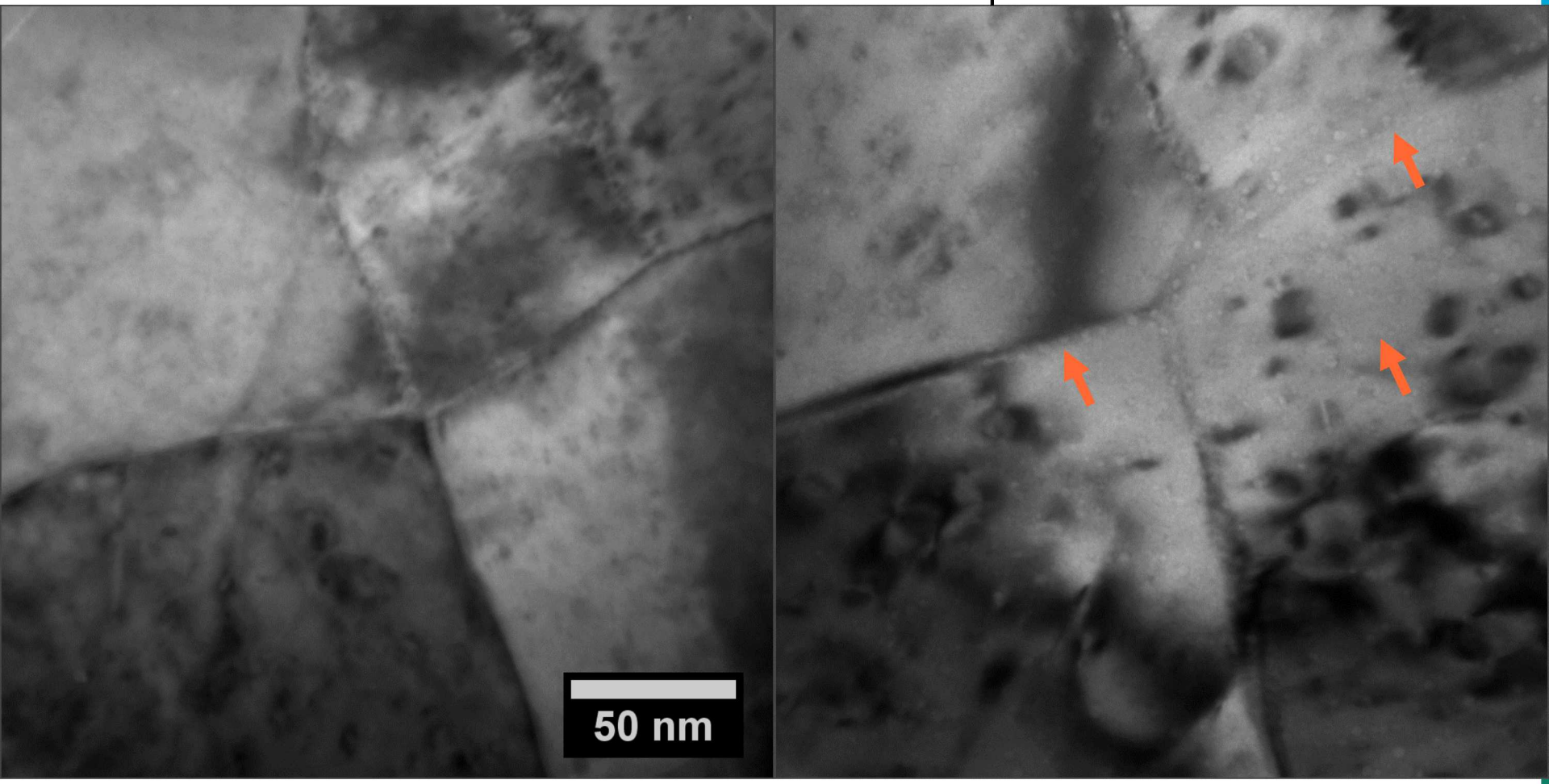
- Cannot observe early nucleation stages in the TEM
- Preferential GB nucleation → diffusion before trapping/bubble nucleation

Helium implantation into palladium resulted in temperature dependent microstructures

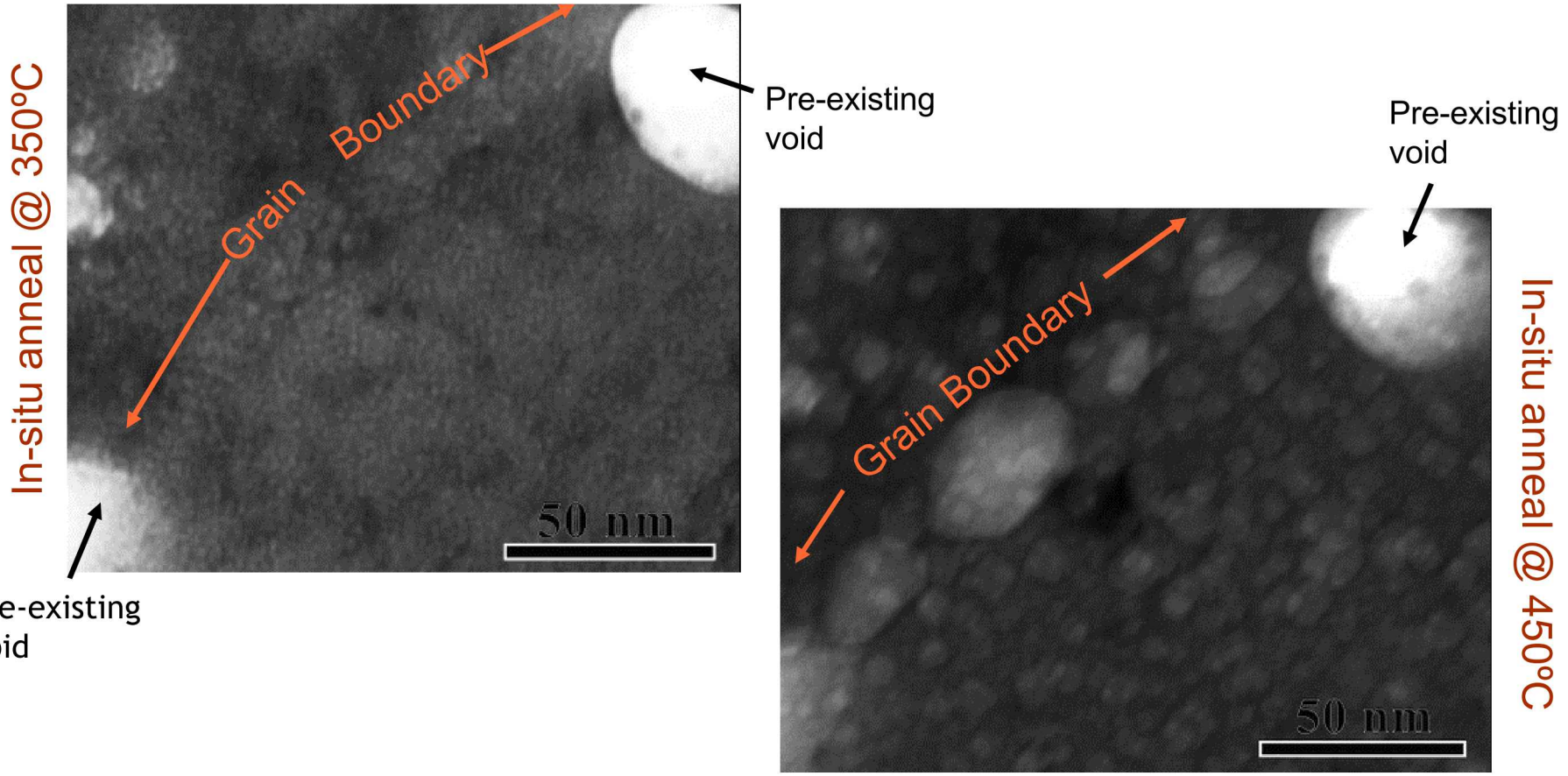
400ºC, $T/T_m = 0.37$

Pristine Material

After He implantation to ~3.6×10^{18} He/cm^2



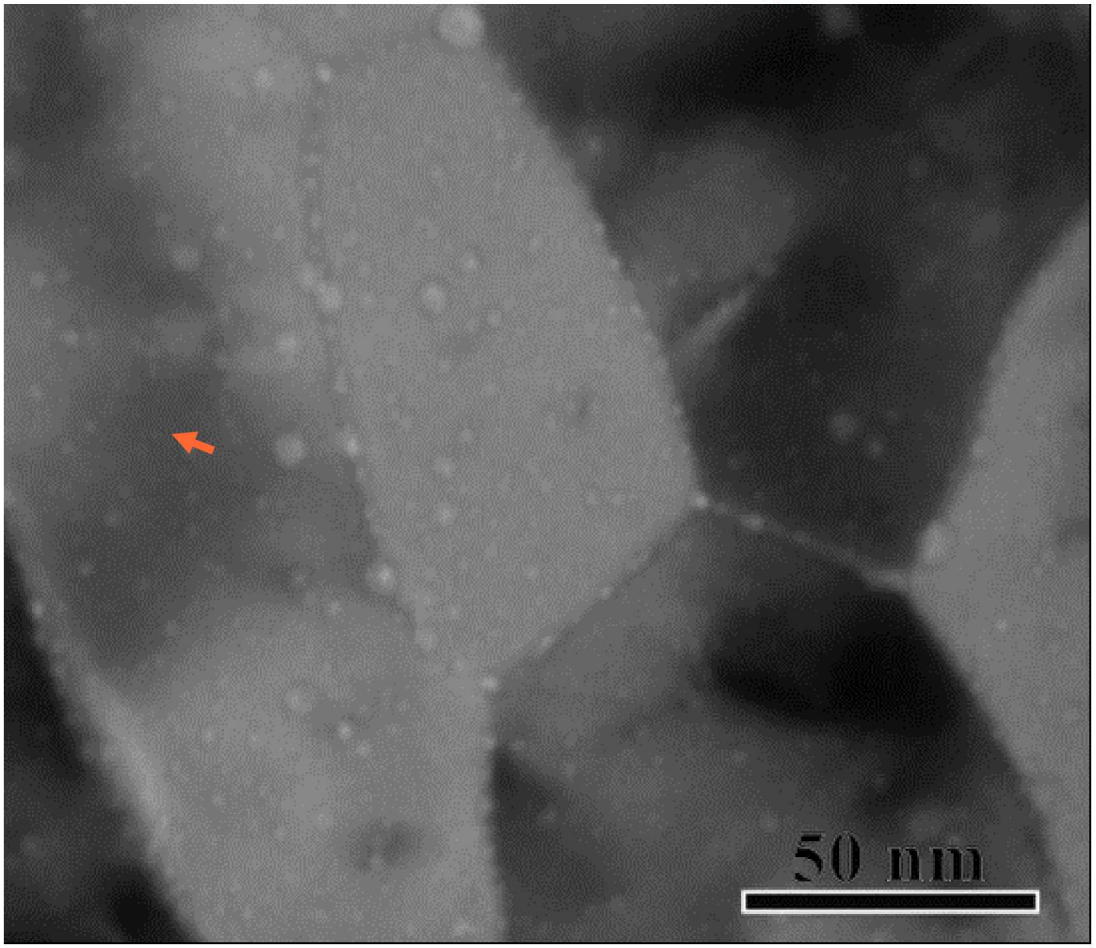
Cavities evolved into faceted structures during in-situ annealing of the sample implanted at room temperature



- Blisters form at boundaries by absorbing nearby cavities
- Large faceted cavities form inside the grains by absorbing smaller bubbles and possibly He from the matrix
- Blisters eventually burst, leaving behind a denuded zone at the boundary. Material remains in-tact.

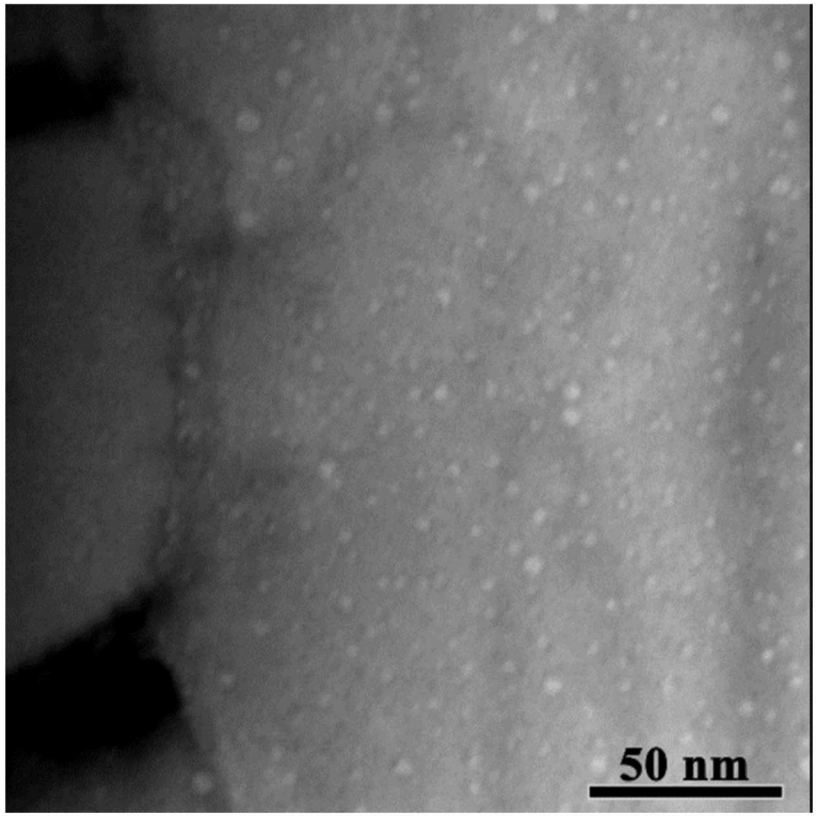
After 250°C implantation with a low fluence of helium, growth occurred due to bubble coalescence under annealing

In-situ annealing at 500ºC, $T/T_m = 0.42$

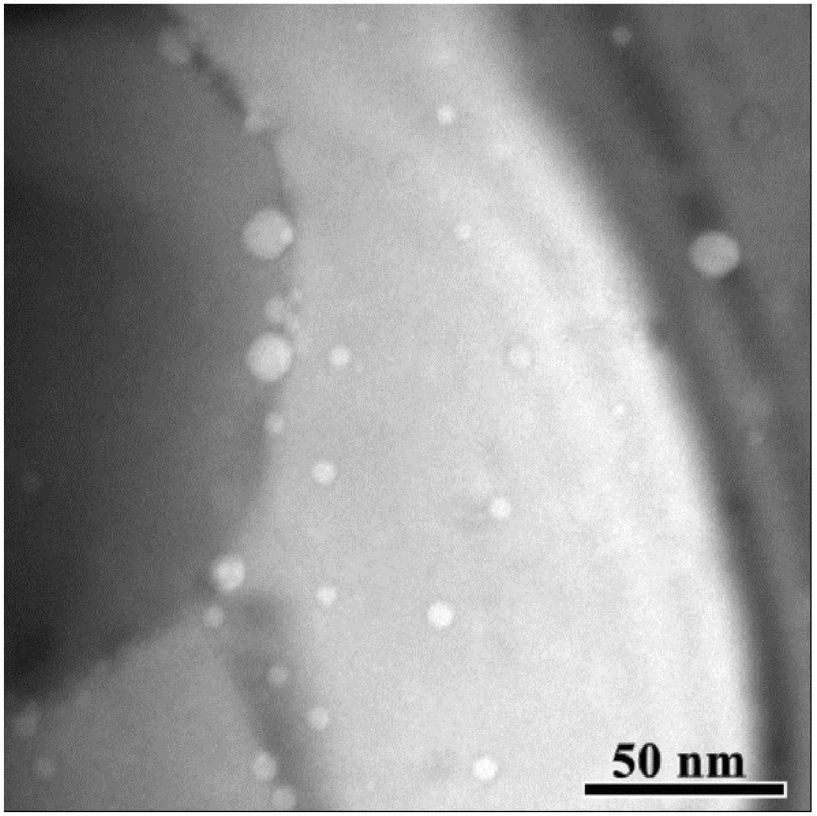


After 400°C implantation with a low dose of helium, growth occurred due to bubble coalescence under annealing

In-situ annealing at 550ºC, $T/T_m = 0.45$



In-situ annealing at 700ºC, $T/T_m = 0.53$



- Bubbles remain trapped at boundaries during grain growth
- Some bubbles appear to be strongly trapped inside grains (e.g. at defects)

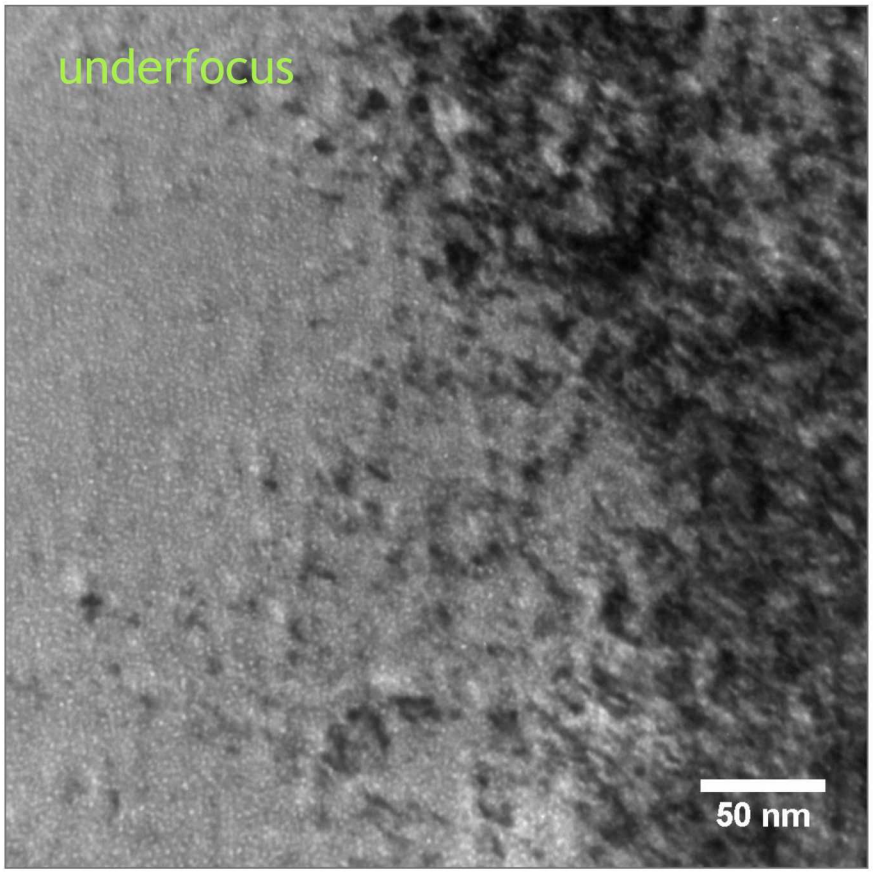
Thermal Aging of Tritium Aged Palladium

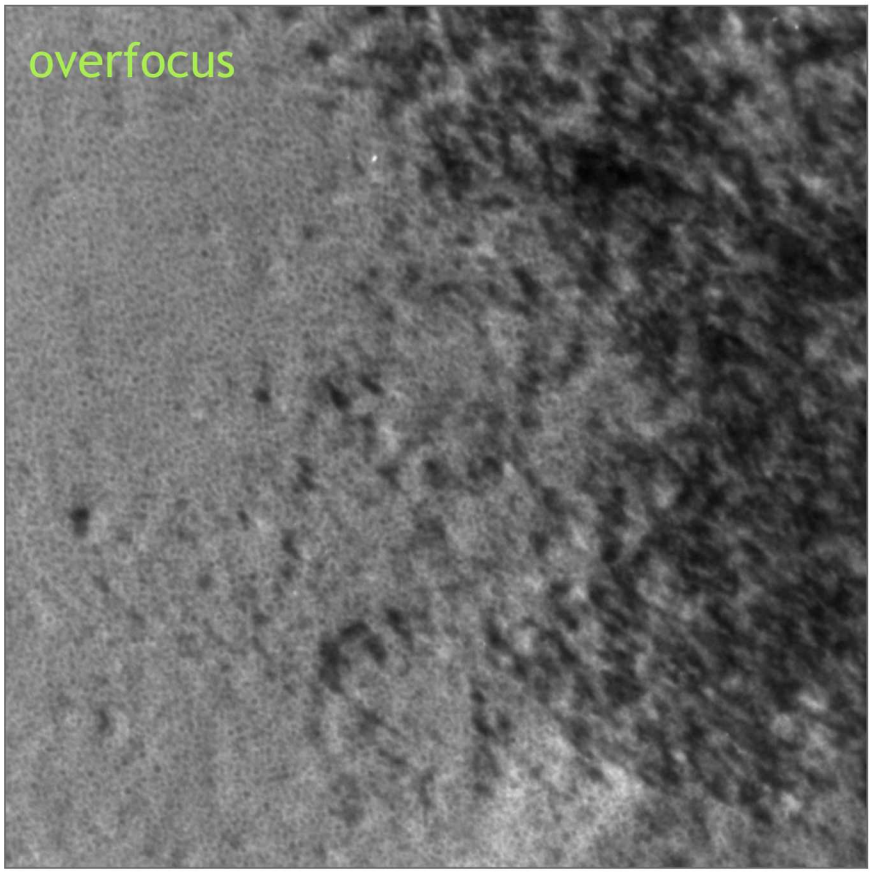
- Tritium aged PdNi sample had bubble distribution similar to was annealed in-situ to accelerate bubble growth

A palladium-nickel alloy was aged under tritium for 3.8 years

- Palladium-5% Nickel alloy was tritiated and aged for 3.8 years at SRNL
- Estimated ~10 at.% ^{3}He

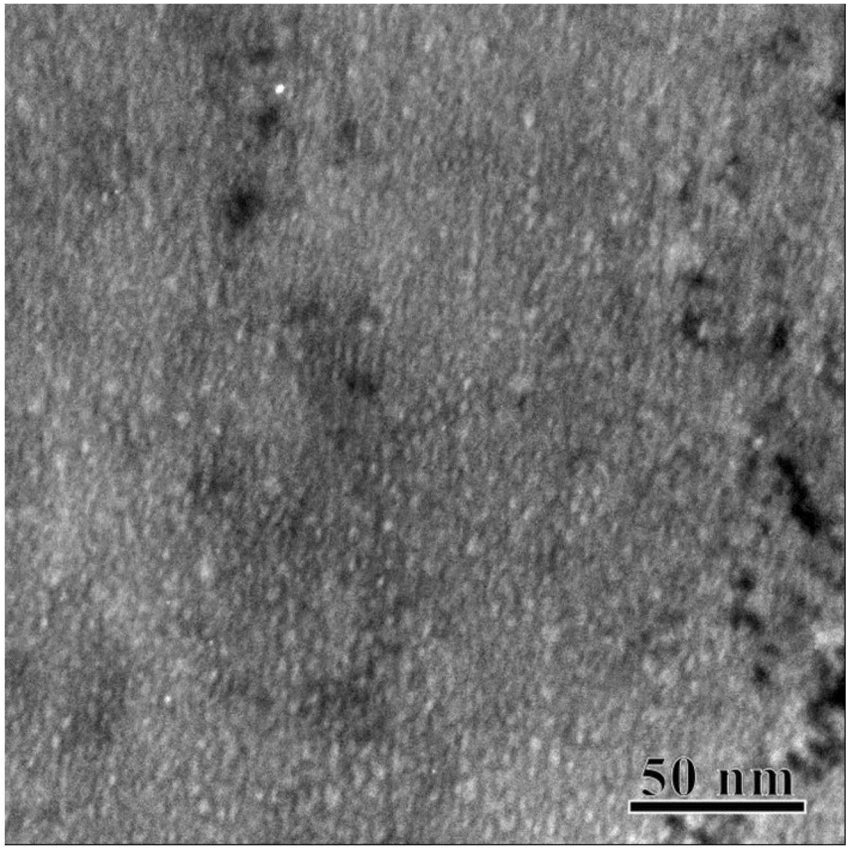
A high density of small, uniformly distributed ^{3}He bubbles (~1.5 nm in diameter) were present in the aged material



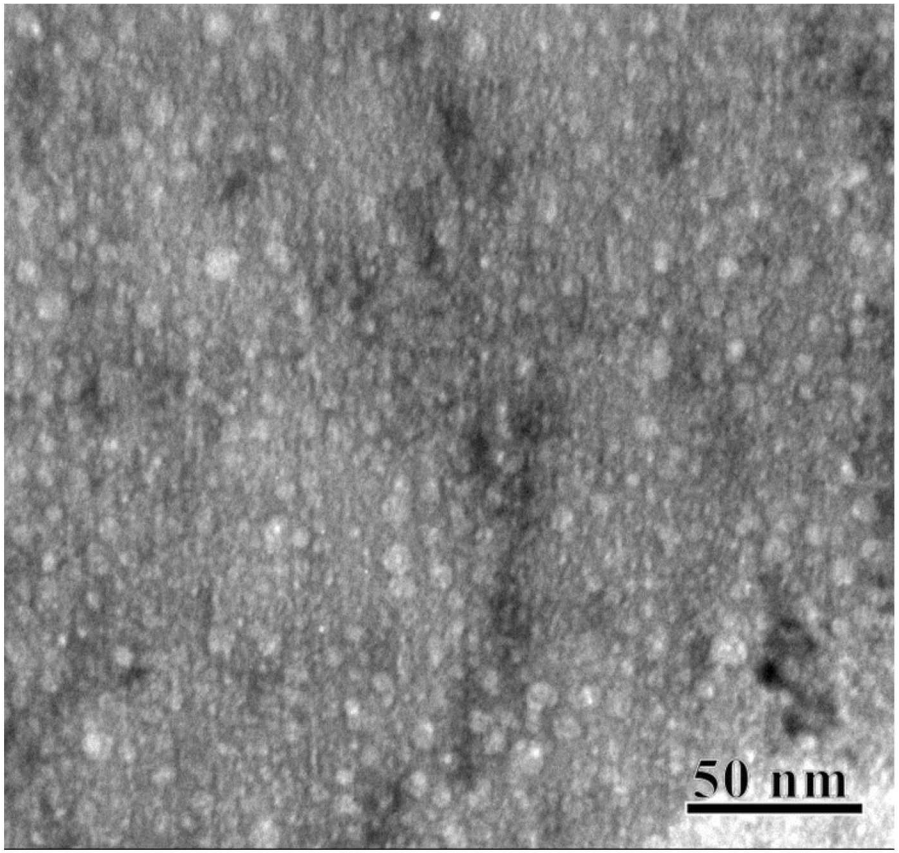


Bubbles began to grow @ 350°C during in-situ annealing aged PdNi sample, and became faceted @ 400°C

In-situ annealing at 350ºC, T/T_m = 0.34



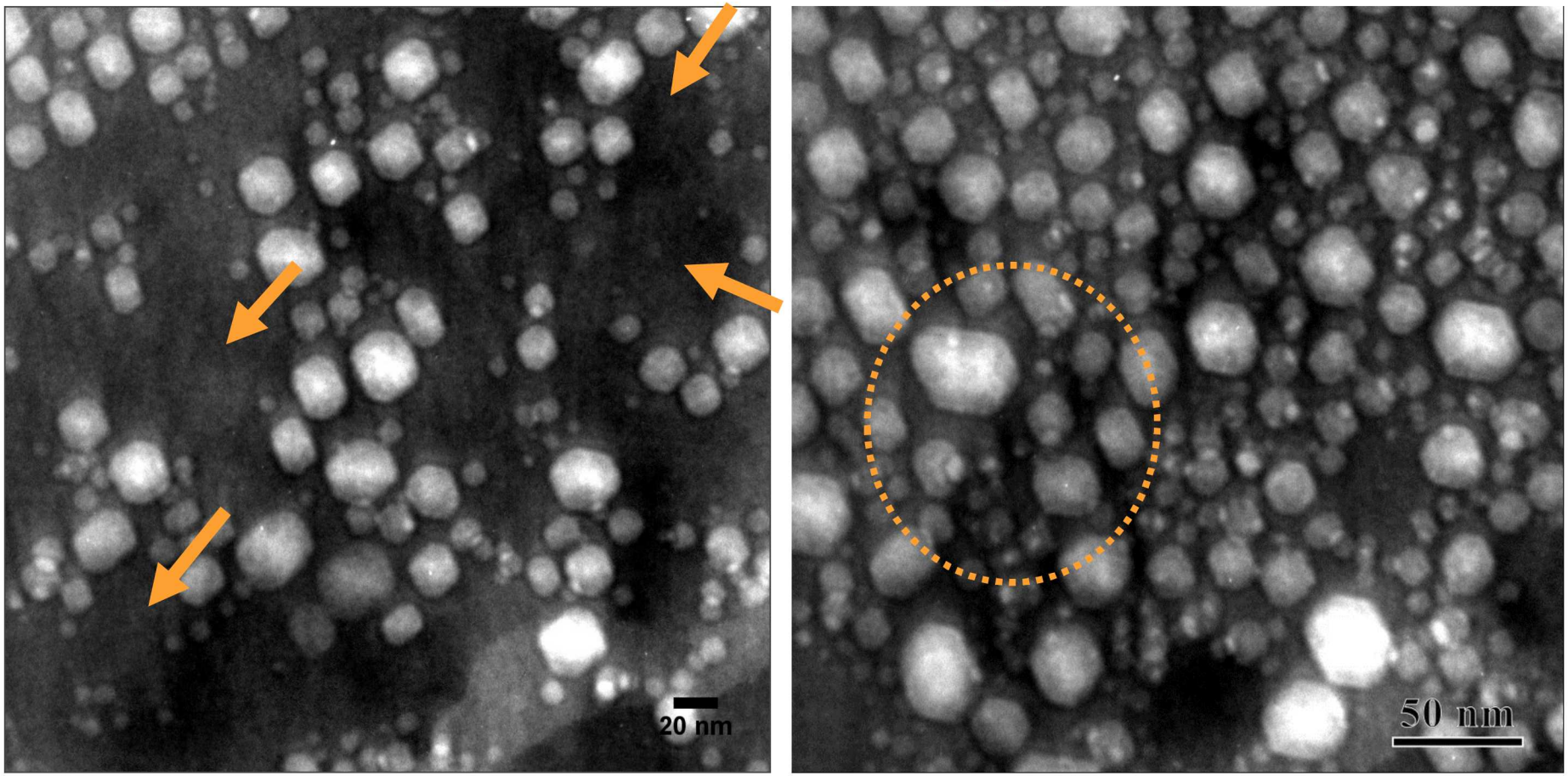
In-situ annealing at 400ºC , T/T_m = 0.37



- Faceted cavities continued to grow with increasing temperature
- Facets indicate that cavities are near equilibrium by T/T_m = 0.37

Cavities grew by absorbing nearby smaller bubbles in aged PdNi

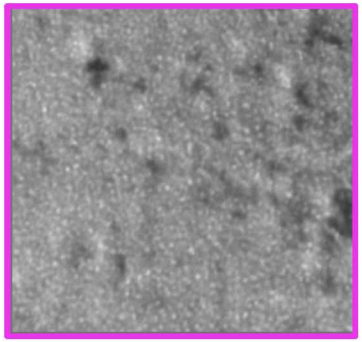
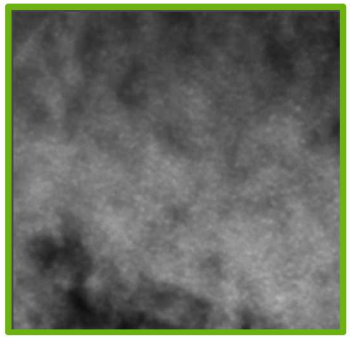
In-situ annealing at 900ºC , $T/T_m = 0.64$



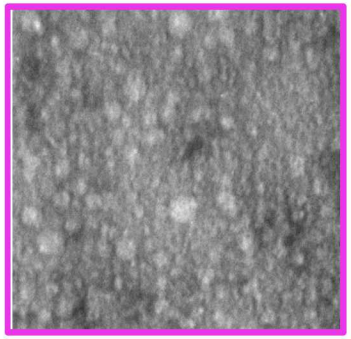
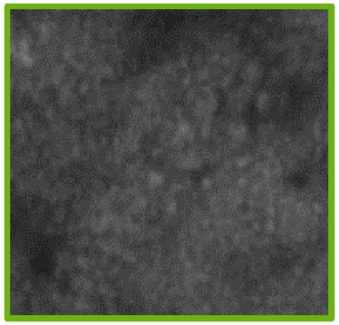
- At high temperature, cavities coalesced or reached the surface, leaving behind denuded zones

Summary of bubble microstructure in helium implanted and tritium aged palladium before and after annealing

- Bubble microstructure was similar in tritium aged sample and sample implanted with helium at room temperature.

- Cavities in tritium aged and RT implanted samples grew by absorption of nearby cavities under annealing.

- Large, faceted cavities were present in tritium aged and RT implanted samples after annealing.