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Title: (U) AFM/STM Plutonium capability, research summary and future plans

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# (U) AFM/STM Plutonium capability, research summary and future plans

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*Laboratory Directed Research and Development – Directed Research*  
*Los Alamos National Laboratory*

# Summary

- Initiated GB-AFM & UHV-STM capability installation at MST-7 (June 2011)
- Authorized for Pu studies on Nov 16, 2015 (LDRD DR, PI-M.Wilkerson)
- Performed surface analysis of:
  - $\delta$ -Pu<7%Ga> coupon (MST-16) (AFM&STM)
  - $\delta$ -Pu<2%Ga> coupon (MST-16) (AFM)
  - PAD-PuO<sub>2</sub> thin film (MPA-11) (AFM)
- Started Pu aging study for Science Campaign 1 (PL - W. Blumenthal)
- Completed AFM instrument upgrade (SC 1)
- Purchased specialized tools for AFM studies under (SC 1):
  - ✓ controlled gaseous environments
  - ✓ controlled temperature 238 - 523K

# Scanning Probe Microscopies (SPM)

## Omicron UHV-STM:

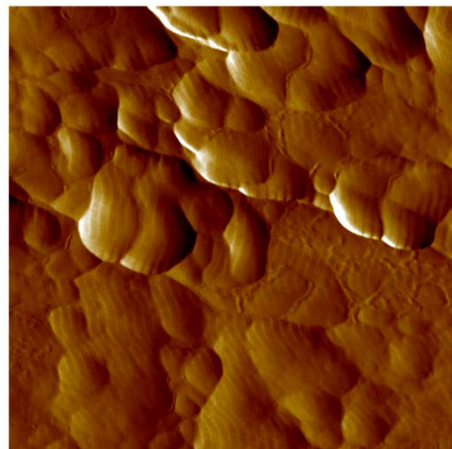
- STS Mode
  - ✓ local electronic structure of both occupied and unoccupied states
- STM Mode
  - ✓ atomic-scale imaging
  - ✓ defect structures (vacancies, steps, adatoms, etc.)
  - ✓ adsorbate binding sites and coadsorbate interactions
  - ✓ surface diffusion
- AES/LEED
  - ✓ surface cleanliness and long-range order

## Bruker Multimode™ AFM:

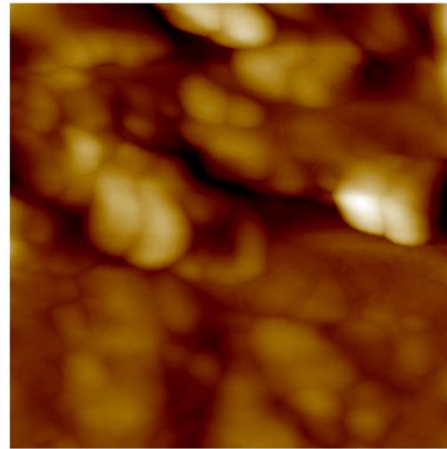
- Height & Amplitude Modes
  - ✓ 3D morphology
- Phase Contrast Mode
  - ✓ qualitative mapping of surface mechanical and chemical properties
- Peak Force Mode
  - ✓ quantitative mapping of surface mechanical properties
- Contact Mode
  - ✓ quantitative mapping of friction coefficients

# AFM 3D topography(amplitude & height modes) and phase imaging of $\delta$ -Pu<7at%Ga> with $1 \times 1 \mu\text{m}^2$ and $75 \times 75 \mu\text{m}^2$ scale

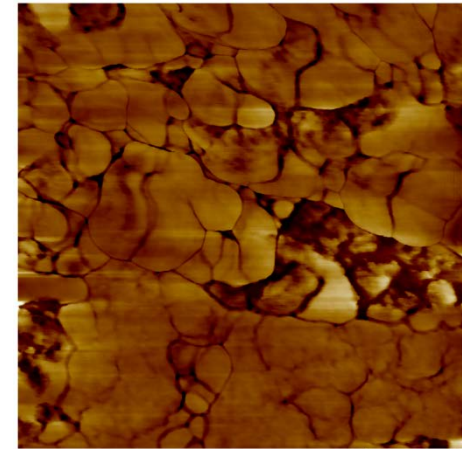
- Glove box-atomic force microscope (GB-AFM)
  - UHP-Ar atmosphere ( $\text{O}_2$  and  $\text{H}_2\text{O} < 1\text{ppm}$ ) at  $\sim 580$  Torr



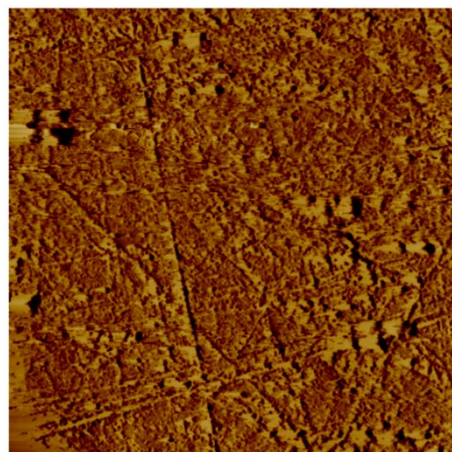
Amplitude 200.0 nm



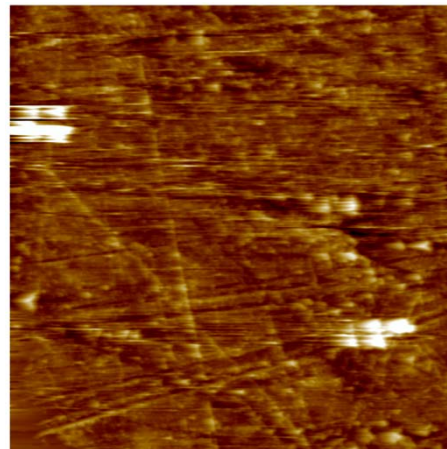
Height 200.0 nm



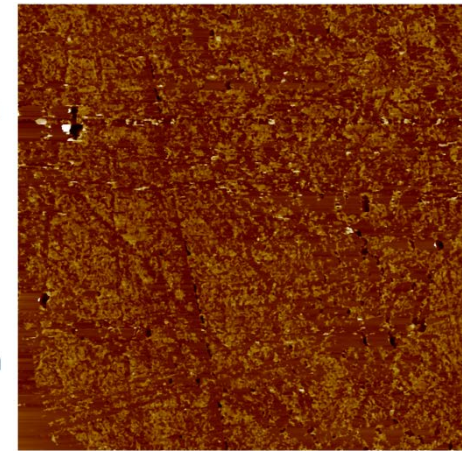
Phase 200.0 nm



Amplitude 20.0  $\mu\text{m}$



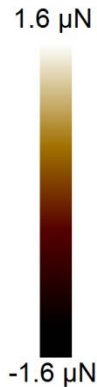
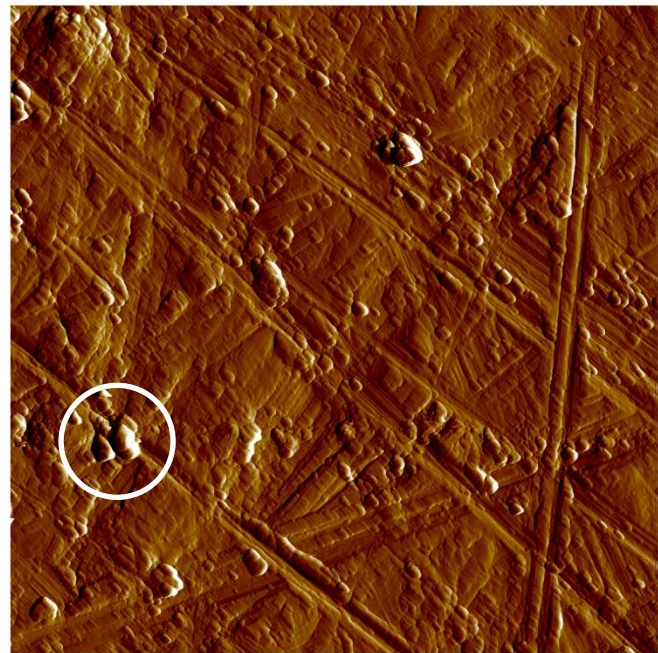
Height 20.0  $\mu\text{m}$



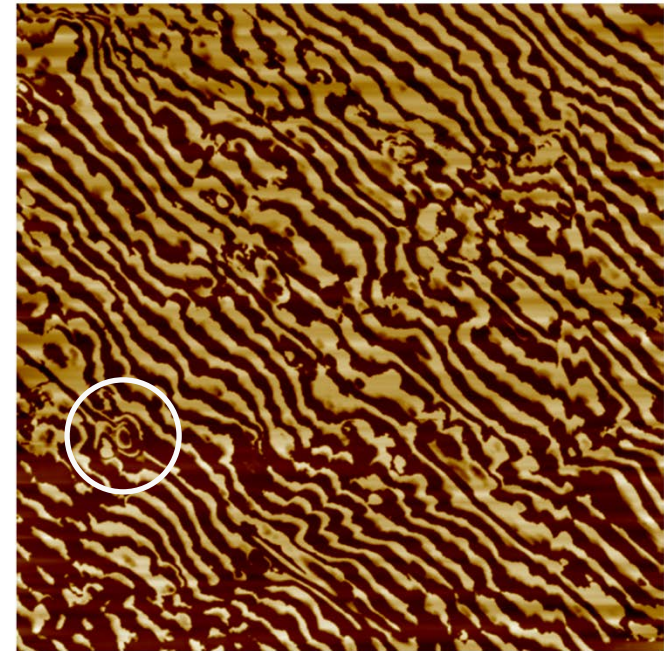
Phase 20.0  $\mu\text{m}$

# Surface mechanical properties of $\delta$ -Pu<2at%Ga>

- Applied Peak Force-Quantitative Nanomechanical Mapping (PF-QNM) Mode enabling quantitative mapping of surface mechanical properties in addition to 3D morphology.



0.0 Peak Force Error 50.0  $\mu$ m



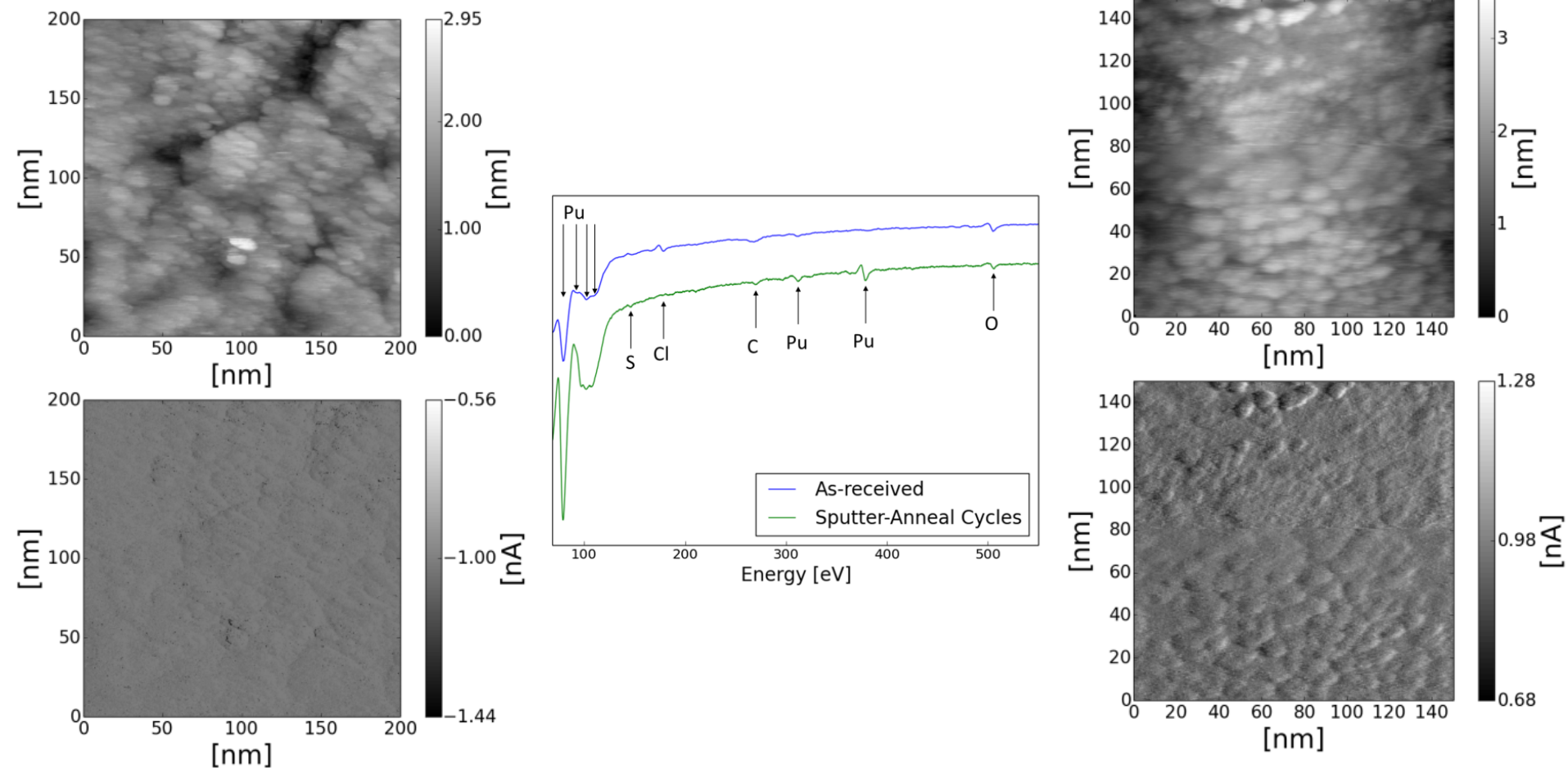
0.0 Deformation 50.0  $\mu$ m

Observations:

- Peak Force Error image (left) provides surface morphology
- Deformation image (right) gives mechanical properties mapping:
  - Striations represent deformation of the surface by the tip (brighter regions correspond to greater deformation or softer material)
  - A few hypotheses are being considered to explain this image

# STM Z-Images (Height) of $\delta$ -Pu<7at%Ga>

- Ultra high vacuum conditions (UHV) at  $\sim 4 \times 10^{-10}$  Torr
- Multiple Sputter Cleaning/Annealing Cycles



# Future Plans Pu-AFM/STM capabilities

- ❑ Ensure these capabilities and expertise remain state-of-the-art for the long-term:
  - Search is underway for 2 postdoctoral researchers
- ❑ Obtain surface image with atomic scale resolution (STM):
  - Develop techniques for samples with atomically smooth Pu surface
- ❑ Correlate local surface microstructure/mechanical properties with surface chemical composition (AFM vs SIMS/XPS/AES/EMPA/SEM):
  - Develop collaboration with LLNL and continue collaboration with MST-16
- ❑ Inform theory/modeling on relationship between surface structure-composition and corrosion initiation:
  - Determine nascent corrosion sites with atomic scale (STM) and mesoscale (AFM) resolution