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Title: Role of Interfaces in the Design of Ultra-high Strength, Radiation Damage Tolerant Nanocomposites

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Role of Interfaces in the design of ultra-high strength, radiation damage tolerant nanocomposites

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The combination of high strength and high radiation damage tolerance in nanolaminate composites can be achieved when the individual layers in these composites are only a few nanometers thick and contain special interfaces that act both as obstacles to slip, as well as sinks for radiation-induced defects. The morphological and phase stabilities and strength and ductility of these nano-composites under ion irradiation are explored as a function of layer thickness, temperature and interface structure. Magnetron sputtered metallic multilayers such as Cu-Nb and V-Ag with a range of individual layer thickness from approximately 2 nm to 50 nm and the corresponding 1000 nm thick single layer films were implanted with helium ions at room temperature. Cross-sectional Transmission Electron Microscopy (TEM) was used to measure the distribution of helium bubbles and correlated with the helium concentration profile measured vis ion beam analysis techniques to obtain the helium concentration at which bubbles are detected in TEM. It was found that in multilayers the minimum helium concentration to form bubbles (approximately 1 nm in size) that are easily resolved in through-focus TEM imaging was several atomic %, orders of magnitude higher than that in single layer metal films. This observation is consistent with an increased solubility of helium at interfaces that is predicted by atomistic modeling of the atomic structures of fcc-bcc interfaces. At helium concentrations as high as 7 at.%, a uniform distribution of 1 nm diameter bubbles results in negligible irradiation hardening and loss of deformability in multilayers with layer thicknesses of a few nanometers. The control of atomic structures of interfaces to produce high helium solubility at interfaces is crucial in the design of nano-composite materials that are radiation damage tolerant. Reduced radiation damage also leads to a reduction in the irradiation hardening, particularly at layer thickness of approximately 5 nm and below. The strategies for design of radiation-tolerant structural materials based on the knowledge gained from this work will be discussed.
This research is funded by US DOE, Office of Basic Energy Sciences.

Role of Interfaces in the design of ultra-high strength, radiation damage tolerant nanocomposites

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Acknowledgements: Nathan Mara (nanoindentation, CINT user project)

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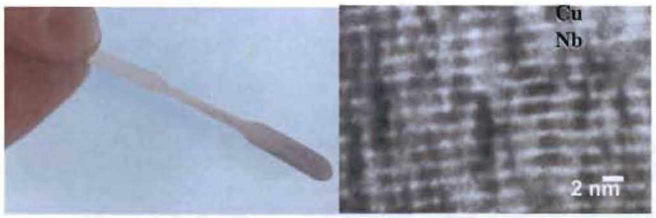
Objectives

- Determine the critical concentration of implanted helium at which bubbles nucleate at different interfaces.
- Explore the effect of ion implantation induced defects (particularly He bubbles) on irradiation hardening.

Approach

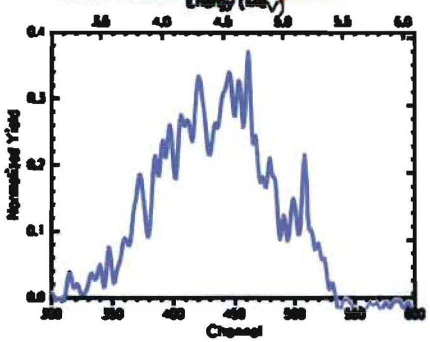
Synthesis of nanostructured materials (CINT)



Ion irradiation (IBML)

Characterization

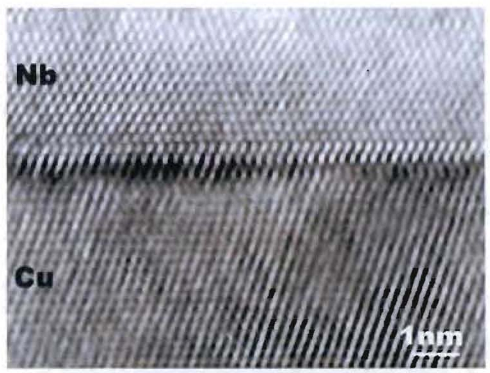
Ion-beam analysis



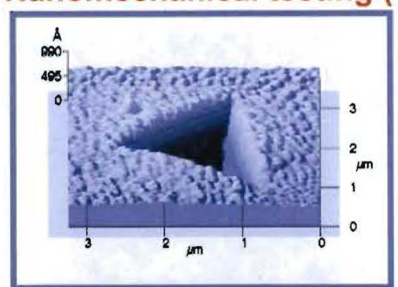
He concentration

Depth He

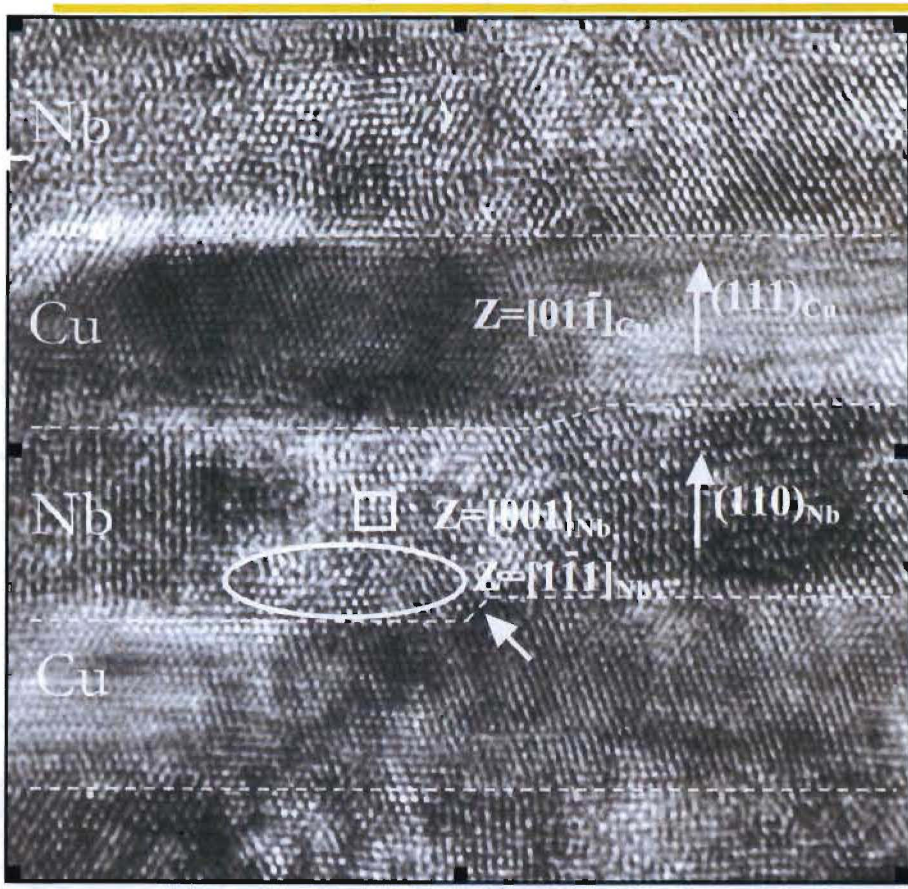
HRTEM (EML)



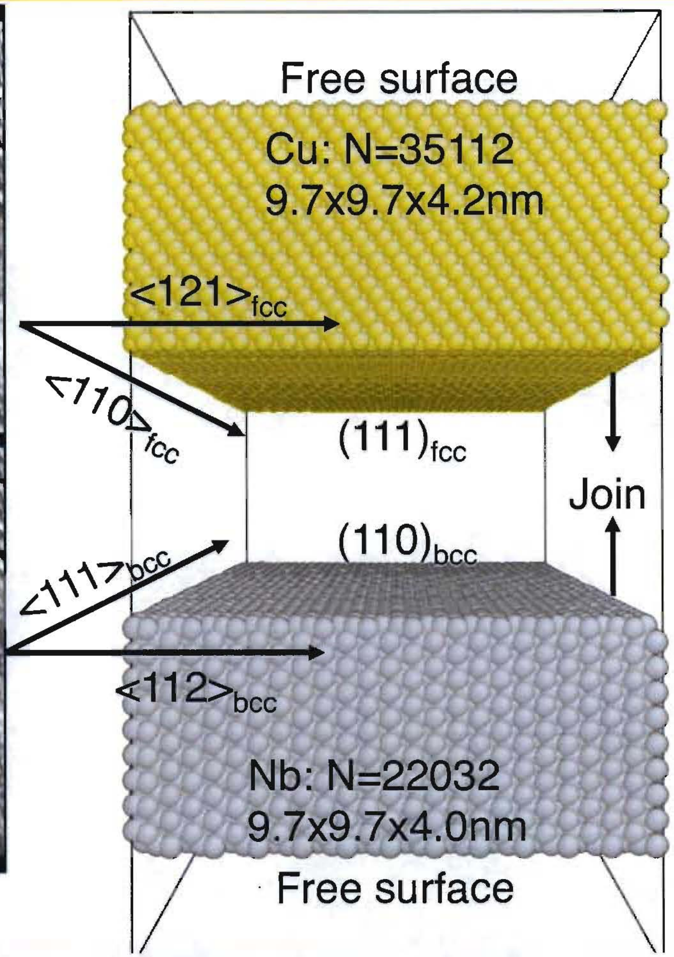
Nanomechanical testing (CINT)



FCC-BCC Model System:
integrate experiments with atomistic modeling



At 300K, ion beam mixing is not expected in immiscible Cu-Nb system. [Ref: Averback, et al., Appl. Phys. A (1986)].



Large misfit strain (≈ 12%)

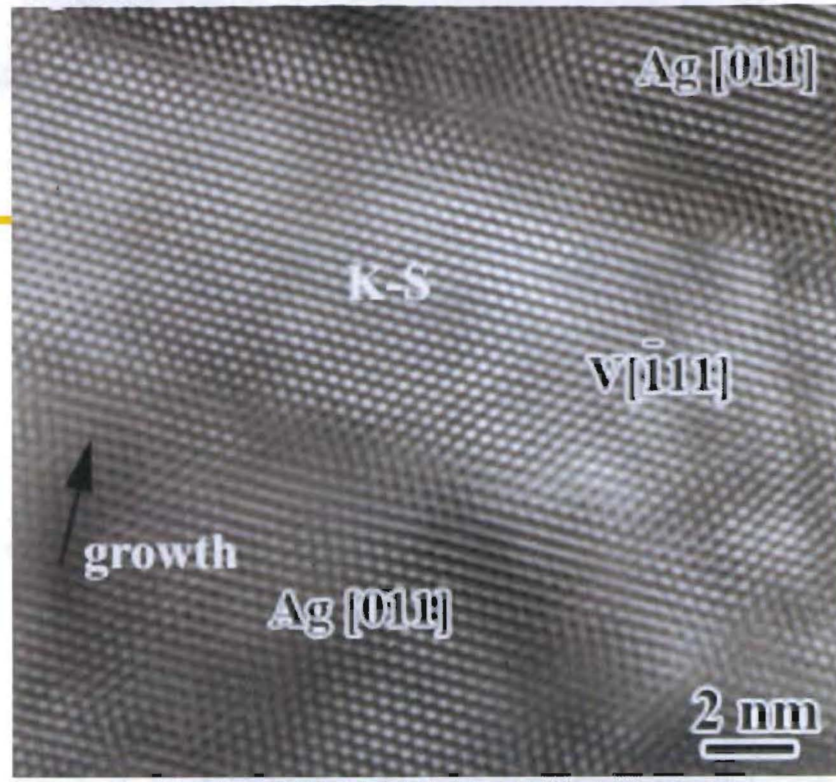
Positive heat of mixing

Atomistic models created by joining fcc-Cu and bcc-Nb in the experimentally observed Kurdjumov-Sachs (KS) orientation relation (OR)

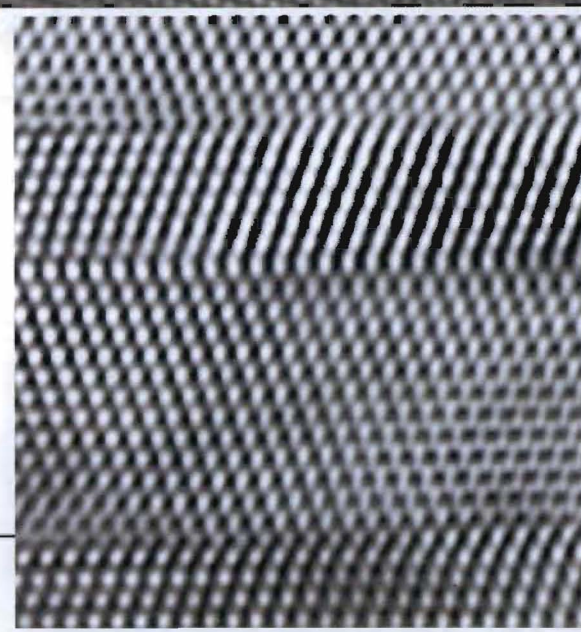
Materials Investigated

- FCC-BCC multilayers: Kurdjumov-Sachs orientation relationship: {111}fcc // {110}bcc // interface <011>fcc // <111>bcc.

V-Ag (ε = 9.7%), Cu-Nb (ε = 11%), Cu-Mo (ε = 6.2%), Cu-V (ε = 2.4%).



- Σ3{111} coherent twin boundary in Cu

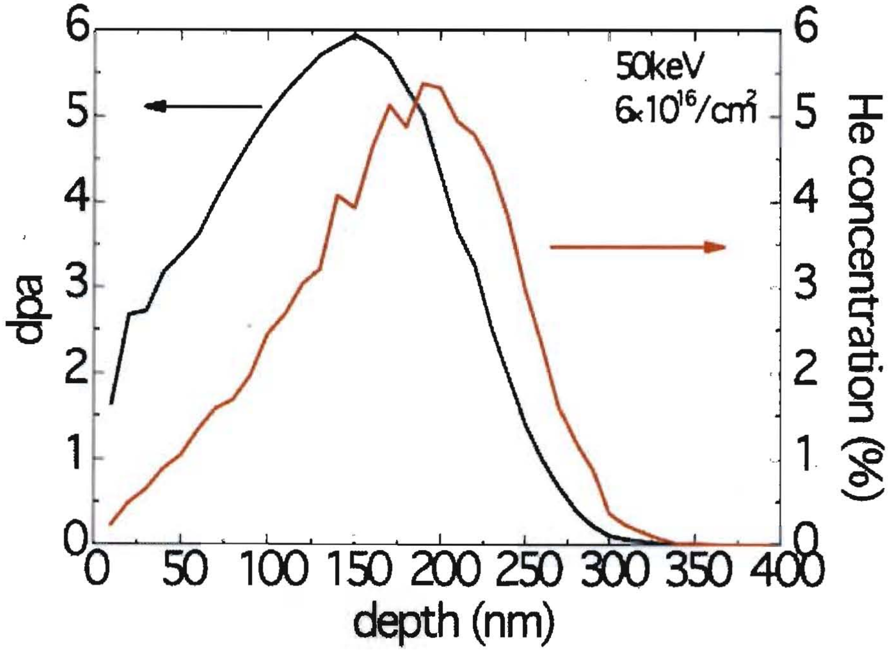


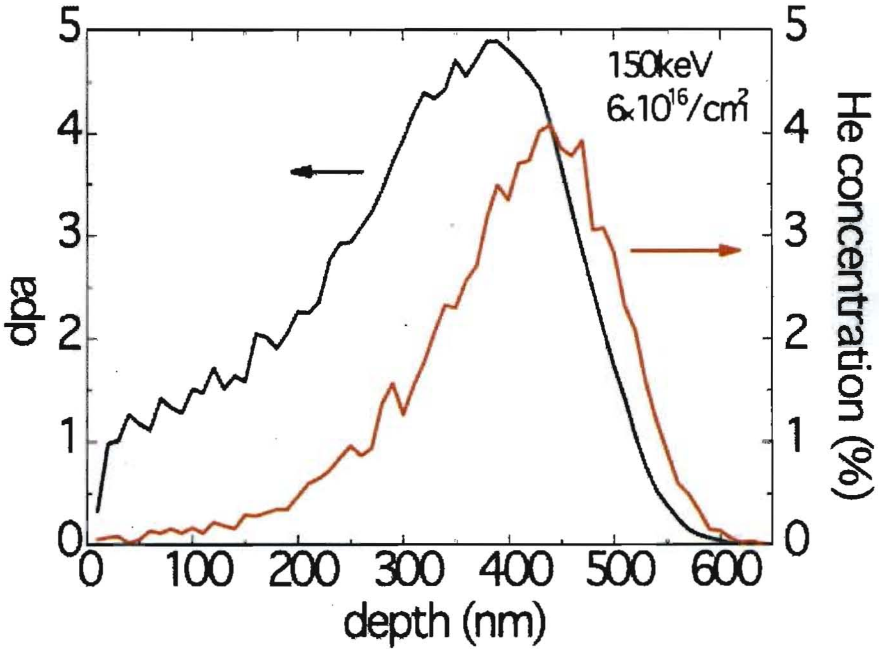
- Single layer metal metals (Cu, Nb,...)

Helium Ion Implantation Experiments

(room temperature implants)

Calculations using software SRIM (Stopping and Range of Ions in Matter), James Ziegler





Profiles were measured experimentally by a variety of techniques:

Elastic Recoil Detection (ERD)

Nuclear Reaction Analysis (NRA): implants were He^{3+}

Secondary Ion Mass Spectroscopy (SIMS)

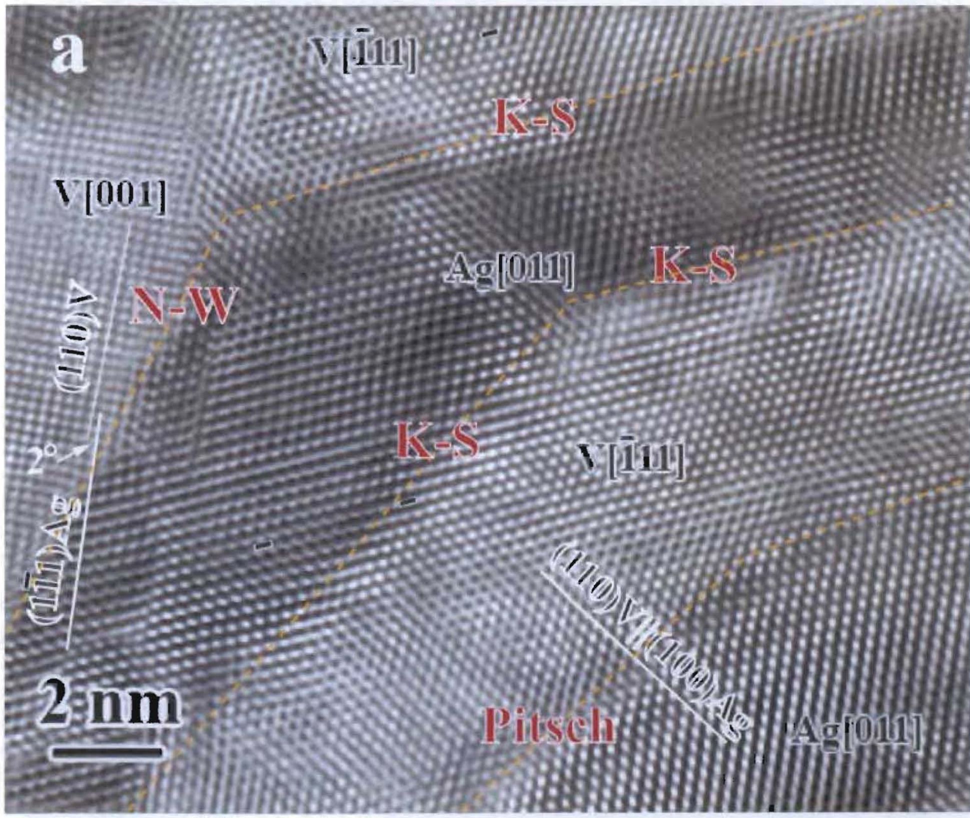
Multiple orientation relationships observed in V-Ag

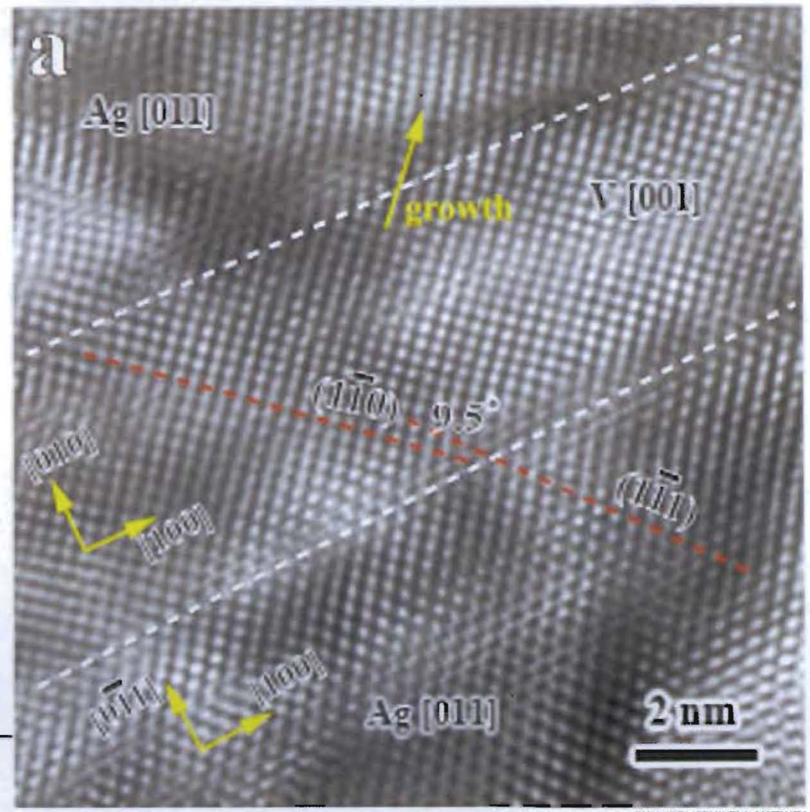
Kurdjumov-Sachs: $\{111\}_{fcc} \| \{110\}_{bcc}$ & $\langle 011\rangle_{fcc} \| \langle 111\rangle_{bcc}$

Nishiyama-Wasserman: $\{111\}_{fcc} \| \{110\}_{bcc}$ & $\langle 011\rangle_{fcc} \| \langle 001\rangle_{bcc}$

Bain Orientation: $\{100\}_{fcc} \| \{100\}_{bcc}$ & $\langle 011\rangle_{fcc} \| \langle 001\rangle_{bcc}$

Pitsch orientation: $\{100\}_{fcc} \| \{110\}_{bcc}$ & $\langle 110\rangle_{fcc} \| \langle 111\rangle_{bcc}$

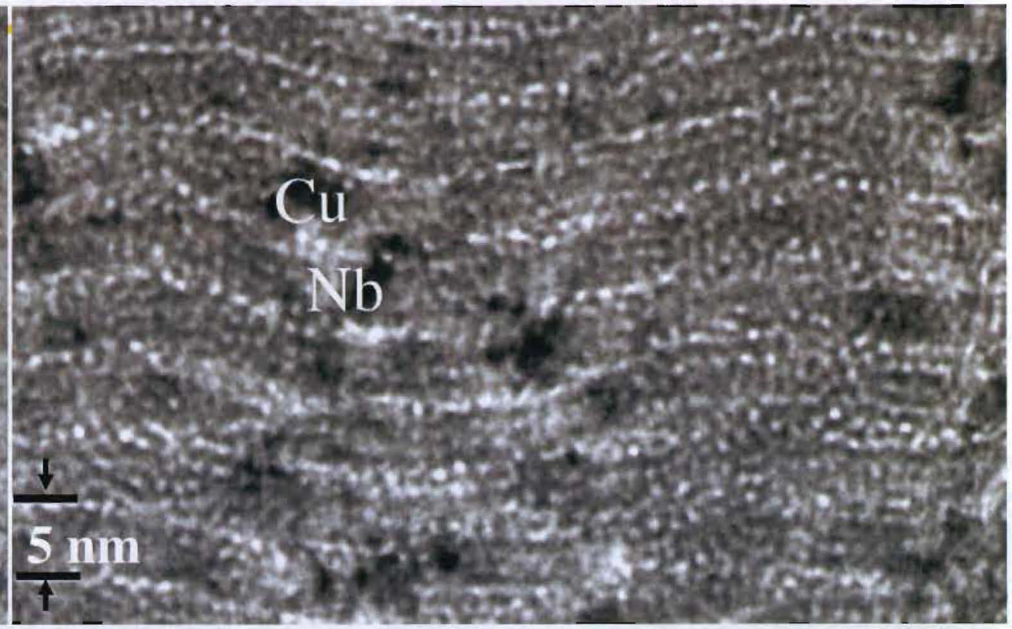




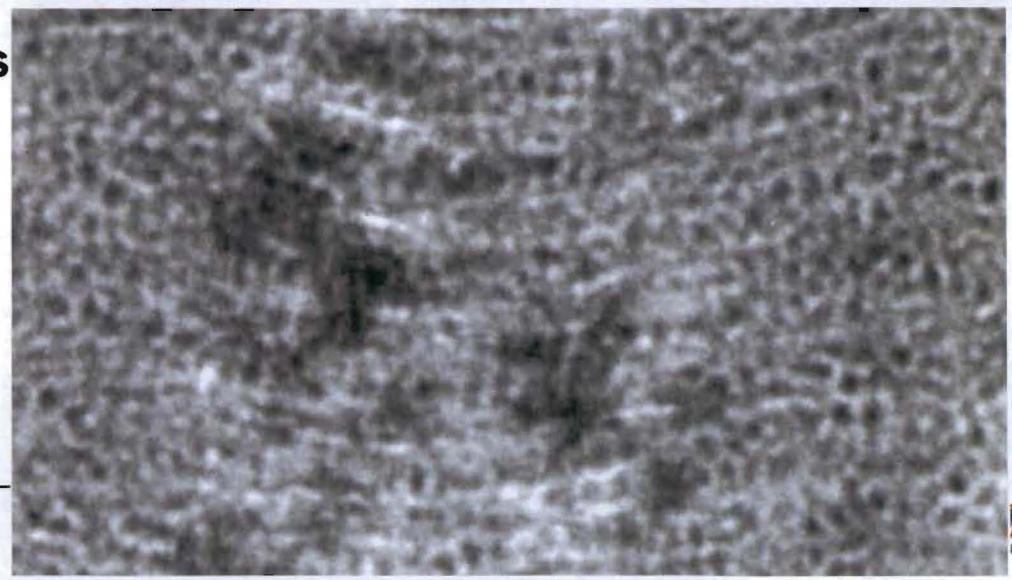
Through-focus imaging in TEM is used to detect helium bubbles

Near-focus

Under-focus



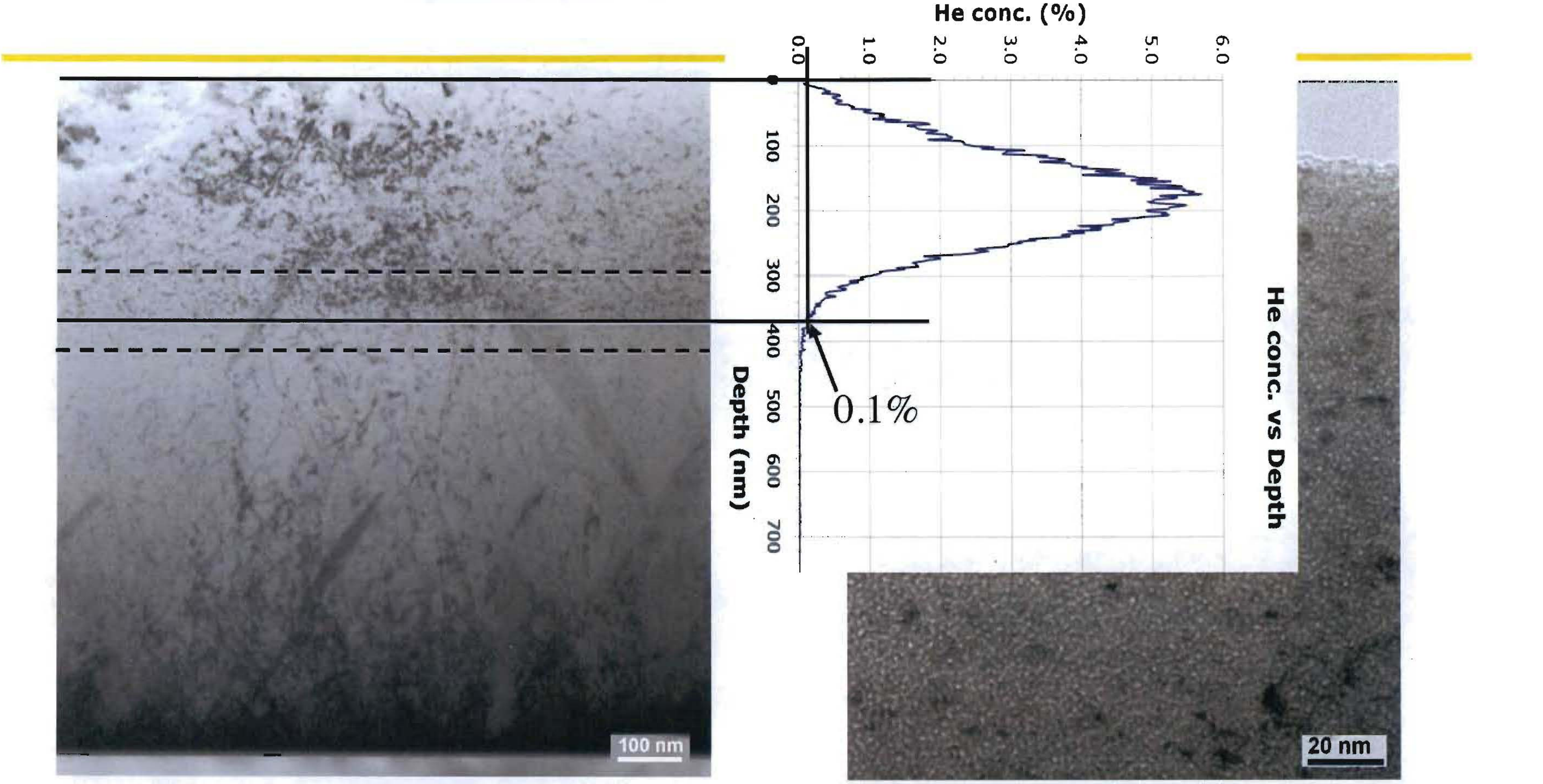
Over-focus



Room temperature He implantation,
Energy: 35 keV, Dose: $1 \times 10^{17}\ /cm^2$;

He Ion Implantation Damage in Pure Cu

He ions at 35 keV and 1 x 10^{17}/ cm^2

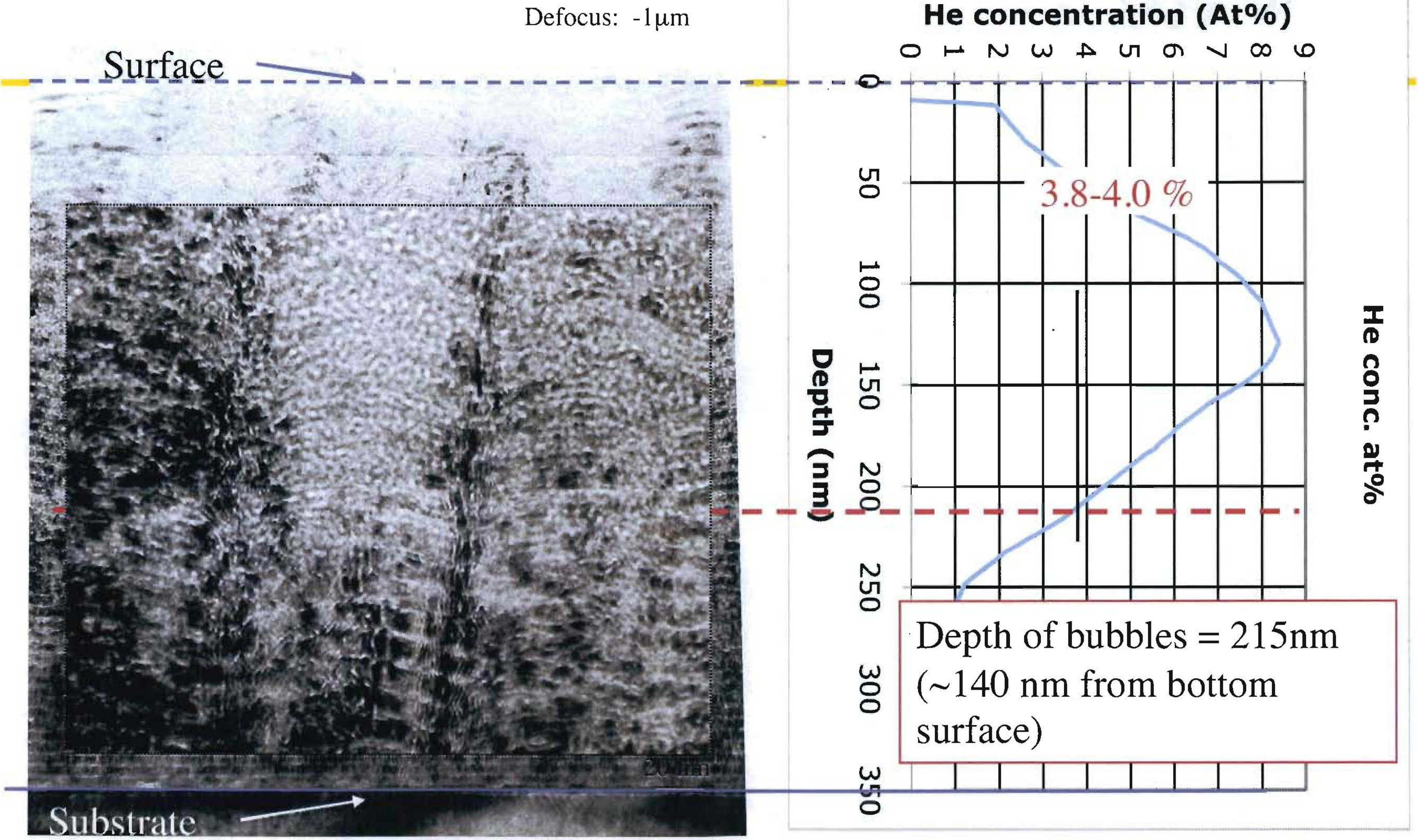


BF TEM image of the full thickness of a pure Cu film -

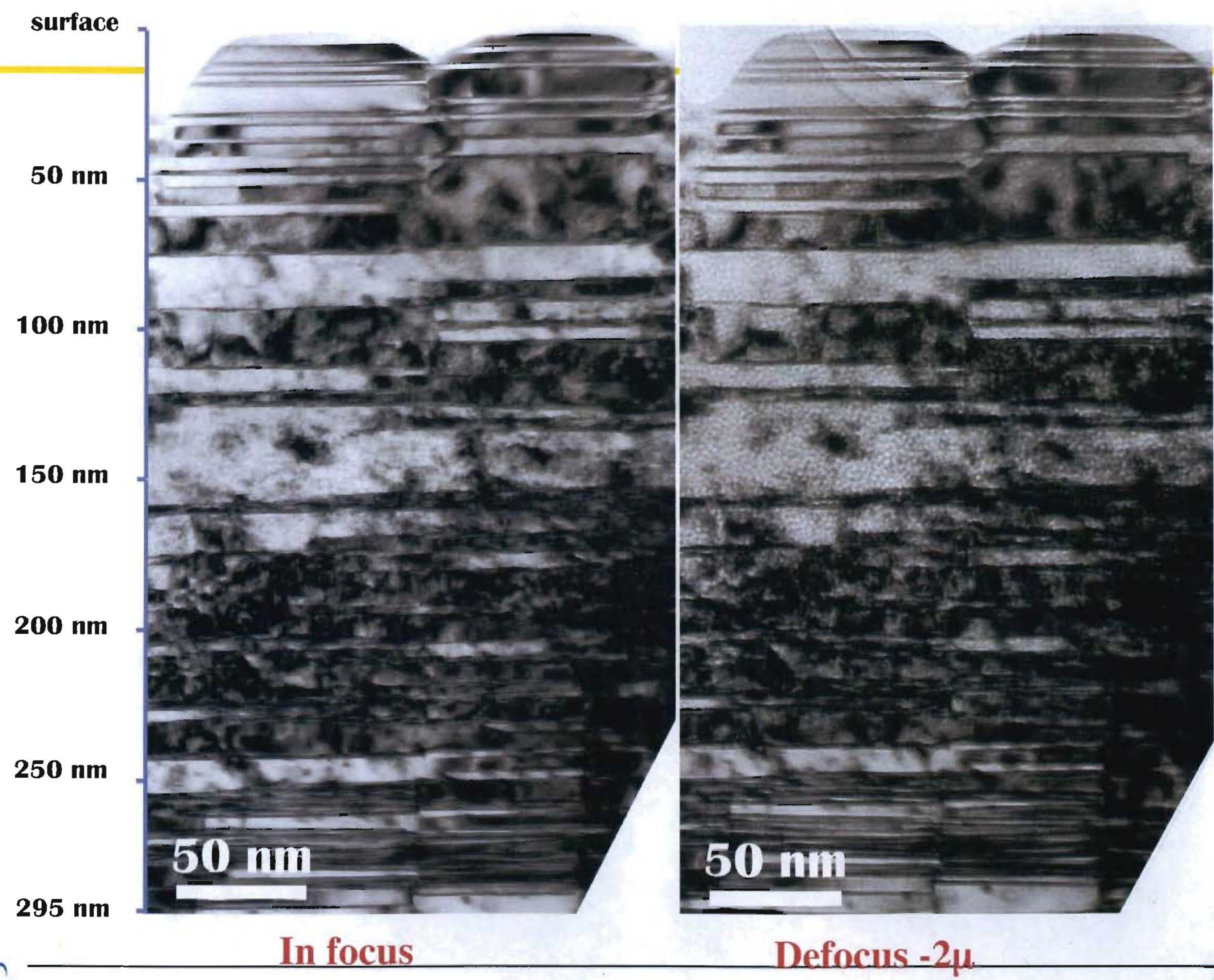
Upper region of the film - He bubbles start to form near the top surface

Bubbles detected at helium concentrations << 0.1 at.%

In 2.5 nm Cu/Nb multilayers, bubbles are detected when Helium concentration exceeds ≈ 4 at.% (twice that of 5 nm multilayers)

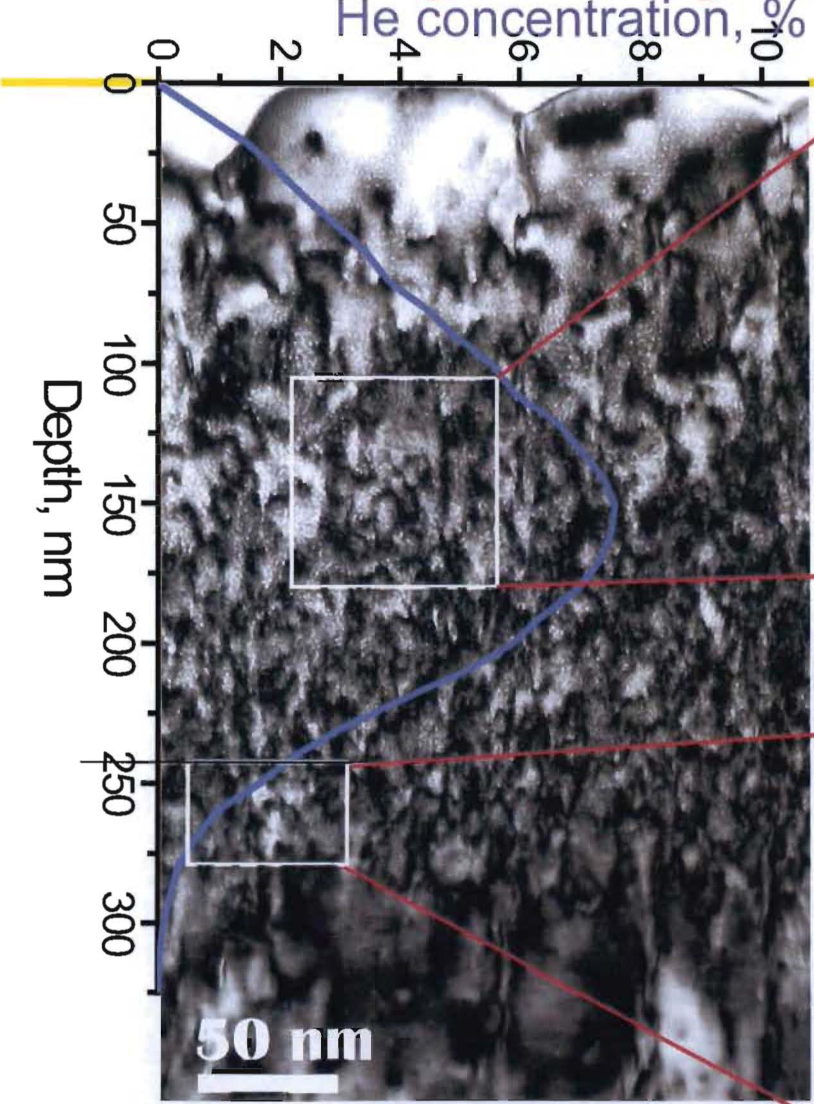


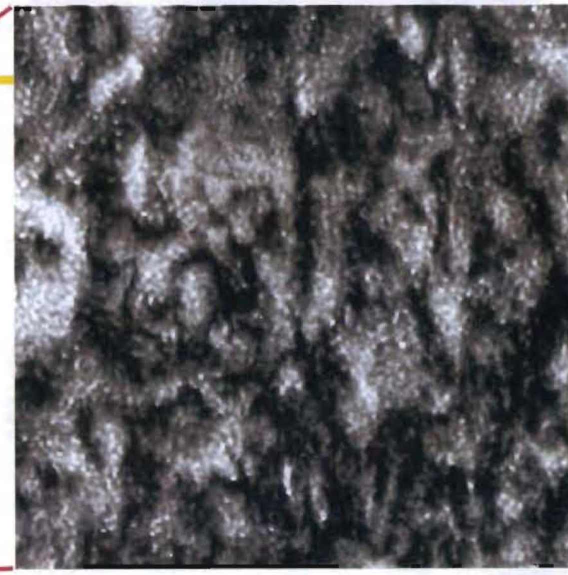
Bubble formation in Nano-twinned Cu



Bubble distribution vs He concentration

Twinning has no significant effect on He solubility





Bubbles visible even at very low He^{3}+ concentration: << 1%

Critical Helium Concentration at which Bubbles are Resolved

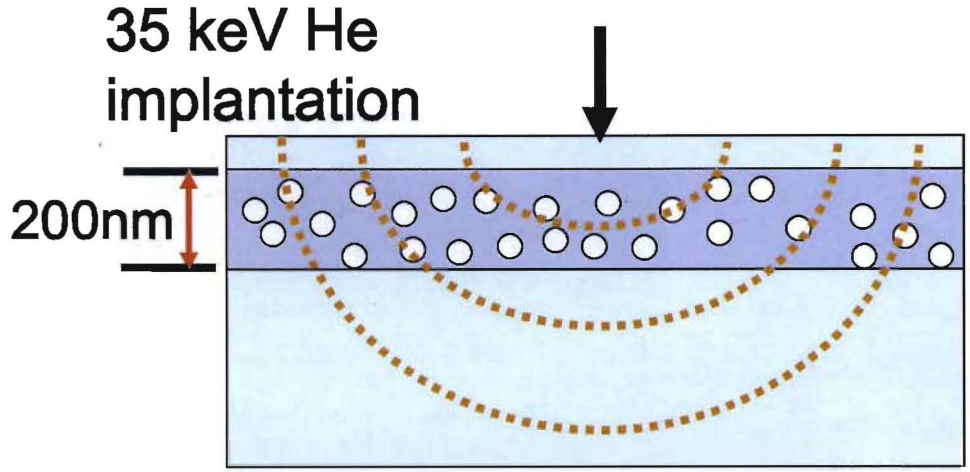
Pure metals ≈ 100-1000 appm

Σ3{111} coherent twin boundary <0.1 at.%

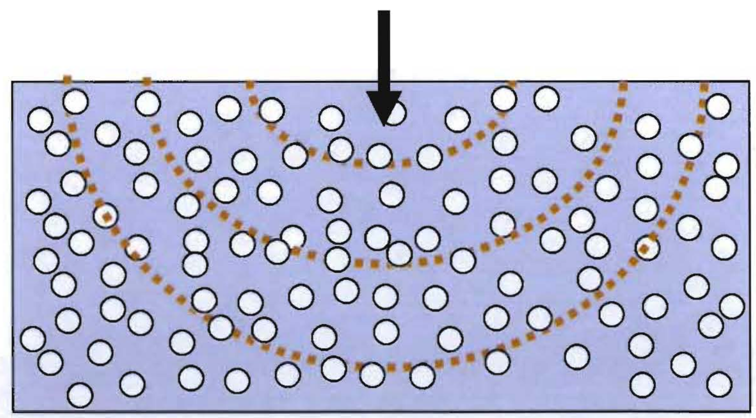
Cu-V (2.5 nm) ≈ 1 at.%
Cu-Mo (5 nm) ≈ 0.7 at.%; estimate ≈ 1.4 at.% for 2.5 nm layers
Cu-Nb (2.5 nm) ≈ 4 at.%
V-Ag (2.5 nm) ≈ 4-5 at.%

Objective #2: Explore the effect of ion implantation induced defects (particularly He bubbles) on irradiation hardening

Implantation with a single ion energy produces a narrow zone containing He bubbles.



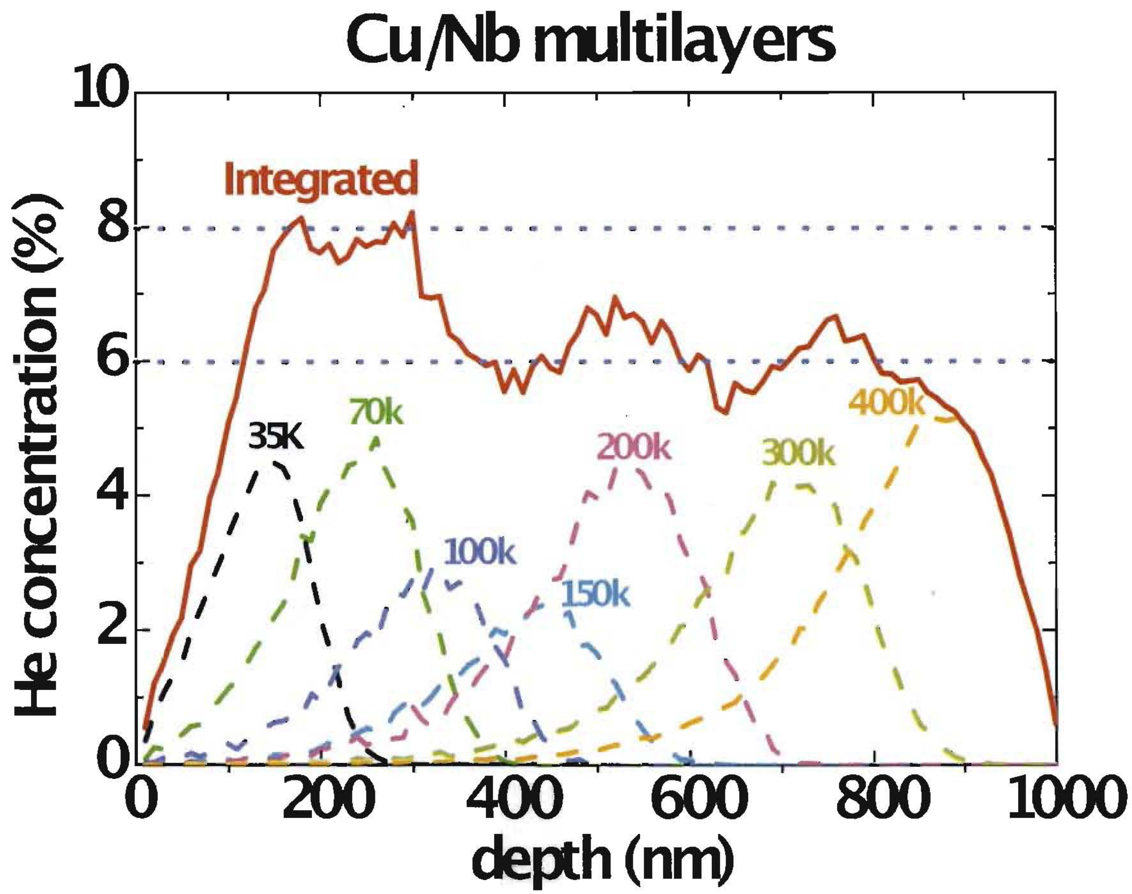
Desired bubble distribution for measuring mechanical response.



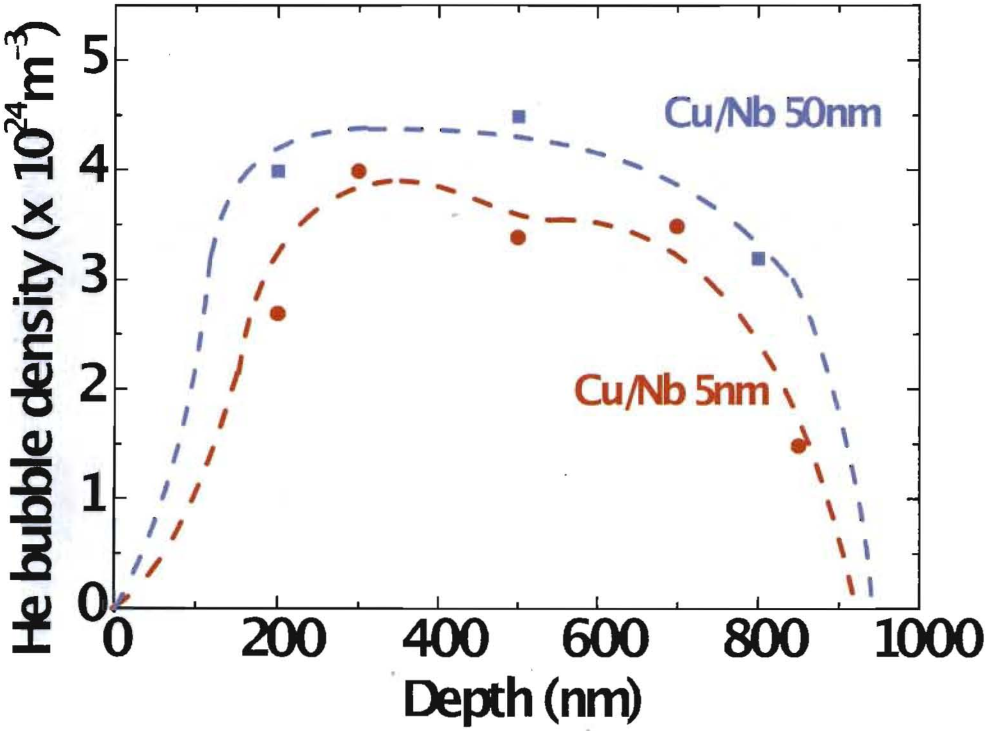
Experiment design: A series of implants of different energies were used to produce uniform helium bubble distribution over 1 um depth

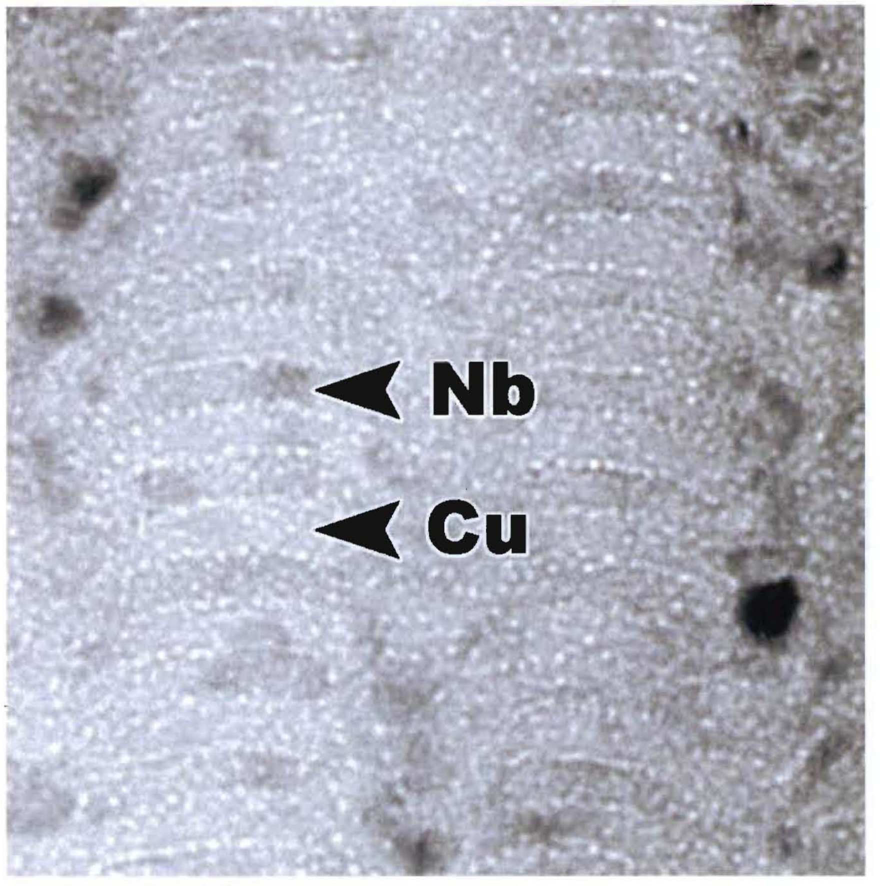
Sample: Cu, Nb,
Cu/Nb 50 nm, Cu/Nb 5 nm,
Cu/Nb 2.5 nm.

#1: 400 keV - 9 $\times 10^{16}$ ion·cm^{-2}
#2: 300 keV - 7 $\times 10^{16}$ ion·cm^{-2}
#3: 200 keV - 7 $\times 10^{16}$ ion·cm^{-2}
#4: 150 keV - 3.5 $\times 10^{16}$ ion·cm^{-2}
#5: 100 keV - 4 $\times 10^{16}$ ion·cm^{-2}
#6: 70 keV - 6 $\times 10^{16}$ ion·cm^{-2}
#7: 35 keV - 4 $\times 10^{16}$ ion·cm^{-2}



He bubble density as measured from TEM images decreases with decreasing layer thickness

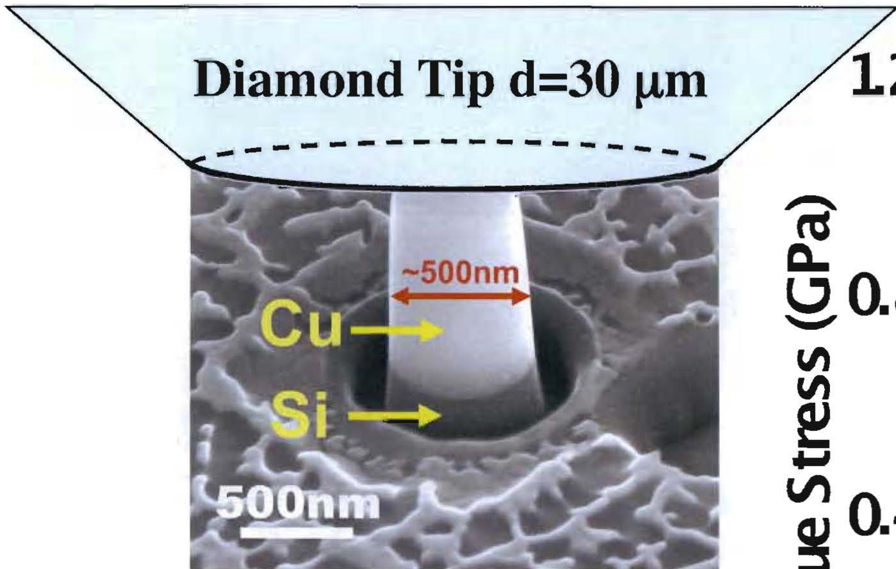




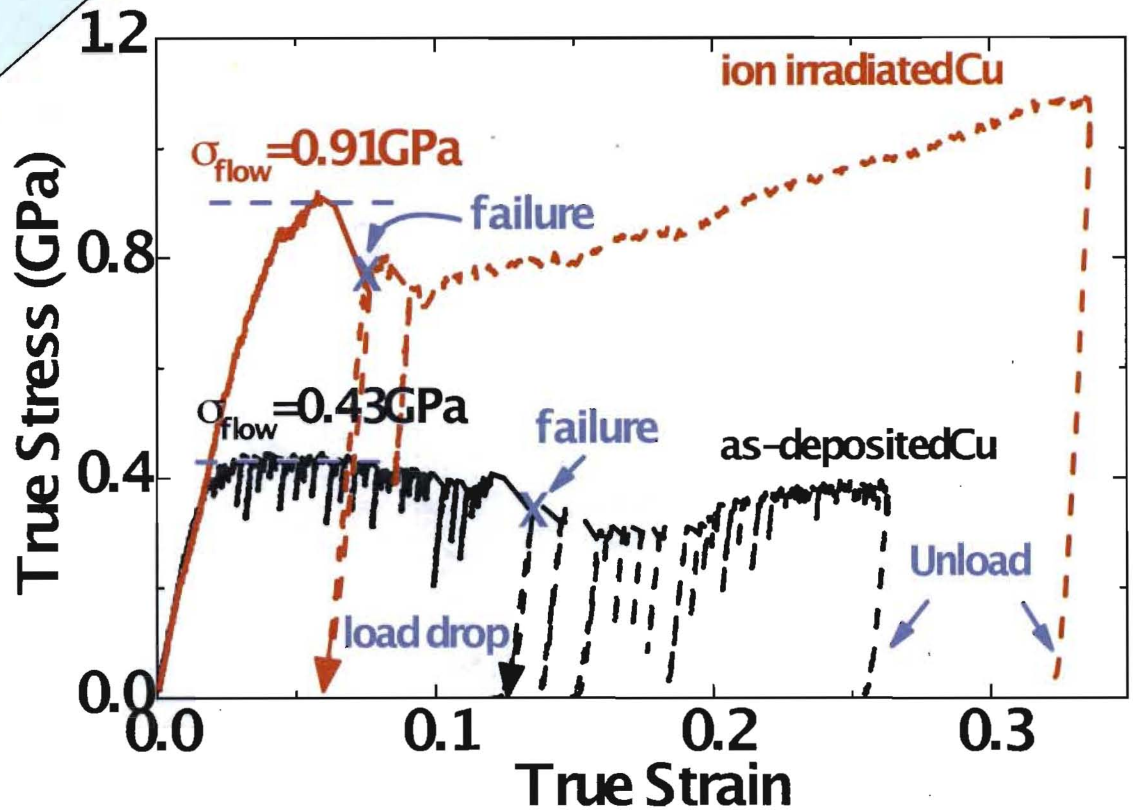
Assuming the thickness of sample is ~ 30nm

Pillar compression test – sputtered Cu with He bubbles

Significant hardening with reduced deformability

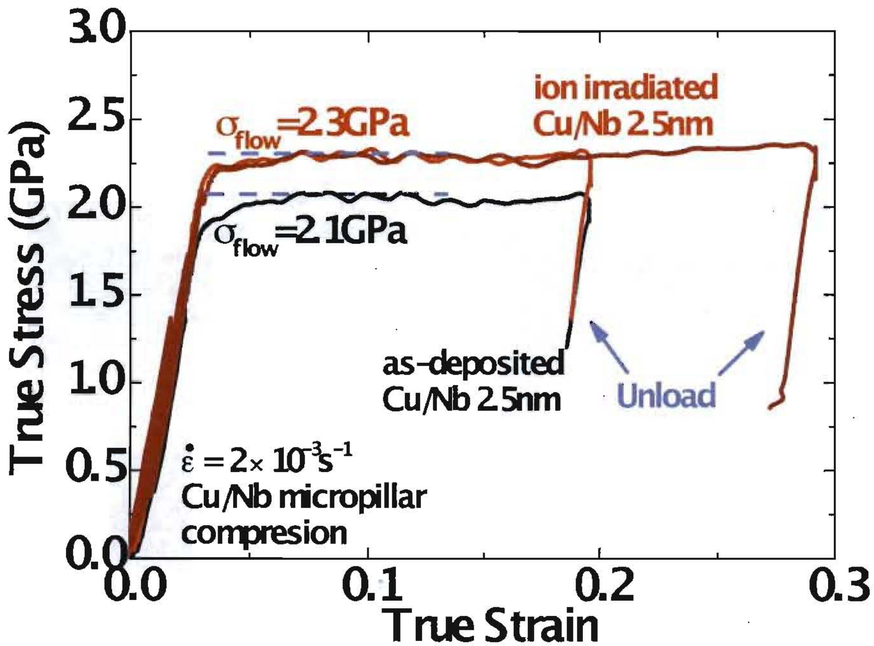


Displacement control
(Hysitron Tribo Indenter)

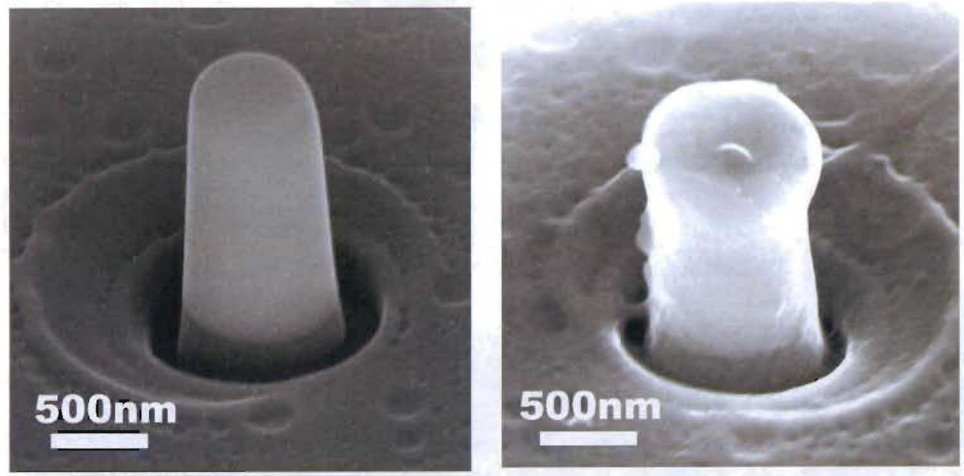


Test methodology: Uchic, Dimiduk, Florando, Nix, *Science* (2004).

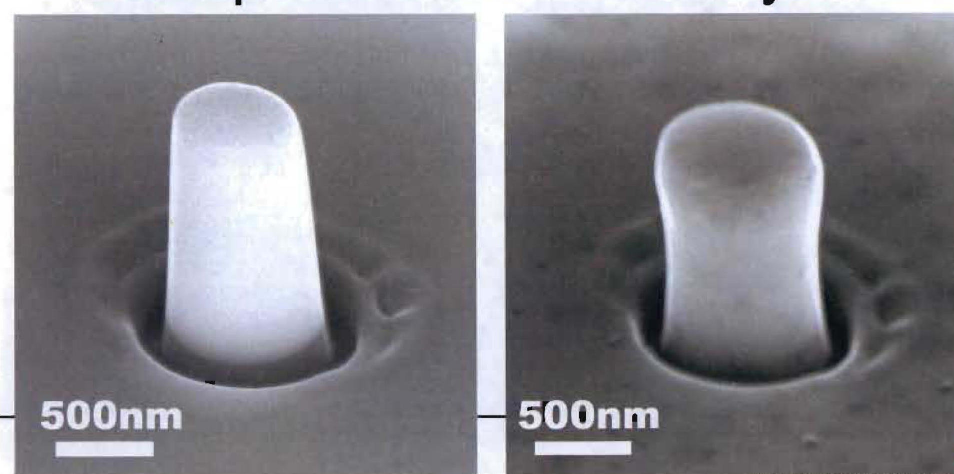
In multilayers, irradiation hardening decreases with decreasing layer thickness



Ion irradiated Cu/Nb 2.5nm multilayers

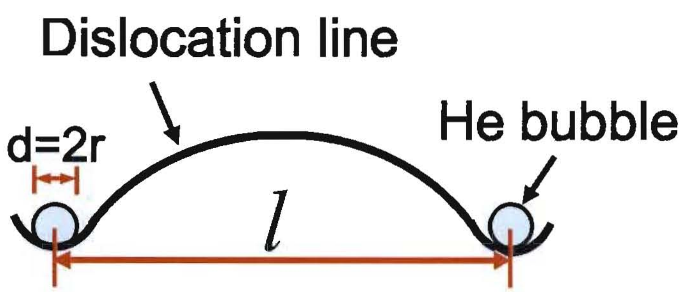


As-dep Cu/Nb 2.5nm multilayers



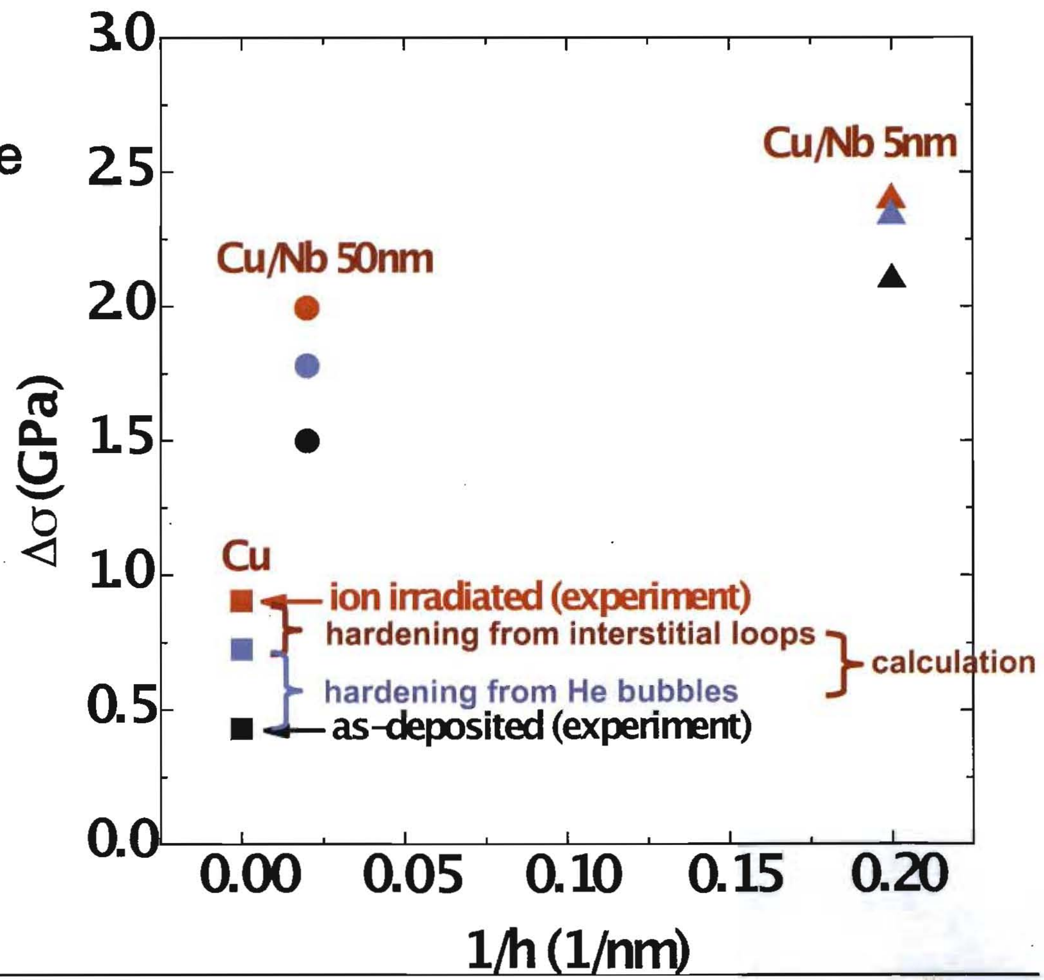
Irradiation Hardening Mechanisms

- Pressurized He bubbles



$$\Delta\tau = \alpha \frac{2\xi}{b(l-2r)}$$

$$= \alpha \frac{\mu b}{2\pi(l-2r)} \ln\left(\frac{r}{b}\right)$$



Summary

- The critical He concentration at which bubbles are resolved in TEM is orders of magnitude higher in fcc-bcc nanolayered composites than in pure metals. This implies increased He solubility at fcc-bcc interfaces that may originate from the atomic structure of the interface. By comparison, coherent twin boundaries appear to have low solubility for helium.
- Density of radiation induced bubbles and interstitial loops, and barrier strength of bubbles decrease with reducing layer thickness. Consequently, the magnitude of hardening in helium ion implanted multilayers decreases significantly with decreasing layer thickness.