Influence of Fast Neutron Irradiation on Nanostructured Metals / Alloys

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NSUF workshop 06/2010

Idaho National Laboratory

NC STATE UNIVERSITY

Collaboration with INL

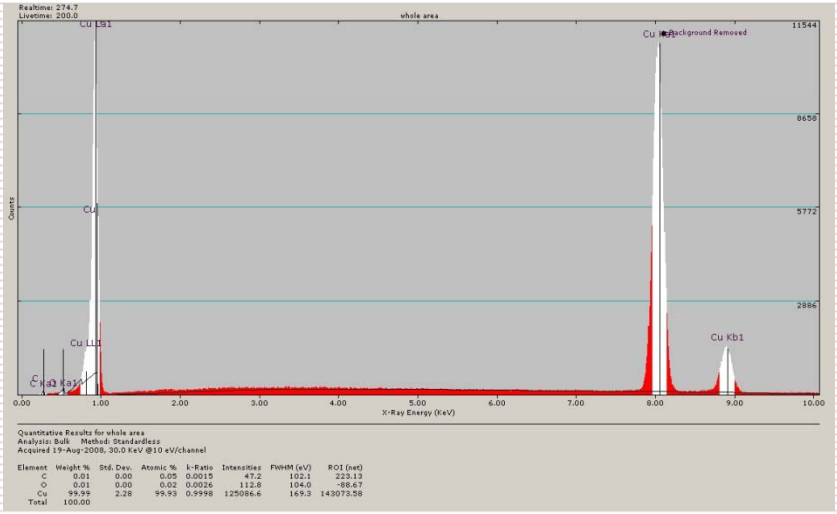
- "Influence of Fast Neutron Irradiation on Nanocrystalline Metals" was one of the first ***four*** experiments chosen under ATR/NSUF program
- Samples of nanocrystalline copper, nickel and carbon steel have been irradiated at the ATR at two different damage levels (1dpa and 2 dpa)
- Initial irradiation and post-irradiation testing at NCSU/PULSTAR (~$6x10^{17}$ n/cm^2 / 0.34 dpa)
- Samples were inserted in the ATR on March, 2009
- Irradiation at ATR has the following benefits:
  - Achieving high damage levels in short times (very short time compared to PULSTAR)
  - ATR is equipped with a hot cell for testing irradiated samples

Key Project Personnel

- DOE Federal Project Director: *Bob Boston*
- Project Manager: *Davis Duncan (current), Julie Foster (former)*
- Principal Investigator: *Doug Porter*
- Designer/Fabricator: *Tim Hyde*
- QA Manager: *Carla Dwight (Project Quality Engineer: Dana Cooper)*
- ATR Engineering POC: *James Williams*
- ATR Nuclear Safety Engineering POC: *Terry Tomberlin*
- Nuclear Operations Management: *Max Heberling*
- Facility Managers: *Martin McDonough, ATR; Dave Swank, IRC Manager for Fabrication; Richard Cain, HFEF; Marsha Lambregts, MFC Analytical Laboratory; Tom O'Holleran, MFC EML*
- Irradiation Testing Manager: *Frances Marshall*
- ATR NSUF Manager: *Mitch Meyer*

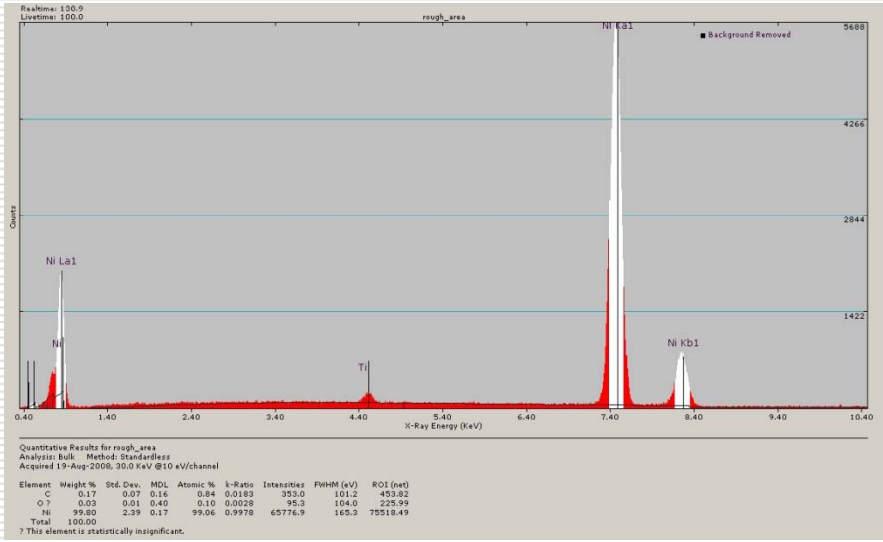
Irradiation at ATR

- Materials: nc-Cu, nc-Ni, uf- Carbon Steel



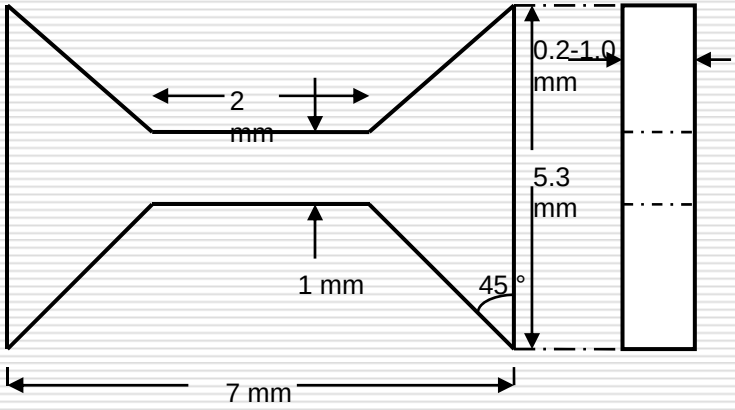
SEM-EDS nc-Cu

SEM-EDS nc-Ni

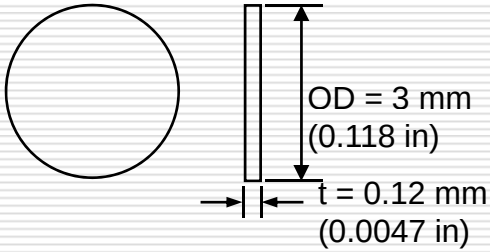


Irradiation at ATR

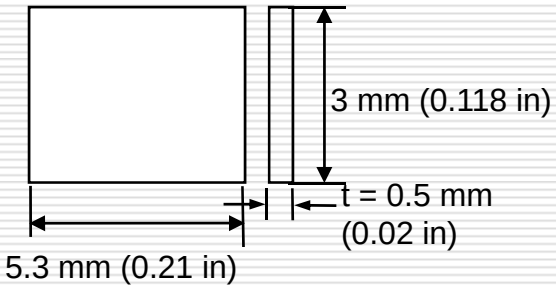
- Materials: nc-Cu, nc-Ni, uf- Carbon Steel.
- Samples:



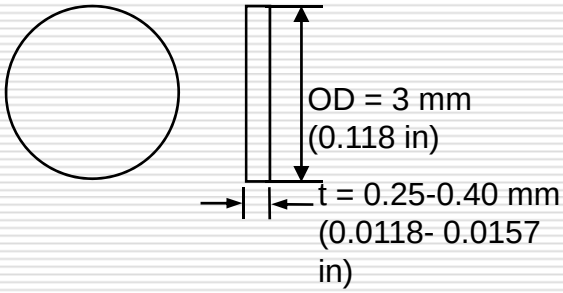
Sub-size tensile sample



TEM specimen



Hardness sample



SPT specimen

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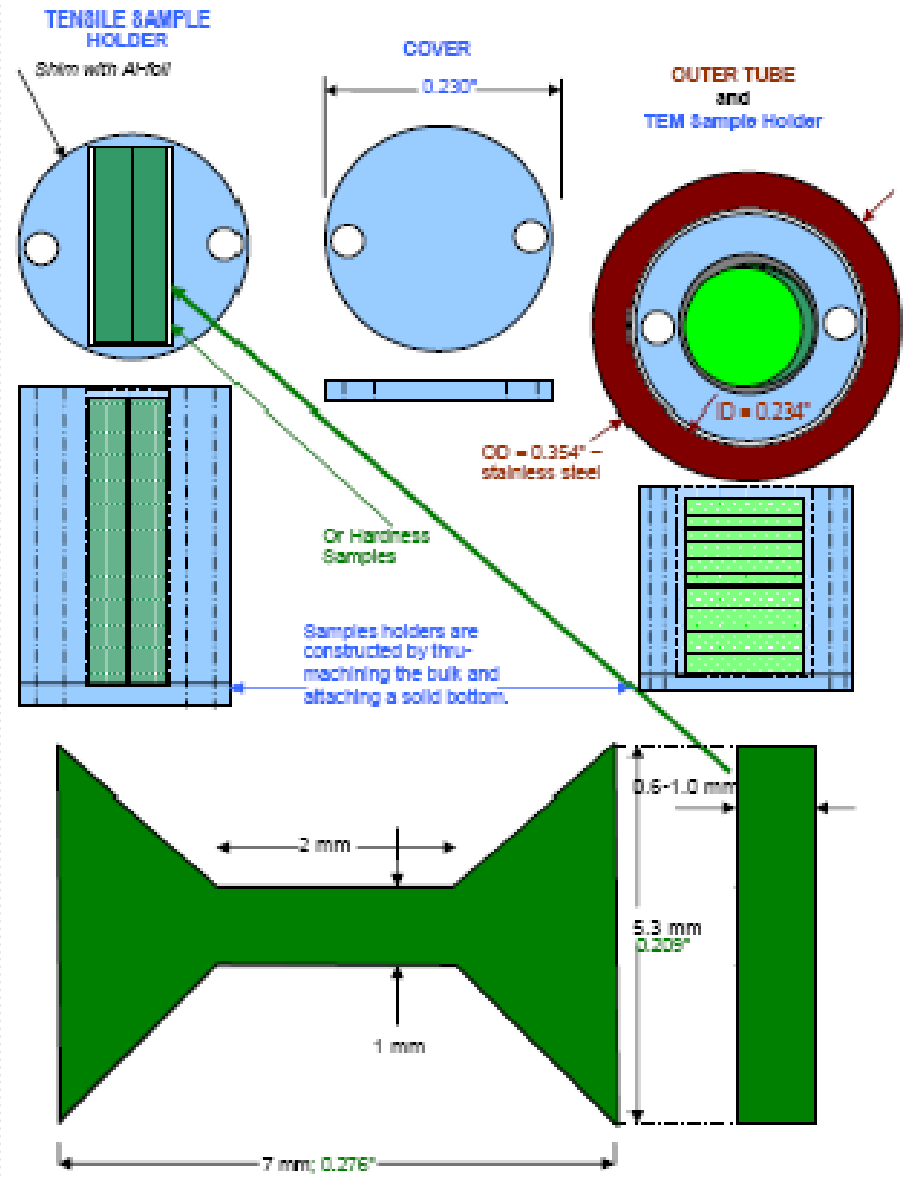
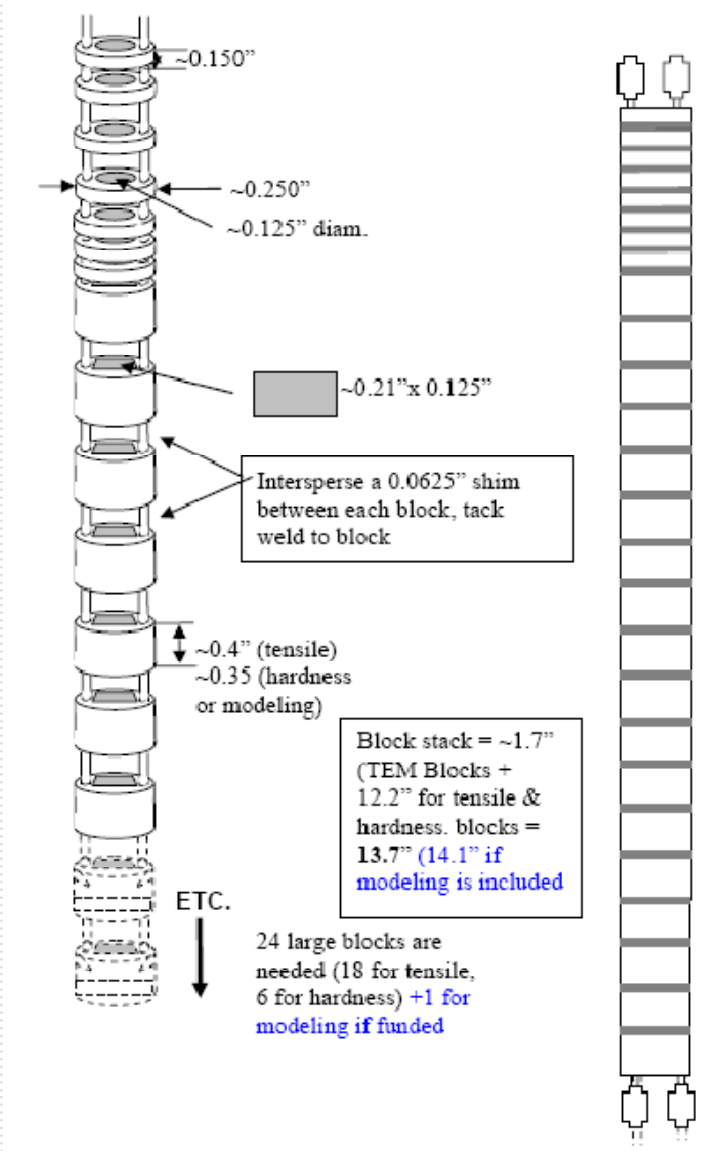
Irradiation at ATR: Sample/Holder Design



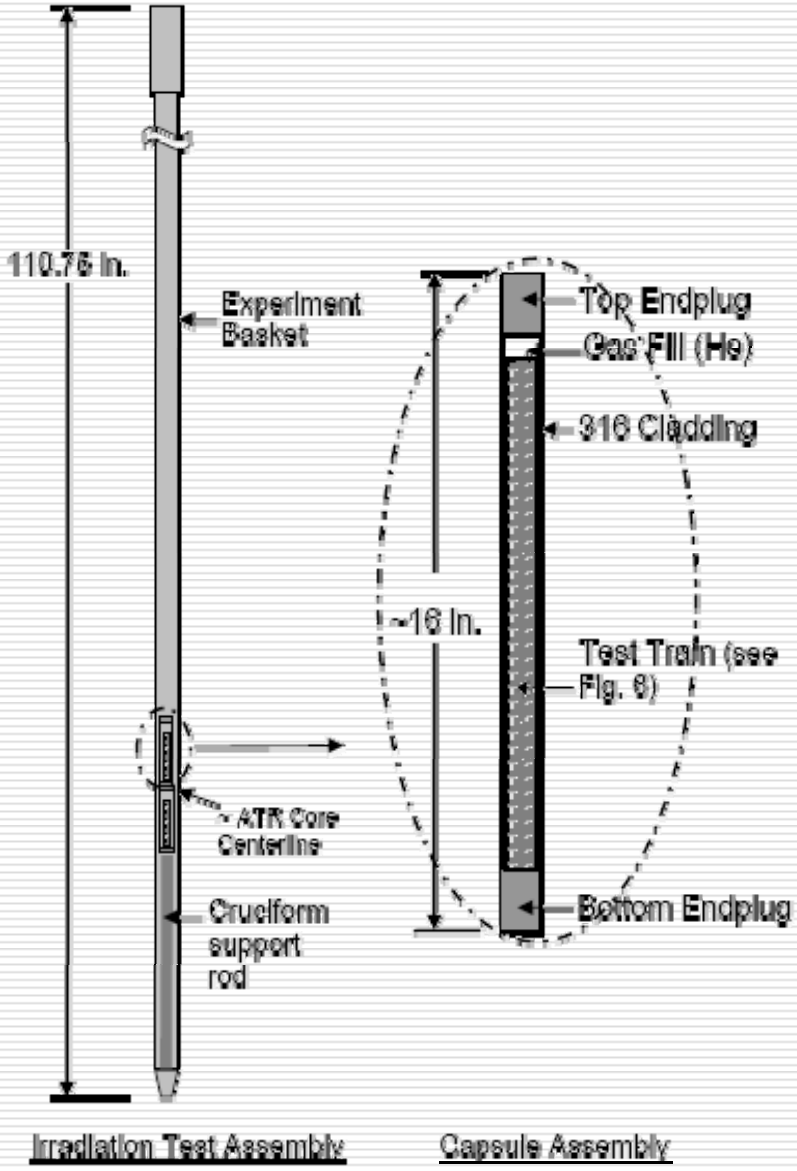
Sample/Holder design Coordination:
Capsule Tube and End Plugs

Irradiation at ATR



Test Train Base Design

Each test train was sealed in a stainless steel capsule to prevent contact with the water coolant



NCSU Experiment Irradiation Test Assembly for ATR East Flux Trap Position

Project Major Milestone

Finalize Experiment Configuration	July, 2008
Complete Hardware Fabrication	October, 2008
Complete Experiment Assembly	November, 2008
Ship & Insert Experiment in ATR	March, 2009
Irradiation – ATR Irradiation – PULSTAR (part II)	March 2009 – Jan 2010 April 2010 – Dec 2010
Post-irradiation Examination – I PIE – II (ATR & PULSTAR)	Feb 2010 – Jul 2010 CY2011
Final Project Report	Aug 2011
Project Closeout Complete	Aug 2011

Irradiation Experiment Conditions

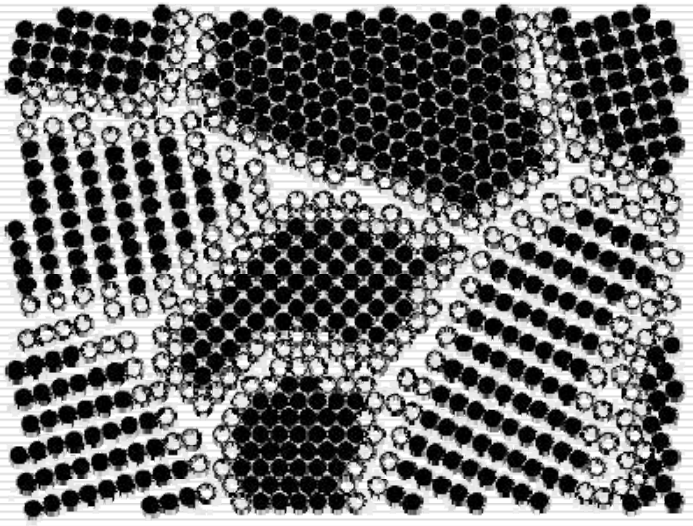
- Irradiation Position: the center position of the EFT (E-7).
- Design Temperature: < 200°C
- Calculated Temperature: using the MCNP Coupled with ORIGEN2 (MCWO) analysis methodology.
- Thermal Hydraulic Analysis: T = 65°C - 85°C
- Damage levels:
  - NCSU-1 capsule: *1 dpa (two ATR cycles)*
  - NCSU-2 capsule: *2 dpa (four ATR cycles)*

Irradiation Experiment Conditions

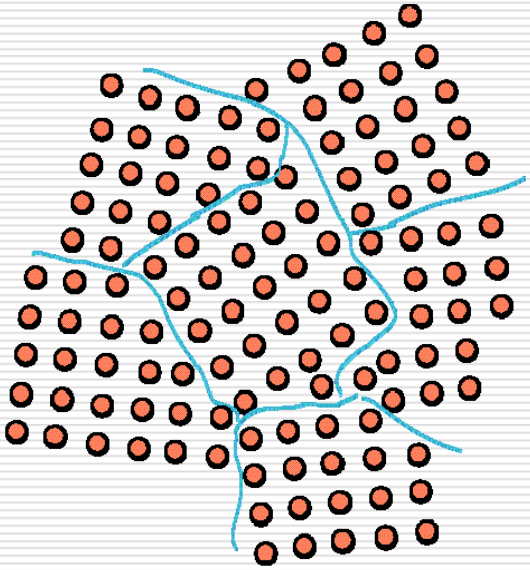
- Post-Irradiation Examination (PIE):
  - Microstructure (TEM, XRD)
  - Mechanical Testing (Tensile, Microhardness)
  - SPT (U Idaho – R. Prabhakaran, INL)
  - AFM (to be conducted at NCSU)

Introduction

- Highly radiation resistant materials are needed for new generation of nuclear reactors
- Nanostructured metals are characterized by the presence of large volume of interfaces / grain boundaries that may act as sinks for radiation-produced defects
- Nanostructured metals ***are thus expected to*** possess radiation tolerant characteristics



Nanostructured



Conventional

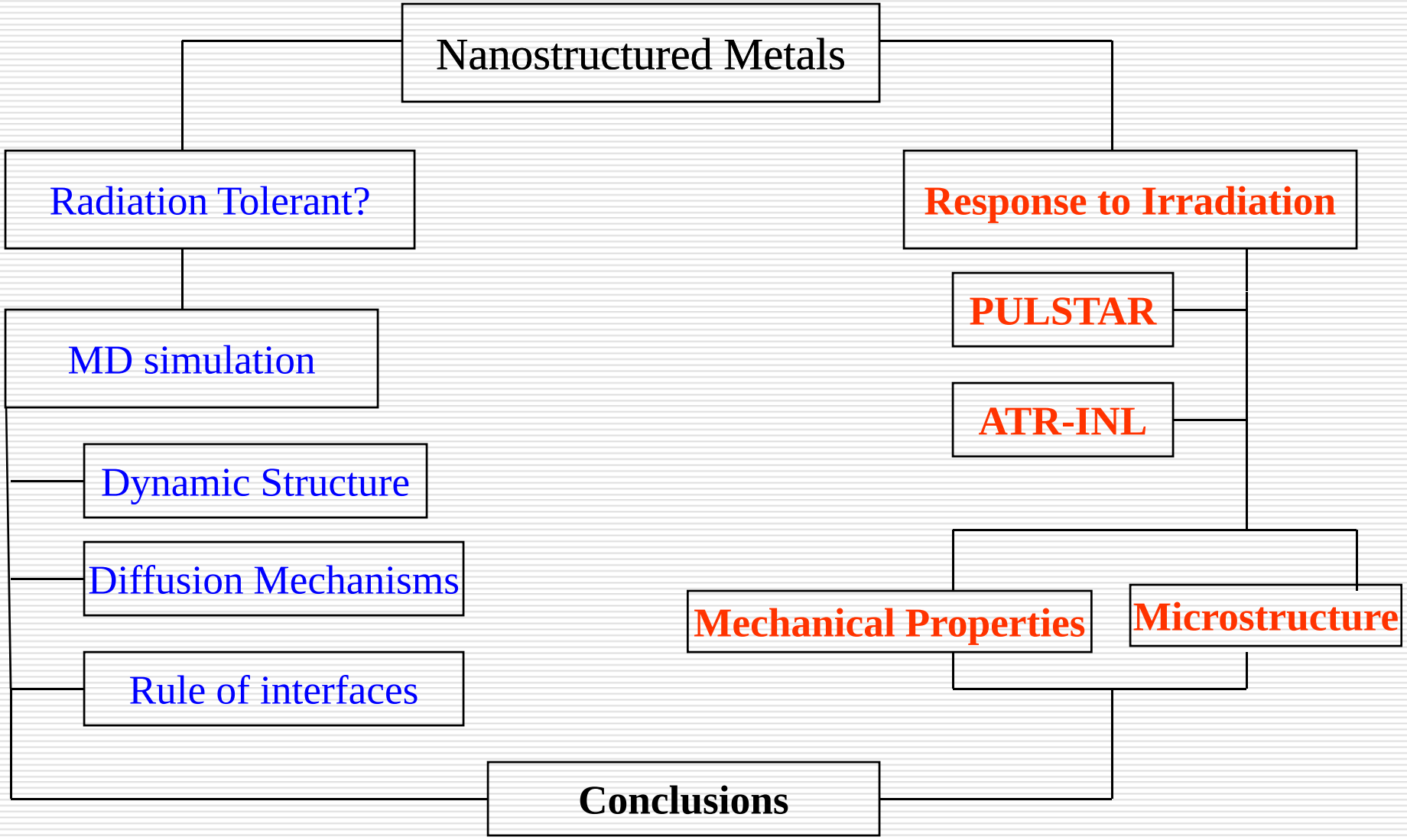
Objectives

1. To investigate radiation effects on mechanical properties of nanostructured metals

2. To compare radiation effects on nanostructured metals vs conventional metals

3. To understand deformation mechanisms of nanocrystalline materials and effect of neutron irradiation

Research Scope



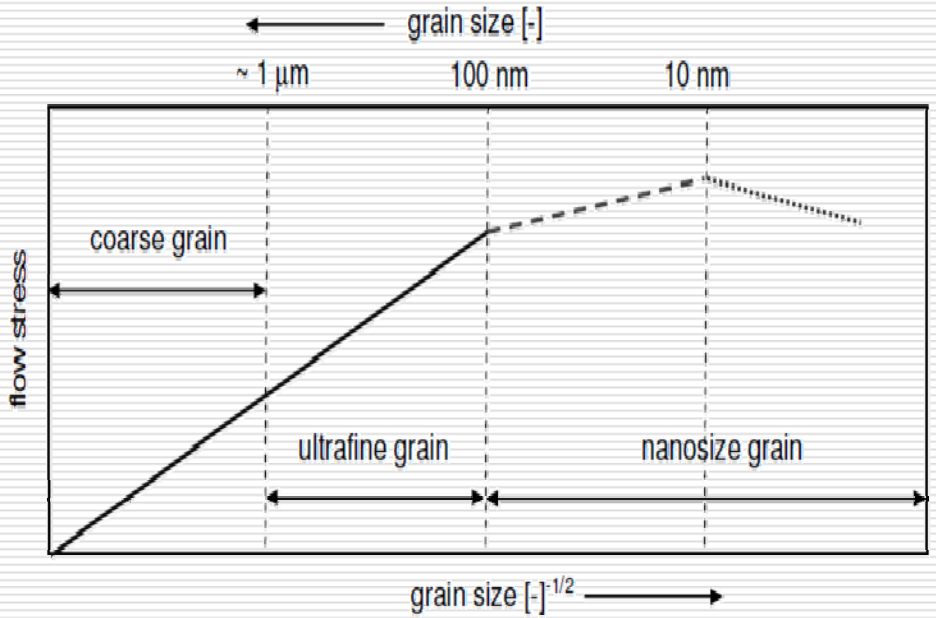
Phase One

Irradiation at NC State University

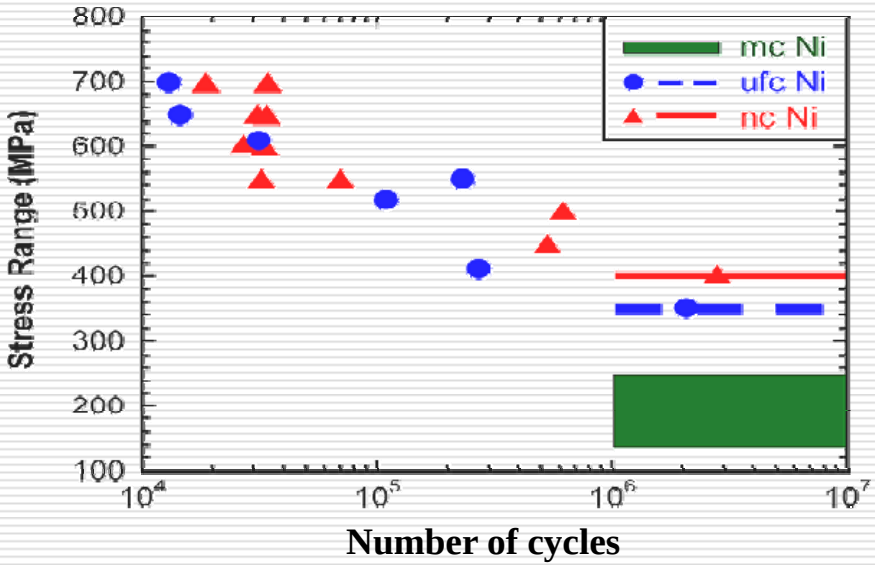
Status: Completed

Mechanical Properties of Nanostructured Metals

- Some appealing properties of nanostructured metals
  - Ultra-high yield and fracture strengths
  - Superior wear resistance



Katrina et al./ METAL 2009, 19. – 21. 5. 2009, Hradec nad Moravici



K. S. Kumar et al./ Acta Materialia 51 (2003) 5743-5774

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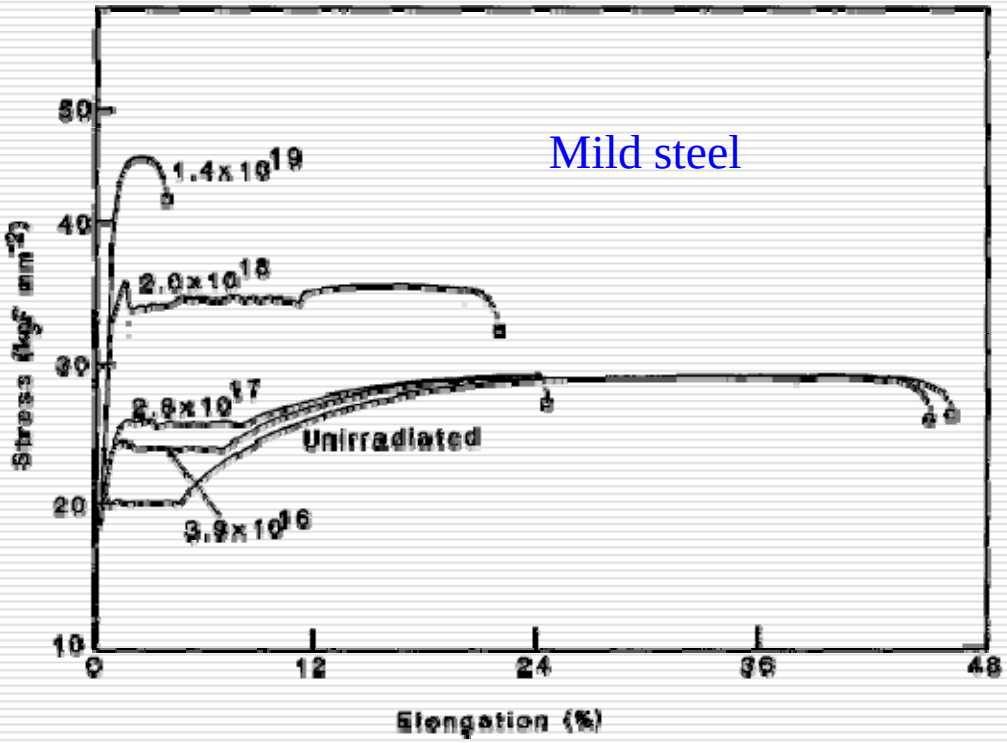
Radiation Impacts on Conventional Materials

- Irradiation induced defects
  - Vacancies
  - Interstitials
  - Thermal spikes
  - Displacement spikes
  - Dislocation loops
  - Voids
  - Cavities.
- These defects strongly affect the mechanical properties



K. L. Murty, Materials Science and Engineering, 59 (1983) 207-215

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Radiation Impacts on Nanostructured Metals

- Nanostructured Ni and Cu-0.5Al_2O_3 samples synthesized by severe plastic deformation (SPD). Damage levels were *0.56 dpa for Ni* and *0.91 dpa for Cu-0.5Al_2O_3*.

- Nanostructured Ni
  - Highly strained regions were observed in irradiated samples
  - The average grain size *decreases* following proton irradiation to 0.56 dpa
  - Twin boundaries were observed in irradiated material with higher density than it in the unirradiated material.

- Nanostructured Cu-0.5Al_2O_3
  - Cu-0.5Al_2O_3 exhibited *grain growth* as a consequence of irradiation.
  - Stacking fault and *low density of dislocations* were observed in the irradiated Cu-0.5Al_2O_3.

N. Nita, R. Schaeublin, M. Victoria. Journal of Nuclear Materials 329-333 (2004) 953-957

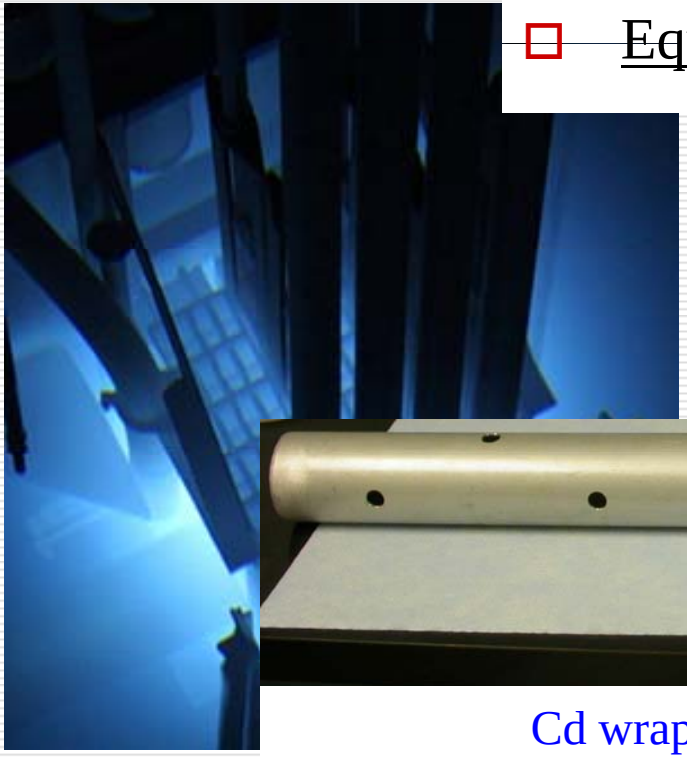
Phase One

Irradiation at NC State University

- The average fast flux component was found to be 6.6×10^{11} n/cm^2.s ch reactor
- Irradiation time: 249.87 hrs
- Equivalent Fluence: 5.9×10^{17} n/cm^2

^{58}Ni (n , p) ^{58}Co

- *Cd* wrapped *Al* irradiatio reduced thermal neutron abso



Cd wrapped Al irradiation column

Irradiation jig

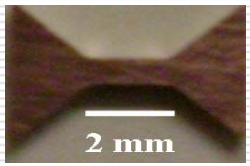
Materials and Samples

Material

- Nanostructured copper
- In fusion reactors, Cu is considered to be a good candidate for first-wall applications

Samples

1. Sub-size tensile samples (Tensile testing)
2. Square samples (TEM / XRD / Microhardness ….etc)

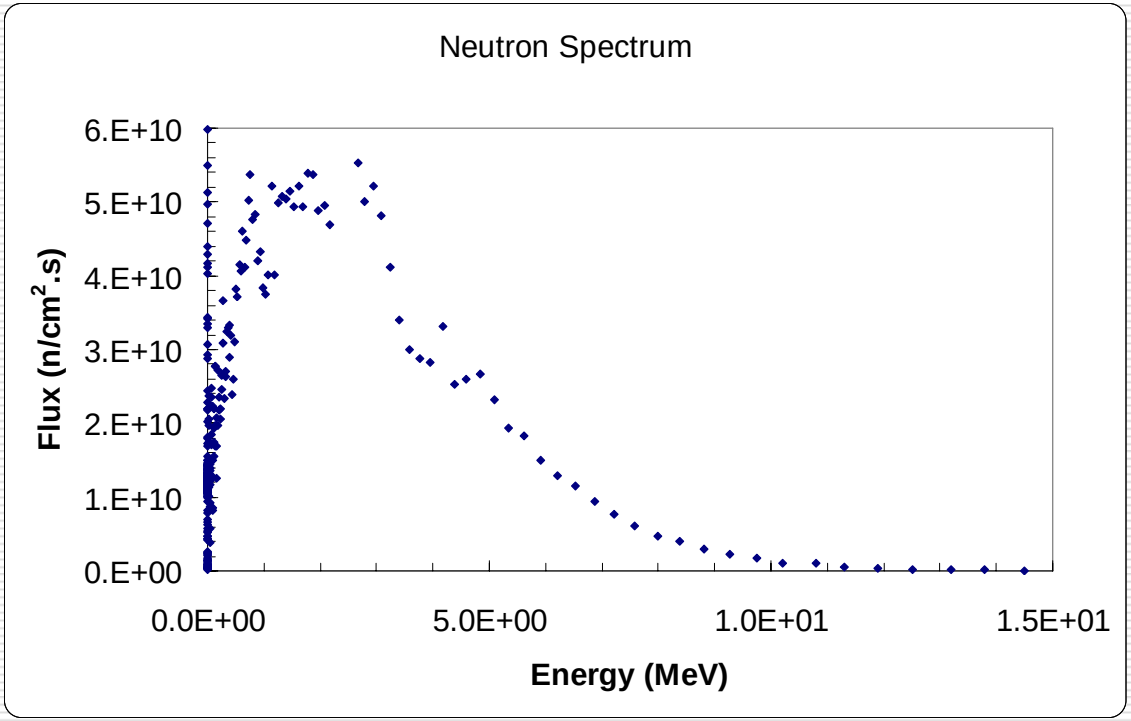


(1)

(2)

dpa Calculations

- Neutron Spectrum simulation (MCNP)*



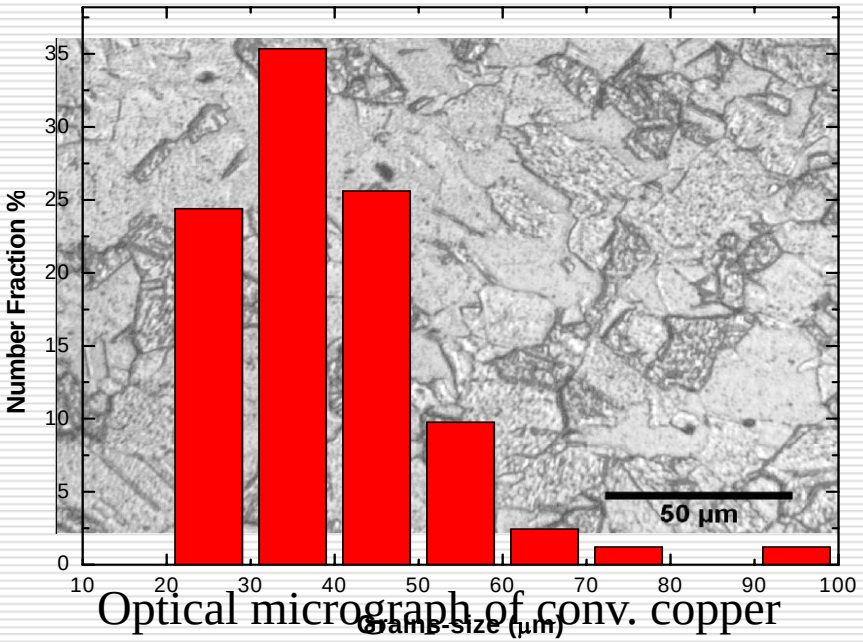
- ***dpa*** = 0.34 (estimated using Kinchin-Pease model)
- Cross-sections data was generated via the *"Evaluated Nuclear Data File (ENDF)"* provided by Brookhaven National Laboratory (BNL)

Integration of flux from E = 2 MeV to the end of spectrum = 6.1×10^{11} (n/cm^2.s) which is of good agreement with the activation data

* Acknowledging Mr. Trey Hathaway for providing the MCNP data

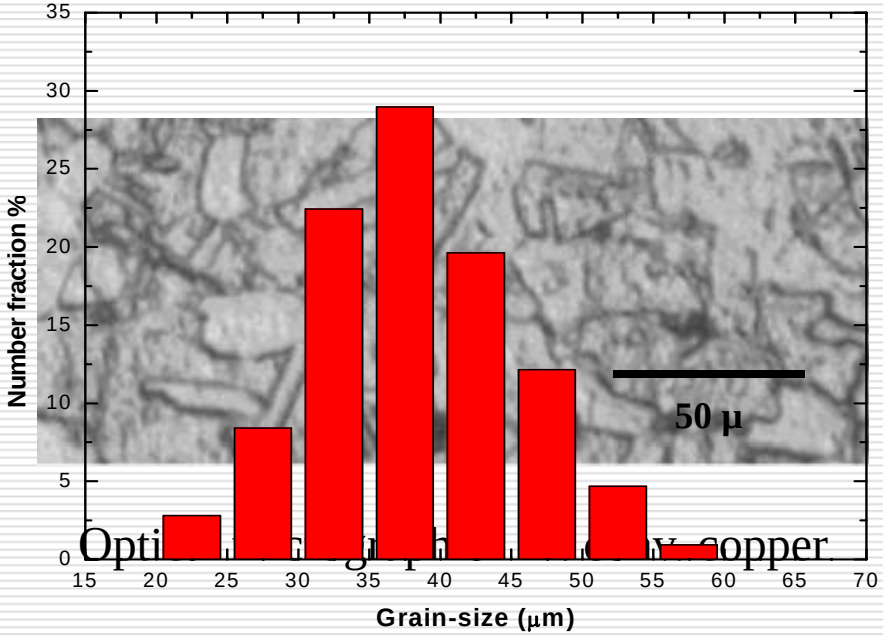
Microstructure Examination

- Conventional Copper



Optical micrograph of conv. copper

GSD of conventional copper showing average GS of (38.24±12.24) µm



Optical micrograph of irradiated conv. copper

GSD of irradiated conventional copper showing average GS of (39.1+7.1) µm

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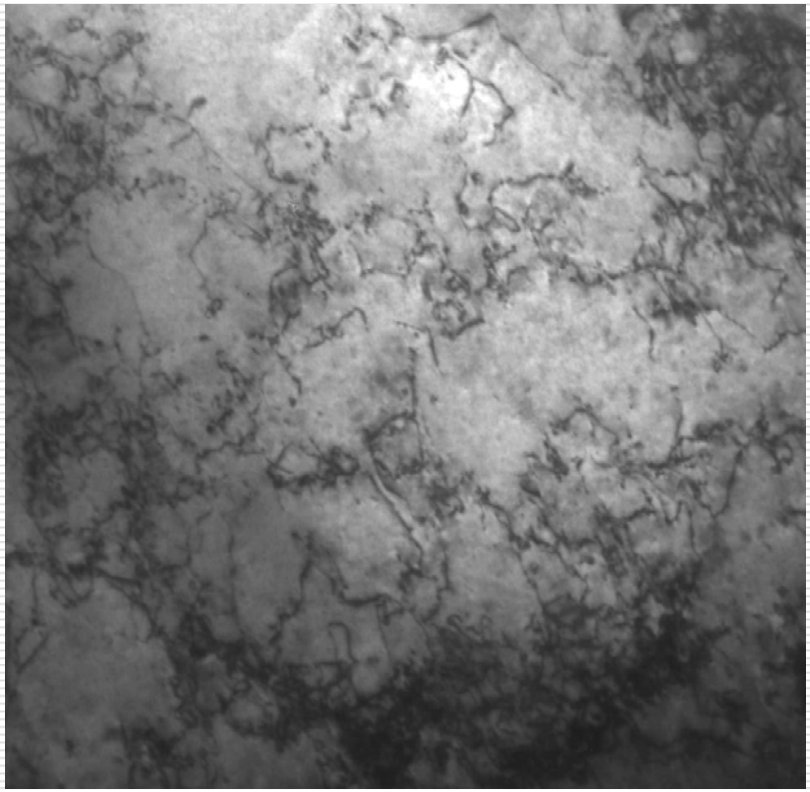
Microstructure Examination

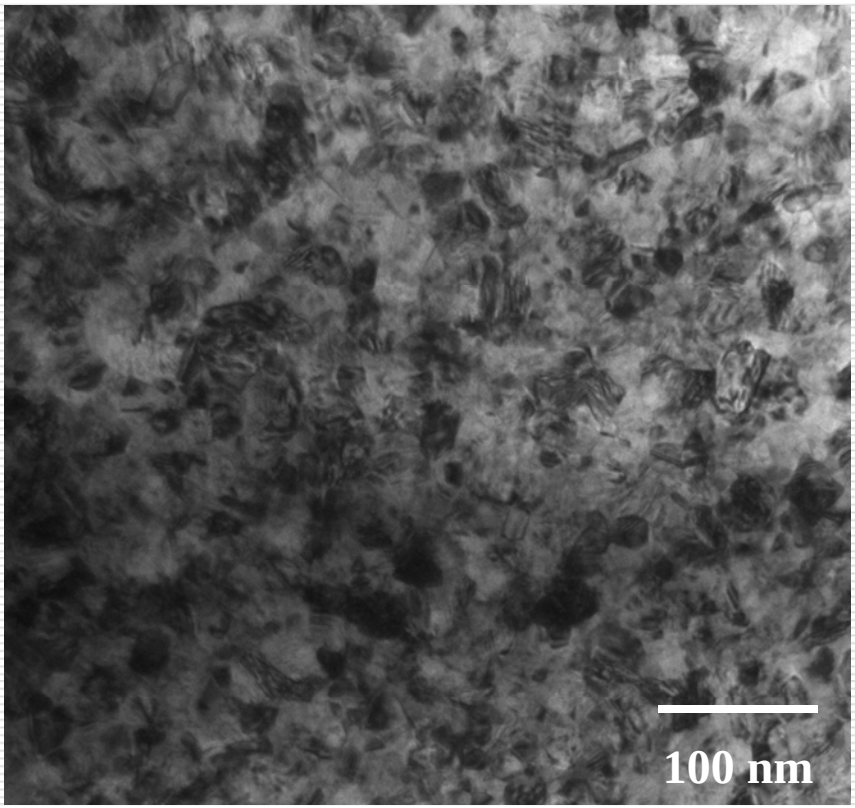
- Conventional copper - Irradiated



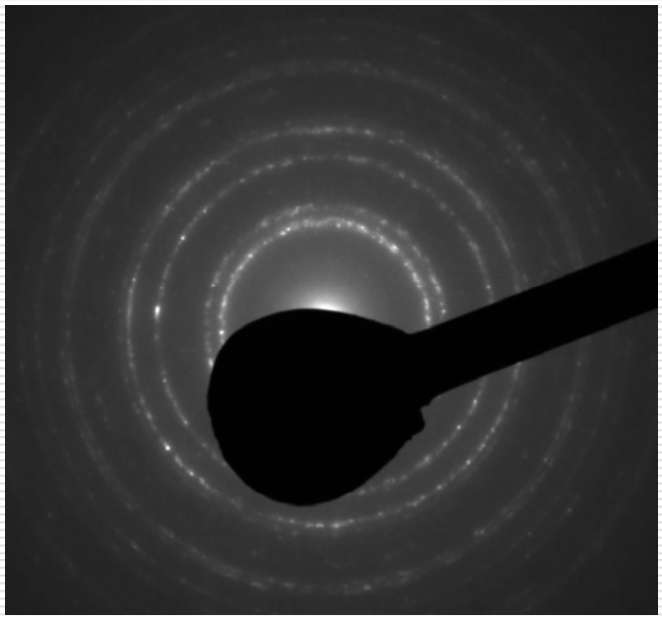
BF TEM micrograph of irr. conv. Cu

Microstructure Examination

- Nanostructured Copper



Bright Field (BF) TEM
of unirradiated nc-Cu

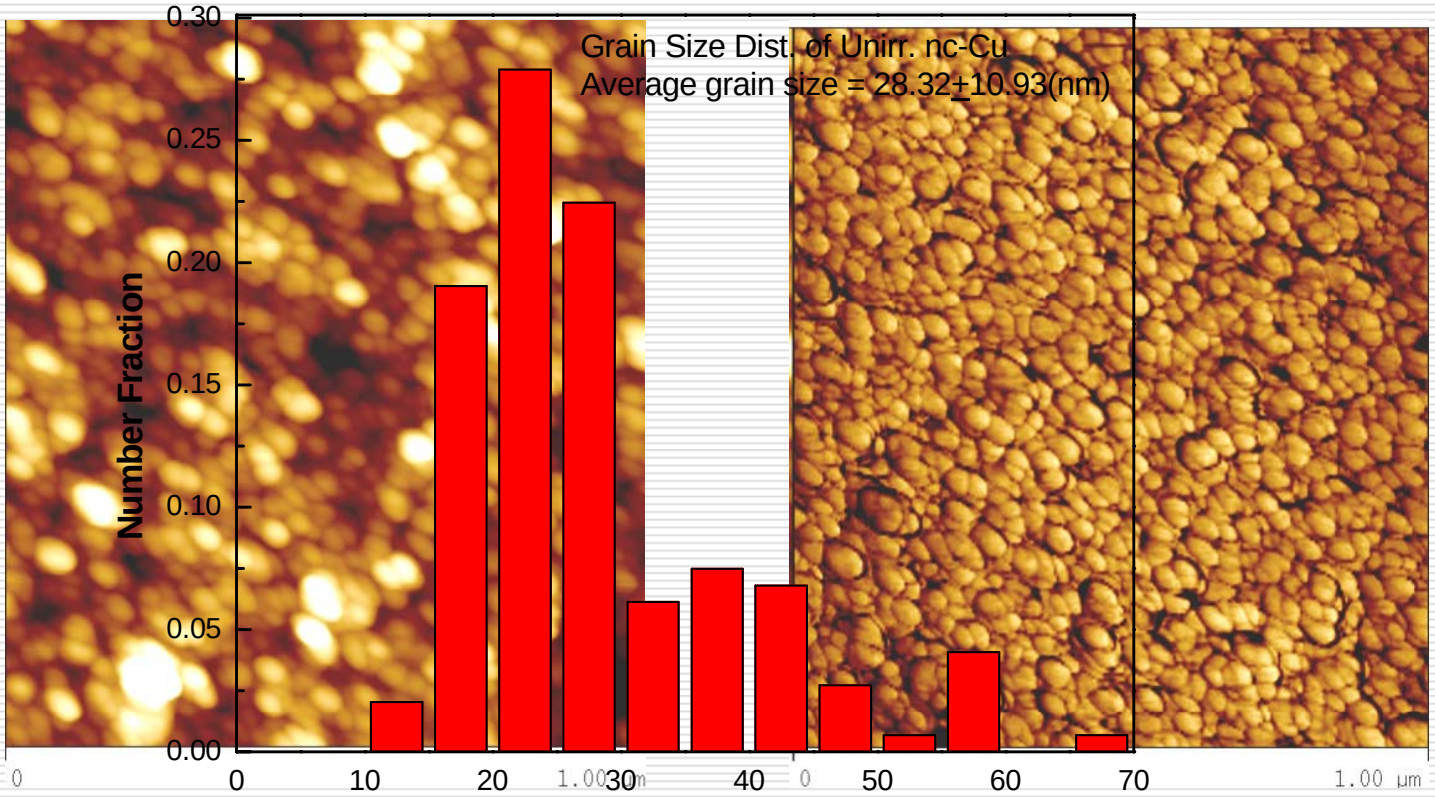


Diffraction pattern of unirradiated nc-Cu

The complete circles in the diffraction pattern indicate the majority of the grains are at the nano-scale.

Microstructure Examination

- Nanostructured Copper

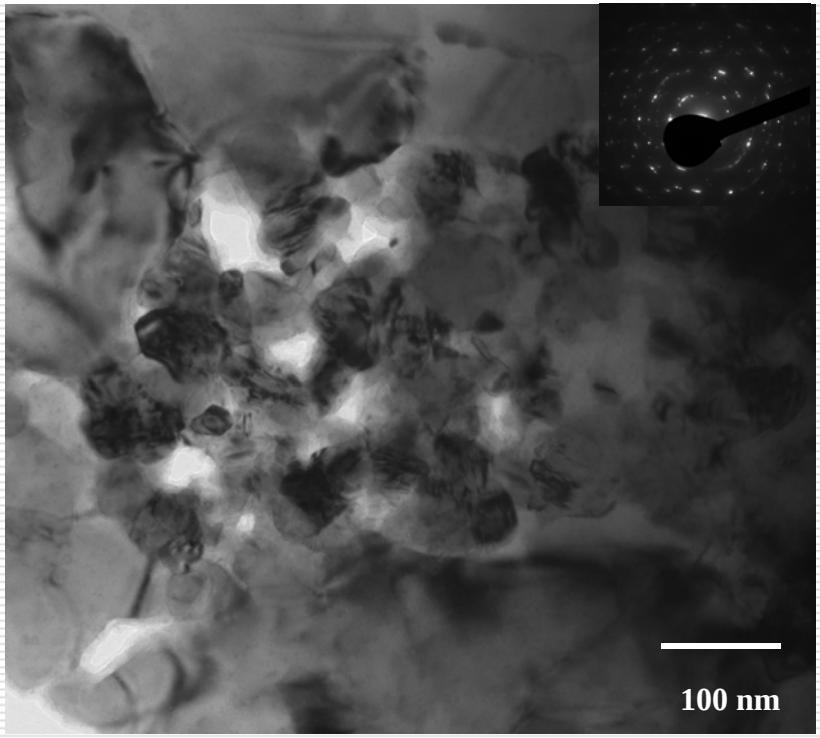


AFM of nanostructured copper

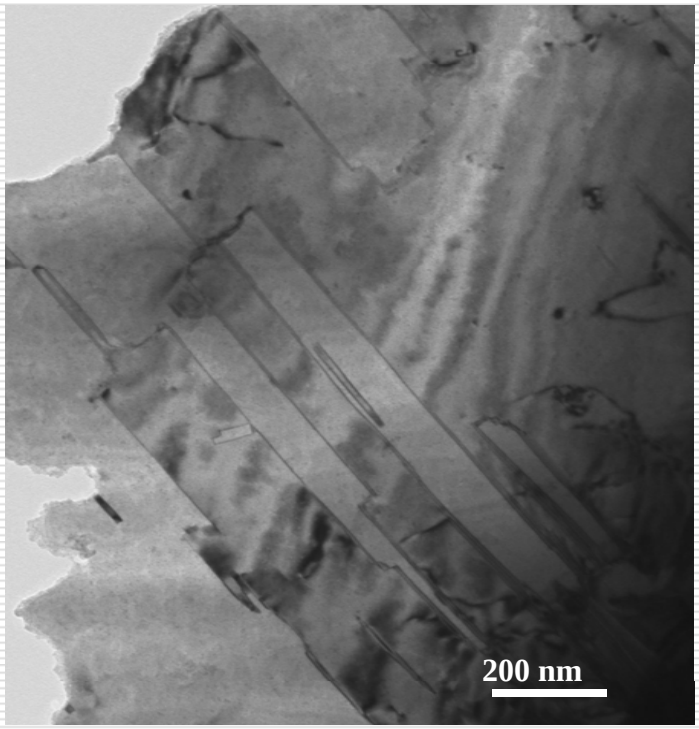
GSD of unirr. nc-Cu showing average grain size of (28.32±10.93) nm

Microstructure Examination

- Irradiated nc-Copper



BF TEM of irr. Nc-Cu

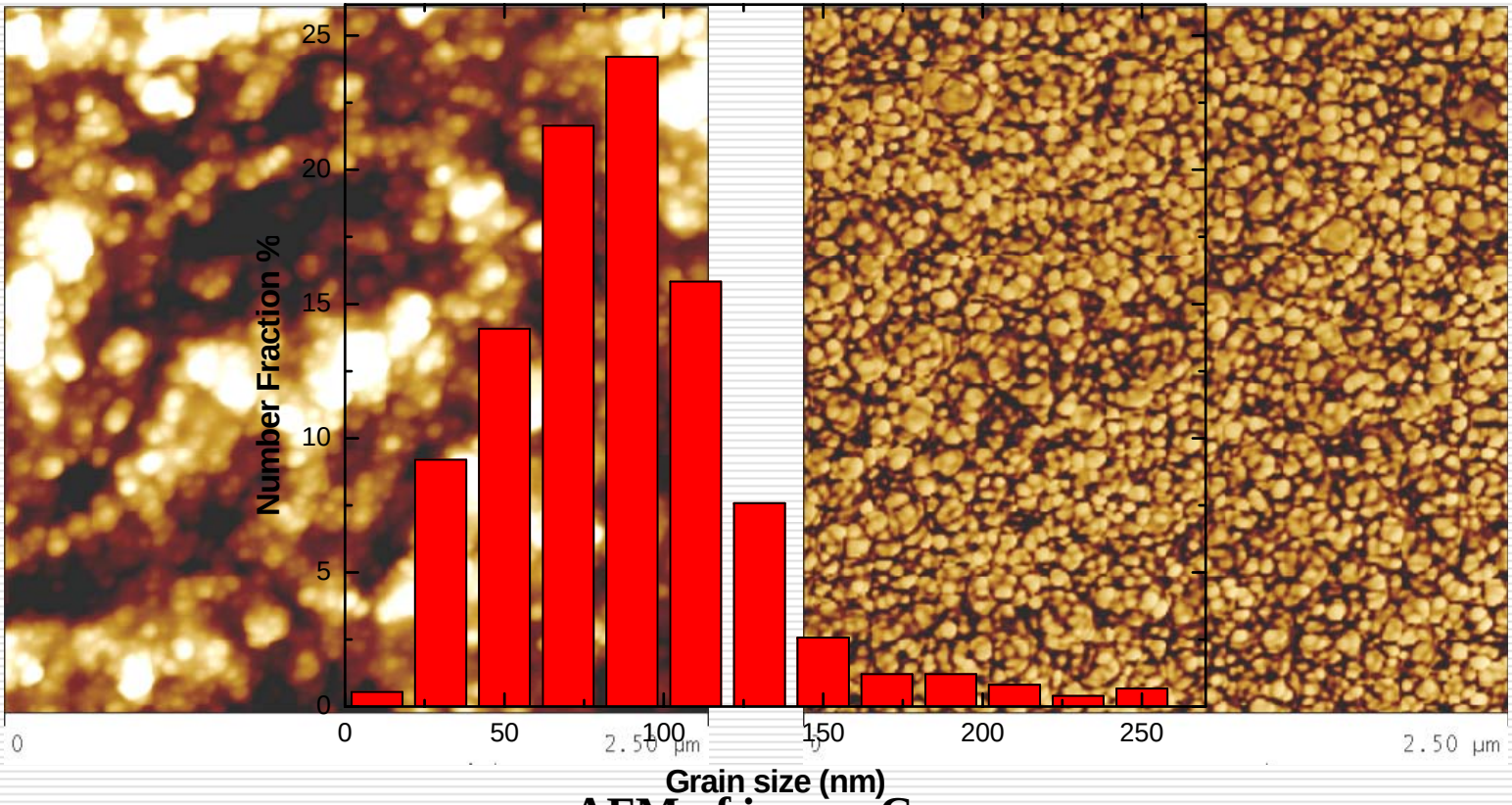


BF TEM of irr. nc-Cu showing dislocation and twin structures

Microstructure Examination

- Irradiated nc-Copper

Note increased grain size from 28 to 87 nm



AFM of irr. nc-Cu
GSD of irr. nc-Cu showing average grain size of (86.72± 38.65) nm.

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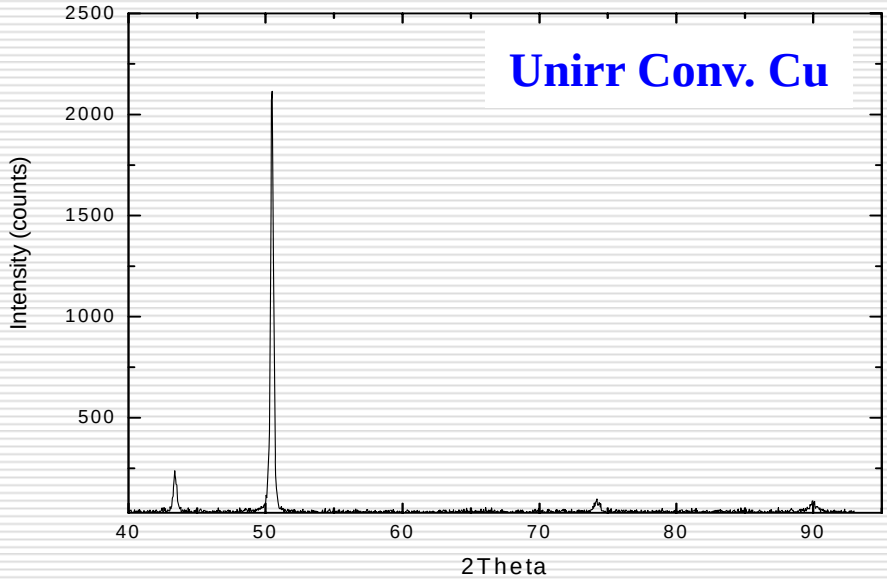
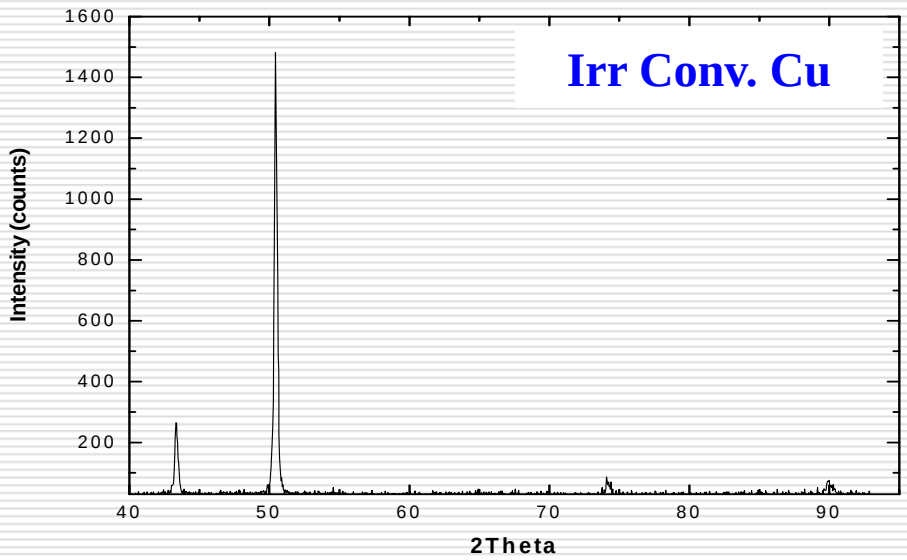
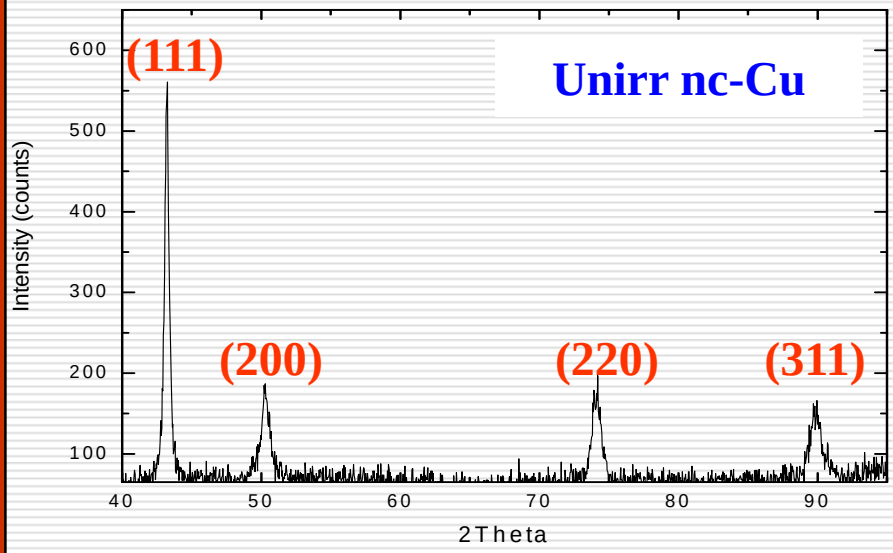
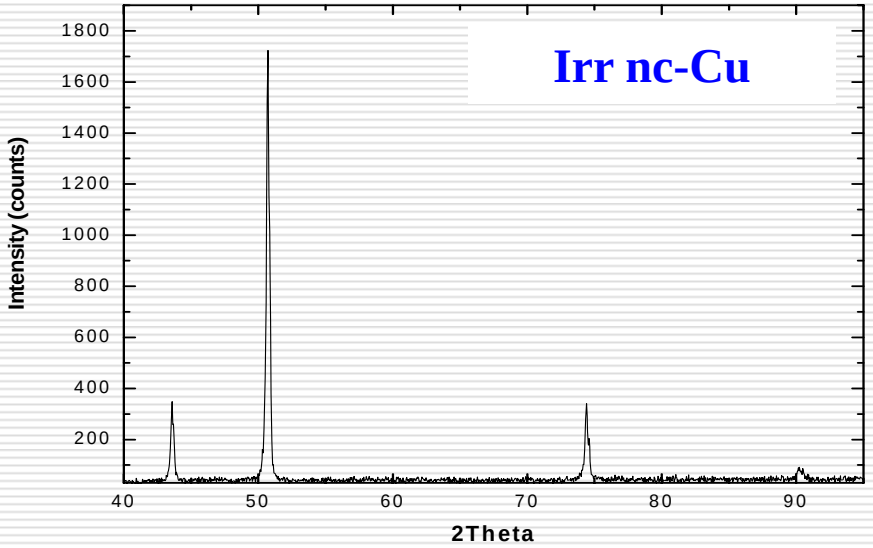
XRD

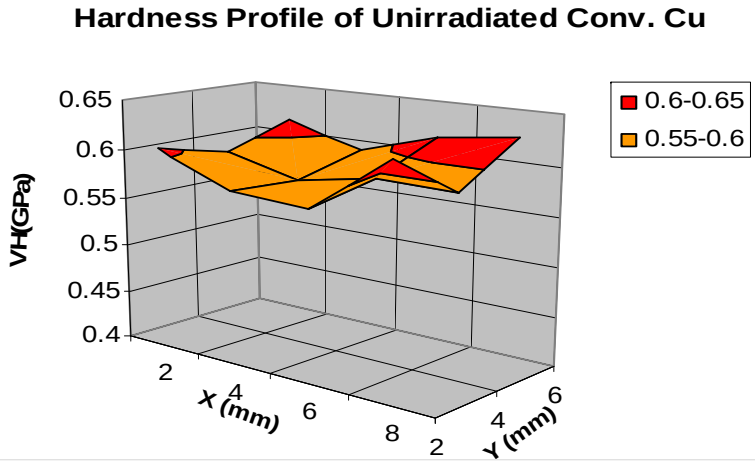

Unirr Conv. Cu
Intensity (counts)
2Theta


Irr Conv. Cu
Intensity (counts)
2Theta

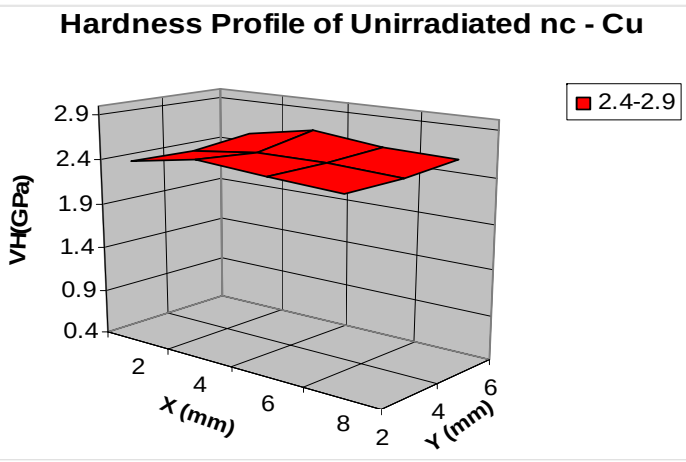

Unirr nc-Cu
(111)
(200)
(220)
(311)
Intensity (counts)
2Theta


Irr nc-Cu
Intensity (counts)
2Theta

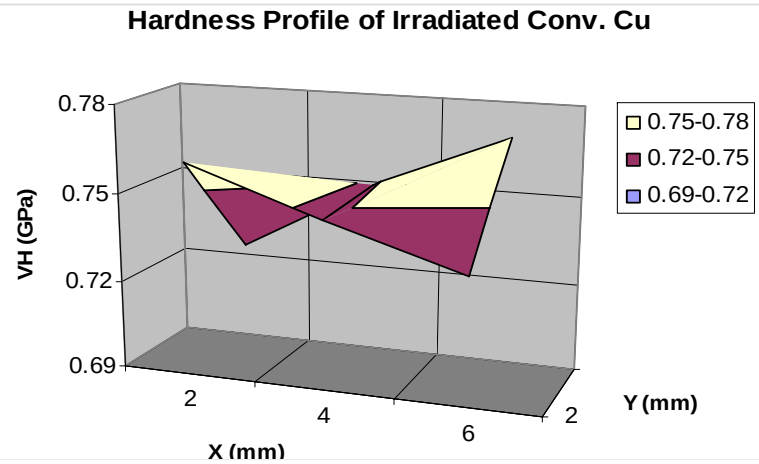
Microhardness Measurements



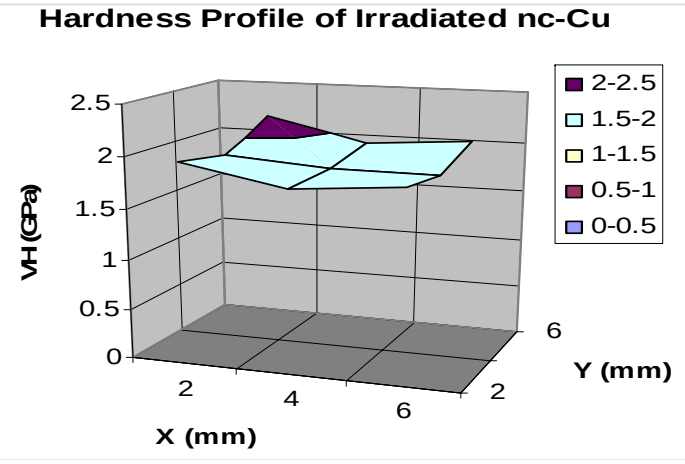
VH = 0.596±0.02 (GPa)



VH = 2.51±0.05 (GPa)



VH = 0.747±0.01 (GPa)



VH = 1.91+0.11 (GPa)

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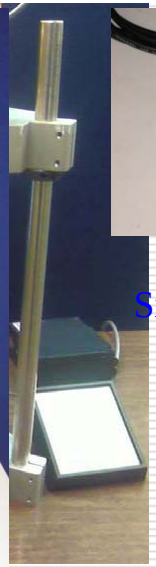
Mechanical Testing

- A miniature tensile tester was built for tensile testing

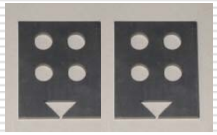
Step motor

Smart step

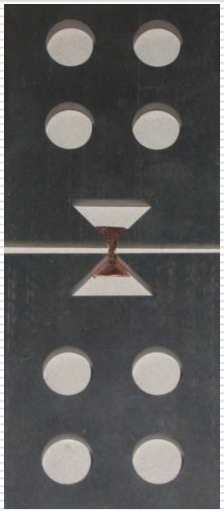
Main body



Signal conditioner

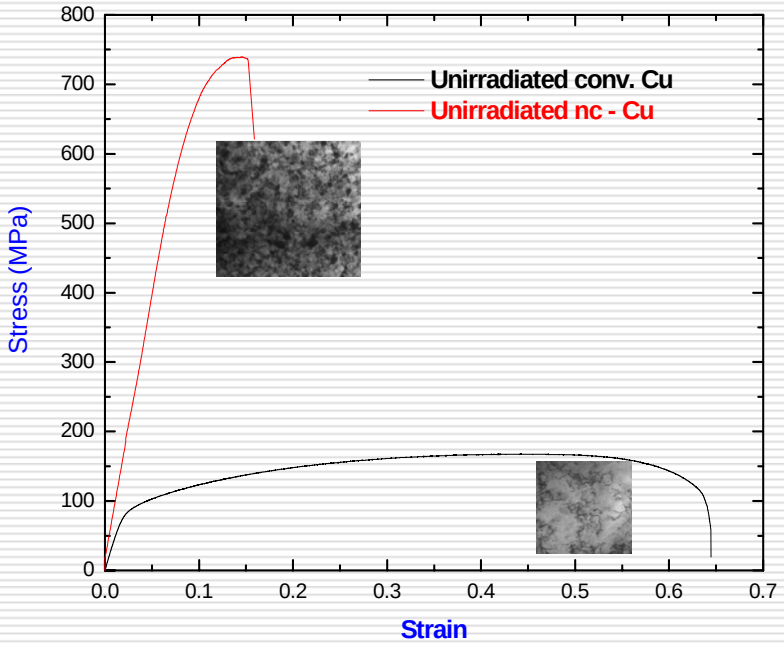


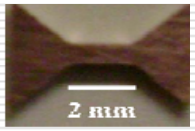
Grips



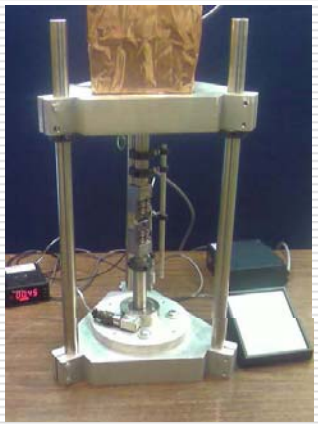
(Miniature Tensile Tester)

Mechanical Properties (Tensile Testing)

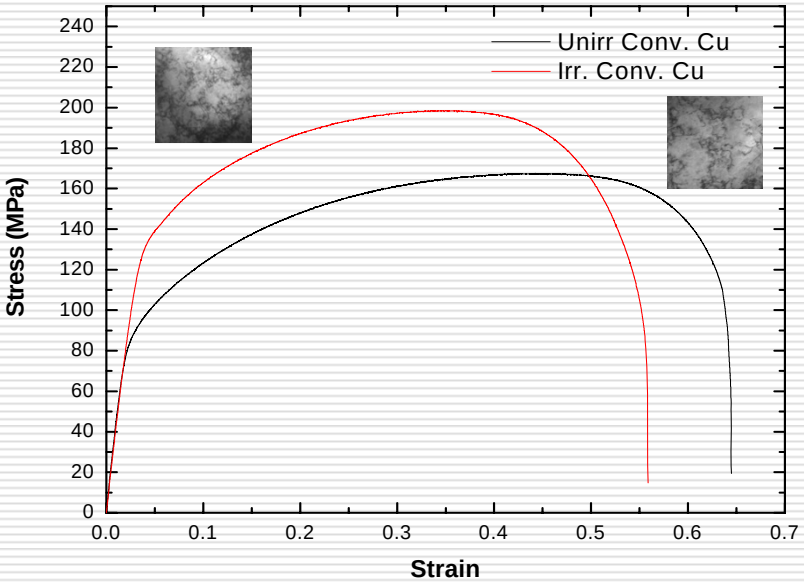


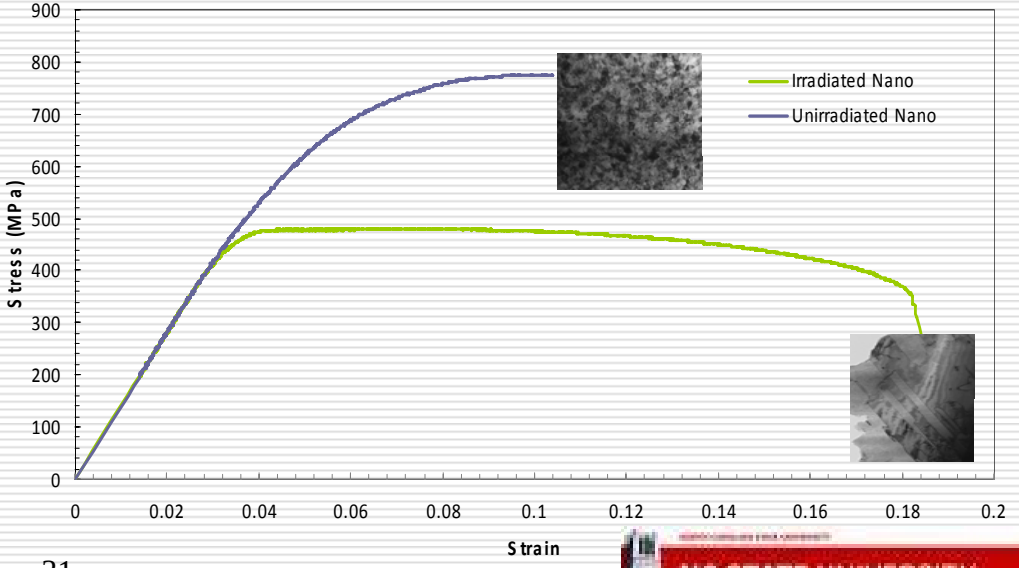


Instron Frame



Mini. Tensile Tester





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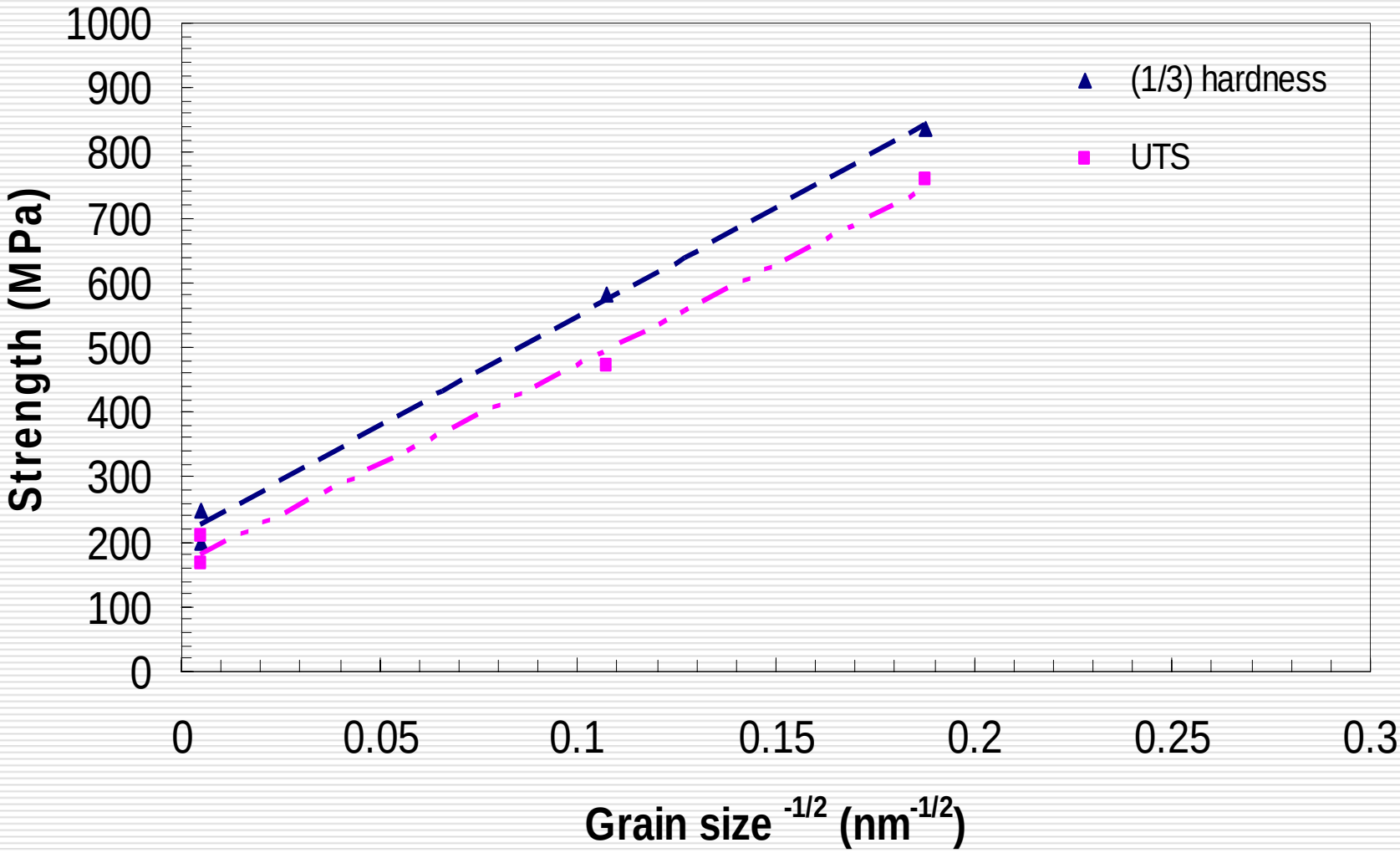
Summary

Material	Yield (MPa)	UTS (MPa)	e_u	n
Unirr. Conv Cu (6 samples)	82±5	167.53±8	0.27	0.24
Irr. Conv Cu (3 samples)	170.5±23	209.58±11	0.18	0.165
Unirr. nc – Cu (5 samples)	487±20	759.6±30	0.083	0.079
Irr. nc – Cu (3 samples)	370±40	470±13	0.054	0.052

Grain Size Effect



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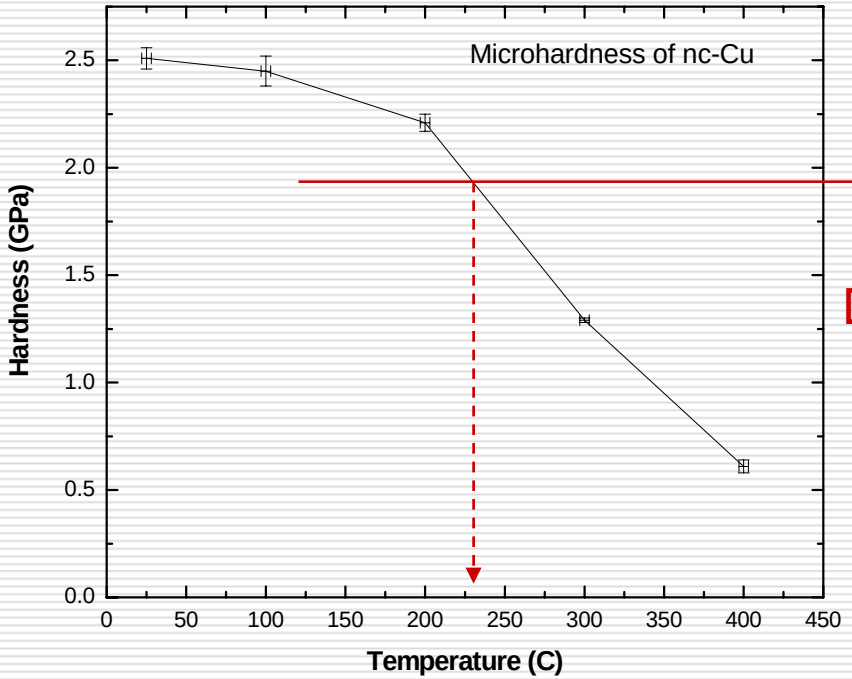
Discussion – PULSTAR results

- **Conventional Copper**
  - Irradiated conventional copper agrees with the commonly observed radiation hardening and embrittlement
  - The strain hardening exponent, n, decreased as expected
- **Nanostructured Copper**
  - nc-Cu became softer after irradiation (yield strength, UTS and microhardness decreased following irradiation)
  - Low dislocation density and twin structure were observed
  - Grain size measurements revealed in-reactor grain-growth (as noted earlier in irradiated nanostructured materials)

Annealing Effects on nc-Cu



Microhardness measurements of nc-Cu vs annealing temperature

Irr. nc-Cu

- Irradiated nc-Cu seems to have microhardness close to samples annealed at *200°C*
  - ***Grain growth due to Self heating***
  - ***Irradiation-induced grain growth***

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Discussion

- Both conventional and nc-Cu exhibit radiation hardening - Supported by TEM observations of dislocations and twin structure in irradiated nc-Cu
- Radiation-induced grain growth related to radiation-induced collision cascade at atomistic level
- Radiation effect in nc-Cu is governed by two processes:
  1. Radiation-induced grain growth
  2. Radiation hardening

The End