Influence of Fast Neutron Irradiation on Nanostructured Metals / Alloys

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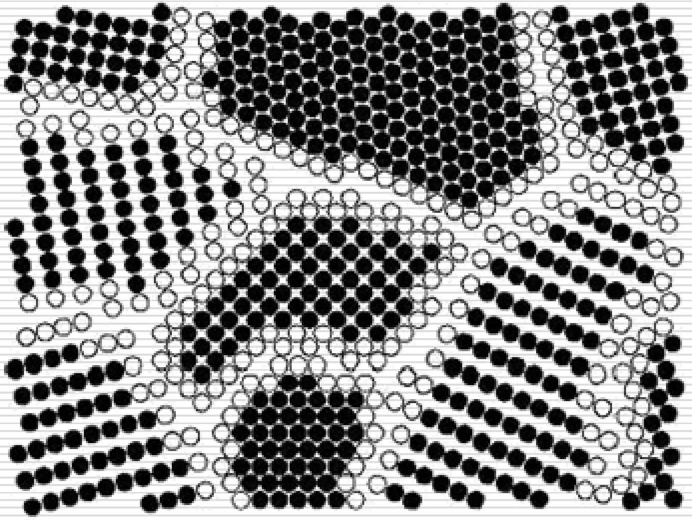
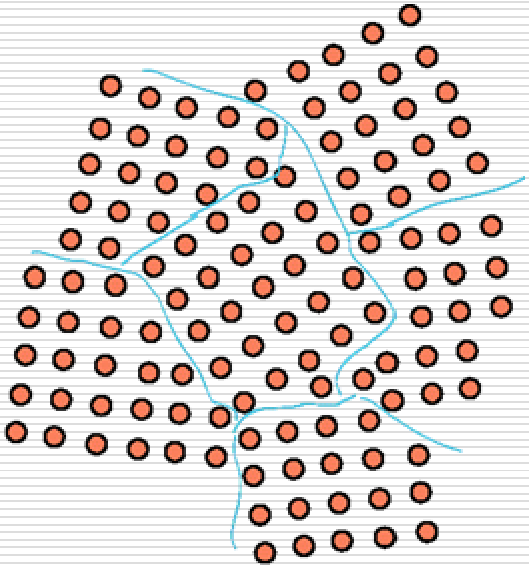
Introduction

- 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

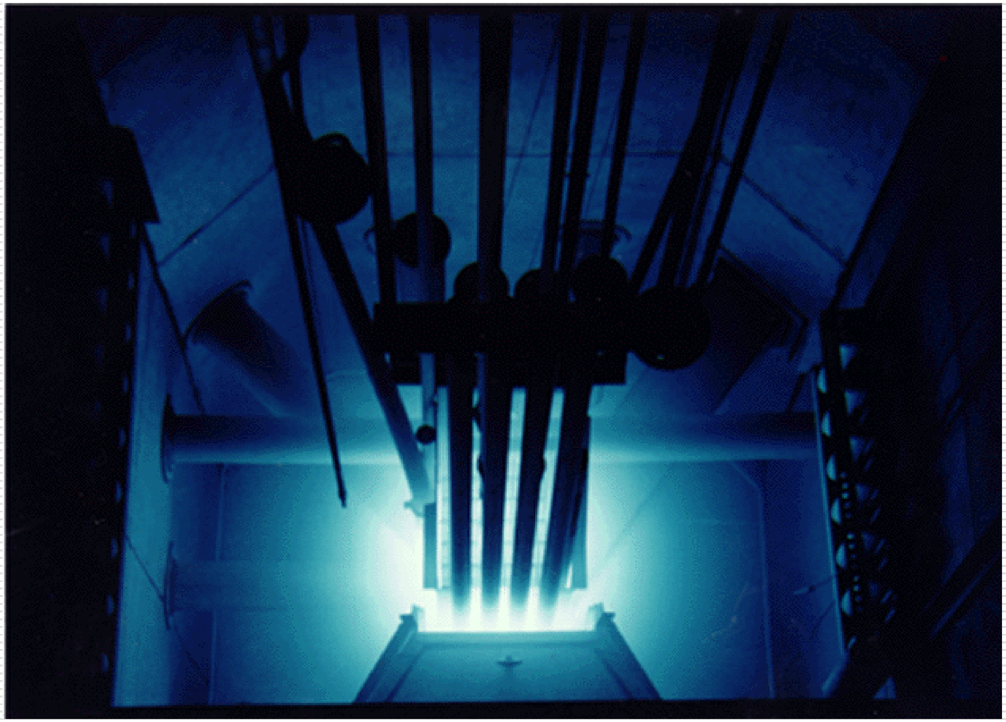
1. To investigate radiation effects on mechanical properties of nanostructured metals

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

3. To understand deformation mechanisms of nanocrystalline materials and effect of neutron irradiation on them
   * PULSTAR reactor at NC State University for low dpa
   * ATR at Idaho National Laboratory for higher dpa

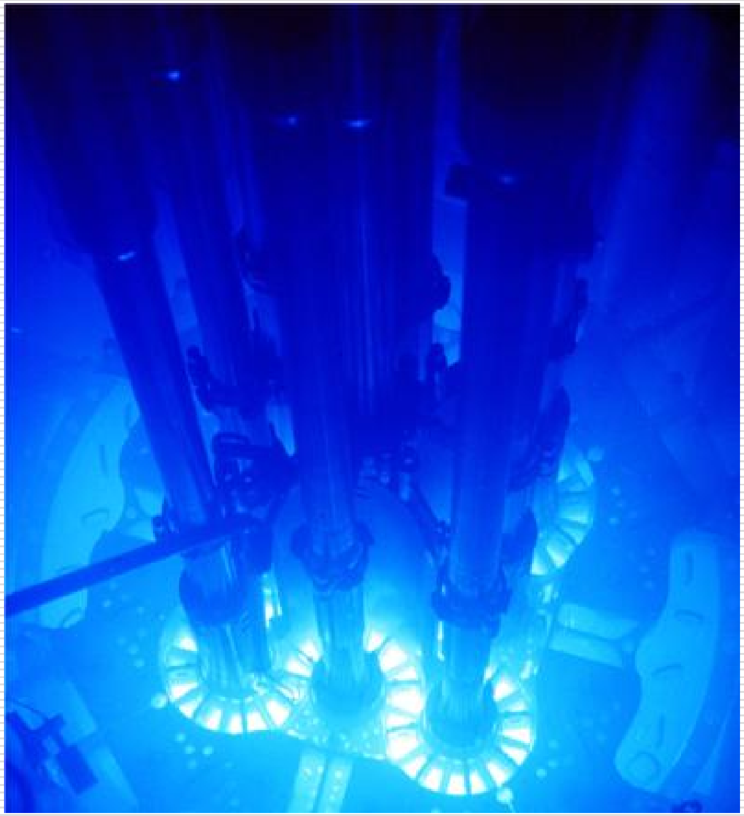
NC STATE UNIVERSITY

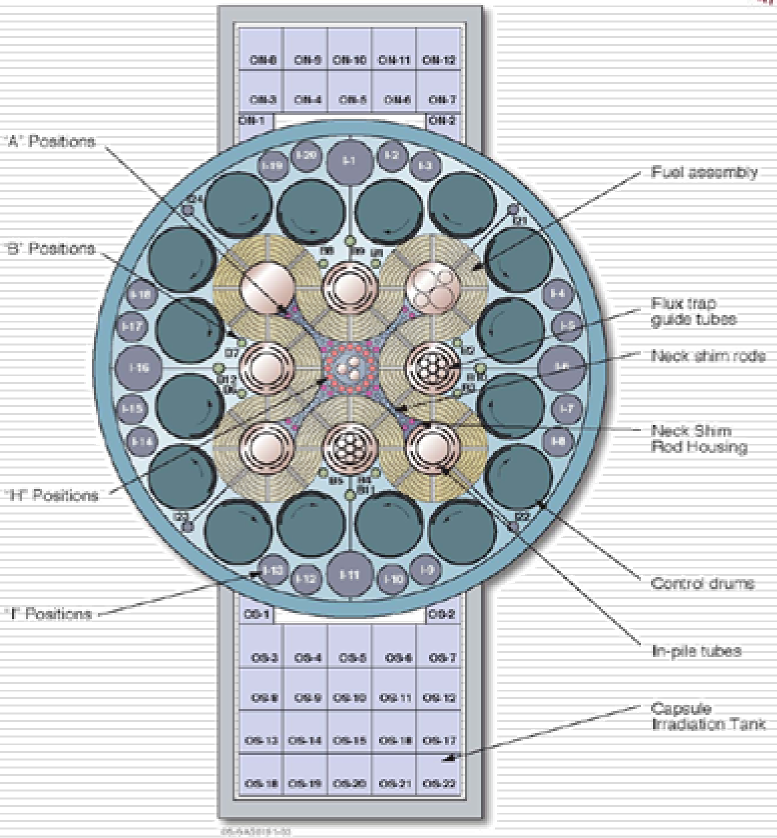
PULSTAR at NC State University



The PULSTAR, 1 MWth, reactor at NC State University used for low fluence/dpa level irradiation experiments

The ATR at INL





The Advanced Test Reactor at INL represents a unique irradiation facility equipped with *hot-cell* for post-irradiation examination

Research Scope

Nanostructured Metals

Radiation Tolerant?

MD simulation

- Dynamic Structure
- Diffusion Mechanisms
- Rule of interfaces

Response to Irradiation

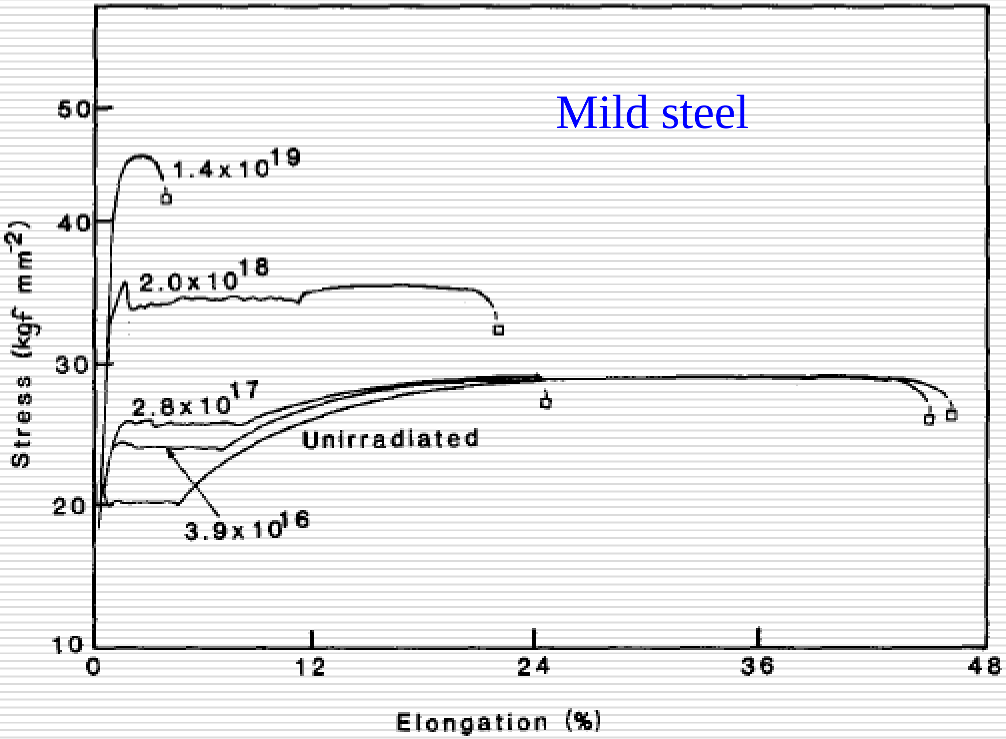
- PULSTAR
- ATR-INL
- Mechanical Properties
- Microstructure

Conclusions

Radiation Impacts on Conventional Materials

- Irradiation induced defects
  - Vacancies
  - Interstitials
  - Thermal spikes
  - Displacement spikes
  - Dislocation loops
  - Voids
  - Cavities.

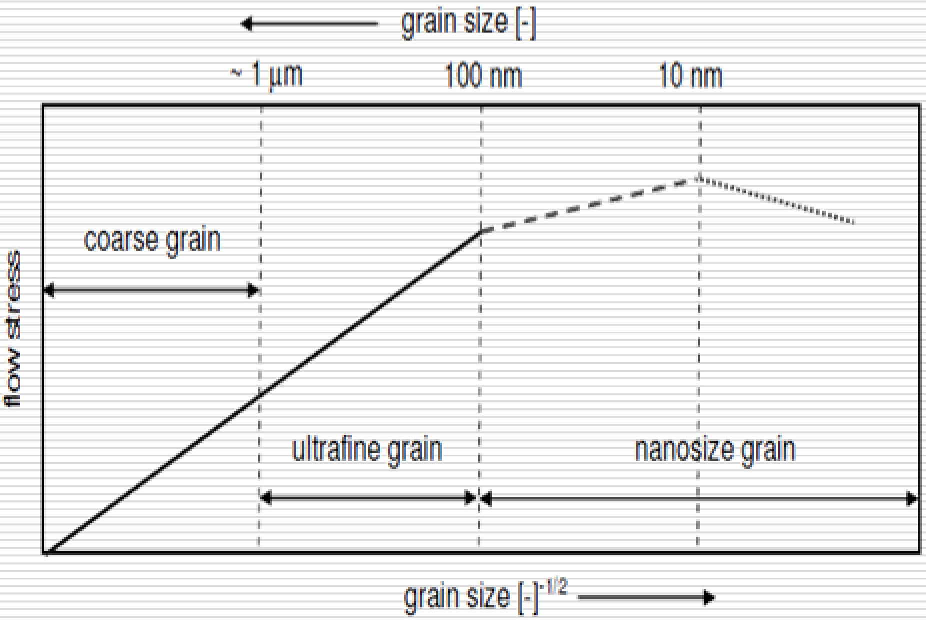
- These defects strongly affect the mechanical properties – increased strength and decreased ductility known as ***radiation hardening and embrittlement***



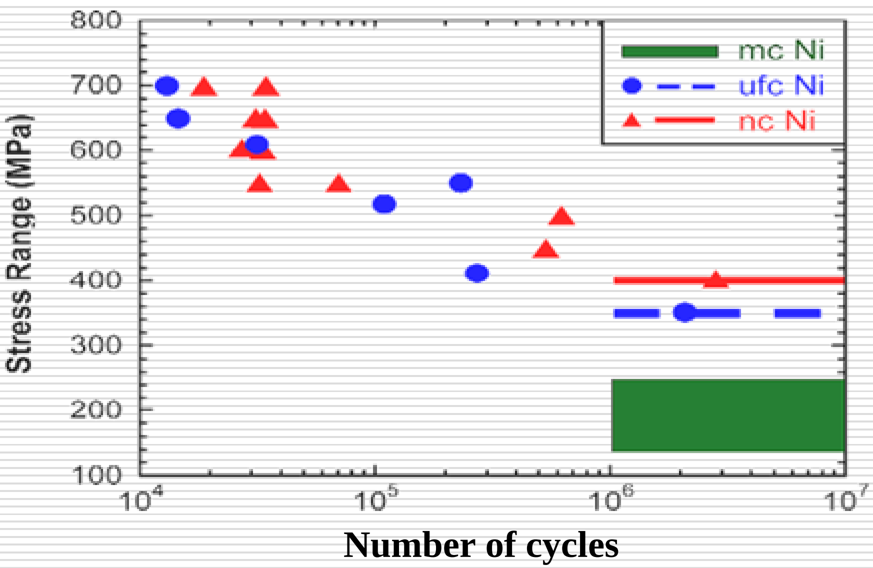
K. L. Murty, Materials Science and Engineering, 59 (1983) 207-215

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

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*.

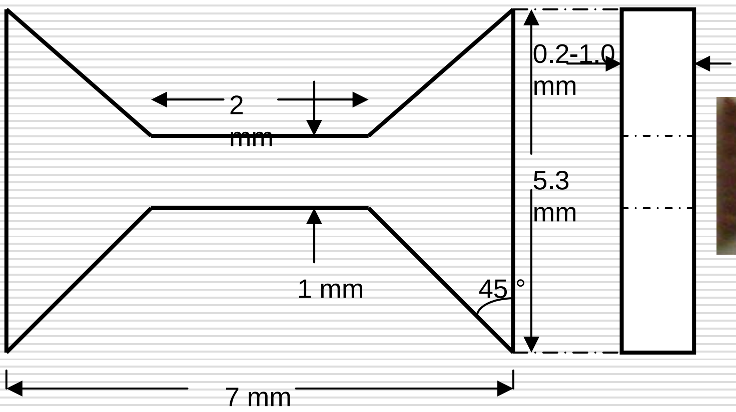
- Nanostructured Ni
  - Highly strained regions were observed in irradiated samples
  - The average grain size *decreases* following proton irradiation to 0.56 dpa
  - Twin boundaries were observed in irradiated material with higher density than it in the unirradiated material.

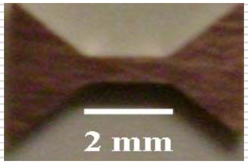
- Nanostructured Cu-0.5Al_2O_3
  - Cu-0.5Al_2O_3 exhibited *grain growth* as a consequence of irradiation.
  - Stacking fault and *low density of dislocations* were observed in the irradiated Cu-0.5Al_2O_3.

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

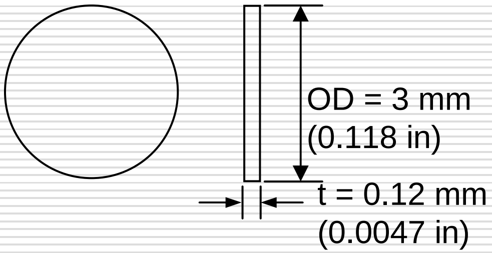
Materials & Samples for Irradiation

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



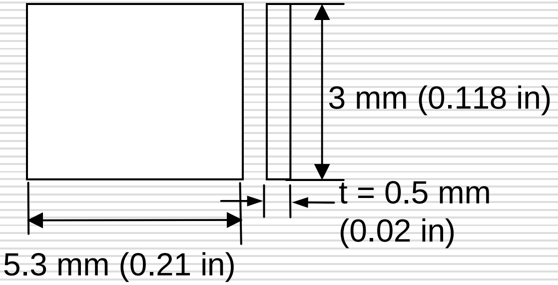


(1)

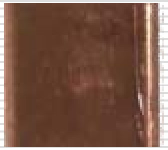


TEM specimen

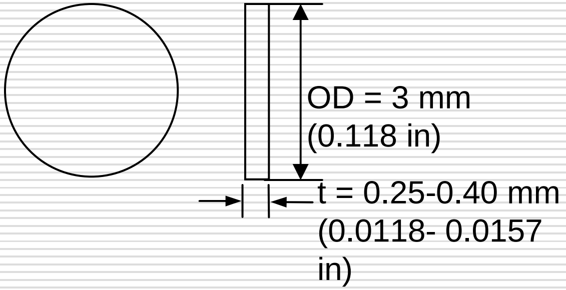
Sub-size tensile sample



Hardness sample



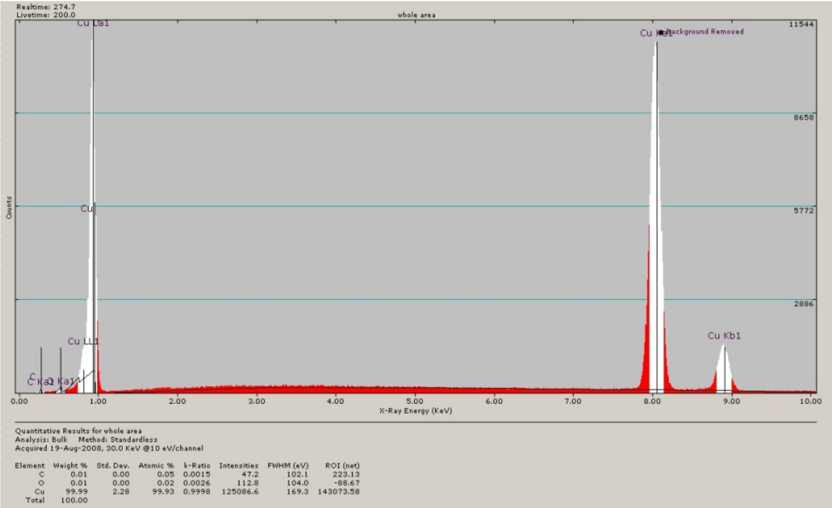
(2)



SPT specimen

Materials

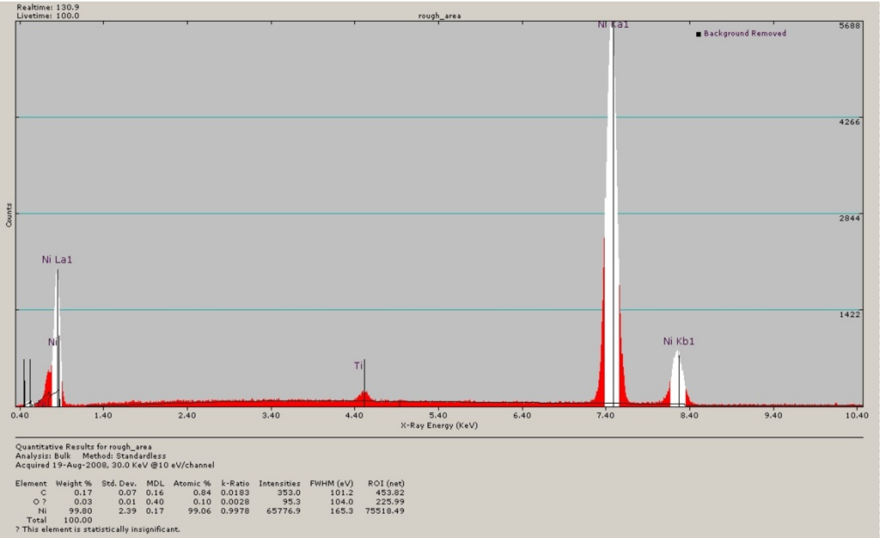
- nc-Cu, nc-Ni, uf- Carbon Steel



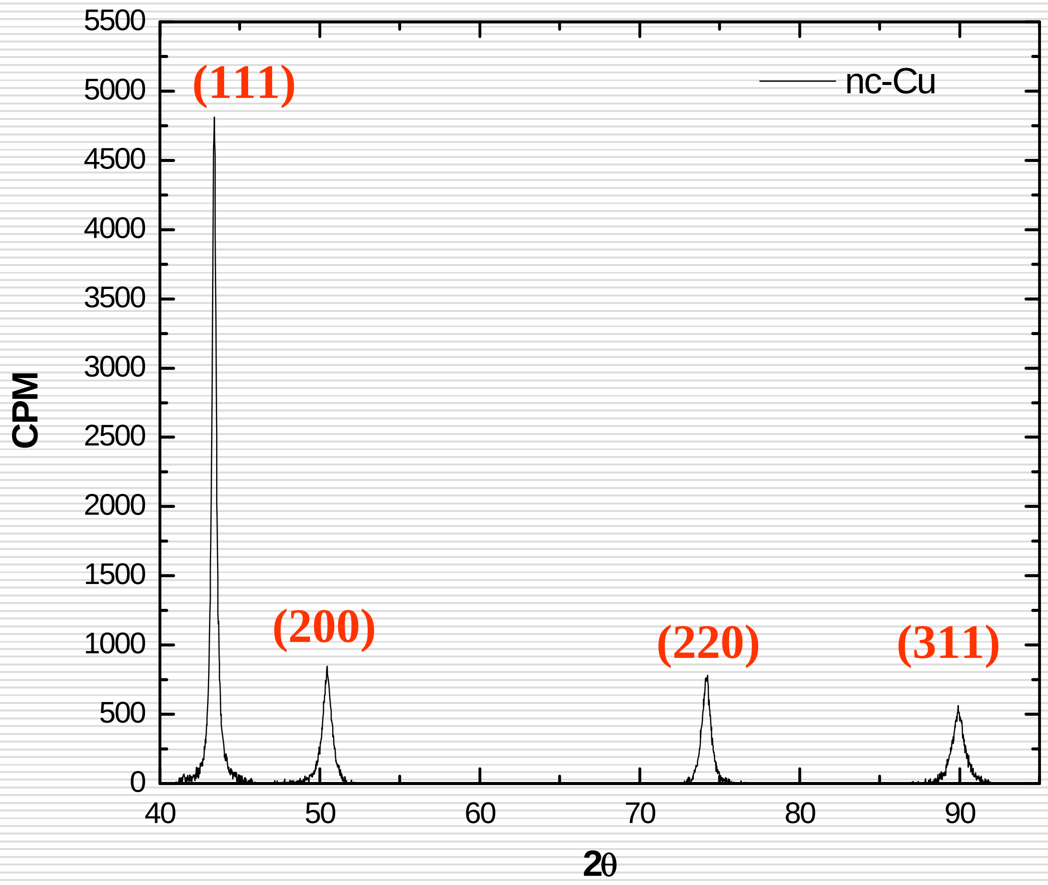
Compositions of Materials

SEM-EDS nc-Cu

SEM-EDS nc-Ni

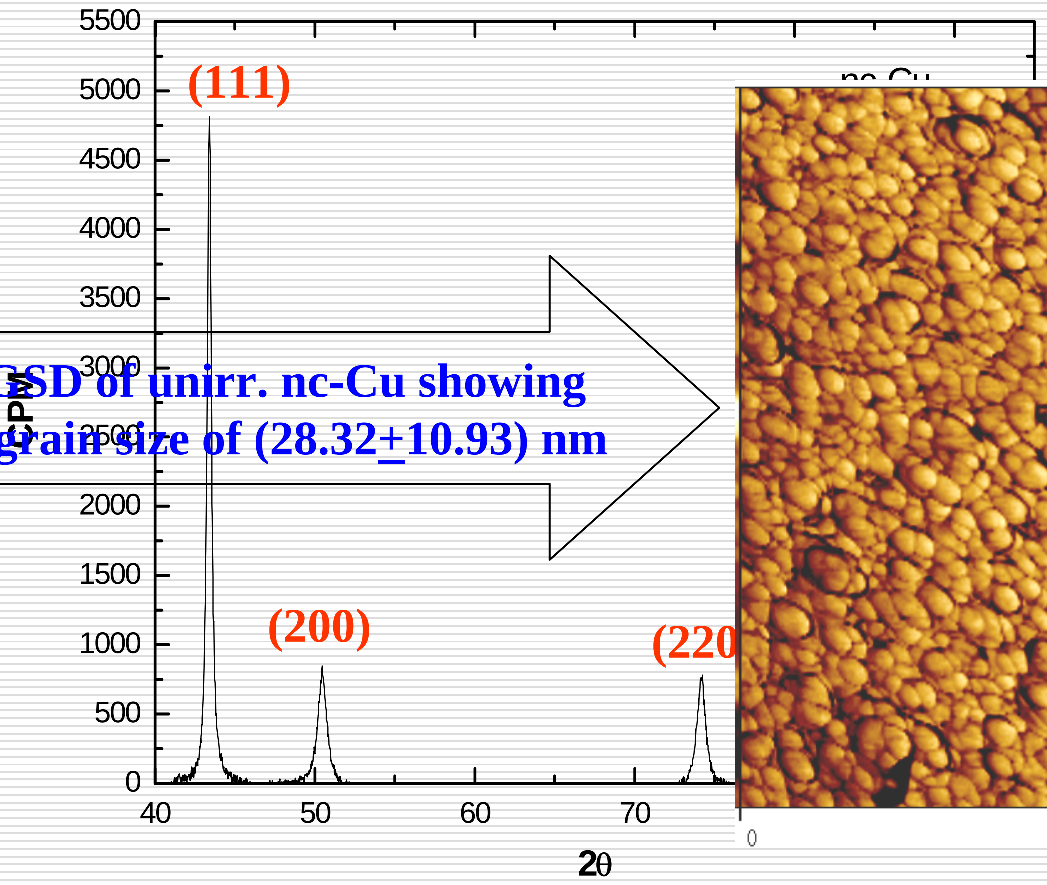


XRD Analysis

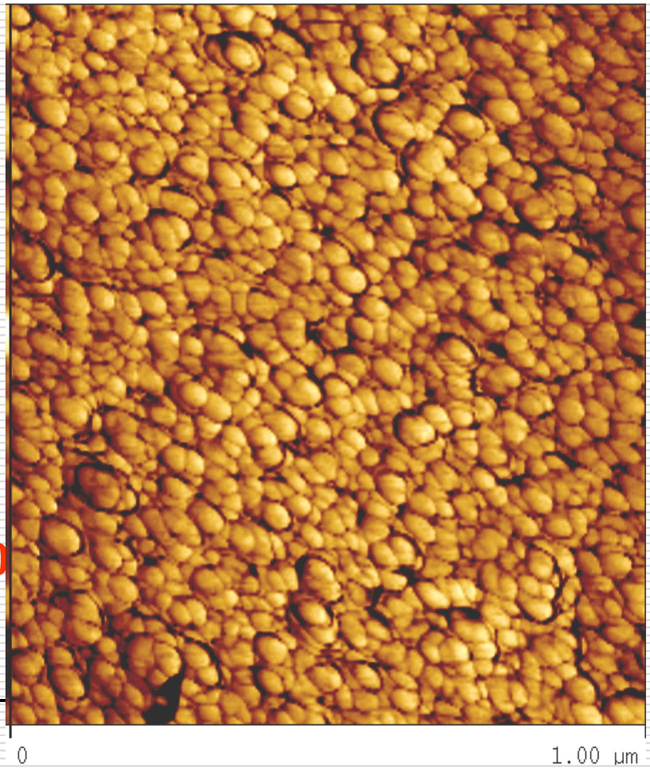


XRD of nc-Cu - grain size = 40.11 nm

XRD Analysis – Grain Size Measurement



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



XRD of nc-Cu - grain size = 40.11 nm

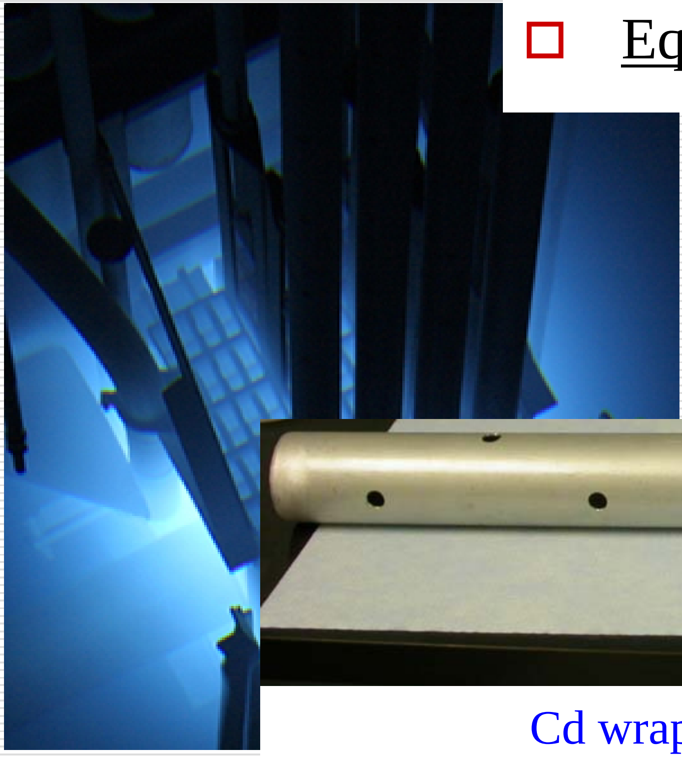
Irradiation at NC State University

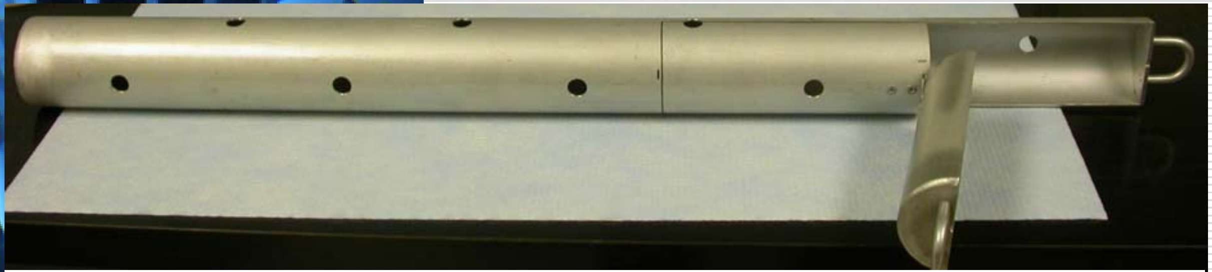
ch reactor

- The average fast flux component was found to be 6.6x10^{11} n/cm^2.s
- Irradiation time: 249.87 hrs
- Equivalent Fluence: 5.9x10^{17} n/cm^2

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

- *Cd* wrapped *Al* irradiatio
  eliminated thermal neutron ab

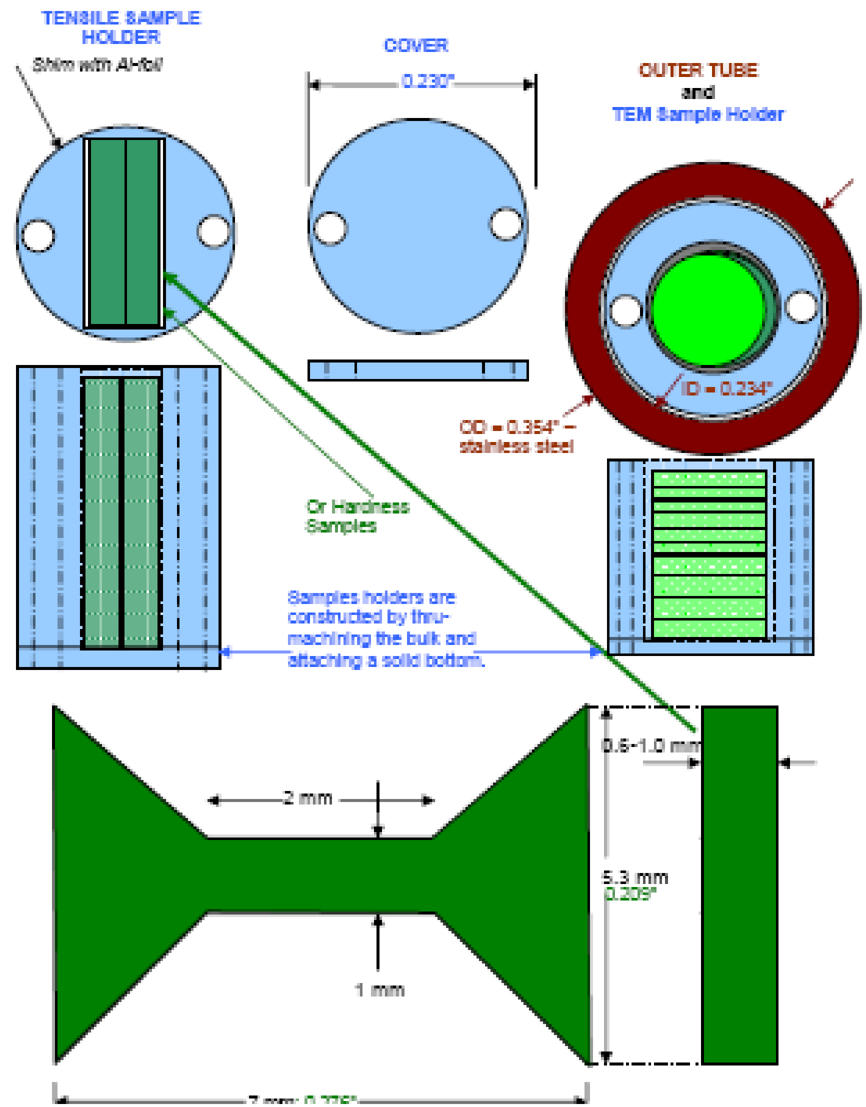


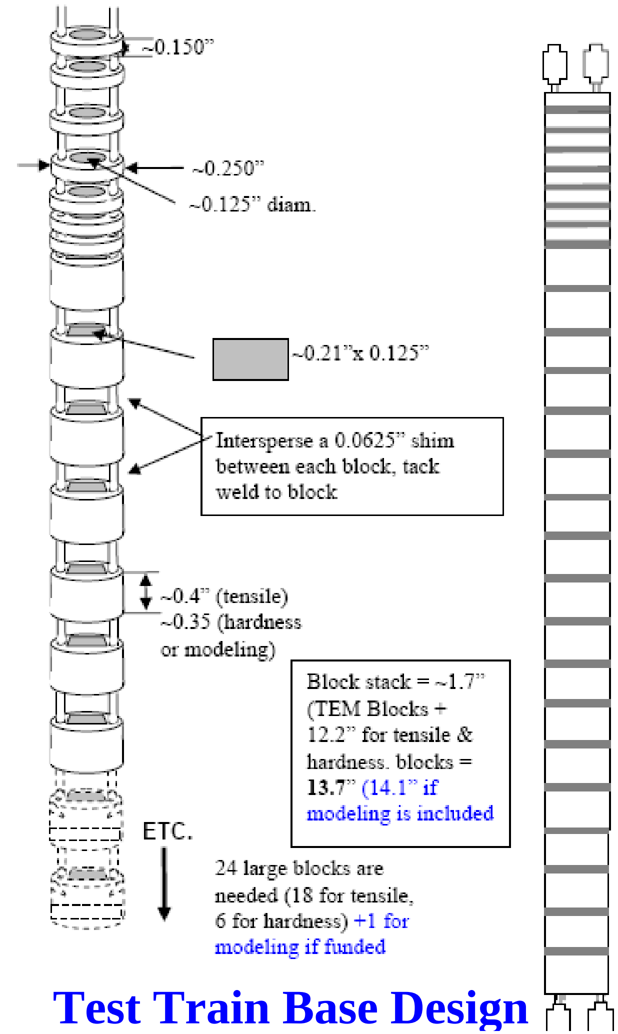


Cd wrapped Al irradiation column

Irradiation jig

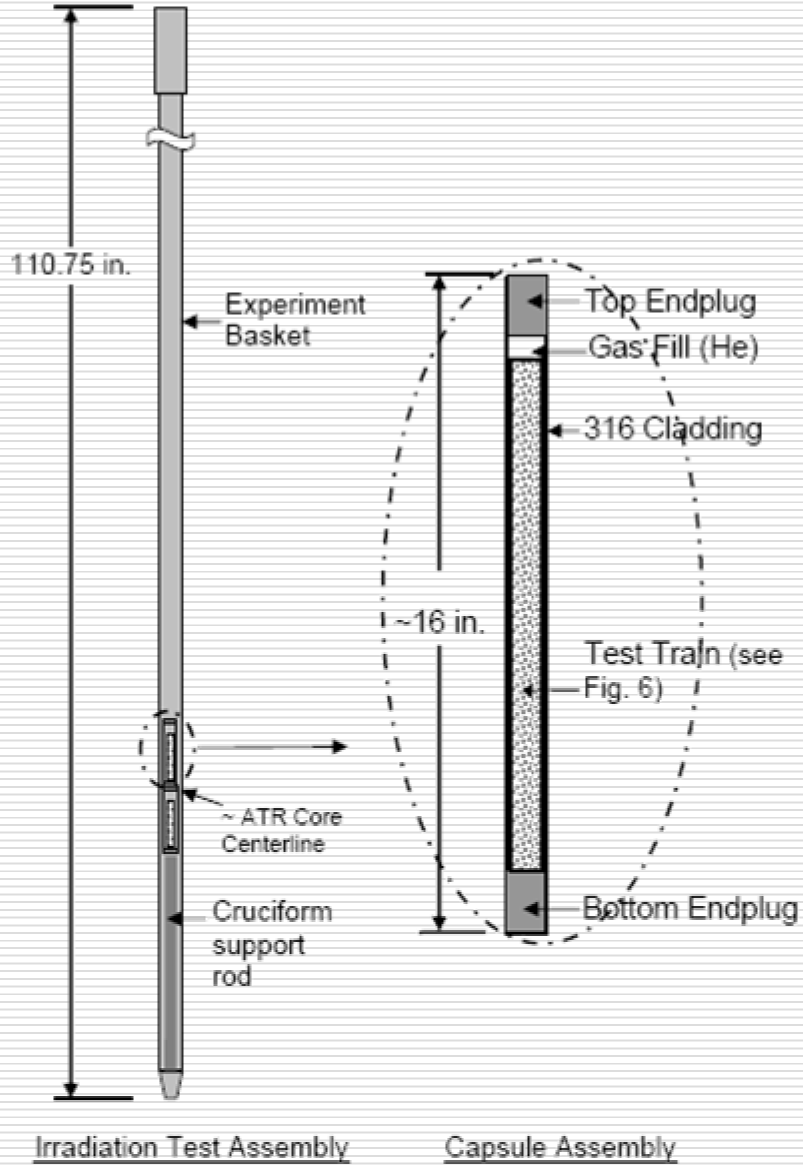
Irradiation at ATR: Sample/Holder Design





Test Train Base Design

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



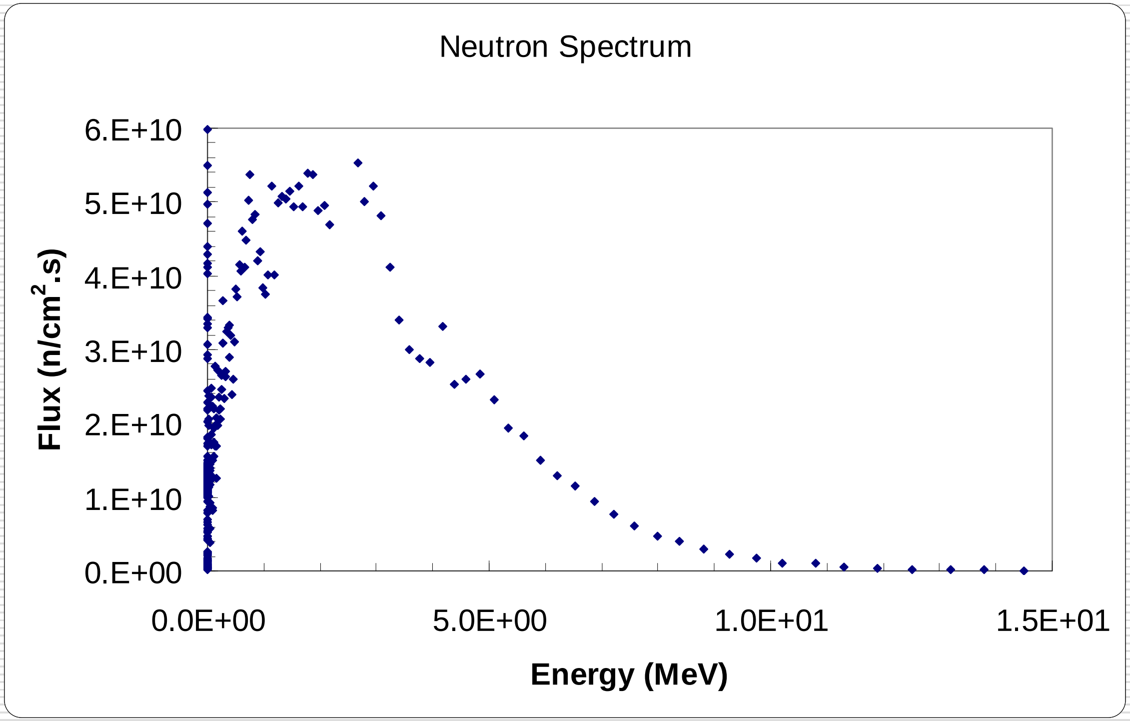
NCSU Experiment Irradiation Test Assembly for ATR East Flux Trap Position

Irradiation Experiment Conditions * ATR *

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

dpa Calculations - PULSTAR

- 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

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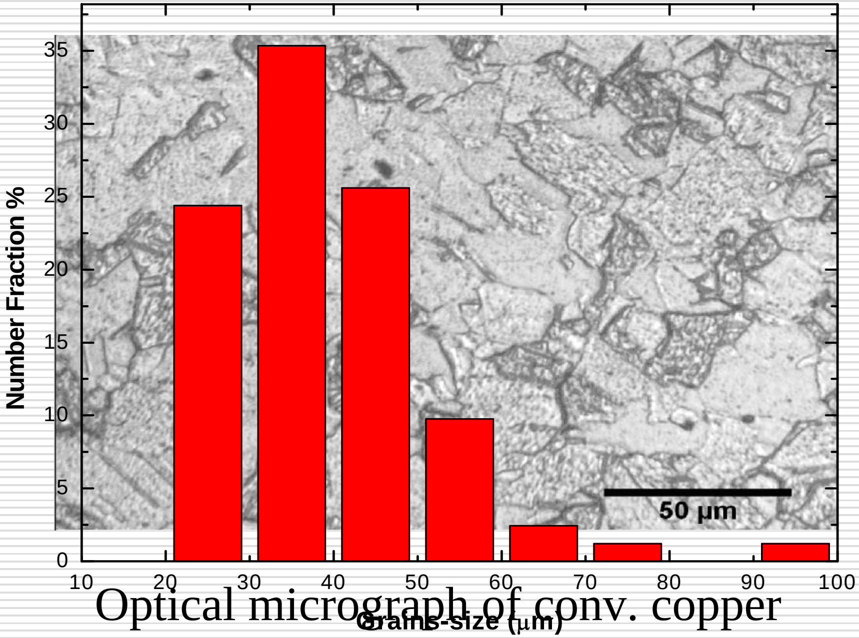
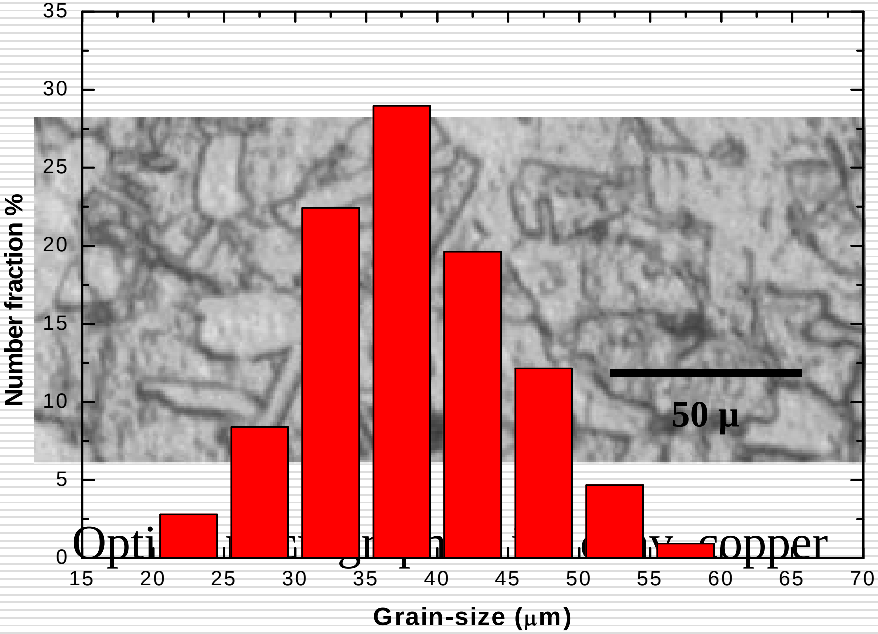
Microstructure Examination

- Conventional Copper



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

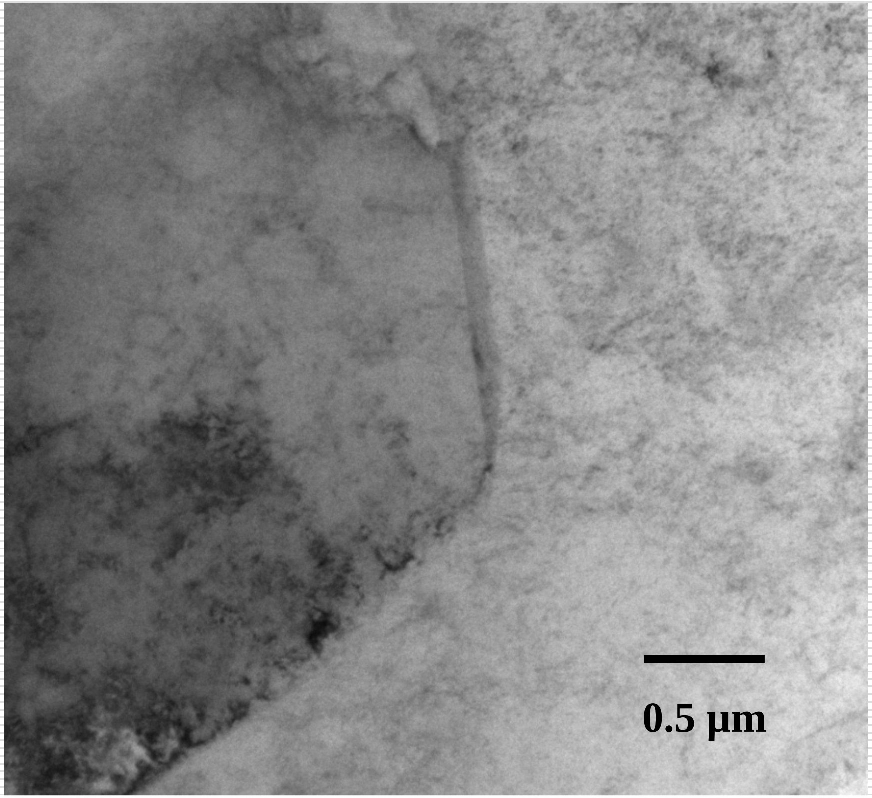


GSD of irradiated conventional copper
showing average GS of (39.1+7.1) µm

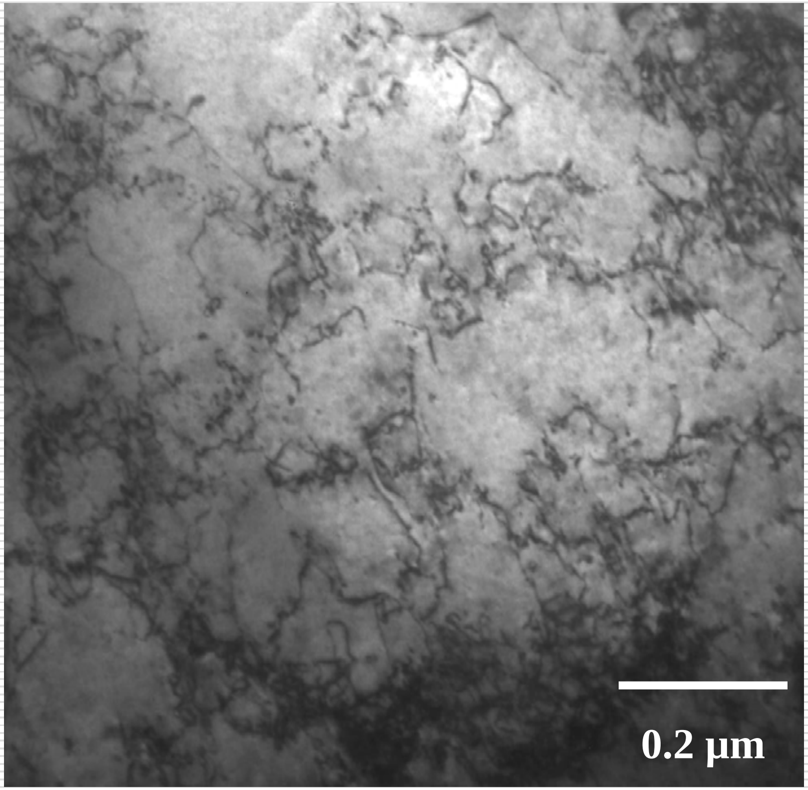
PULSTAR Irradiation – 0.34 dpa

Microstructure Examination

- Conventional copper



BF TEM micrograph of conv. Cu

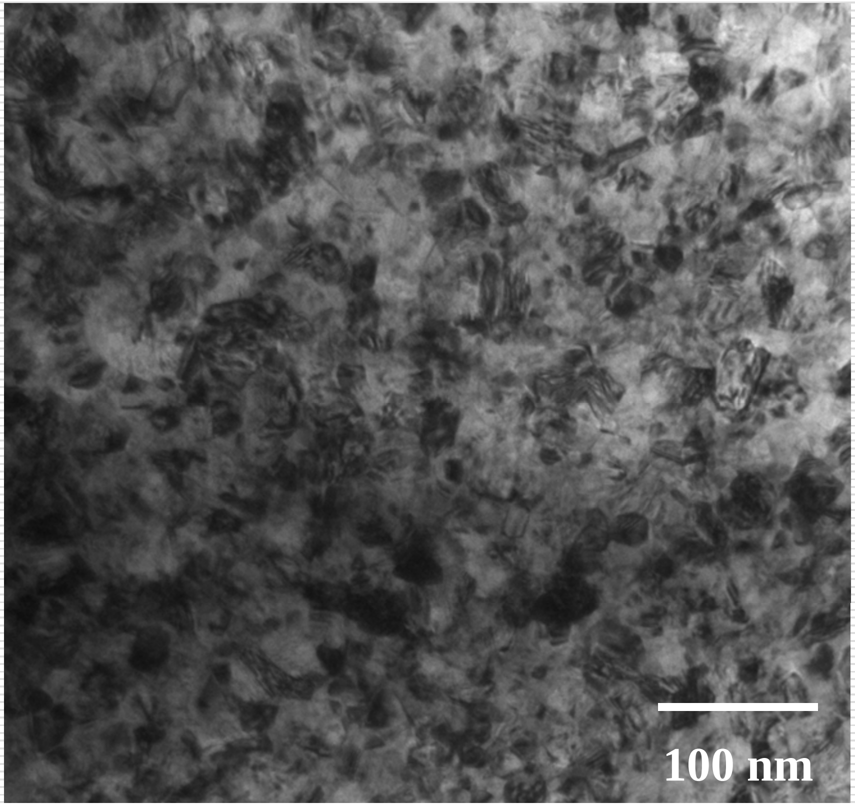


BF TEM micrograph of irr. conv. Cu

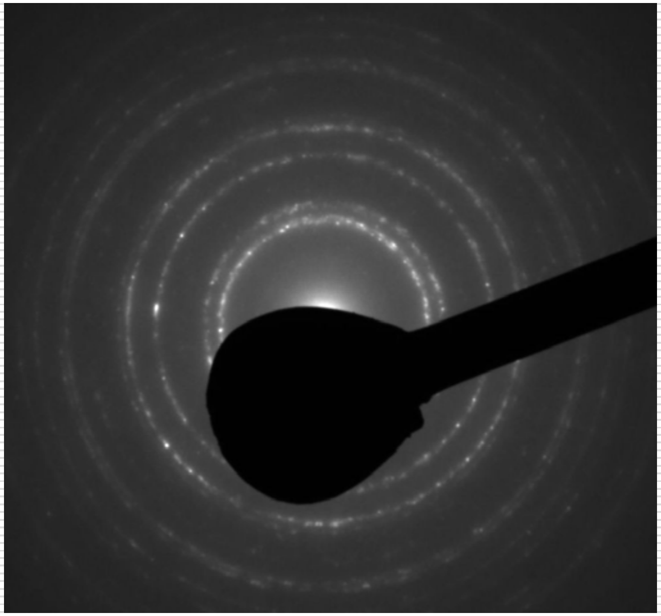
PULSTAR Irradiation – 0.34 dpa

Microstructure Examination

- Nanostructured Copper



Bright Field (BF) TEM
of unirradiated nc-Cu

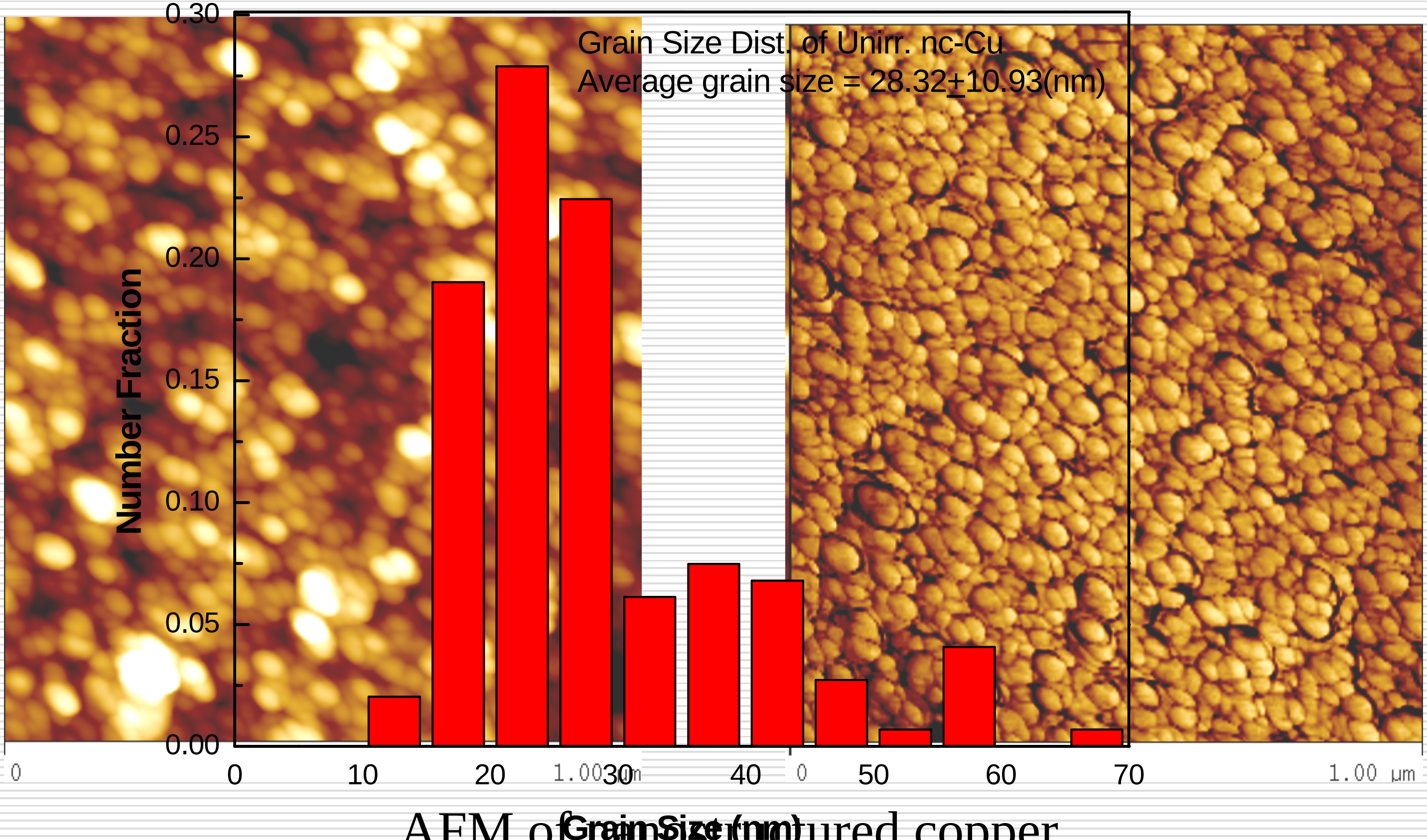


Diffraction pattern of unirradiated nc-Cu

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

Microstructure Examination

- Nanostructured Copper

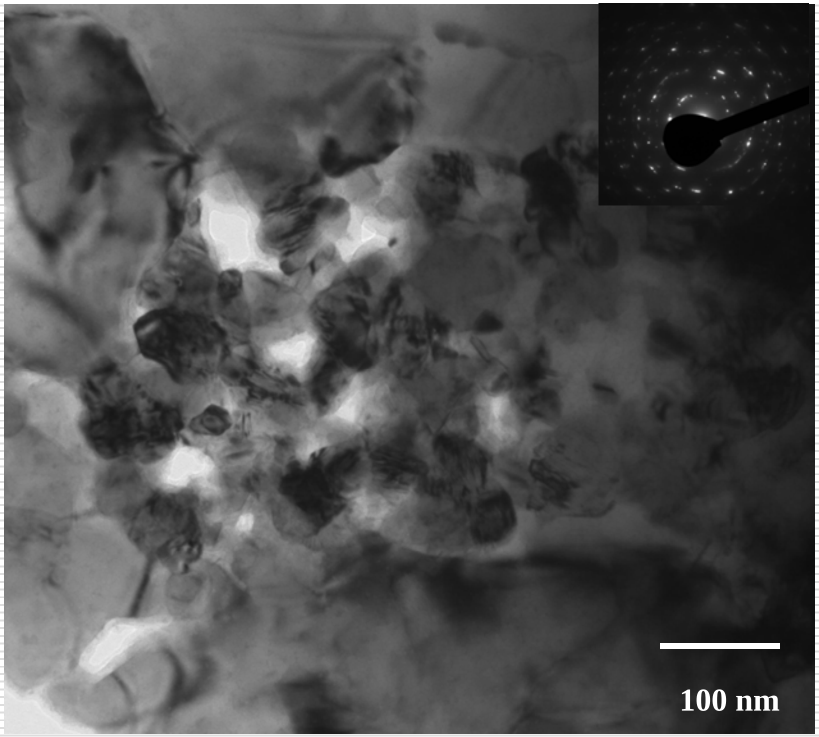


AFM of nanostructured copper

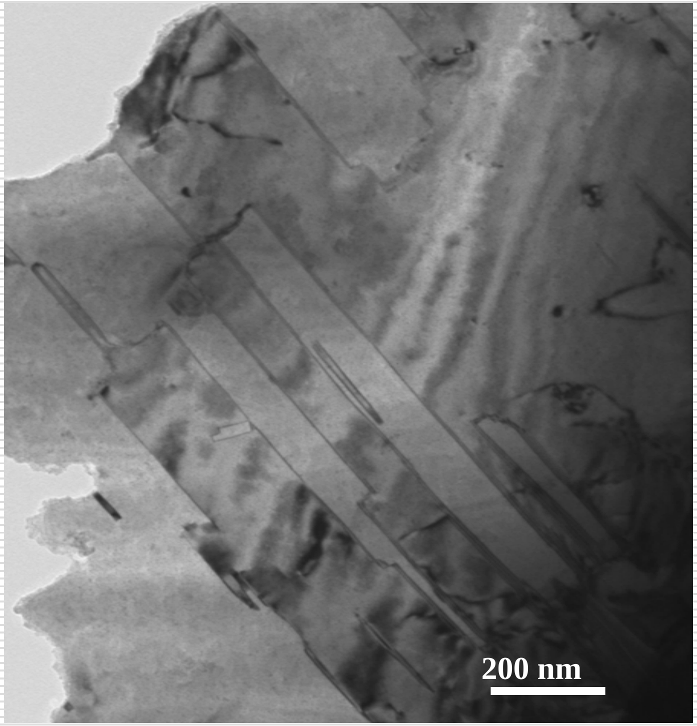
GSD of unirr. nc-Cu showing average grain size of (28.32±10.93) nm

Microstructure Examination

- Irradiated nc-Copper



BF TEM of irr. Nc-Cu

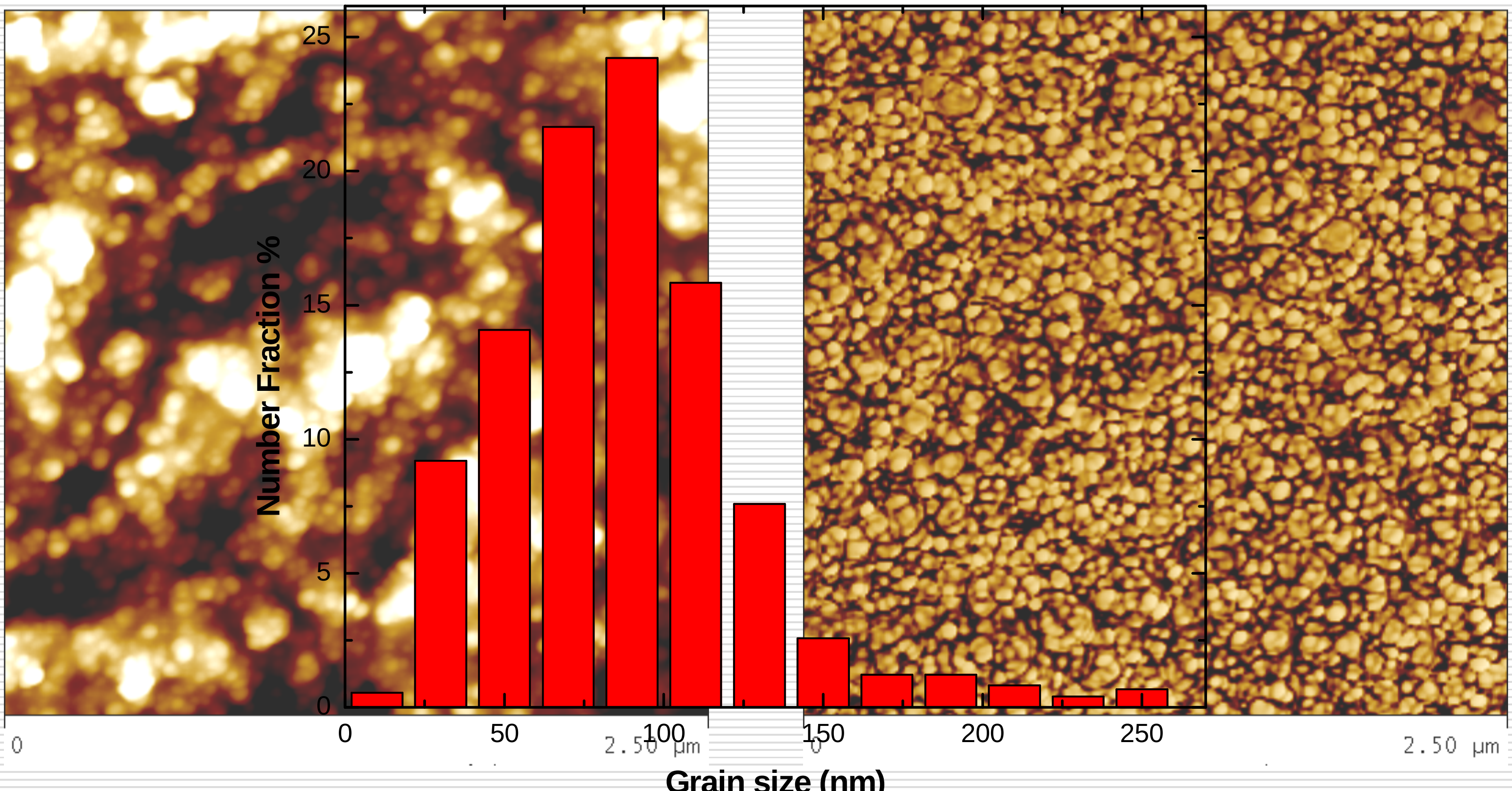


BF TEM of irr. nc-Cu showing dislocation and twin structures

PULSTAR Irradiation – 0.34 dpa

Microstructure Examination

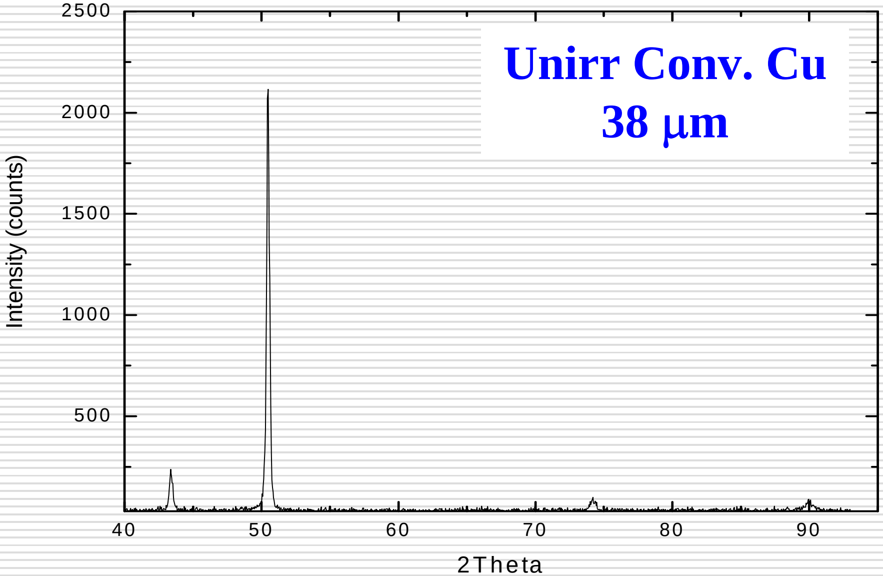
Irradiated (0.34 dpa) nc-Copper   Note increased grain size from 28 to 87 nm



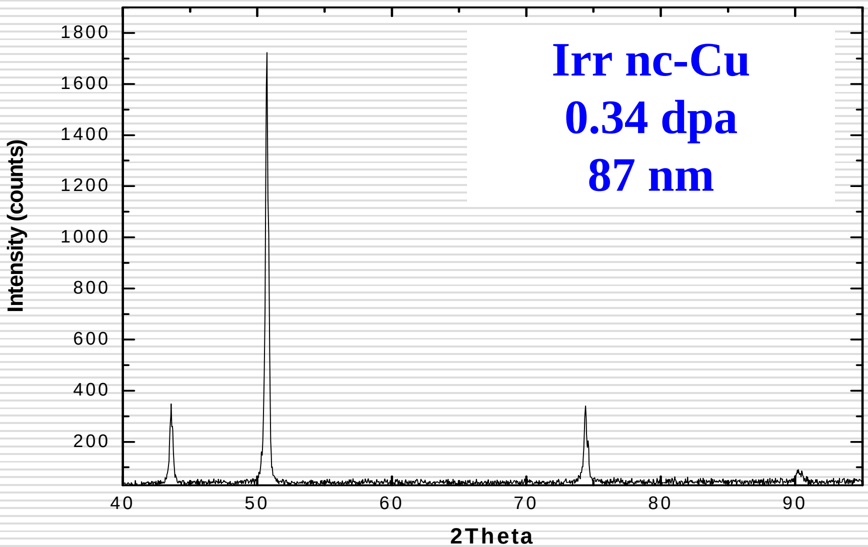
AFM of irr. nc-Cu

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

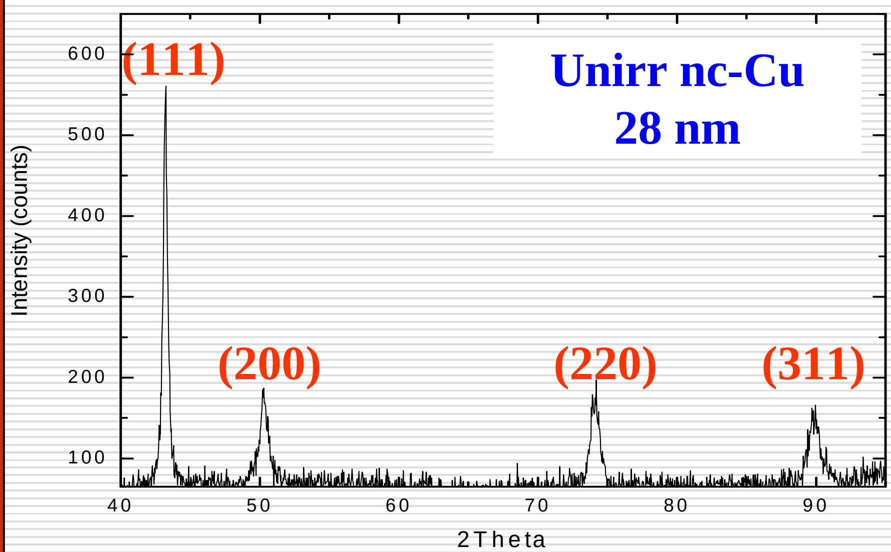
XRD



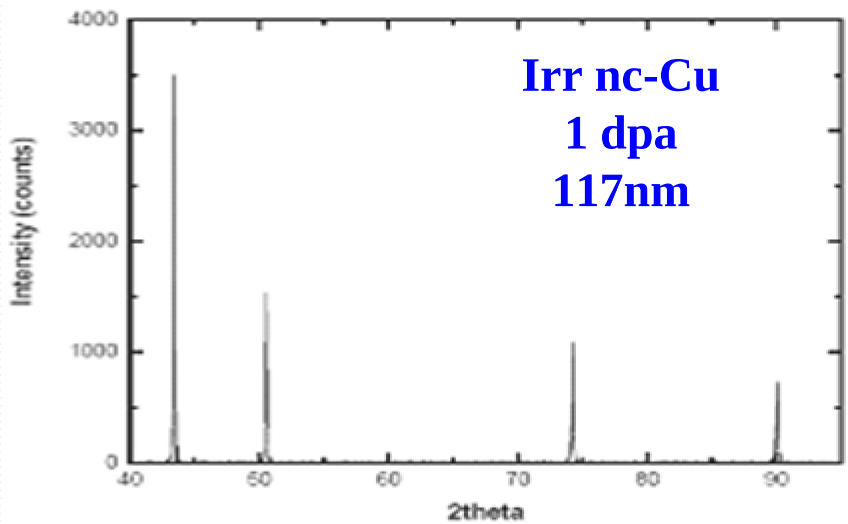
Unirr Conv. Cu
38 µm
Intensity (counts)
2500
2000
1500
1000
500
40
50
60
70
80
90
2Theta



Irr nc-Cu
0.34 dpa
87 nm
Intensity (counts)
1800
1600
1400
1200
1000
800
600
400
200
40
50
60
70
80
90
2Theta

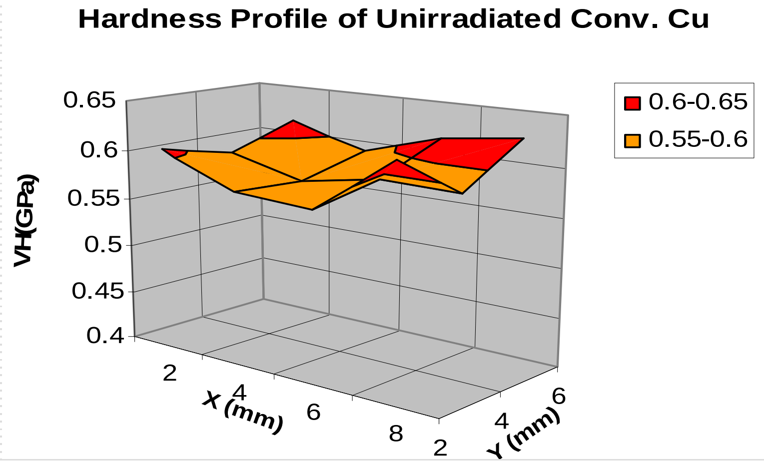


Unirr nc-Cu
28 nm
(111)
(200)
(220)
(311)
Intensity (counts)
600
500
400
300
200
100
40
50
60
70
80
90
2Theta

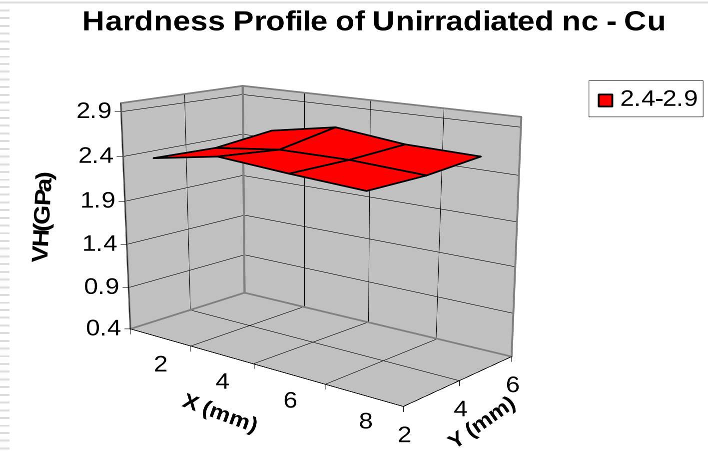


Irr nc-Cu
1 dpa
117nm
Intensity (counts)
4000
3000
2000
1000
0
40
50
60
70
80
90
2theta

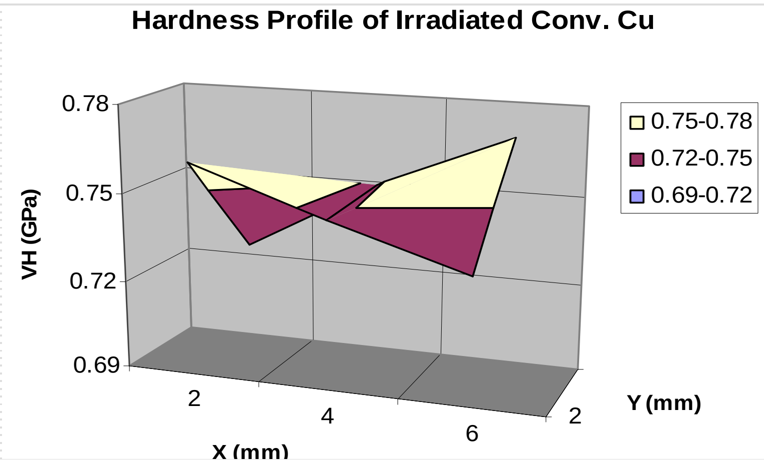
Microhardness Measurements



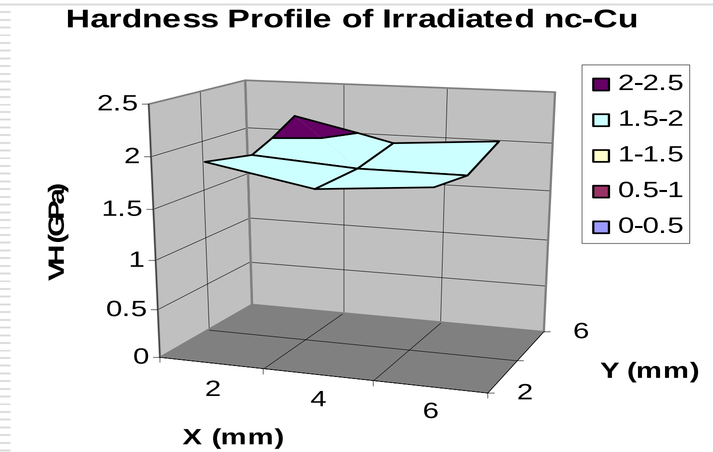
VH = 0.596±0.02 (GPa)



VH = 2.51±0.05 (GPa)



VH = 0.747±0.01 (GPa)

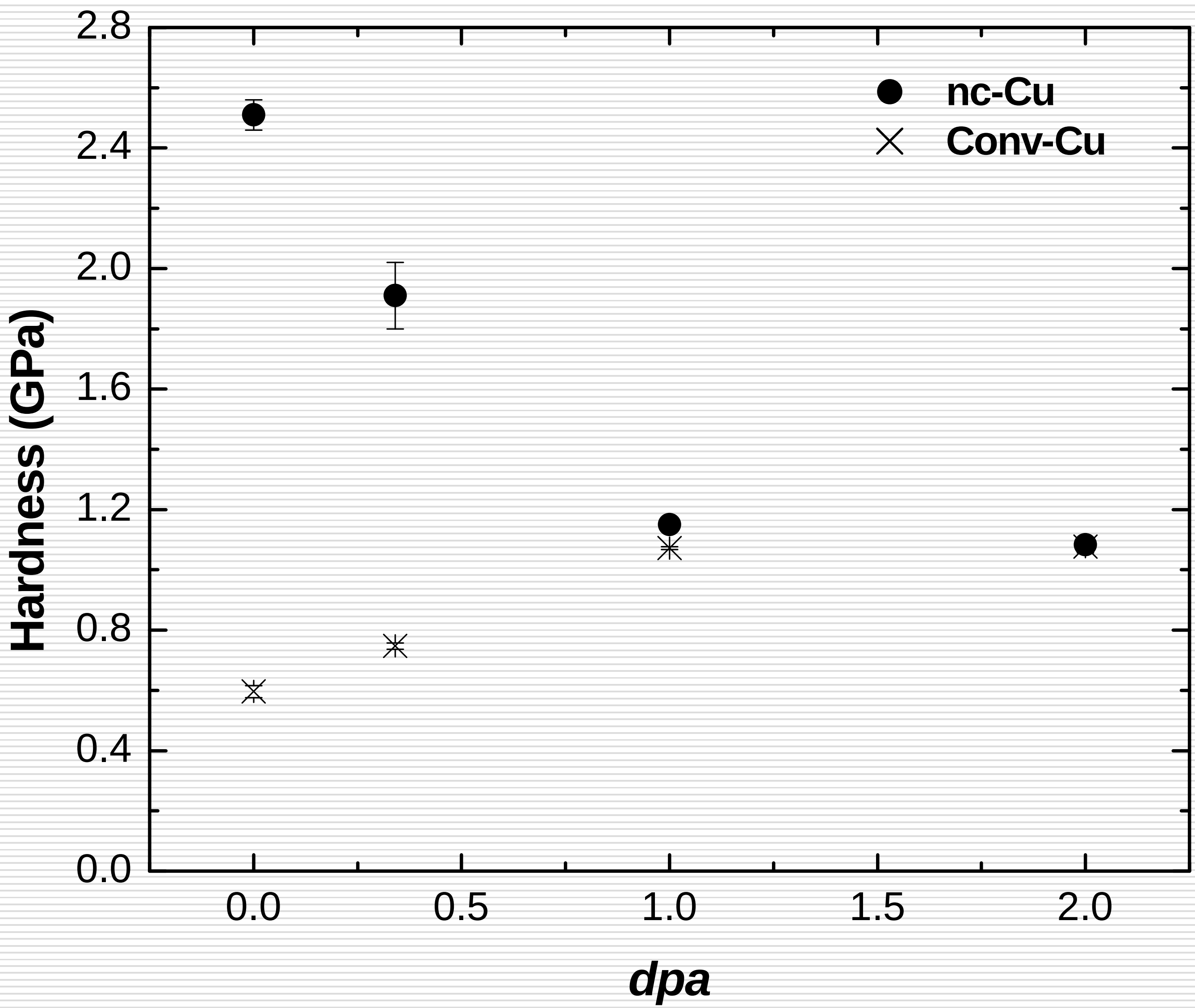


VH = 1.91+0.11 (GPa)

0.34 dpa

Note decreased hardening following radiation exposure

Microhardness Measurements



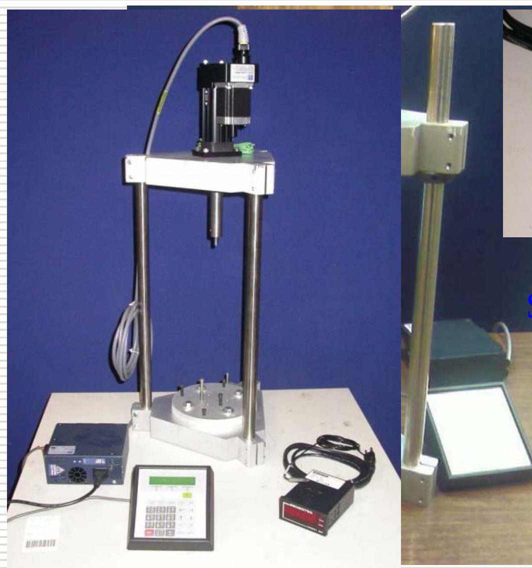
Hardness variation vs. *dpa*

Mechanical Testing

- A miniature tensile tester was built for tensile testing

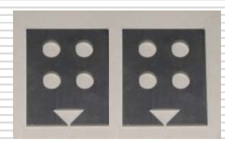
Step motor

Smart step

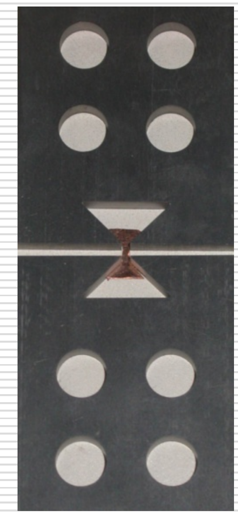


Main body

Signal conditioner

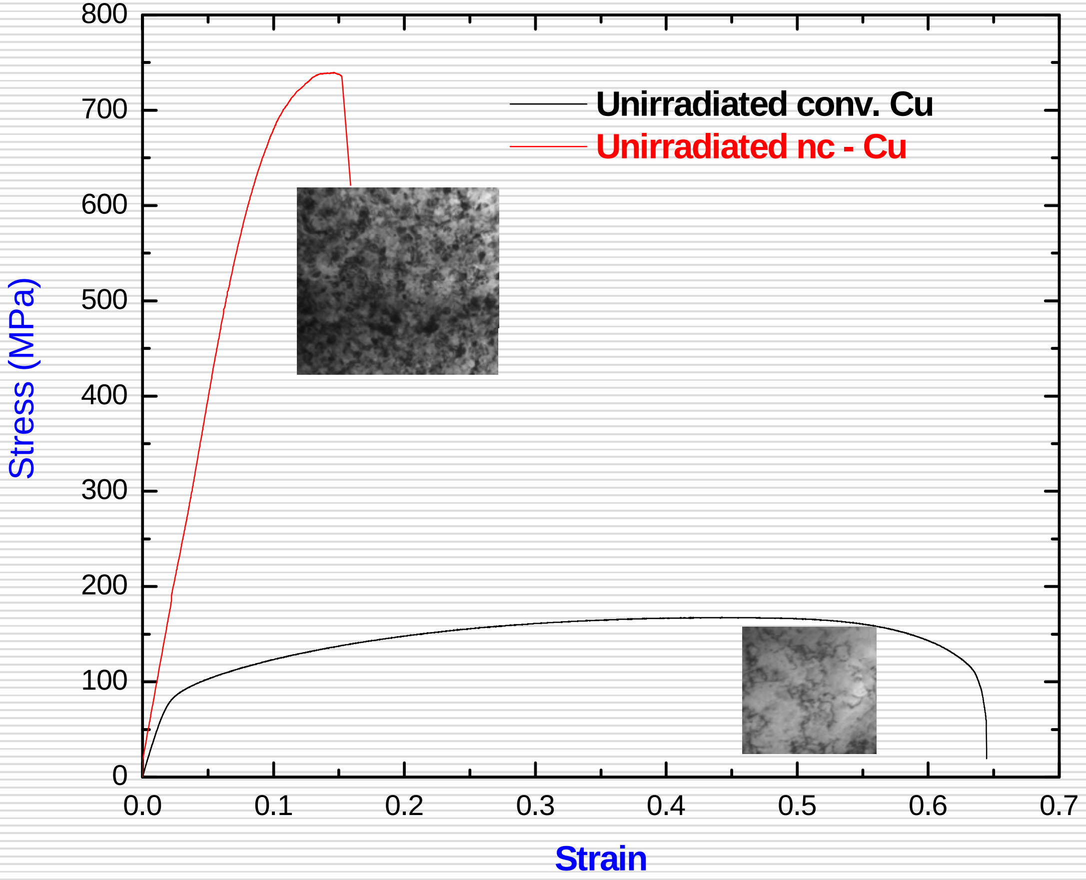


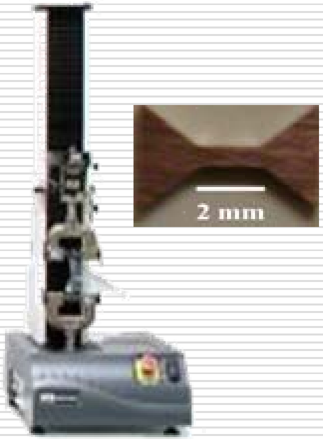
Grips



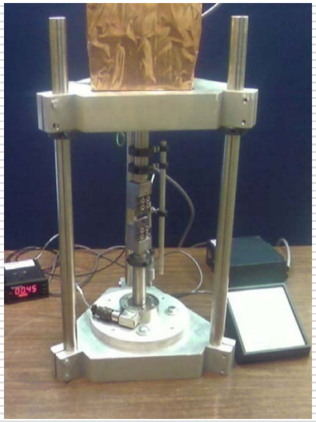
(Miniature Tensile Tester)

Mechanical Properties (Tensile Testing)





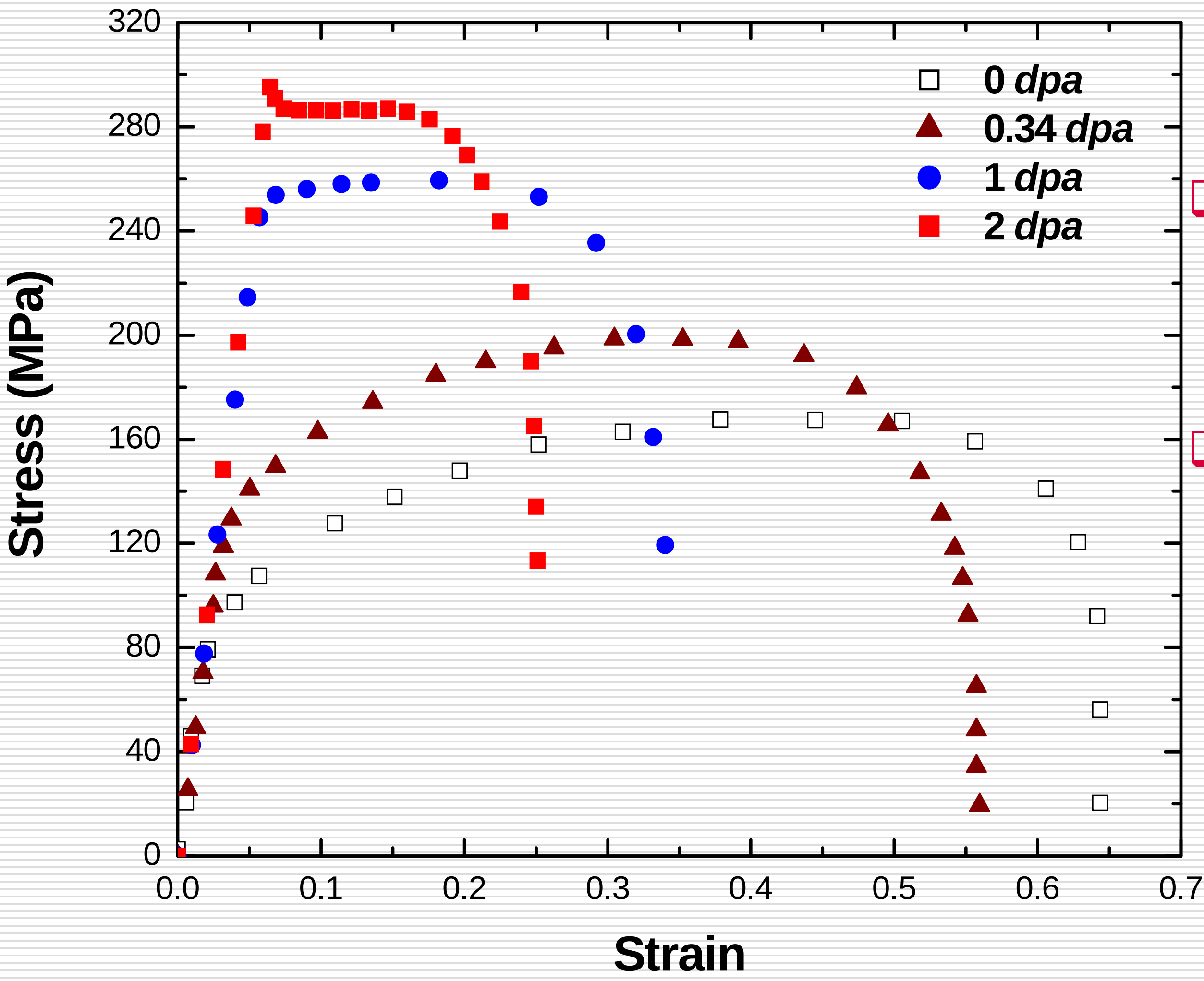
Instron Frame



Mini. Tensile Tester

Mechanical Testing

- Conventional Copper



- Radiation hardening
- Yield Phenomenon at 2 *dpa*

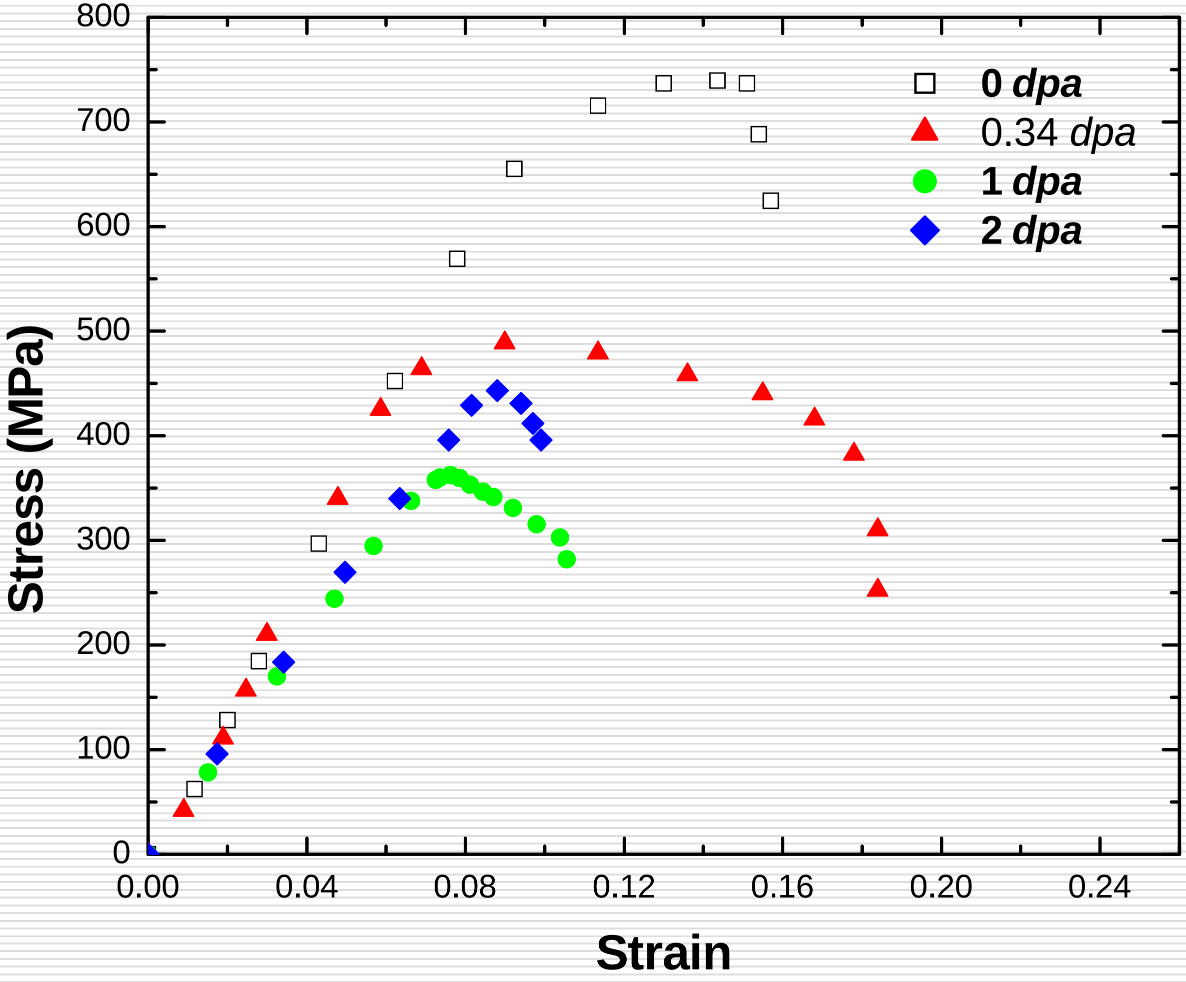
Summary of Mechanical Properties

- Conventional Copper

dpa	S_y (MPa)	UTS (MPa)	e_u%	e_t%	n
0	82±5	167.5±8	36.5	63.4	0.24
0.34	170.5±23	209.6±11	27.3	55.5	0.165
1	233.3±11	258.5+16	11.6	30.4	0.109
2	288.4±27	288.4±27	7.2	22.2	0.069

Mechanical Testing

- nc - Copper



- Radiation softening up to 1 *dpa* competes with radiation hardening
- Radiation hardening dominates at 2 *dpa*

Summary of Mechanical Properties

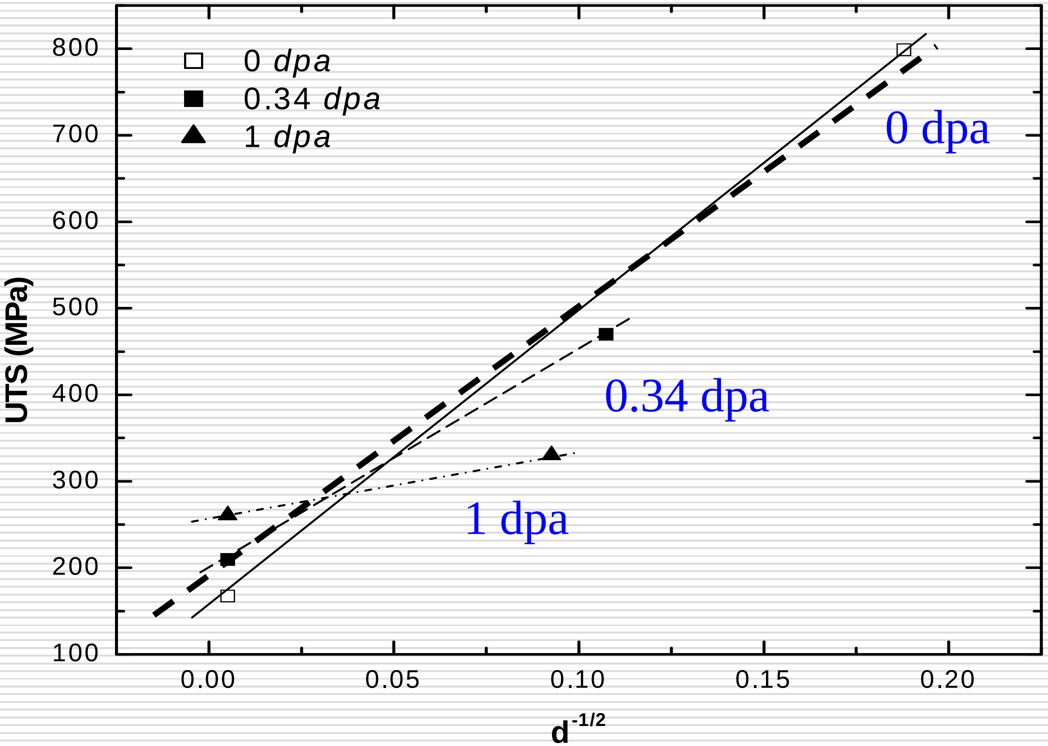
- nc - Copper

dpa	S_y (MPa)	UTS (MPa)	e_u%	e_t%	n
0	487±20	759.6±30	5.2	7.5	0.079
0.34	370±40	470.6±13	1.2	17.5	0.052
1	307.8±2	365.37+3	0.75	4.12	0.007
2	382.3±46	451.2±11	0.6	3.44	0.006

Hall-Petch Relationship

$$\sigma = \sigma_o + \frac{k_y}{\sqrt{d}} = \sigma_i + \sigma_s$$

d=grain-size,
σ_i=friction hardening and
σ_s=source hardening



Although the data follow general trend of Hall-Petch relation, note the decreased slope with increased radiation dose (dpa)

Ongoing Work

1. TEM investigation of samples irradiated at 1, 2 dpa

2. DSC to investigate phase transition and grain growth in nc-Cu

3. SEM of fracture surfaces of tested tensile samples

Summary & Conclusions

- **Conventional Copper**
  - Irradiated conventional copper agrees with the commonly observed radiation hardening and embrittlement
  - The strain hardening exponent, n, decreased as expected
  - Yield phenomenon is observed at 2 dpa.
- **Nanostructured Copper**
  - nc-Cu became softer after irradiation (yield strength, UTS and microhardness decreased following irradiation)
  - Radiation hardening appears to take place at 2 dpa
  - Low dislocation density and twin structure were observed
  - Grain size measurements revealed in-reactor grain-growth
- Further research is called for in characterizing the grain-size variation of radiation hardening