



Idaho State
UNIVERSITY

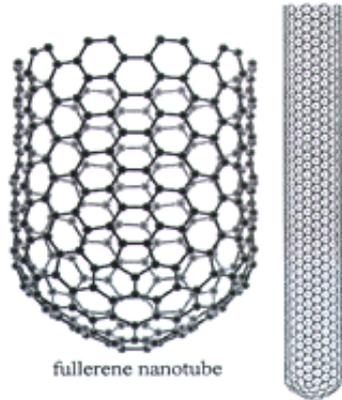
Enhancing irradiation tolerance of steels via nanostructuring by innovative manufacturing techniques

Haiming Wen

Idaho State University

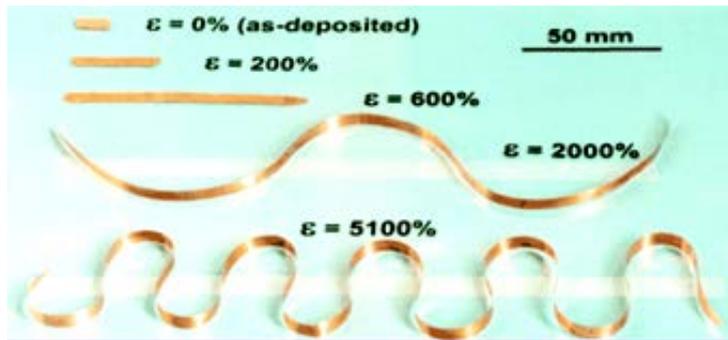
Why We Need to Go into NANO

Carbon Nanotubes



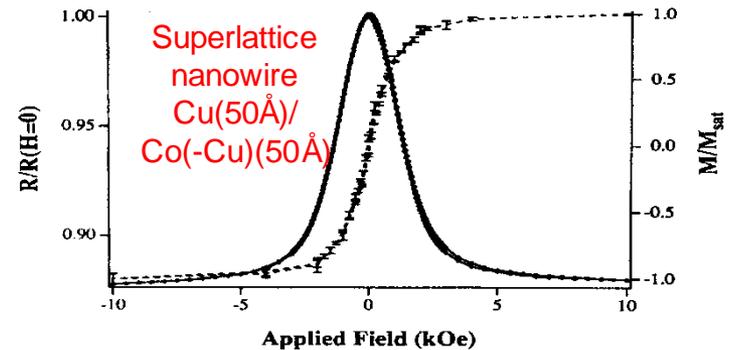
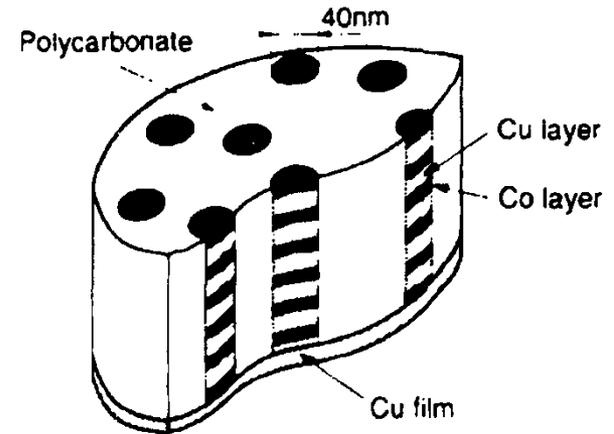
Conductive fine probe
with a few nm diameter

Superextensibility of nc-Cu at room T



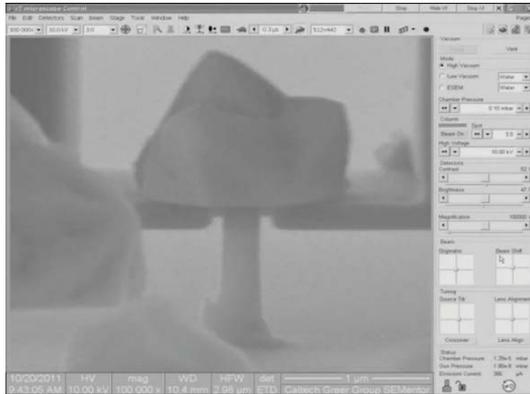
Lu et al. Science (2000)

Giant Magnetoresistance (GMR)

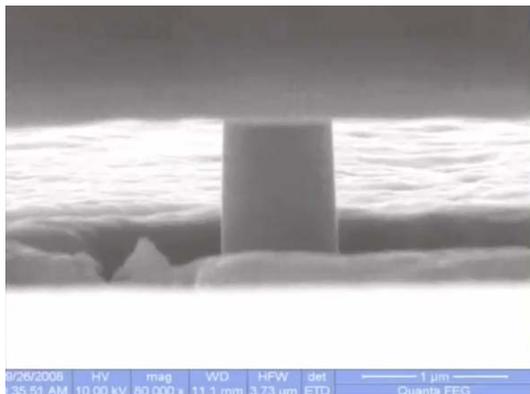


Schwarzacher, et al,
IEEE Trans. Magnetism (1996)

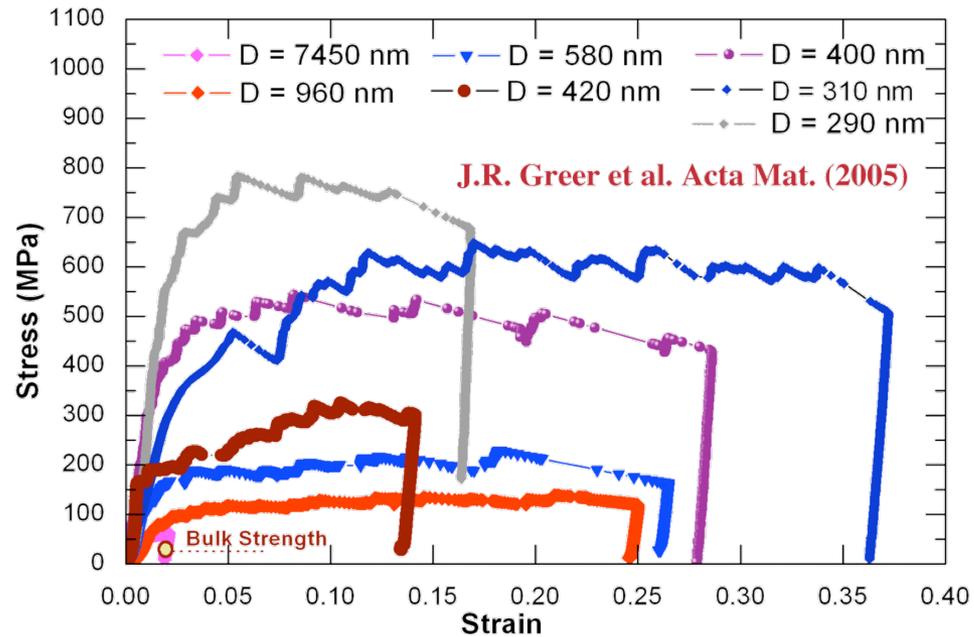
Why We Need to Go into NANO



1 micron



1 micron

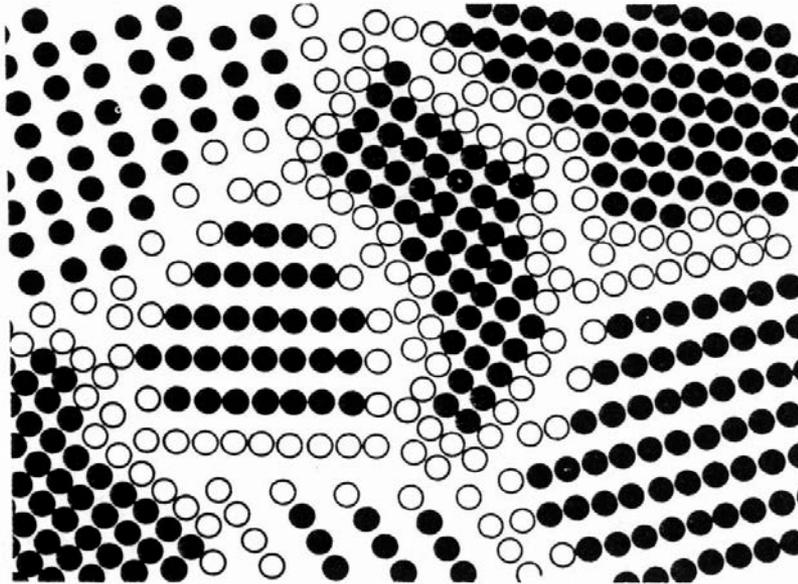


Decreasing Diameter

Single-crystalline metals

Smaller is Stronger

Concept of bulk nanostructured materials

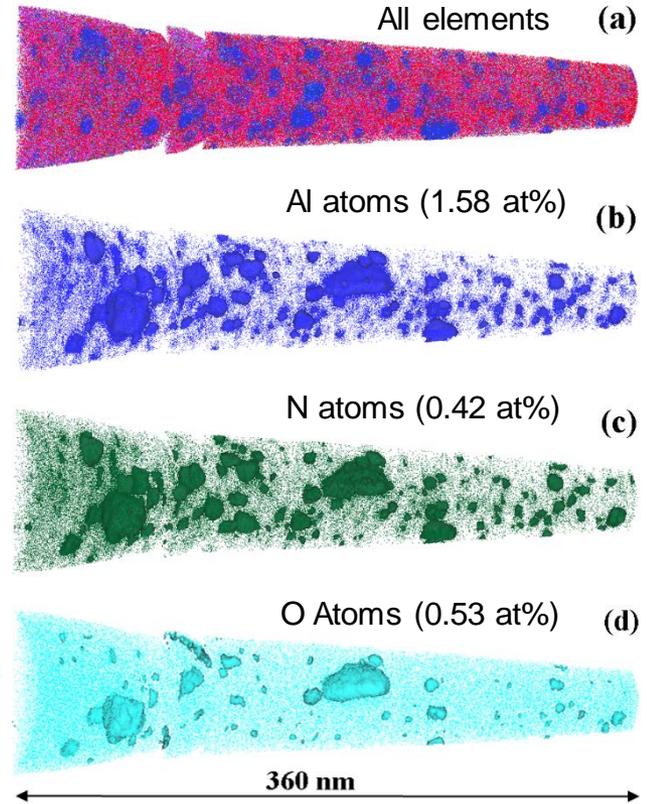
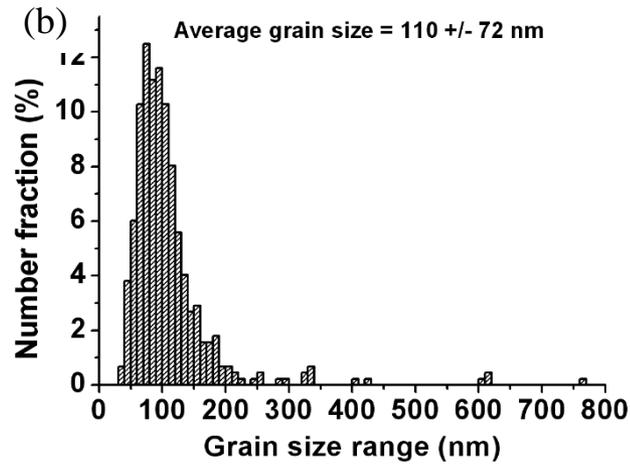
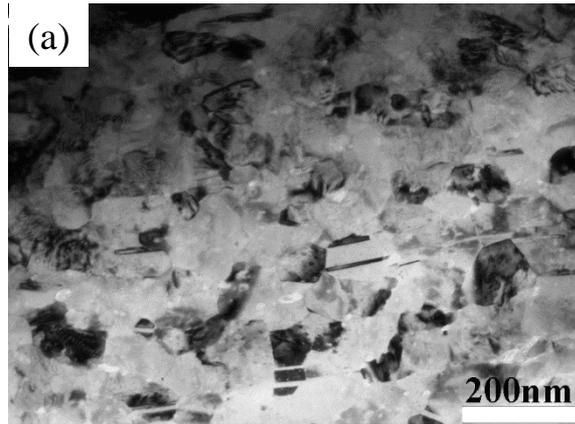


Single or multiple-phase polycrystals with structural features (grain, subgrain, dislocation cell, twin, etc.) smaller than 100 nm

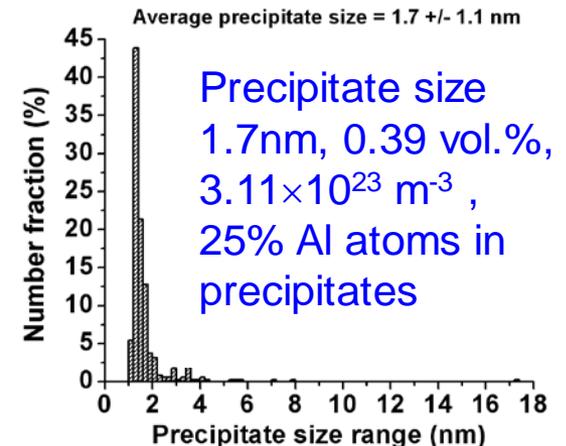
- $D=5$ nm, fraction of GBs=50%
- $D=100$ nm~ 1 μ m, ultrafine grained materials; $D=1$ ~ 10 μ m, fine grained materials; $D>10$ μ m, coarse grained conventional materials

H. Gleiter, in Proceedings of the second Risø International Symposium on Metallurgy and Materials Science, 1981, Denmark: Risø National Laboratory, Roskilde

High-strength bulk nanostructured brass



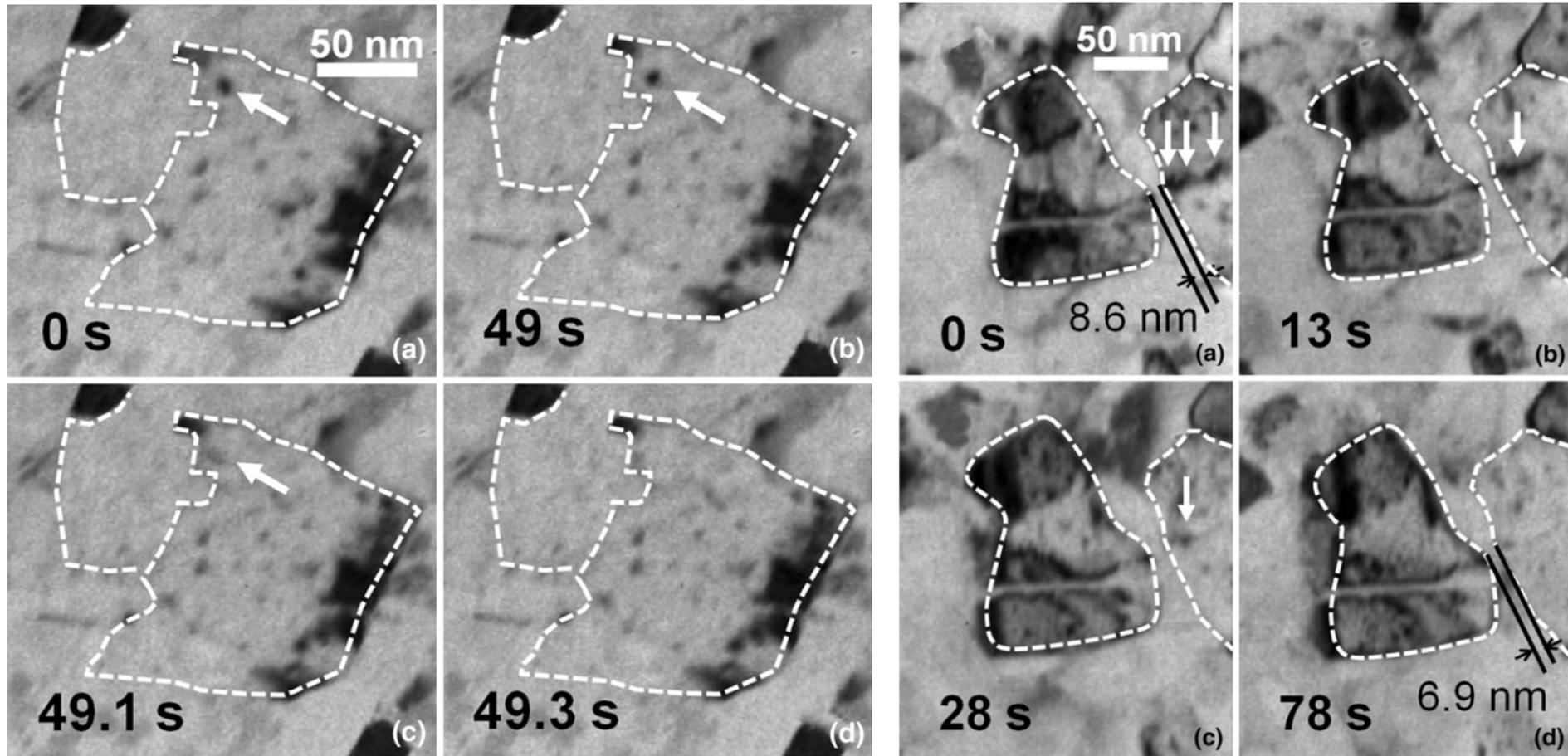
| Alloy composition | Stacking fault energy (mJ/m ²) | Processing condition | Yield strength (MPa) |
|-------------------------------|--|---|---------------------------|
| CuZn30 | 7 | Liquid nitrogen temperature dynamic plastic deformation | 717 |
| CuZn30 | 7 | HPT + cold rolling | 690 |
| CuZn30 | 7 | HPT | ~ 962 (1/3 microhardness) |
| CuZn30 | 7 | Cryomilling + room temperature milling | 877 |
| Cu-12.1 at % Al – 4.1% at% Zn | 7 | Cryomilling + room temperature milling | 1067 |
| Cu-16 at.% Al | 6 | ECAP | 740 |
| Cu-16 at.% Al | 6 | HPT | 820 |
| Cu-11 at.% Zn | 33 | ECAP | ~ 500 |



Wen HM, Lavernia EJ, Scripta Mater 67 (2012) 245

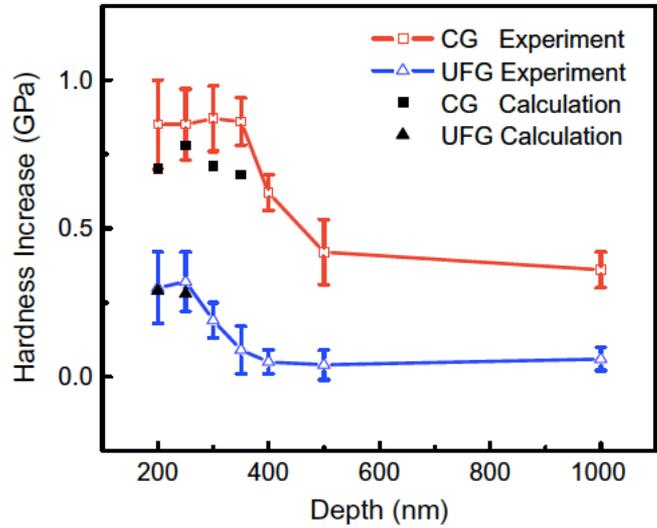
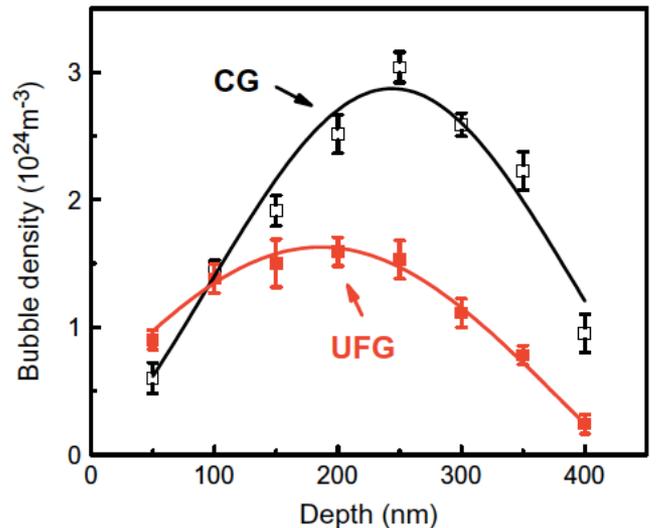
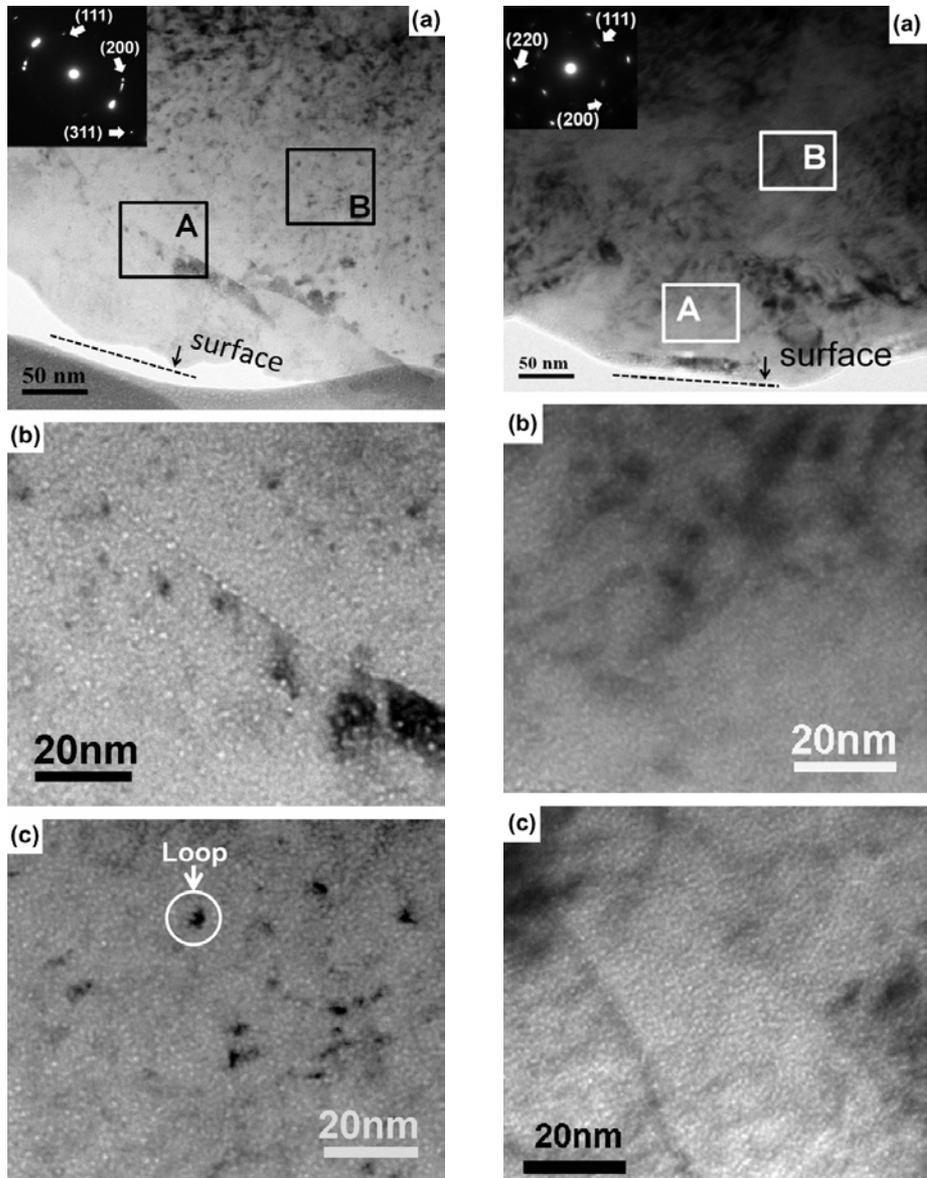
Wen HM, Lavernia EJ, et al, Acta Mater 61 (2013) 2769

GBs as sinks for irradiation defects



- In-situ TEM imaging during ion irradiation of NC Ni films
- Grain boundaries as sinks for irradiation-induced dislocation loops and segments

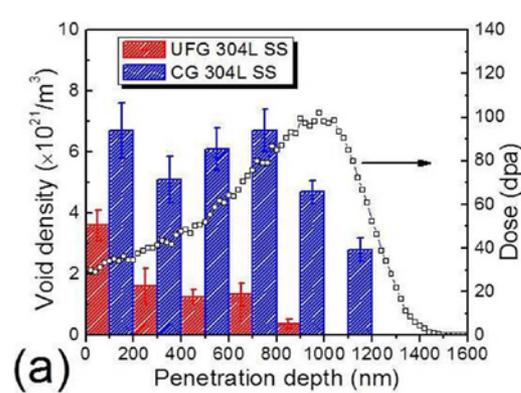
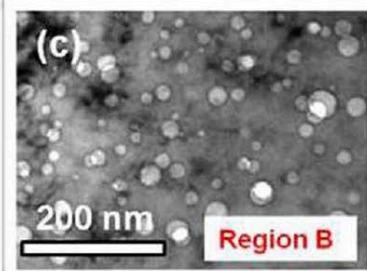
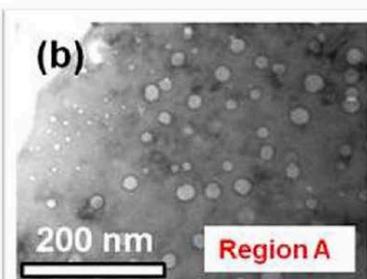
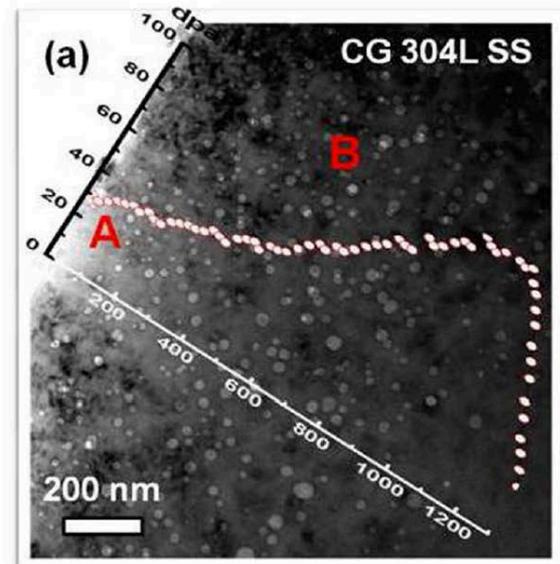
Radiation resistance of UFG Fe-Cr-Ni alloy



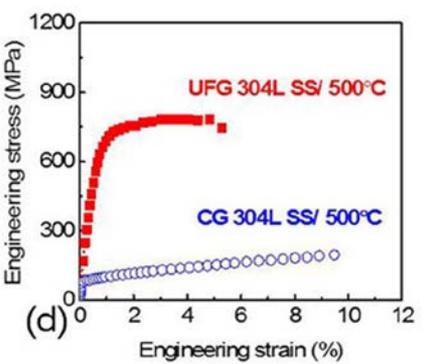
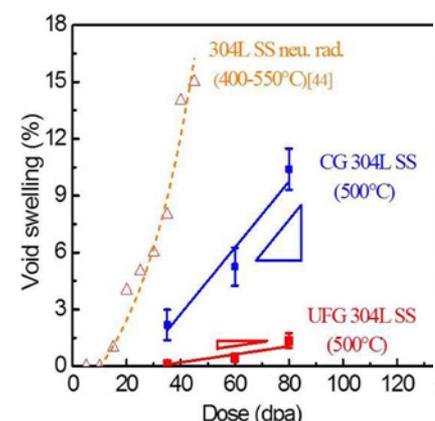
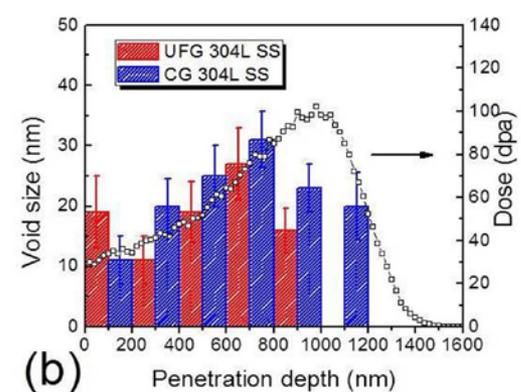
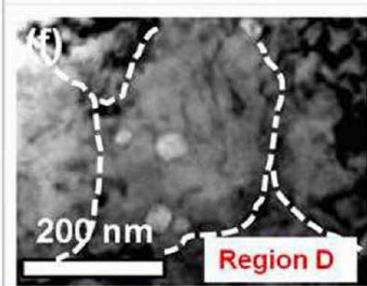
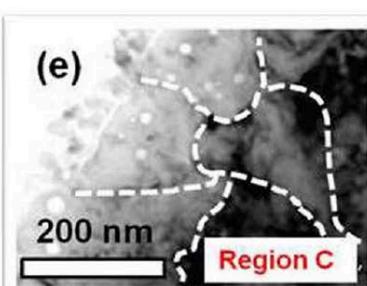
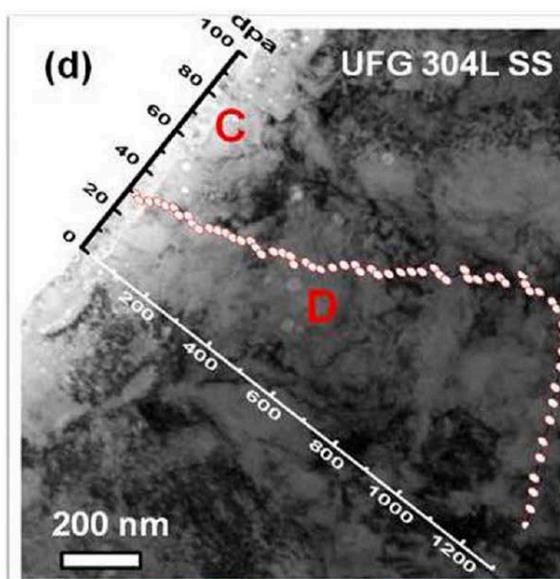
Sun C, et al., J Nucl Mater 420 (2012) 235

- Reduced He bubble density and irradiation hardening in UFG sample

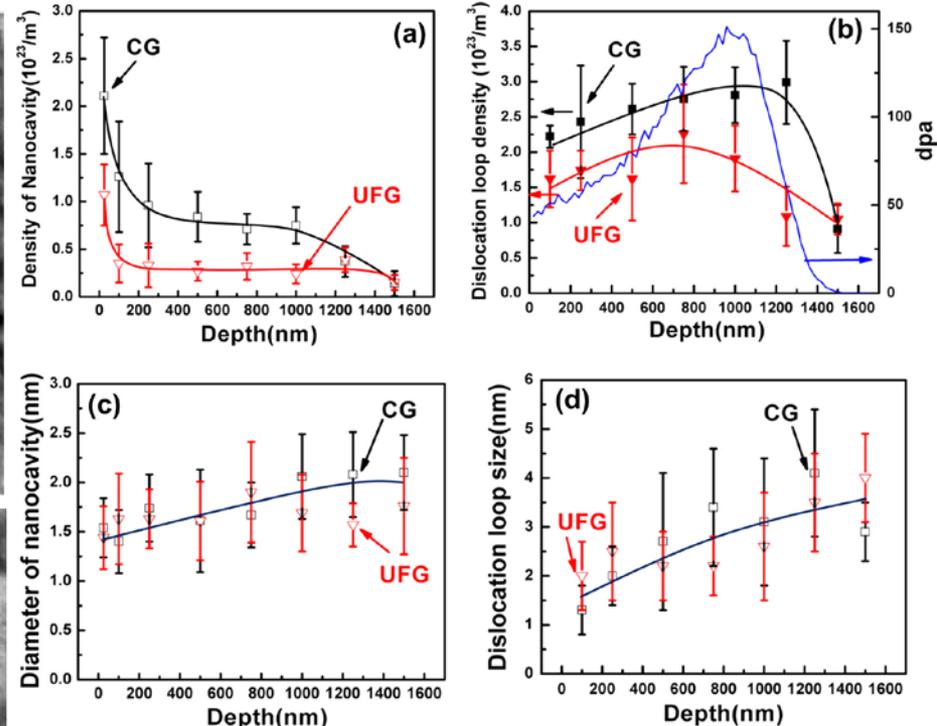
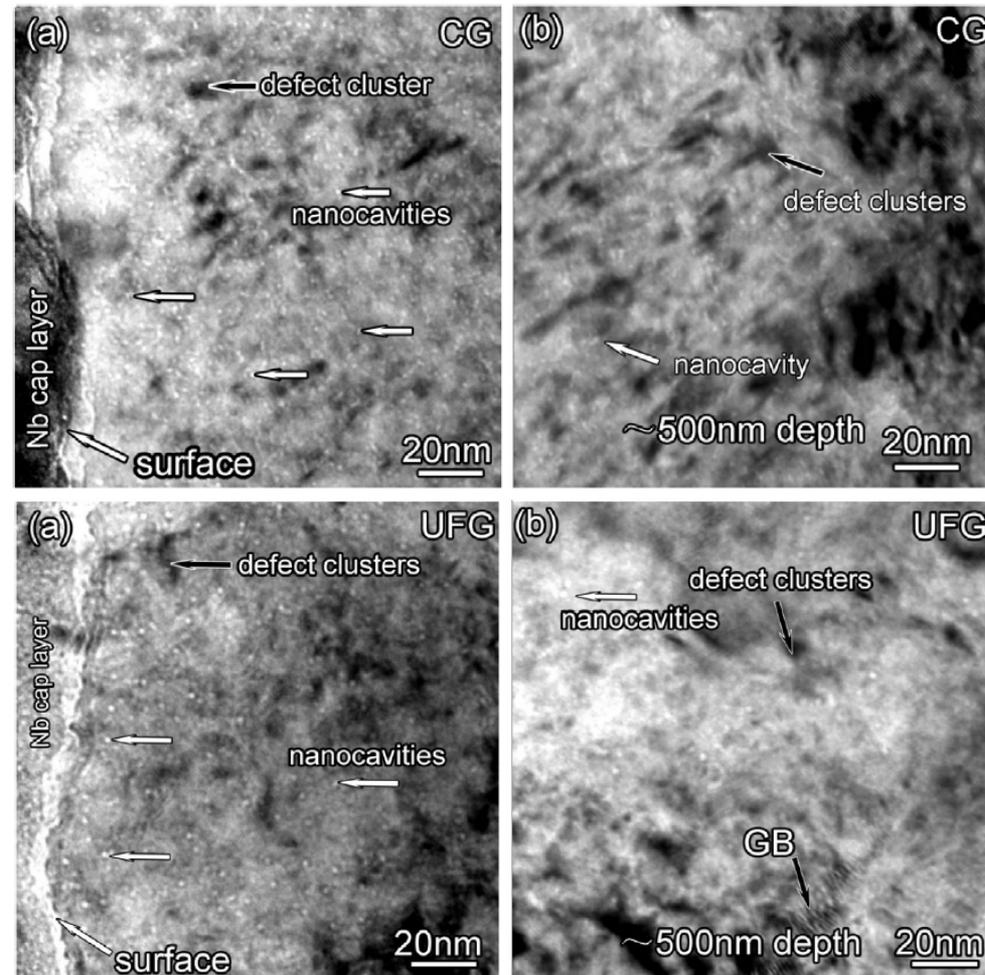
Radiation resistance of UFG 304 steel



- Much smaller void density and void swelling in UFG sample
- Much higher strength of UFG sample

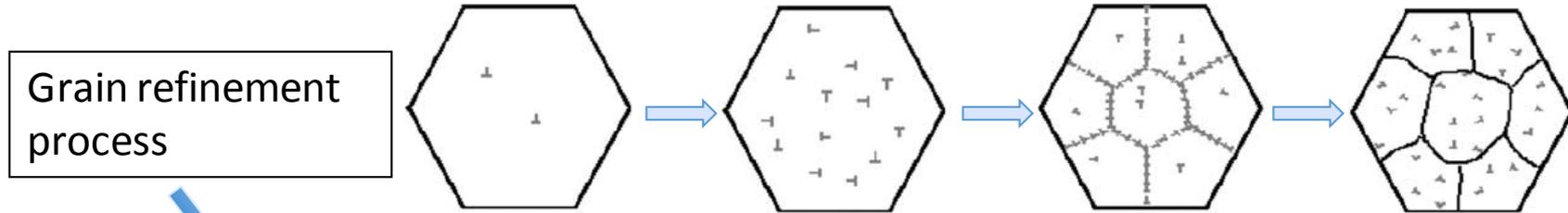


Radiation resistance of UFG T91 steel



- Lower density of nanocavity and dislocation loops in UFG sample

Manufacturing of bulk nanostructured metals



Grain refinement process

Top-down approach

severe plastic deformation (SPD)

equal-channel angular pressing (ECAP)

high pressure torsion (HPT)

Bottom-up approach

Step I: producing particles or agglomerates

inert gas condensation

Mechanical milling

chemical methods

Step II: consolidating particles or agglomerates

CIP/HIP

quasi-isostatic forging

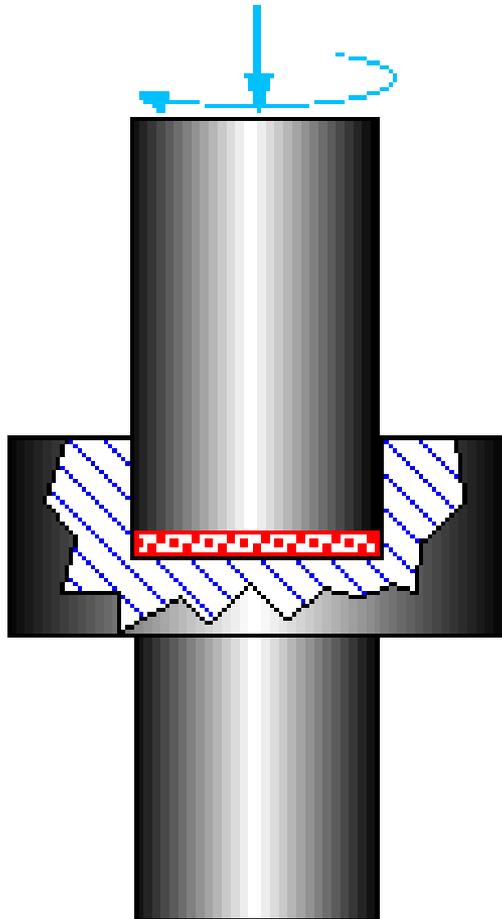
spark plasma sintering

SPD { ECAP

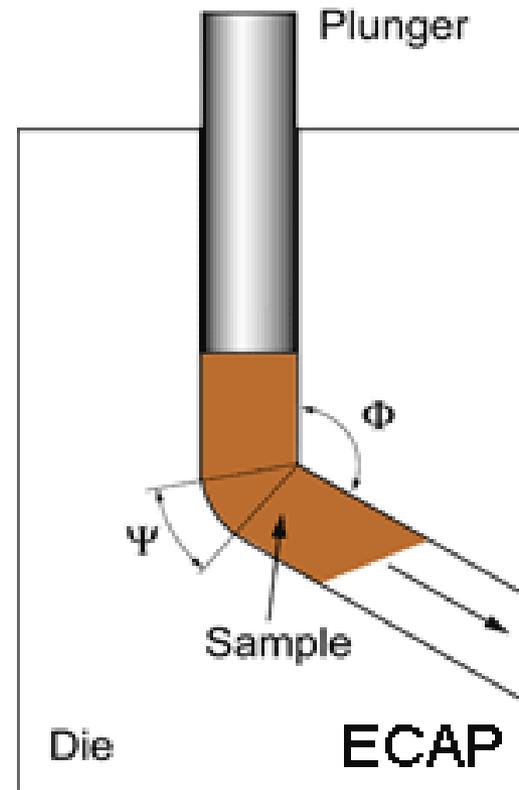
HPT

Severe plastic deformation (SPD)

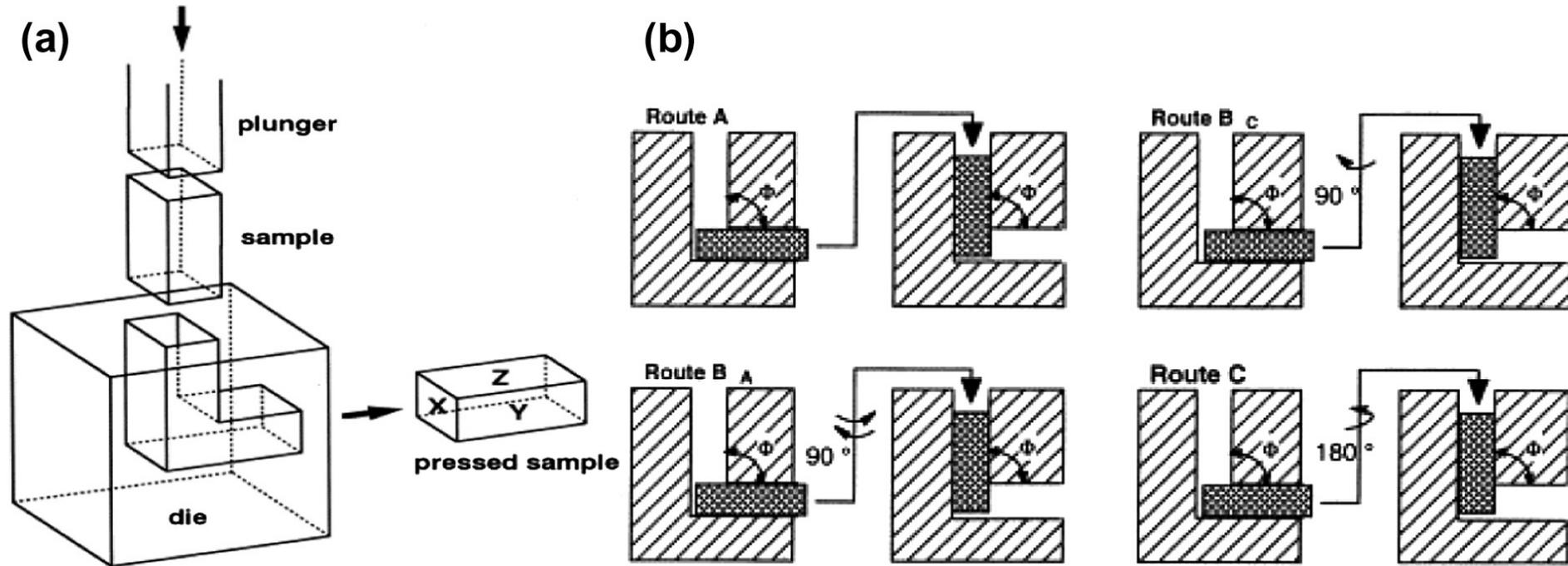
High pressure torsion
(HPT)



Equal-channel angular pressing
(ECAP)



Equal-channel angular pressing



New project

- **Enhancing irradiation tolerance of steels via nanostructuring by innovative manufacturing techniques**

- **Team members:**

PI: Haiming Wen

Co-PIs: James I. Cole (INL), Yongfeng Zhang (Co-PI, INL)

Isabella J. van Rooyen (INL)

NSUF Technical Lead: James K. Jewell

Other ISU team members: Ishtiaque Robin (graduate student) and a postdoc (to be hired)

- **Project duration:** 10/1/2016 – 09/30/2021
- **Funding amount:** \$500 K + NSUF facility access in the value of ~\$2.4 million

Objectives

- Establish/enhance our fundamental understanding of irradiation effects in ultrafine-grained or nanocrystalline steels produced by ECAP or HPT.
- Assess the potential applications of ECAP and HPT in fabricating materials for applications in current and advanced reactors

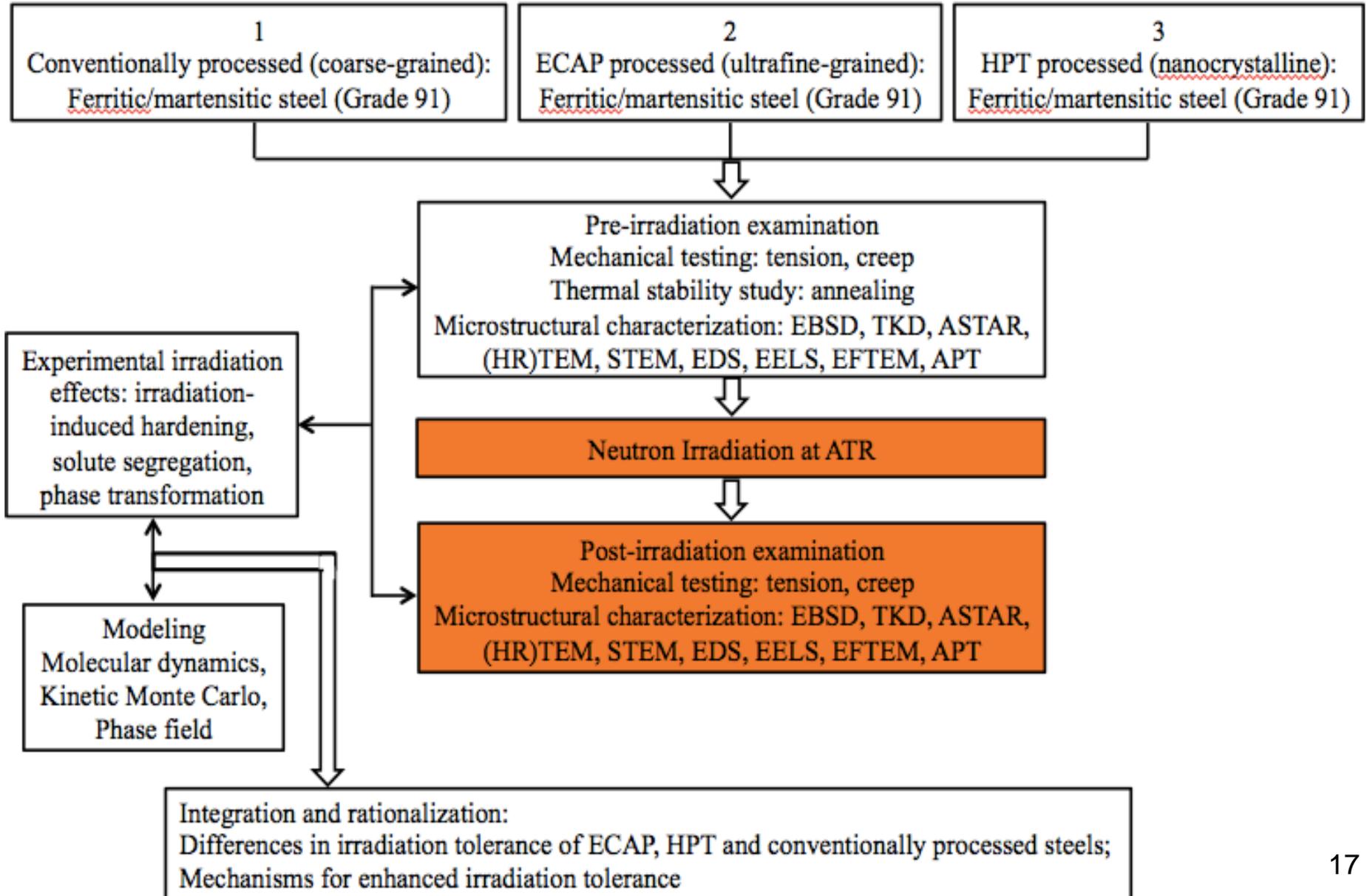
Novelty

- UFG or NC austenitic or F/M steels have not been neutron irradiated, and their performance under neutron irradiation is not established.
- This work will establish the performance of UFG and NC variants of reactor structural and cladding steels produced by ECAP or HPT, under neutron irradiation at relevant reactor operating temperatures, which has not previously been accomplished.

Samples

- Sample set 1: conventionally processed (coarse-grained) F/M Grade 91 steel
- Sample set 2: ECAP processed (ultrafine-grained) F/M Grade 91 steel
- Sample set 3: HPT processed (nanocrystalline) F/M Grade 91 steel
- Sample set 4: conventionally processed (coarse-grained) austenitic 316 steel
- Sample set 5: ECAP processed (ultrafine-grained) austenitic 316 steel
- Sample set 6: HPT processed (nanocrystalline) austenitic 316 steel
- Sample set 7: conventionally processed (coarse-grained) austenitic 304 steel
- Sample set 8: ECAP processed (ultrafine-grained) austenitic 304 steel
- Sample set 9: HPT processed (nanocrystalline) austenitic 304 steel
- Sample set 10: conventionally processed (coarse-grained) ferritic Kanthanl D
- Sample set 11: ECAP processed (ultrafine-grained) ferritic Kanthanl D
- Sample set 12: HPT processed (nanocrystalline) ferritic Kanthanl D

Project flow chart



Pre-irradiation characterization

- ***Mechanical testing***
tensile and creep tests
- ***Microstructural characterization***
grain sizes/morphologies, dislocations, grain boundary characteristics, solute segregation at grain boundaries, pre-existing precipitates, and phase boundaries.
- ***Thermal stability study: annealing***

Neutron irradiation

- Irradiation conditions
 - 300 °C: 2 dpa, 6 dpa;
 - 500 °C: 2 dpa, 6 dpa
- Non-instrumented standard capsule experiments
- Melt wires to monitor temperature

Post-irradiation characterization

- *Mechanical testing*

tensile and creep (?) tests

irradiation-induced hardening and decrease in ductility

irradiation effects less significant in UFG and NC samples

- *Microstructural characterization*

- ✓ Neutron irradiation induced defects (such as dislocation loops), irradiation-induced solute segregation and precipitation, grain boundary characteristics, interaction between irradiation-induced defects and grain boundaries, and irradiation-induced solute segregation at grain boundaries in relation to the specific characteristics of grain boundaries

- ✓ (HR)TEM, STEM-EDS, STEM-EELS, APT, EBSD, TKD, PED

Modelling

- Irradiation-induced segregation
molecular dynamics simulations
lattice kinetic Monte Carlo
- Thermal stability of nanocrystalline alloys before and after irradiation
phase field based MARMOT code
- Irradiation-induced hardening
molecular dynamics simulations

Integration and rationalization

- Feasibility assessment of applications of ECAP and HPT in fabricating materials with improved performance for current and advanced reactors
- Irradiation effects: irradiation-induced hardening, solute segregation and phase transformation (precipitation)
- Irradiation tolerance as a function of grain size
- Influence of specific characteristics of GBs on interaction between irradiation-induced defects and GBs
- Correlation between specific characteristics of GBs and irradiation-induced solute segregation/precipitation at GBs
- Role of solute segregation/precipitation at GBs in pinning GBs and stabilizing ultrafine-grained and nanocrystalline structures