Probing Nanoscale Damage Gradients in Irradiated Metals using Nano-mechanical Test Techniques

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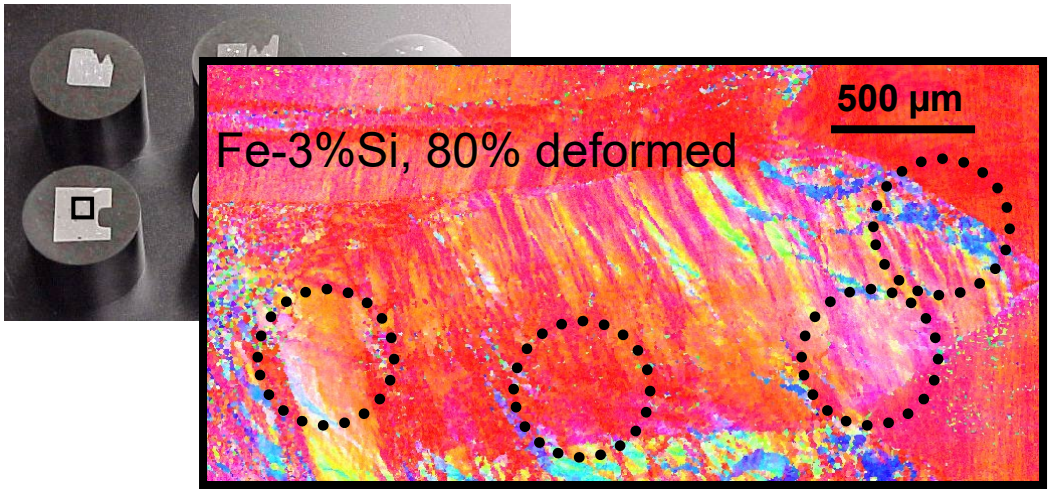
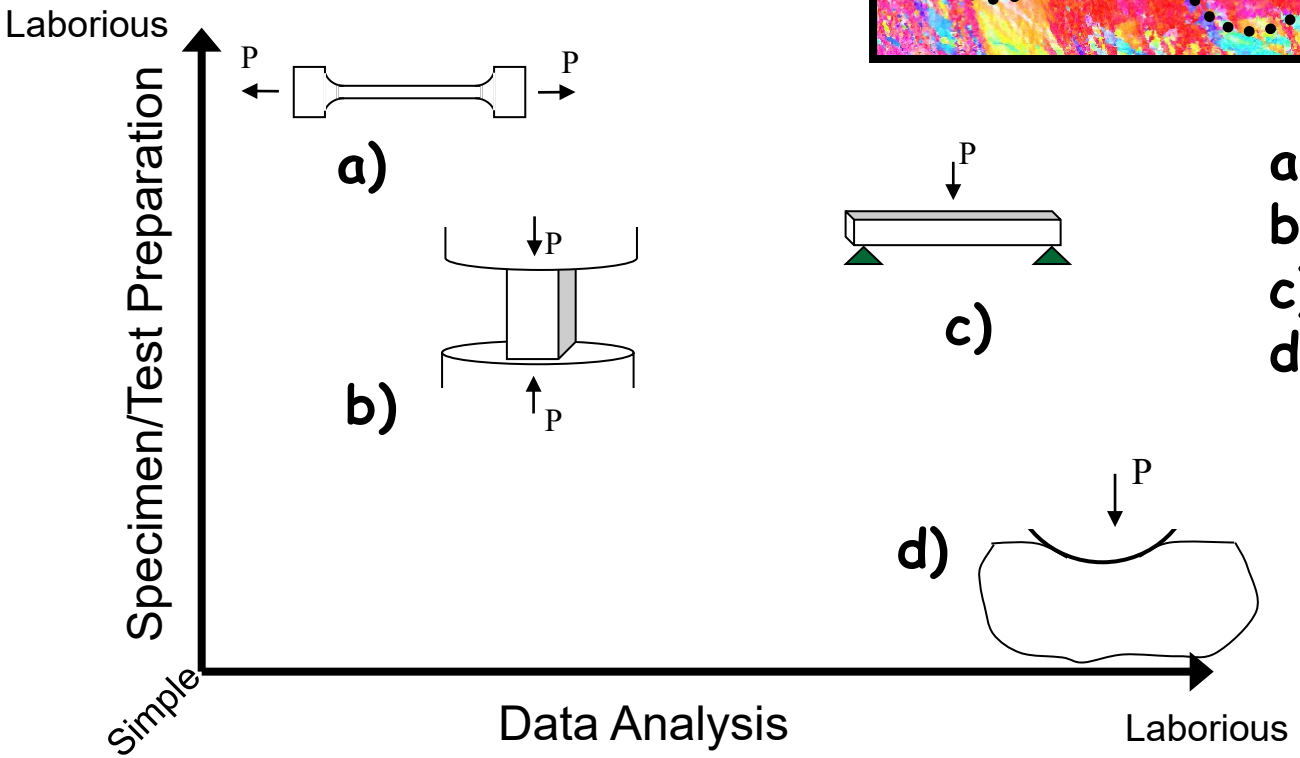
IOWA STATE UNIVERSITY
OF SCIENCE AND TECHNOLOGY

Mechanical Testing Tools at the Micro-scale

Crystal Plasticity models at the micron (grain) scale:

missing information
– grain scale heterogeneity of dislocation density,
both between grains and within the same grain





a) Tensile
b) Compression
c) Bending
d) Indentation

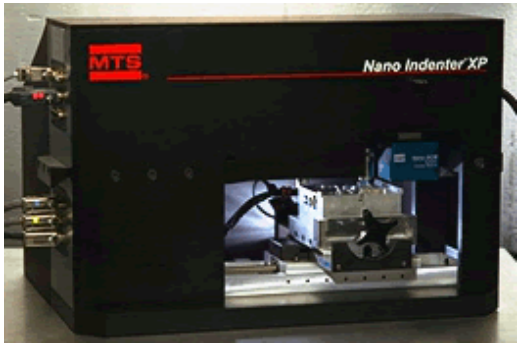
Investigating local mechanical response at the micro- and nano-scales: Capabilities under extreme conditions

Strain rate, per sec (9 orders of magnitude)

10^{4} 10^{3} 10^{2} 10^{1} 10^{0} 10^{-1} 10^{-2} 10^{-3} 10^{-4}

Ex-situ (in-air) indenters

Hysitron TI950 Triboindenter

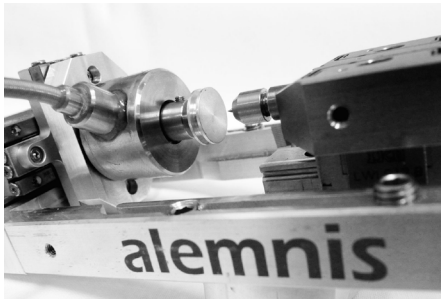


Nanoindenter® XP
LANL Laboratory Education Equipment Gift (LEEG) Program 2017

zwickiLine+

In-situ indenters

Hysitron PI85 SEM PicoIndenter

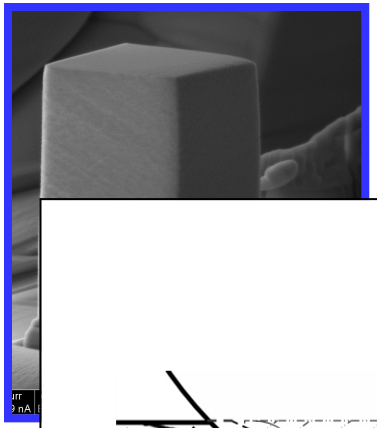


Alemnis Indenter System
DURIP 2021
DOE 2018 NEUP Infrastructure Support

1000 °C Elevated Temp | Cryo Temp -150 °C

~µN Nano 10mN > Load range Micro 2.5N > Meso 2.5 kN

Investigating local mechanical response at the micro- and nano-scales: *In-situ* SEM straining capabilities

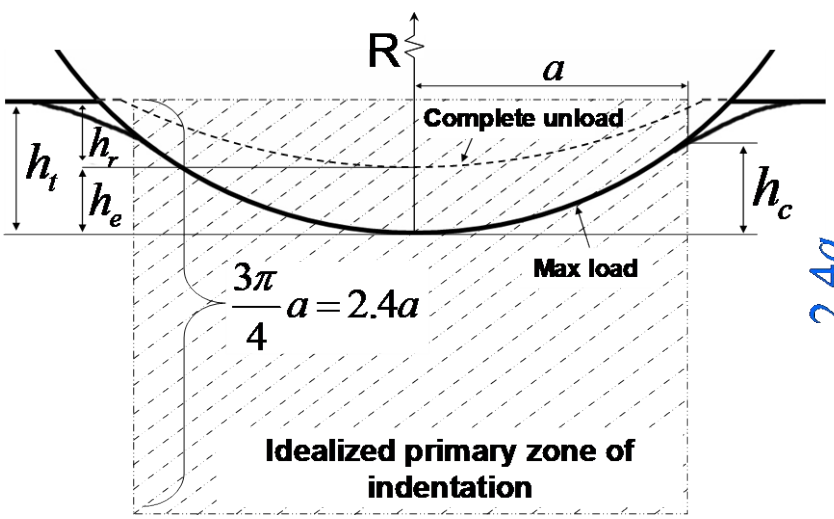


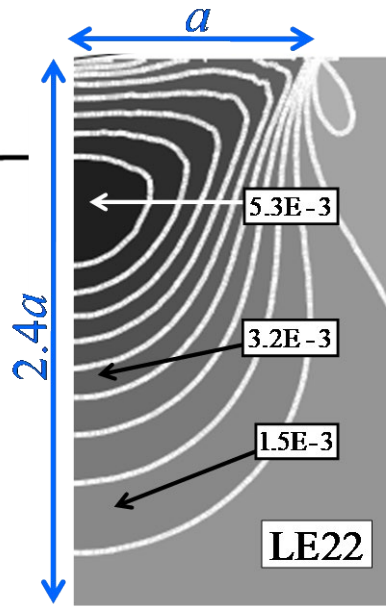
Micro-Compression

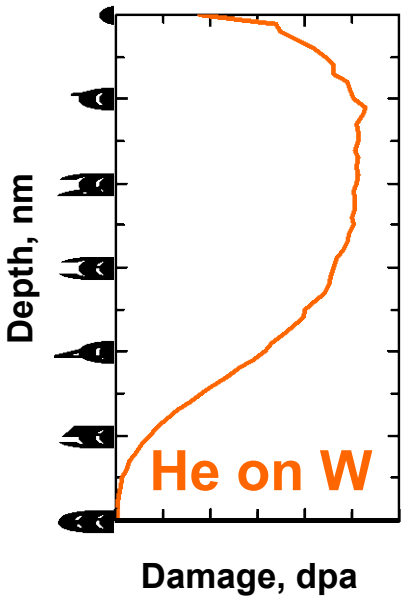
Load Cases **Samples** **Properties**

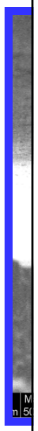
Nanowires,

E, σ_y, σ_f, n



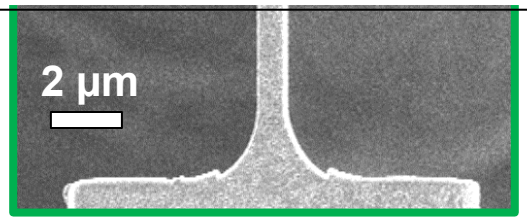






Cantilever
Micro-Bending

Micro-Tension



MEMS/NEMS,
milled, machined
structures

K_c

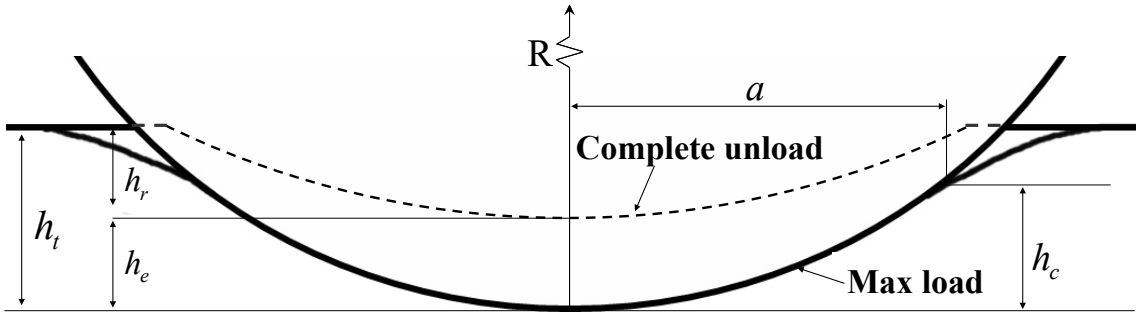
Outline

- Extraction of Spherical Nanoindentation Stress Strain curves
- Indentation on anisotropic samples
  - Characterizing the anisotropy in indentation modulus
  - Characterizing the local indentation yield strength
- Probing nanoscale damage gradients with spherical nanoindentation
- Comparing small scale mechanical test techniques for measuring irradiation hardening

Spherical Nanoindentation Basics

Hertz Principle

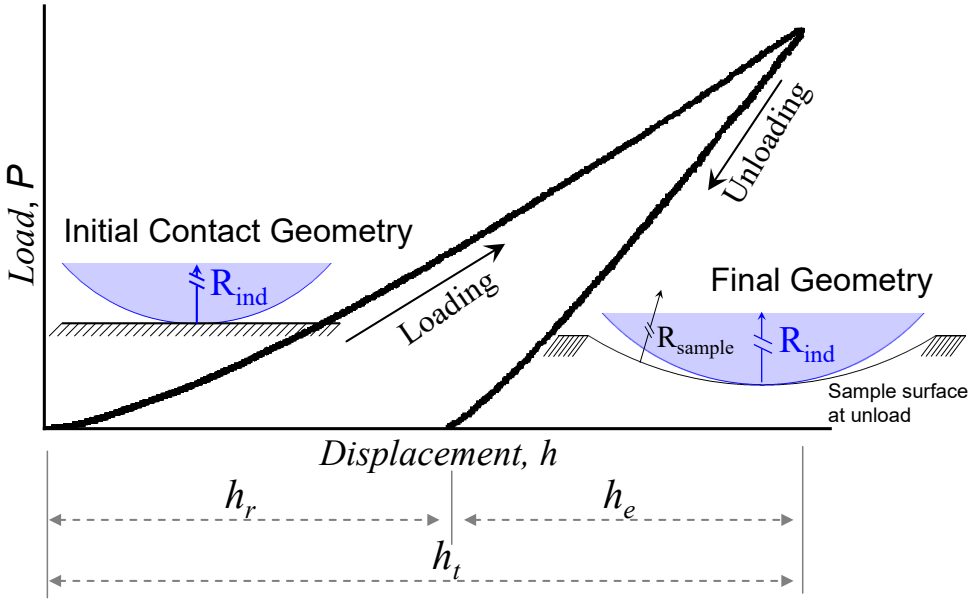
$$h_e = \left(\frac{3P}{4E_{eff}}\right)^{\frac{2}{3}}\left(\frac{1}{R_{eff}}\right)^{\frac{1}{3}}$$



$$\frac{1}{E_{eff}} = \left(\frac{1-v^2}{E}\right)_{sample} + \left(\frac{1-v^2}{E}\right)_{indenter}$$

$$\frac{1}{R_{eff}} = \frac{1}{R_i} + \frac{1}{R_s} \qquad a = fn(R_{eff})$$

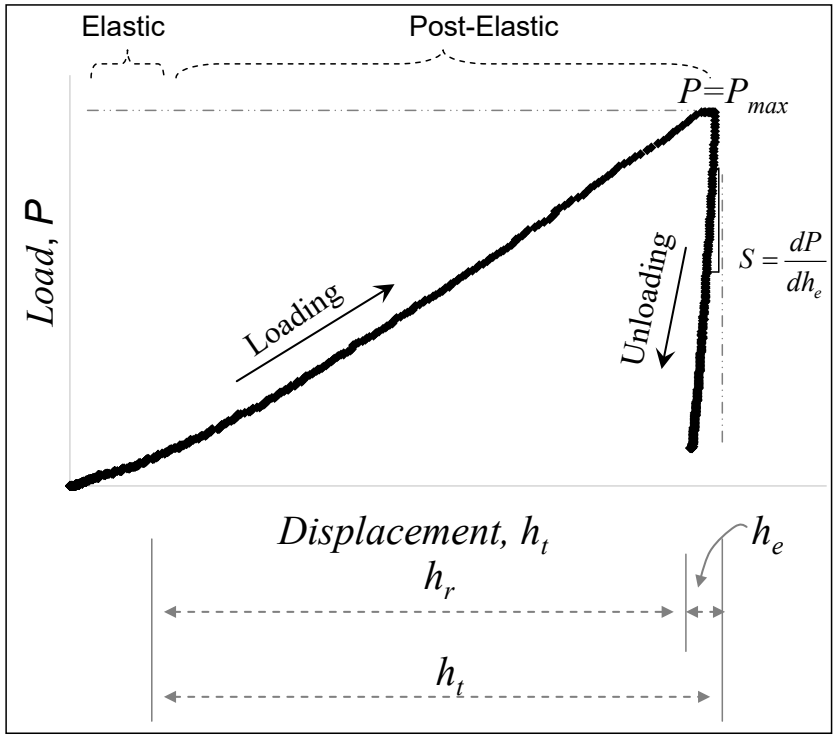
Assumptions: linear elastic, isotropic material response, frictionless contact

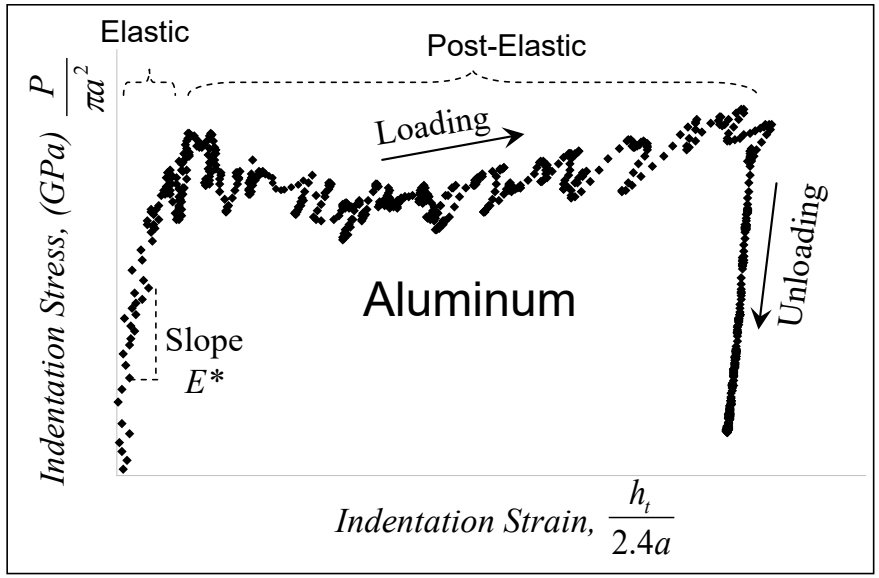


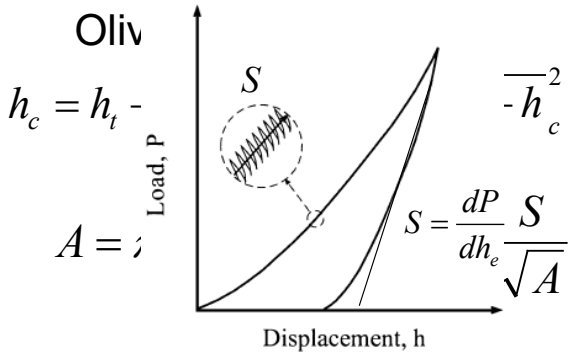
E_{eff} = *Effective Modulus*
E_i = *Modulus of Indenter*
E_s = *Modulus of Sample*
v_i = *Poisson Ratio of Indenter*
v_s = *Poisson Ratio of Sample*
R_{eff} = *Effective indentation radius*
R_i = *Radius of curvature of the indenter tip*
P = *Load*
h_e = *elastic depth*
a = *contact radius*

Hertz, H., *Miscellaneous Papers*. 1896, New York: MacMillan and Co., Ltd. .

Generating Nanoindentation Stress and Strain Curves







a) determine an effective zero point

b) calculate E_{eff} from the initial elastic loading segment where $R_{eff} = R_i$

c) estimate the contact radius $a = \frac{S}{2E_{eff}}$

d) $\sigma_{ind} = E_{eff}\varepsilon_{ind}$

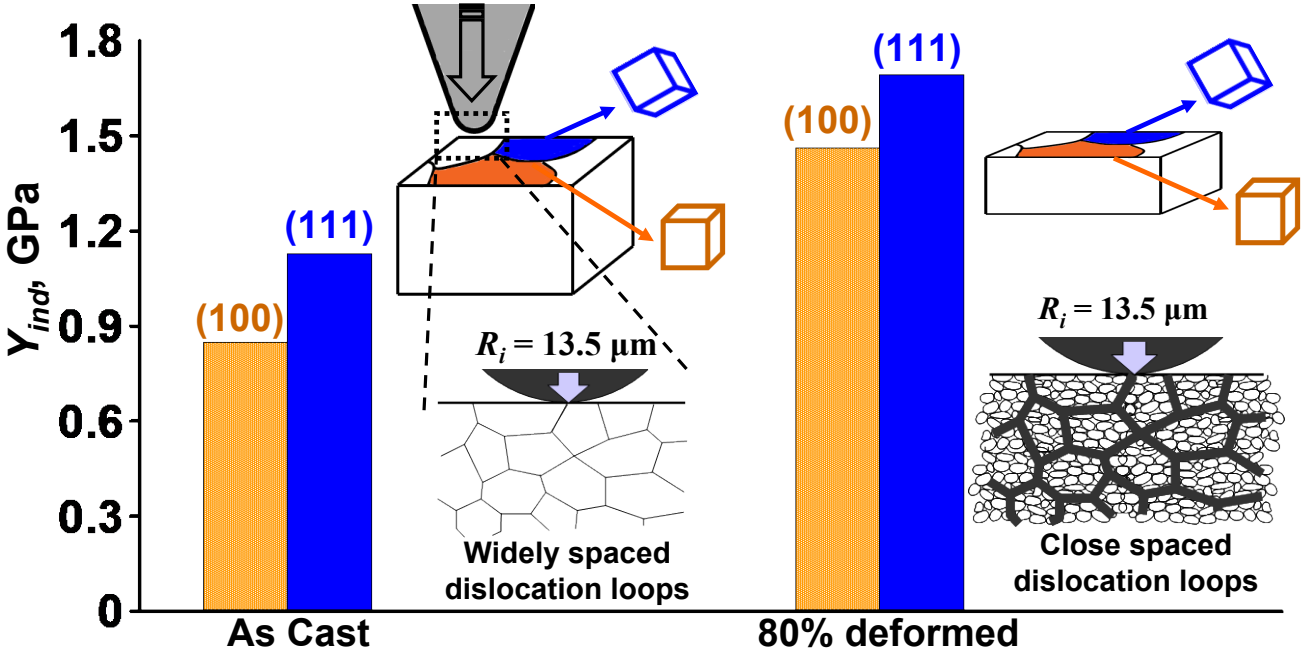
$$\sigma_{ind} = \frac{P}{\pi a^2} \qquad \varepsilon_{ind} = \frac{4}{3\pi}\frac{h_t}{a}$$

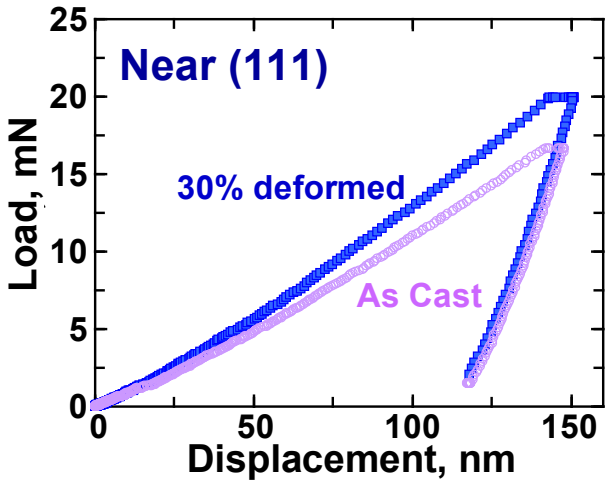
Oliver, W.C. and Pharr, G.M., J.Mat. Res., 1992 7(6): p. 1564-1580.

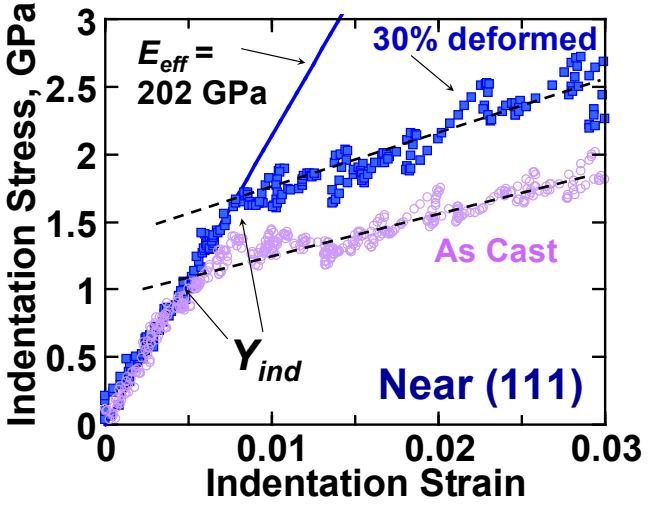
X. Li, B. Bhushan / Materials Characterization 48 (2002) 11–36

S. R. Kalidindi, S. Pathak, Acta Mat., 56 (2008) 3533-42

Nanoindentation on anisotropic samples



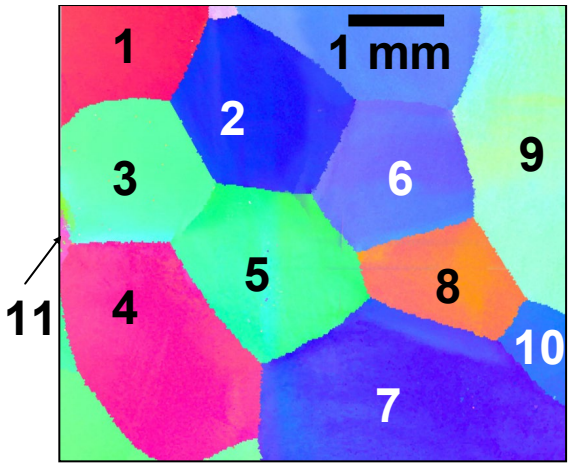


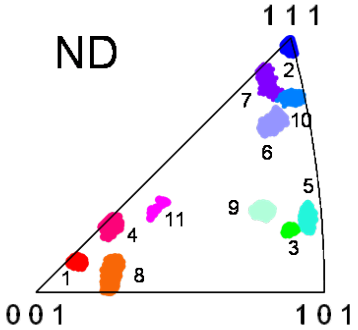


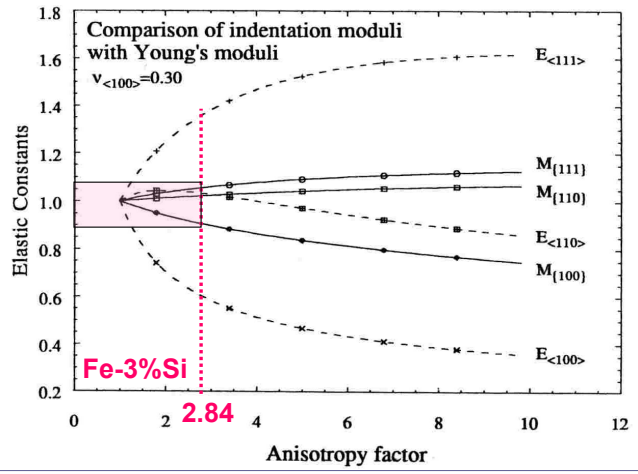
Hertz theory for anisotropic samples, Yield Surface Maps

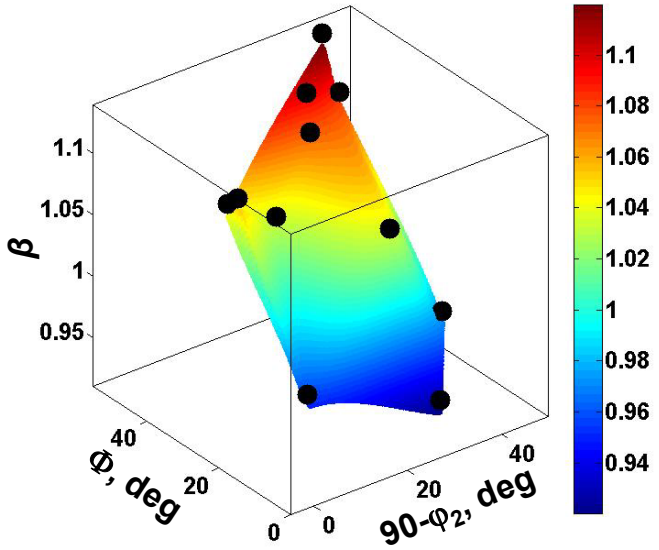
$$\frac{1}{E_{eff}} = \frac{1}{\beta}\left(\frac{1-\nu_s^2}{E_s}\right)_{isotropic} + \left(\frac{1-\nu_i^2}{E_i}\right)_{indenter}$$

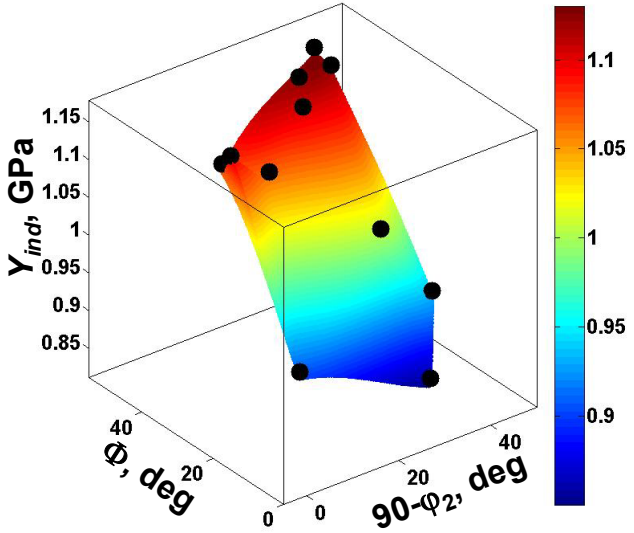
Fe-3%Si As cast











S. Pathak, D. Stojakovic, S.R. Kalidindi. Acta Mat. 2009

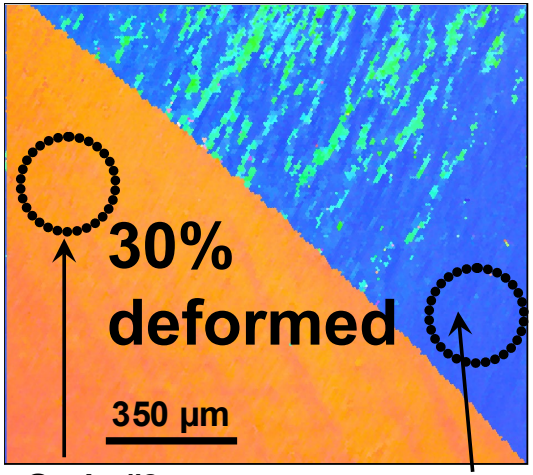
Vlassak, J.J., Nix, W.D., Philosophical Magazine A 1993.

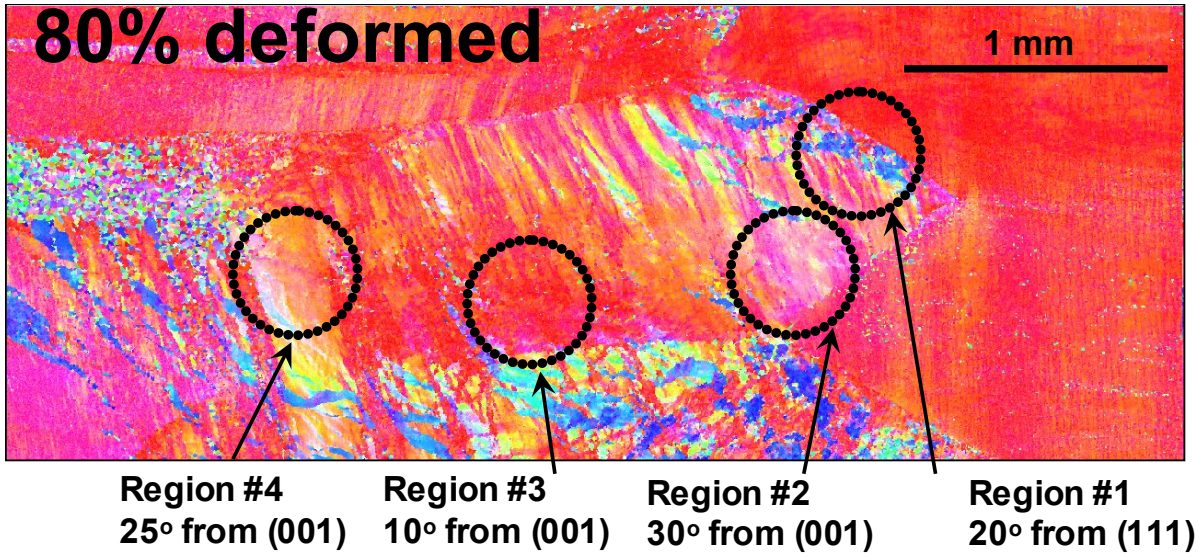
30% deformed Fe-3%Si				
Grain #	**Misorientation**	**Estimated Y_{ind} in the annealed condition, GPa**	**Measured Y_{ind} in the deformed condition, GPa**	**% change in τ_{CRSS}**
1	7° from (111)	1.12	1.31±0.13	16.9%
2	14° from (001)	0.90	1.07±0.05	18.9%
80% deformed Fe-3%Si				
Region #	**Misorientation**	**Estimated Y_{ind} in the annealed condition, GPa**	**Measured Y_{ind} in the deformed condition, GPa**	**% change in τ_{CRSS}**
1	20° from (111)	1.12	1.69±0.06	50.9%
2	30° from (001)	1.00	1.46±0.07	46.0%
4	25° from (001)	0.92	1.37±0.07	48.9%
3	10° from (001)	0.85	1.27±0.08	49.4%

Estimating $\Delta\tau_{CRSS}$ for deformed samples

$$Y_{ind} = M(\Phi, \varphi_2)\,\tau_{CRSS}$$

$$\Delta\tau_{CRSS} \propto \sqrt{\rho}$$

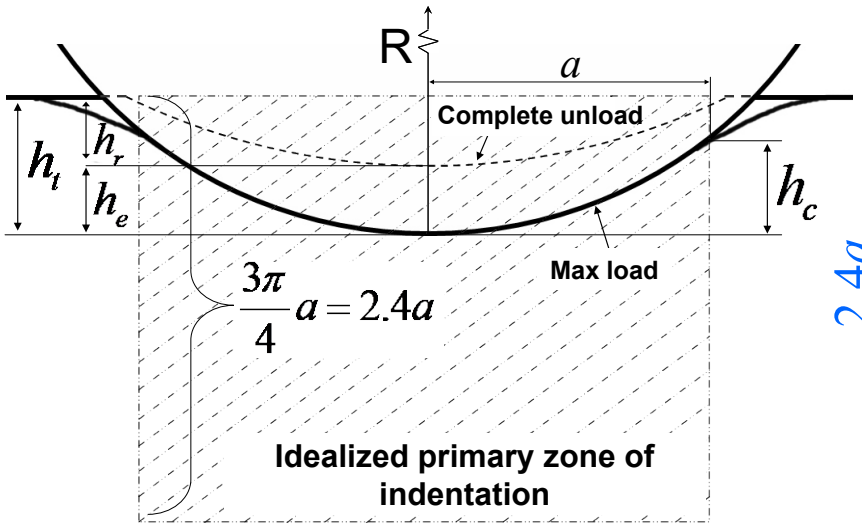


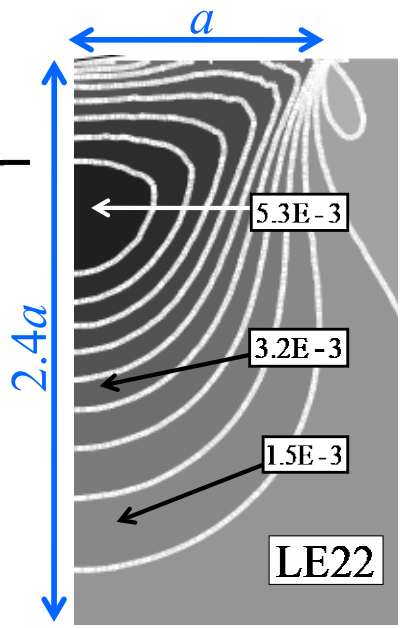


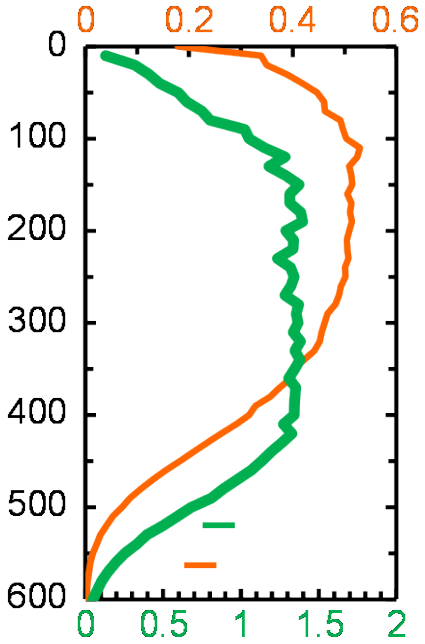
Outline

- Extraction of Spherical Nanoindentation Stress Strain curves
- Indentation on anisotropic samples
- Mechanical response of near grain boundary regions
- Probing nanoscale damage gradients with spherical nanoindentation
- Comparing small scale mechanical test techniques for measuring irradiation hardening

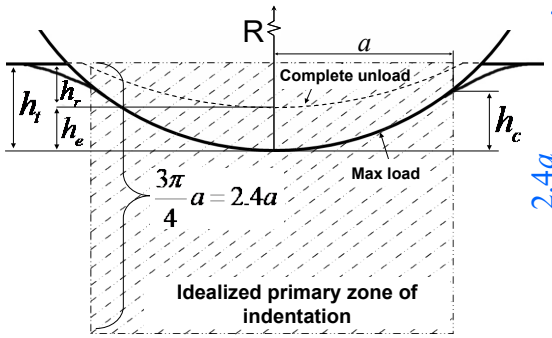
Radiation depth vs. indentation zone

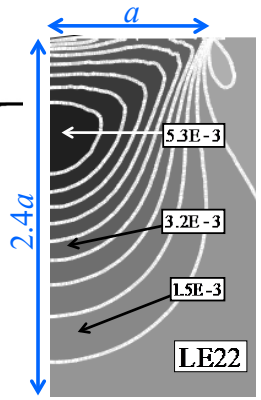


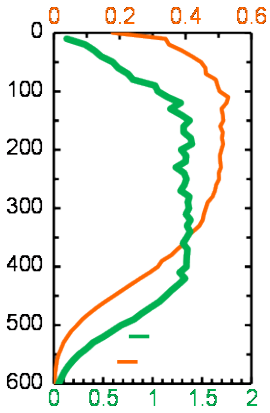


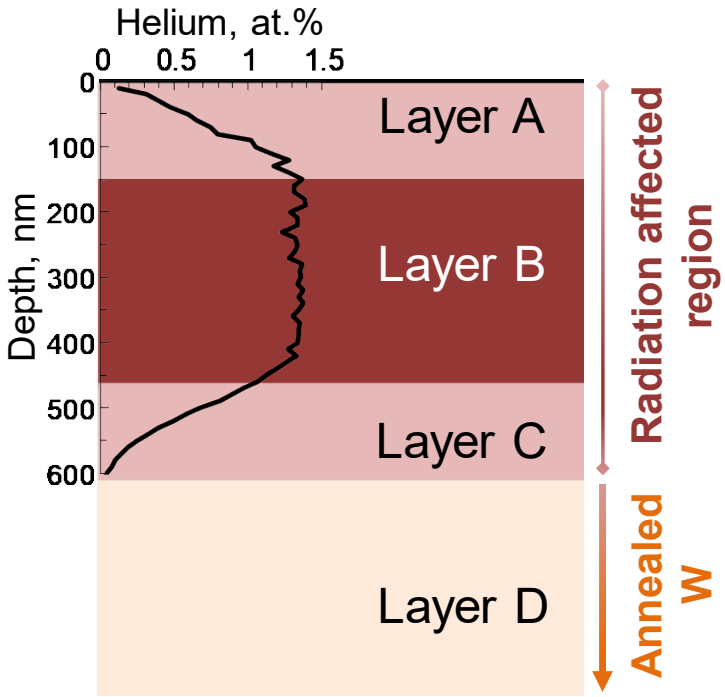


Radiation depth vs. Indentation zone

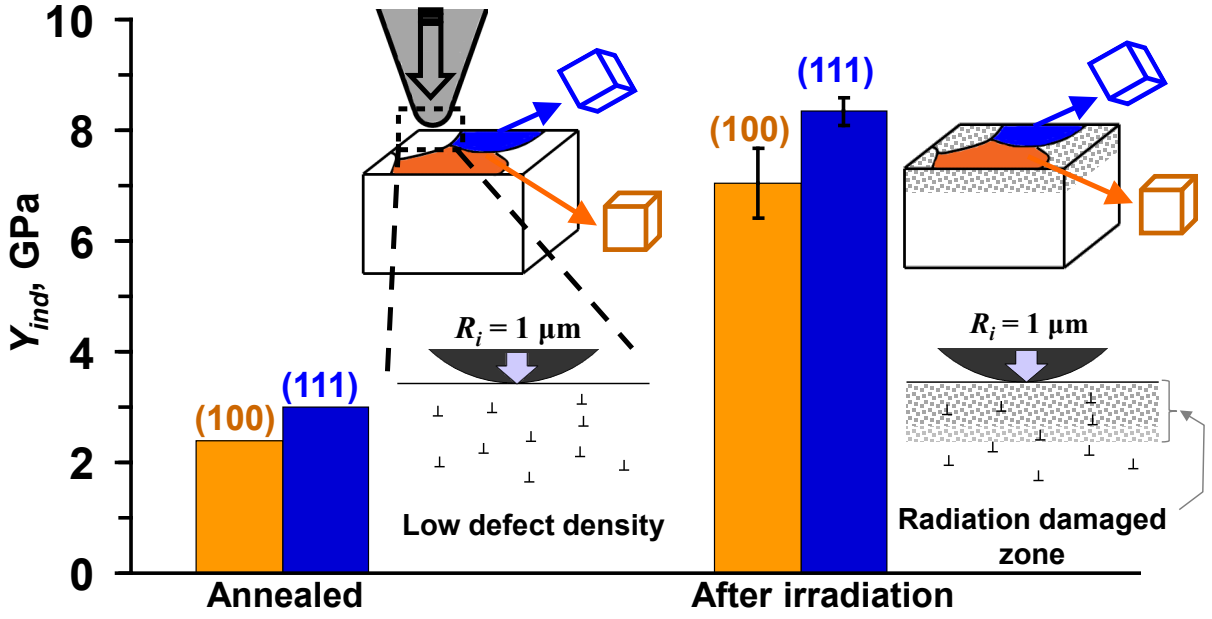


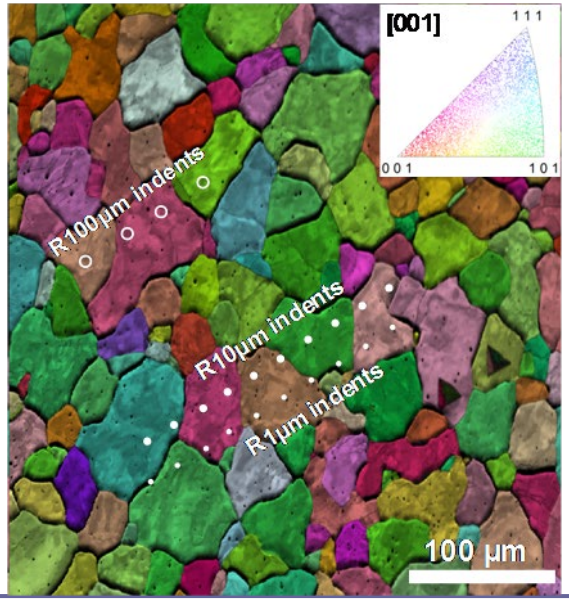


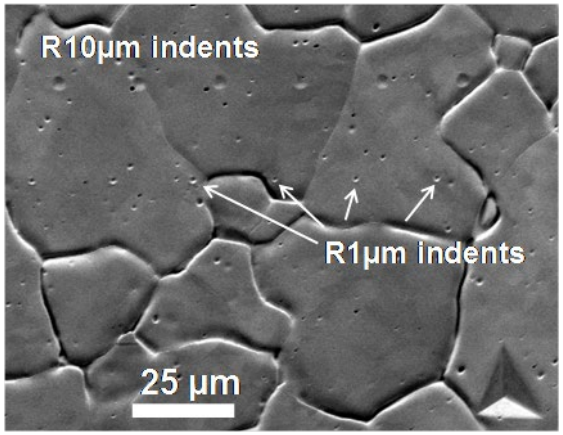




Effect of grain orientation – He on W



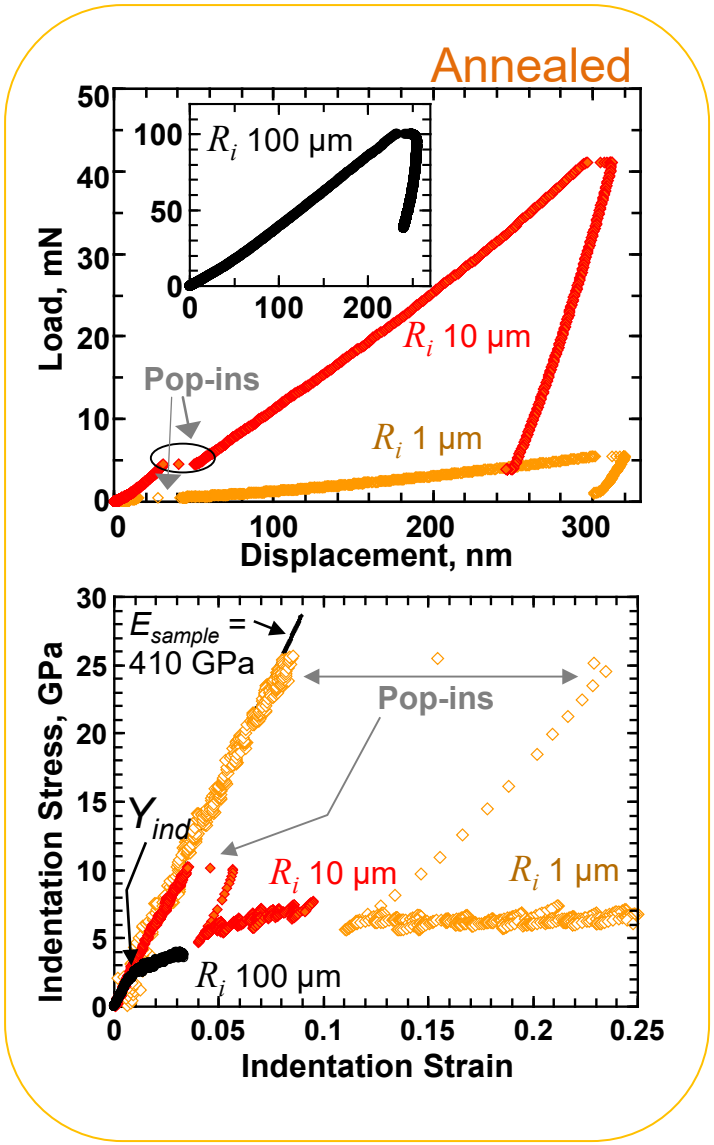


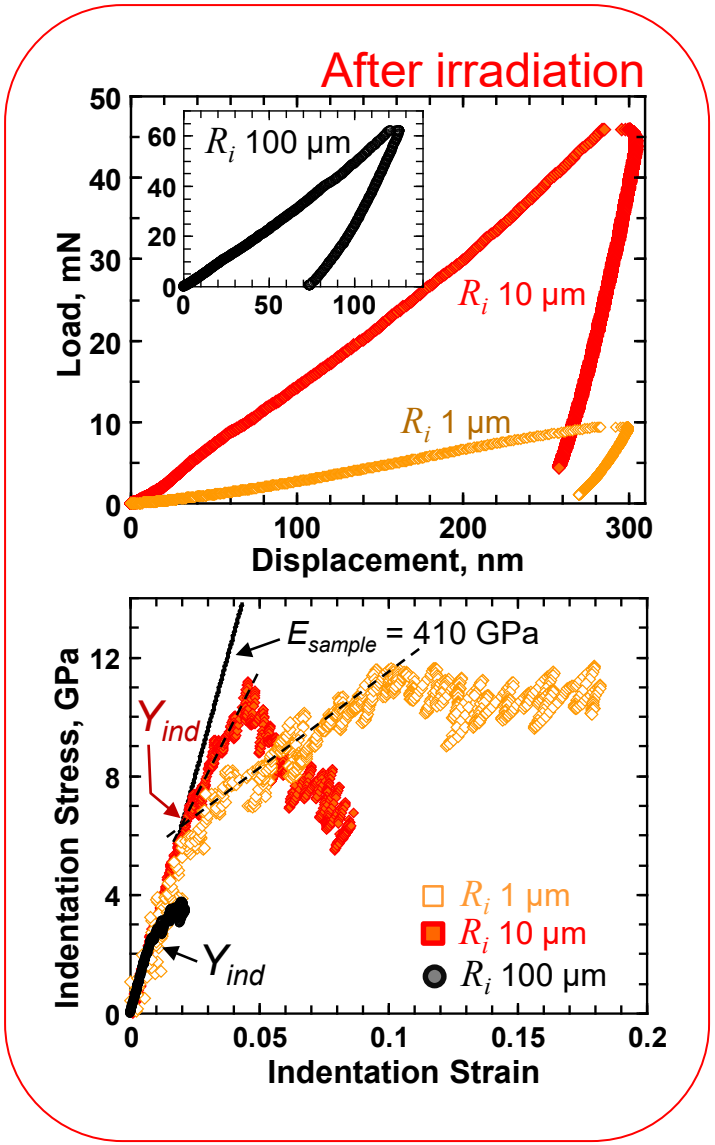


Effect of indenter size, pop-ins disappear after irradiation

Load - Displacement [100] oriented

Indentation Stress-Strain [100] oriented

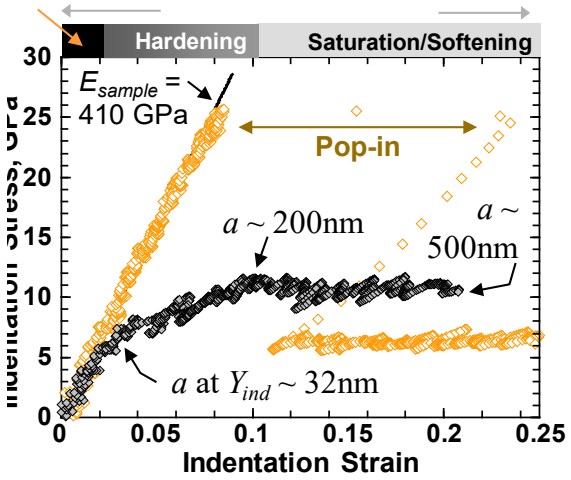




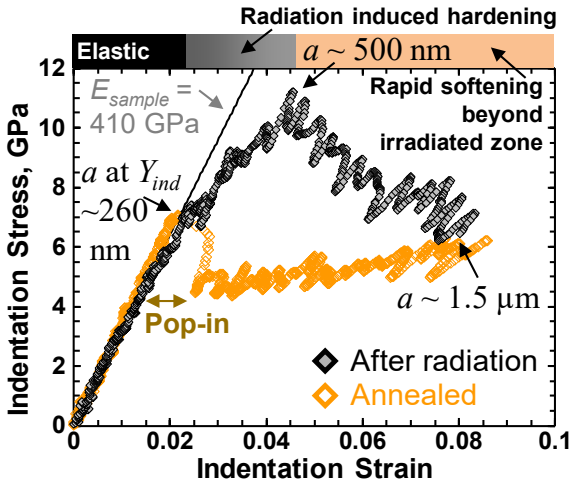
S Pathak et al. MSE:R. (Review paper) 2015

Annealed vs. He irradiated W with varying indenter sizes

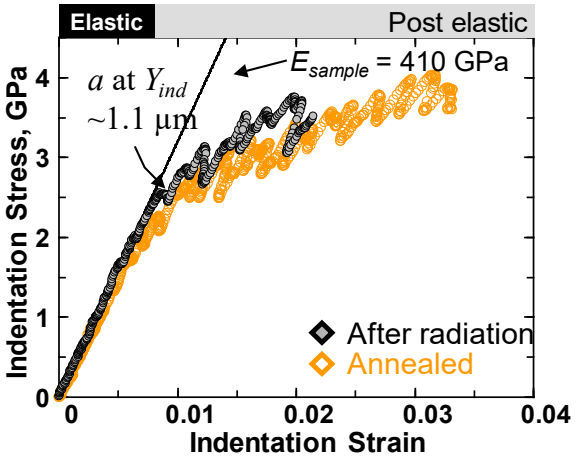
R_i = 1 μm

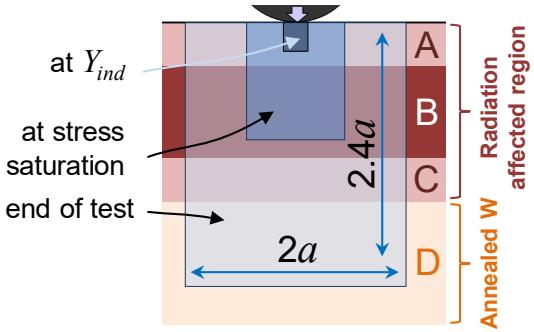


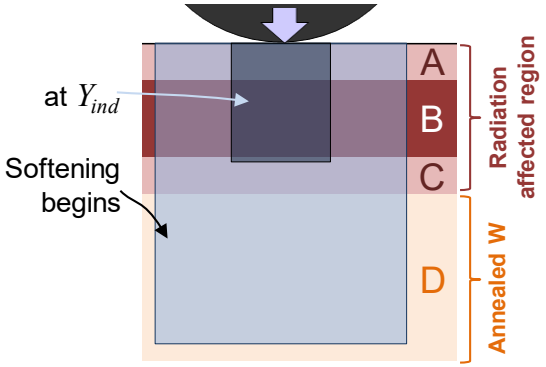
R_i = 10 μm

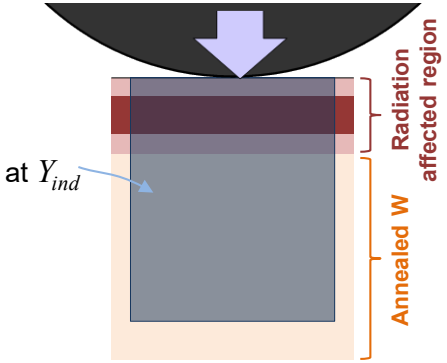


R_i = 100 μm









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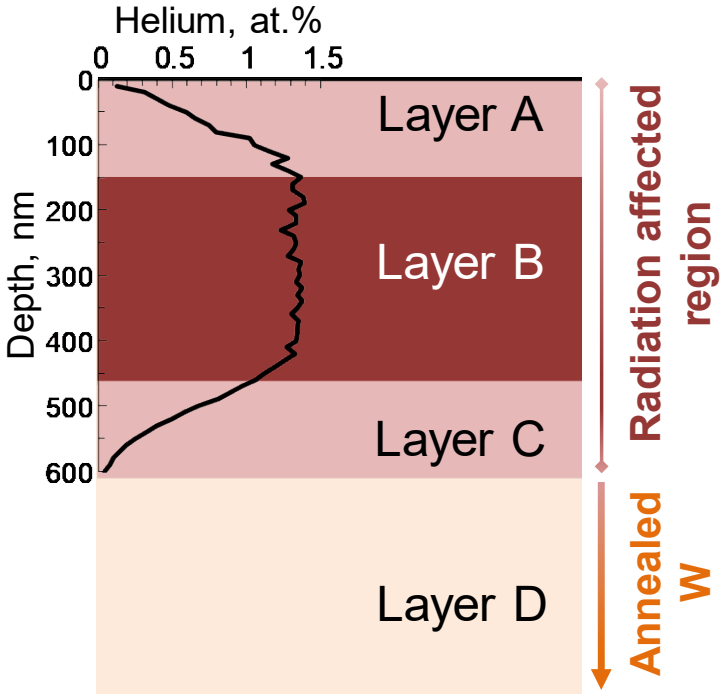
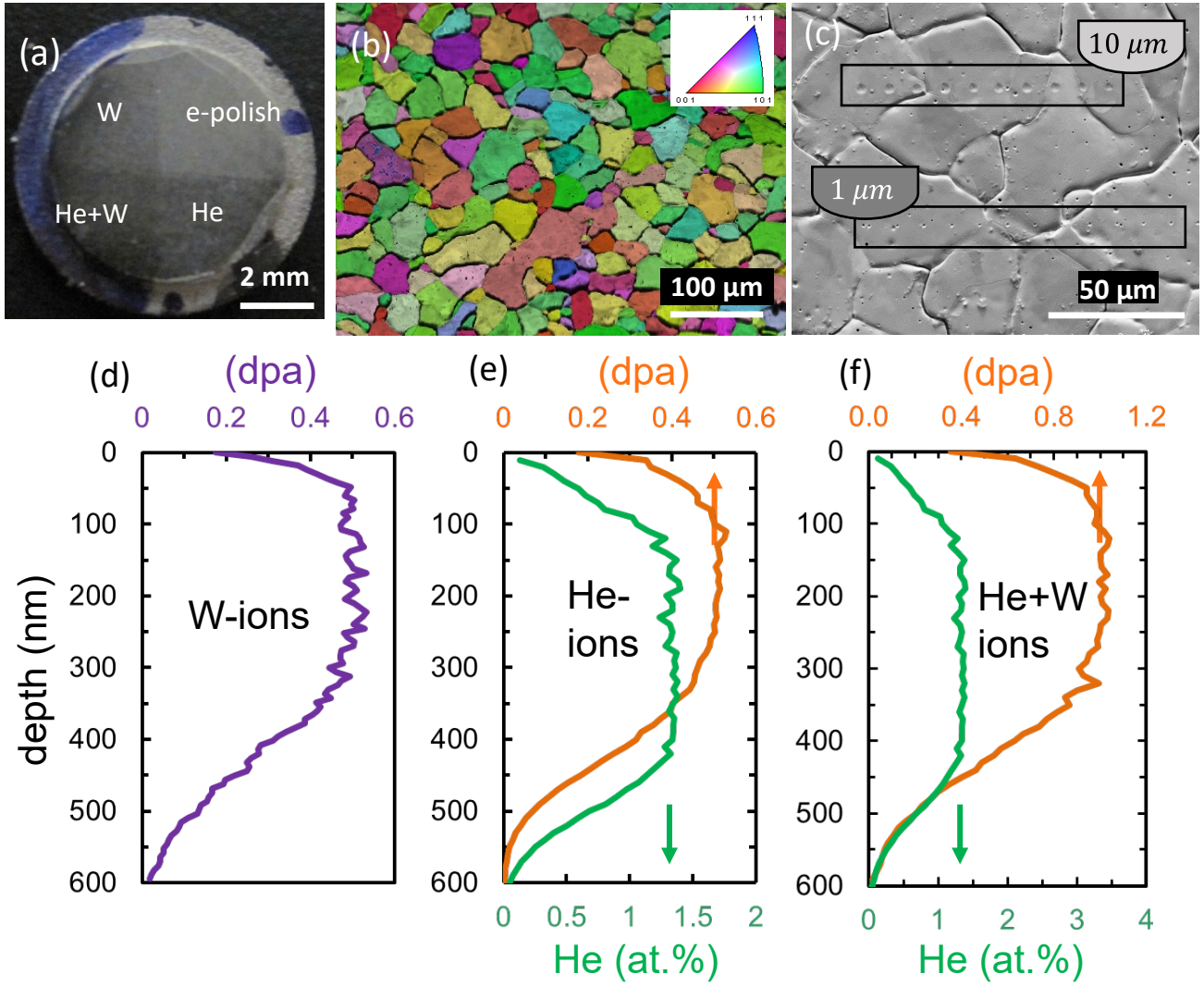
Orientation effect in He irradiated W



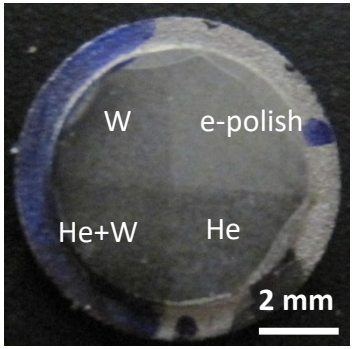
Grain orientation	Layer A Yield stress, GPa	Layer B Yield stress, GPa	Layer D Yield stress, GPa
	Measured as Y_{ind} from R_i=1 μm indenter	Measured as the saturation stress values from R_i=1, μm indenter	Measured as Y_{ind} on annealed W using R_i=100 μm indenter
Near (100)	3.7±0.8	10.1±0.9	2.4±0.4
Near (111)	4.6±0.4	11.3±0.4	3.1±0.3

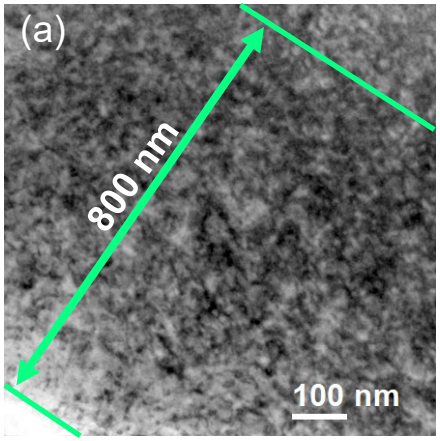
S Pathak et al. 2017. Scientific Reports

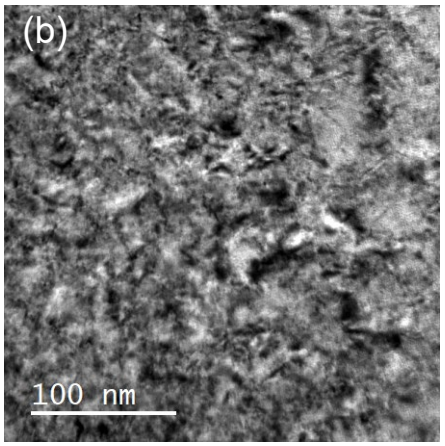
Comparing He, W and He + W ion irradiation on tungsten

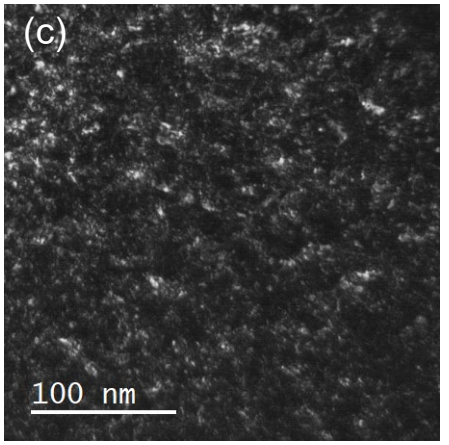


Microstructure of He+W ion irradiation on tungsten

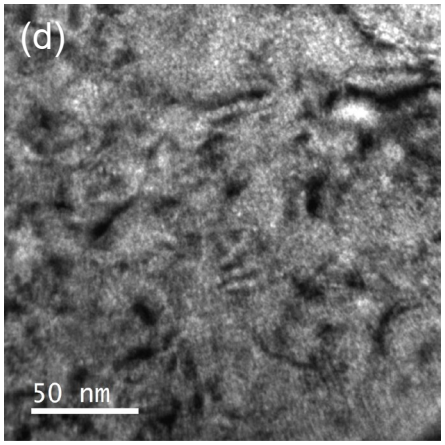


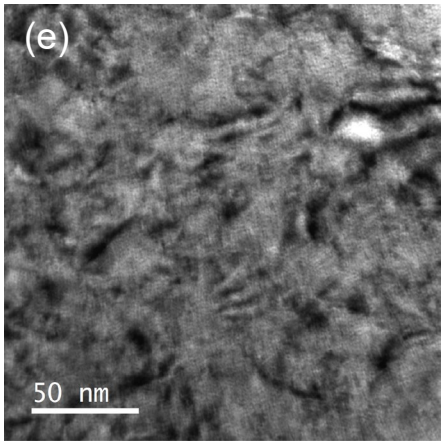


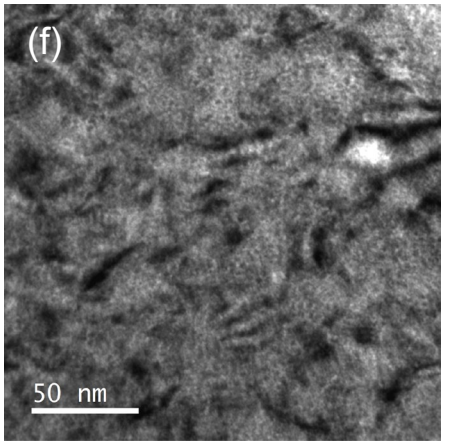




Representative TEM micrographs of the dislocation loops and He bubbles in the irradiated layer for the He+W region







(d-f) under focused, in-focus, and over focused images showing He bubbles.
He bubbles end at a depth of ~ 500 nm

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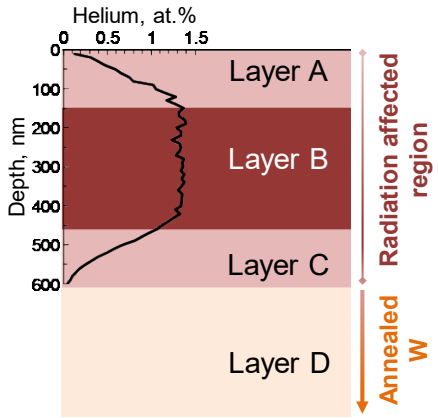
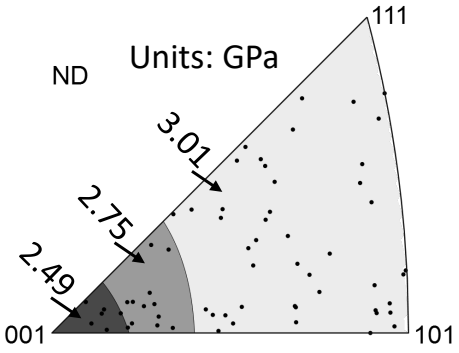
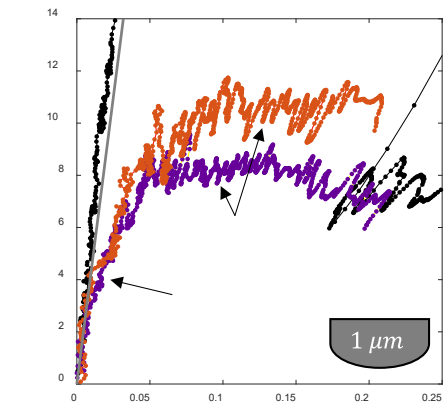
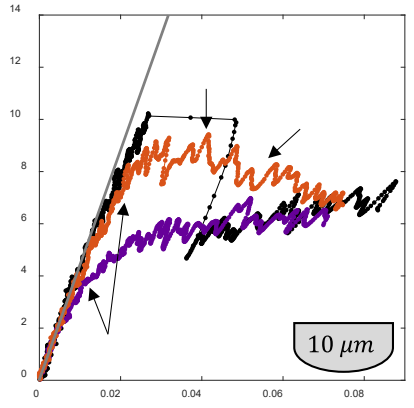
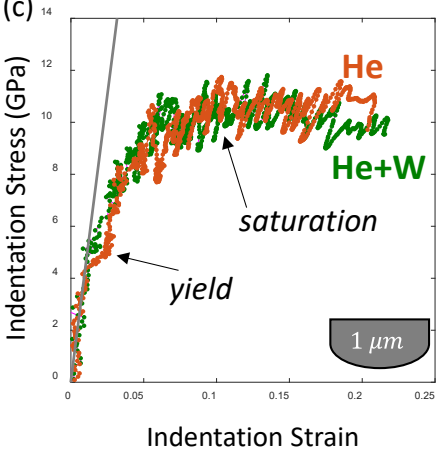
Spherical indentation stress-strain analysis of He, W and He+W ion irradiation on tungsten

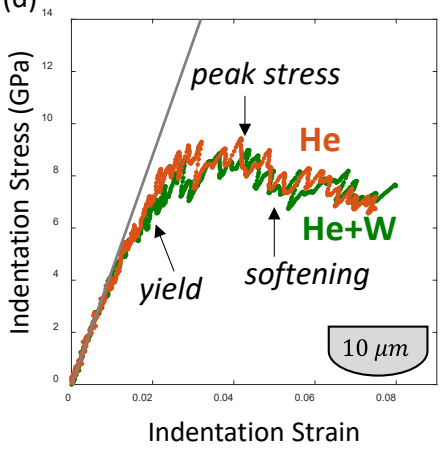












- He implanted tungsten exhibits higher plastic flow strengths compared to W irradiated tungsten,
- there is no significant difference between the indentation response of the He and He+W regions despite twice the dpa damage for the He+W compared to the He region,
- the 1 μm indenter response is very similar for all irradiated conditions up to ~5% indentation strain,
- 1 and 10 μm indenters show different responses for the same irradiated material

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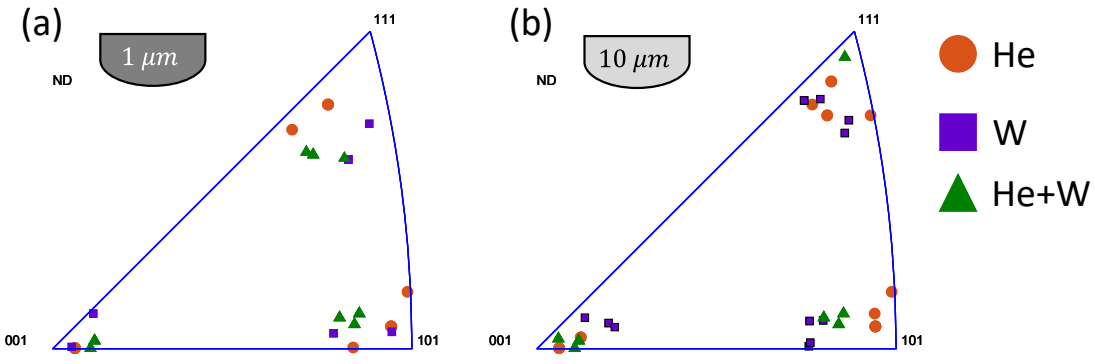
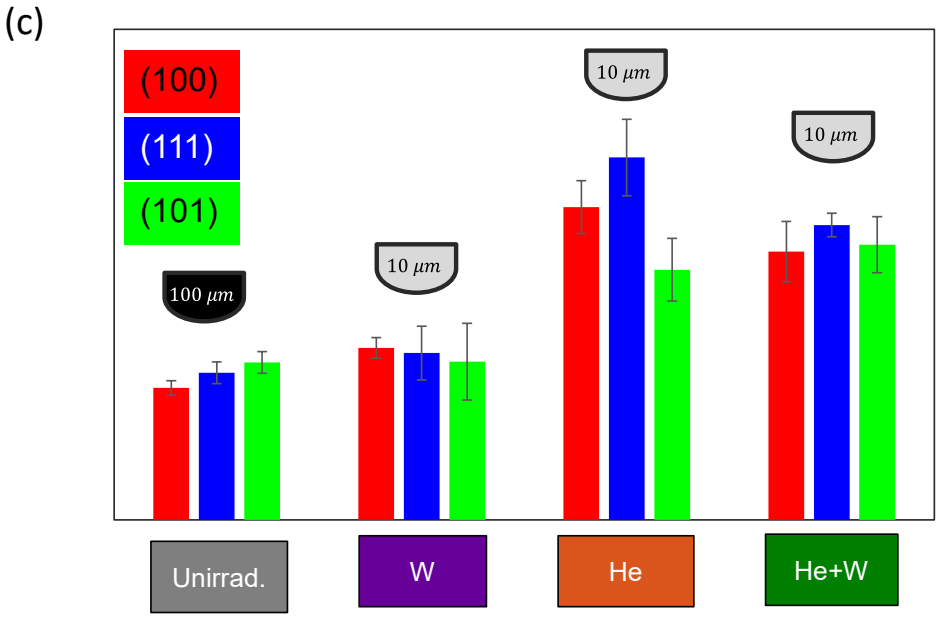
Effect of crystal orientation on the mechanical response of the irradiated material





The orientation trend for the indentation yield strength ($Y_{ind}^{(100)} < Y_{ind}^{(111)} \cong Y_{ind}^{(101)}$) present in the unirradiated material is no longer apparent in the irradiated material

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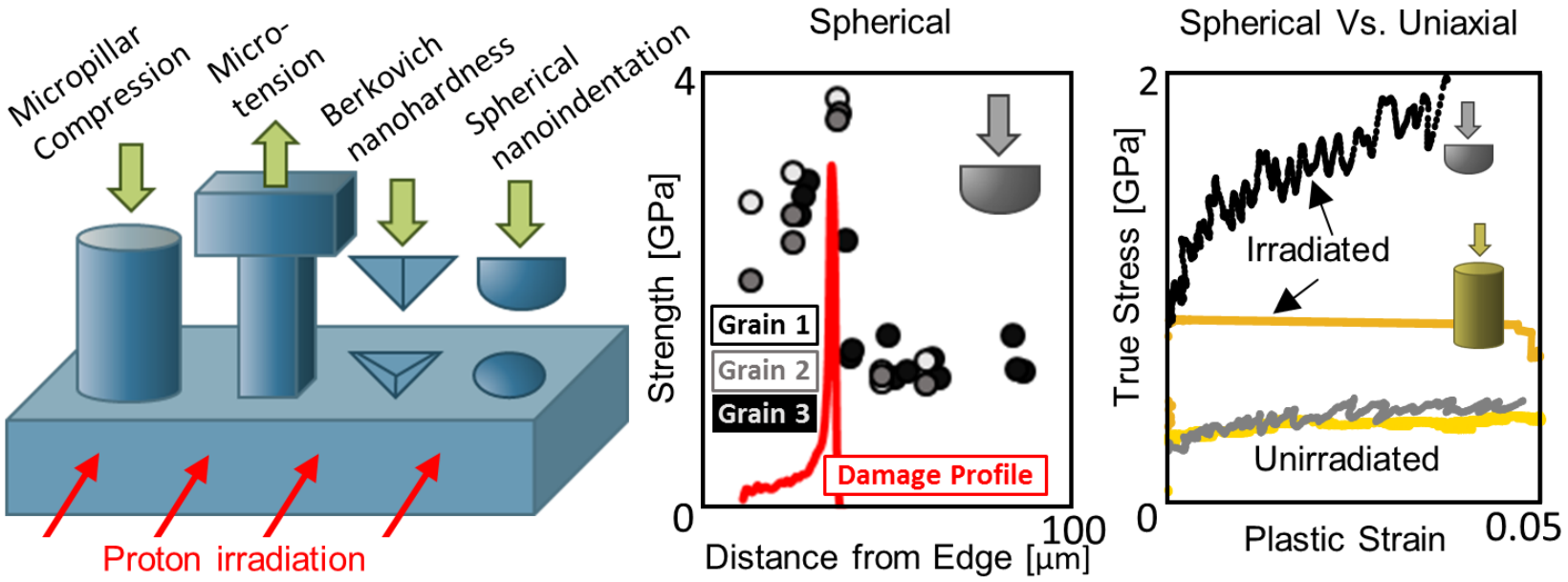
Outline

- Extraction of Spherical Nanoindentation Stress Strain curves
- Indentation on anisotropic samples
- Probing nanoscale damage gradients with spherical nanoindentation
- Comparing small scale mechanical test techniques for measuring irradiation hardening

Proton irradiated 304 stainless steel: A comparison of small scale mechanical test techniques for measuring irradiation hardening



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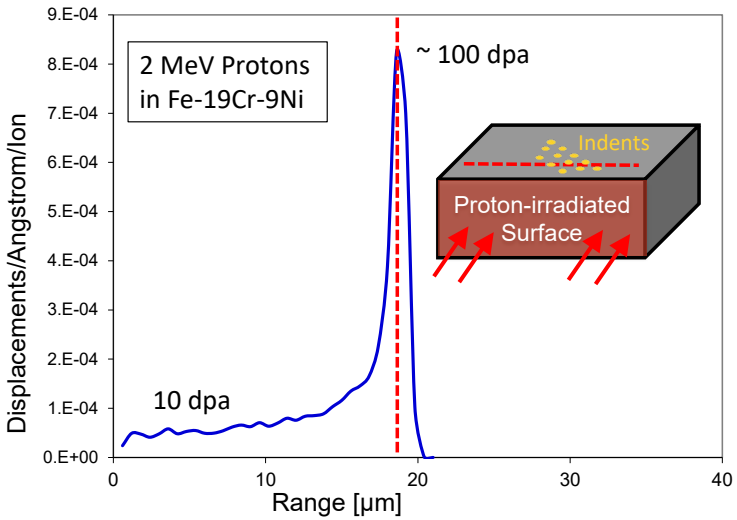
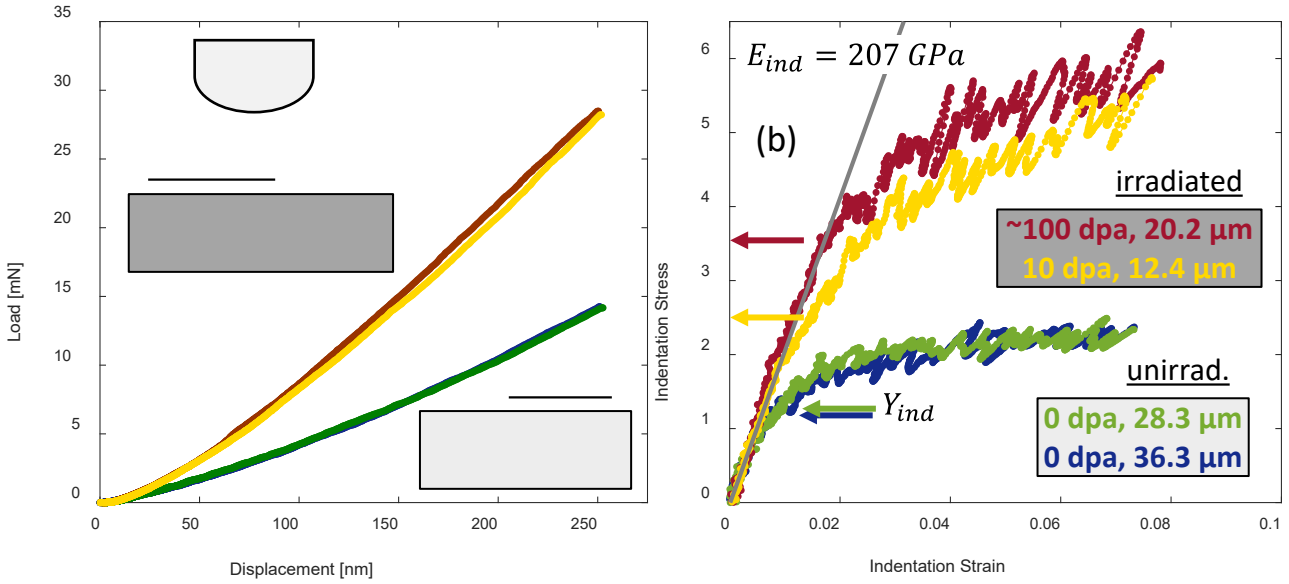
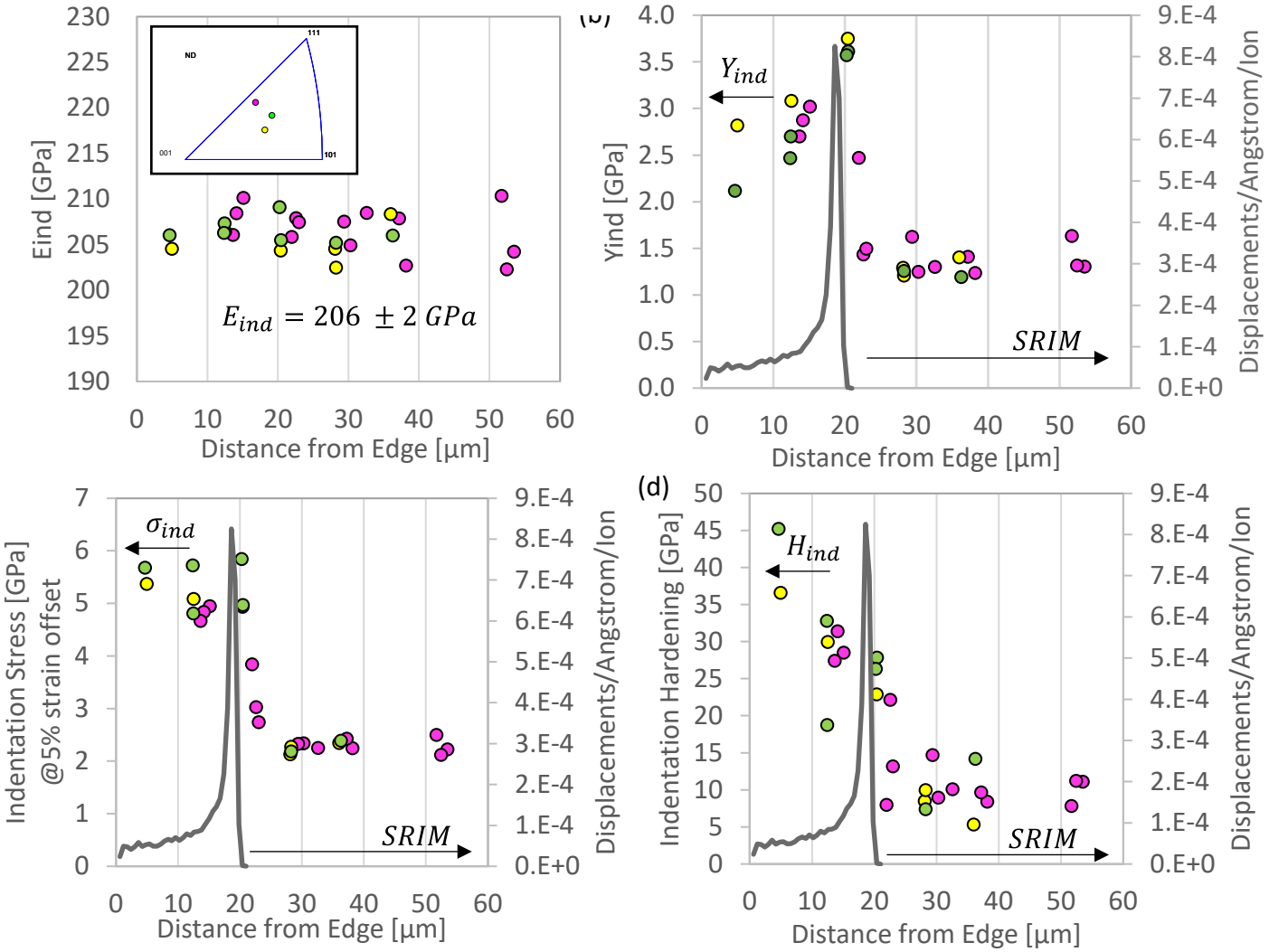
Proton irradiation of 304 stainless steel

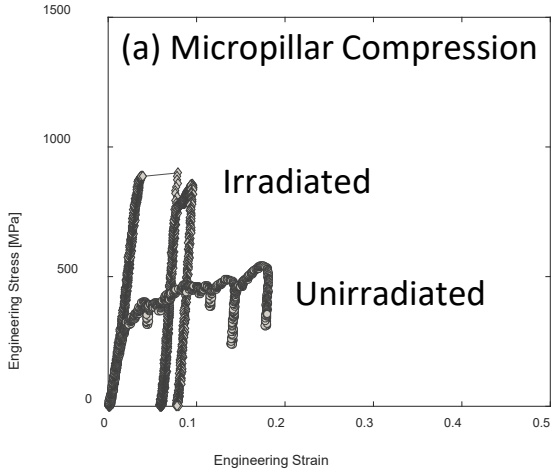


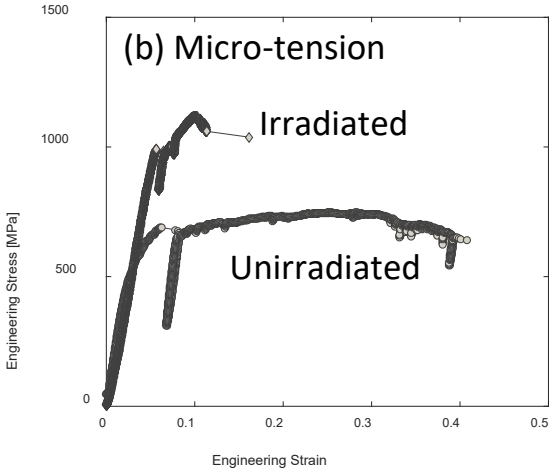


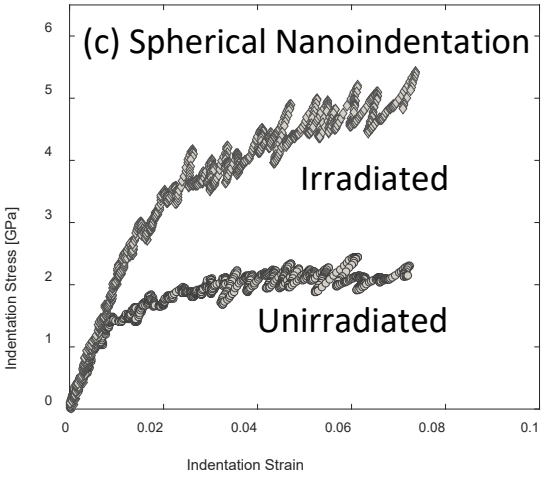
Trends in indentation properties as a function in distance from the edge of the sample



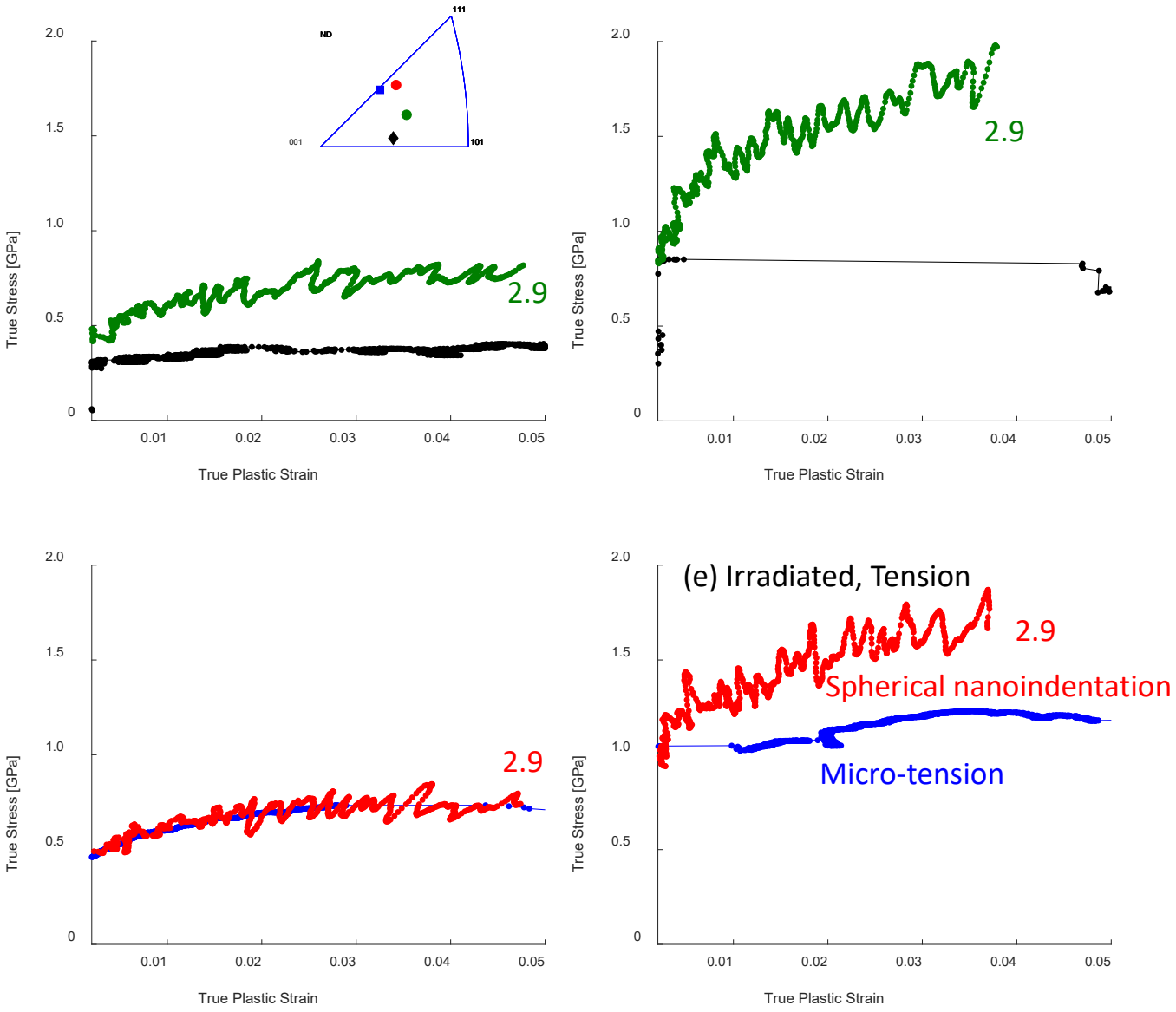
Comparing micro-pillar, micro-tension and spherical indentation





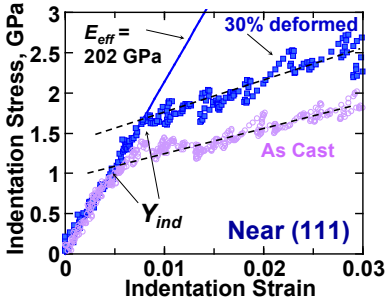


Comparing micro-pillar, micro-tension and spherical indentation

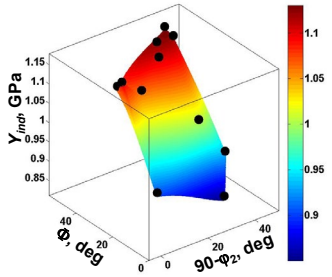


Summary: indentation stress-strain curves

- Indentation stress-strain curves allow us to explore the elastic, yield and post-elastic behavior in a variety of material systems.



- In anisotropic polycrystalline metal samples (Fe-3%Si steel)
  - to characterize the anisotropy (β) in the elastic indentation modulus, and
  - the increase in the indentation yield strength in deformed microstructures



- This technique is now being explored to
  - probe near-interface regions
  - probe the changes in indentation yield strength in ion-irradiated samples