

# Irradiation Creep in Nanostructures

SAND2018-2384C

## Measured Using In-situ TEM

*Shen J. Dillon, Gowtham Jawaharam, Robert S. Averback, and Khalid Hattar*

Sandia Postdoc Collaborators:

*Christopher Barr*

*Daniel Bufford*

*Patrick Price*

Acknowledgements:

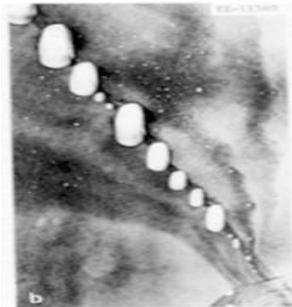
*US DOE DEFG02-05ER46217*

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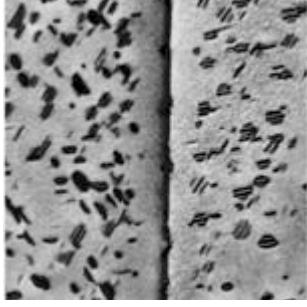
# Example Issues in Nuclear Materials

## Microscopic

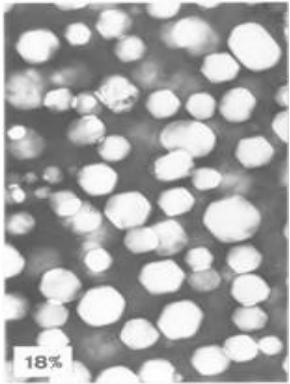
Gas Bubbles



Voids



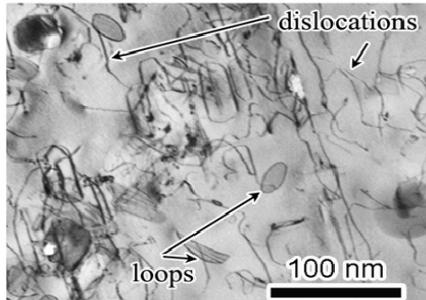
Precipitation



Segregation

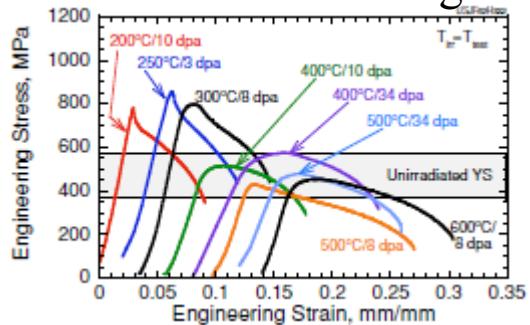


Line Defects

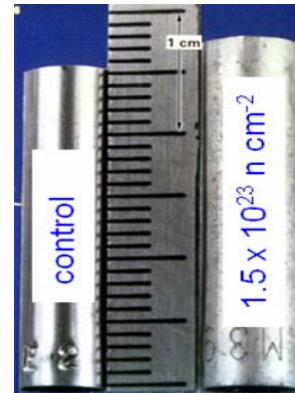


## Macroscopic

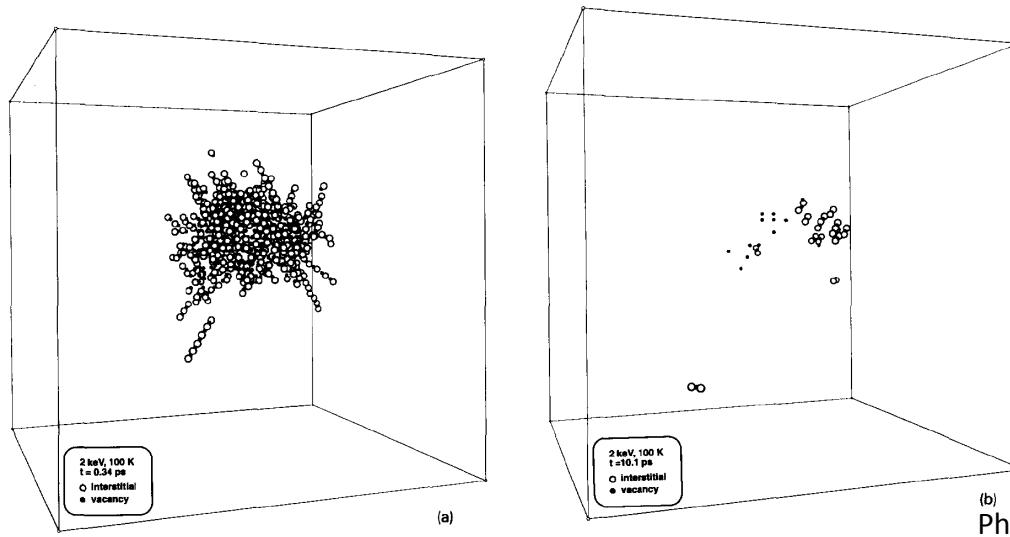
### Radiation hardening



Swelling/Creep

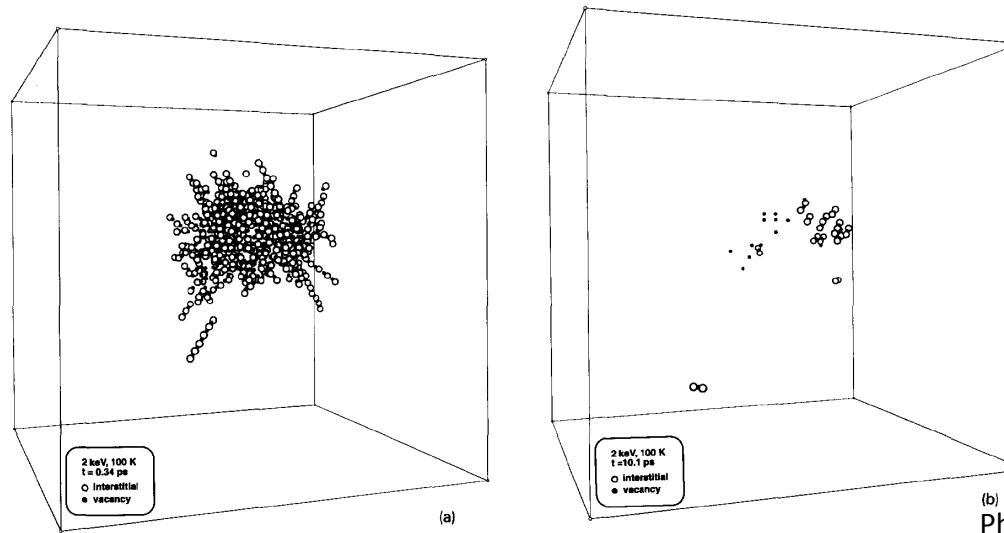


# Point Defect Production and Annihilation



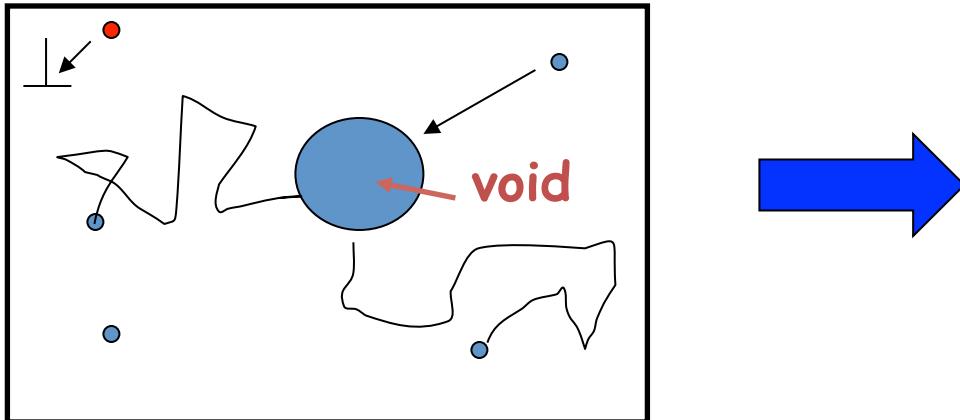
Phythian et al. *J. Nuc. Mater.* (1995)

# Point Defect Production and Annihilation



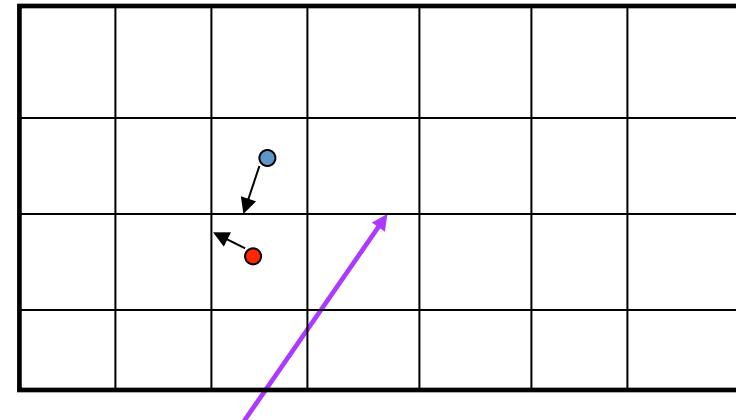
Phythian et al. *J. Nuc. Mater.* (1995)

## Conventional materials



• Vacancies, • interstitials

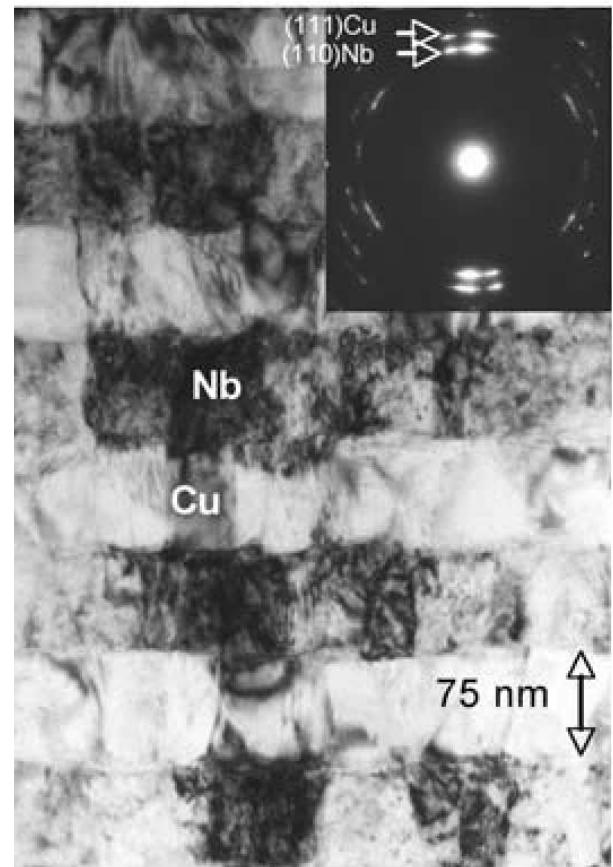
## Nanostructured materials



Interface & Grain boundaries (defect sinks)

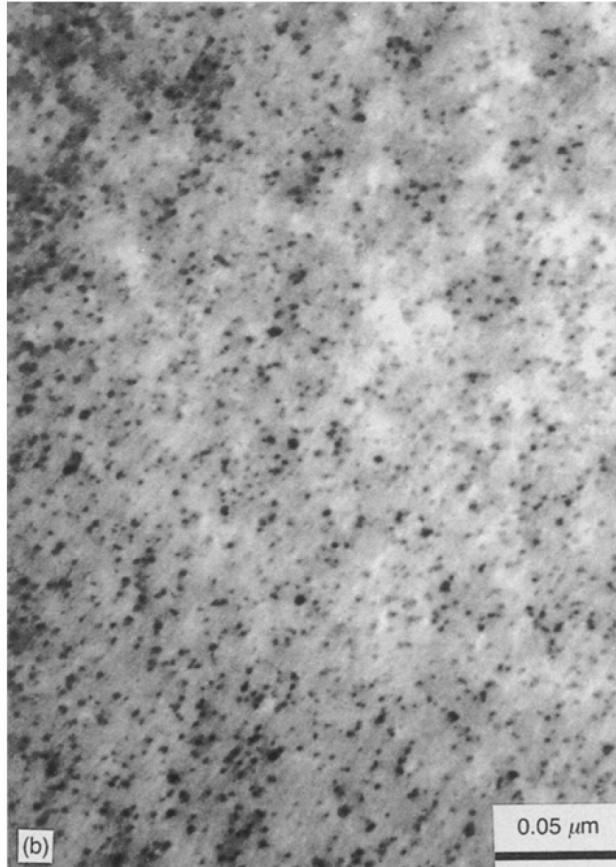
# Nanostructured Alloys

## Nanolaminates



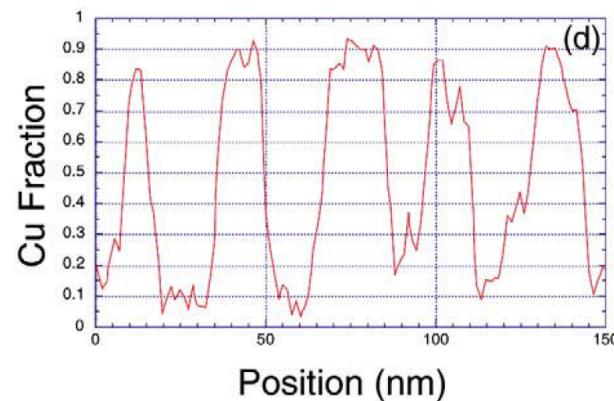
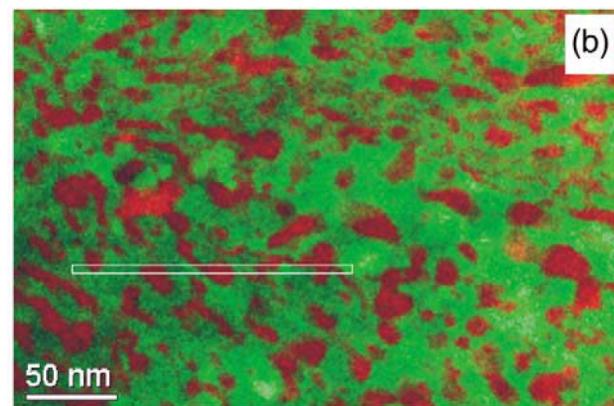
Misra and Hogland, *J. Mater. Sci.* (2007)

## Oxide Dispersion Strengthened



Okuda and Fujiwara, *J. Mater. Sci. Lett.* (1995)

## Self-Organizing Alloys

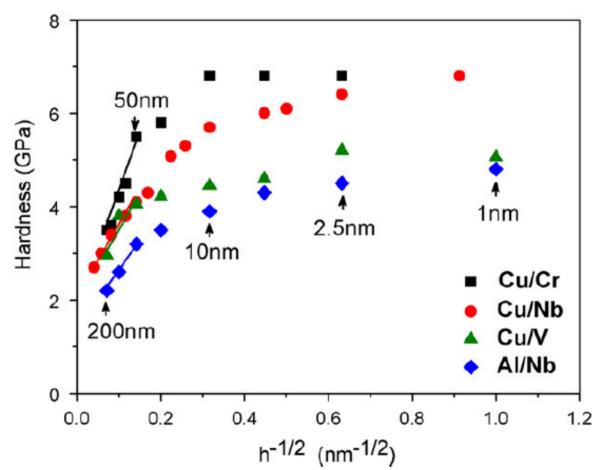
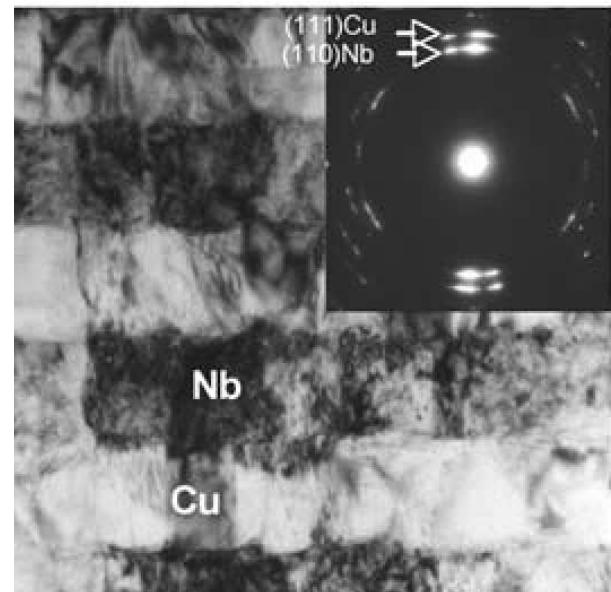


Zghal et al., *Acta Mater.* (2002)

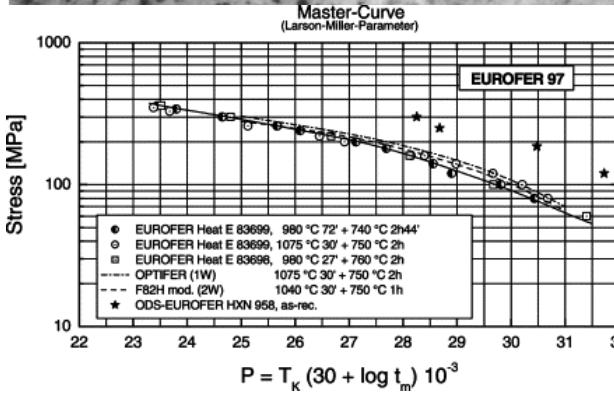
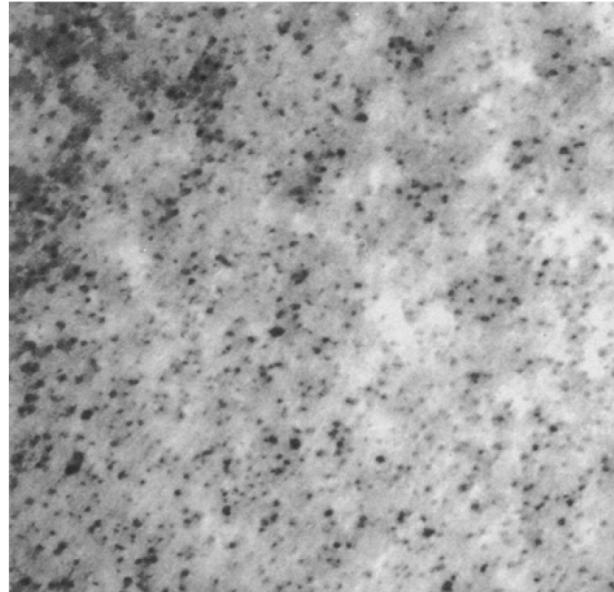
1. Relatively thermally stable
2. High strength
3. High density of interfaces for point defect recombination

# Nanostructured Alloys

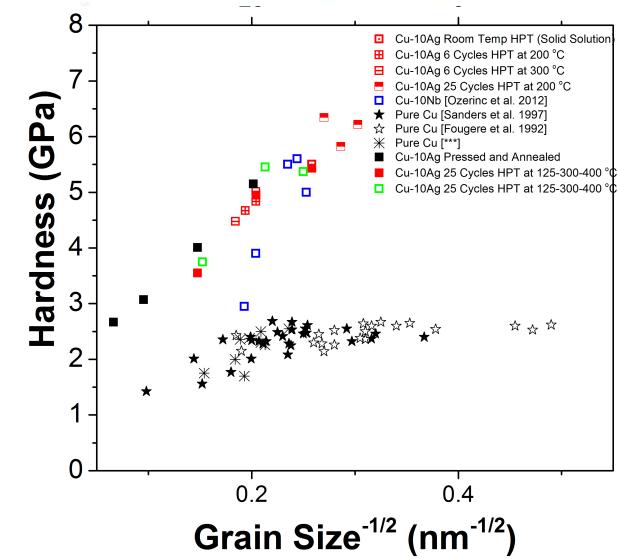
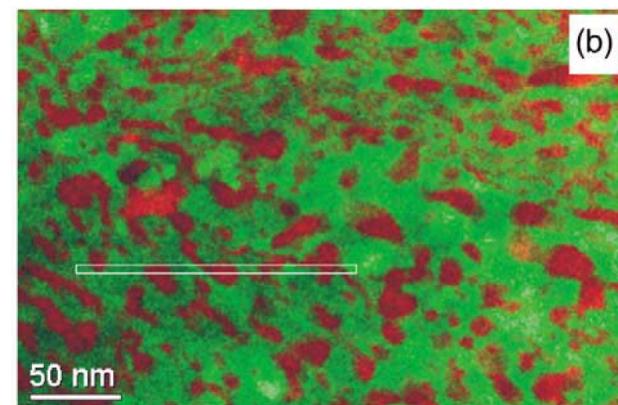
## Nanolaminates



## Oxide Dispersion Strengthened



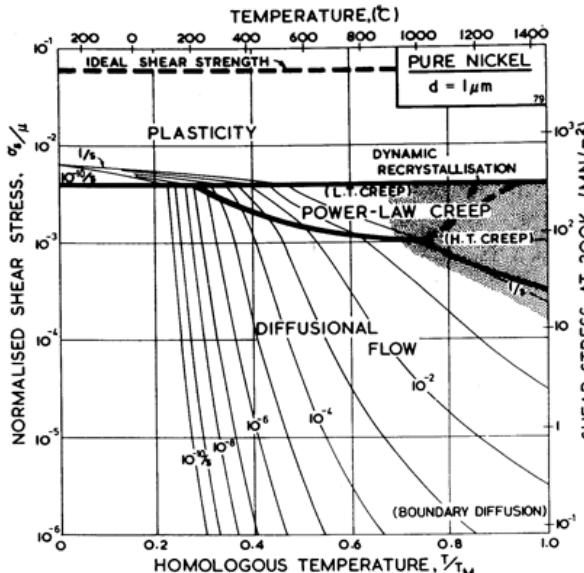
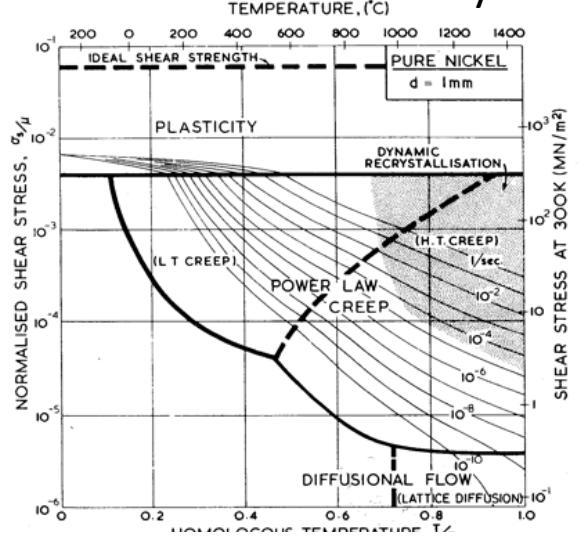
## Self-Organizing Alloys



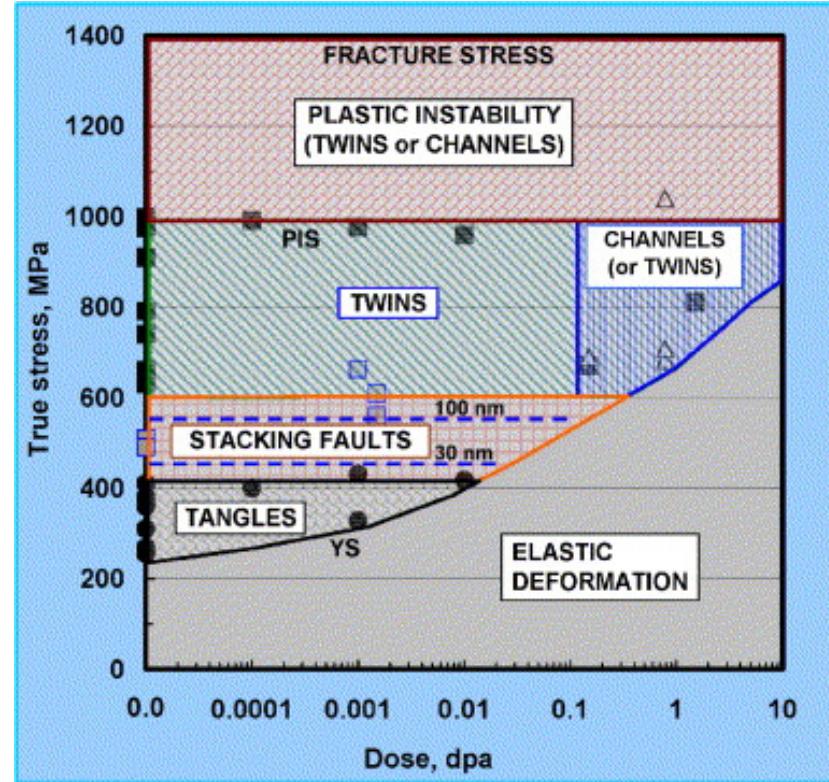
Zghal et al., *Acta Mater.* (2002) [\(link\)](#)  
Arshad, Thesis (2014) [\(link\)](#)

# Size Effects (Nanograin) and Irradiation Induced Creep (IIC)?

Mechanisms effected by size

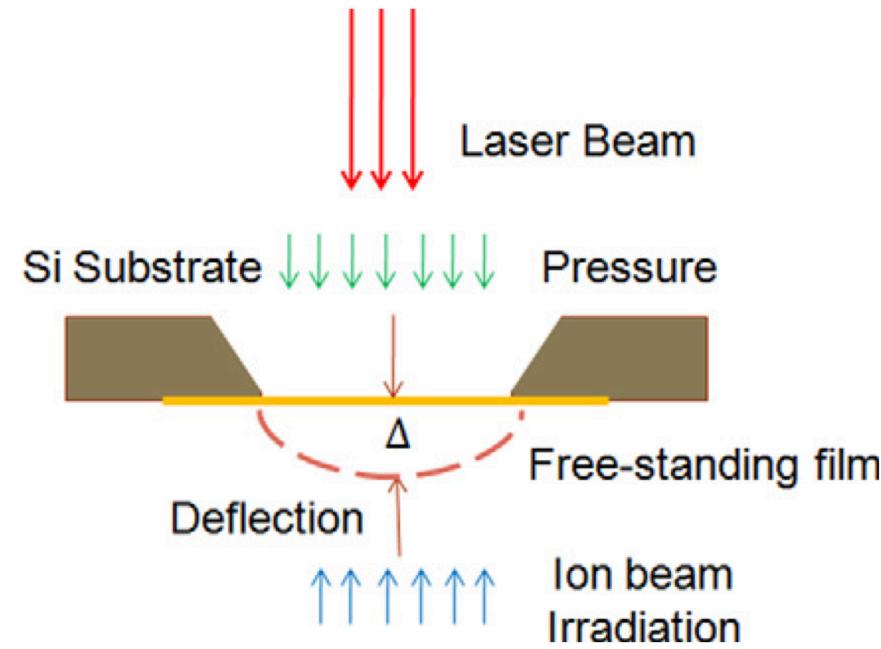
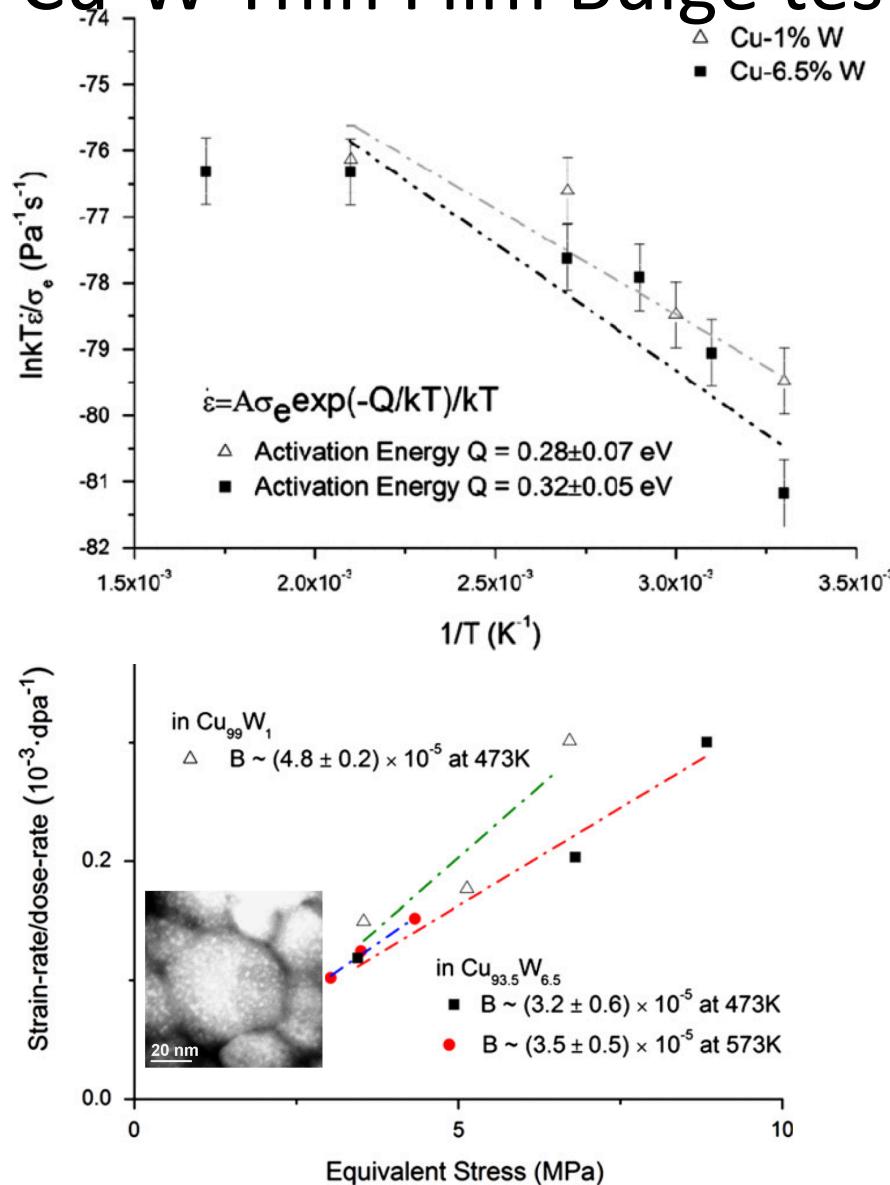


Mechanisms effected by irradiation

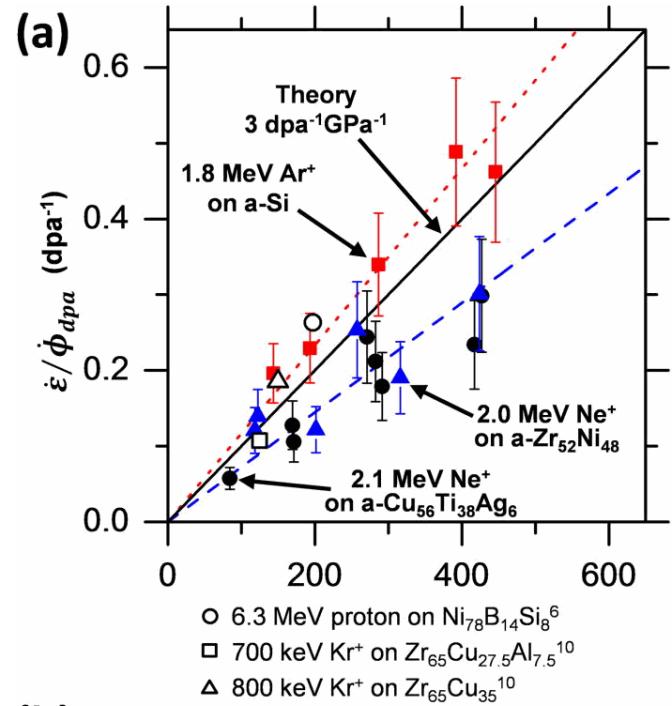
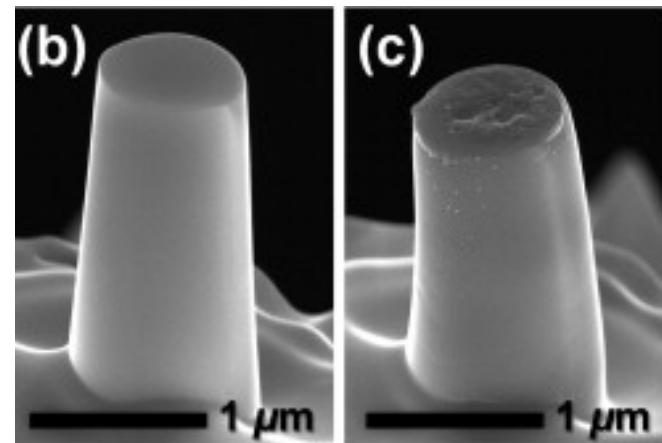
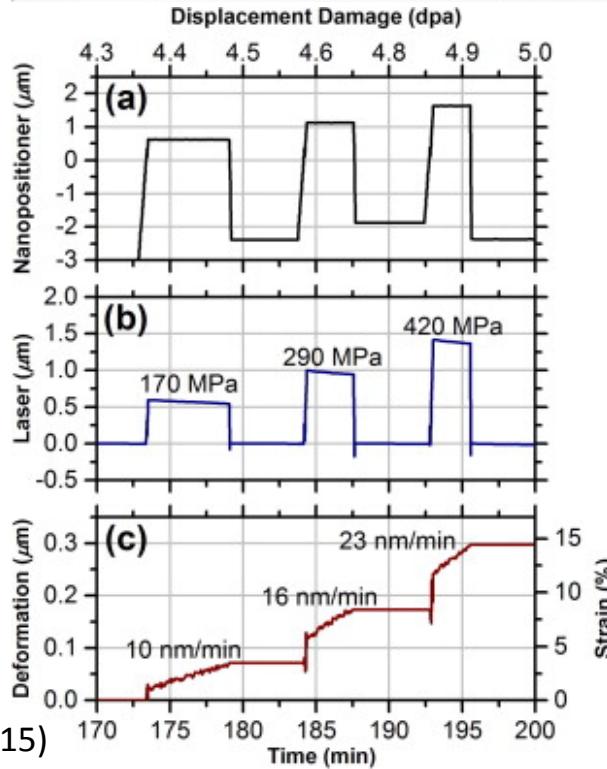
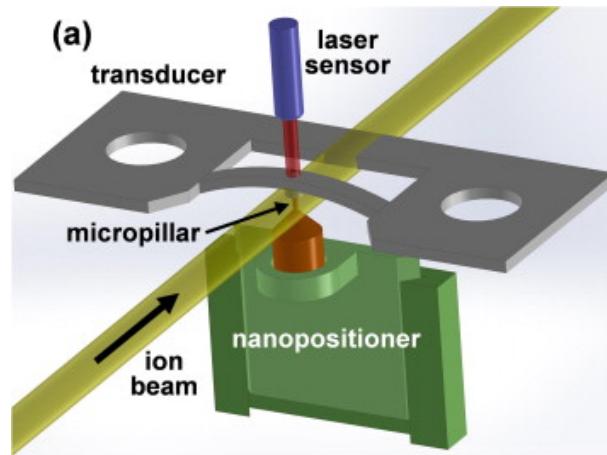


# Nanocrystalline Irradiation Creep Mechanisms

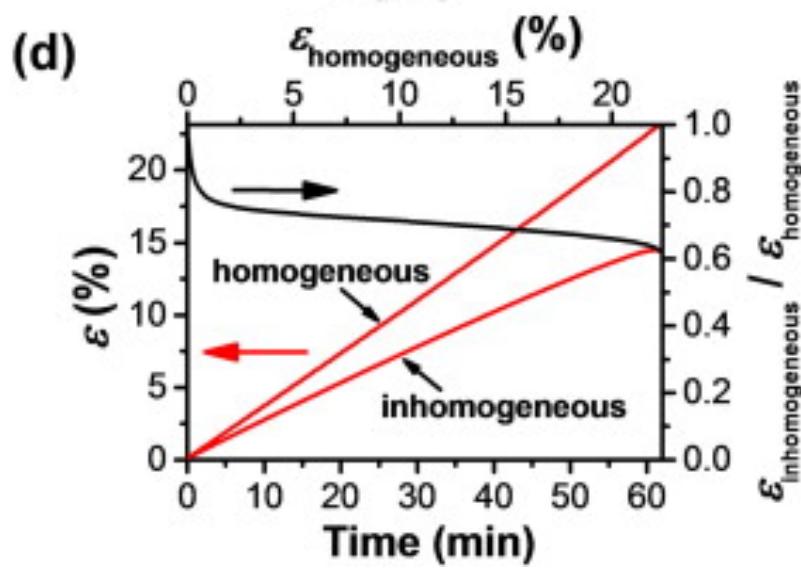
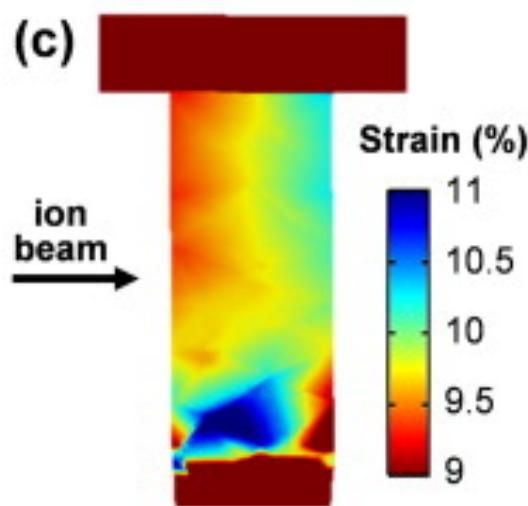
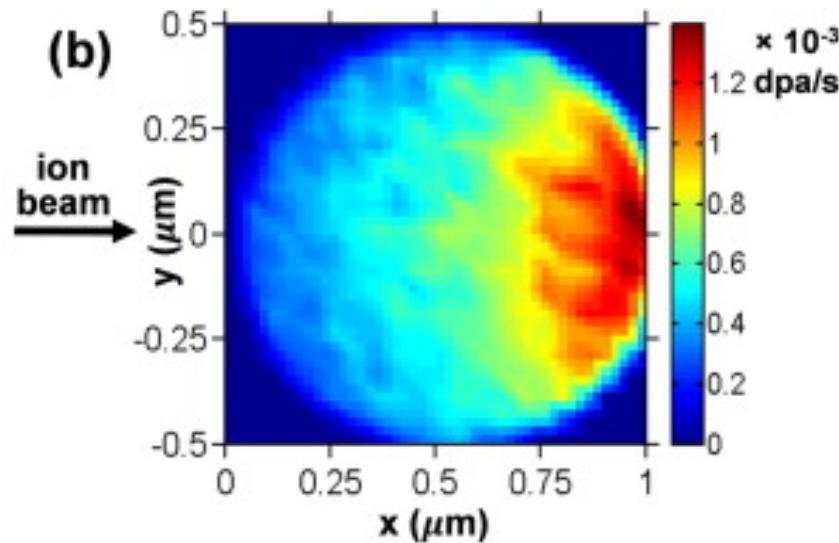
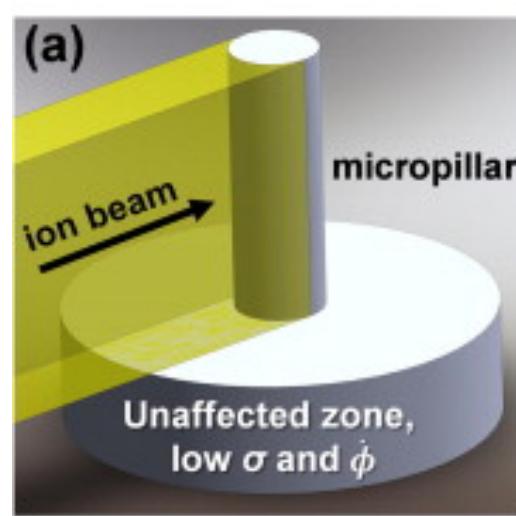
## Cu-W Thin Film Bulge test



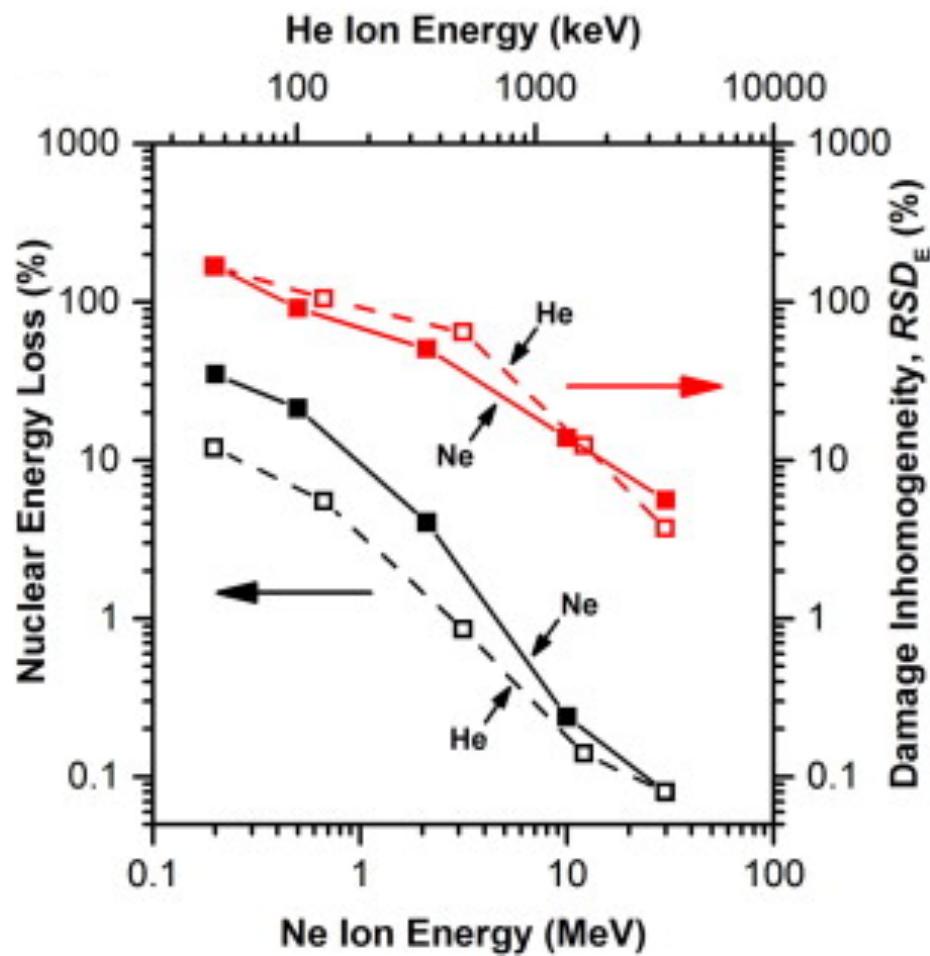
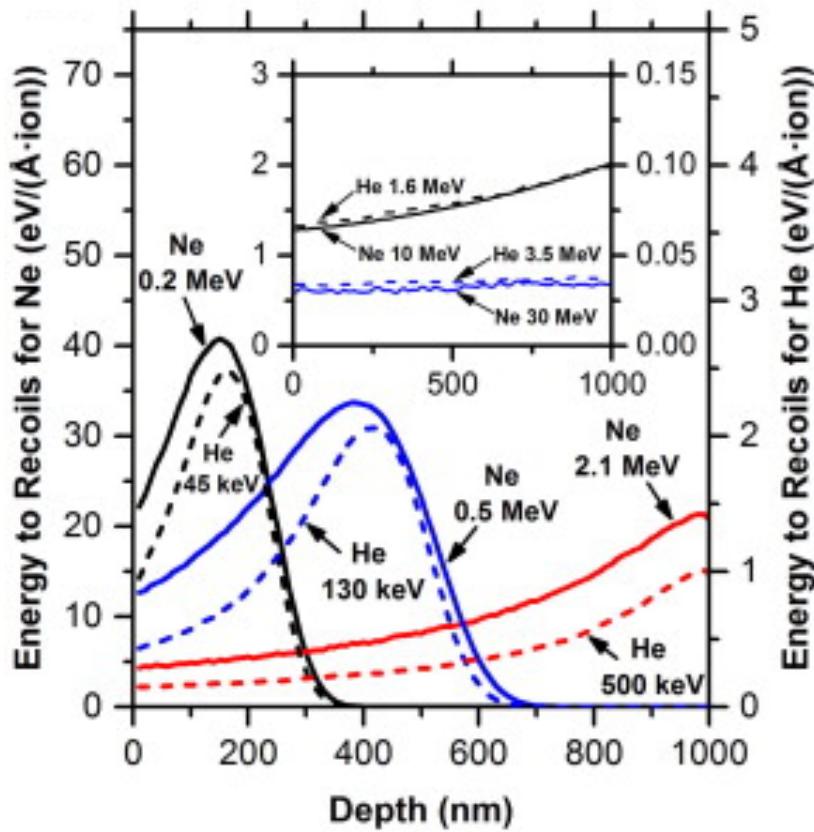
# Nanopillar Experiments



# Experimental Challenges

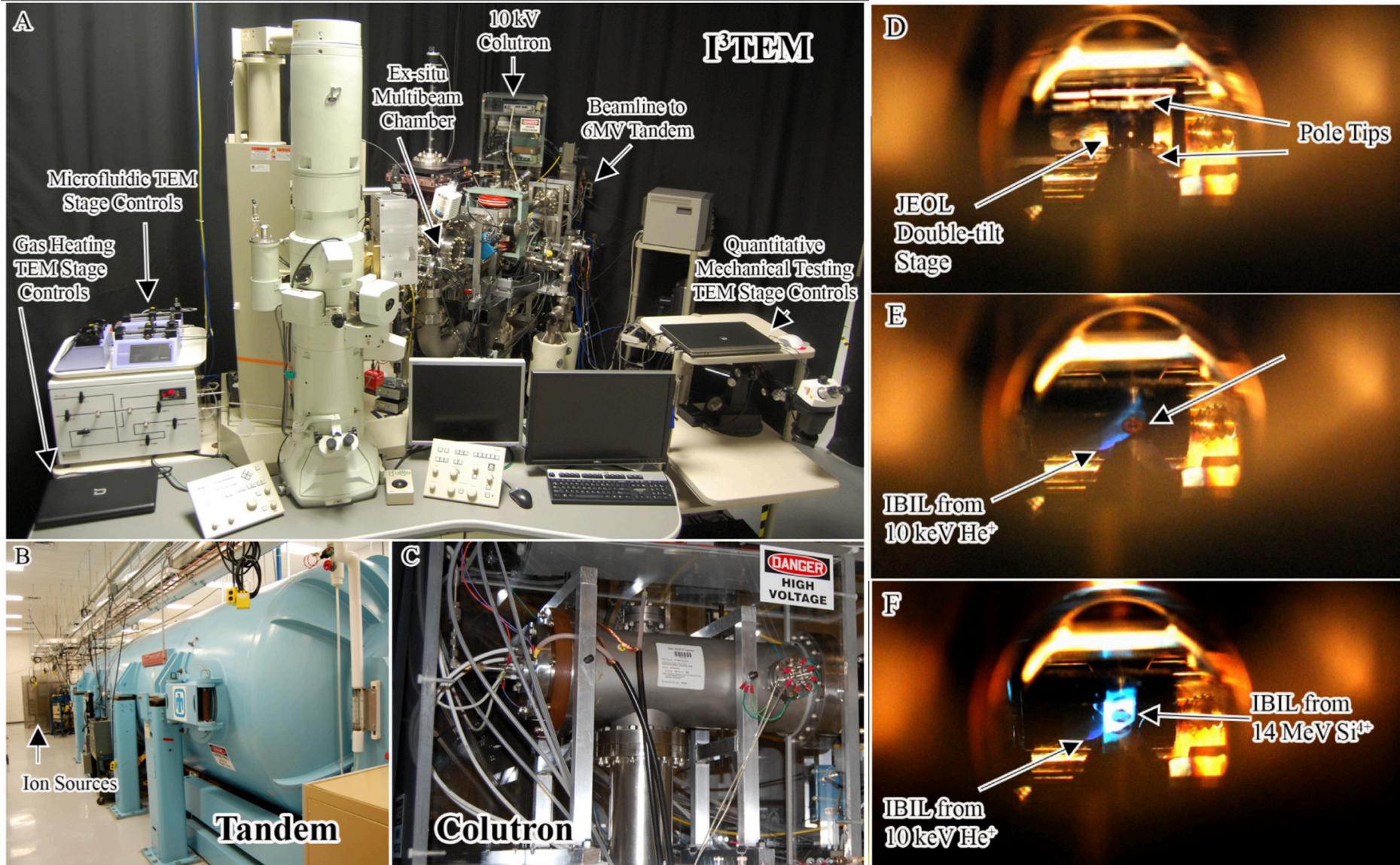


# Experimental Challenges



1. Selection of particle for irradiation to high dose
2. Need for small specimens for homogeneous ion damage
3. Testing of bulk materials (not thin films)

# Measuring Irradiation Creep In-situ Using Sandia National Lab. IBL I<sup>3</sup>TEM



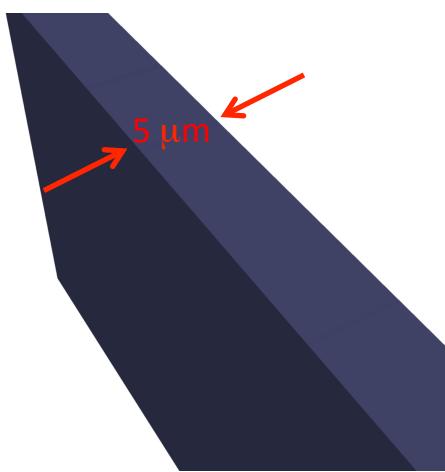
# Goals and Experiments

- Can we measure IIC in the TEM (at high T)?
- Mechanical response of nanograin and nanolaminate materials under irradiation
- Pillar size effect at high T in the sink limited regime (Ag)

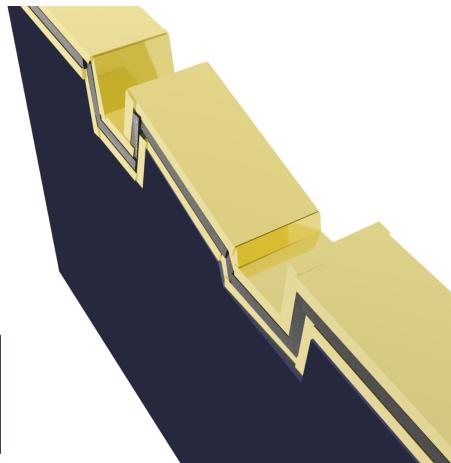
# Sample Preparation and Testing

## Multilayer Samples

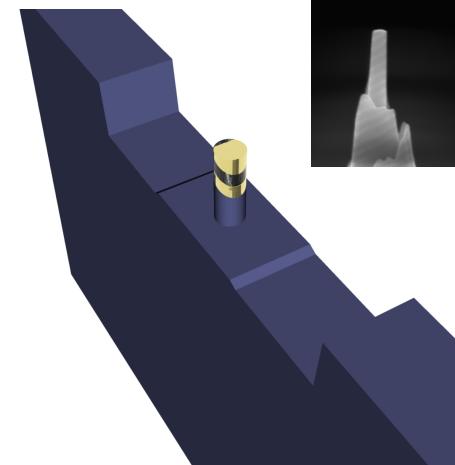
Substrate



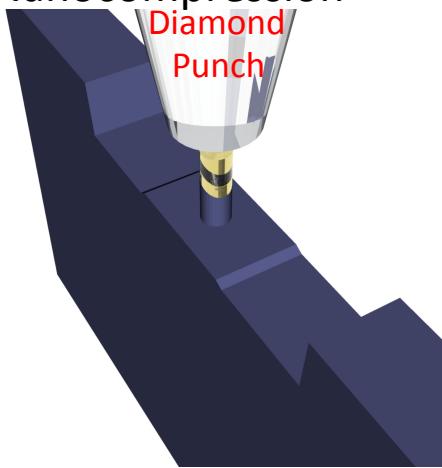
Pre-cut and Grow Films



FIB Sample

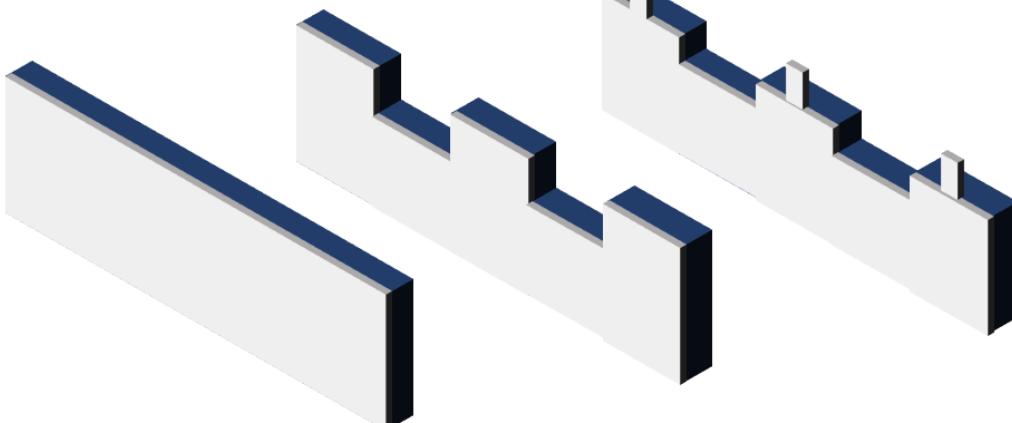


Nanocompression  
Diamond Punch

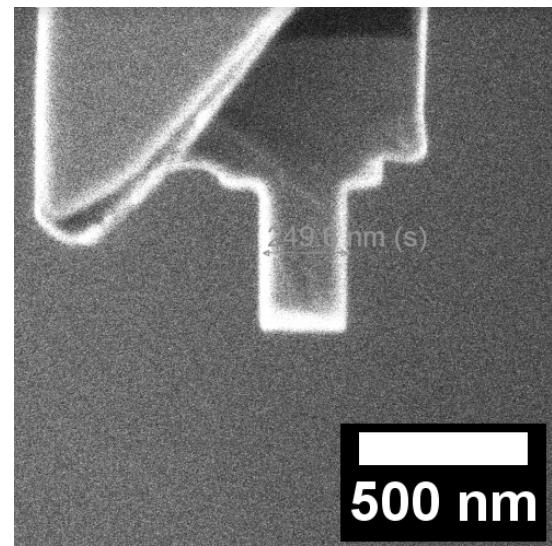


## Ag Samples

Film on surface

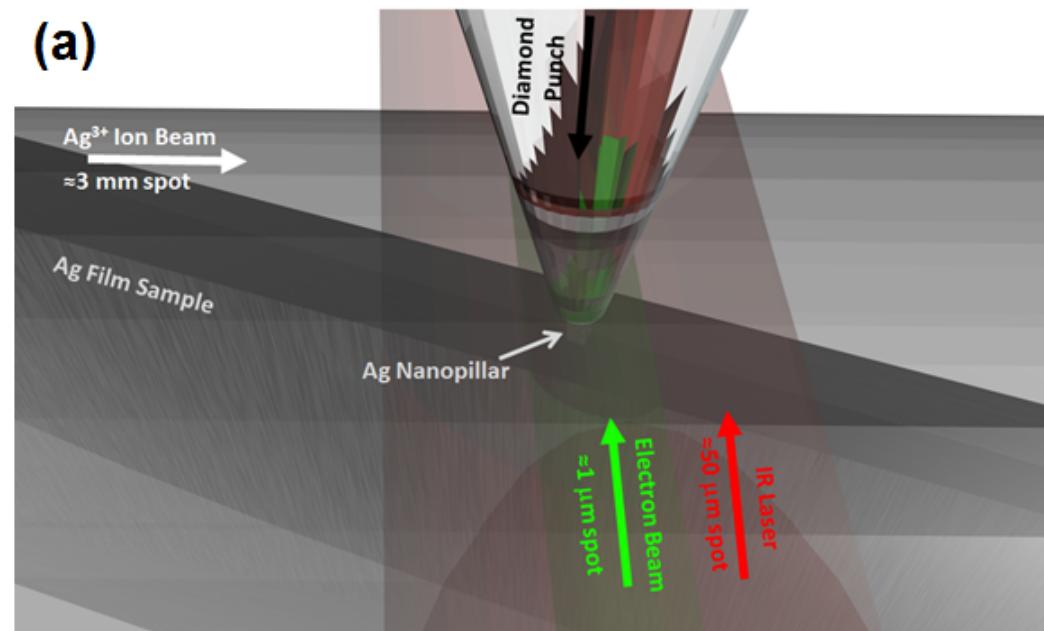


FIB pillars in plane

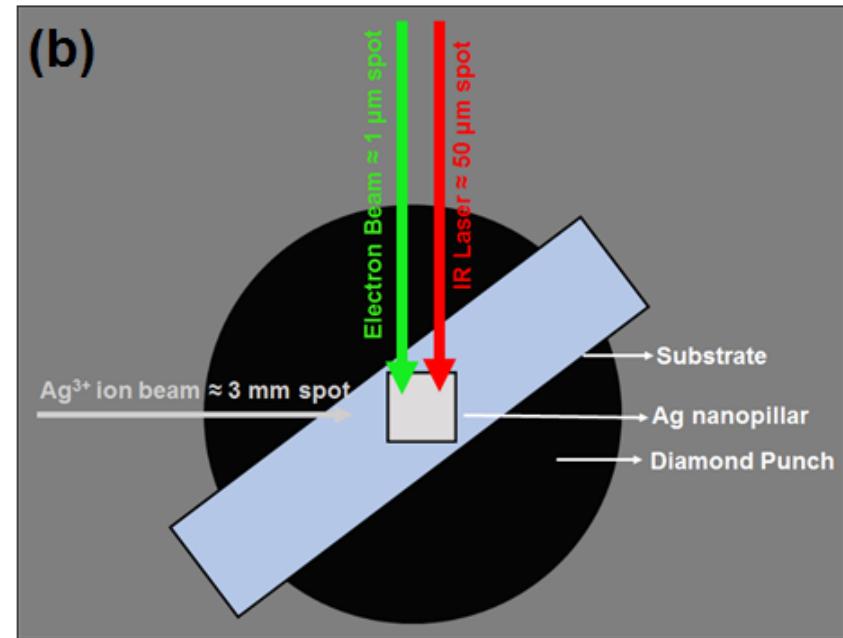


# Loading Geometry

(a)

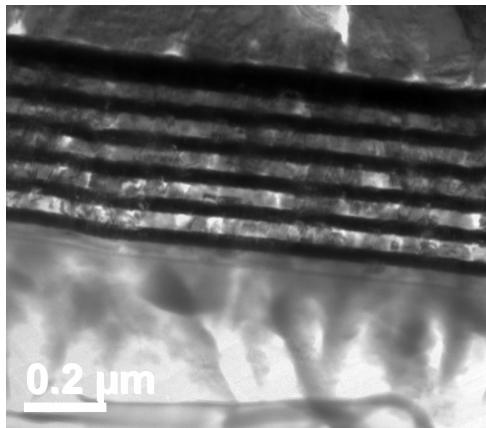


(b)

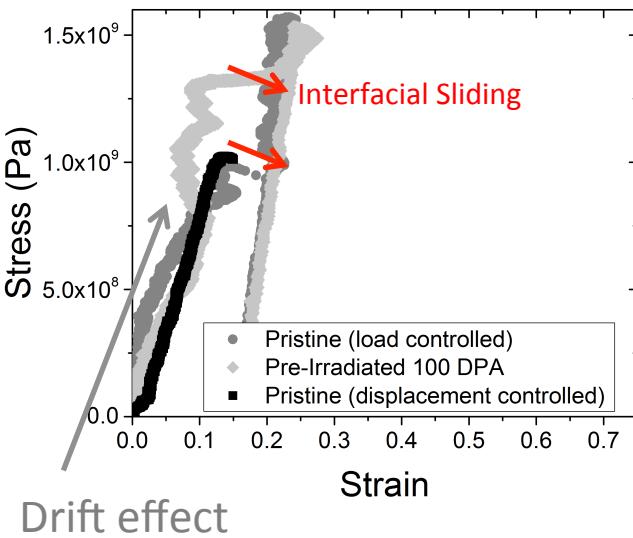


# Irradiation Creep (4 MeV Cu<sup>3+</sup> 10<sup>-2</sup> DPA/s)

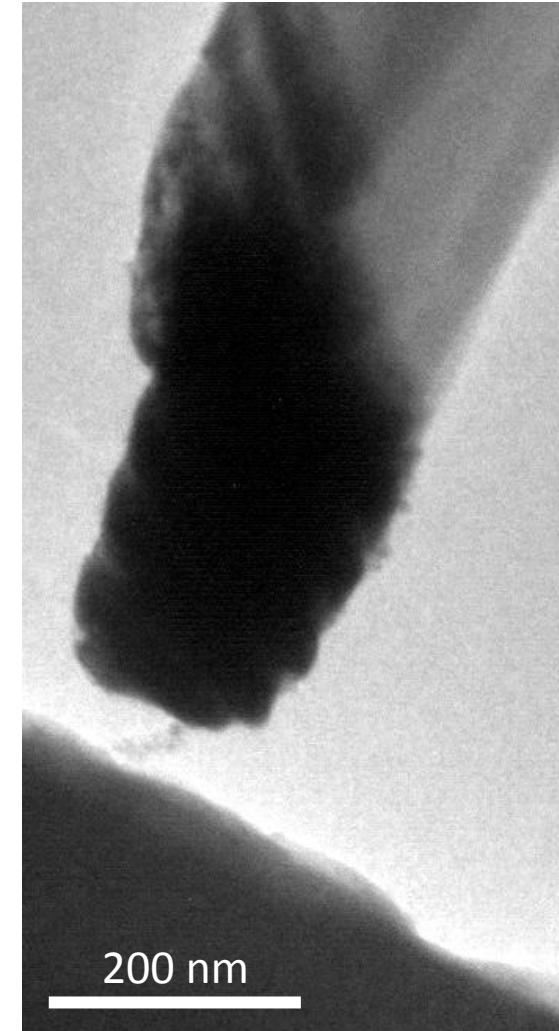
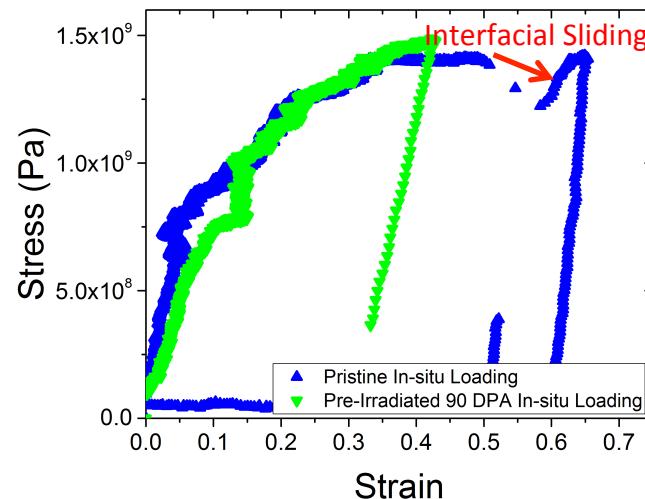
50 nm Cu-W multilayer  
20 Min



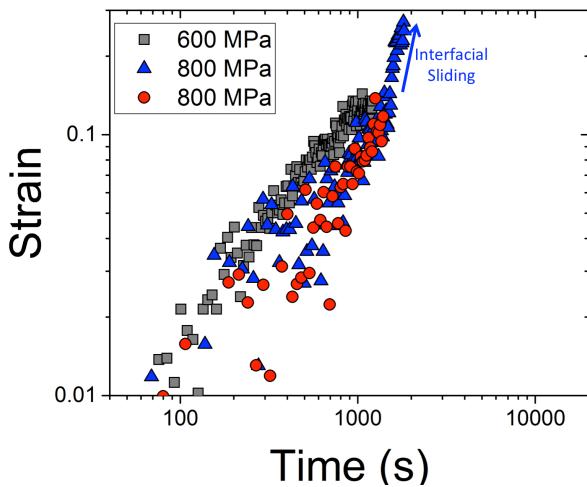
No Irradiation  
(Loading rate 0.6 Mpa s<sup>-1</sup>)



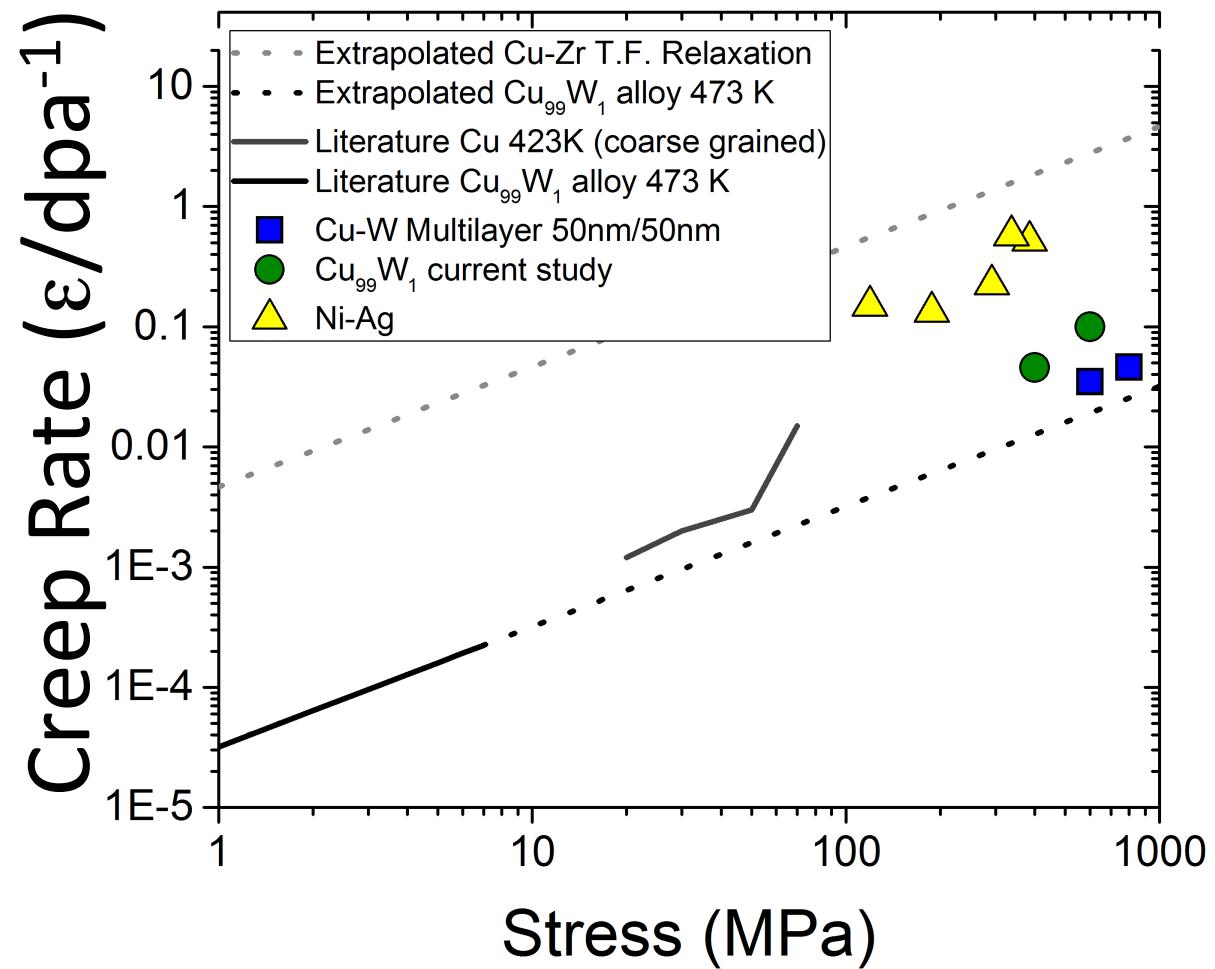
Irradiation Creep  
(Loading rate 0.6 Mpa s<sup>-1</sup>)



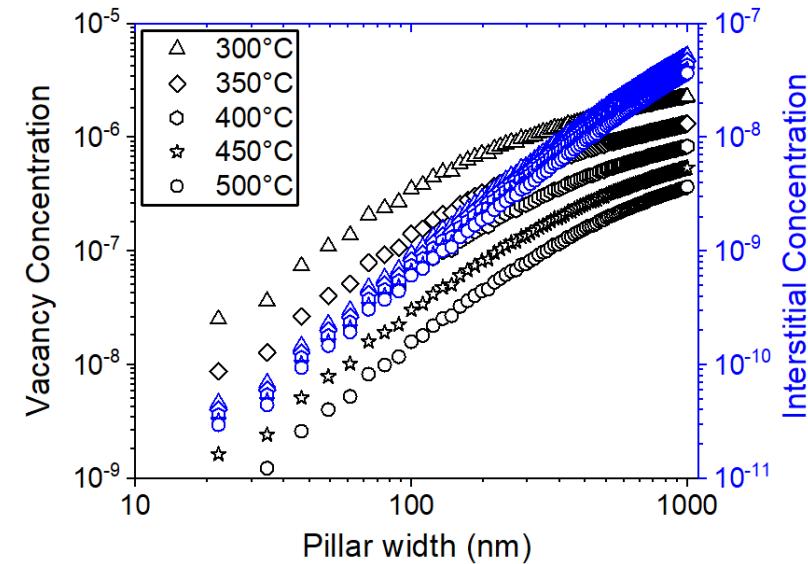
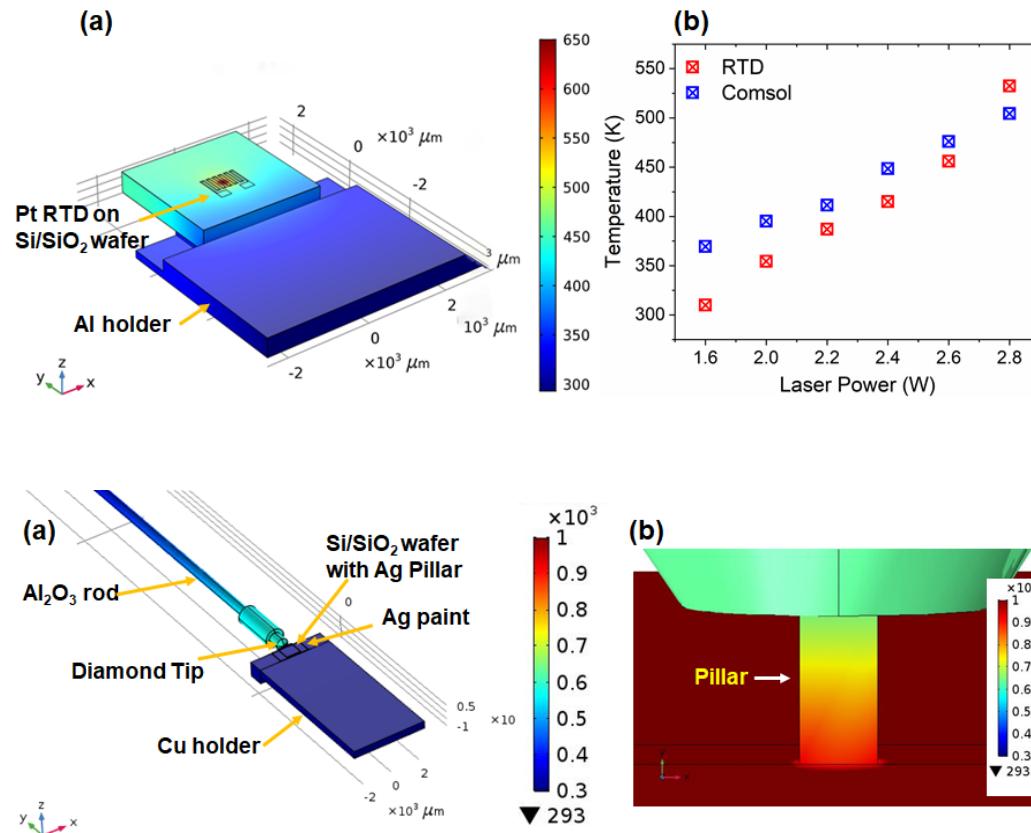
# Comparison of Steady-State Creep Data



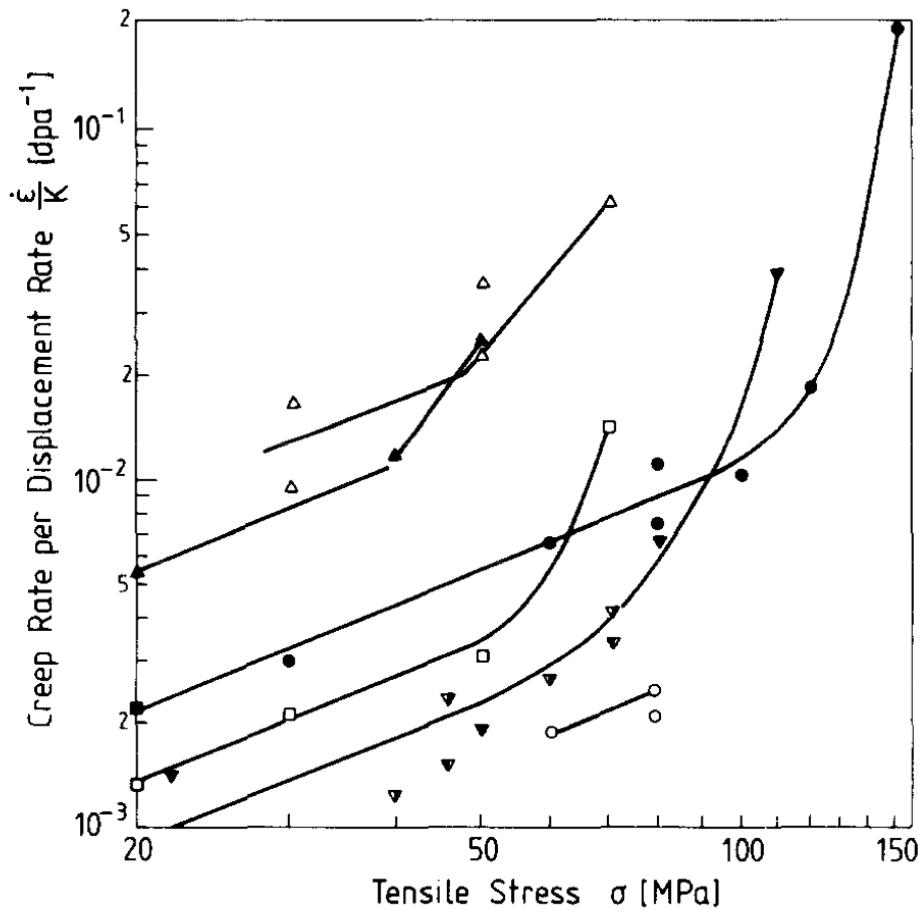
- R.T. IIC rate in nanoliminarites significant at  $<10\% \sigma_y$
- IIC rates in Ni-Ag approaching theoretical maximum
- Mechanism for Cu-W in high stress regime likely different than prior low stress measurements



# High Temperature Experiments on Ag: Approximating Temperature Regime



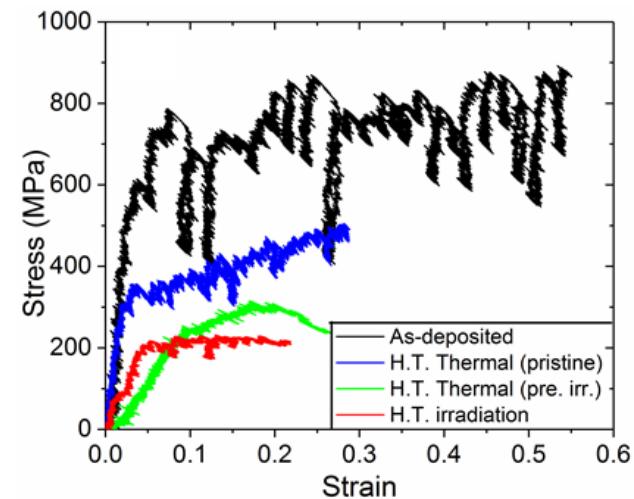
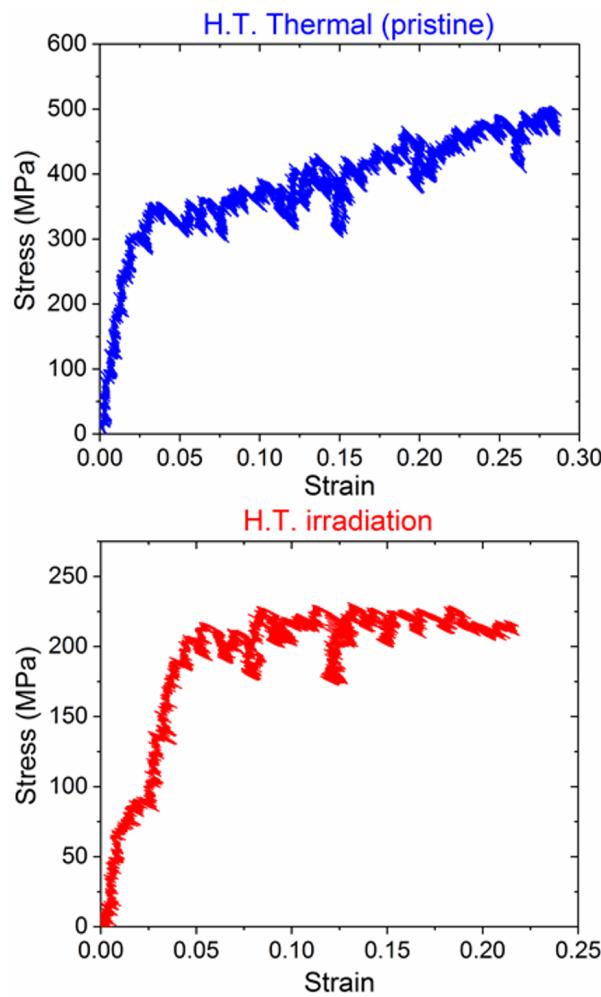
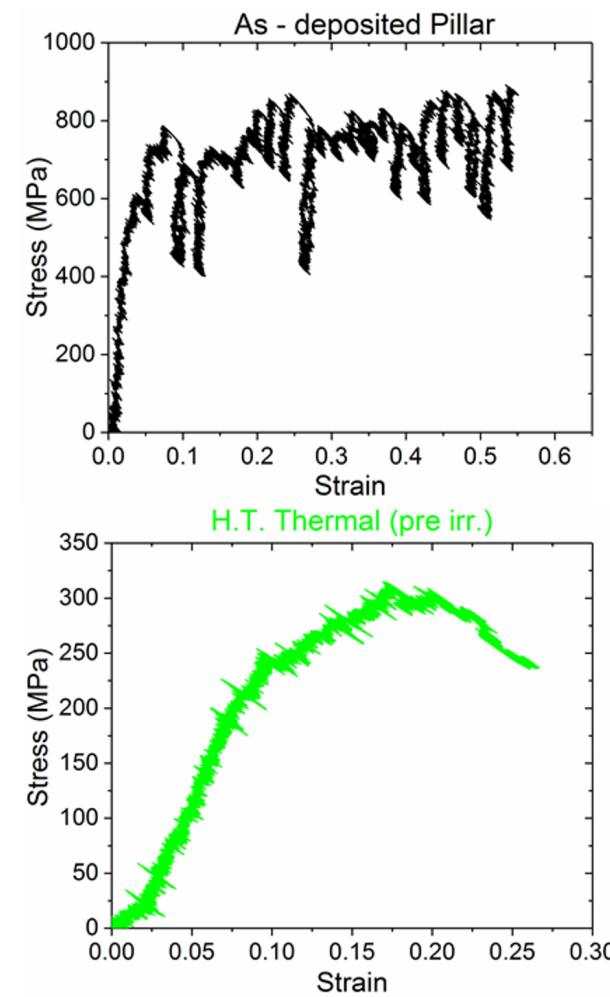
# Prior IIC on Ag



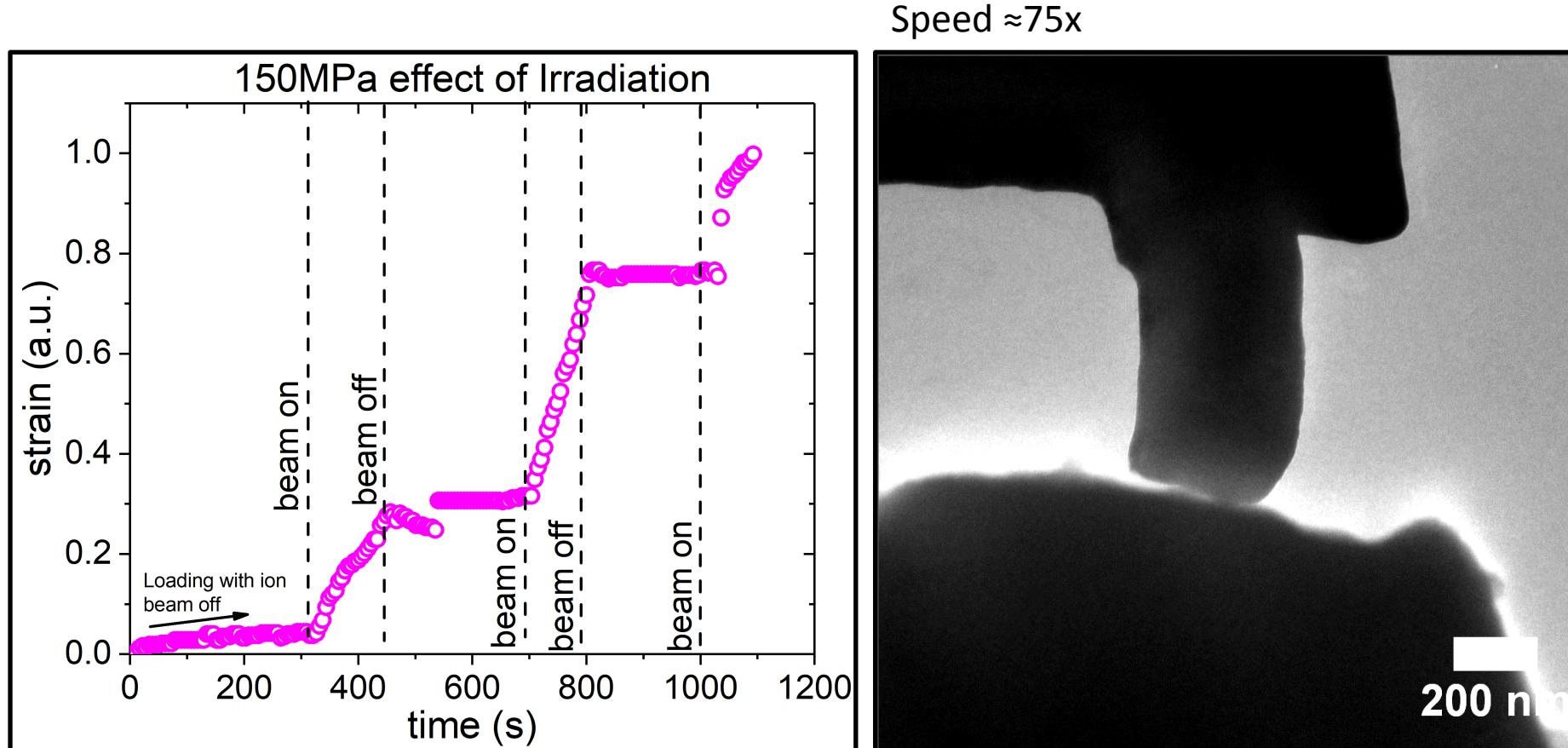
Sink limited regime  
activation energy:  $\approx 0.002$  eV

Fig. 5. Irradiation creep rate per displacement rate as a function of tensile stress of 50  $\mu\text{m}$  foils of 20% c.w. high purity metals at various temperatures. Nickel: 200°C (○), 300°C (●); copper: 150°C (□), 200°C (■); silver: 97°C (△), 150°C (▲); platinum: 220°C (▽), 240°C (▼), 300°C (▼).

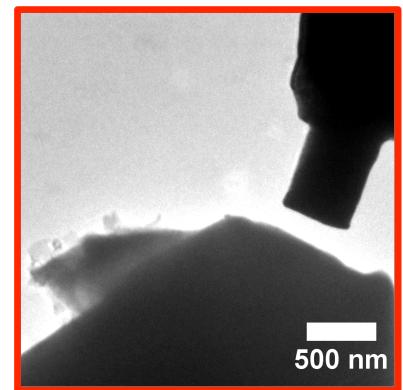
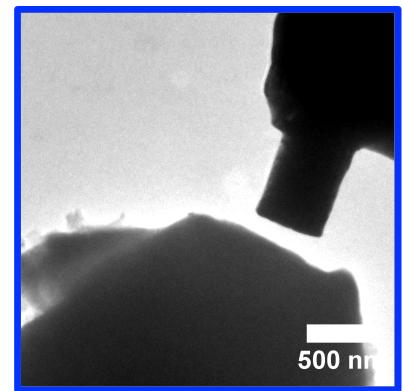
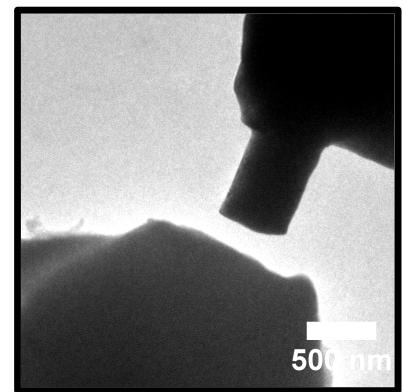
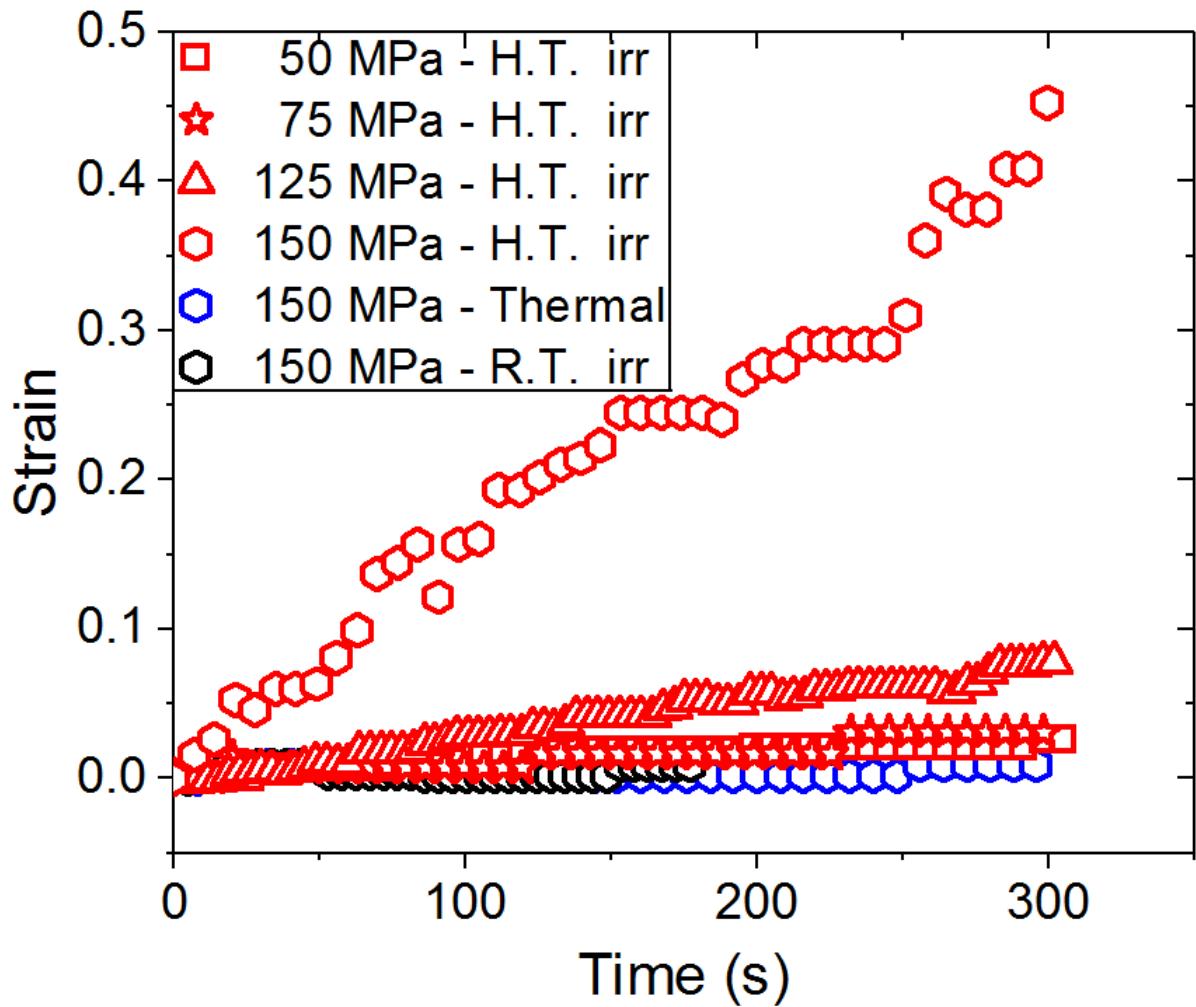
# Stress-Strain: Effect of Irradiation and Temperature



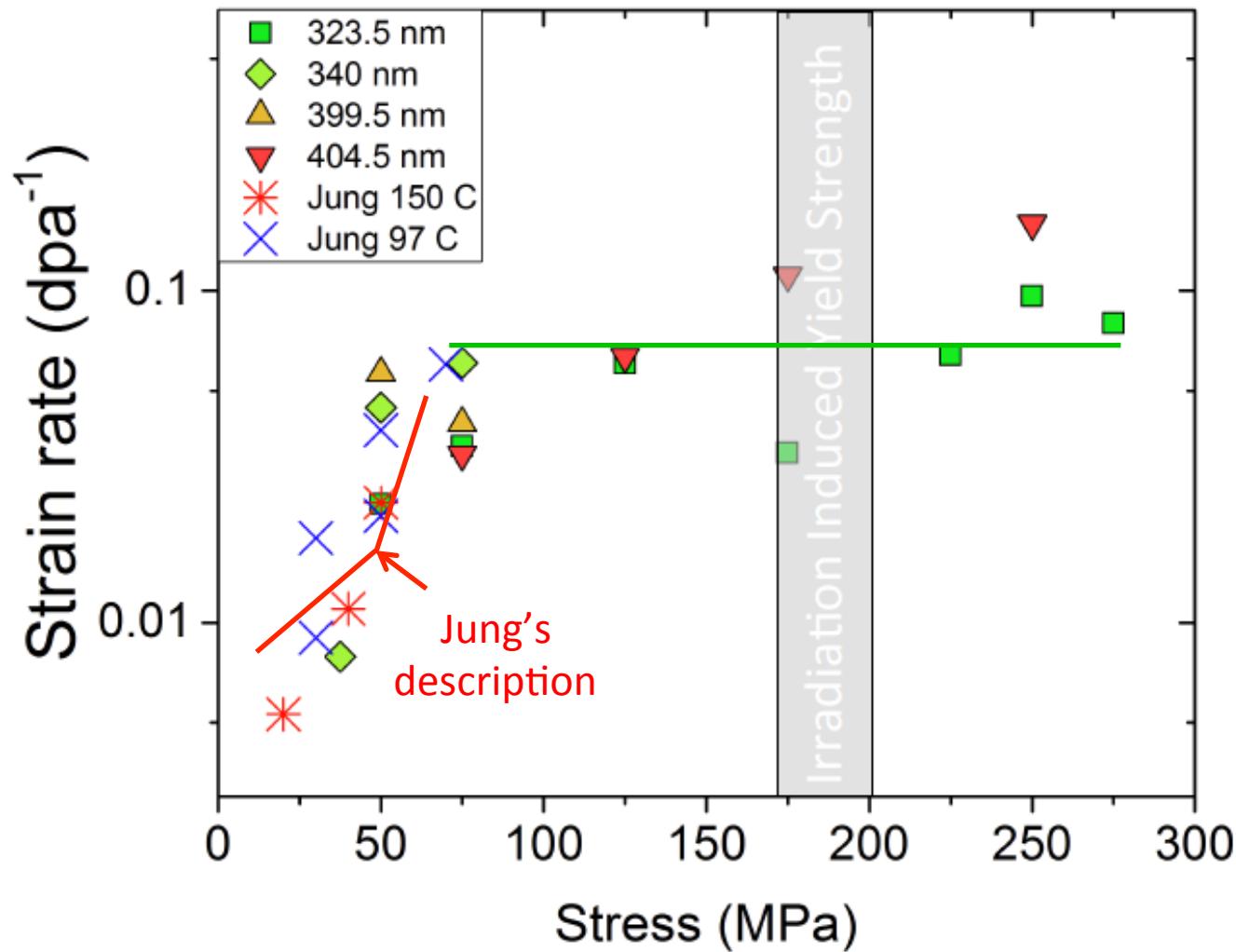
# IIC in Sample with Non-ideal Geometry/Deformation



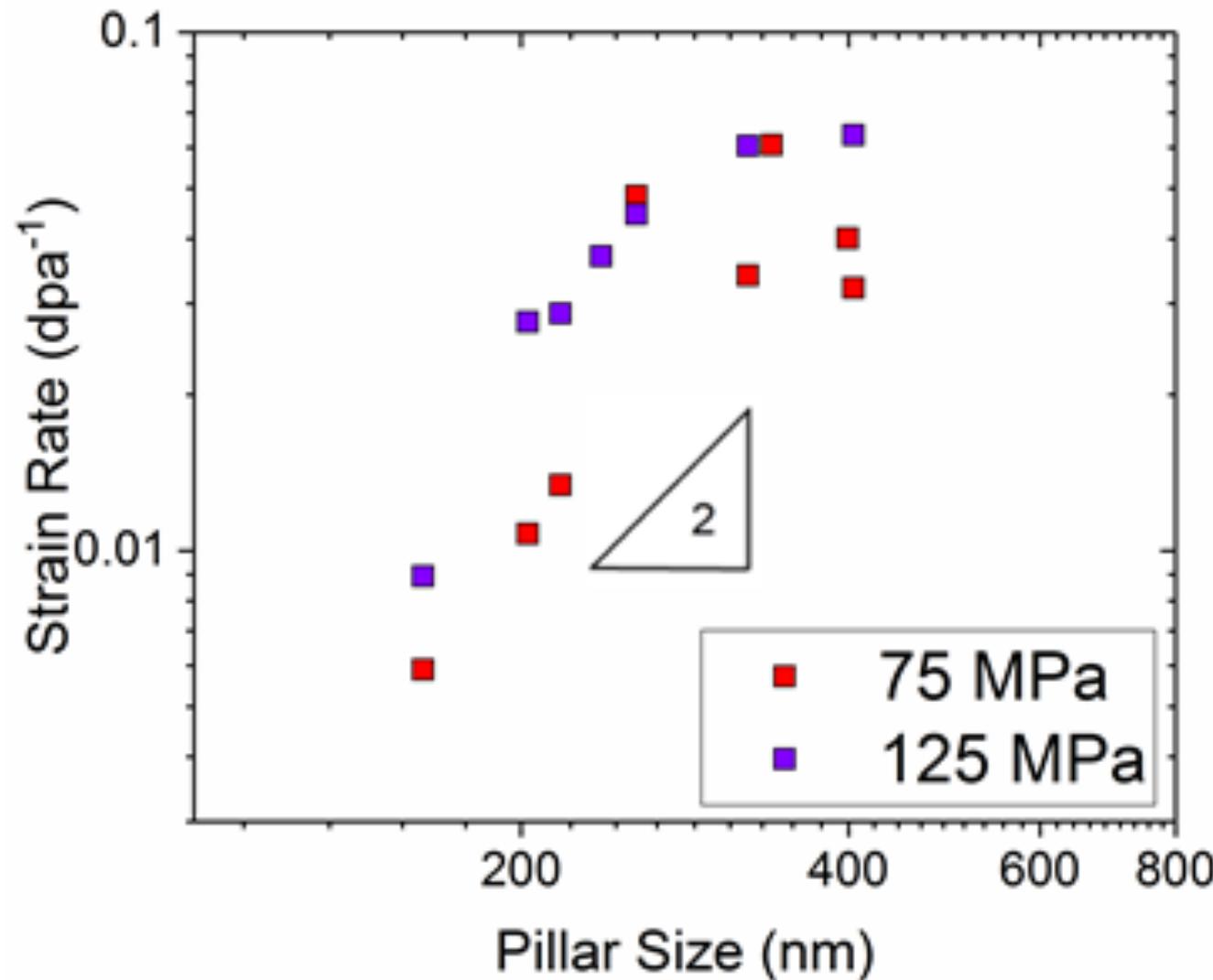
# Steady-State Creep Rates in Ag



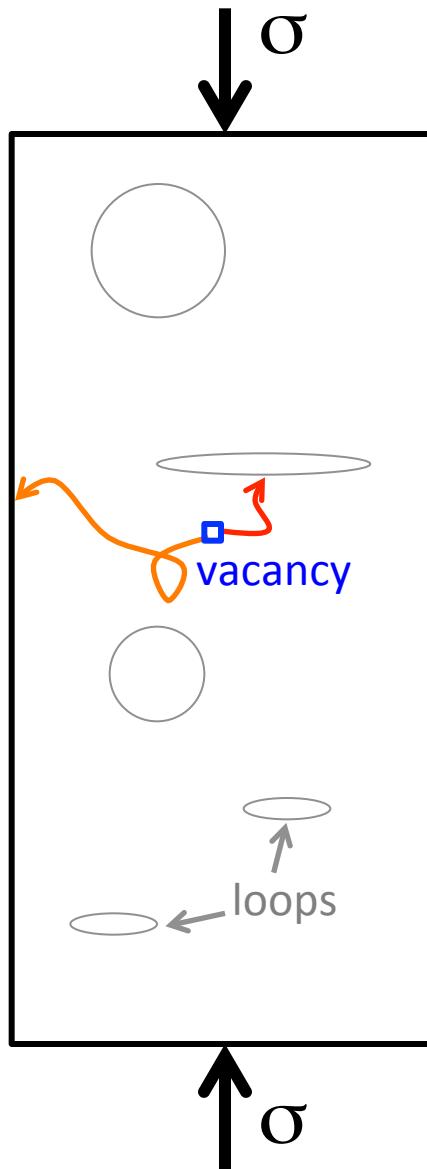
# Effects of Stress on IIC



# Effects of Pillar Size on IIC



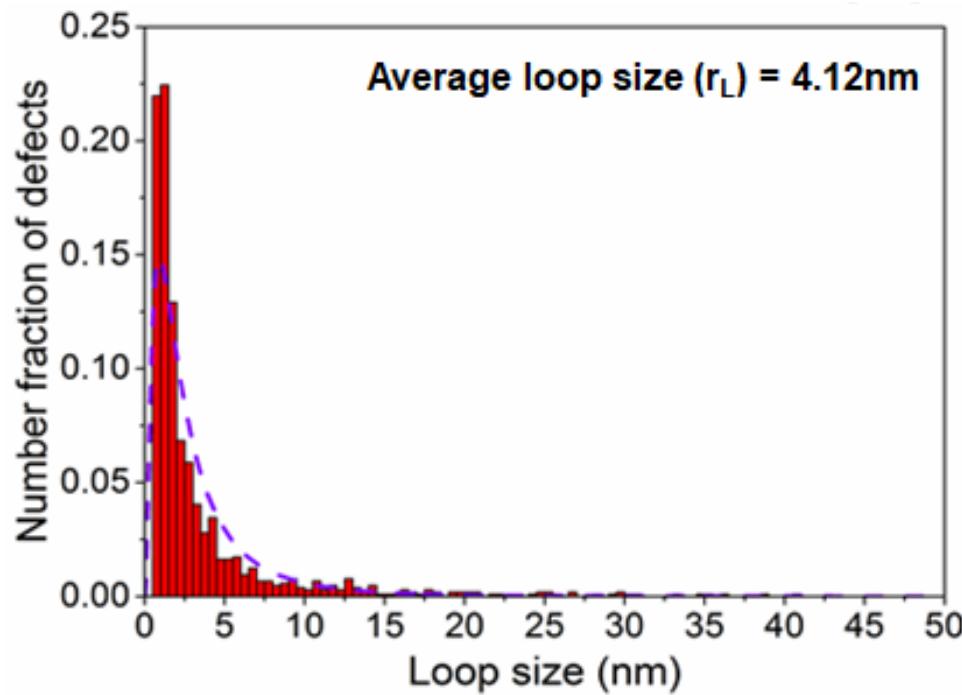
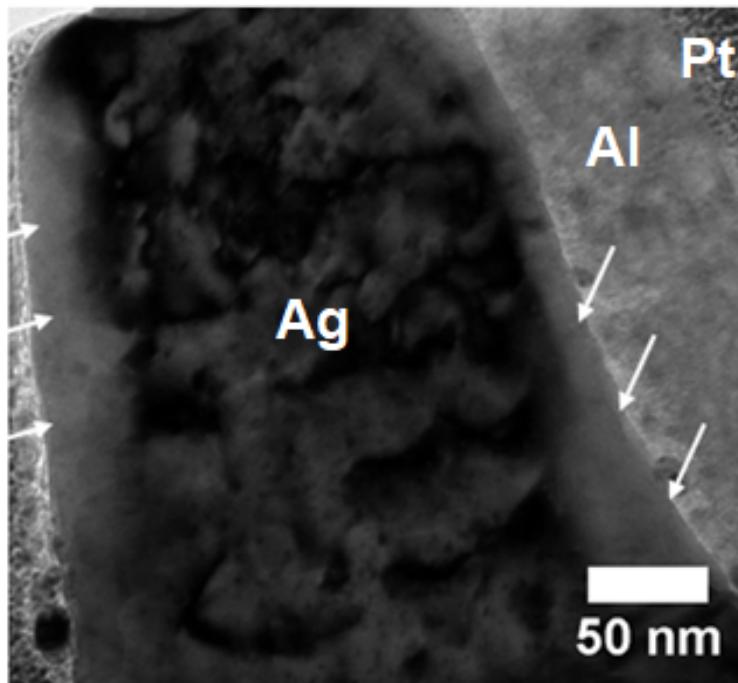
# Size Effect in Sink Limited Regime



Flux to the surface  
does not induce  
creep

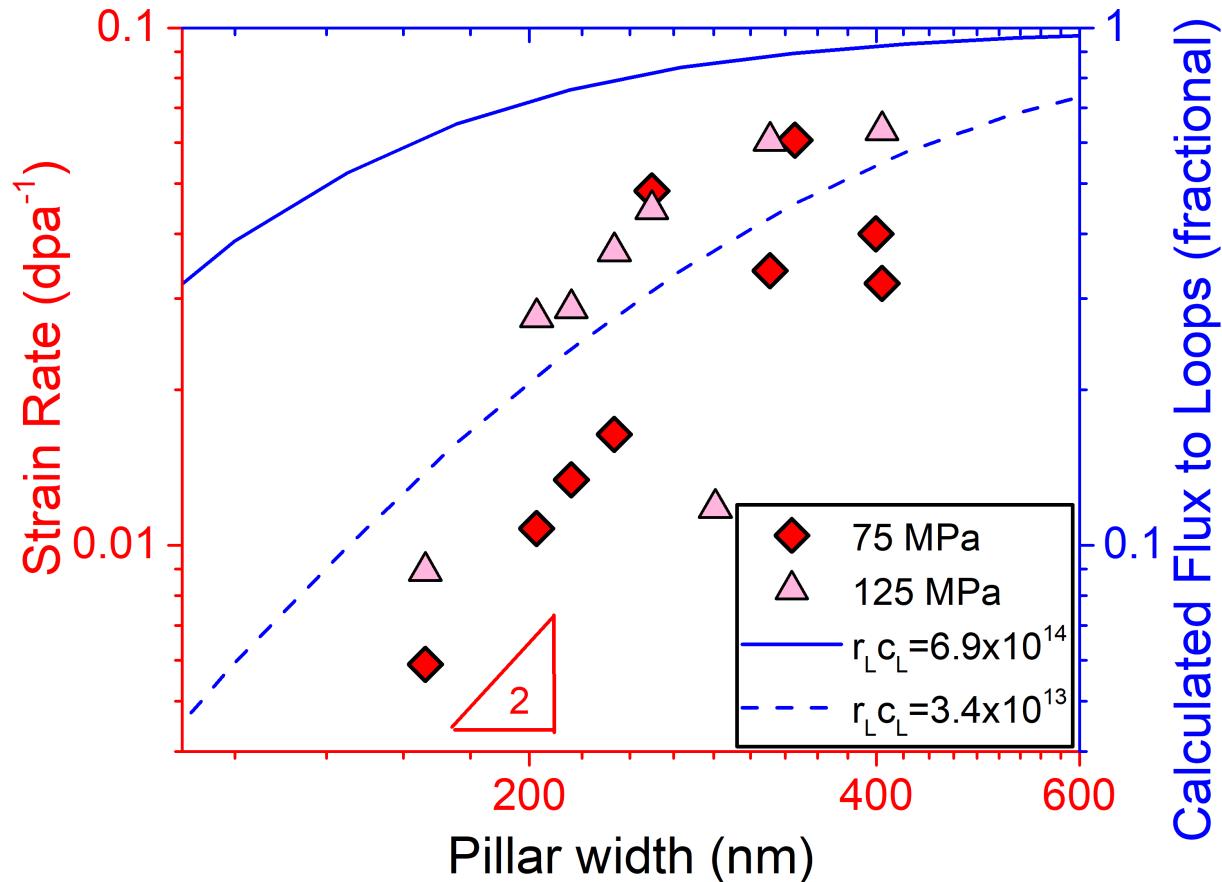
Stress induced  
preferential  
adsorption to loops  
induces creep

# Competition between Dislocations and Interfaces



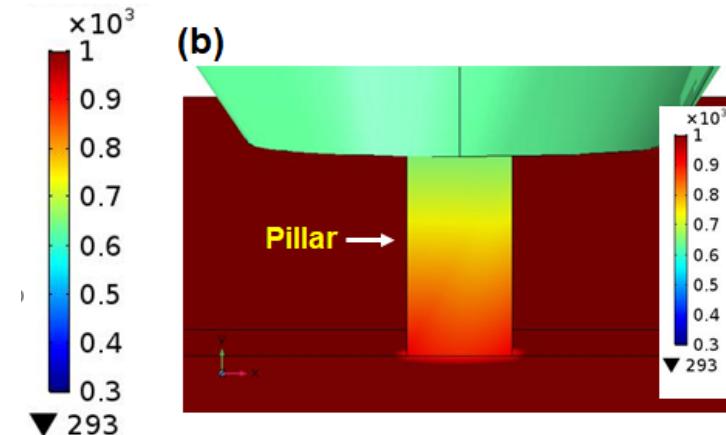
# Fit Predicted from S.S. Rate Equation

$$\frac{\partial c_{i(v)}}{\partial t} = 0 = \xi_{FM} K_o - \frac{4\pi r_{iv}}{\Omega} (D_i + D_v) c_{i(v)} c_{v(i)} - \frac{4\pi r_L}{\ln(8r_L/r_c)} D_{i(v)} c_{i(v)} c_L - \frac{2\pi^2}{L^2} D_{i(v)} c_{i(v)}$$

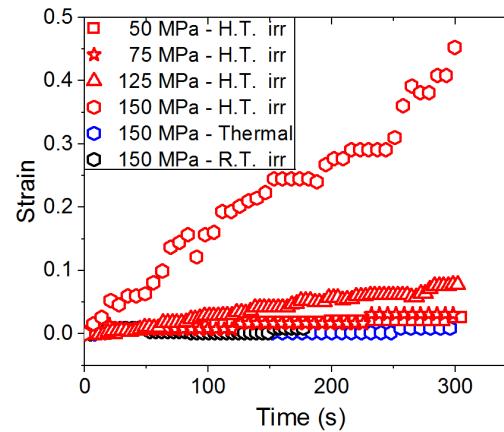


# Remaining Challenges

- Determining temperature and limit the temperature gradient



- Amplifying strain such that it is directly measurable during long (e.g. 1 hour) steady-state creep experiments



# Conclusions

- In situ TEM promising for small scale IIC testing
- IIC in ‘large’ Ag samples comparable to bulk measurements
- Competition between interfaces and loops as point defect sinks quantified through pillar size effects
- R.T. IIC rates may be high in high strength nanostructured materials at a fraction of their yield strengths