

# Irradiation Creep in Nanostructures

## Measured Using In-situ TEM

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Acknowledgements:

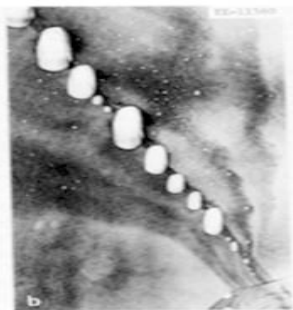
*US DOE DEFG02-05ER46217*

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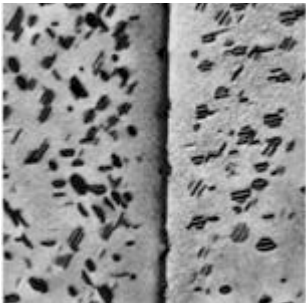
# Example Issues in Nuclear Materials

## Microscopic

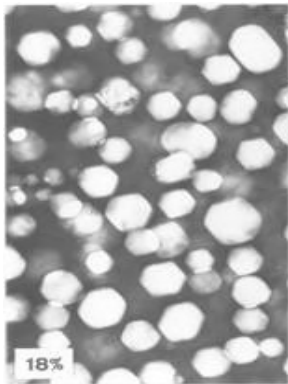
Gas Bubbles



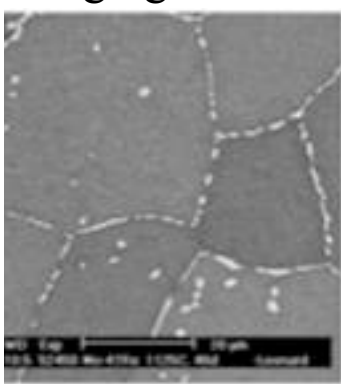
Voids



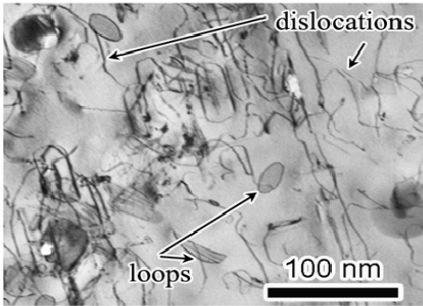
Precipitation



Segregation

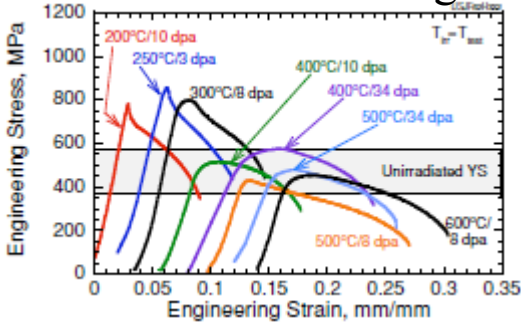


## Line Defects



## Macroscopic

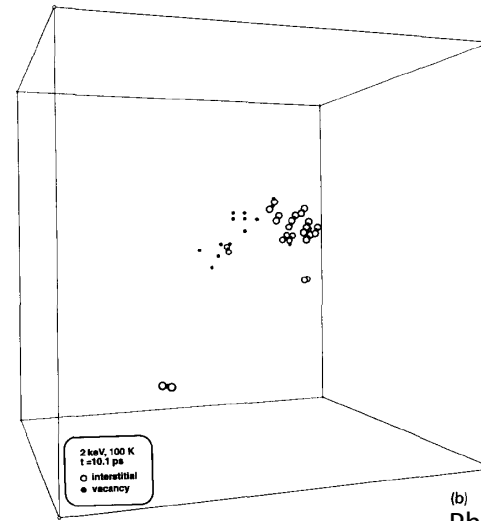
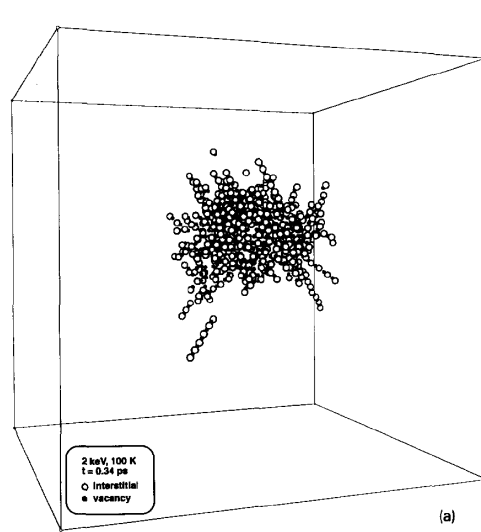
Radiation hardening



Swelling/Creep

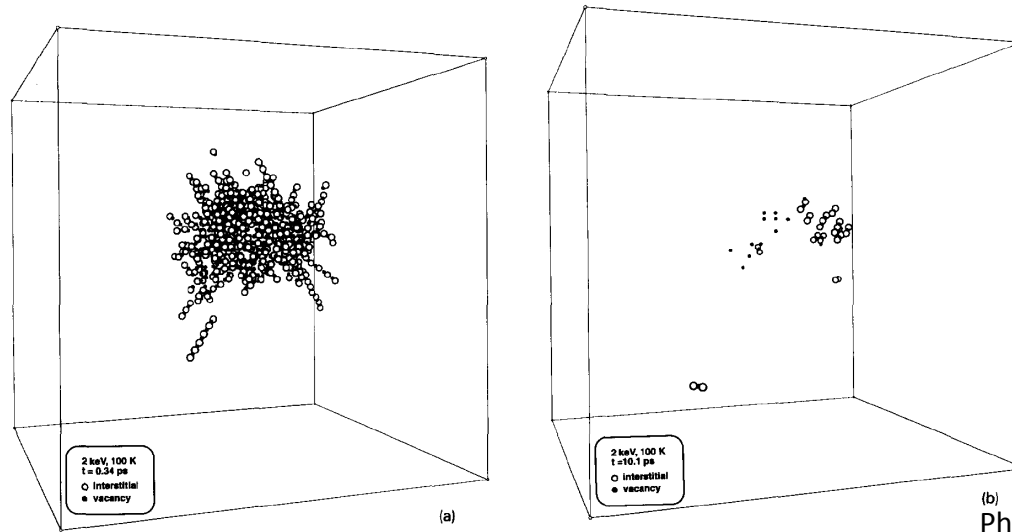


# Point Defect Production and Annihilation



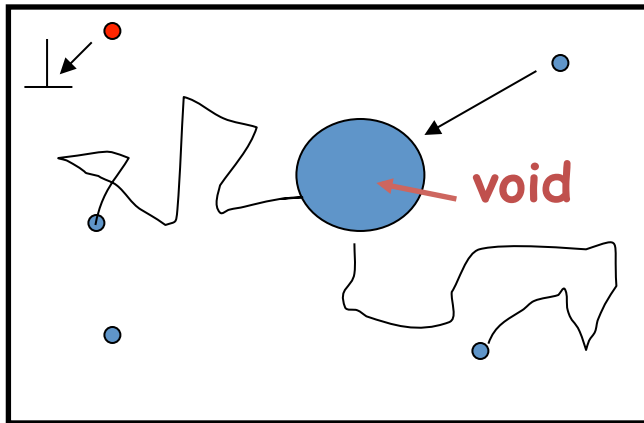
Phythian et al. *J. Nuc. Mater.* (1995)

# Point Defect Production and Annihilation



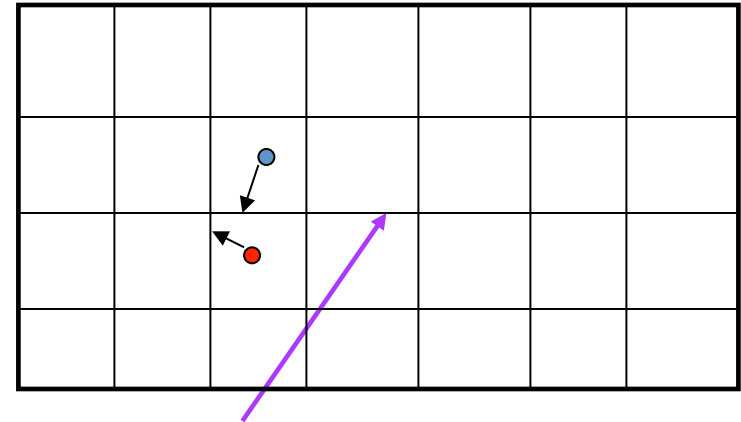
Phythian et al. *J. Nuc. Mater.* (1995)

## Conventional materials



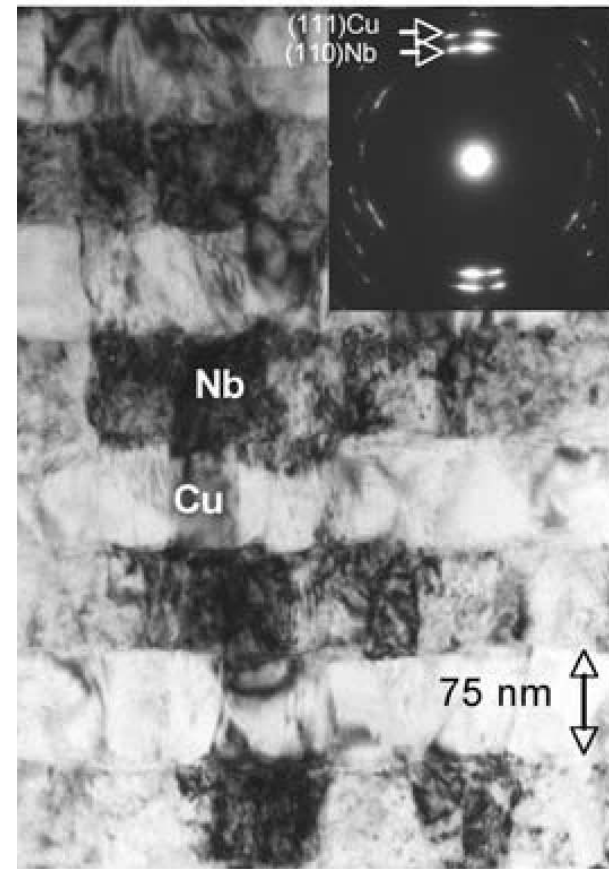
● Vacancies, ● interstitials

## Nanostructured materials



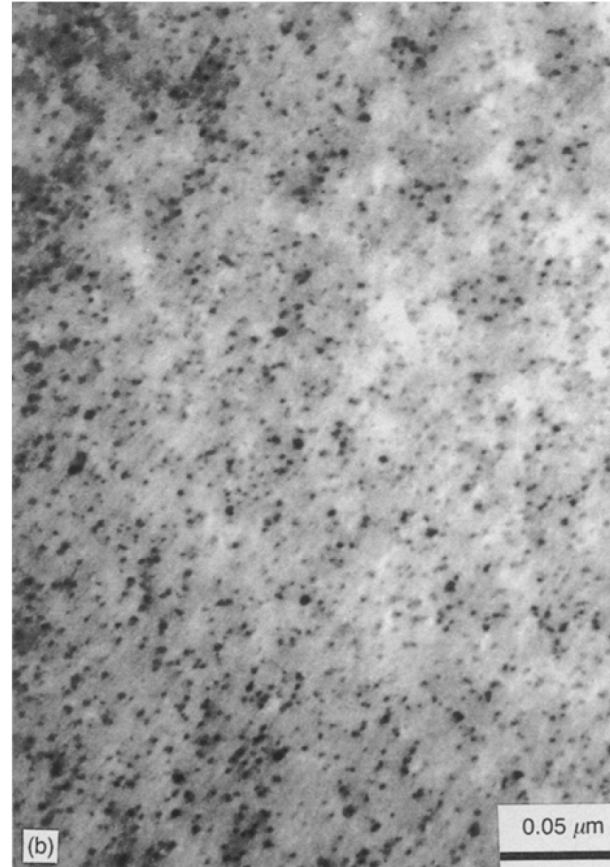
# Nanostructured Alloys

## Nanolaminates



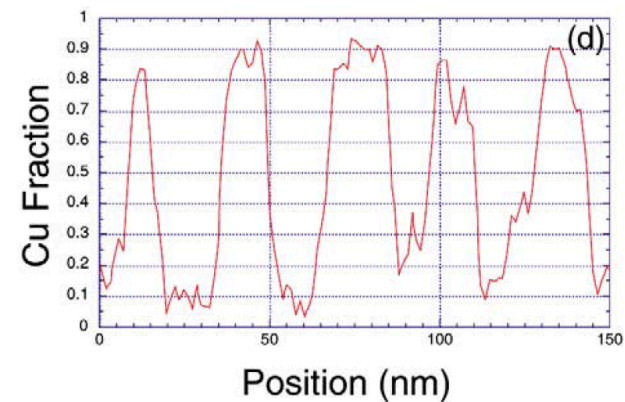
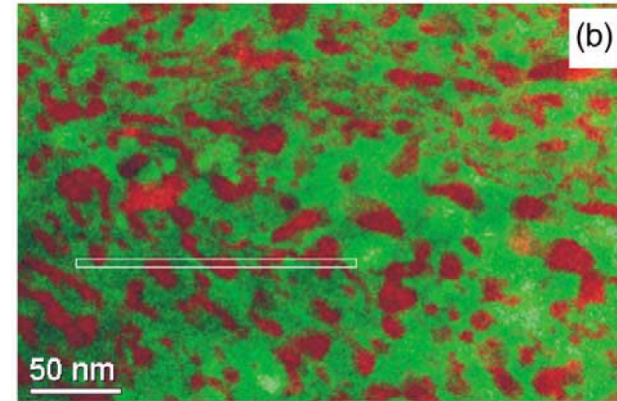
Misra and Hogland, *J. Mater. Sci.* (2007)

## Oxide Dispersion Strengthened



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

## Self-Organizing Alloys

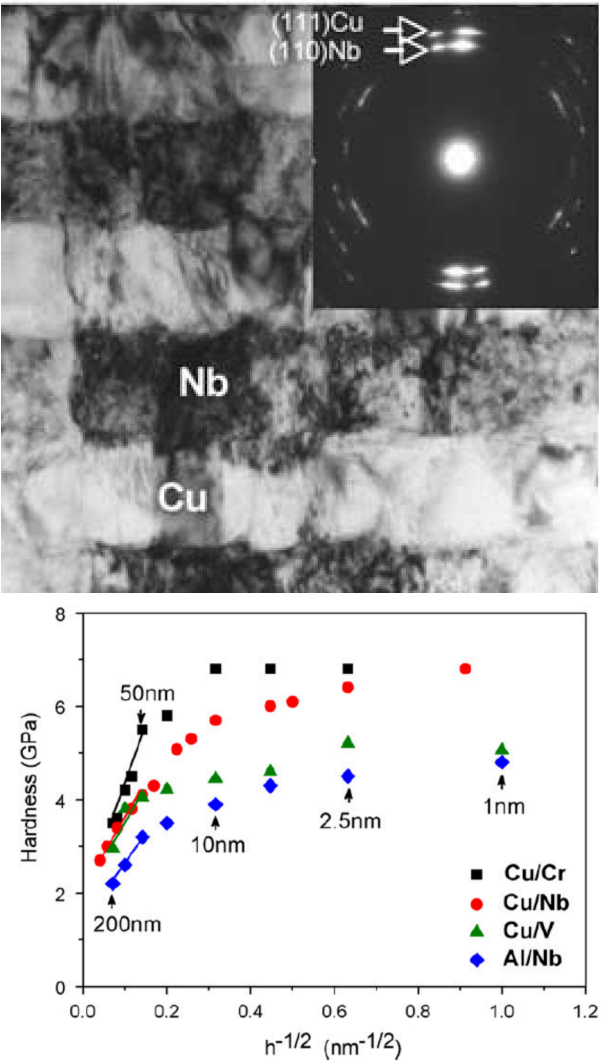


Zghal et al., *Acta Mater.* (2002)

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

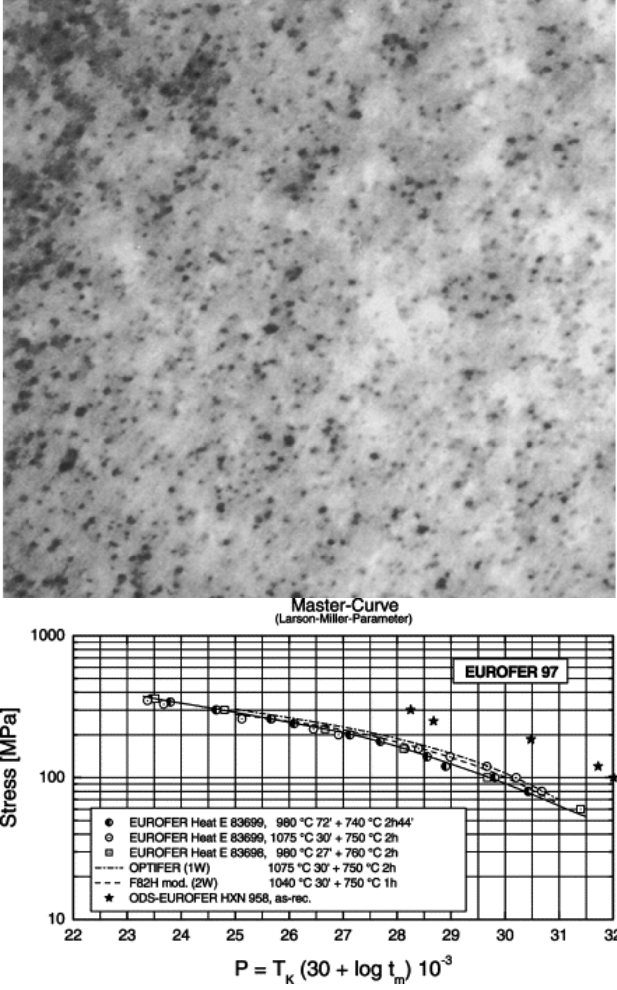
# Nanostructured Alloys

## Nanolaminates



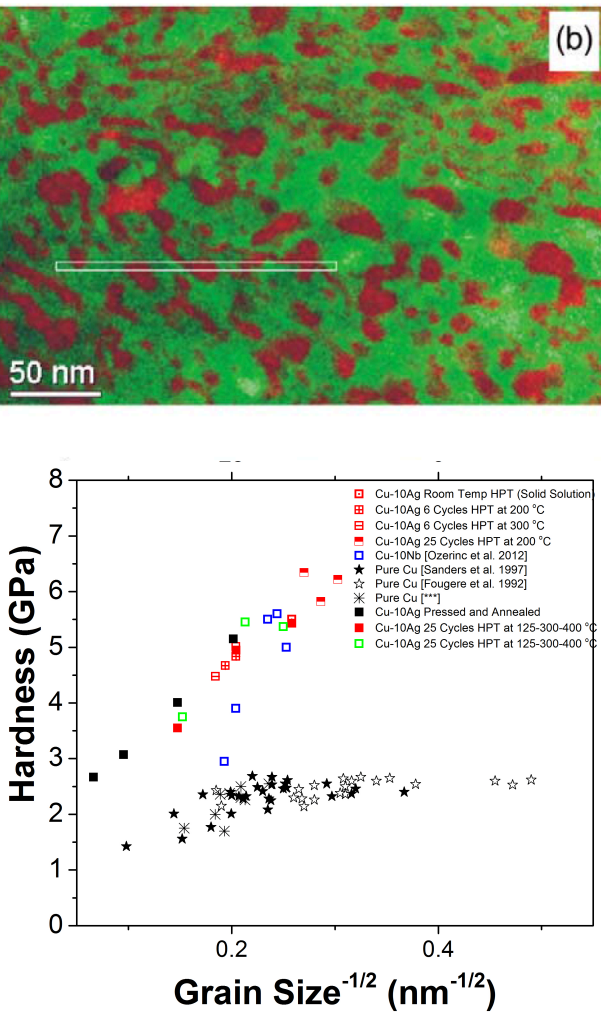
Misra and Hogland, *J. Mater. Sci.* (2007 )  
Fu et al., *Mater. Sci. A* (2008)

## Oxide Dispersion Strengthened



Okuda and Fujiwara, *J. Mater. Sci. Lett.* (1995)  
Lindau et al., *J. Nuc. Mater.* (2002)

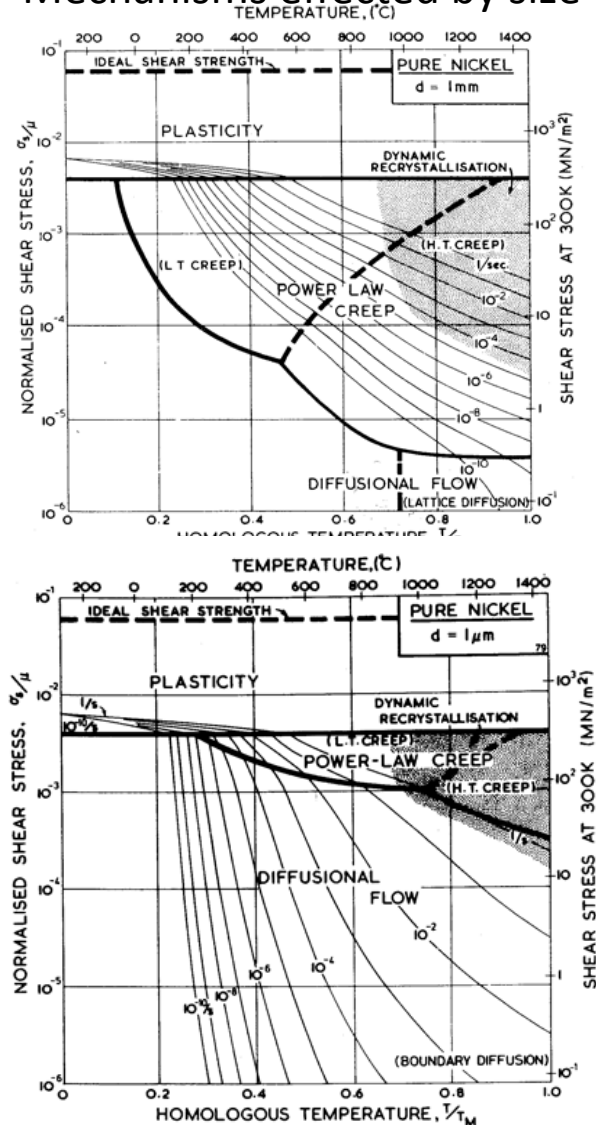
## Self-Organizing Alloys



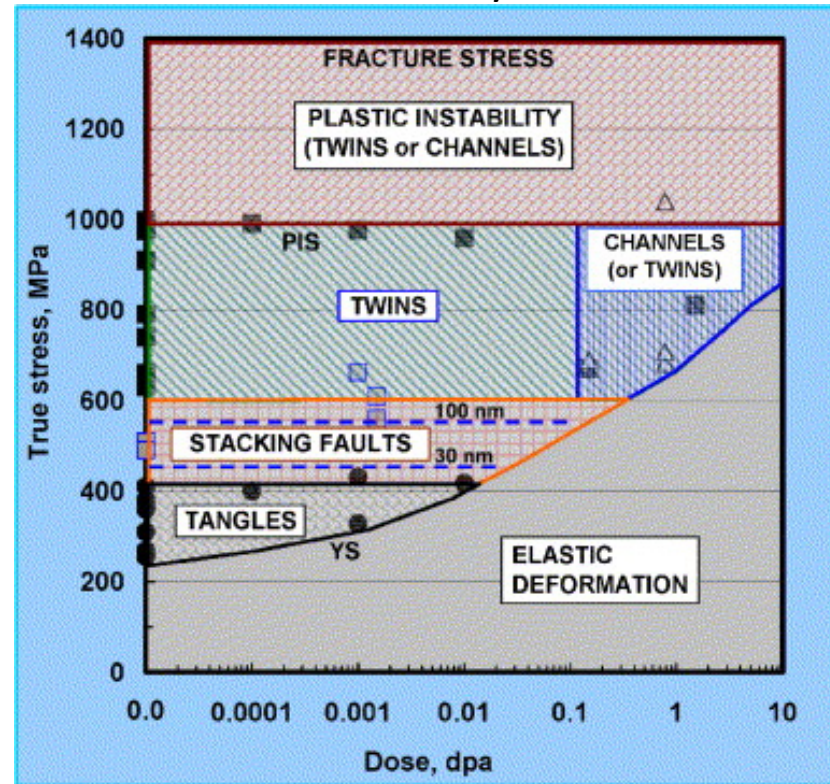
Zghal et al., *Acta Mater.*.. (2002)  
Arshad, Thesis (2014)

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

Mechanisms effected by size

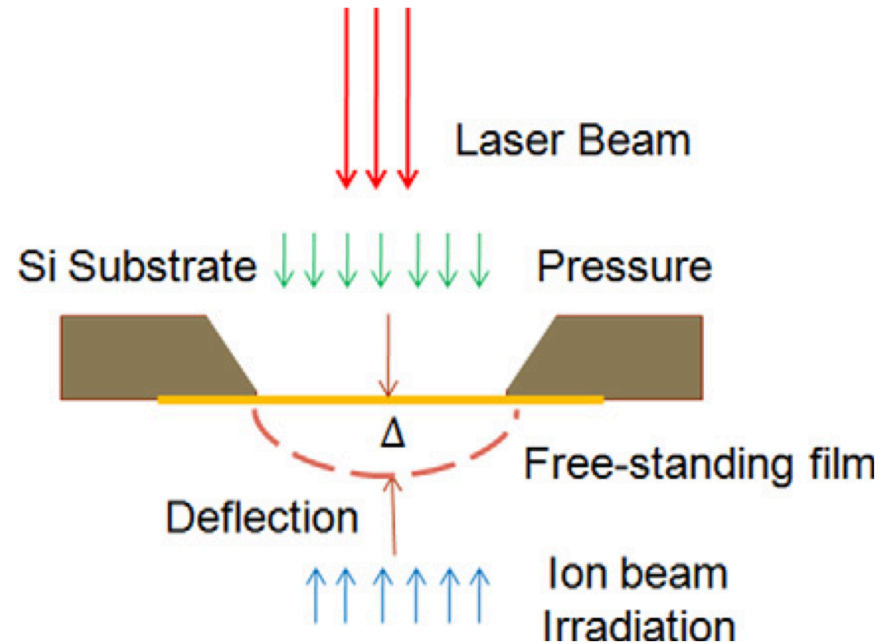
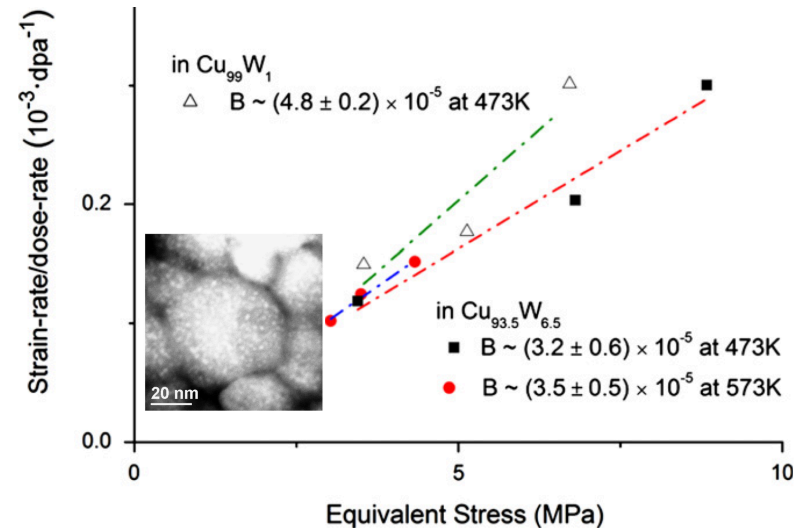
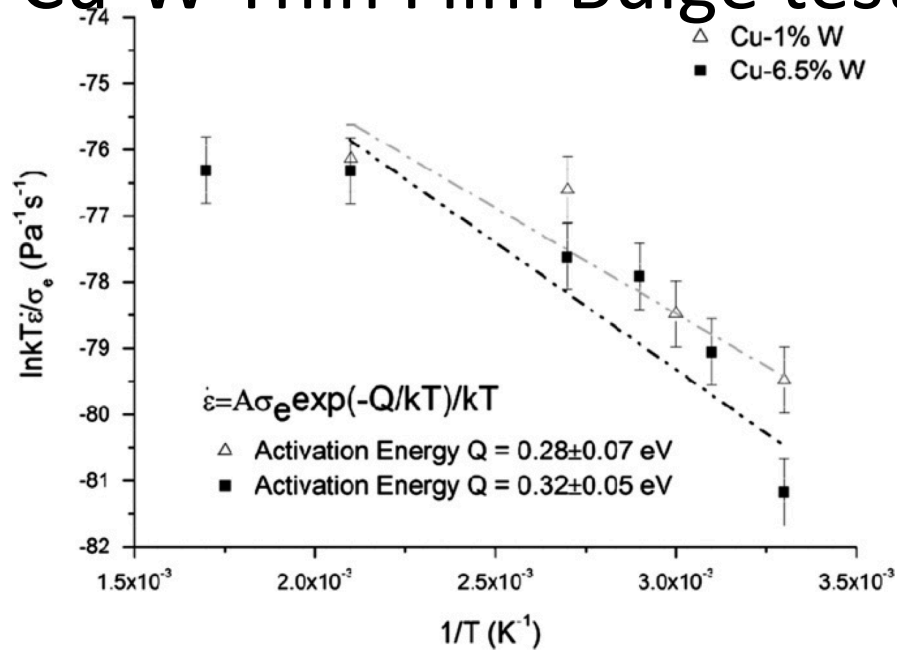


Mechanisms effected by irradiation

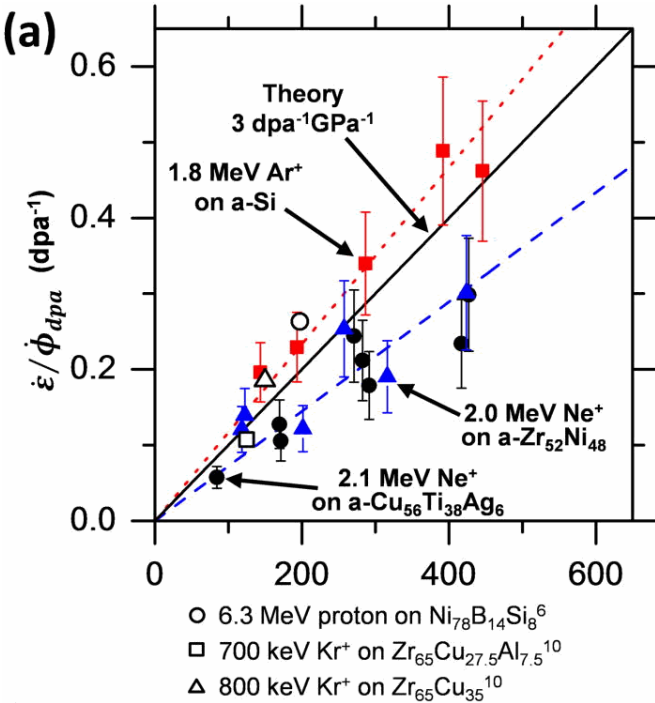
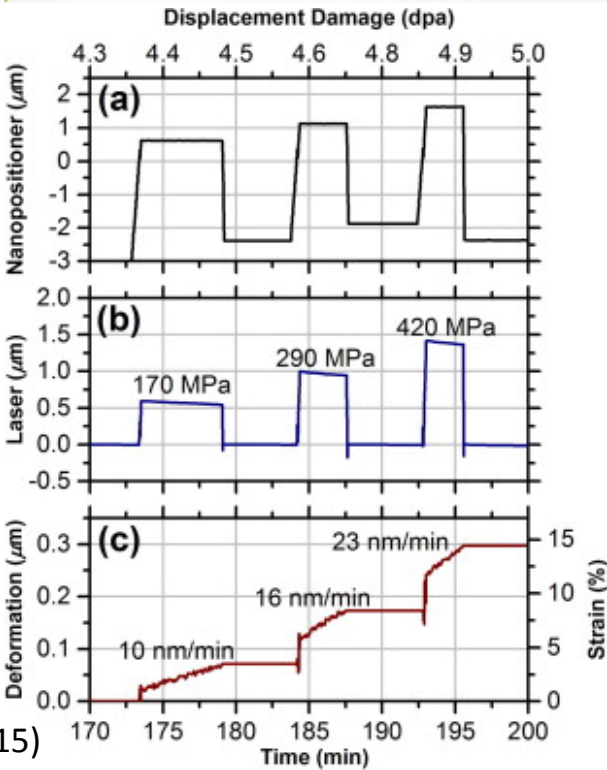
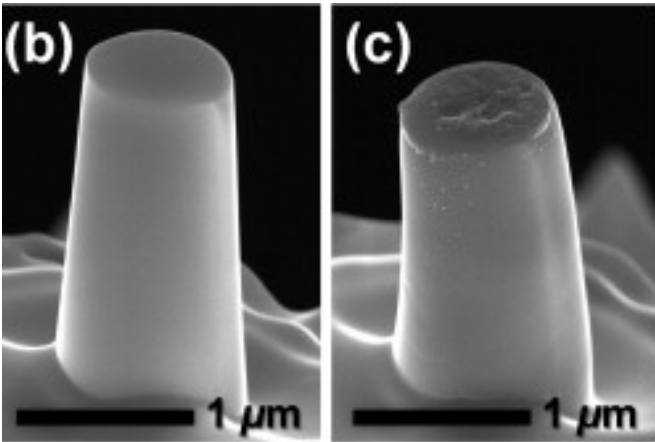
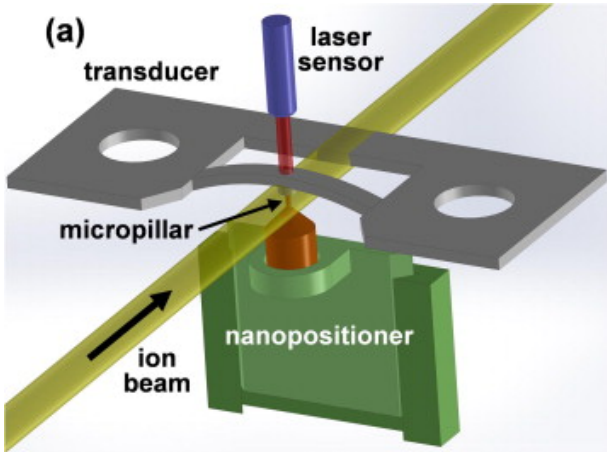


# Nanocrystalline Irradiation Creep Mechanisms

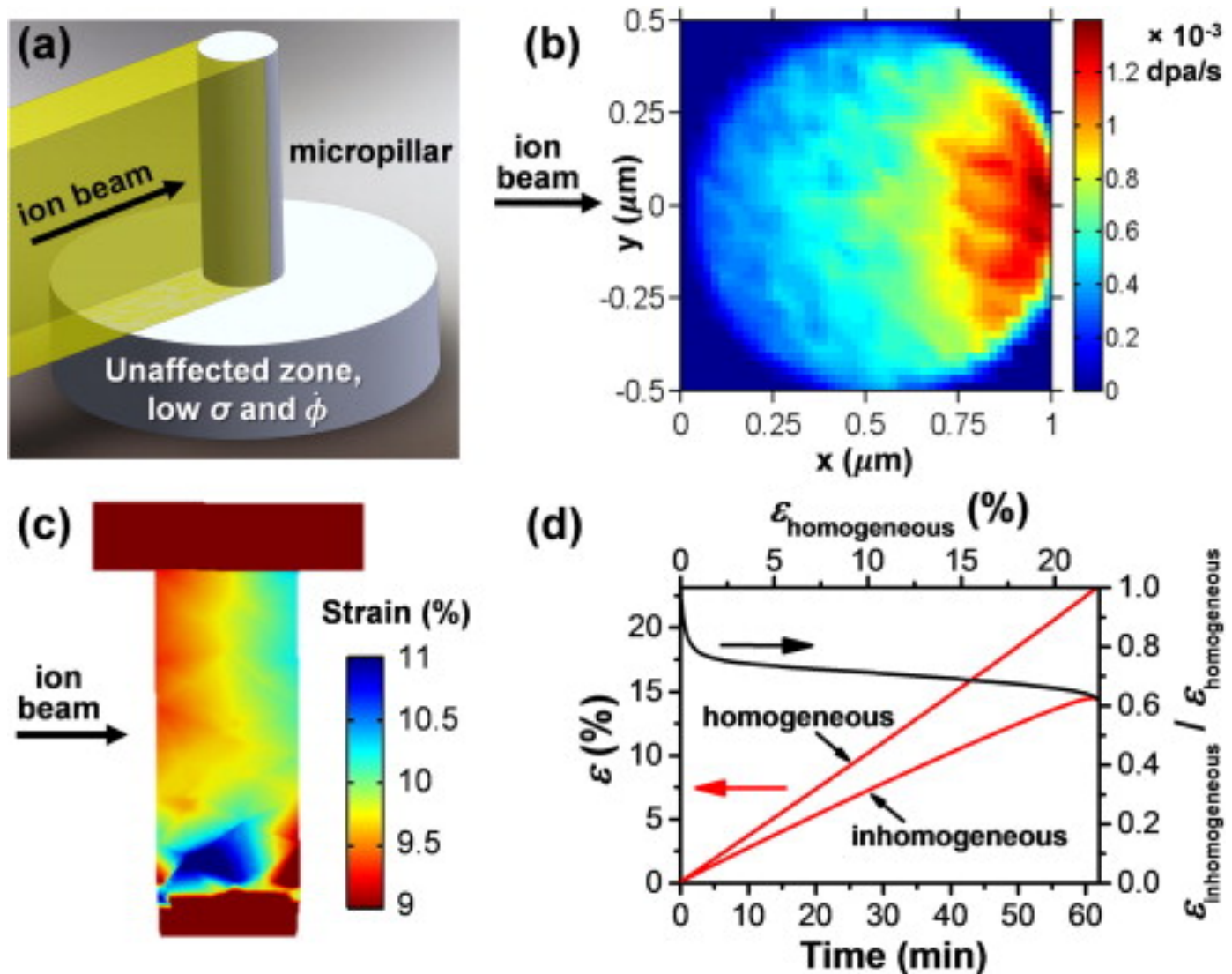
## Cu-W Thin Film Bulge test



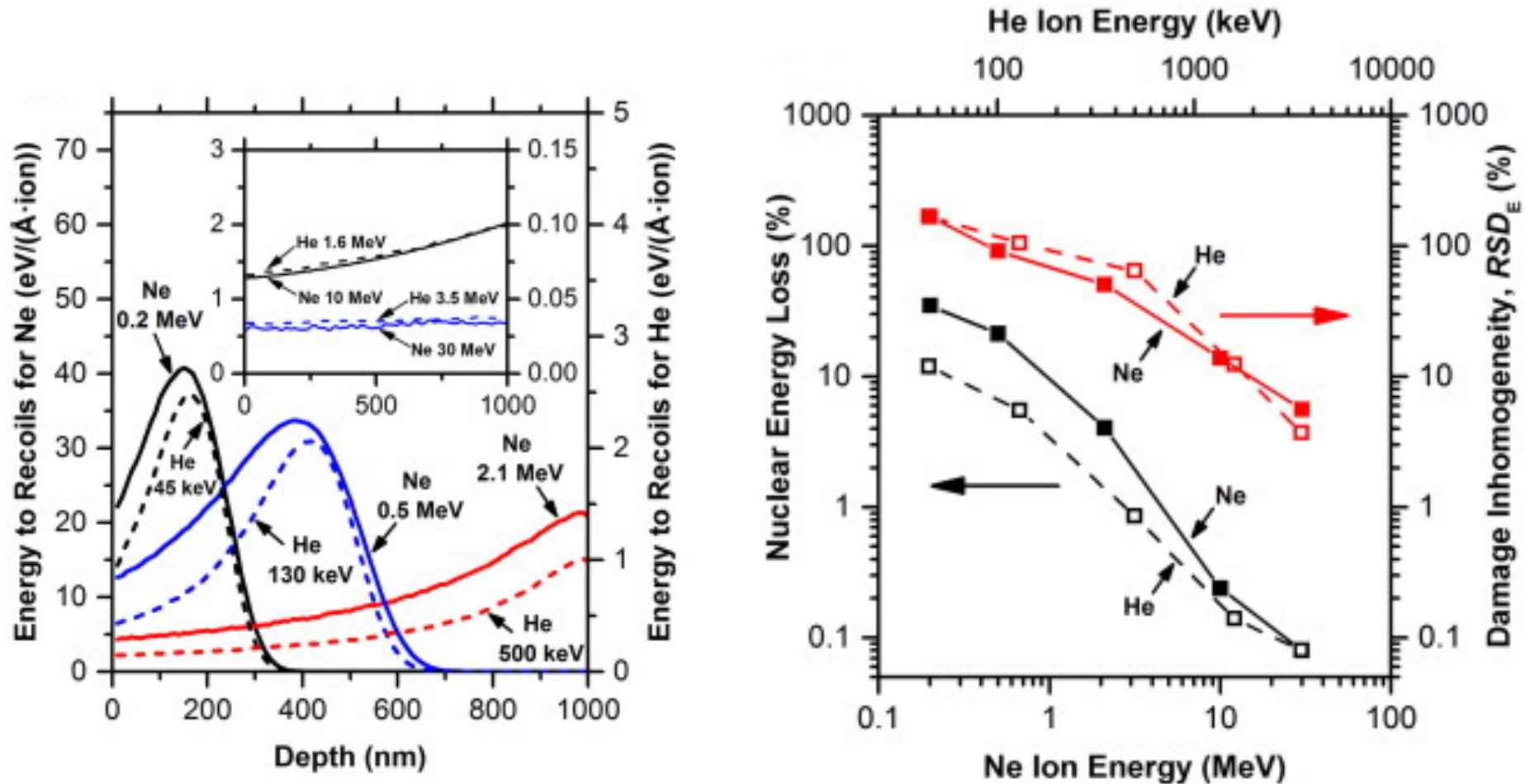
# Nanopillar Experiments



# Experimental Challenges

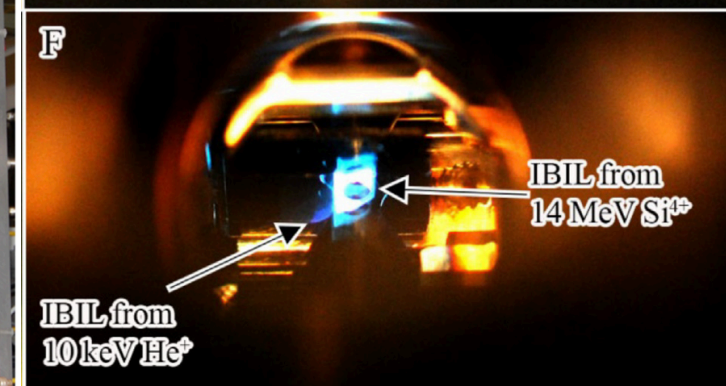
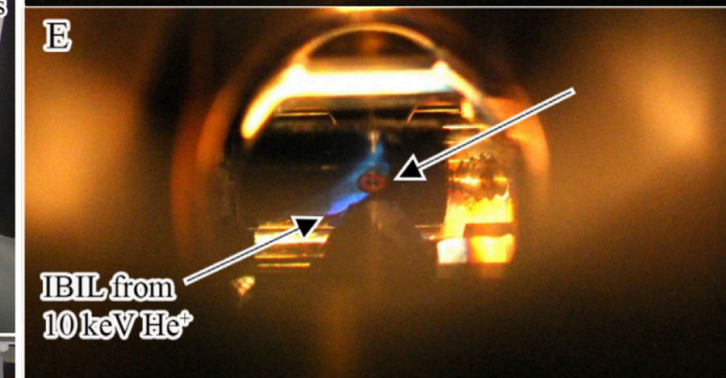
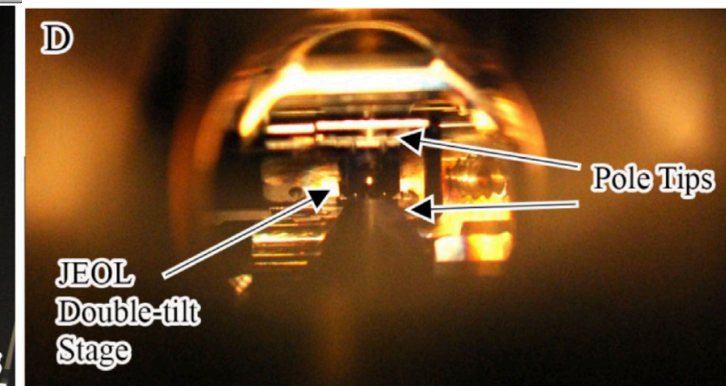
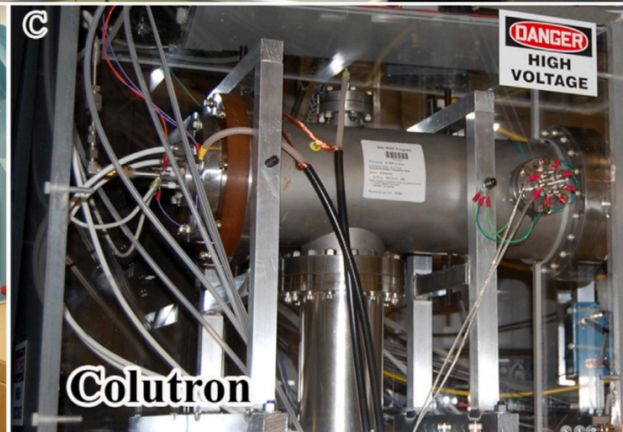
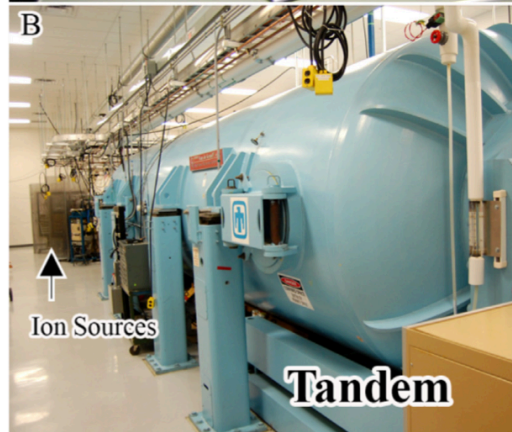
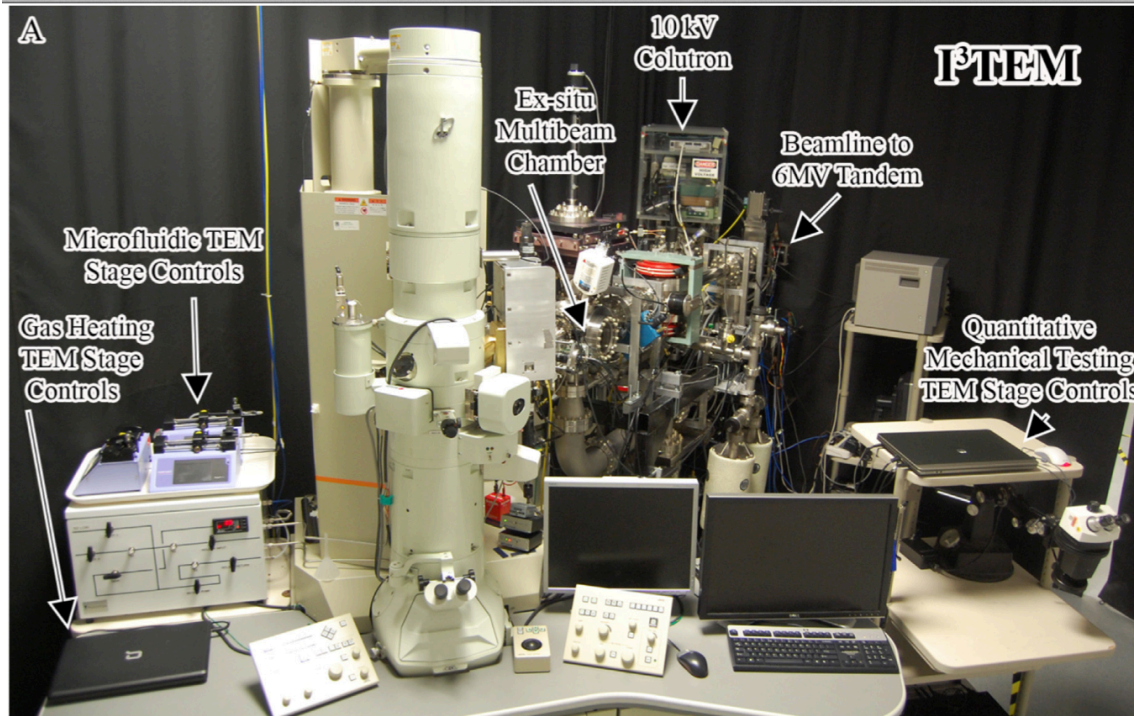


# Experimental Challenges



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



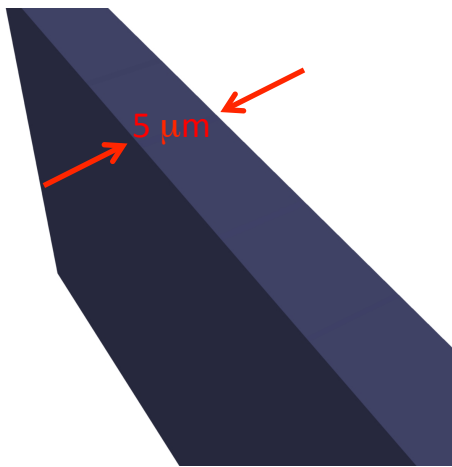
# Goals and Experiments

- 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)

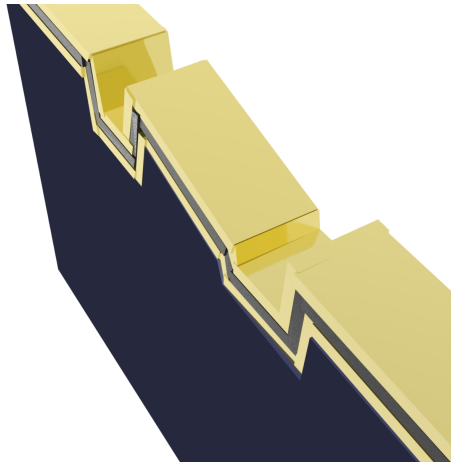
# Sample Preparation and Testing

## Multilayer Samples

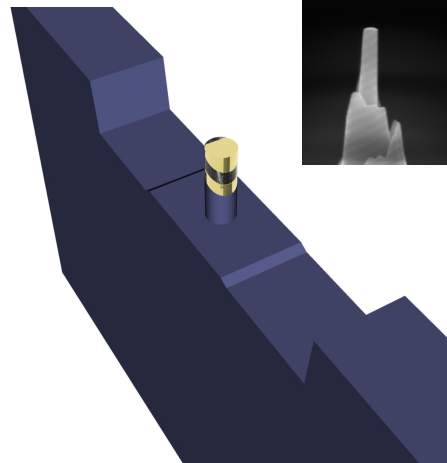
Substrate



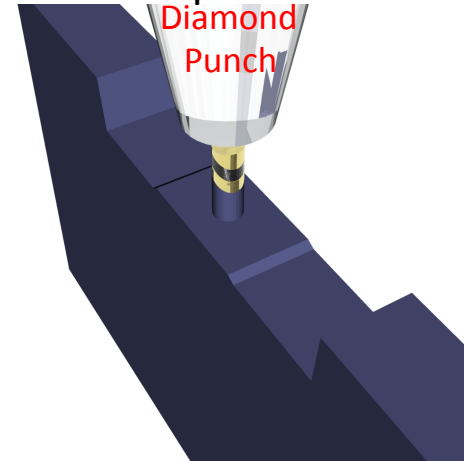
Pre-cut and Grow Films



FIB Sample

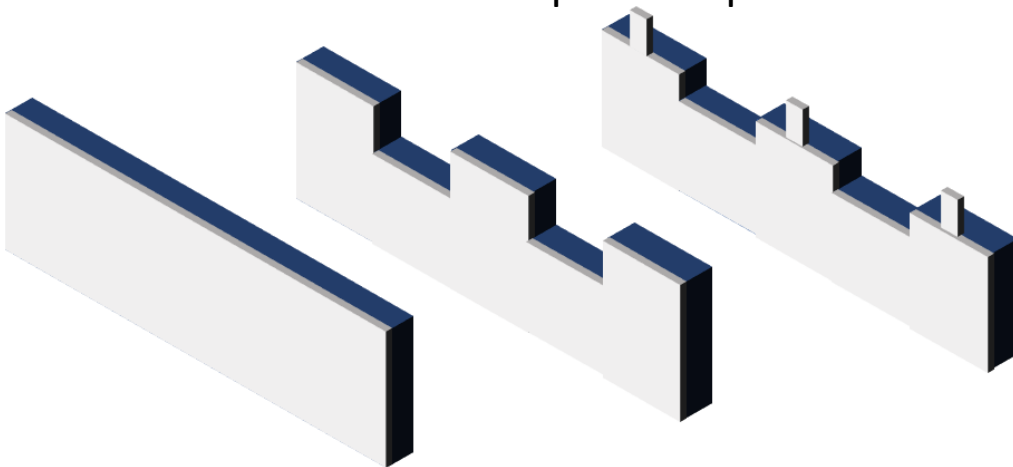


Nanocompression

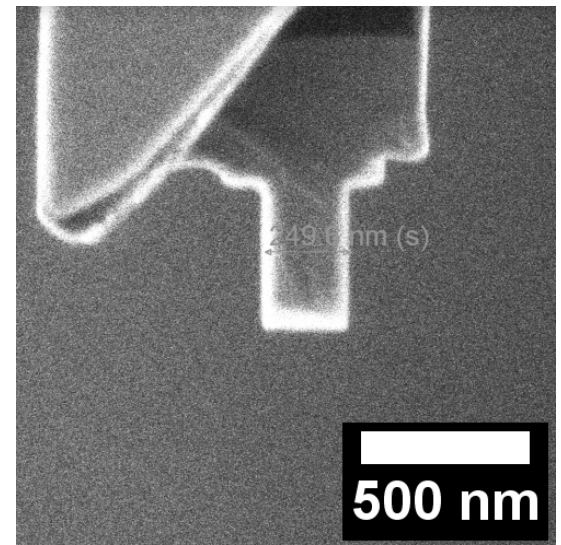


## Ag Samples

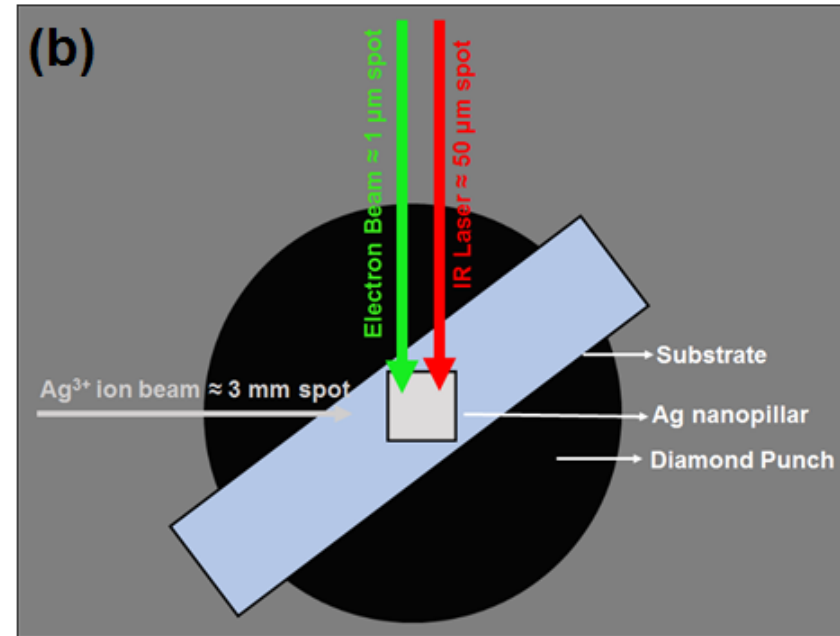
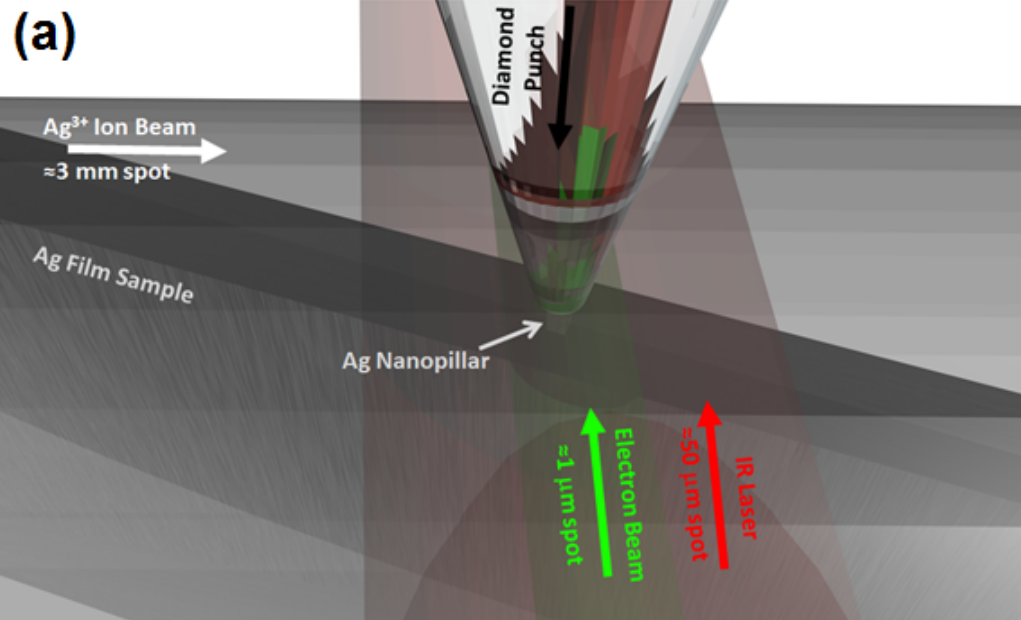
Film on surface



FIB pillars in plane

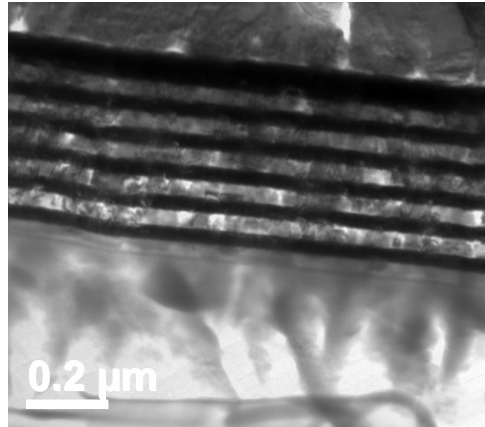


# Loading Geometry

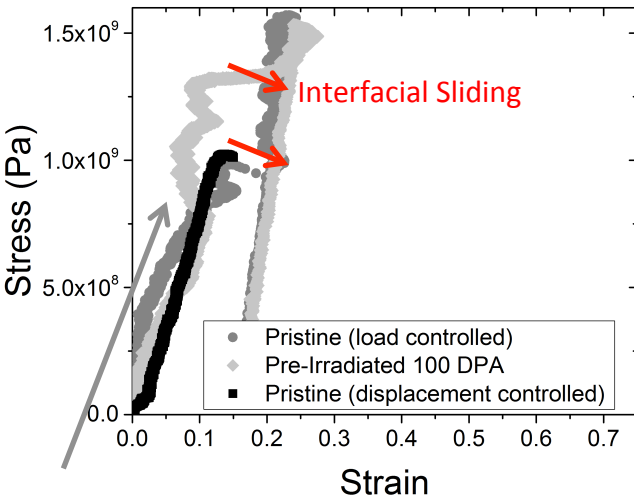


# Irradiation Creep (4 MeV Cu<sup>3+</sup> 10<sup>-2</sup> DPA/s)

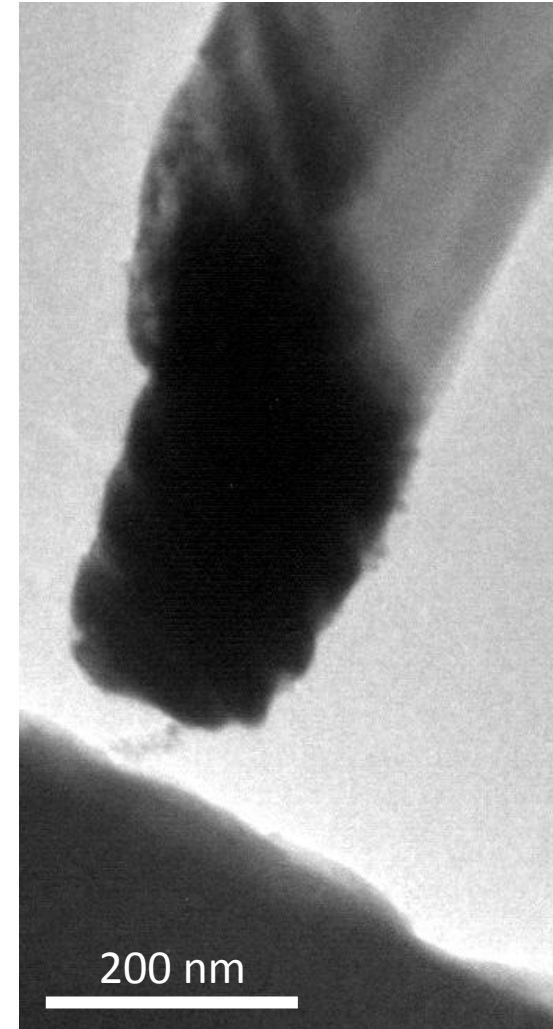
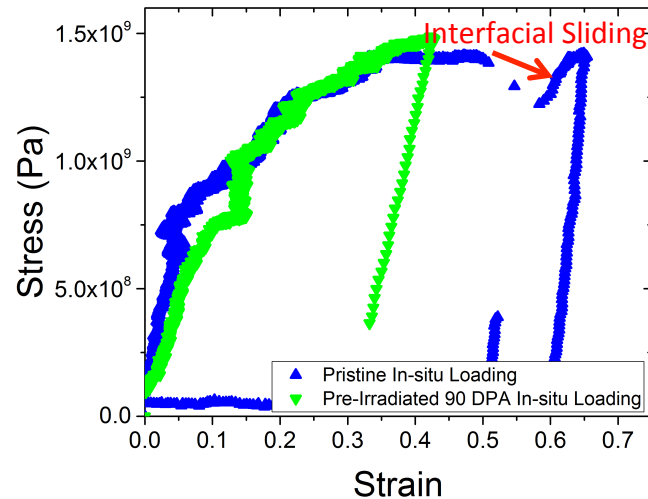
50 nm Cu-W multilayer  
20 Min



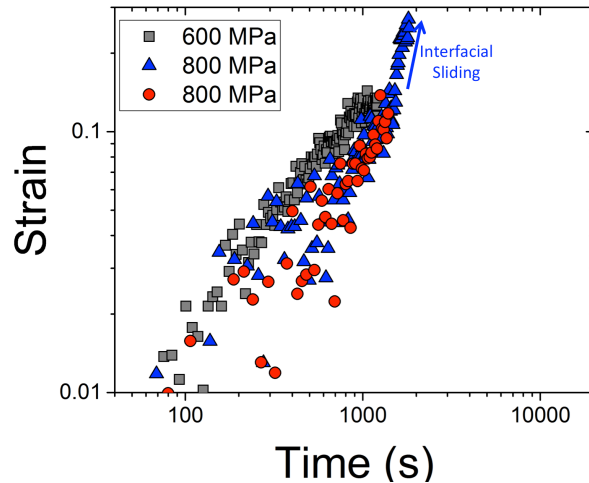
No Irradiation  
(Loading rate 0.6 Mpa s<sup>-1</sup>)



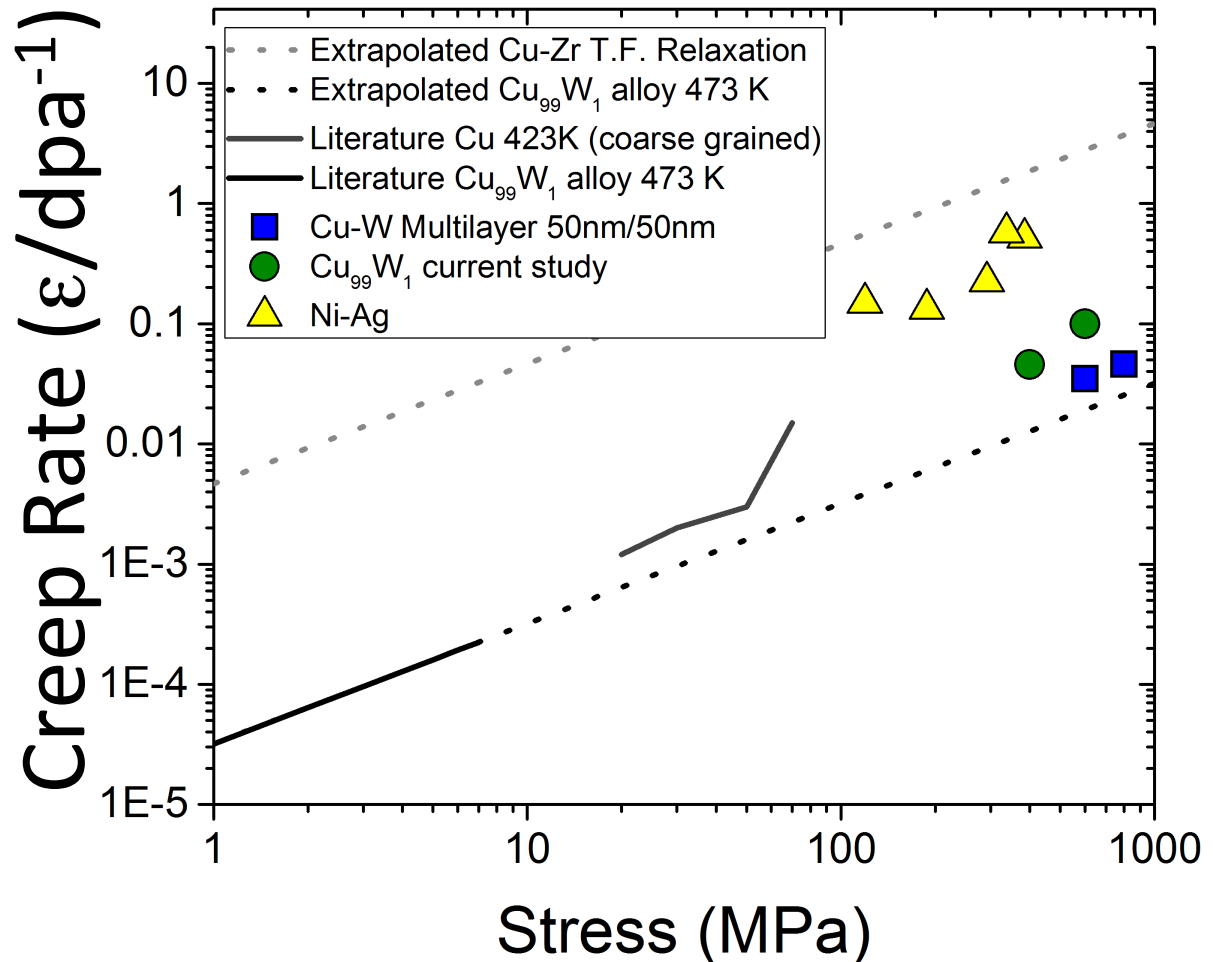
Irradiation Creep  
(Loading rate 0.6 Mpa s<sup>-1</sup>)



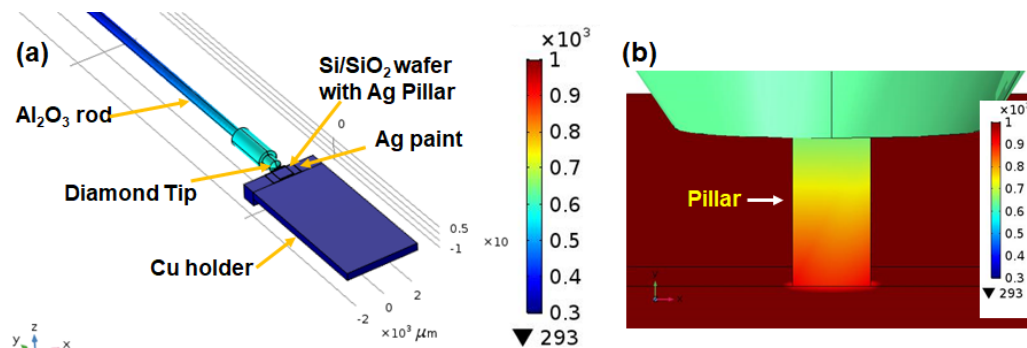
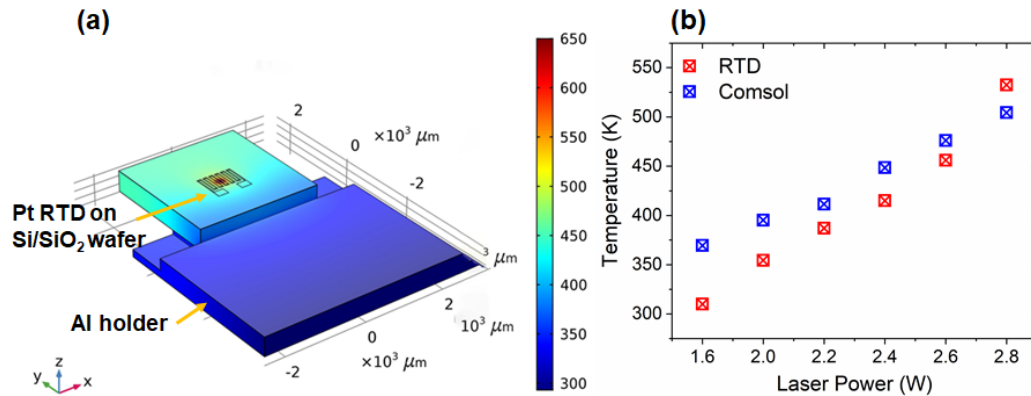
# Comparison of Steady-State Creep Data



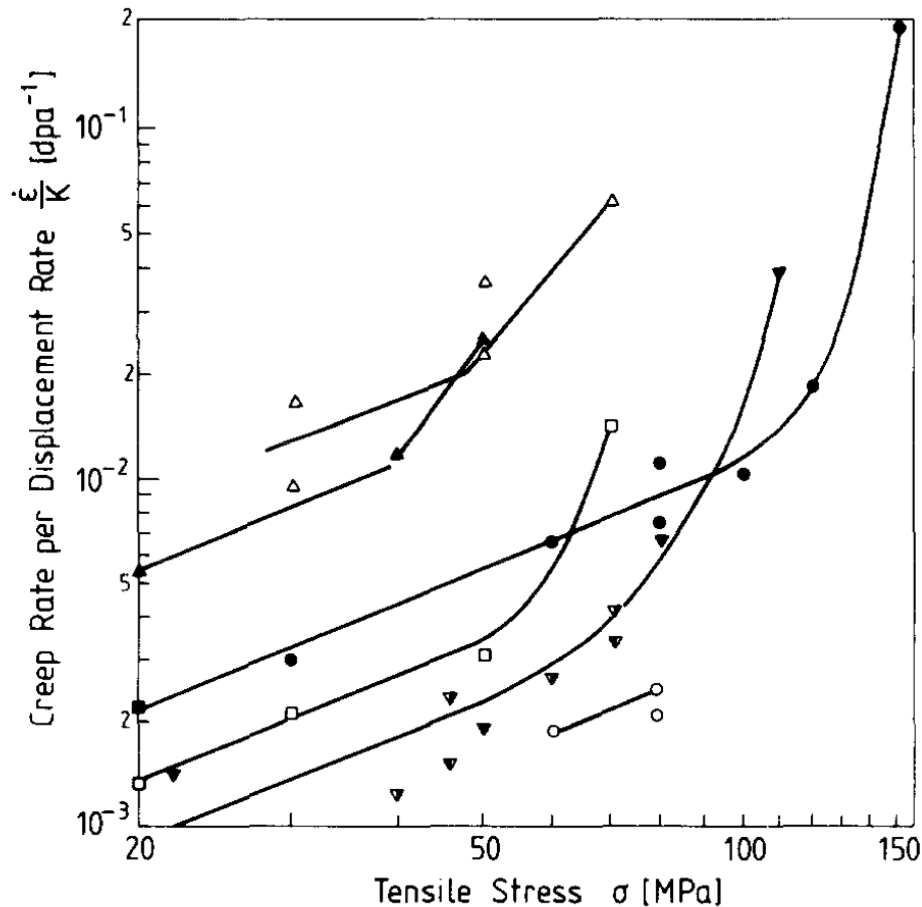
- R.T. IIC rate in nanolaminates significant at  $<10\% \sigma_y$
- IIC rates in Ni-Ag approaching theoretical maximum
- Mechanism for Cu-W in high stress regime likely different than prior low stress measurements



# High Temperature Experiments on Ag: Approximating Temperature Regime



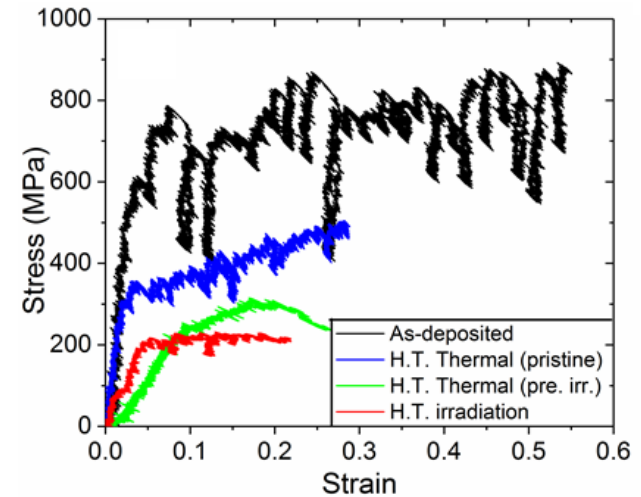
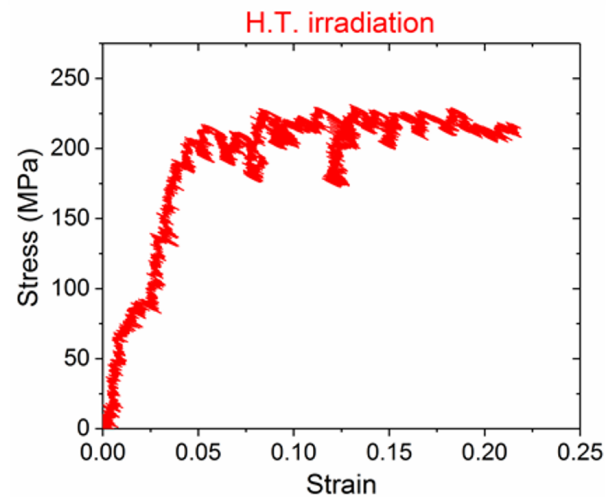
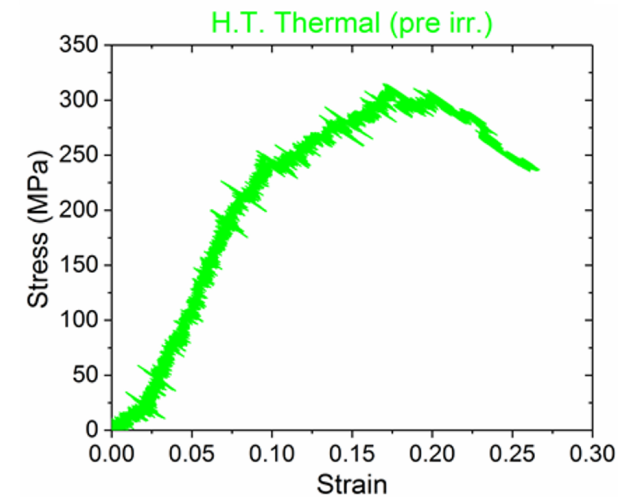
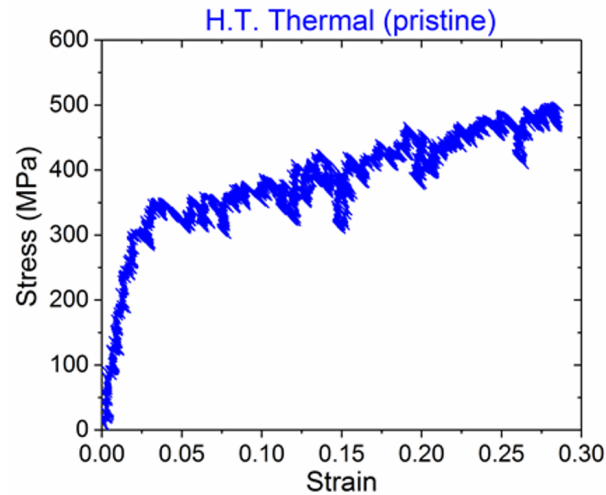
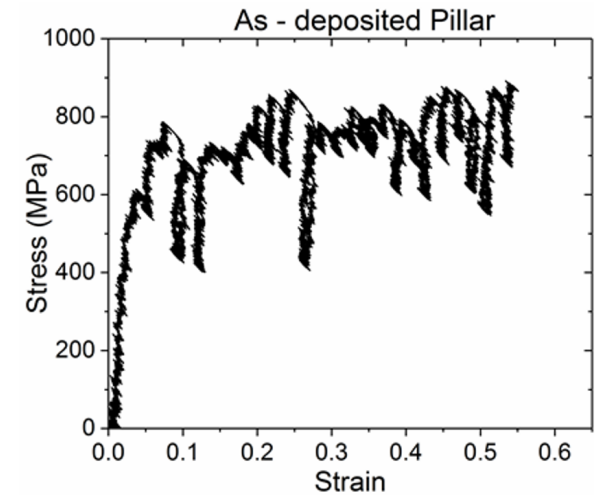
# Prior IIC on Ag



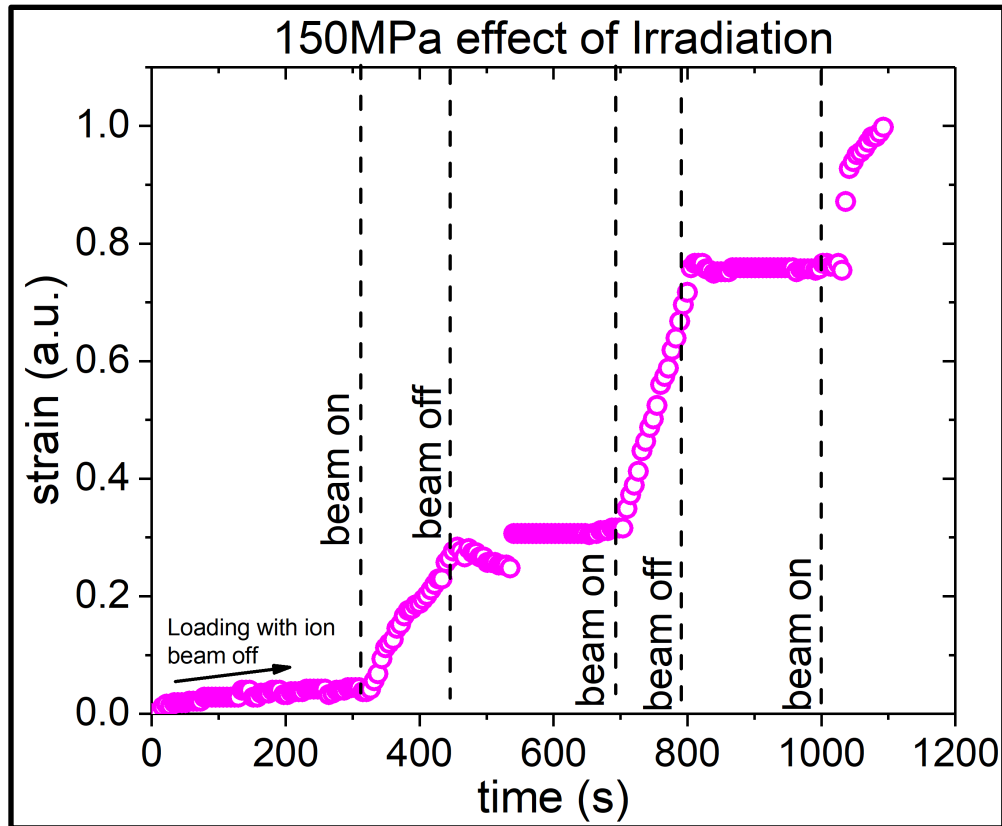
Sink limited regime  
activation energy:  $\approx 0.002$  eV

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 (Δ), 150°C (▲); platinum: 220°C (▼), 240°C (▼), 300°C (▼).

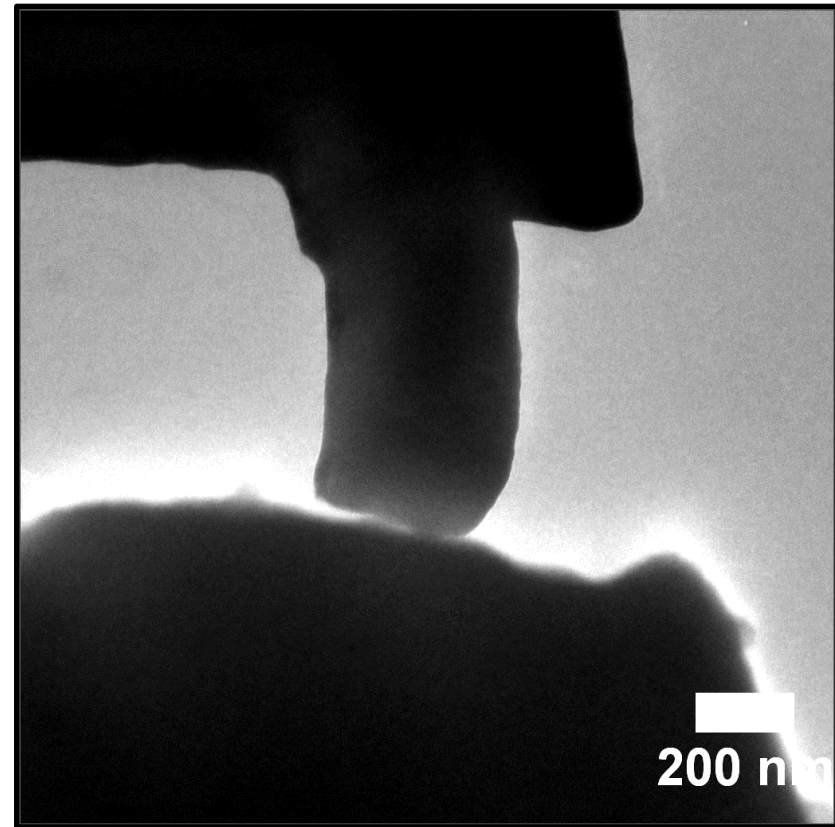
# Stress-Strain: Effect of Irradiation and Temperature



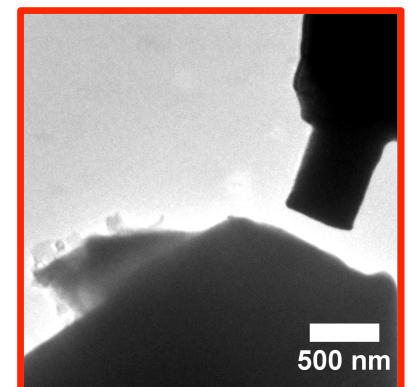
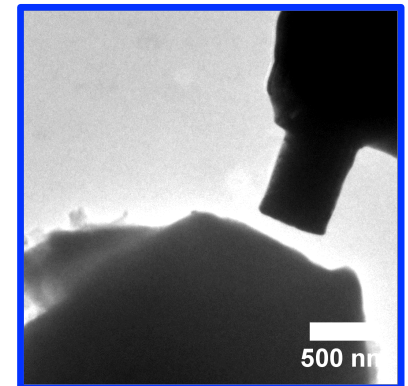
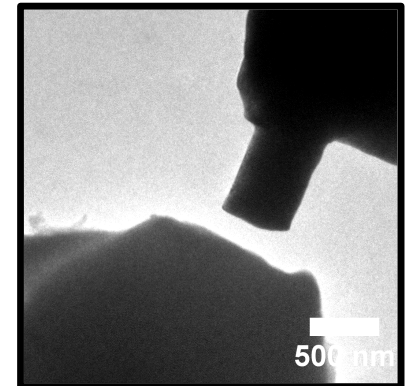
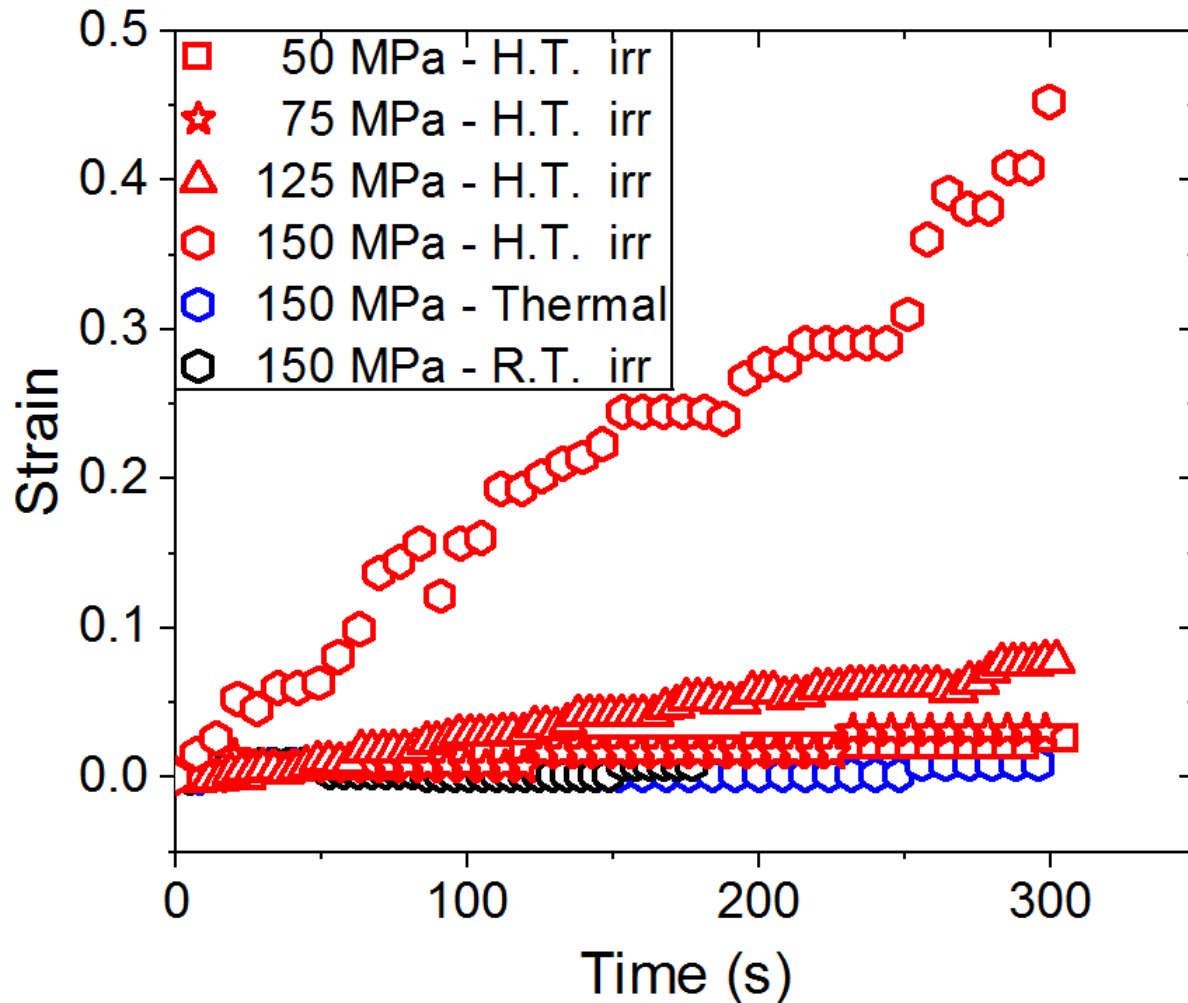
# IIC in Sample with Non-ideal Geometry/ Deformation



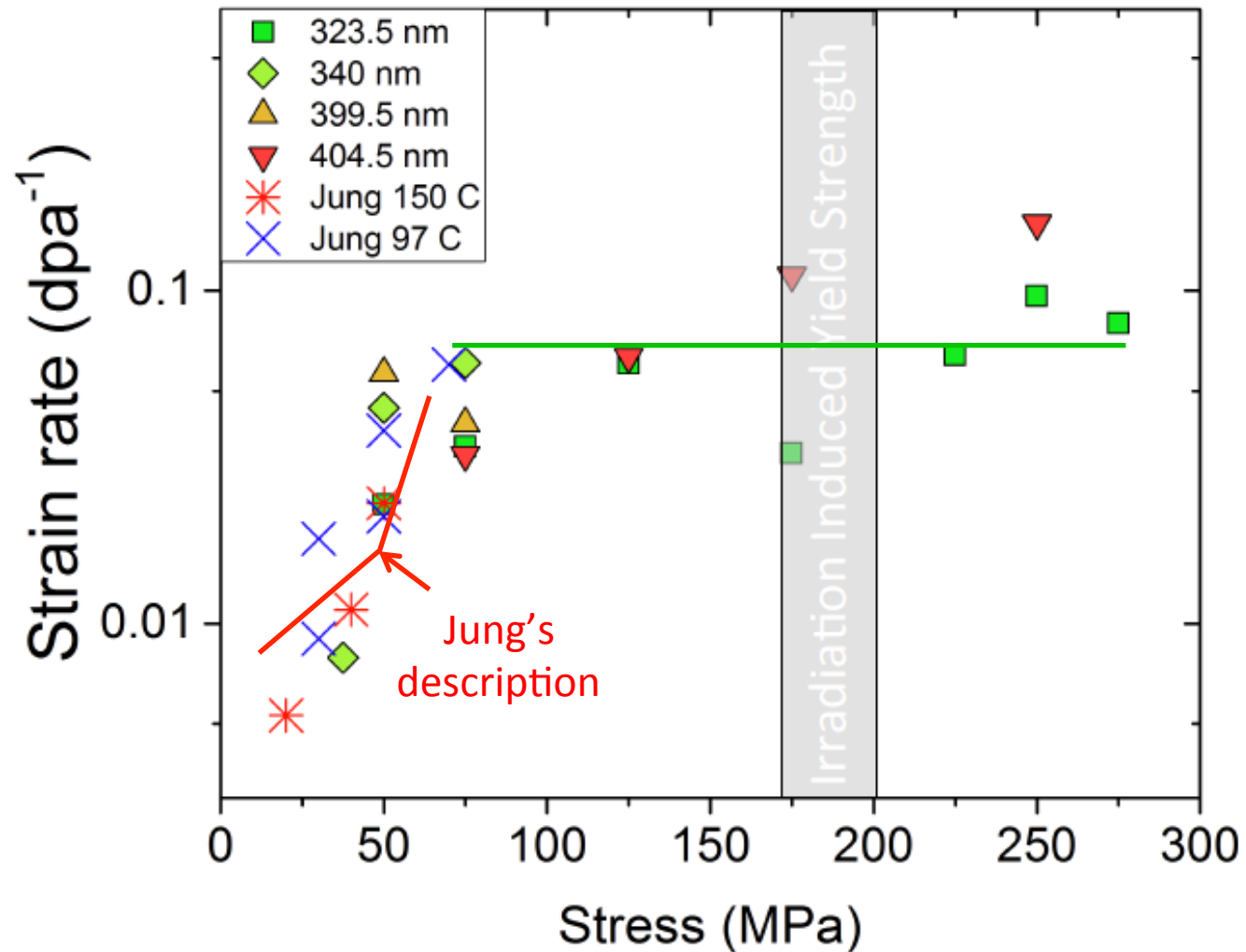
Speed  $\approx 75\times$



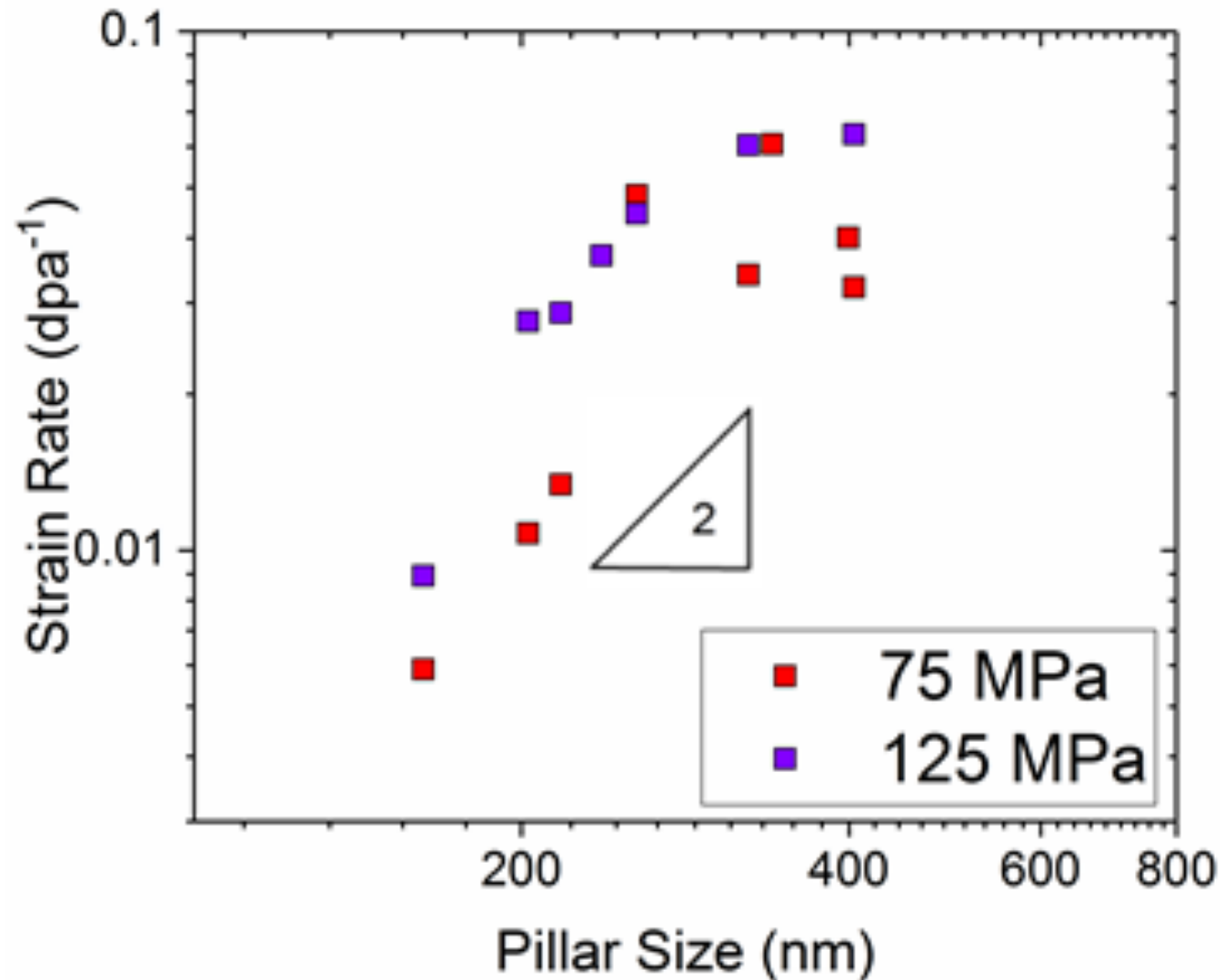
# Steady-State Creep Rates in Ag



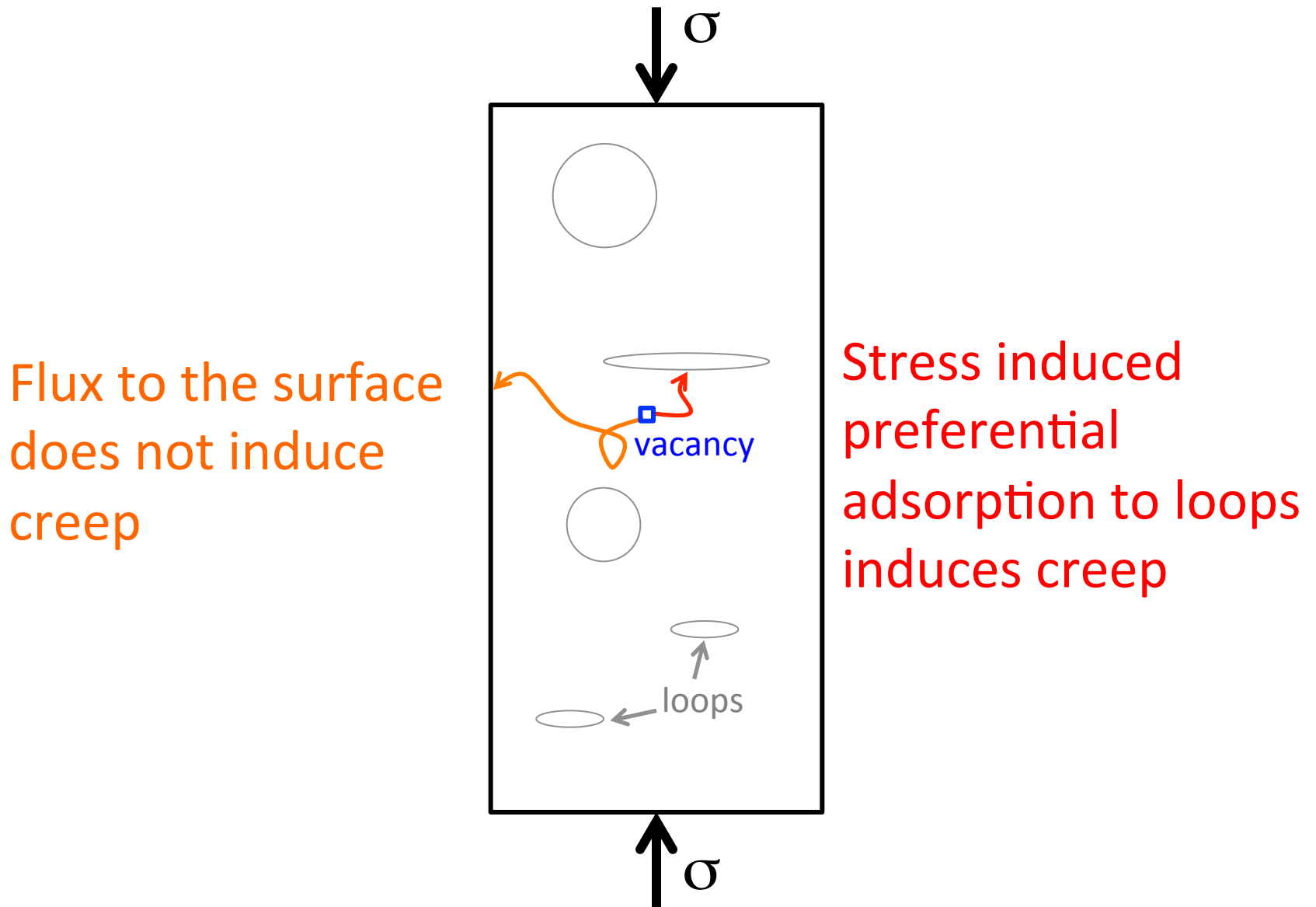
# Effects of Stress on IIC



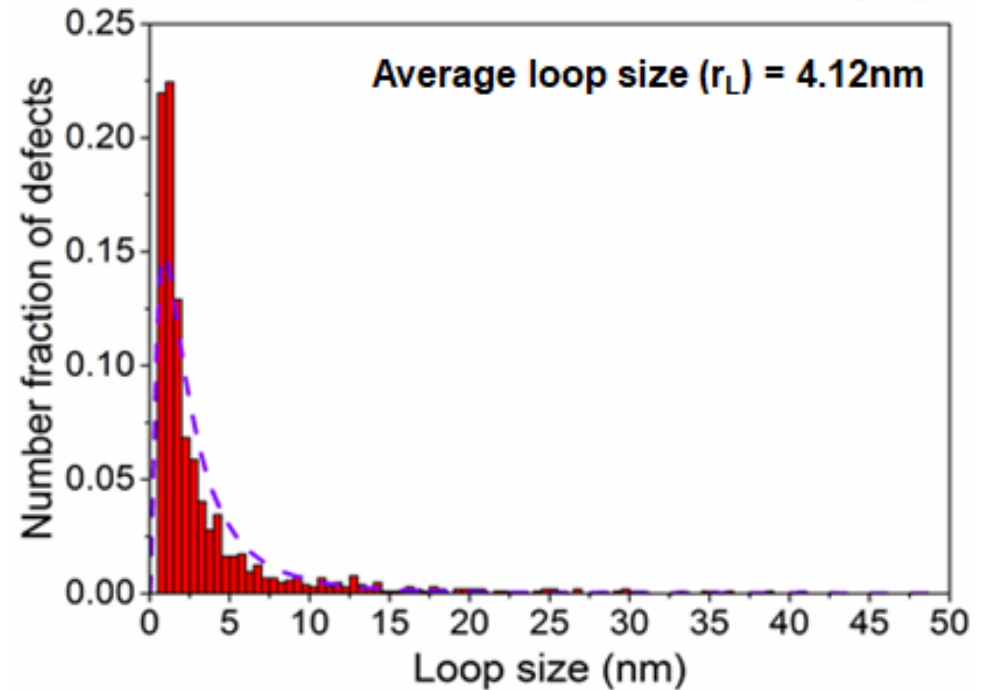
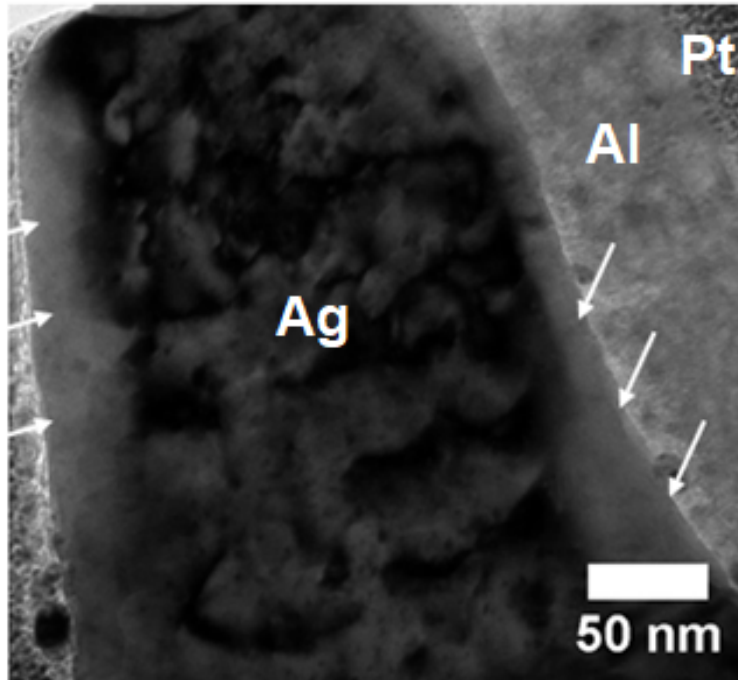
# Effects of Pillar Size on IIC



# Size Effect in Sink Limited Regime

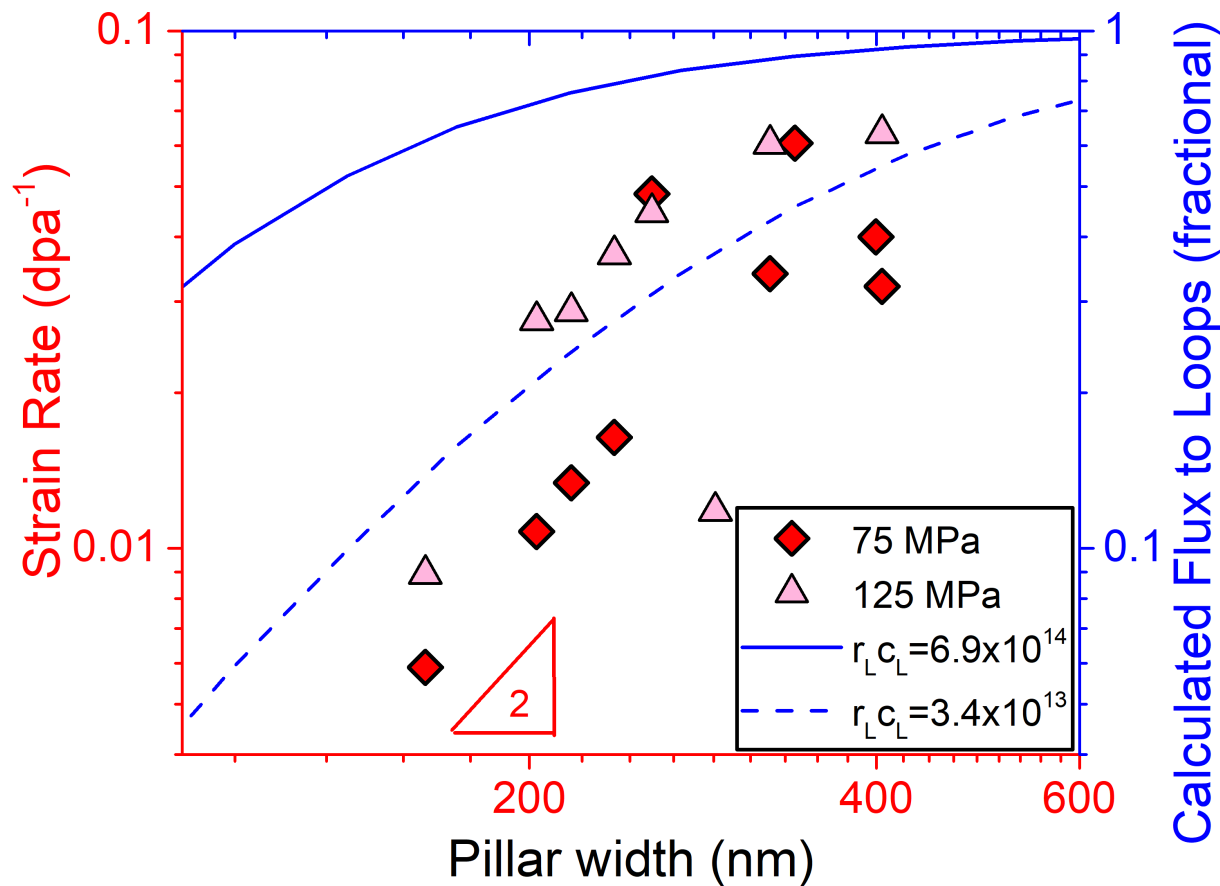


# Competition between Dislocations and Interfaces



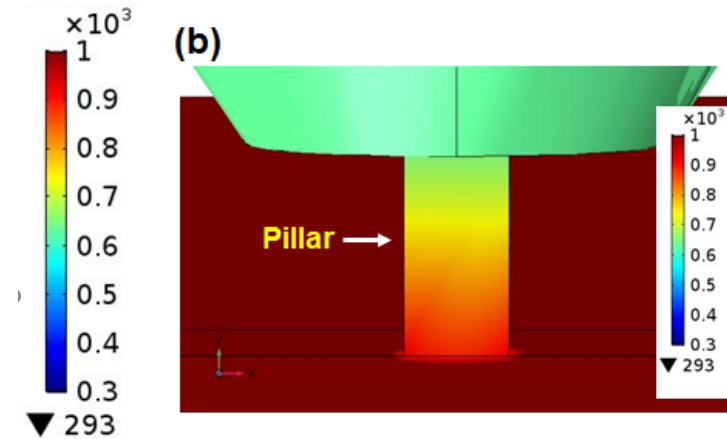
# Fit Predicted from S.S. Rate Equation

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

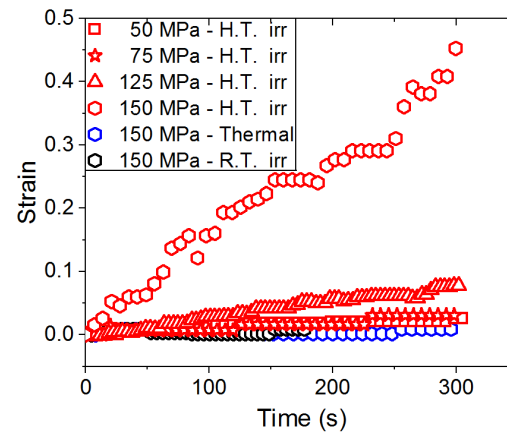


# Remaining Challenges

- Determining temperature and limit the temperature gradient



- Amplifying strain such that is is directly measureable 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