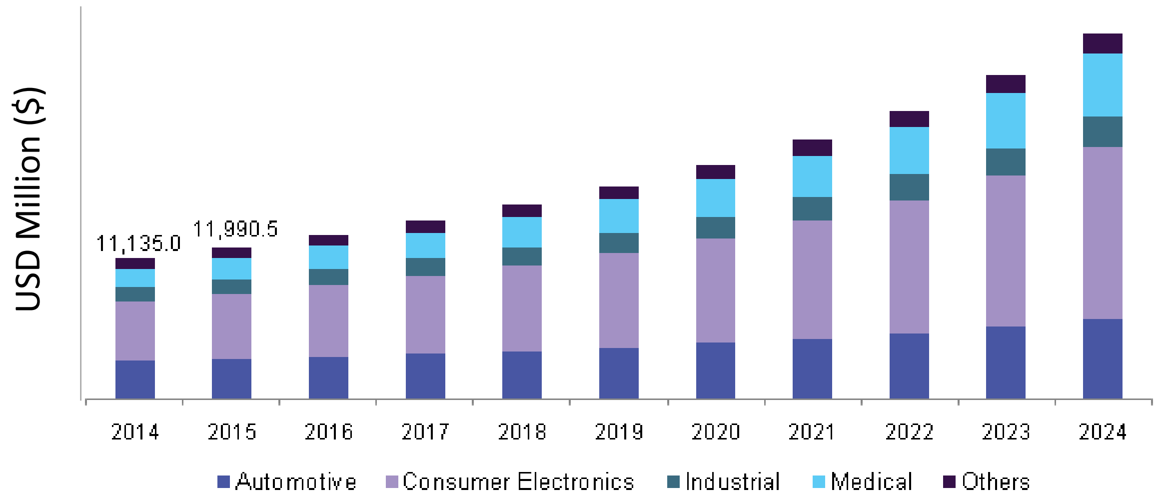
Characterizing Plastic Deformation Mechanisms in Metal Thin Films using *in situ* TEM Nanomechanics

Sandra Stangebye

2023 DOE NNSA SSGF Annual Review

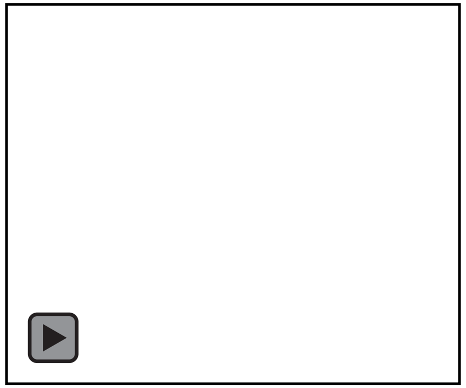
Increased MEMS market drives demand for reliable nanomaterials

MicroElectroMechanical Systems (MEMS)
Global Market



Metallic thin films used for:
- Structural coatings
- Electrical contacts

→ Optimize mechanical properties of metal thin films



*Scale bar: 10 μm

Fine-grained metals pose as viable solution to high-strength & radiation tolerant materials

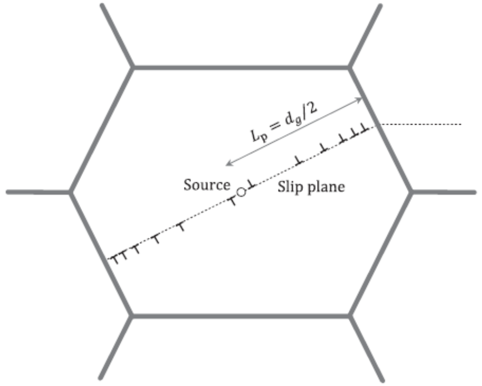
Ultrafine-grained (UFG): 100 nm > *d* > 1μm
Nanocrystalline (NC): *d* < 100 nm

NC & UFG metals exhibit **unique properties** due to the high-volume fraction of grain boundaries (GBs):

Increased Strength

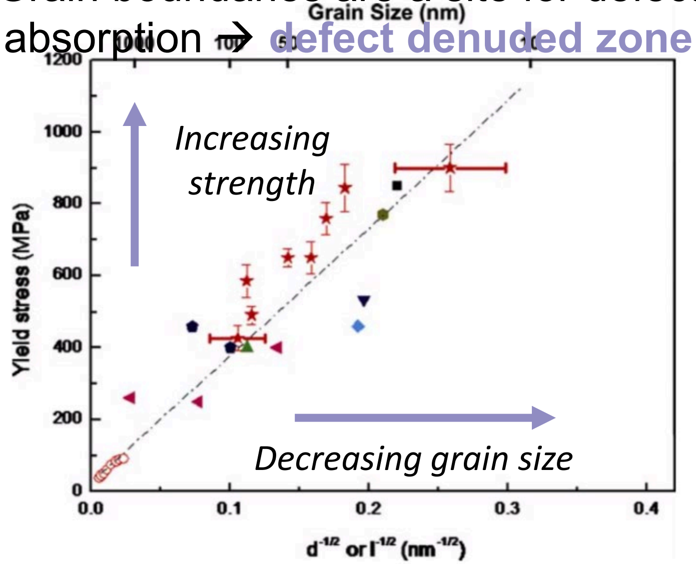
Hall – Petch Law

$$\sigma_y = \sigma_o + \frac{k}{\sqrt{d}}$$



Voyiadjis, George. (2019) *Size Effects in Plasticity: From Macro to Nano.* Academic Press

Grain boundaries are a site for defect absorption → **defect denuded zone**



Li *et al.*, JMPS (2013) 12

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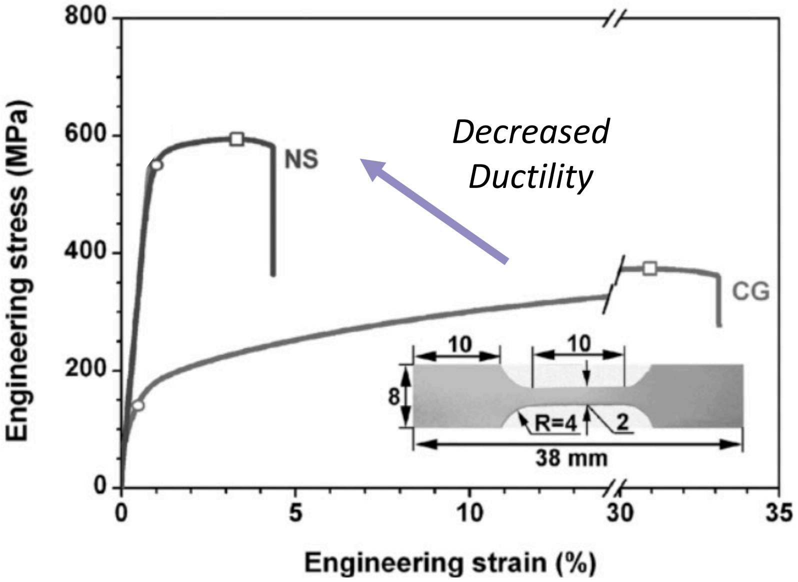
Decreased Ductility



Zhao *et al.*, Adv Mater. 18 (2006) 2280-2283

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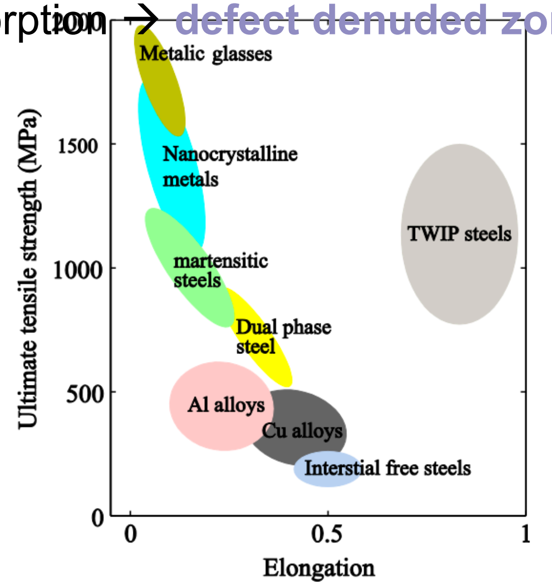

Asaro *et al.*, Acta Mat. 53 (2005) 3369-3382

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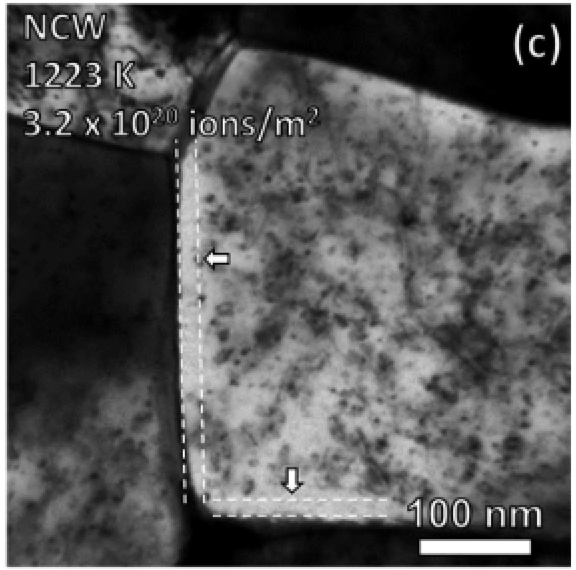
Decreased Ductility

Increased Radiation Tolerance



Enikeev *et al.* Mat. Trans. 60 (2019) 1723-1731

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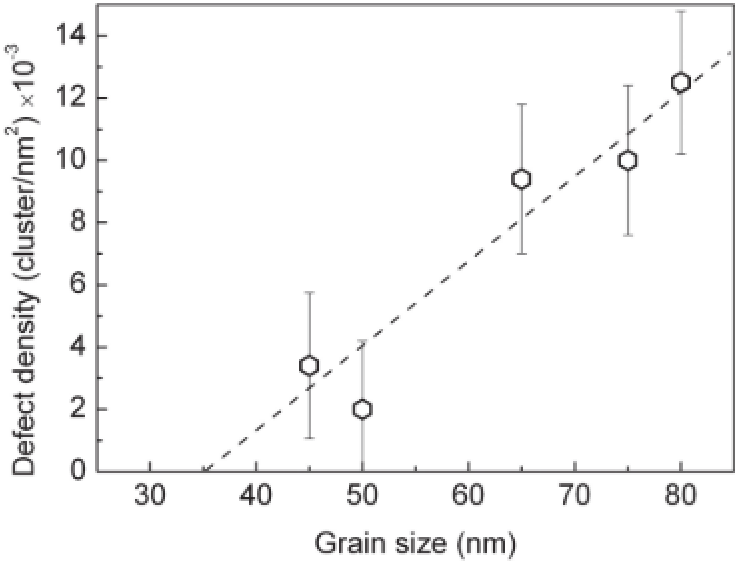
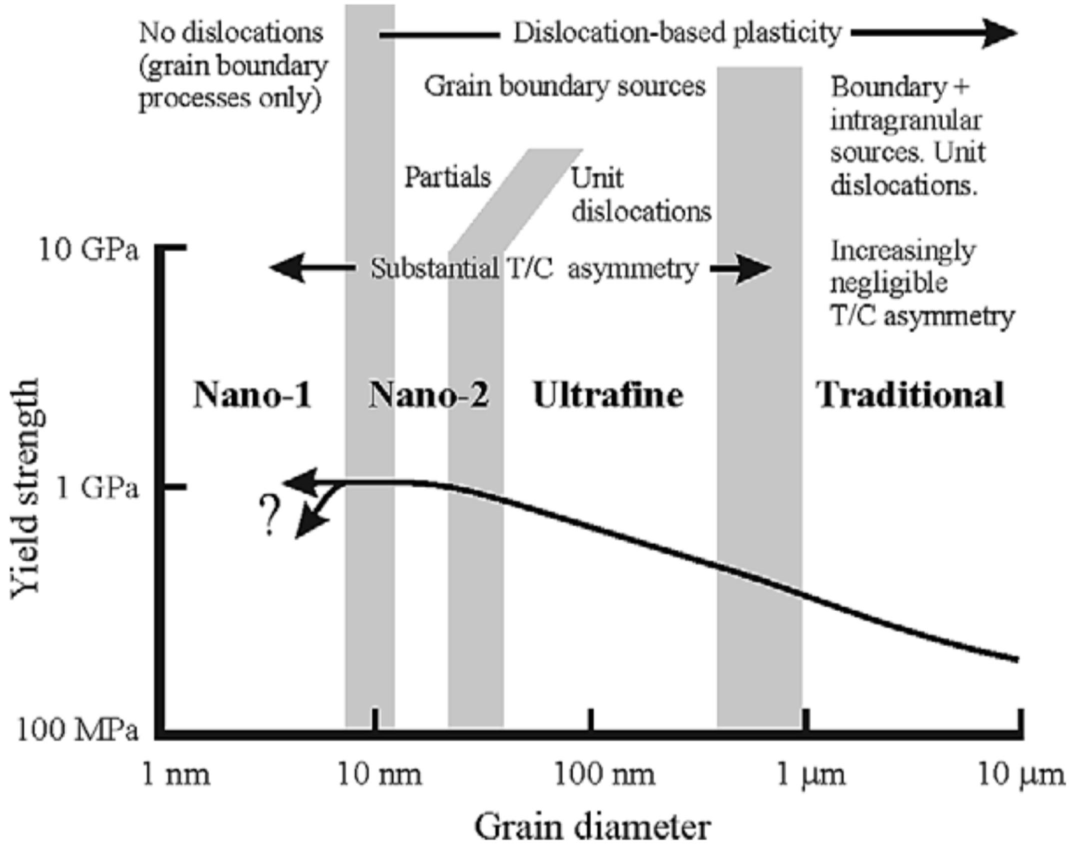


El-Atwani *et al.* Phys. Rev. Mat. 2 (2018) 113604

Deformation Mechanisms: a function of grain size



Cheng *et al.* Acta Mat. 51 (2003) 4505- 4518

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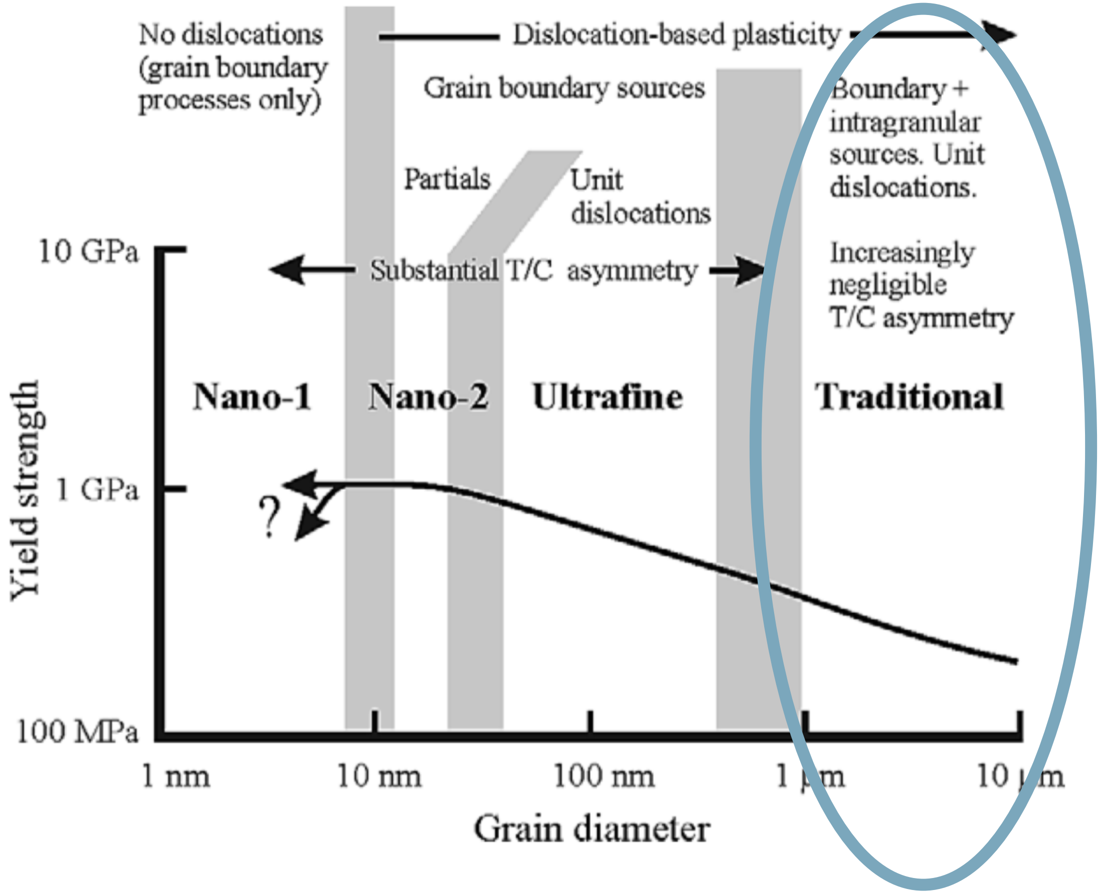
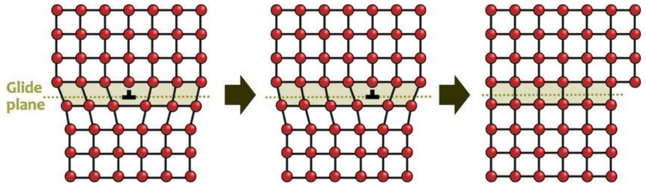


Cheng *et al.* Acta Mat. 51 (2003) 4505- 4518

Traditional grain size regime

Plastic deformation controlled by dislocation glide

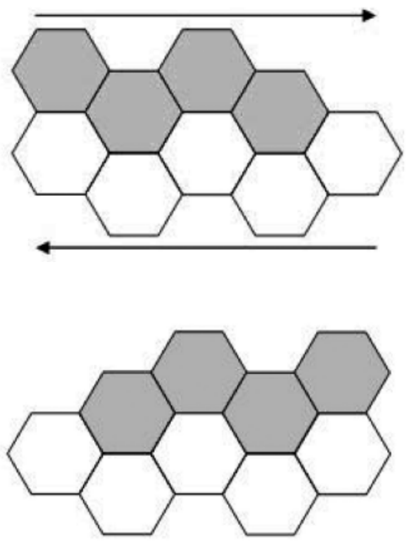


Boioli *et al*. Mult. Mod. of Mantle Rheology (2018)

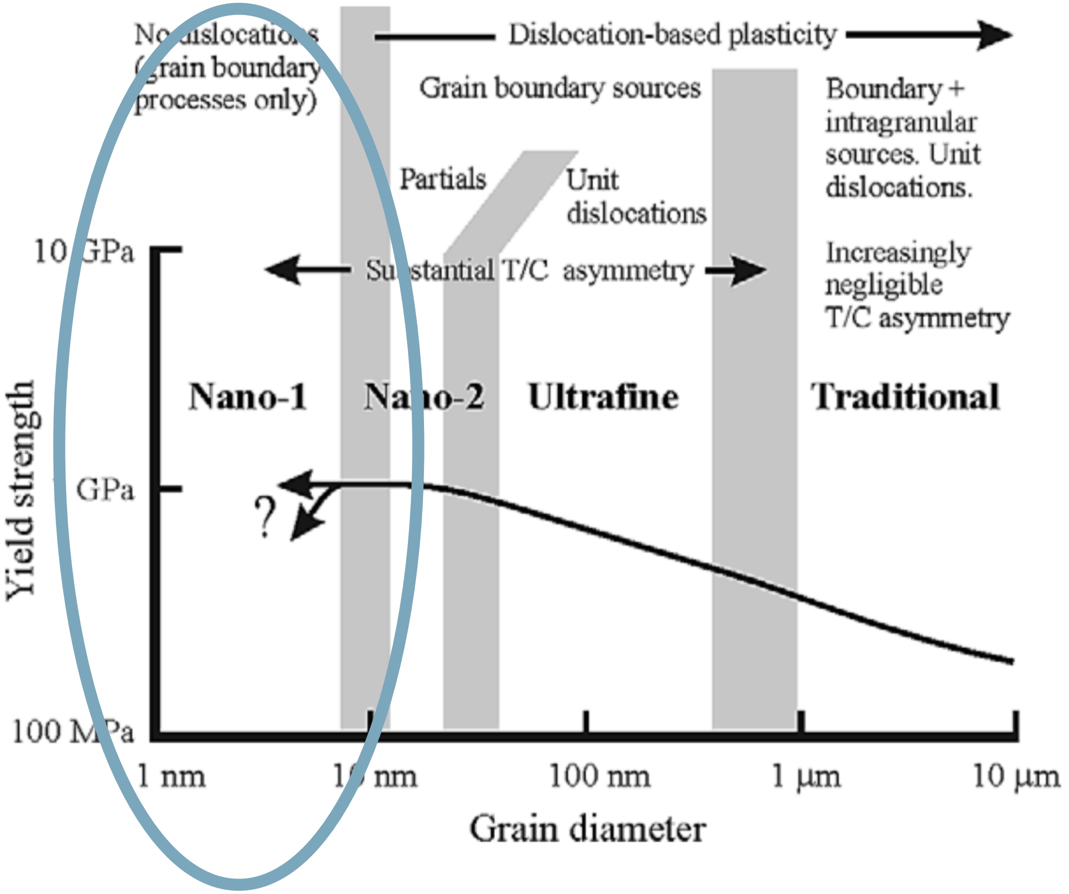
Deformation Mechanisms: a function of grain size

Grain boundary- based deformation:

- GB sliding
- Grain rotation
- GB migration



Meyers *et al.* JOM (2006) 41-48



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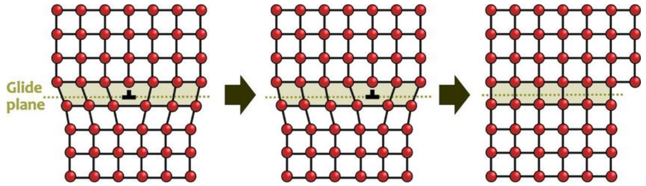

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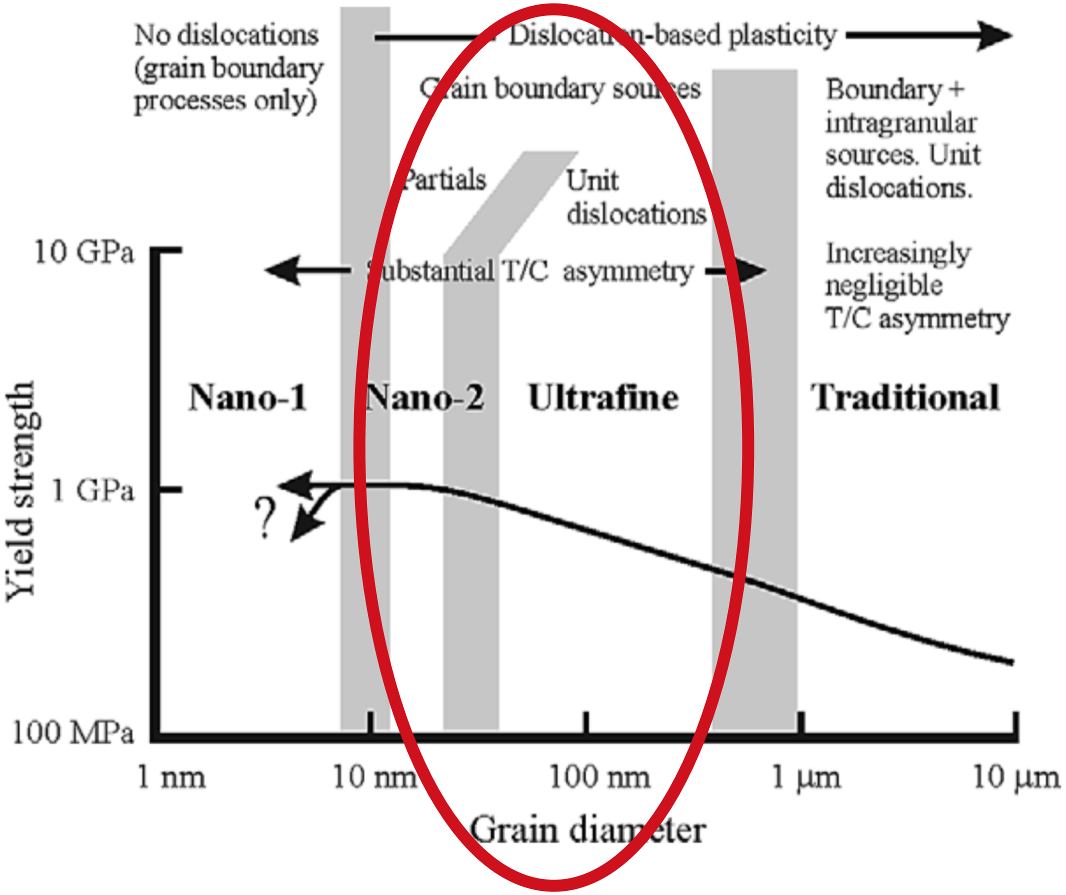
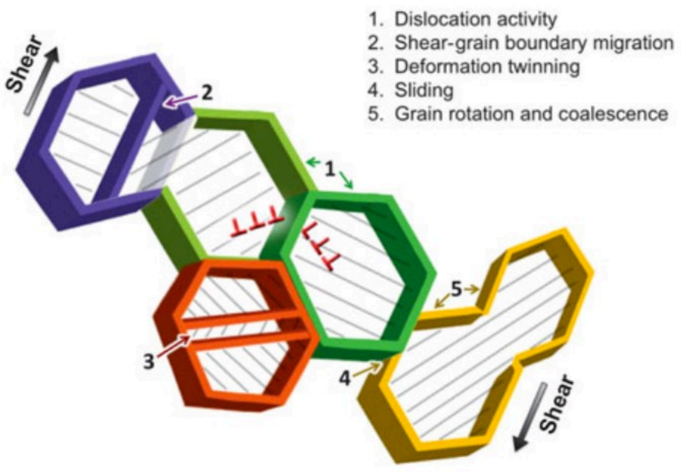


Cheng *et al.* Acta Mat. 51 (2003) 4505- 4518

Deformation mechanisms in UFG grain size regime are not well understood

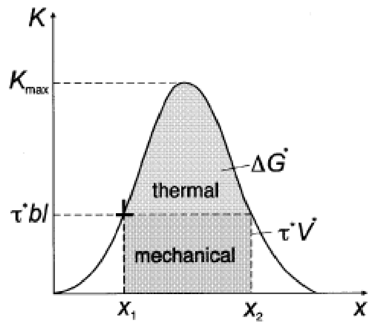
- mixture of dislocation- and GB-based mechanisms occur simultaneously
- GB sources & sinks for dislocations



Yu *et al.* MRS Bulletin 40 (2015) 62-48

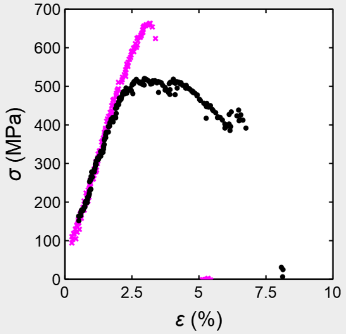
Three approaches required to fully characterize deformation mechanisms

Quantification of Mechanical Properties



Activation Volume V* is a signature parameter associated with deformation mechanism

Diffusion-based mechanism ~ 1

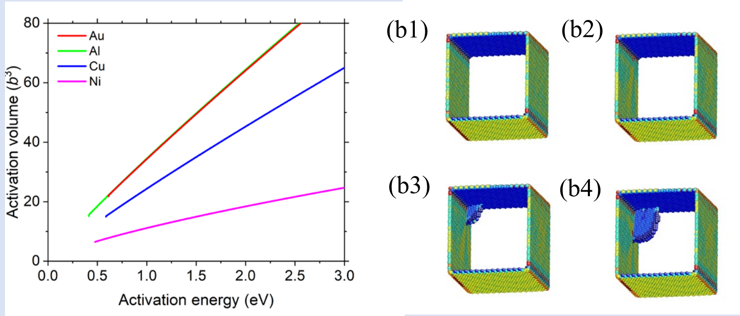


Microstructural Characterization & TEM Observations

Direct observation of dislocation and grain boundary interactions (deformation mechanisms) during straining.

Also used to characterize defect content, grain size, texture, etc.

Atomistic modeling



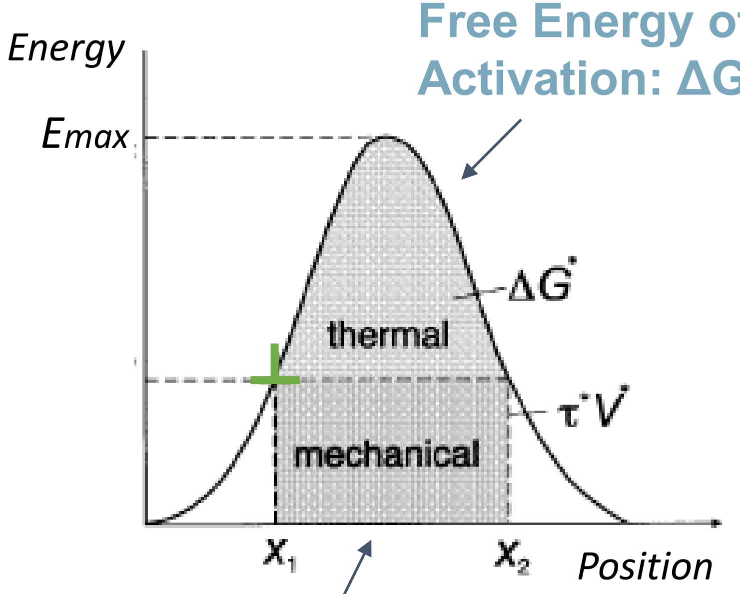
Atomistic models performed on individual mechanisms to determine the expected activation volume values. Compare these with experimentally determined to conclude on the dominant mechanisms.

Rate controlling plastic deformation mechanisms

Deformation mechanisms can be characterized by Activation Volume V^*

Activation volume is a **signature parameter** associated with dislocation mechanisms

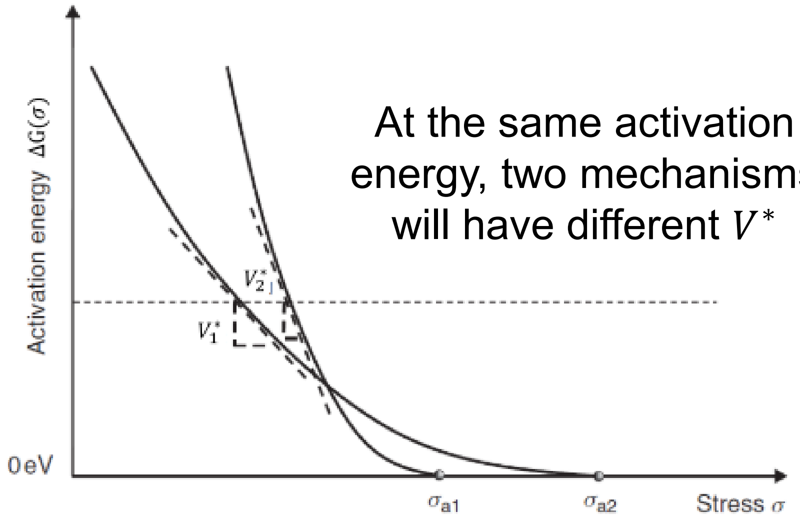
Free Energy of Activation: ΔG*



Mechanical work done by applied load

$$\dot{\gamma}_p = \rho_m b v$$

$$V^* = -\frac{\partial \Delta G^*}{\partial \tau^*}$$



At the same activation energy, two mechanisms will have different V^*

Zhu *et al.* Nano and Cell Mechanics: Fundamentals and Frontiers (2013) 313- 338

Atomistic models can estimate V* for individual mechanisms:

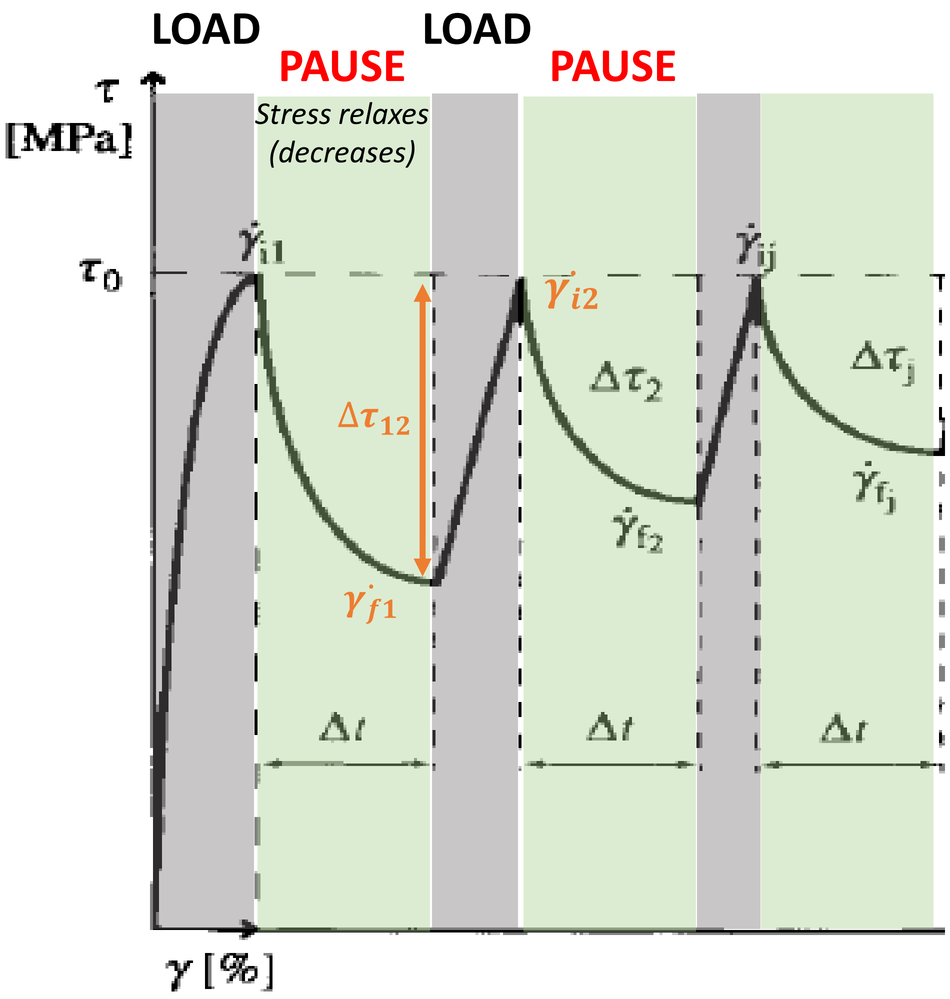
Typical V^* values:

- Diffusion: $\sim 0.1 - 1b^3$
- Cross slip: ~$50b^3$
- Dislocation forest interaction: ~$1000b^3$

(b is Burgers vector)

Introduction to Dislocations, Hull and Bacon (2001)

Activation Volume V^* measured experimentally by performing repeated stress-relaxation experiment



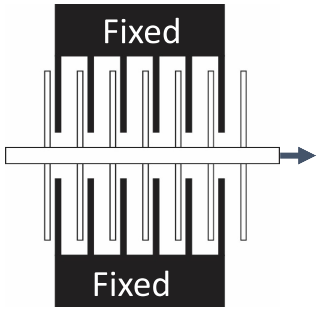
$$\dot{\gamma}_p = \rho_m b v$$

$$V^* = -\frac{\partial \Delta G^*}{\partial \tau^*}$$

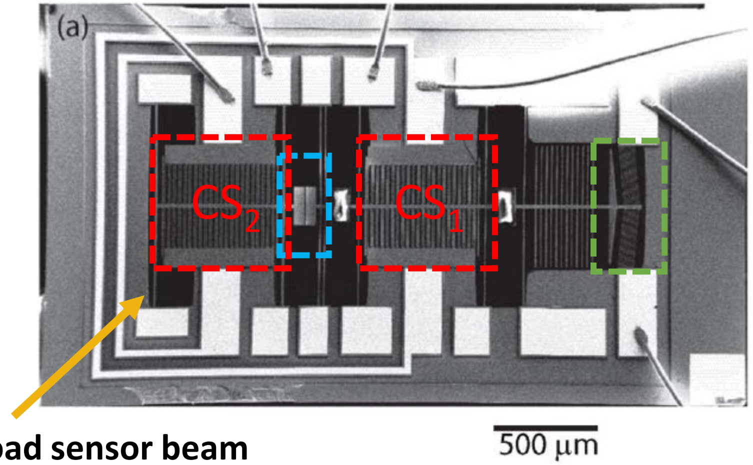
$$V^* = kT\frac{\ln(\dot{\gamma_{i2}}/\dot{\gamma_{f1}})}{\Delta\tau_{12}}$$

Working parameters of MEMS devices used to conduct experiments

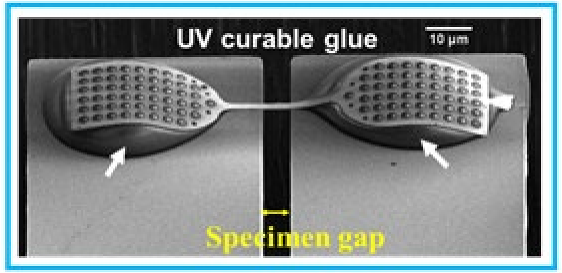
Capacitive displacement sensors

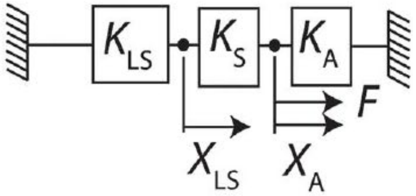


(measuring X_A & X_{LS})

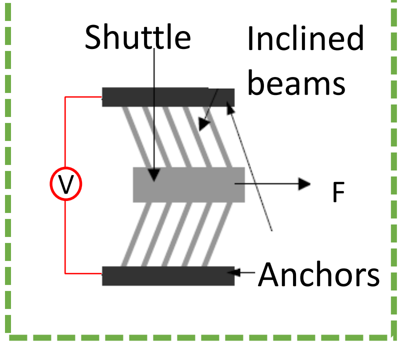


Specimen gap





Thermal actuator



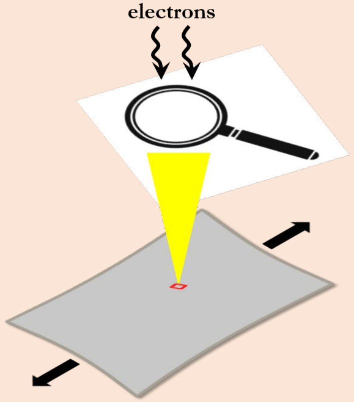
$$F = K_{LS} * X_{LS}$$

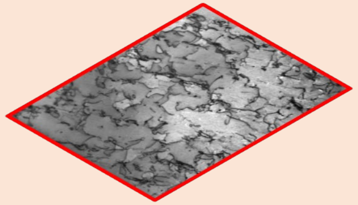
$$X_S = X_A - X_{LS}$$

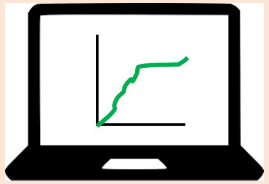
Stress σ

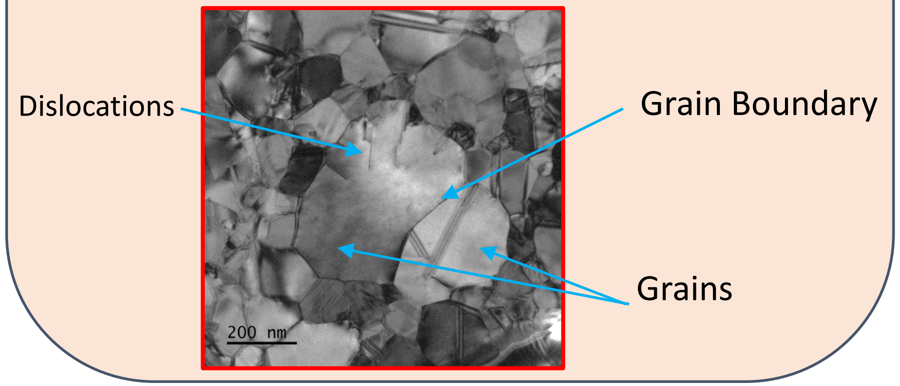
Strain ε

Transmission Electron Microscope

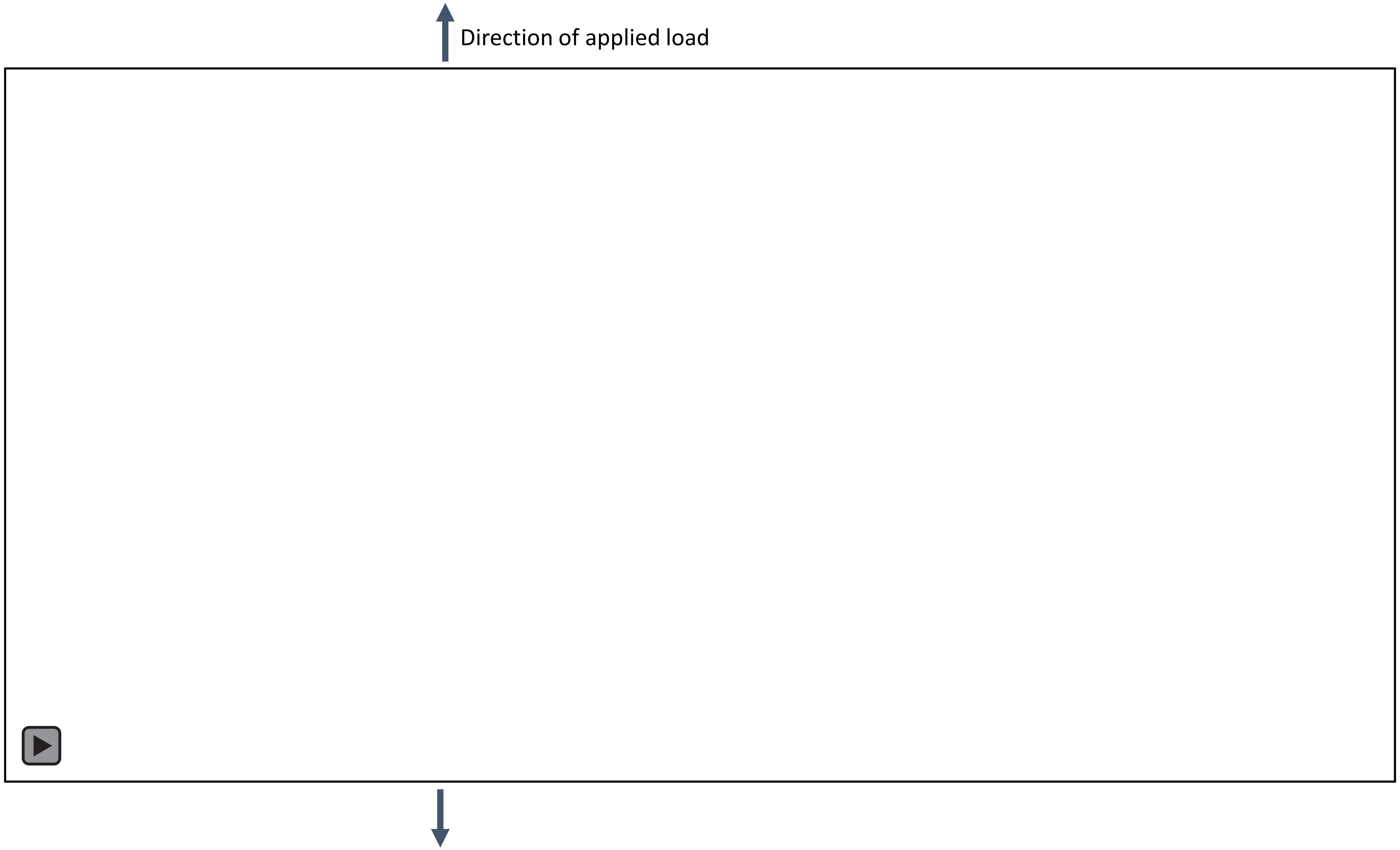






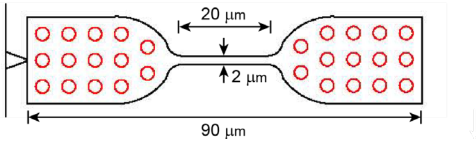


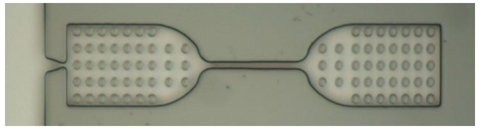
in situ straining and mechanical testing data



Specimen fabrication cleanroom process flow

Optical Lithography

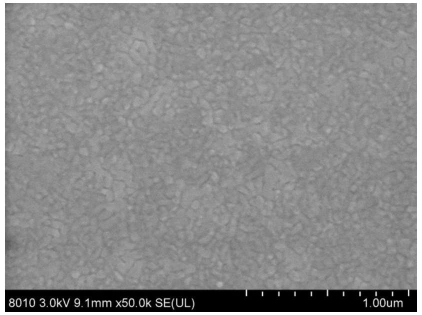




Deposition + Lift Off

E-beam evaporation

Thickness ~ 100-200 nm



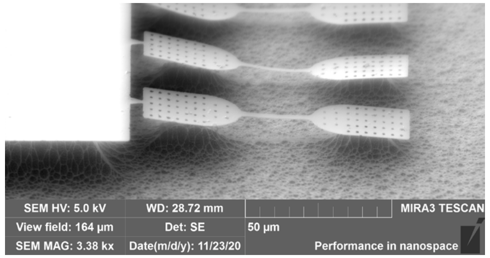
Irradiation

A portion of Au specimens at the Sandia National Laboratory IBL facility

Irradiation parameters:
2.8 MeV Au+
ionsfluence 5.5×10^{13} ions/cm^2 ~1 dpa

Release

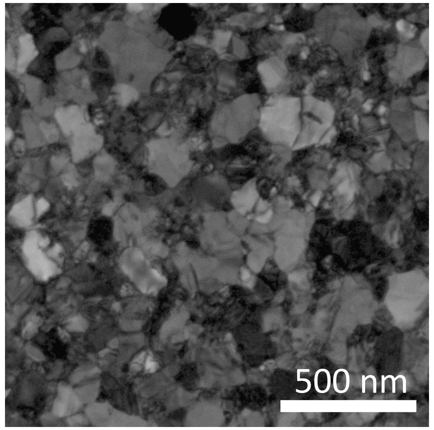
Free-standing thin films



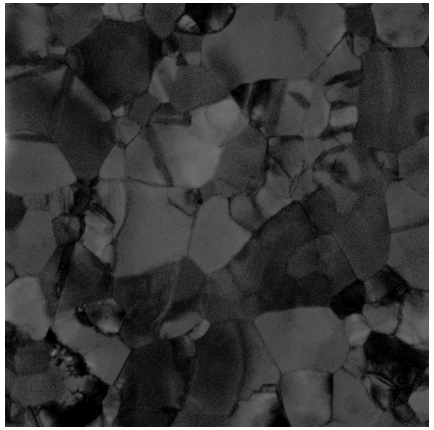
Specimen fabrication cleanroom process flow

Optical Lithography → Deposition + Lift Off → Irradiation → Release

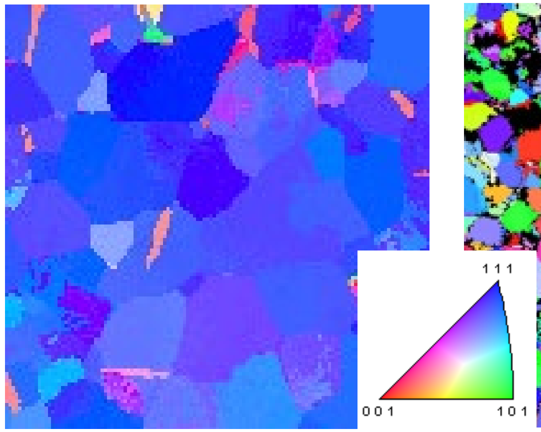
Small grains



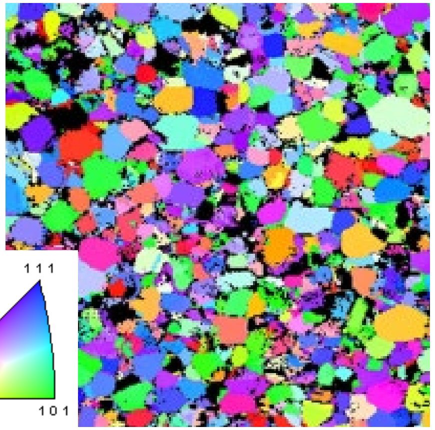
Large grains



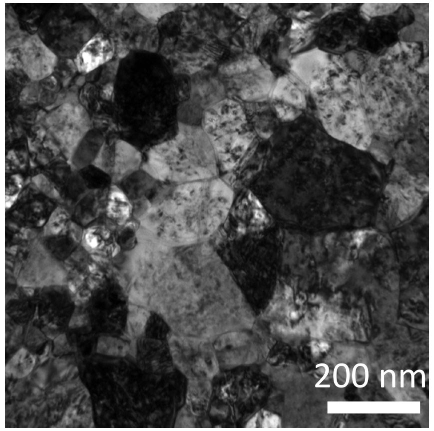
Textured



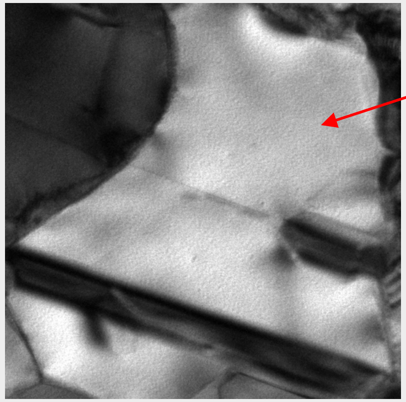
Non-textured



Irradiated

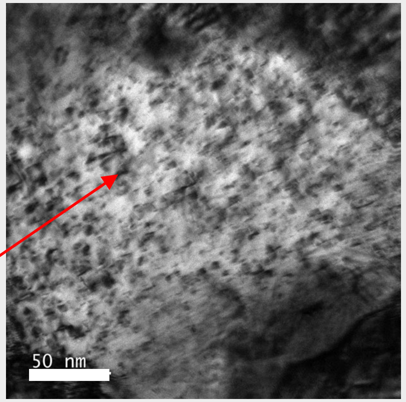


Characterizing and comparing behavior of as-deposited (non-irradiated) and irradiated ultrafine-grained gold



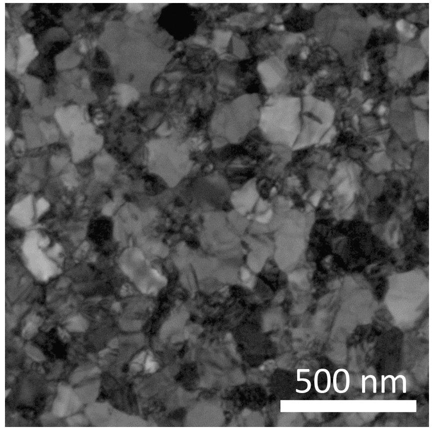
Few defects

Many radiation induced defects

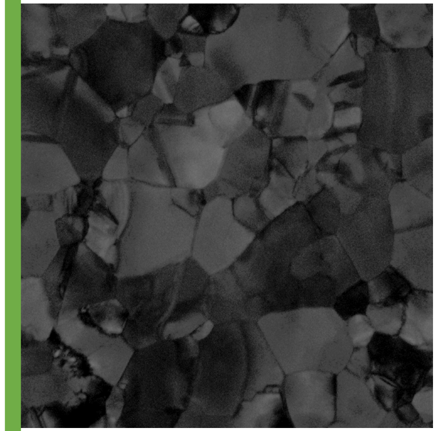


Irradiation parameters:
- 2.8 MeV Au+
- ~1 dpa

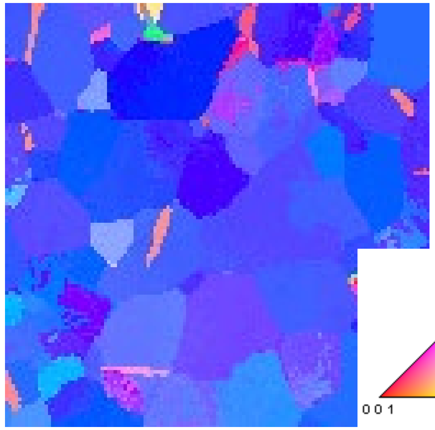
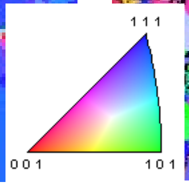
Small grains



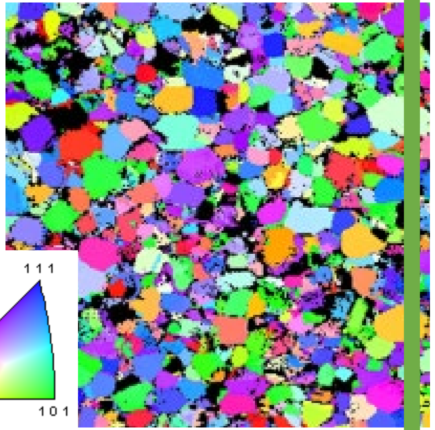
Large grains



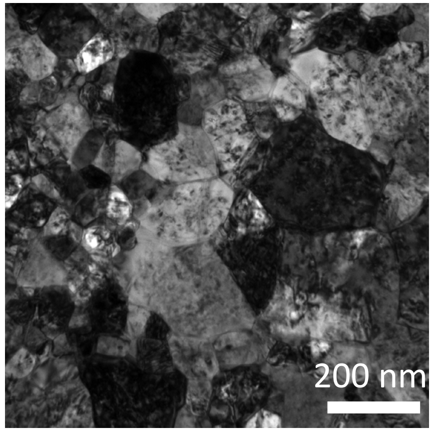
Textured

Non-textured

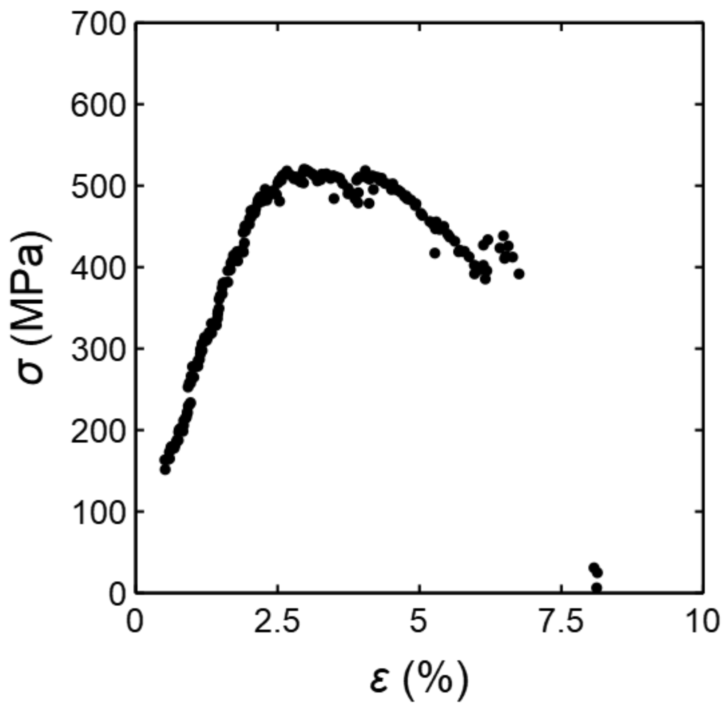


Irradiated

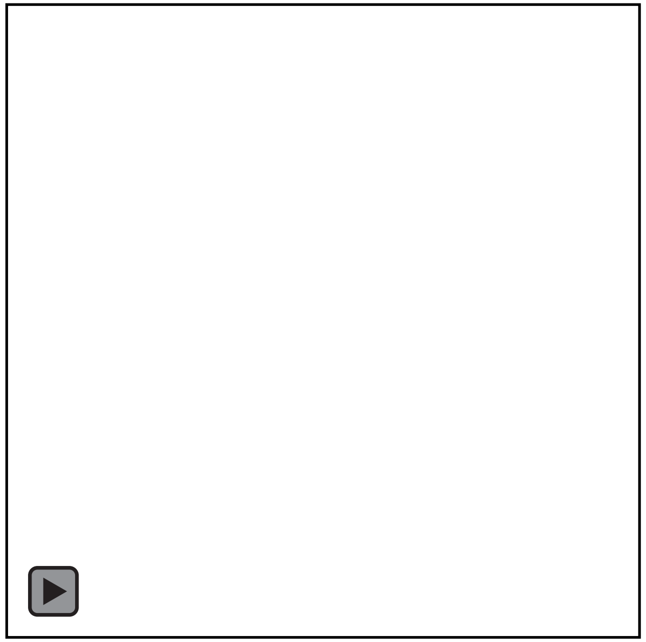


As-Deposited Au: Variety of deformation mechanisms active

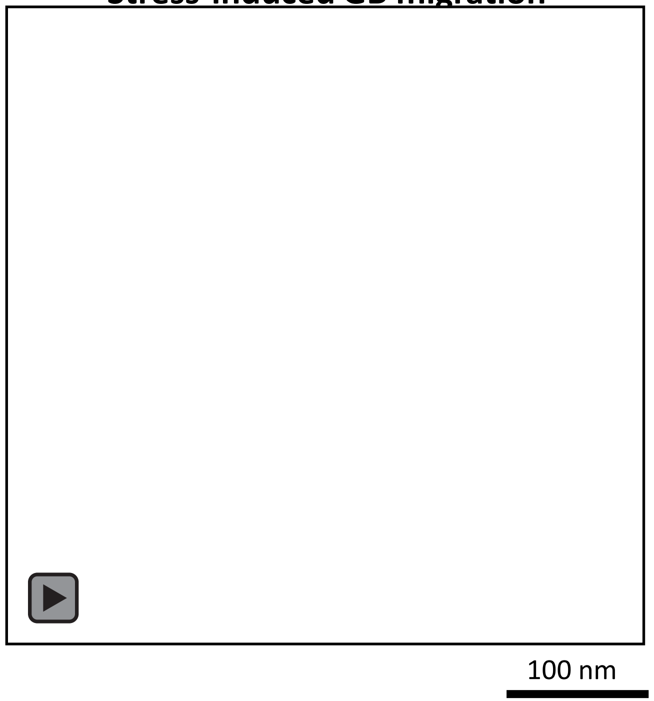
Monotonic response



Dislocation emission & absorption at GB



Stress-induced GB migration

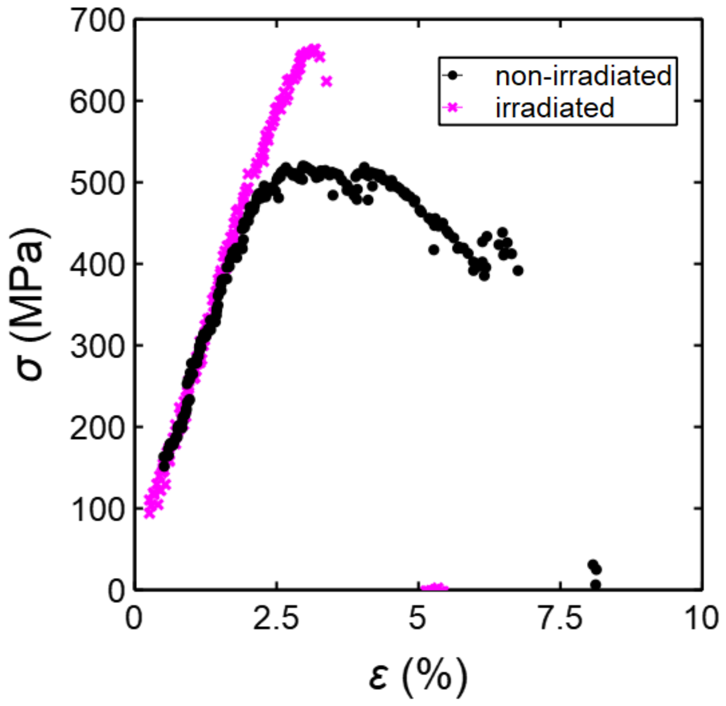


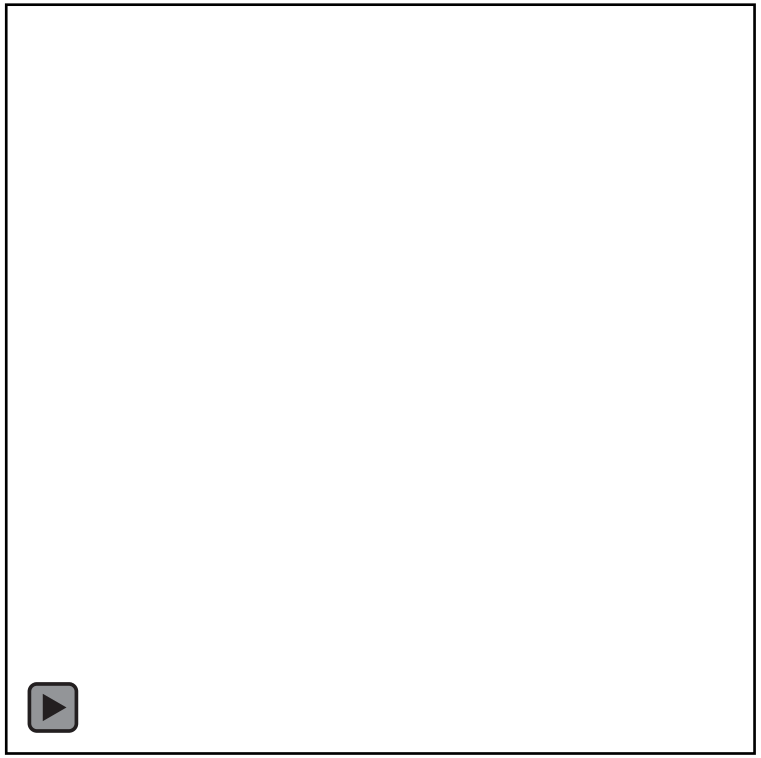
Both **dislocation-** and **grain boundary-based** mechanisms active

Which is rate-controlling?

Irradiated: Increased strength and decreased ductility due to radiation damage-induced dislocation pinning

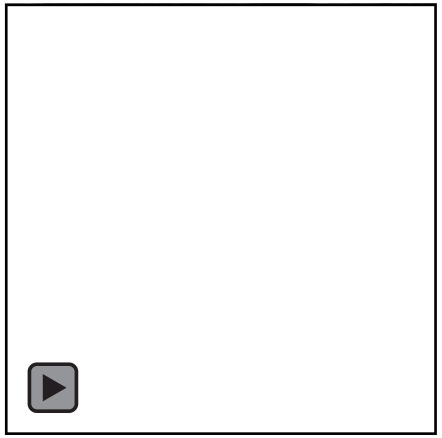
Monotonic response





200 nm

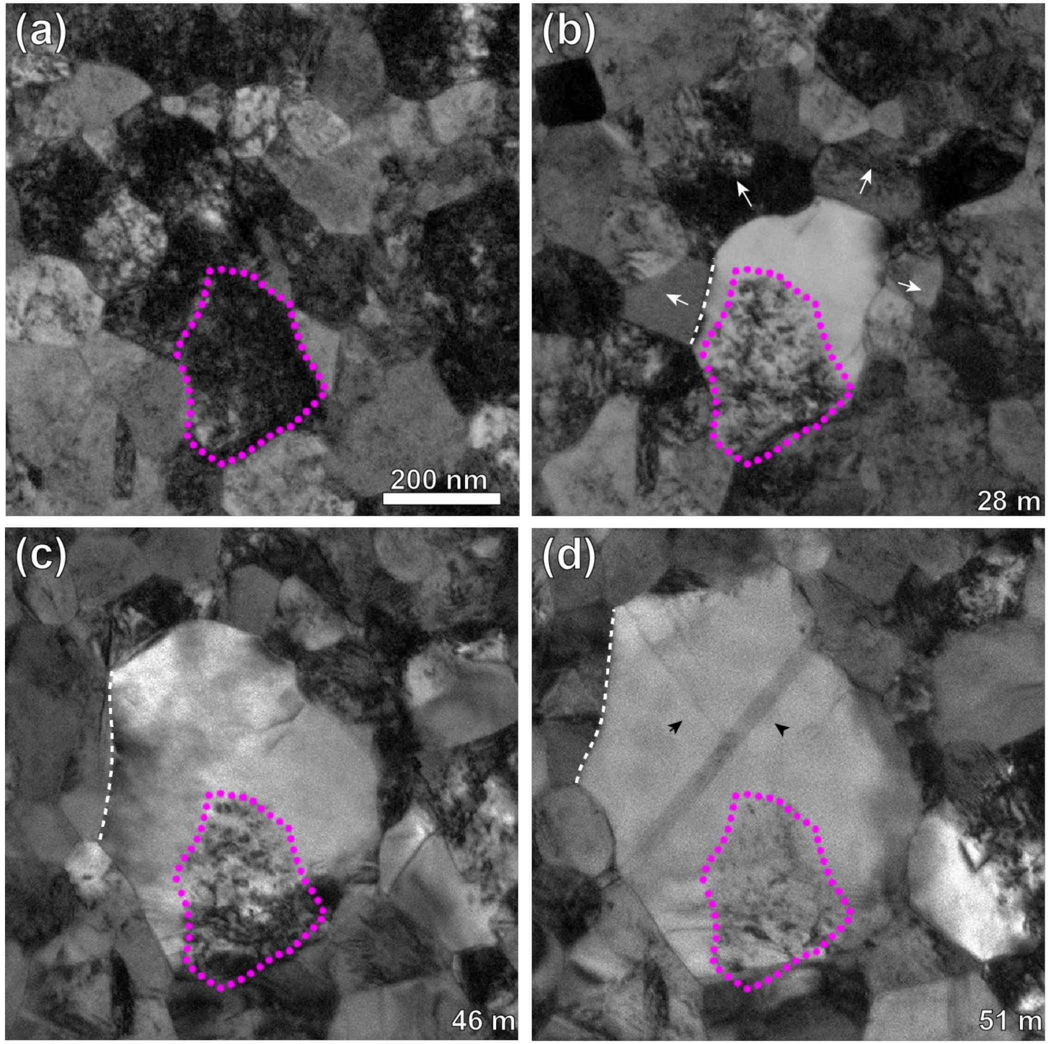
Dislocations 'crawl' through grain interiors due to repeated pinning/de-pinning on radiation defects

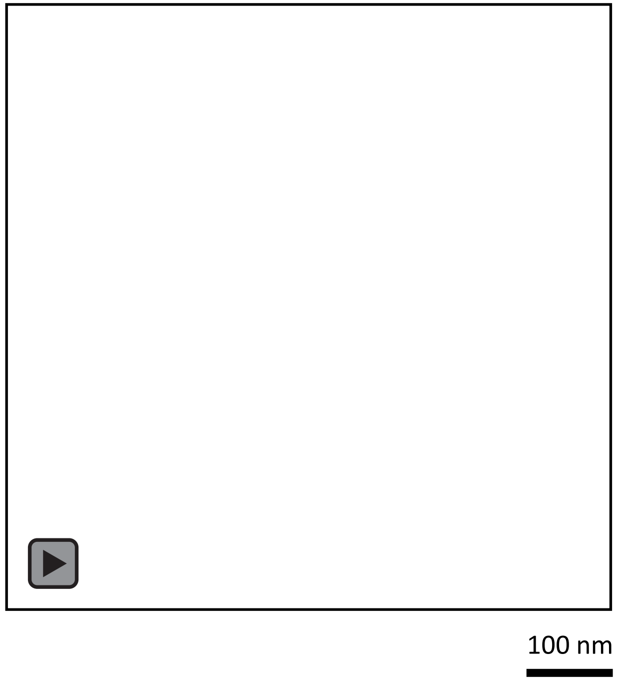


100 nm

Stangebye *et al.*, Nano Letters 23 (8) 3282-3290

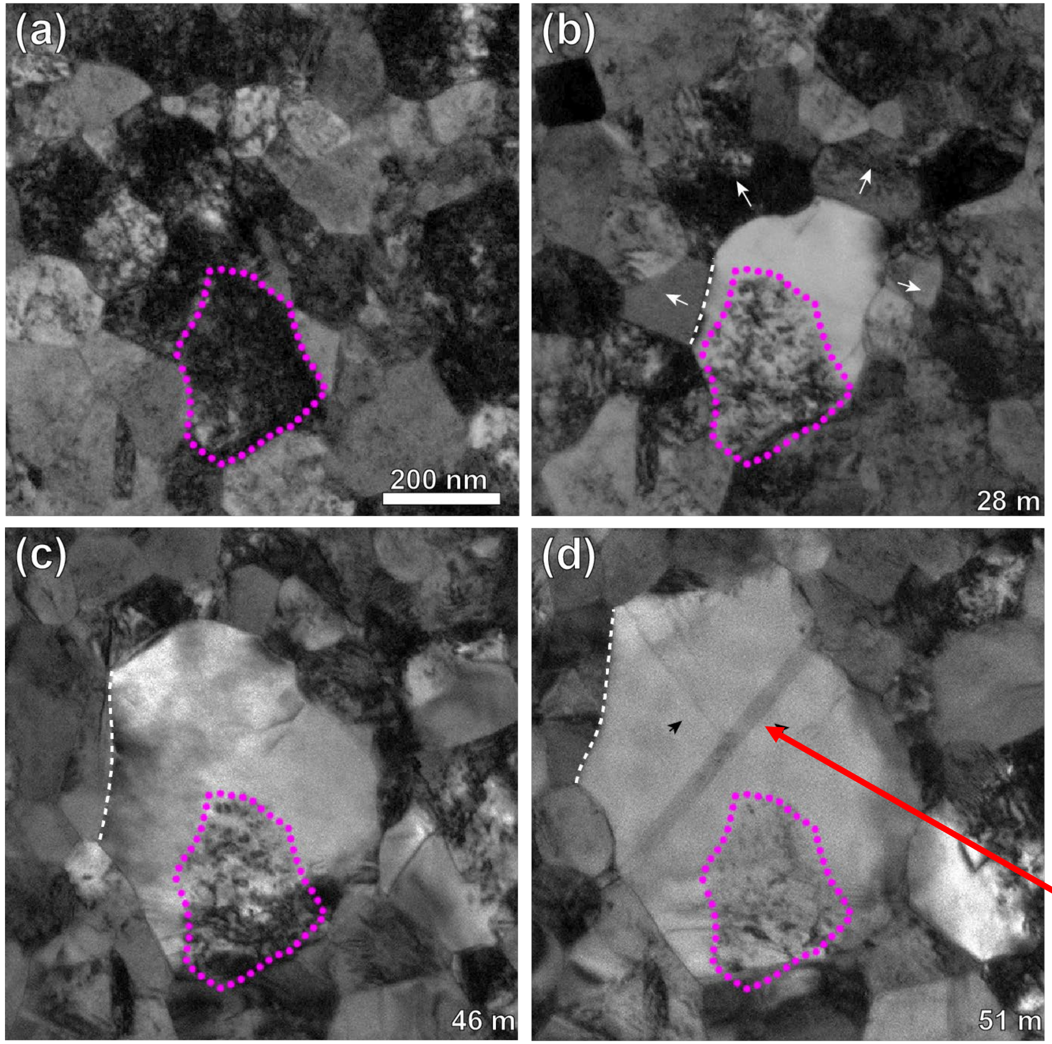
Stress-induced grain boundary migration leads to defect free regions

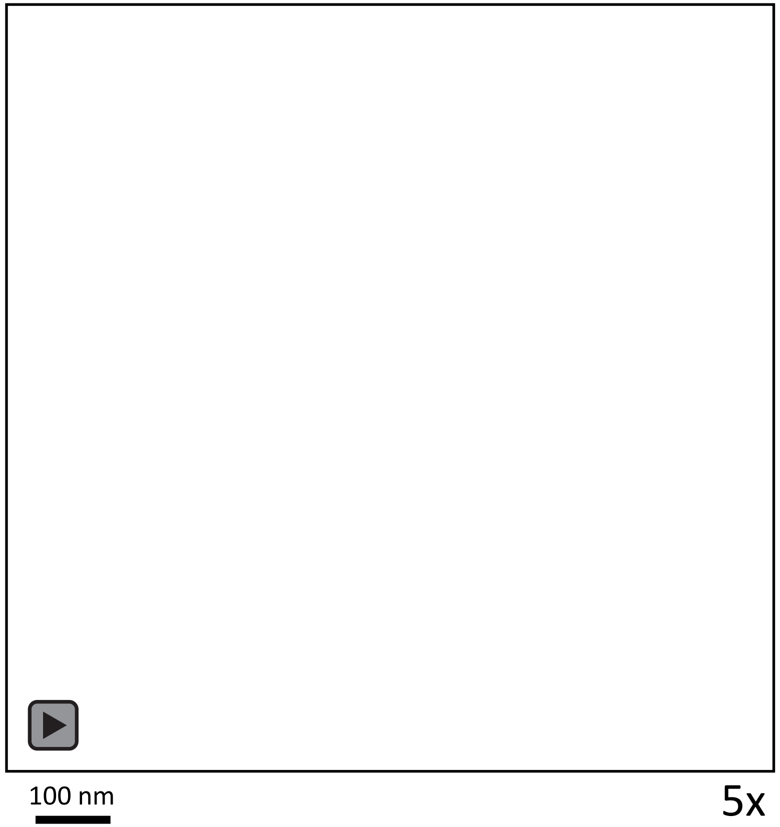




Stangebye *et al.*, Nano Letters 23 (8) 3282-3290

Stress-induced grain boundary migration leads to defect free regions





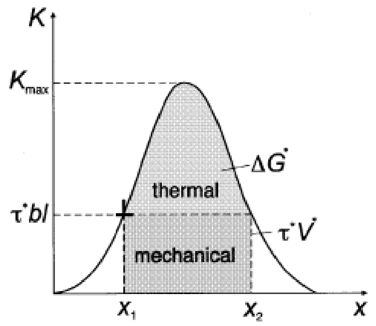
Defect free regions can now support **extended dislocation glide**

Which is rate-controlling?

Stangebye *et al*., Nano Letters 23 (8) 3282-3290

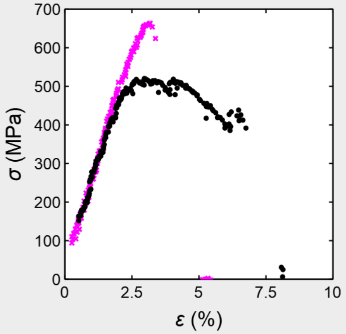
Three approaches required to fully characterize deformation mechanisms

Quantification of Mechanical Properties



Activation Volume V* is a signature parameter associated with deformation mechanism

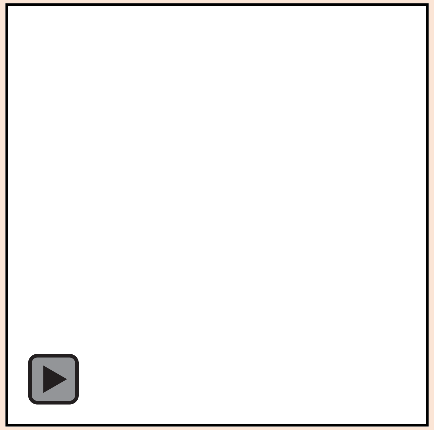
Diffusion-based mechanism ~ 1



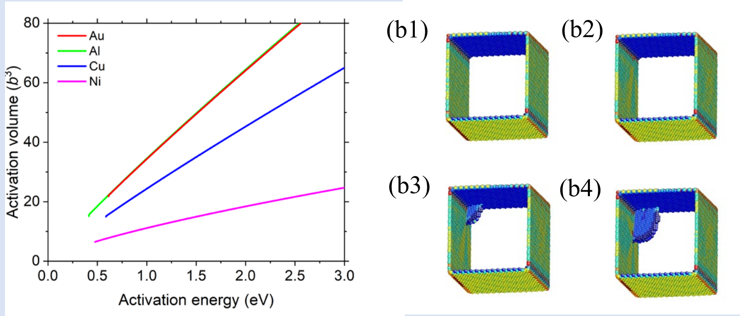
Microstructural Characterization & TEM Observations

Direct observation of dislocation and grain boundary interactions (deformation mechanisms) during straining.

Also used to characterize defect content, grain size, texture, etc.



Atomistic modeling

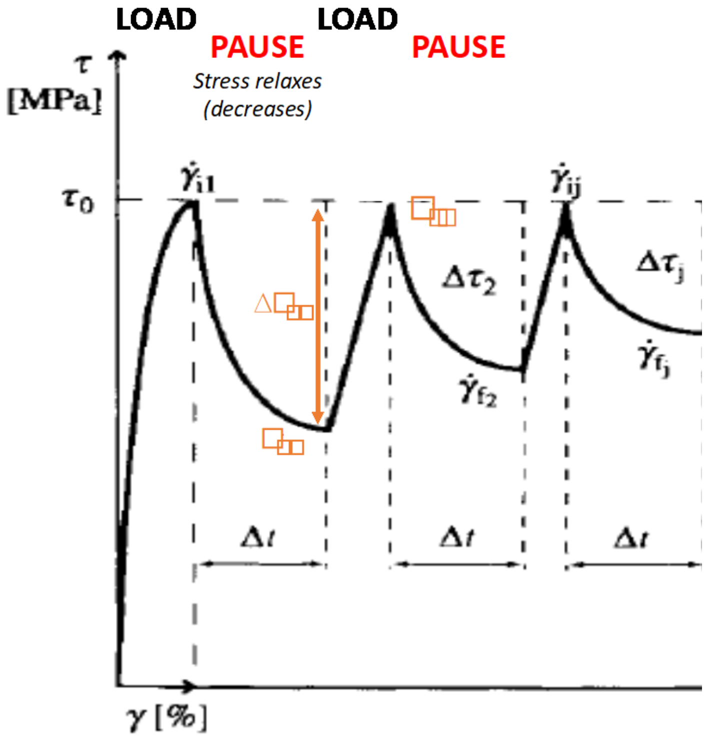


Atomistic models performed on individual mechanisms to determine the expected activation volume values. Compare these with experimentally determined to conclude on the dominant mechanisms.

Rate controlling plastic deformation mechanisms

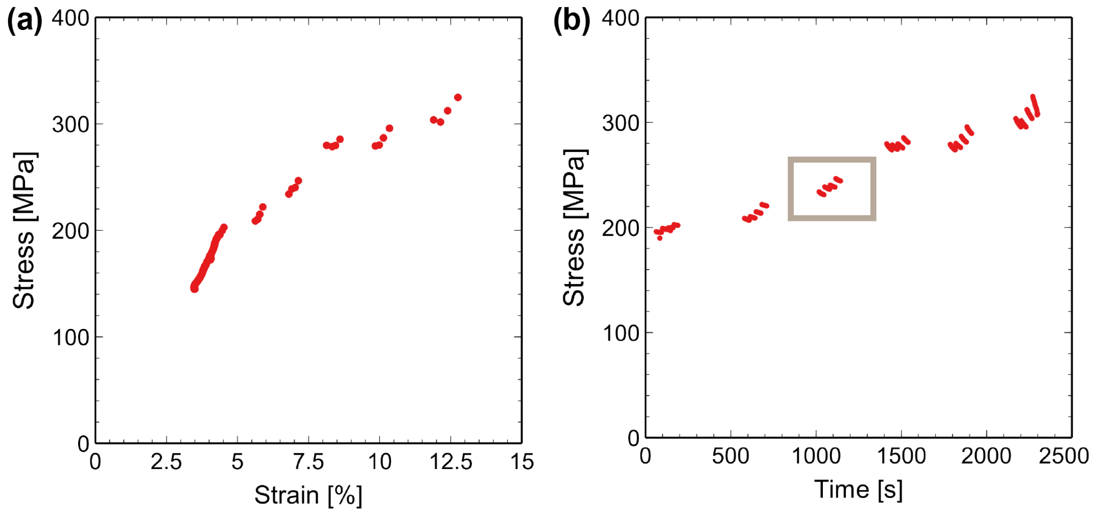
In situ TEM Activation Volume V* measurements

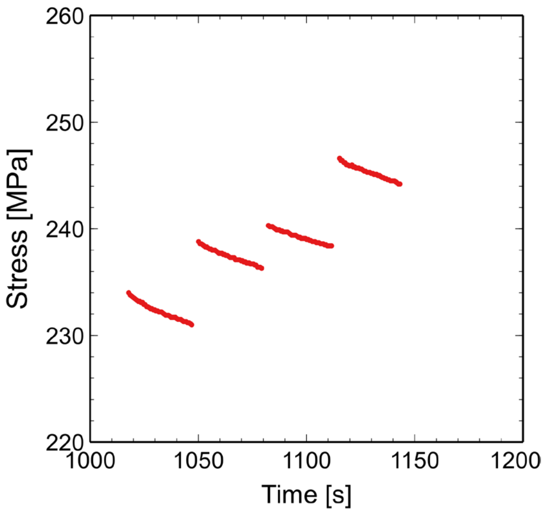
$$V^* = kT\frac{\ln(\dot{\gamma}_{i2}/\dot{\gamma}_{f1})}{\Delta\tau_{12}}$$

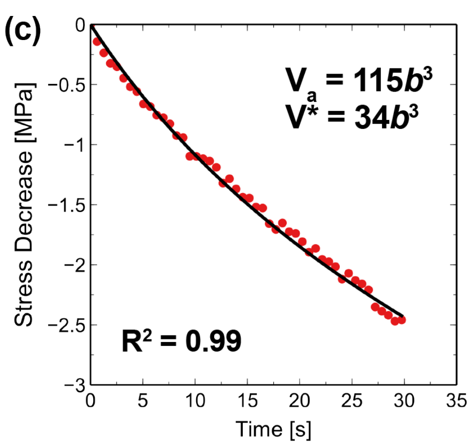


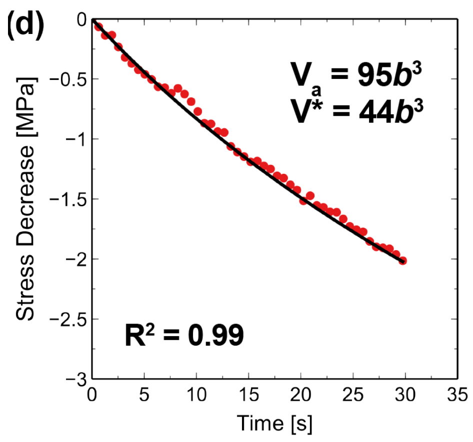
Stress-Strain

Relaxation Segments

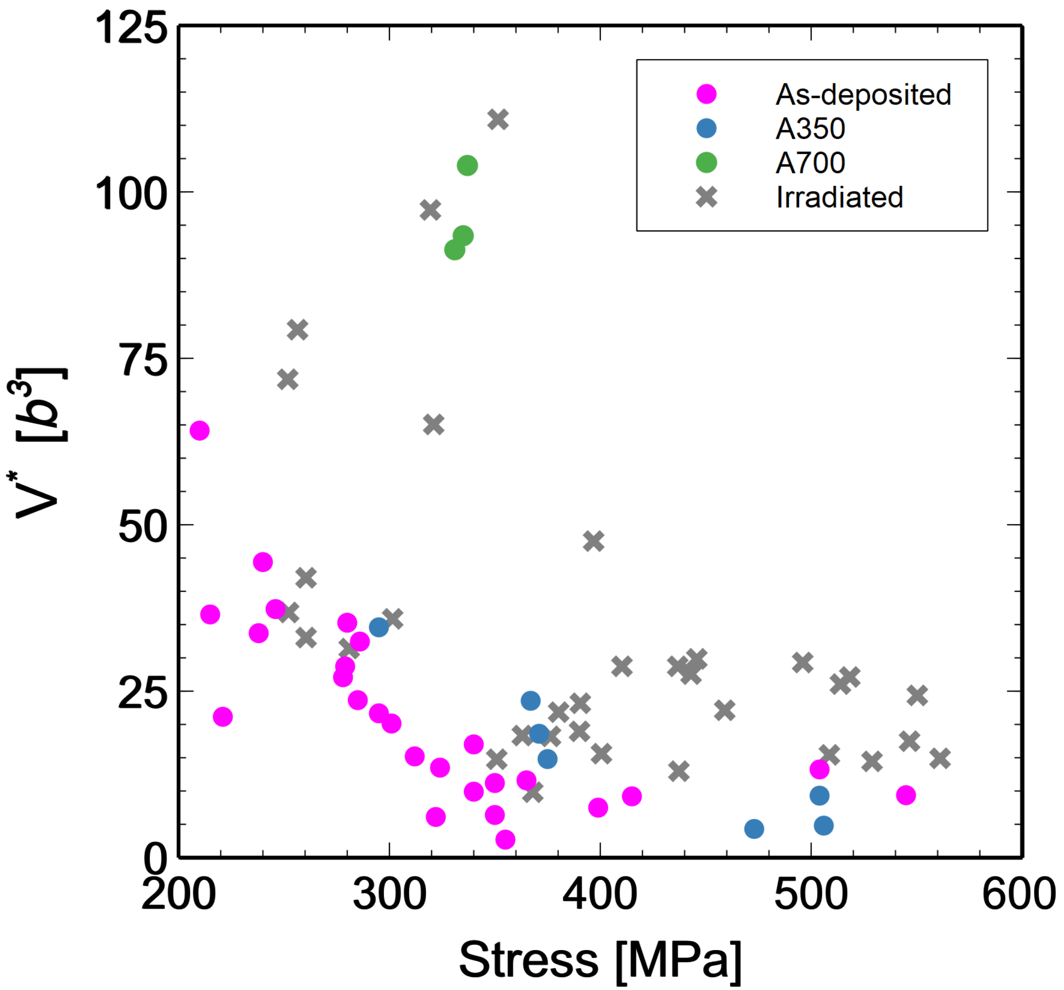








UFG Au: All V^* Measurements



	d_{avg} (nm)	V^* ($\sigma < 350$ Mpa)	V^* ($\sigma > 350$ Mpa)
As-deposited	142	25 ± 14	9 ± 3
Irradiated	189	57 ± 28	22 ± 8

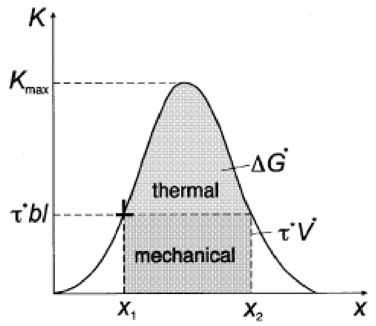
V^* increases with increasing grain size

V^* increases with radiation

V^* decreases with increasing stress

Three approaches required to fully characterize deformation mechanisms

Quantification of Mechanical Properties



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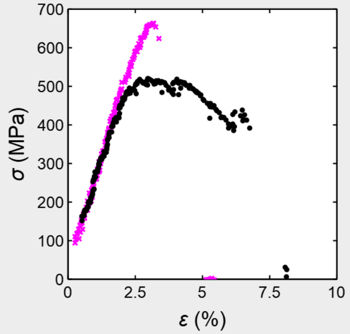

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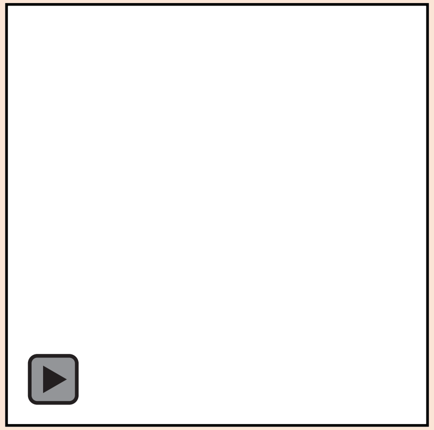

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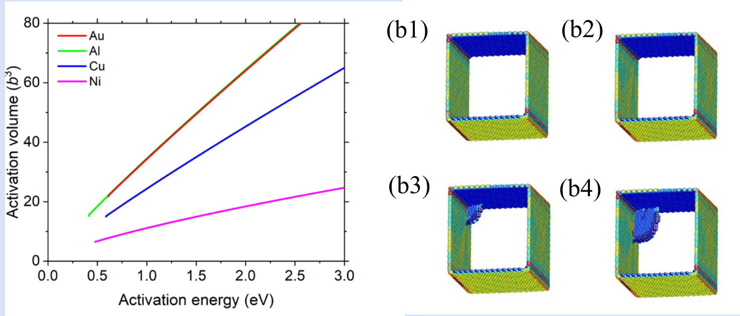

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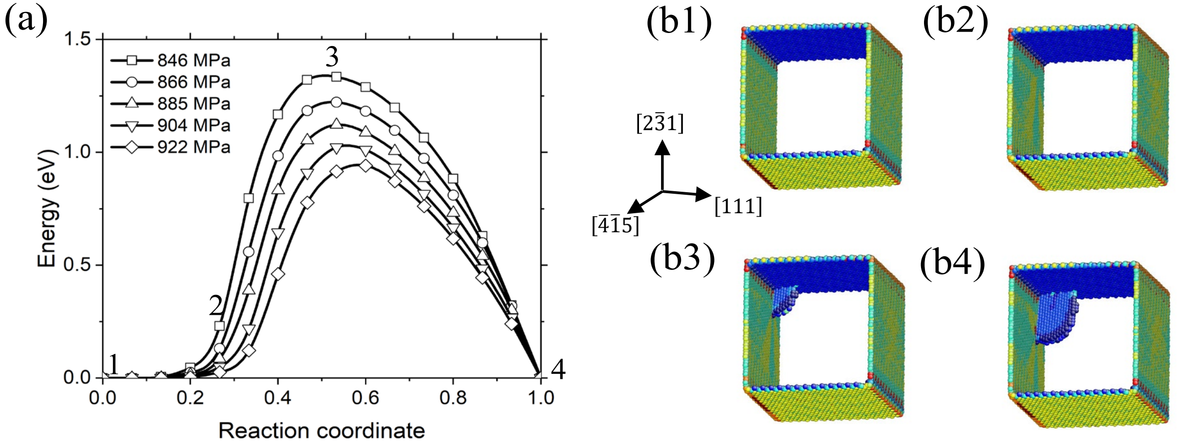
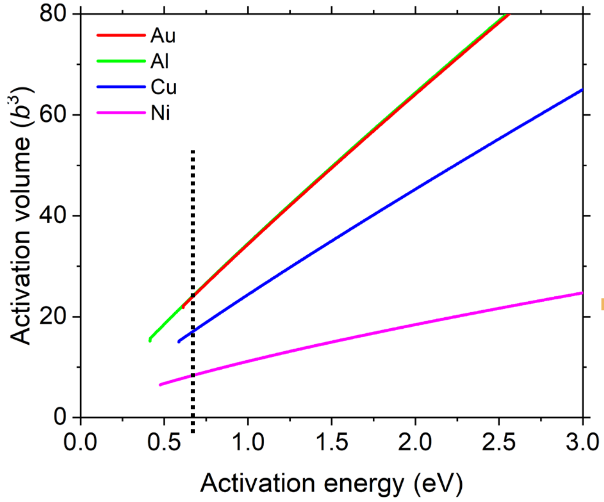
Rate controlling plastic deformation mechanisms

FENEB calculations of dislocation mechanisms give V* > 15b^3

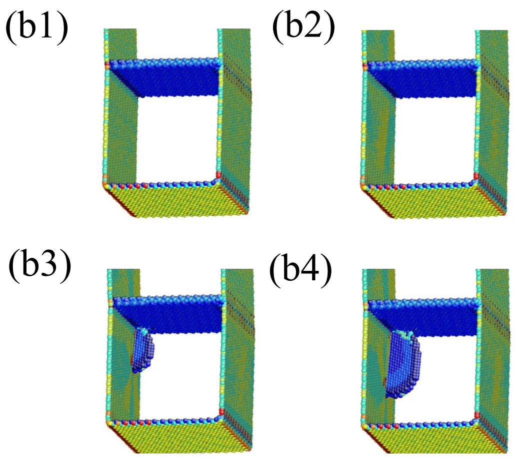
Surface dislocation nucleation

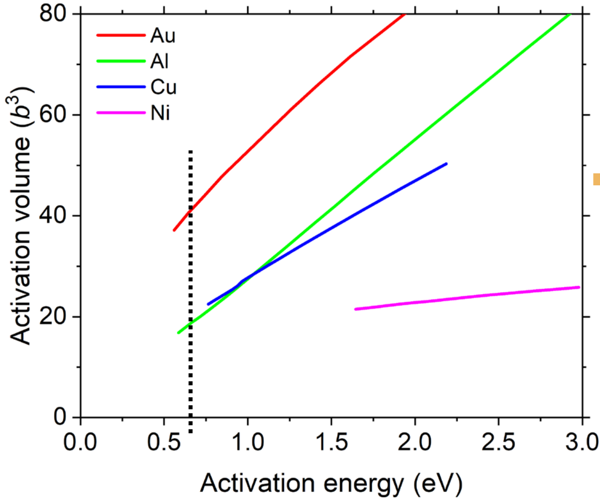




V_C ~ 20 b^3

Grain boundary dislocation nucleation



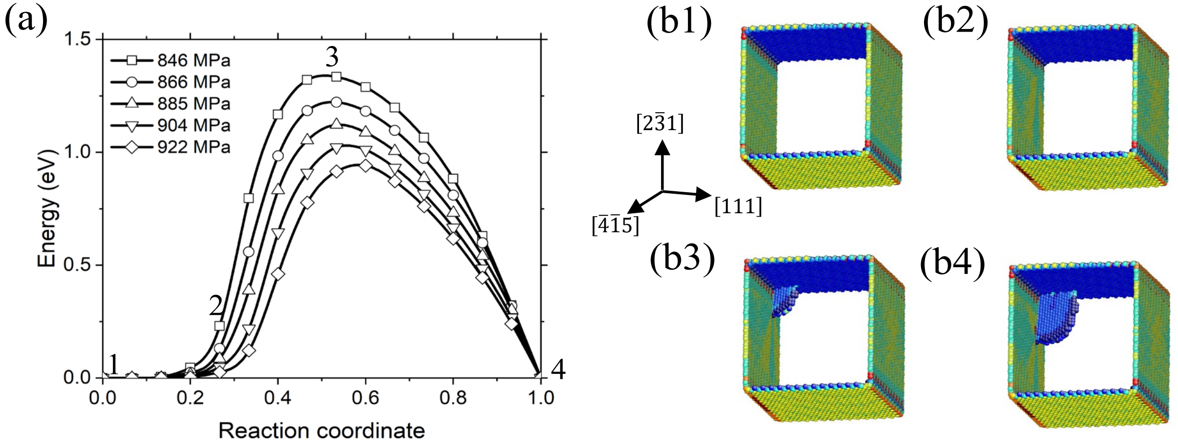


V_C ~ 40 b^3

Zhang, Ding *et al.*, Acta Mat. 237 (2022) 118155

FENEB calculations of dislocation mechanisms give V* > $15b^3$

Surface dislocation nucleation



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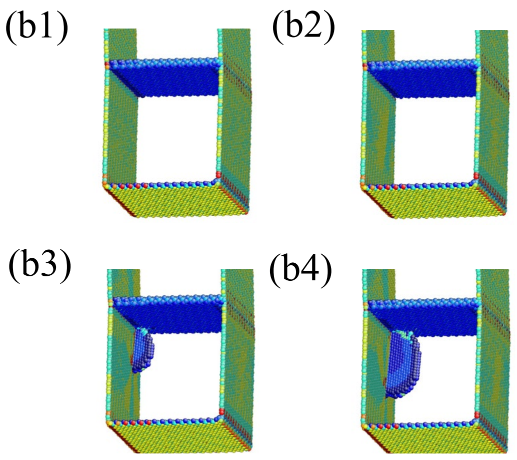
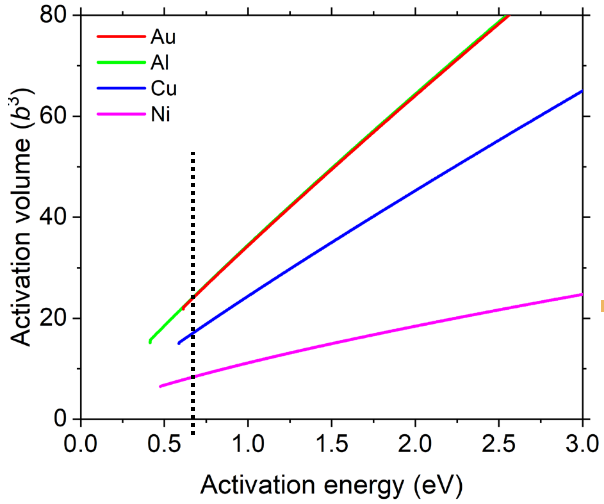
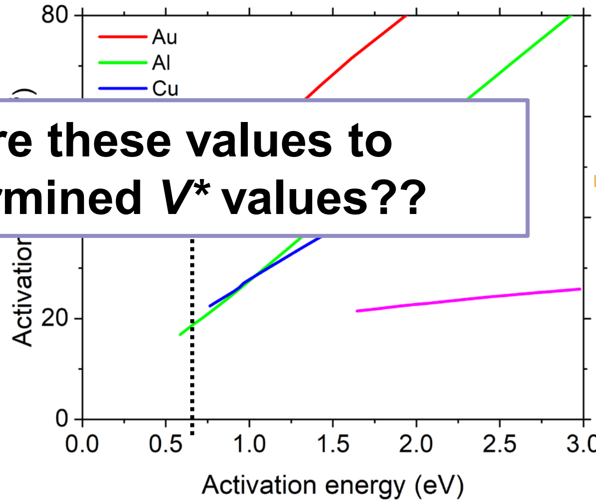




V_C ~ 20 b^3

How do we compare these values to experimentally determined *V values??**



V_C ~ 40 b^3

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Scaling required to account for grain size difference

- Experimentally: grain size ~ 140 – 760 nm
- Simulations: grain size ~ 10 nm

Hall-Petch-type relationship between grain size (*d*) and *V**:

Conrad's model:

$$\frac{1}{V^*} = \frac{1}{V_i} + \frac{M^2 \mu b}{K_{H-P}\sqrt{d}} \frac{1}{V_c}$$

Experimentally measured *V**

Activation Volume inside coarse grains

$V_i \sim lb^2$

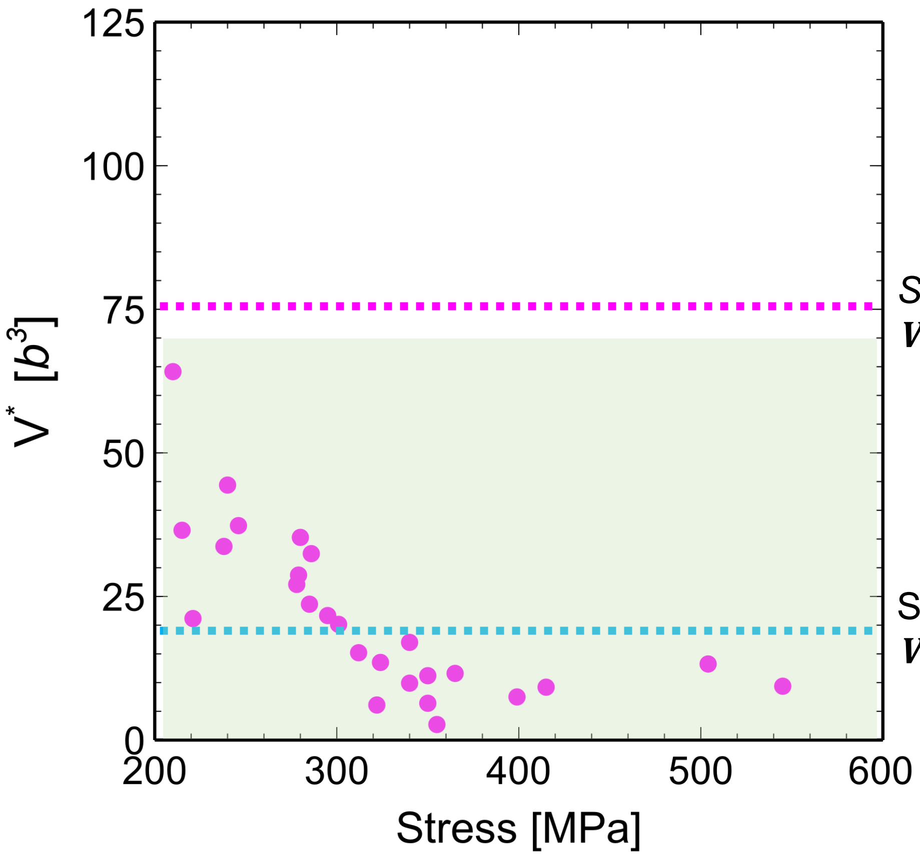
$\sim 250 - 500\ b^3$

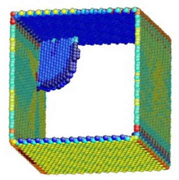
Activation Volume of process at grain boundaries

$$V^* \sim \sqrt{d}\, V_c$$

Experimentally-determined V^* **related** to atomically-determined values via a **grain size factor**

Dislocation nucleation processes cannot explain low *V** values





Scaling for grain size:

$$V^* \sim \sqrt{d}\, V_c$$

Dislocation nucleation processes cannot explain the measured small V^* → not the rate-controlling mechanisms

Other rate-controlling mechanisms with smaller V_c are operating

This technique is a powerful tool used to characterize the plastic deformation mechanisms controlling the mechanical properties of NC and UFG metal thin films

01

Determine the dominant active deformation mechanisms through *in situ* TEM observations

Multiple deformation modes are active, including both dislocation and GB-based

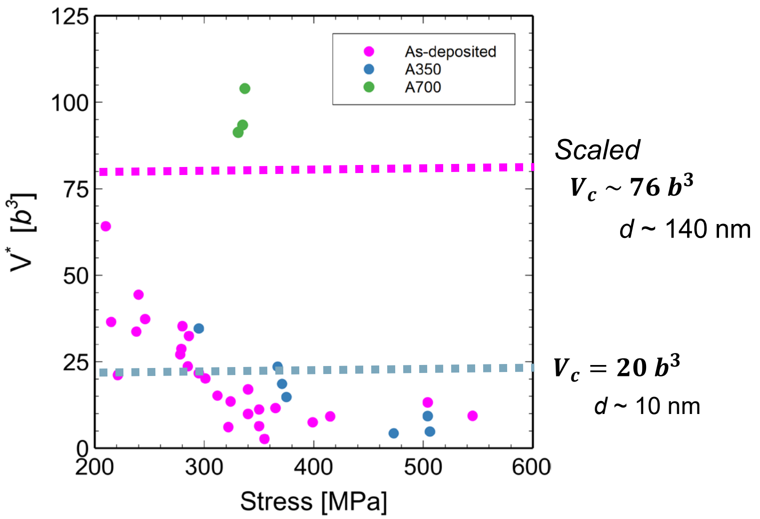
02

Characterize how irradiation damage alters deformation mechanisms

The application of stress can be an effective means of removing radiation damage

03

Relate sample-level experimentally measured V^* to individual rate-limiting unit processes



Other intergranular rate-controlling mechanisms with smaller V_c are operating

Acknowledgement

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Collaborators: Dr. Ting Zhu (ME), Dr. Yin Zhang, Dr. Xing Liu, Kunqing Ding

Committee Members: Dr. Naresh Thadhani (MSE), Dr. Hamid Garmestani (MSE)

Others: Dr. Yong Ding (GT), Dr. Khalid Hattar (Sandia), Dr. Eric Lang (Sandia, UNM)

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DOE NNSA SSGF #DE-NA0003960

SSGF

STEWARDSHIP SCIENCE GRADUATE FELLOWSHIP

What's next??

Working on R&D for Radar systems!

Raytheon Technologies