Experimental Solid Mechanics Across Spatial and Temporal Scales

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Outline

- **A fascinating wave propagation experiment** to investigate failure initiation and kinetics in ceramics
- **Mesoscale stochastic models** to bridge atomistic processes and continuum measurements
- **Functionality driven evolution of materials**, role of hierarchies in natural ceramics … and inferring material design rules
- **Seeing the invisible**: failure in low dimensional materials

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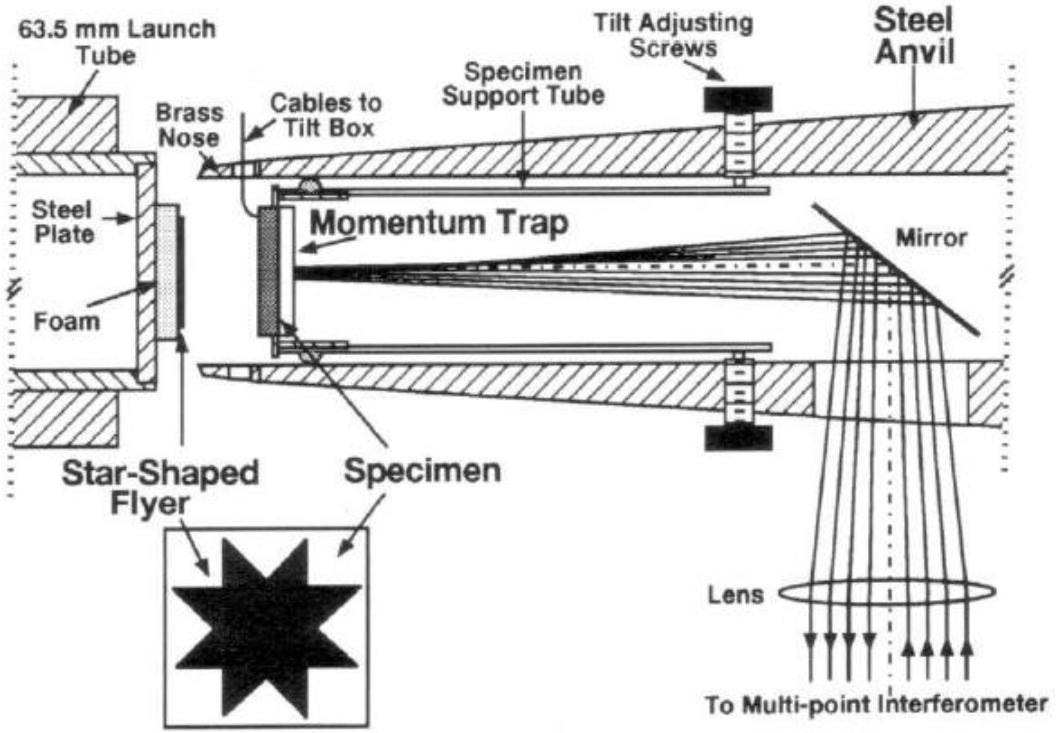
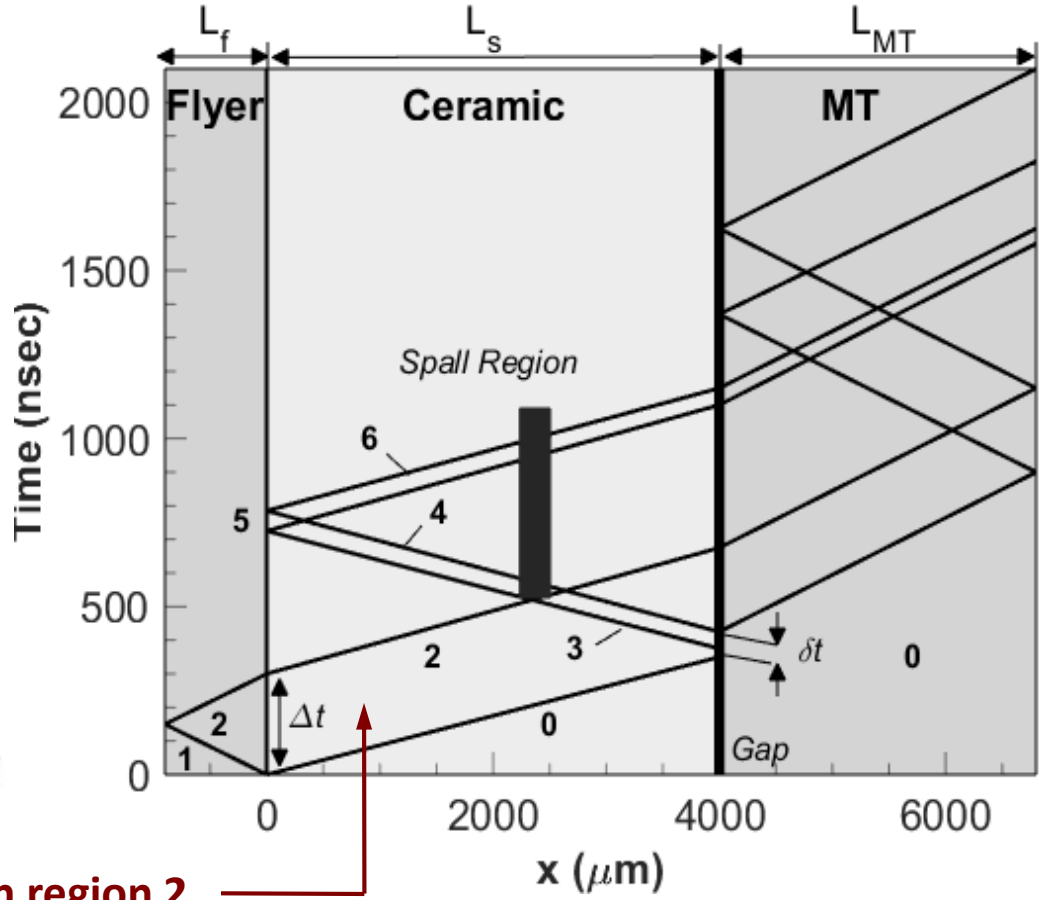
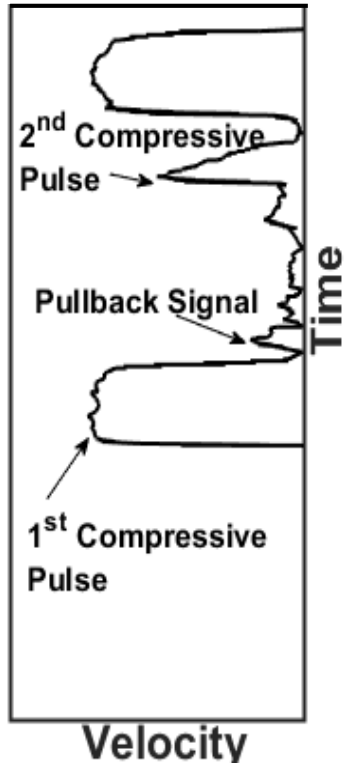
- Seeing the invisible: failure in low dimensional materials

Stress Waves – Damage Initiation and Kinetics

Rod Clifton's soft recovery impact experiment



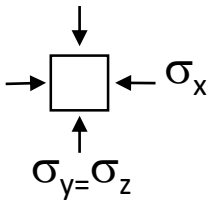




Elastic stress state in region 2

$\sigma_y=[\nu/(1-\nu)]\,\sigma_x$; $p=\frac{1}{3}\left[\frac{1+\nu}{1-\nu}\right]\sigma_x$

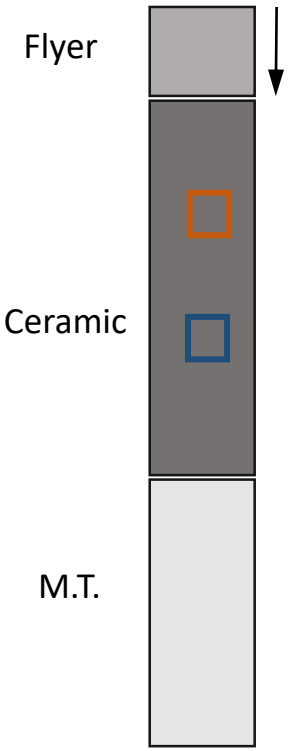
$2\tau=\sigma_x-\sigma_y$

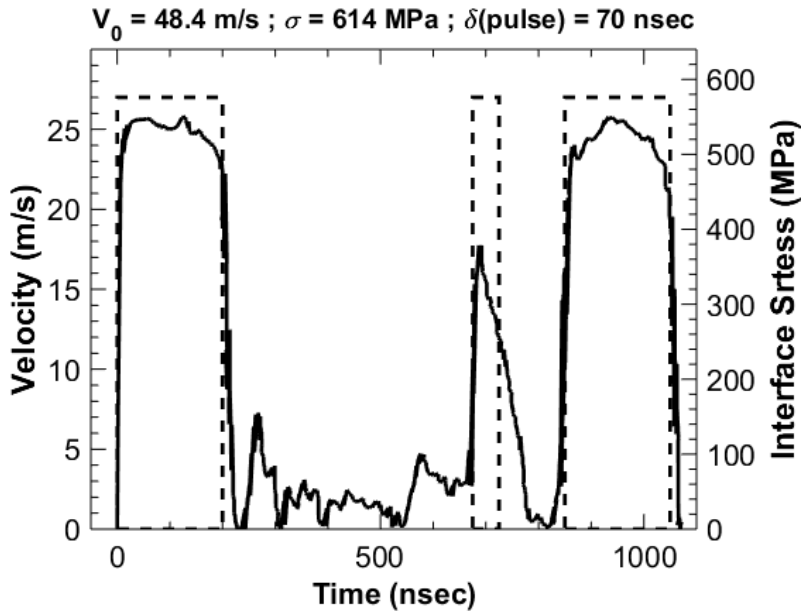


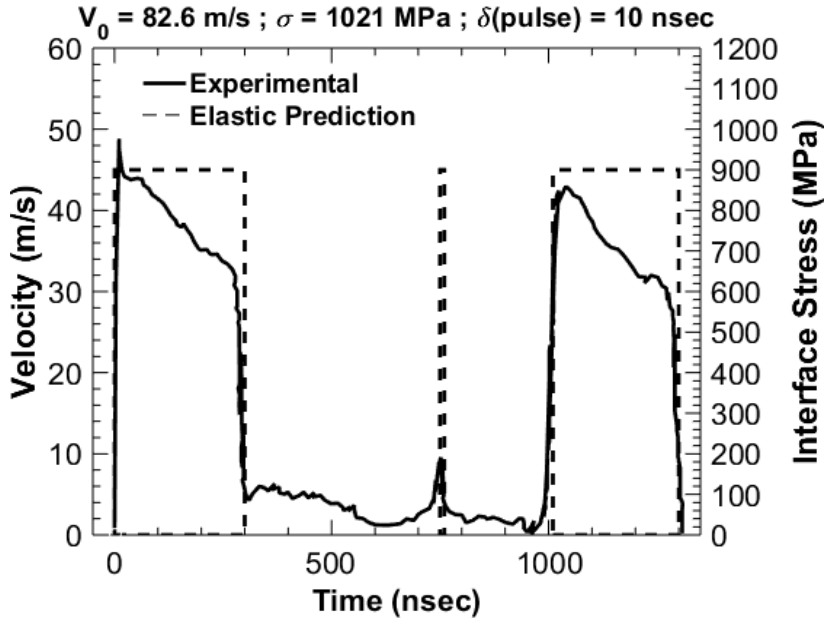
Raiser, Clifton, Ortiz, *Mech. Mater.*, 10, 1990

Kumar and Clifton, *J. Appl. Phys.*, 1979

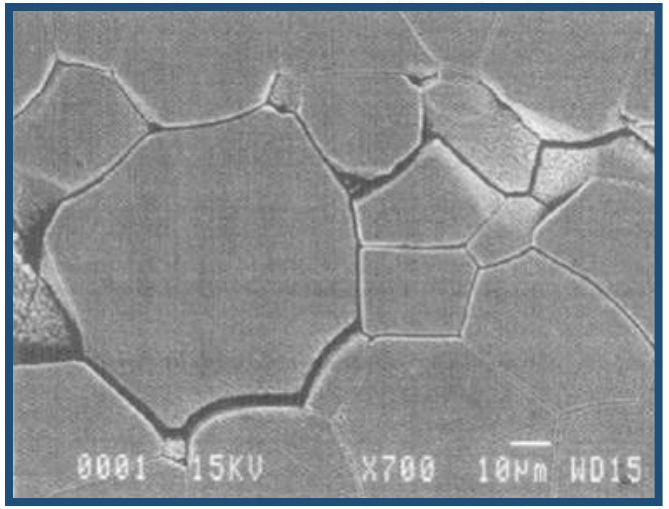
Velocity Profiles for 99.9% Al_2O_3



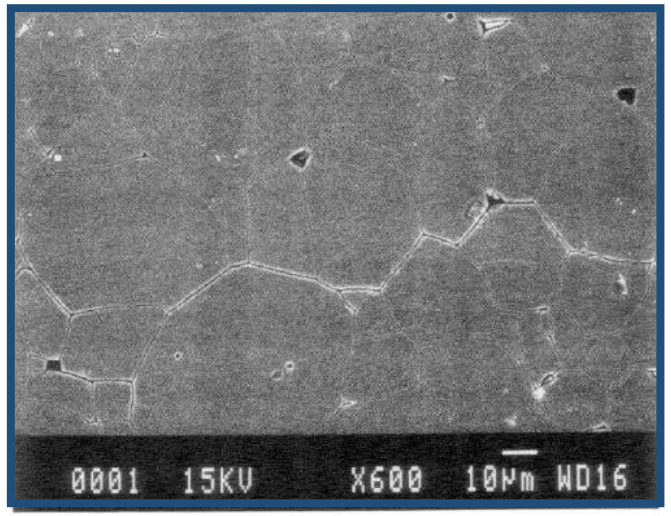




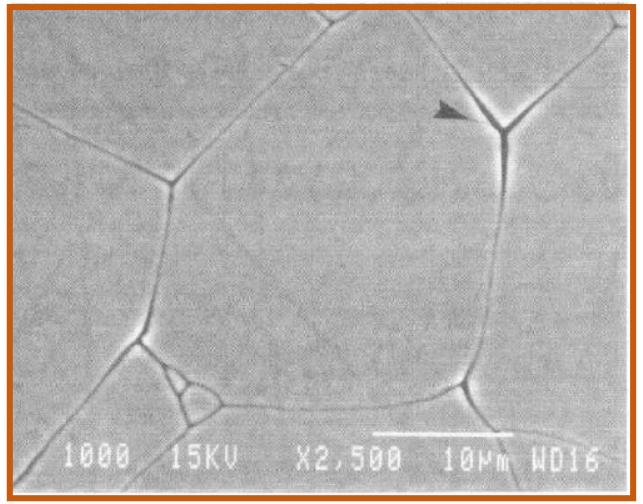
Tensile Damage (δ_t=70 ns)



Tensile Damage δ_t=10 ns



Compressive Damage (Δ_t=300 ns)

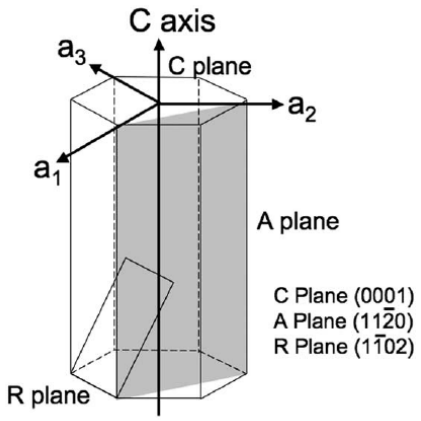


Espinosa, Raiser, Clifton, Ortiz, J. Hard Materials, 3, 1992

Raiser, Clifton, Ortiz, *Mech. Mater.*, 10, 1990

Dynamic Yield and Failure of Al_2O_3

Single Crystal Sapphire

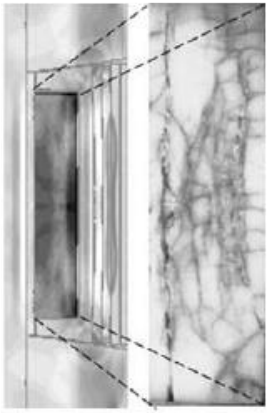


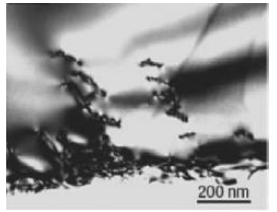
$\sigma_{y=}\sigma_z$

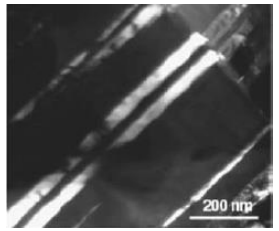
σ_x

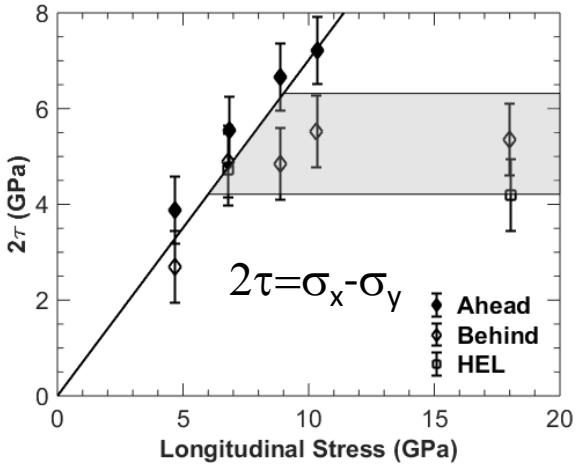
$\sigma_y=[\nu/(1-\nu)]\ \sigma_x$

$$p = \frac{1}{3}\left[\frac{1+\nu}{1-\nu}\right]\sigma_x$$

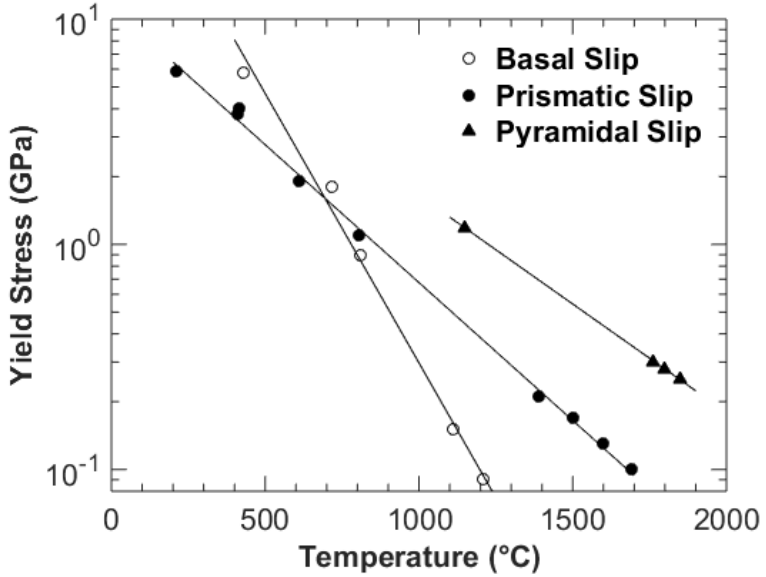








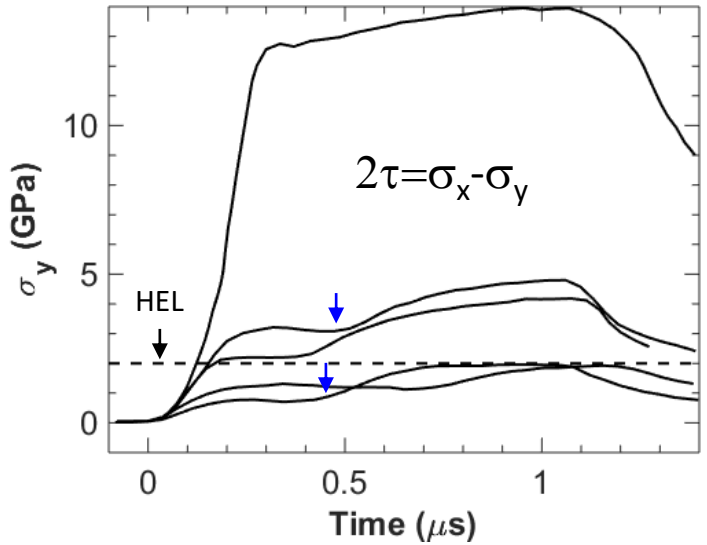
Bourne et al., *JAP*, 102, 2007



The dynamic yield stress of Al_2O_3: $\sigma_{HEL} \approx 6\ GPa$

Grain plasticity at room temperature is limited. Pyramidal and basal slip requires shear stresses in excess to the σ_{HEL}

The tensile (spall) strength of Al_2O_3 is $\approx 0.2 - 0.6\ GPa$ and it is a function of initial compressive pulse amplitude and duration



How do we explain compressive damage below σ_{HEL}?

How do we understand the coupling between compressive and tensile inelasticity?

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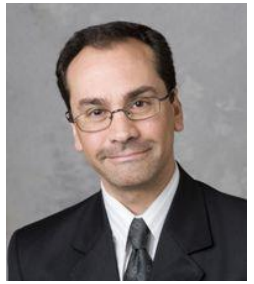
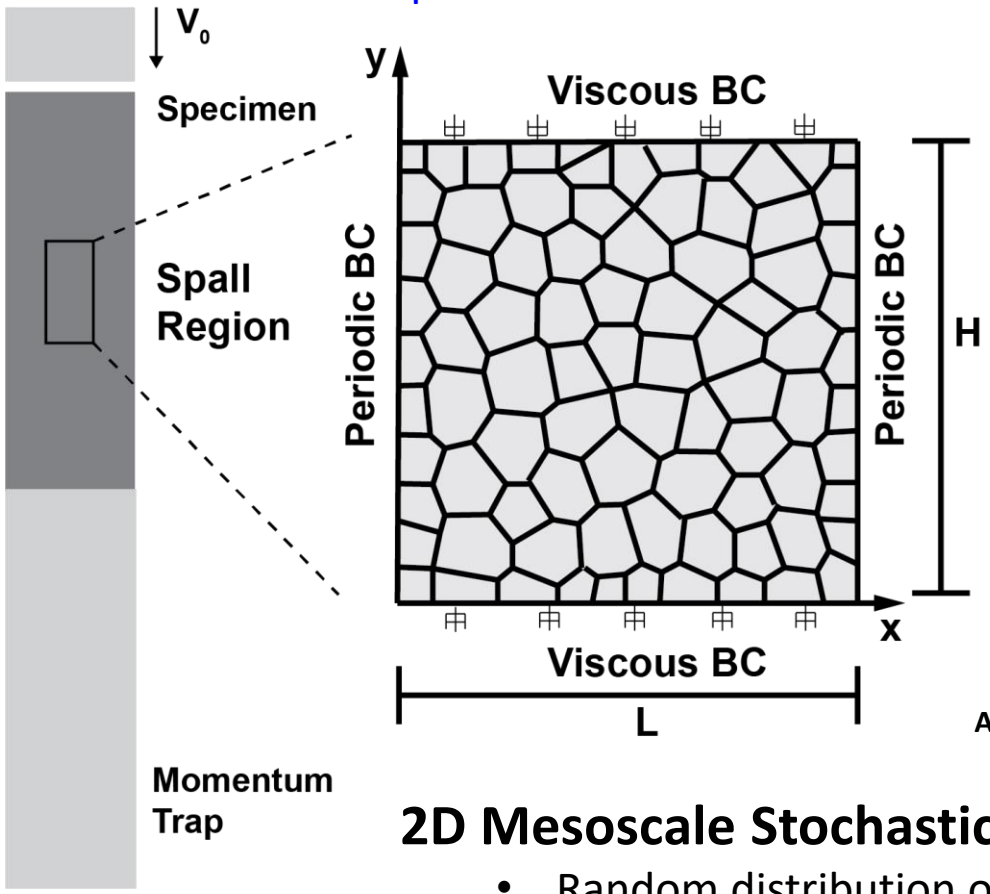
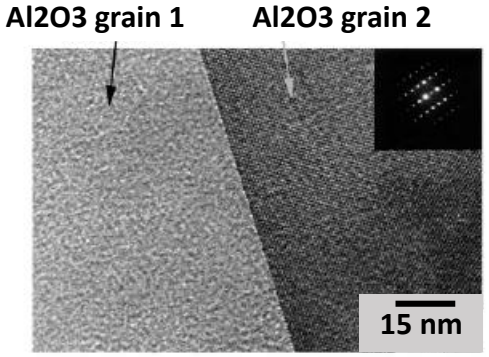
Mesoscale Stochastic Model

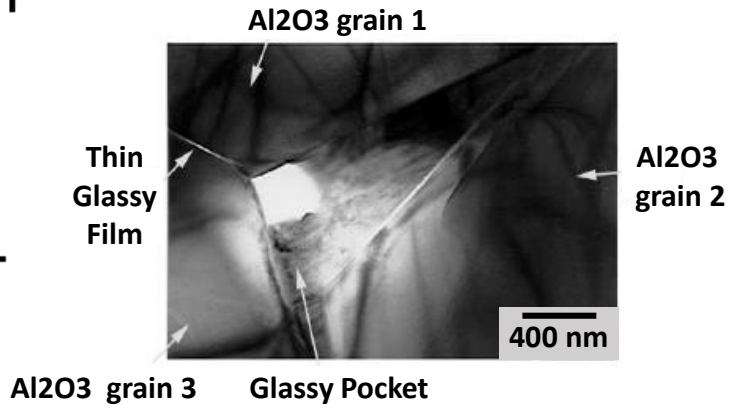
Pablo Zavattieri

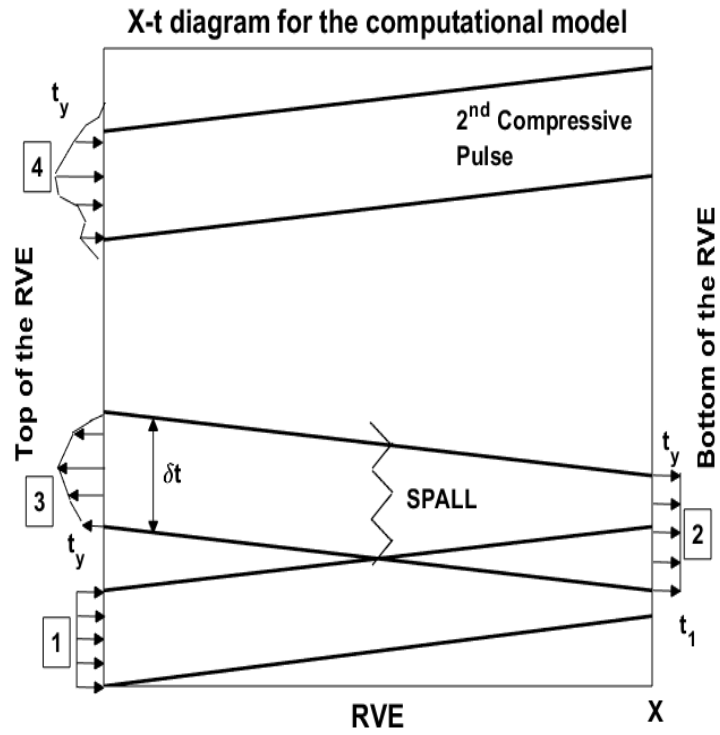


Representative Volume Element







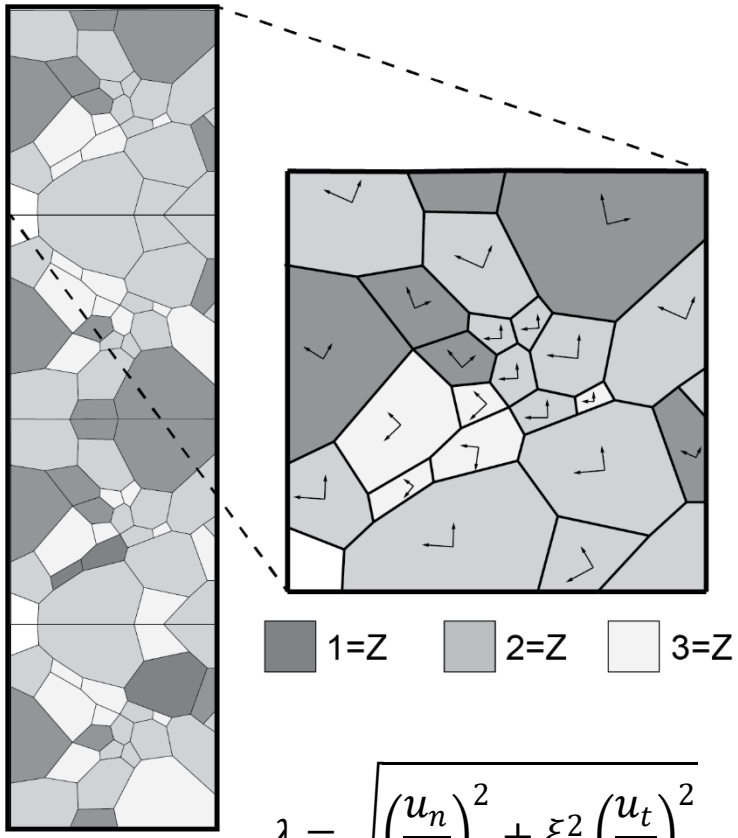


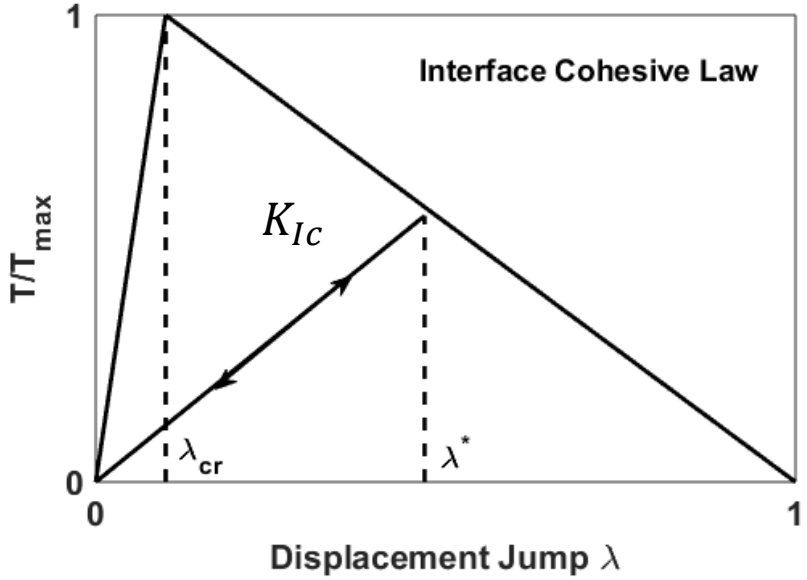
2D Mesoscale Stochastic Model

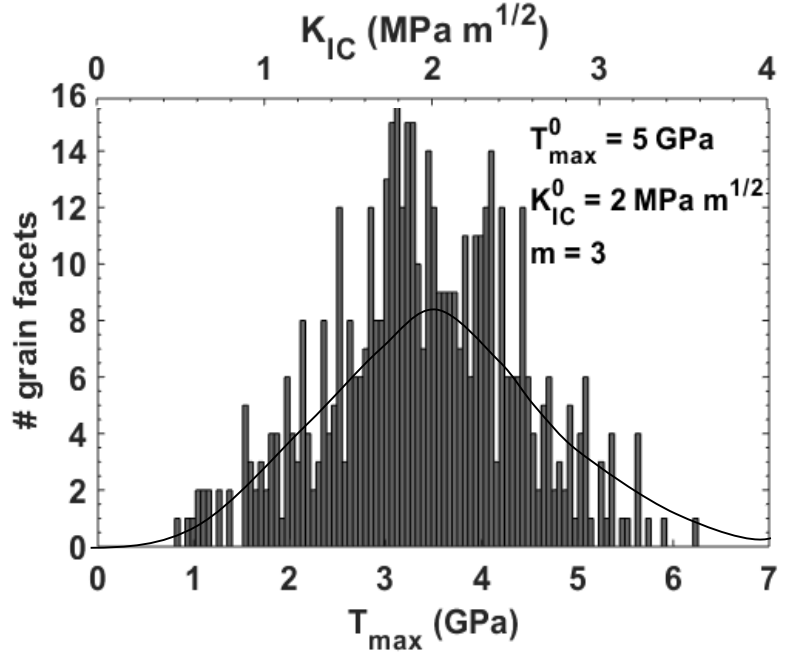
- Random distribution of grains size and crystal orientation
- Elastic anisotropic grains, stress fluctuations including stress concentration at triple points
- Random distribution of grain boundary strength and toughness
- Inclusion of heterogeneities (e.g., glassy phase)

Zavattieri and Espinosa, "Grain Level Analysis of Crack Initiation and Propagation in Brittle Materials," *Acta Materialia*, Vol. 49, 2001

Crystal Elastic Anisotropy and Weibull Interface Strength/Toughness







$$f(T_{max}) = \frac{m(T_{max})^{m-1}}{(T^0_{max})^m}\, exp\left[-\left(\frac{T_{max}}{T^0_{max}}\right)^m\right] ;\ T_{max} > 0$$

$$\lambda = \sqrt{\left(\frac{u_n}{\delta_n}\right)^2 + \xi^2\left(\frac{u_t}{\delta_t}\right)^2}$$

$$\xi^2 = {K_{Ic}}/{K_{IIc}}$$

$$\alpha = \xi^2\left({\delta_n}/{\delta_t}\right)$$

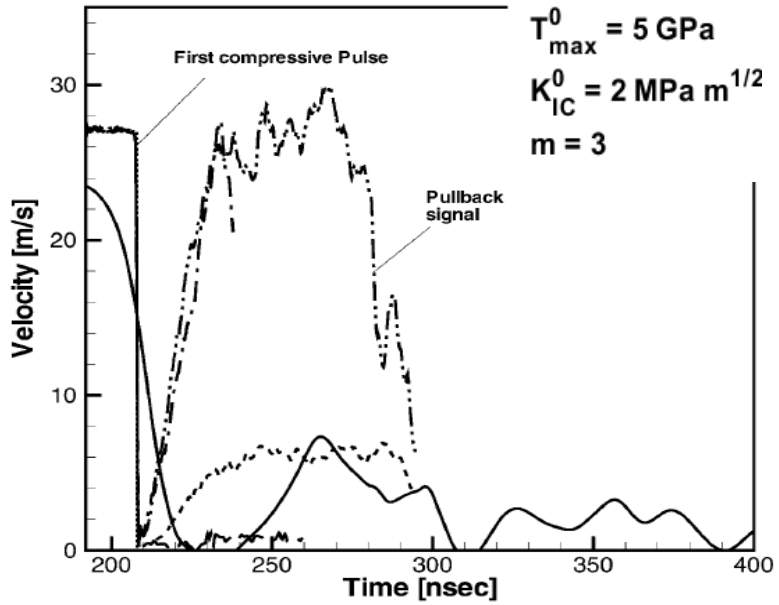
T_n: normal traction ; u_n: normal displ. jump

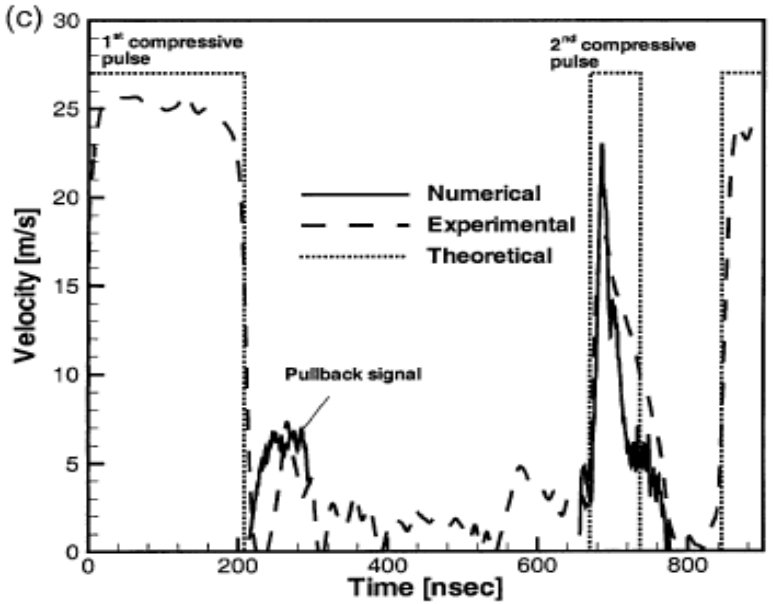
T_t: transverse traction ; u_t: transverse displ. jump

$$T_n = \frac{u_n}{\delta_n}\frac{T_{max}}{\lambda_{cr}} ;\ T_t = \alpha\,\frac{u_t}{\delta_t}\frac{T_{max}}{\lambda_{cr}} \qquad 0 \le \lambda \le \lambda_{cr}$$

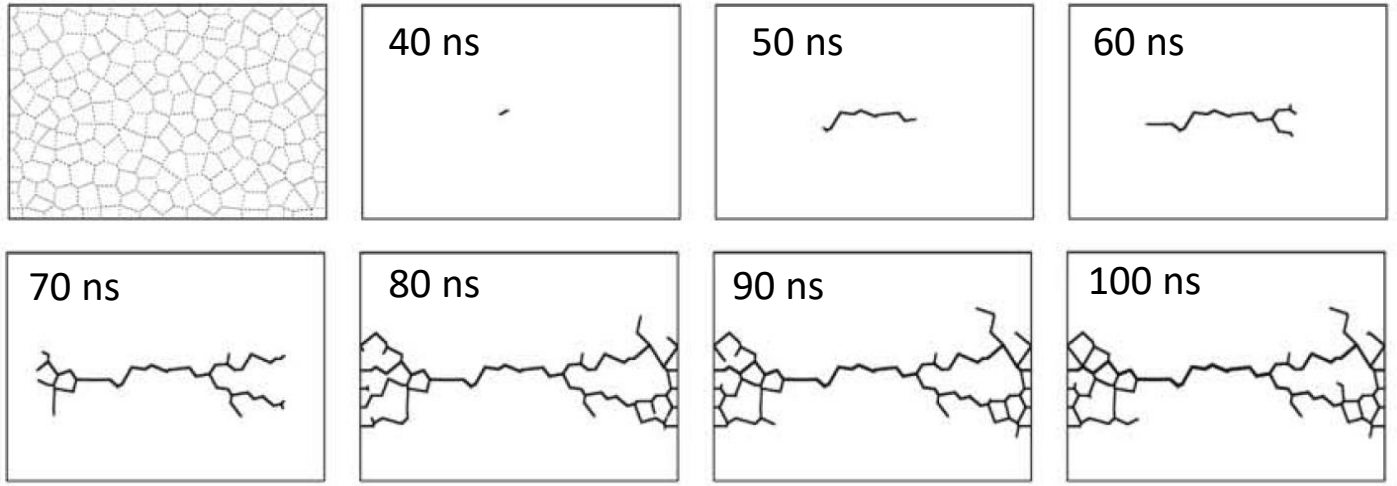
$$T_n = T_{max}\,\frac{u_n}{\delta_n}\,\frac{1-\lambda}{\lambda\,(1-\lambda_{cr})} ;\ T_t = \alpha\,T_{max}\,\frac{u_t}{\delta_t}\,\frac{1-\lambda}{\lambda\,(1-\lambda_{cr})} \qquad \lambda_{cr} \le \lambda \le 1$$

Damage Kinetics and Crack Speeds

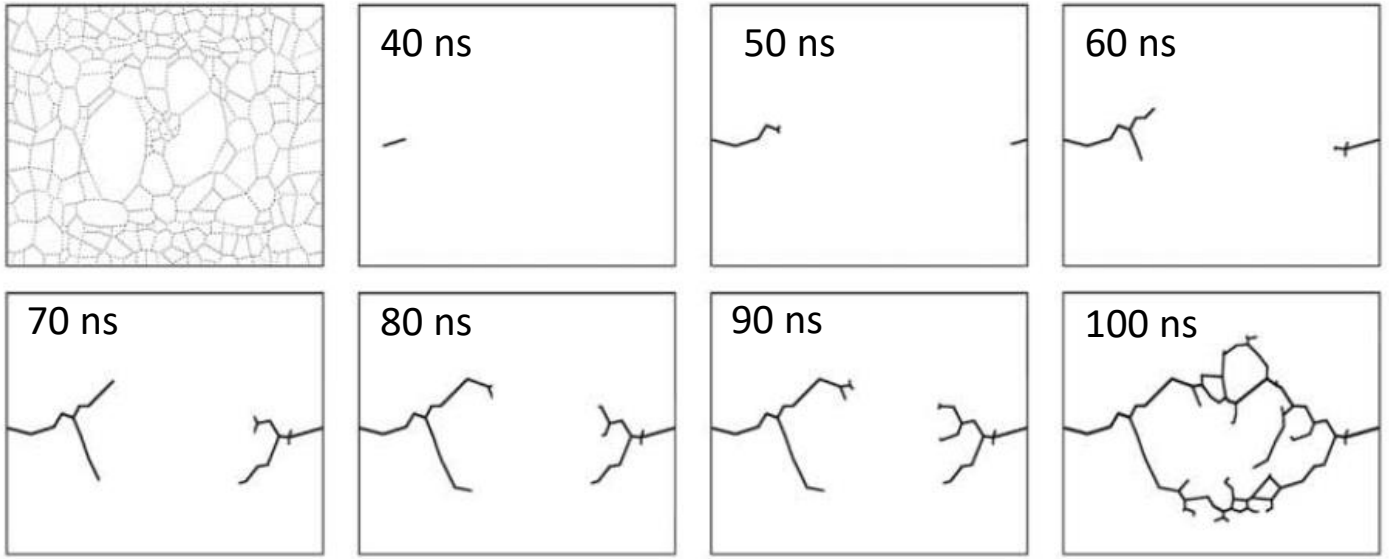




Uniform Grain Size

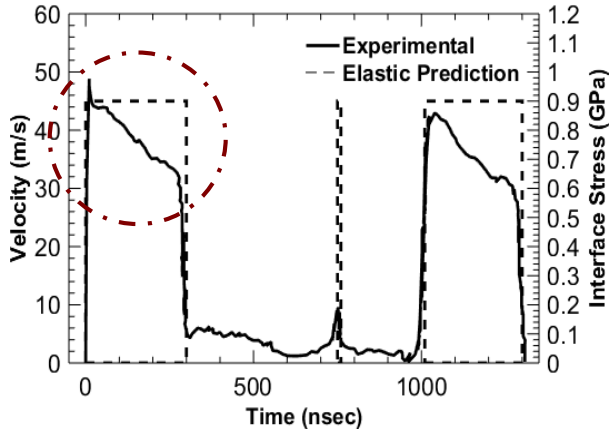


With Grain Size Distribution

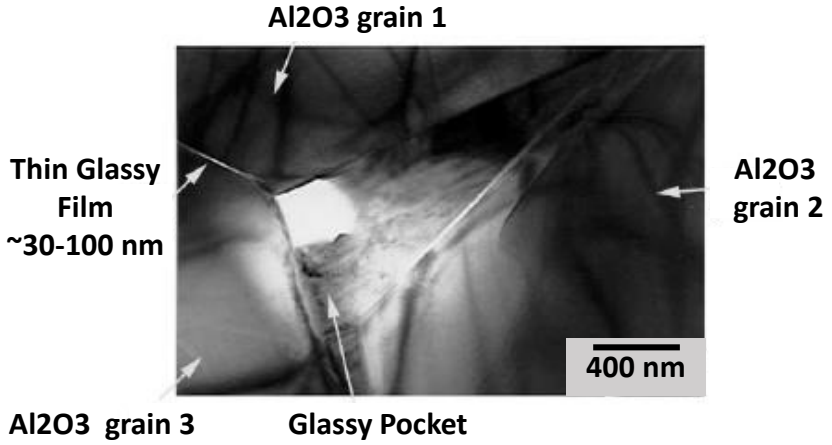


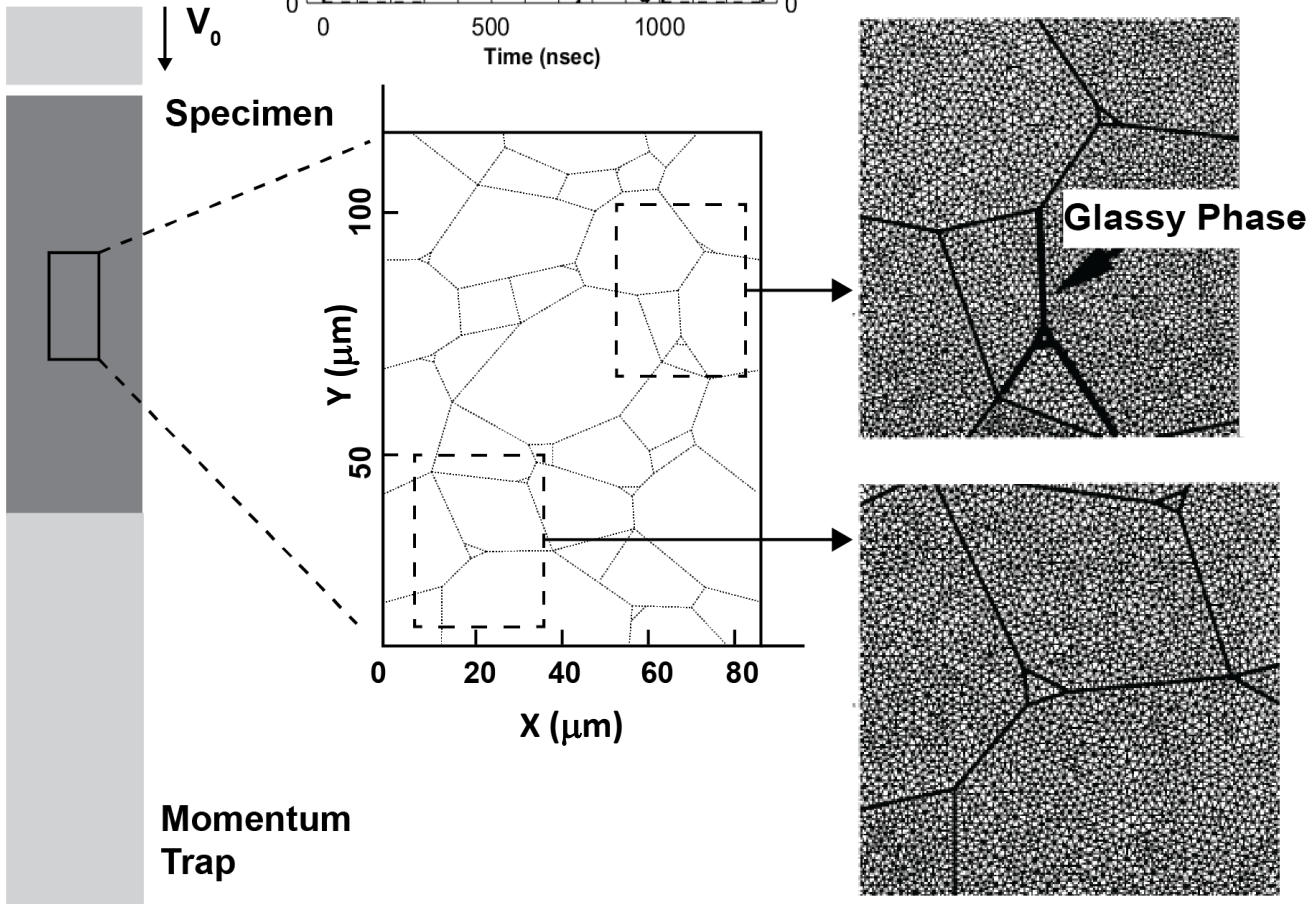
For a dynamically loaded crack: $\dot{a} = \beta\, c_R \left[1 - (K_{IC}^D / K_{app})\right]$; $\beta \sim 0.3$

Damage in Compression Below HEL – Mechanism?

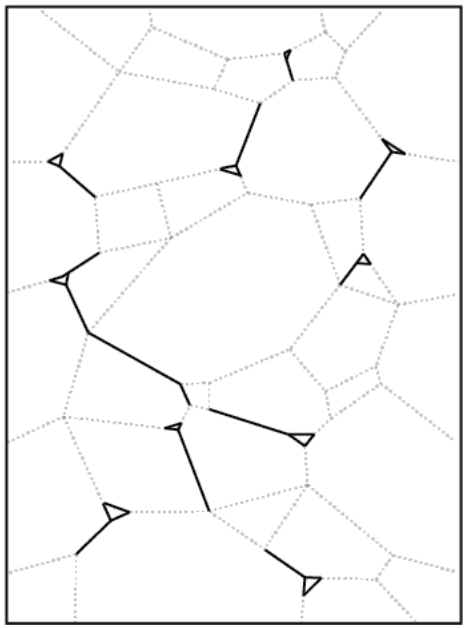


A radical hypothesis: flow of the glassy phase

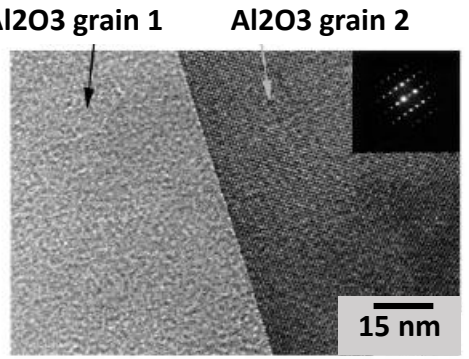




Glassy Phase



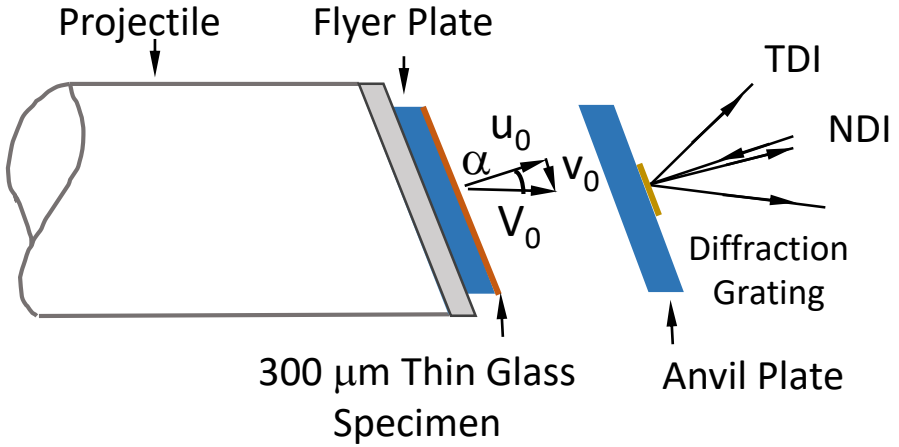
TEM Images



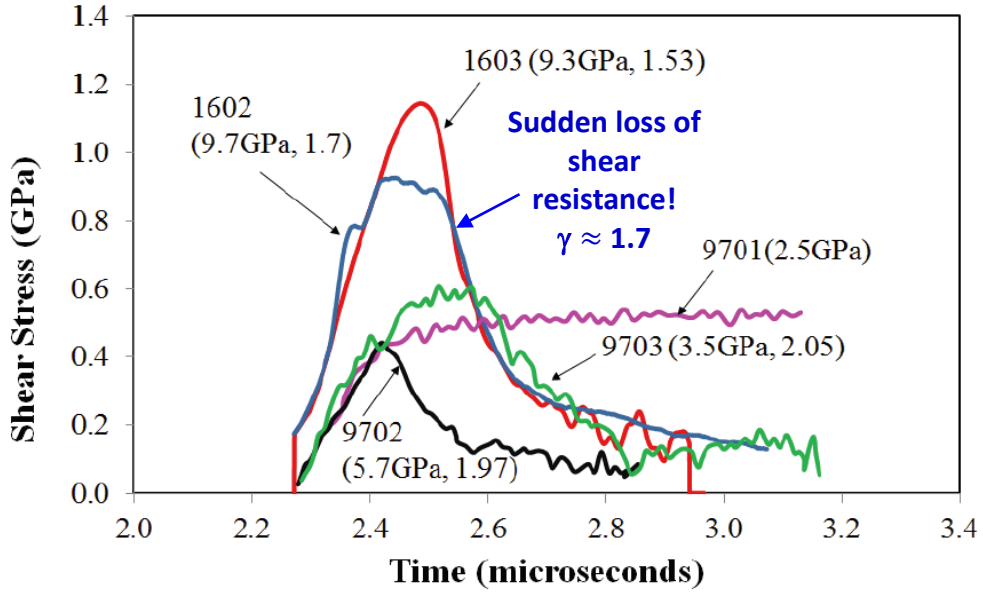
What is the constitutive law of the glassy phase?

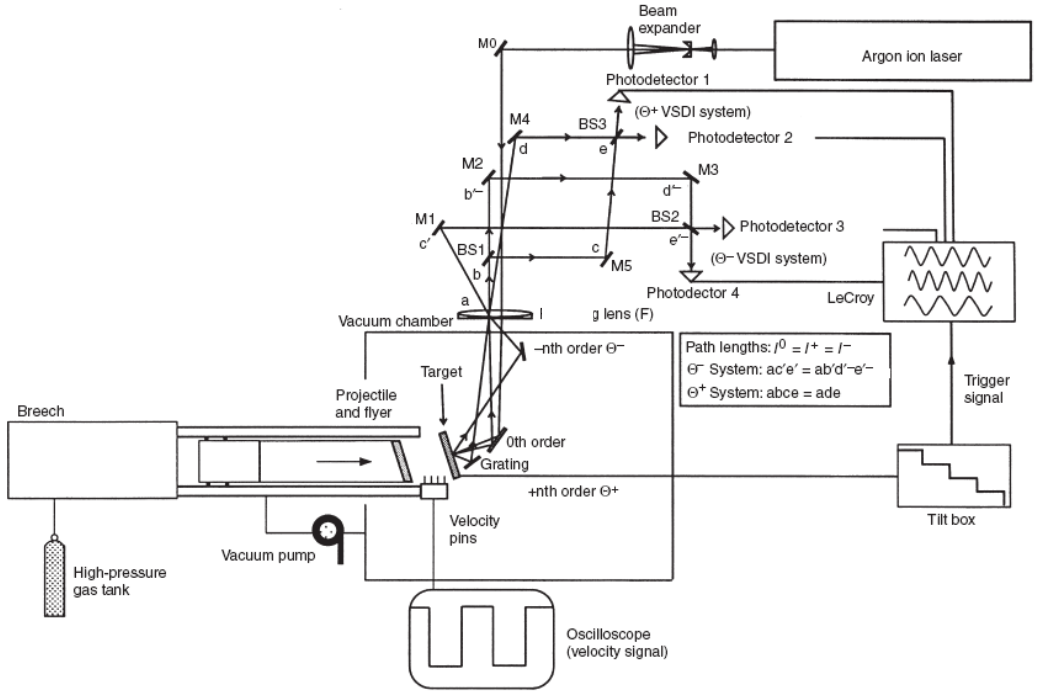
Zavattieri and Espinosa, *Acta Materialia*, Vol. 49, 2001

HSR Compression-Shear Response of Thin Wafers of Silicate Glass



Clifton and Klopp, ASM Metals Handbook, 1985





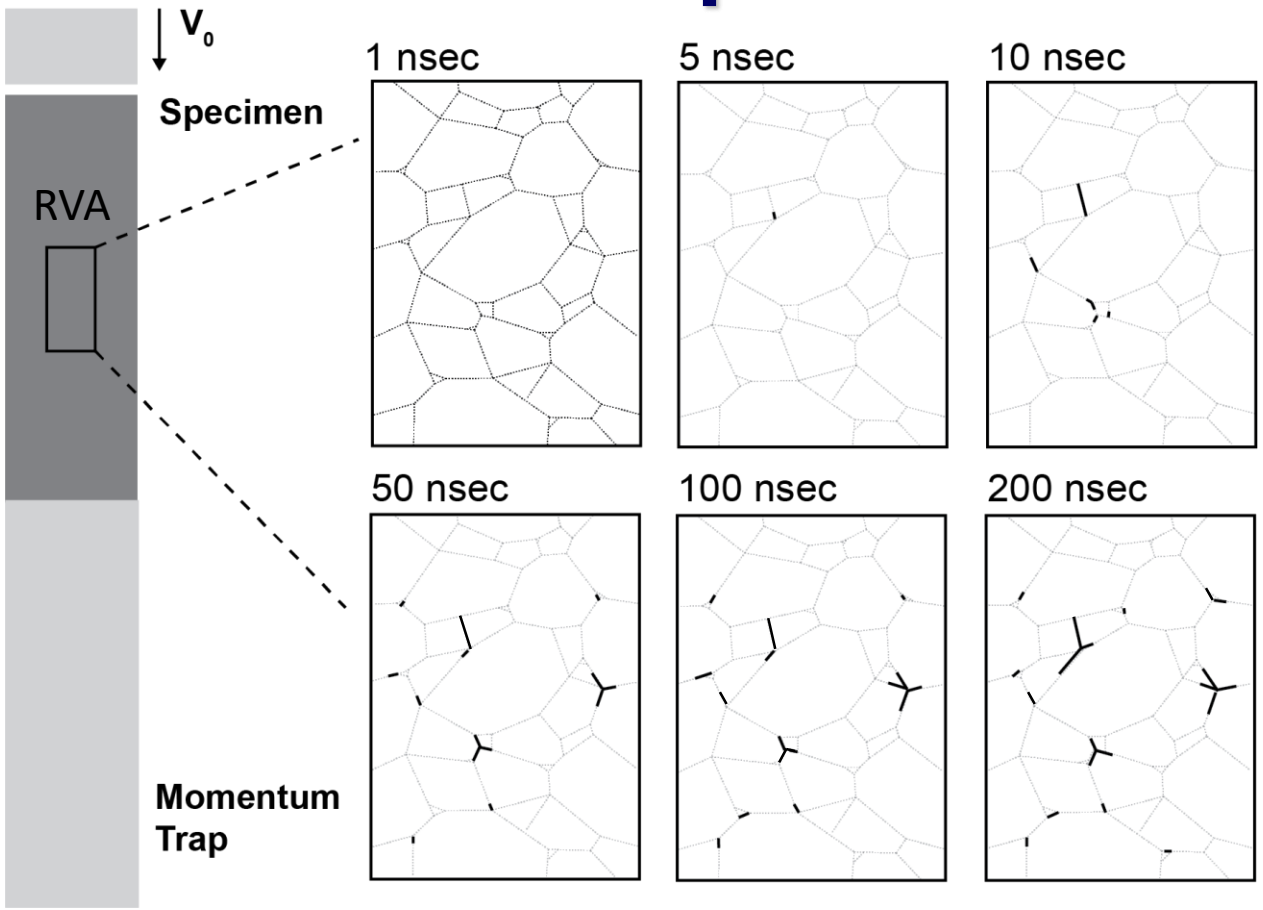
Espinosa et al. J. Am. Ceram. Soc., 80, 1997

Exp. No.	α	V_0 (m/s)	Pressure (GPa)	Shear Stress (GPa)
91-08	18°	212	11	0.88
91-11	15°	162	8.5	0.85
91-10	18°	116	6	0.7

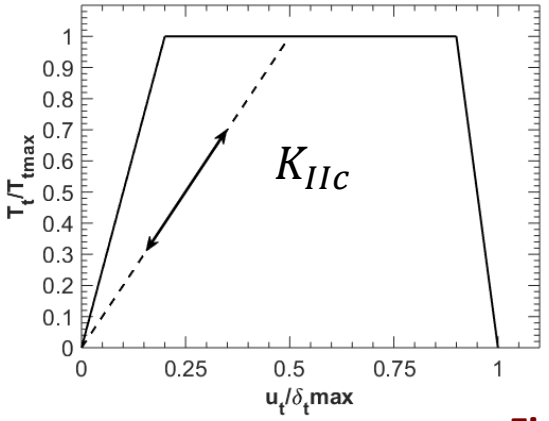
The molecular mechanism remains unknown!

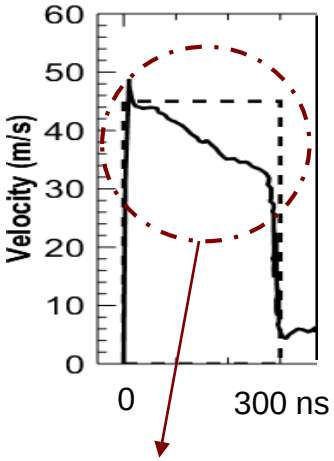
S. Sundaram and R.J Clifton, "Flow Behavior of Soda-lime Glass at High Pressures and High Strain Rates" AIP Conference Proceedings, 1998.
T. Jiao, C. Kettenbeil, G. Ravichandran, and R.J. Clifton, "Experimental Investigation of the Shearing Resistance of Soda-Lime Glass at Pressures of 9 GPa and Strain Rates of 10^6 s^{-1}, AIP Conference Proceedings, 2018

Compressive Pulse Attenuation and Formation of Triple Junction Microcracks

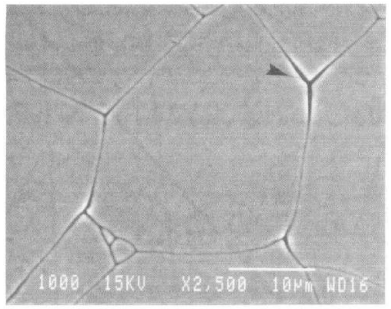


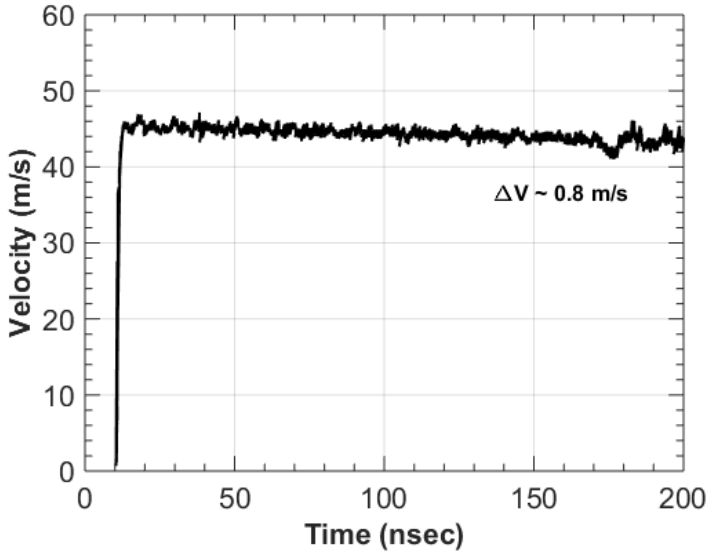
Transverse component of the cohesive law





First Compressive Pulse





Mesoscale as a link between atomistic and continuum constitutive laws

Zavattieri and Espinosa, *Acta Materialia*, Vol. 49, 2001

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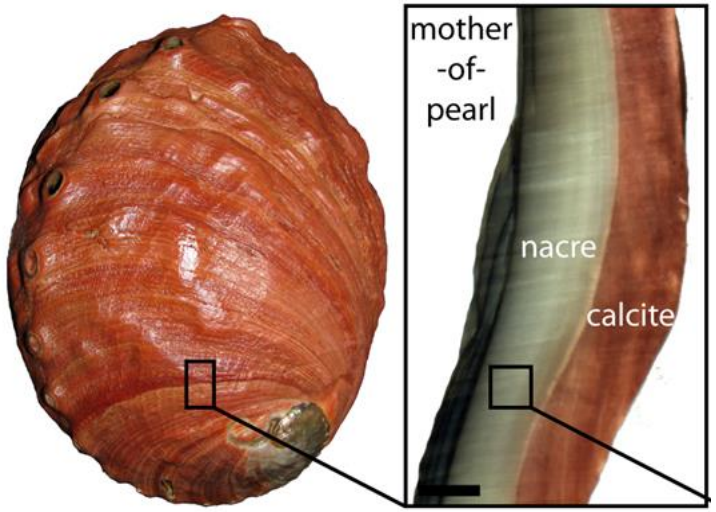
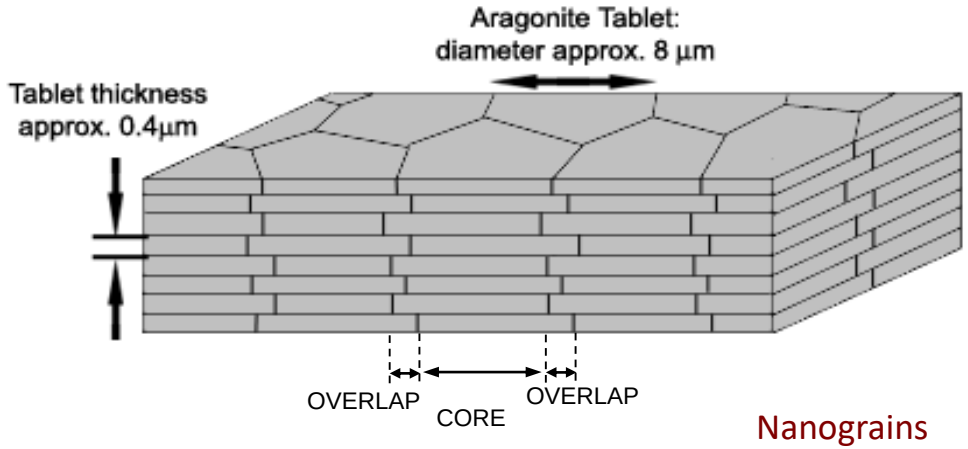
Sea Shells Hierarchical Structure

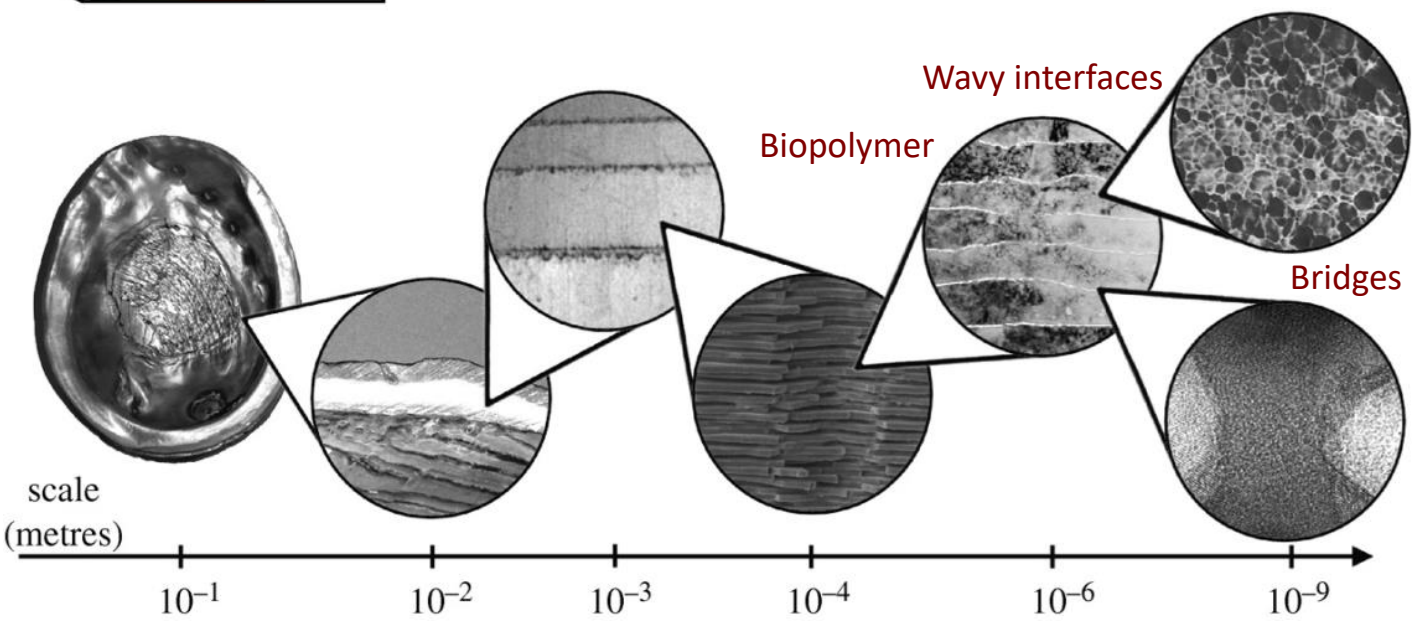
Francois Barthelat

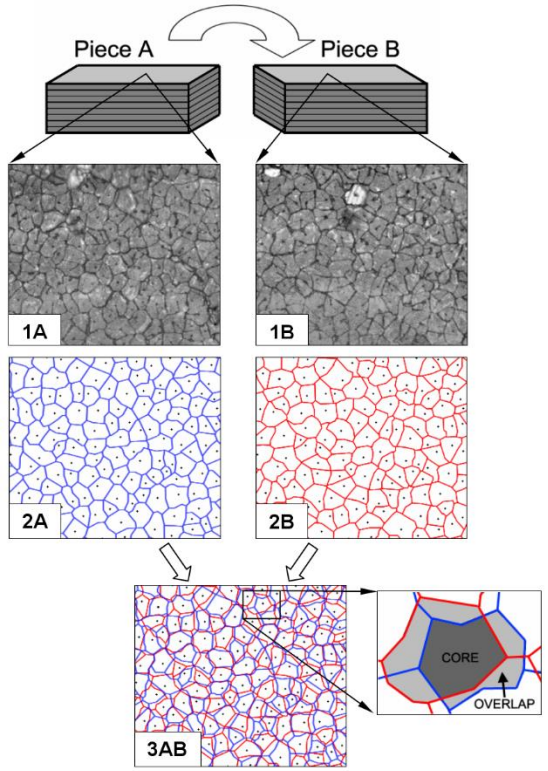
Red abalone (*Haliotis Rufescens*)



Nacre: a composite with 95% ceramic and 5% biopolymer

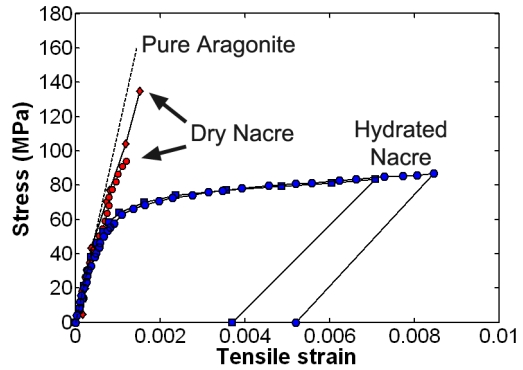


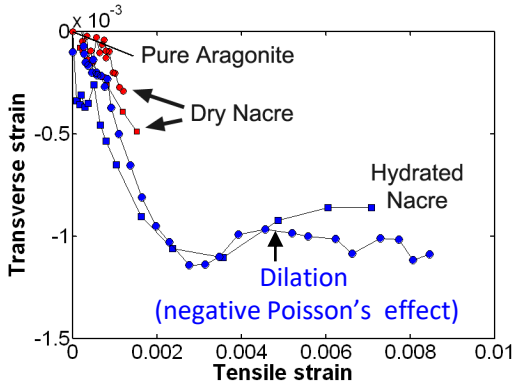




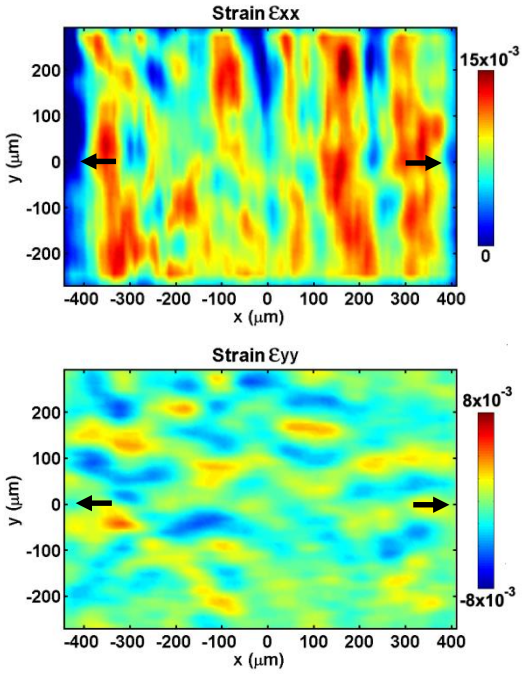
Barthelat, Espinosa et al., *JMPS*, 2007

Tensile Behavior of Nacre

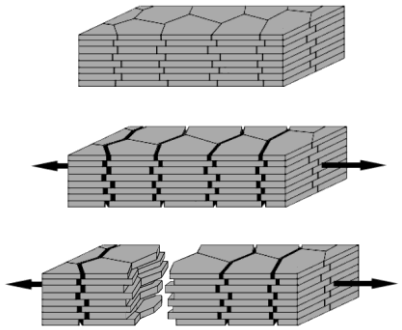




Strain fields from image correlation

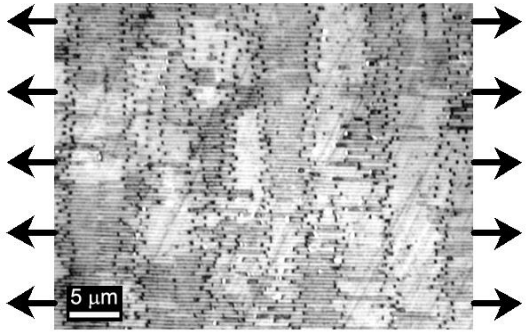


Main mechanism: Tablet sliding



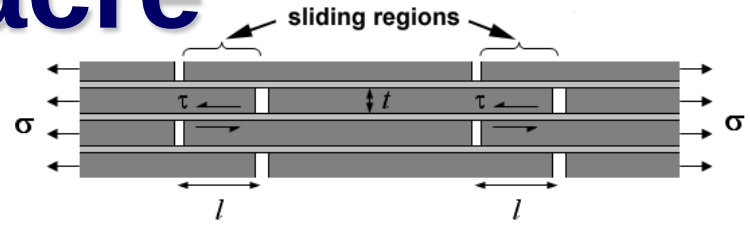
Barthelat, Espinosa et al., *JMPS*, 2007

Optical Image



Diffusive sliding

Shear lag model



For given material properties (E and G) and geometry (h and b), *the elastic strain energy* is maximized when $\lambda L \sim 3.278$, i.e., when:

$$L = L^* \sim 3.278/\lambda \sim 2.318\sqrt{\frac{Ehb}{G}} \quad ; \quad \lambda = \sqrt{\frac{2G}{Ehb}}$$

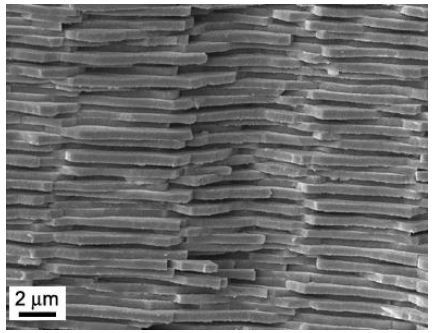
For tablet *fracture* simultaneously with the *shear failure of matrix:*

$$L^{**} = \frac{\sigma_f}{\tau_f} b$$

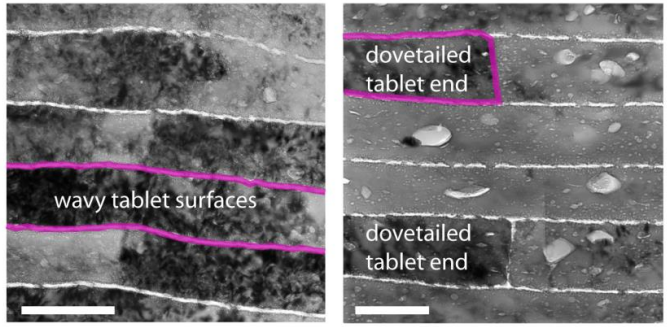
Nacre => $L^* \sim 1.59$ μm ; $L^{**} \sim 1.69$ μm

X. Wei, M. Naraghi, and H.D. Espinosa, *ACS Nano*, 2011
Gao H., et al., *PNAS*, 2003

Fracture surface (SEM)

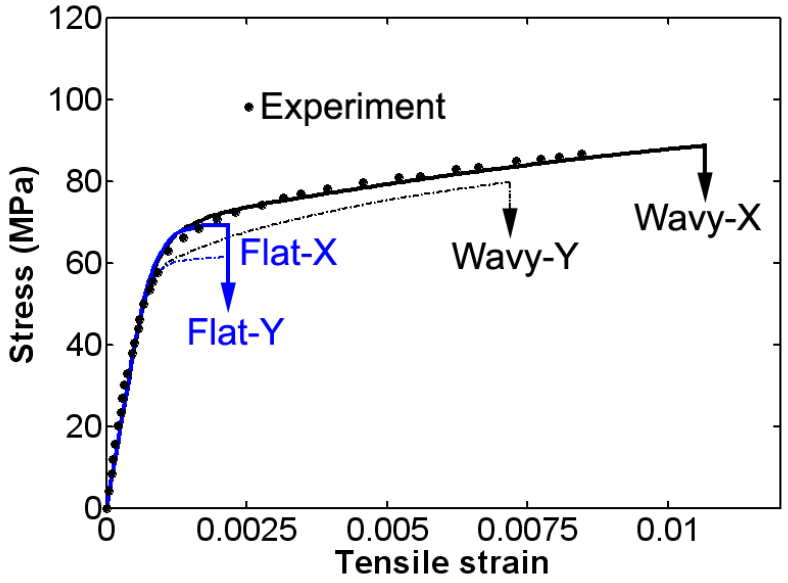


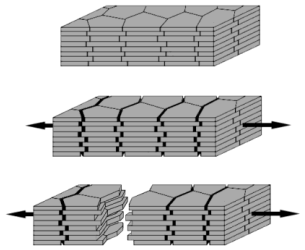
Wavy interfaces (TEM)

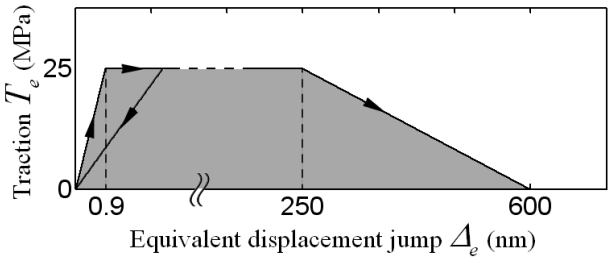


- What is the mechanism for diffusive sliding?
- What is the dilation source?

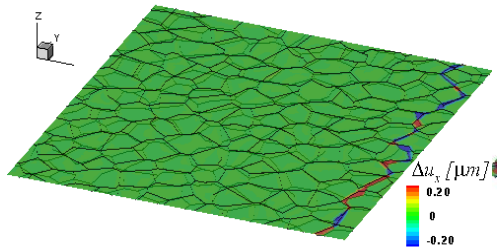
Exploring the Interface Waviness Hypothesis with a Mesoscale Model



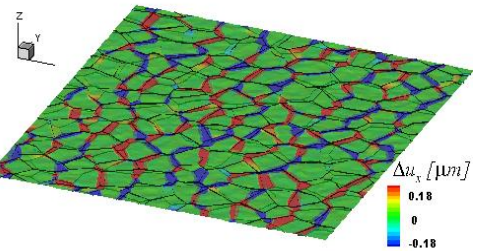


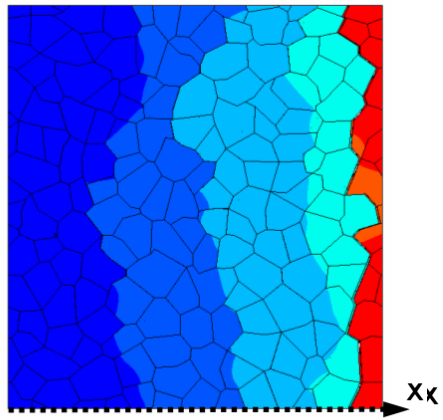


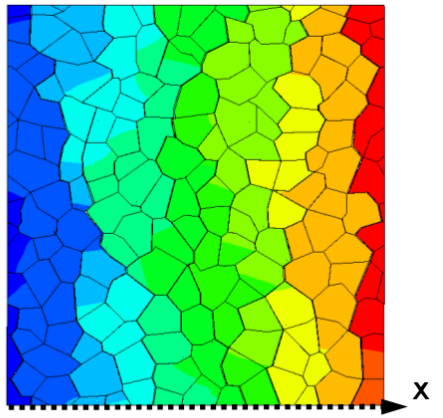
Flat-X

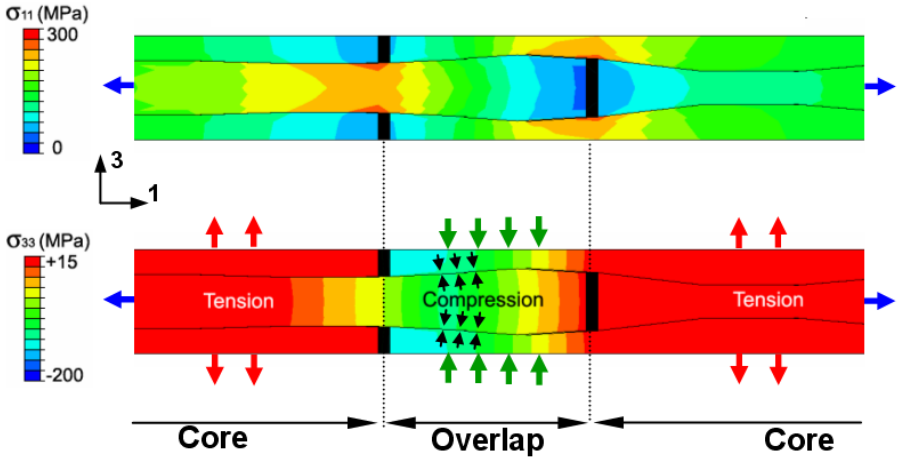


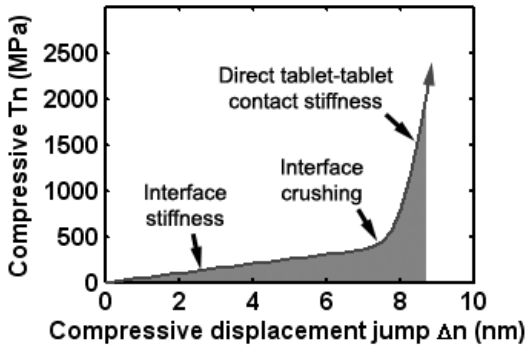
Wavy-X

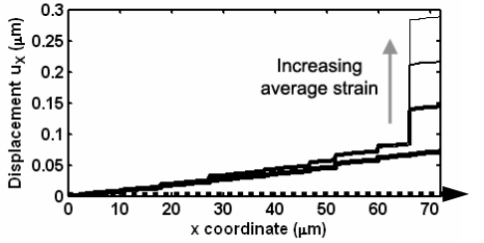


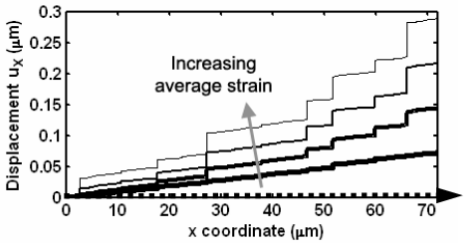










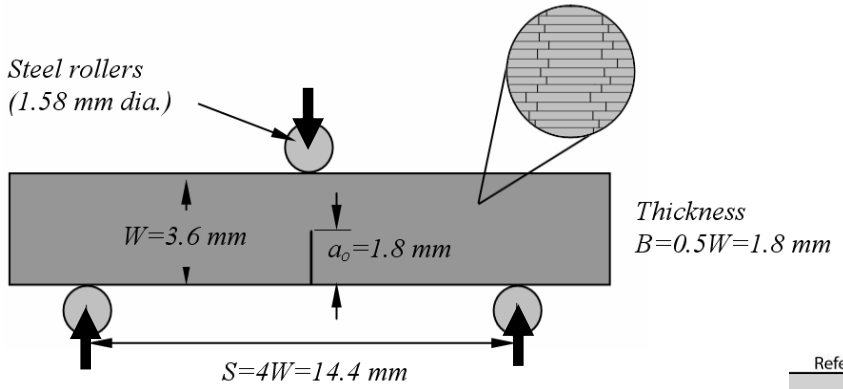


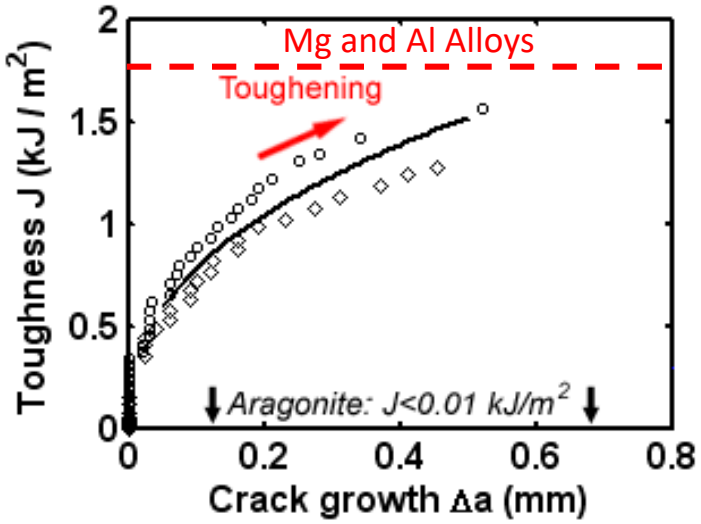
Barthelat, Tang, Espinosa et al., *JMPS,* 2007

Diffusive tablet sliding is predicted

How Does Diffusive Sliding Impact Toughness?

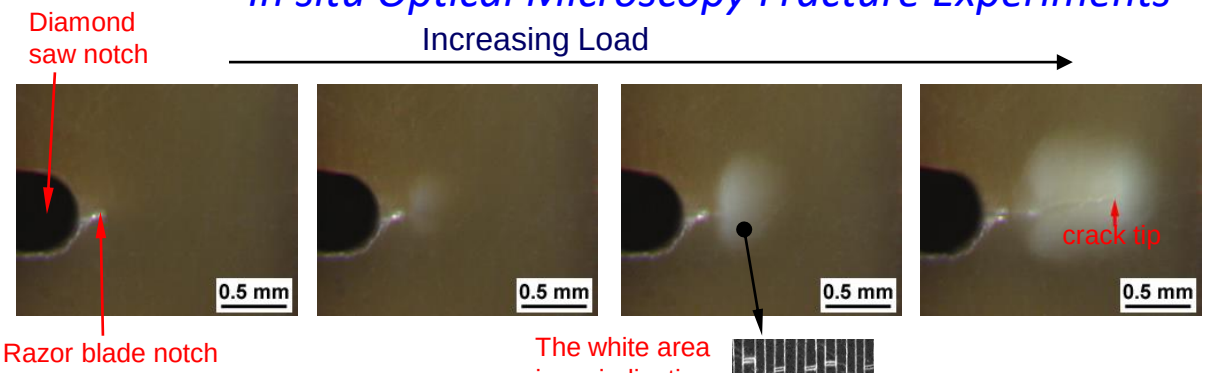
Single Edge Notched Bent (SENB) specimen (ASTM E-1820)





Barthelat and Espinosa, *Exp. Mech.*, 2007

In situ Optical Microscopy Fracture Experiments



The white area is an indication of tablet sliding

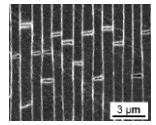
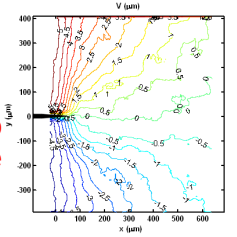
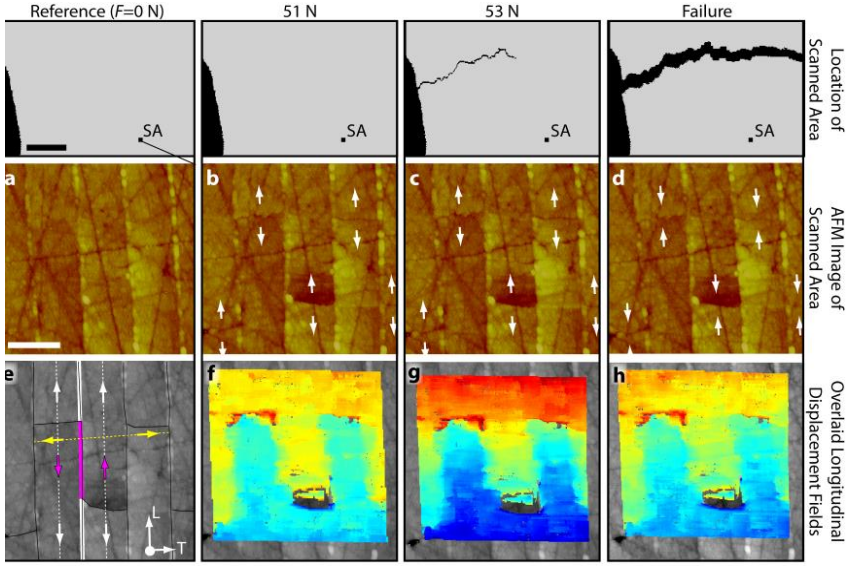
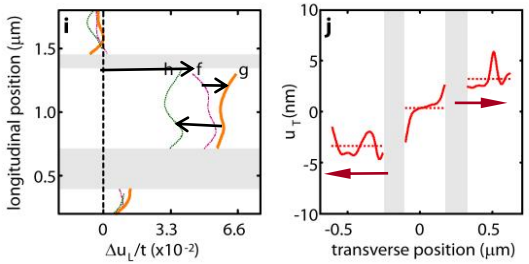


Image correlation was used to monitor crack advance





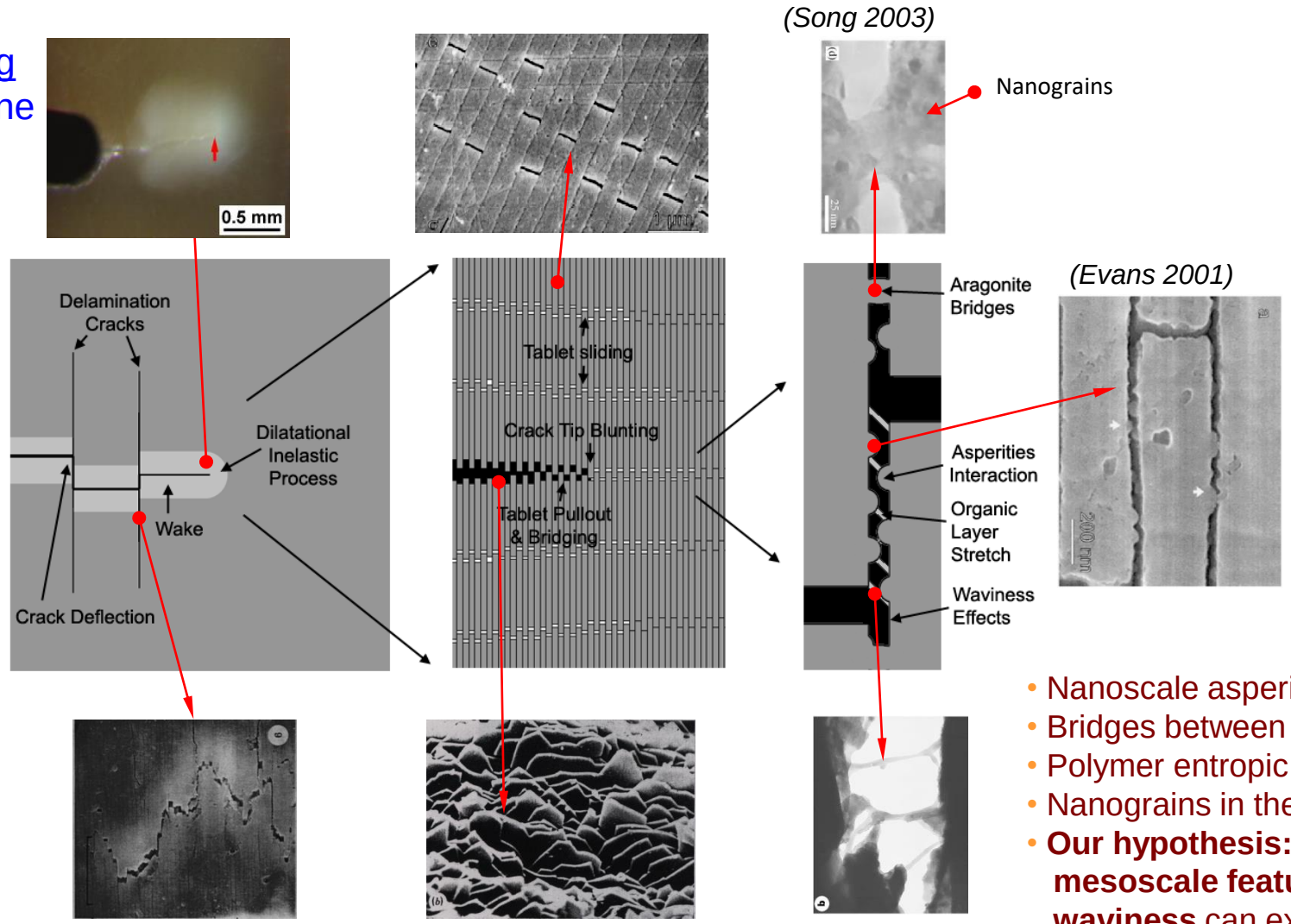


In situ AFM Fracture Experiments
Tablet sliding and
Dilation at crack tips

Espinosa et al., *Nature Communications*, Vol. 2, 2011

What Makes Nacre Tough?

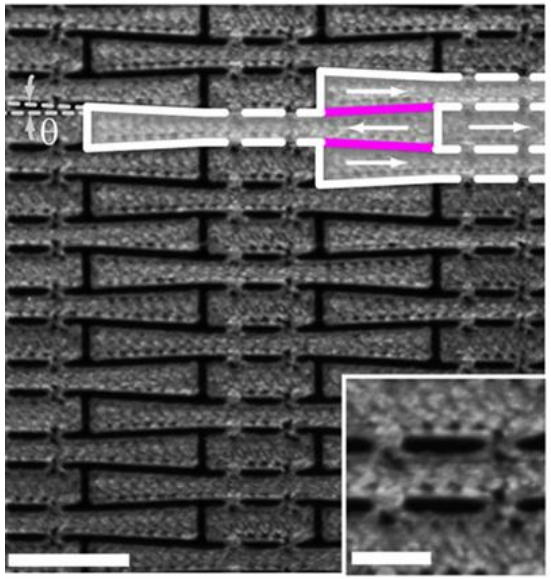
Diffusive tablet sliding
Millimeter Process Zone

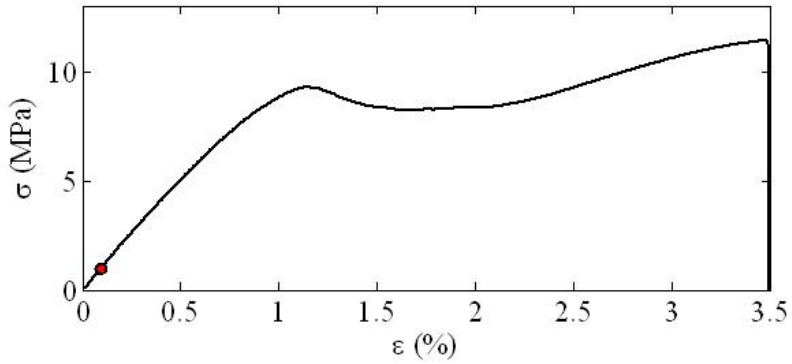


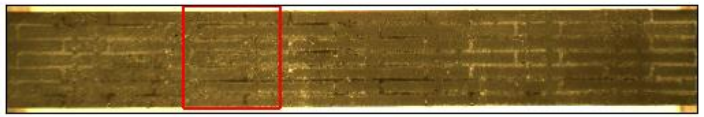
- Nanoscale asperities (Evans)
- Bridges between tablets (Meyers)
- Polymer entropic hardening (Boyce)
- Nanograins in the tablets (Li)
- **Our hypothesis:**
  mesoscale features including tablet waviness can explain high diffusive damage and toughness

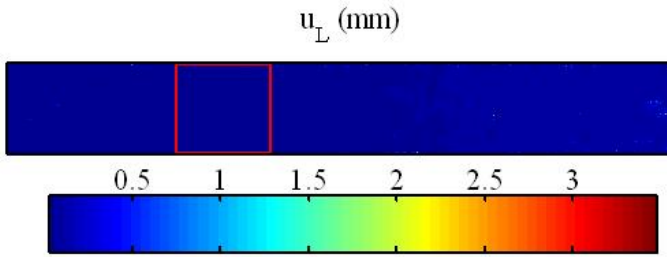
Wegst, Ritchie, et al, Nat. Materials, 2015

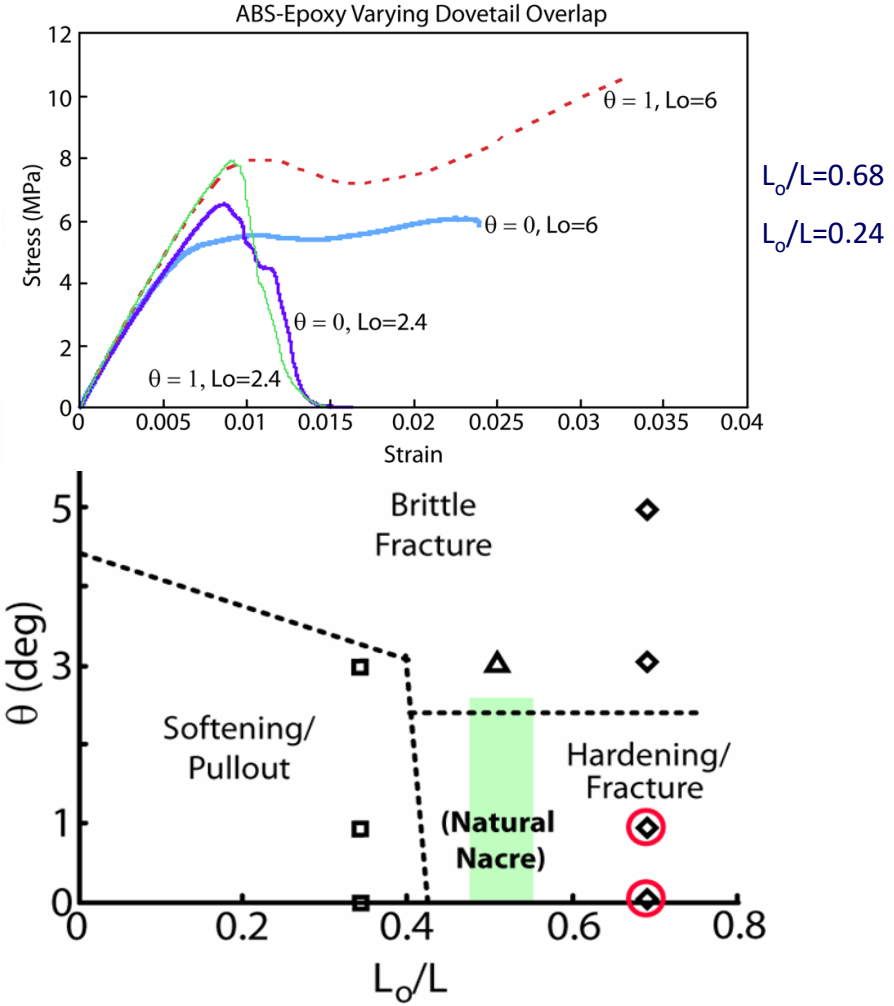
Additive Manufacturing of Nacre-like ABS-Epoxy Composite









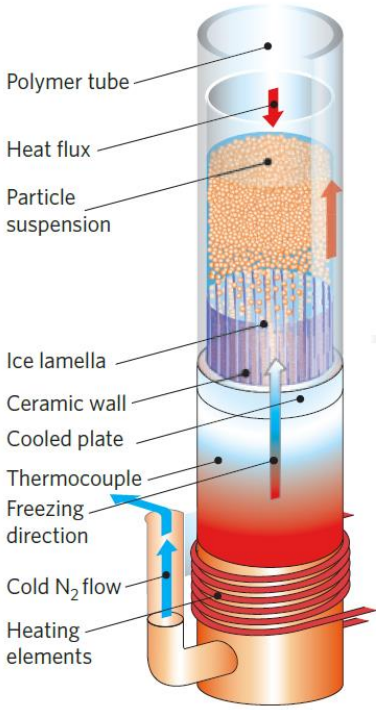


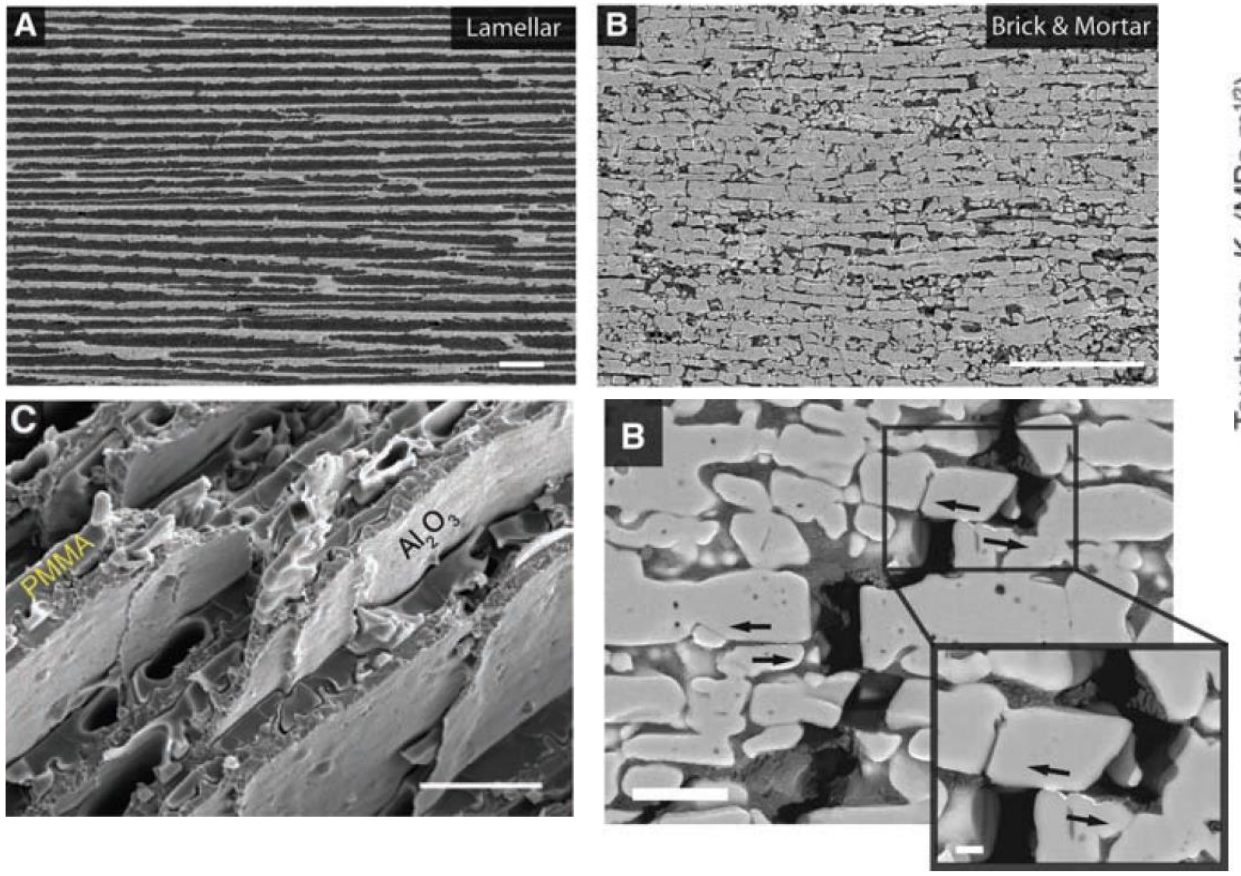
Espinosa, Zavattieri, et al., *Nature Communications*, Vol. 2, 2011

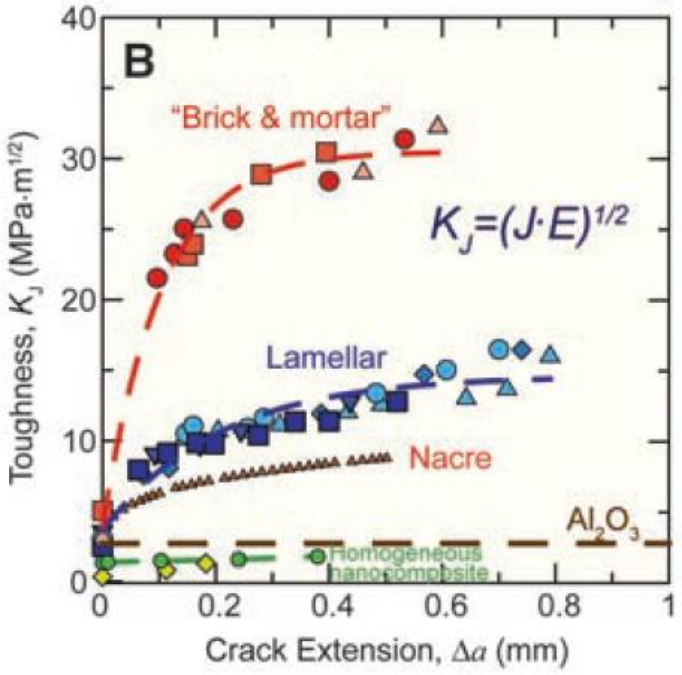
Processing of Nacre-like Tough Composites - Freeze Casting

Munch, Ritchie et al., *Science,* Vol. 322, 2008

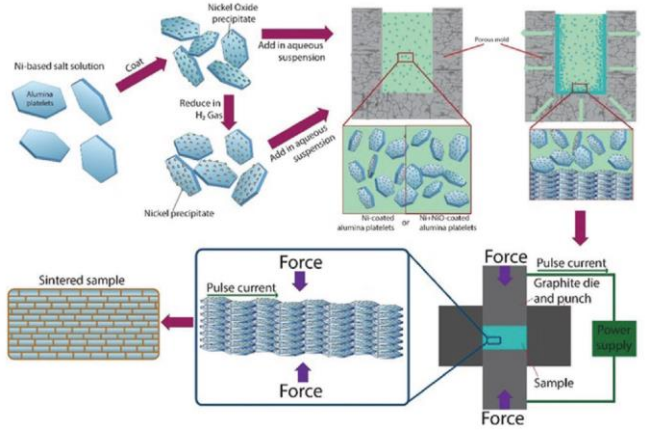
Al_2O_3/PMMA







Ni-coated Al_2O_3: K_J=20 MPa m$^{1/2}$

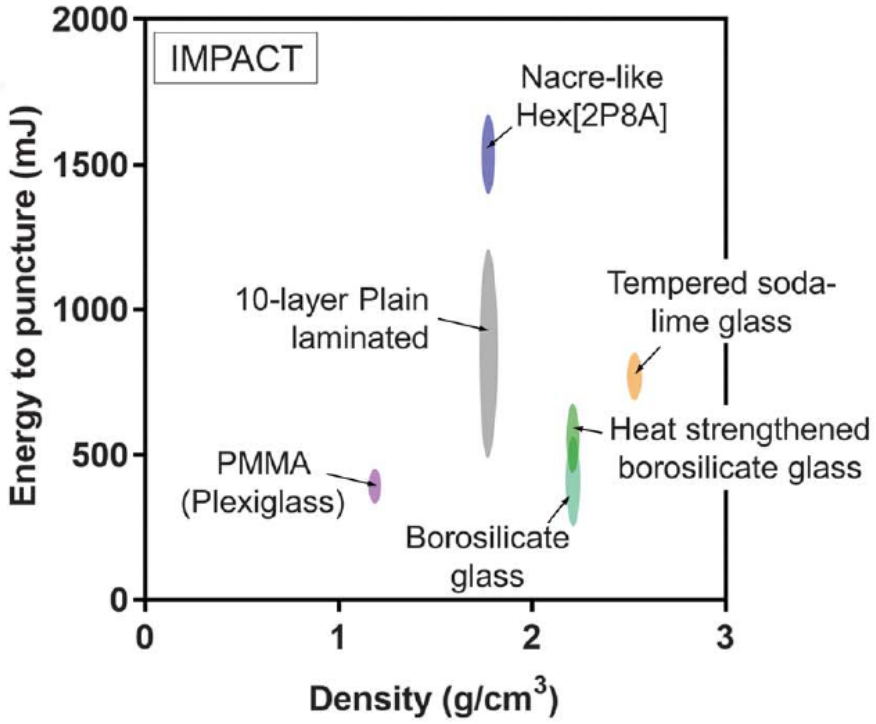


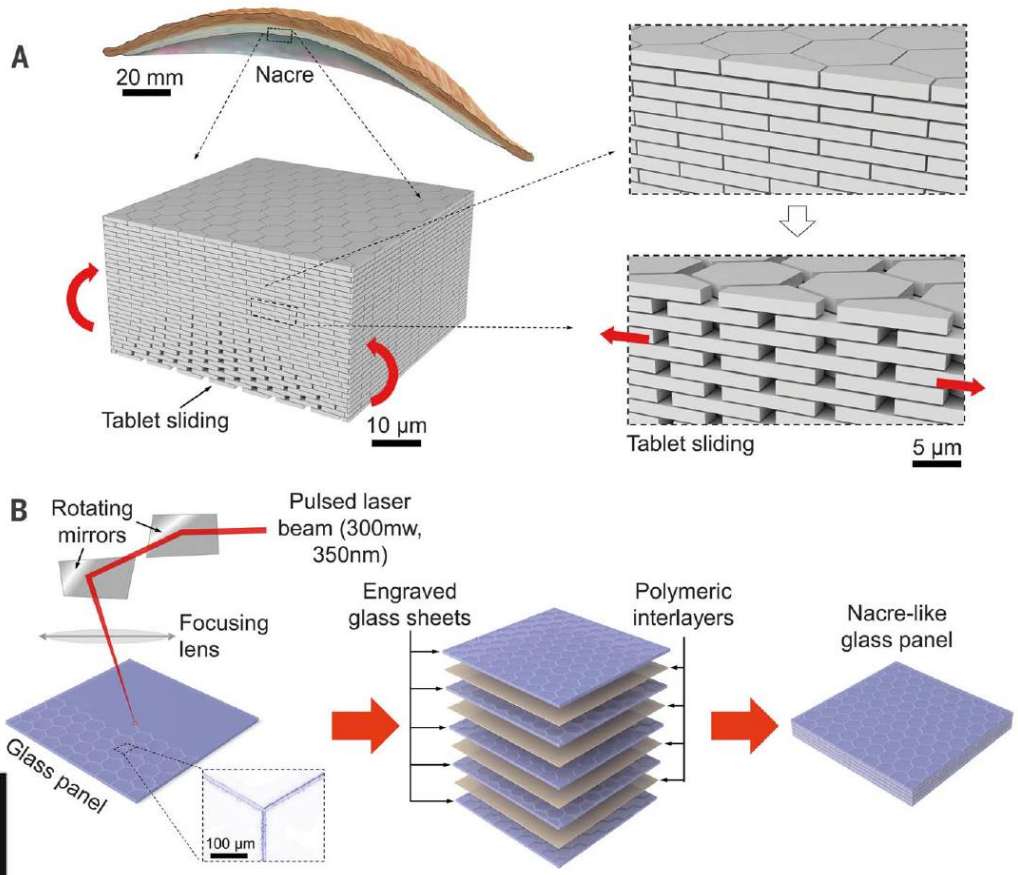
- Control of architecture is limited
- Diffusive mechanism not observed
- Ceramic phase (~65 vol.%) much lower than in Nacre
- Much thicker polymer interlayers (cannot exploit nanoscale effects)

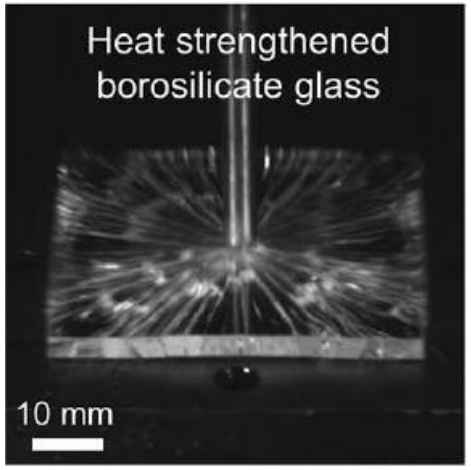
Wat, Ritchie et al., *Small*, 15, 2019

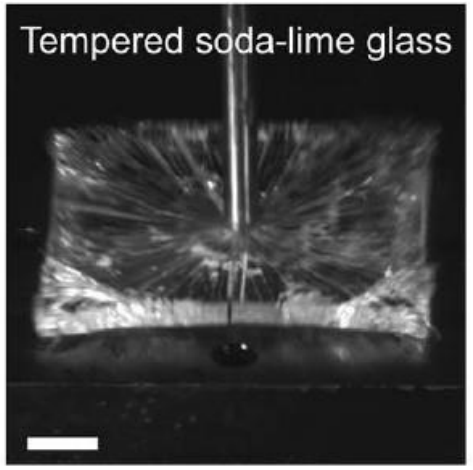
Impact Resistance Nacre-Like Glass

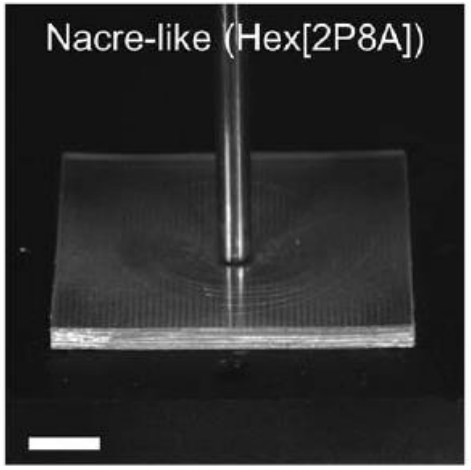
F. Barthelat











Z. Yin, F. Hannard, F. Barthelat, *Science, 2019*

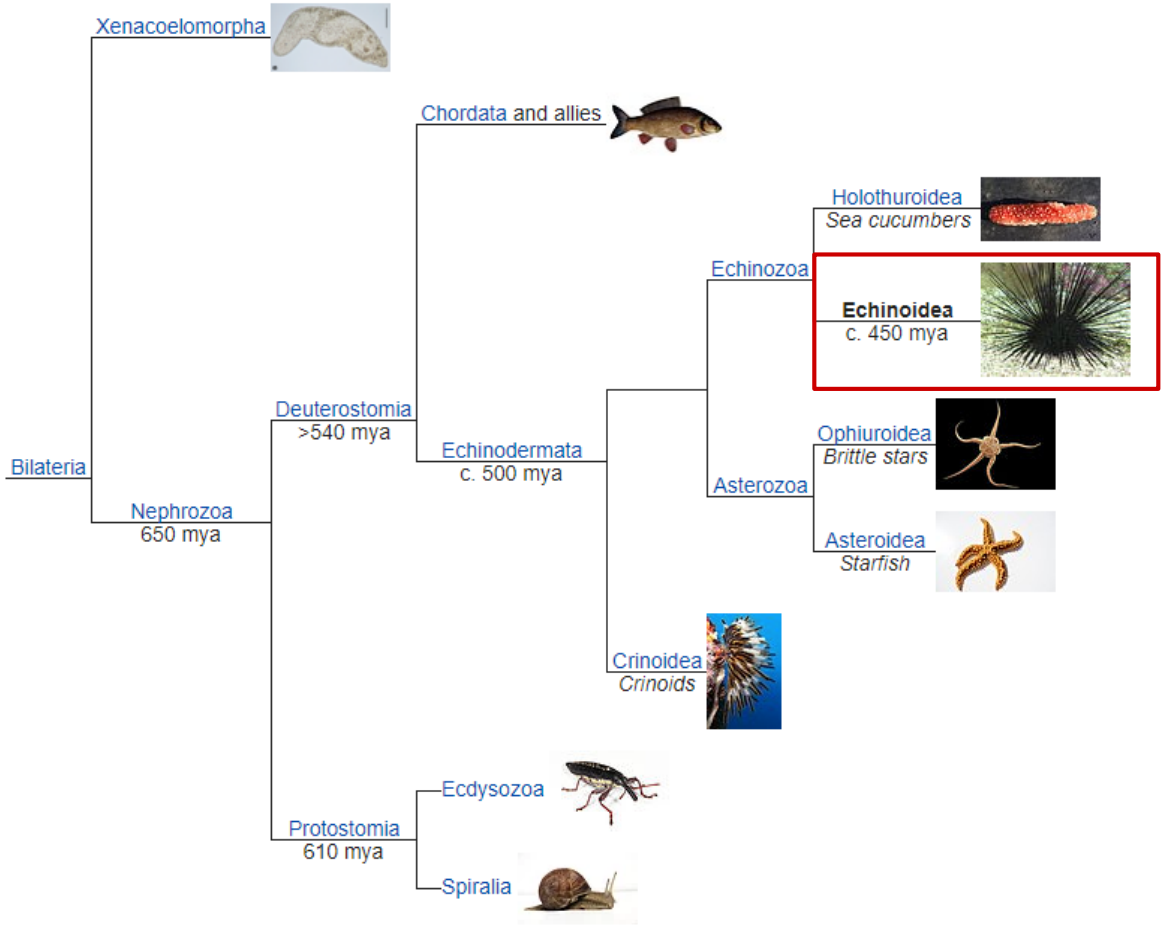
Beyond Brick and Mortar

Natural Ceramic Composites with More Complex Spatial Structures

The Pink Sea Urchin

- Have heavily mineralized teeth in a jaw with a dome form
- The jaw is known as **Aristoteles's apparatus**
- Teeth present a complex architecture of fiber and laminated composites

A. Zaheri

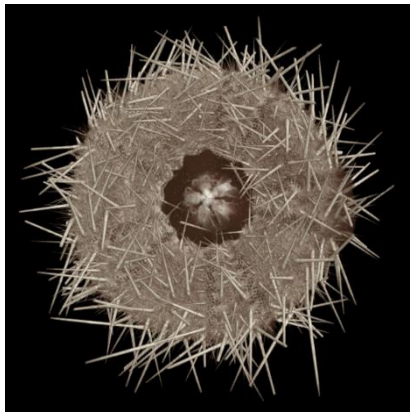


Sea Urchin Phylogeny

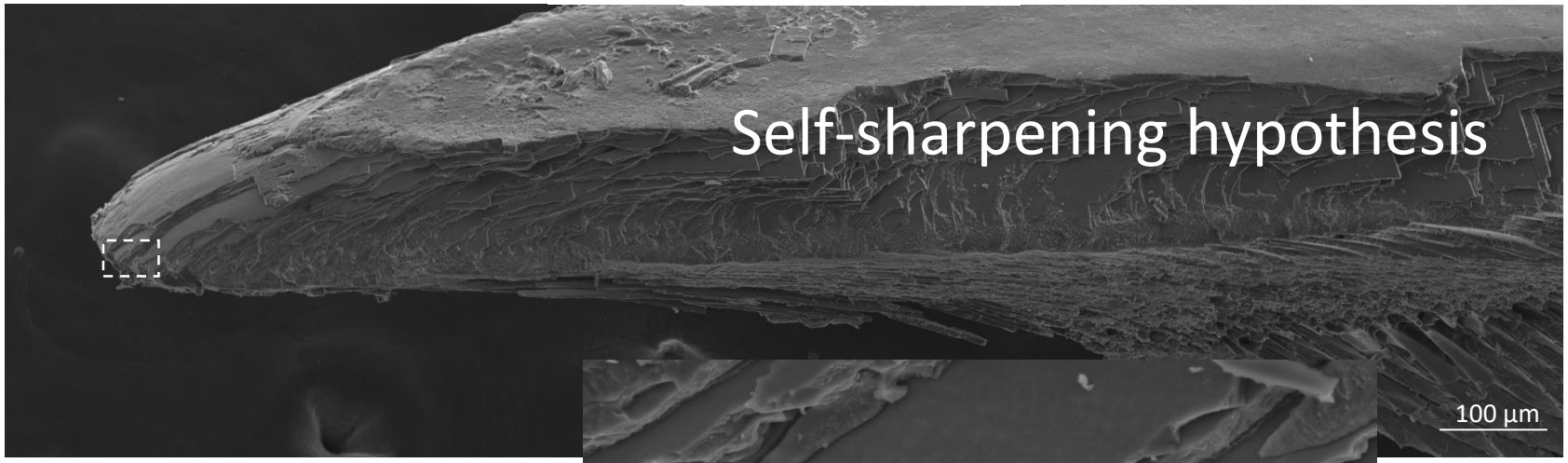
In collaboration with J. Mckittrick

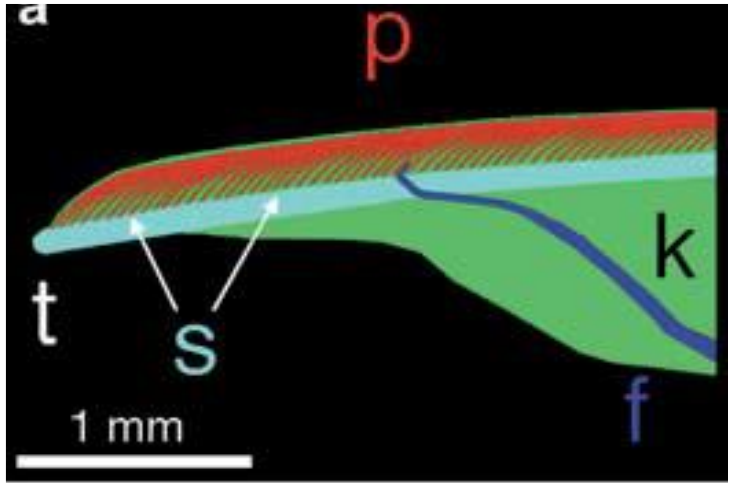
Frank, M. B., et al. *JoVE (Journal of Visualized Experiments)*, 110, 2016

Pink Sea Urchin Tooth Structure

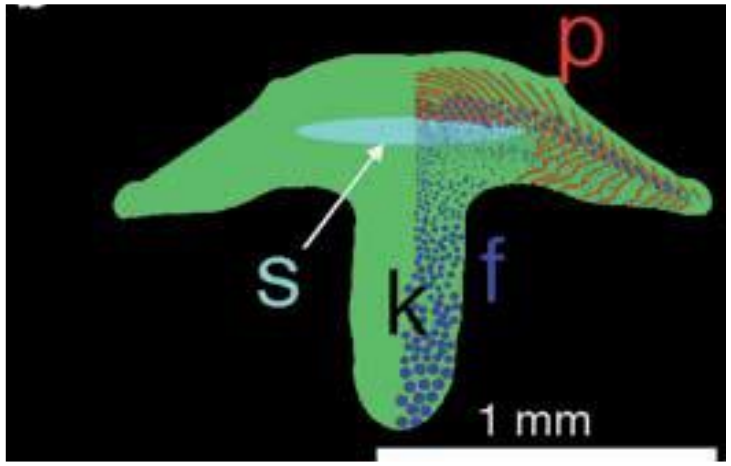


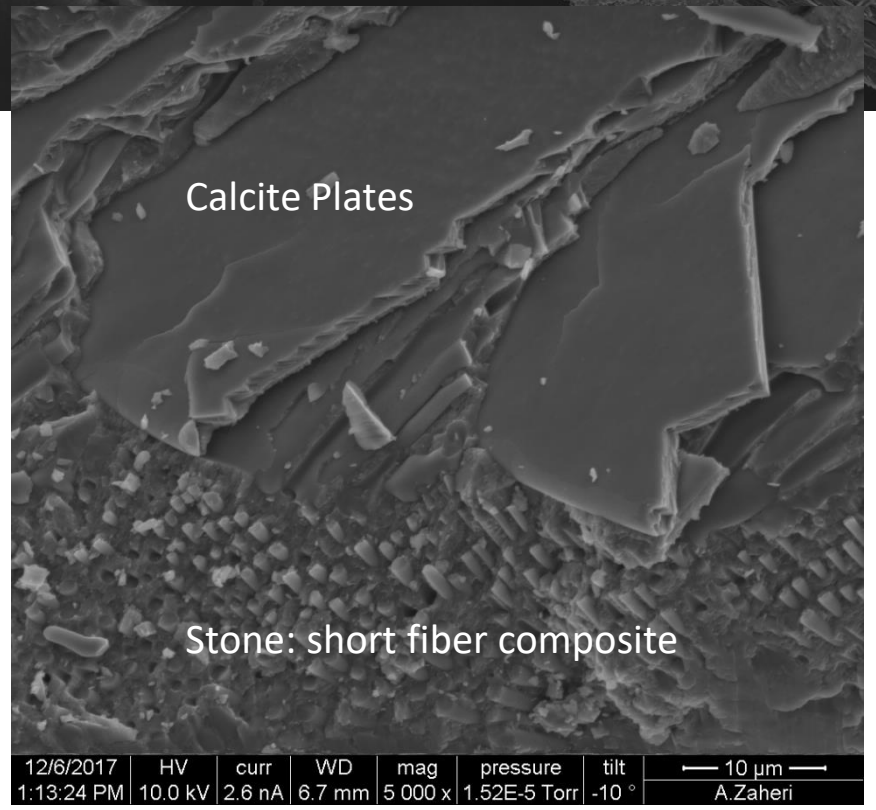
Animal's jaw and teeth





S: Stone region
p: Plate region
k: Keel region



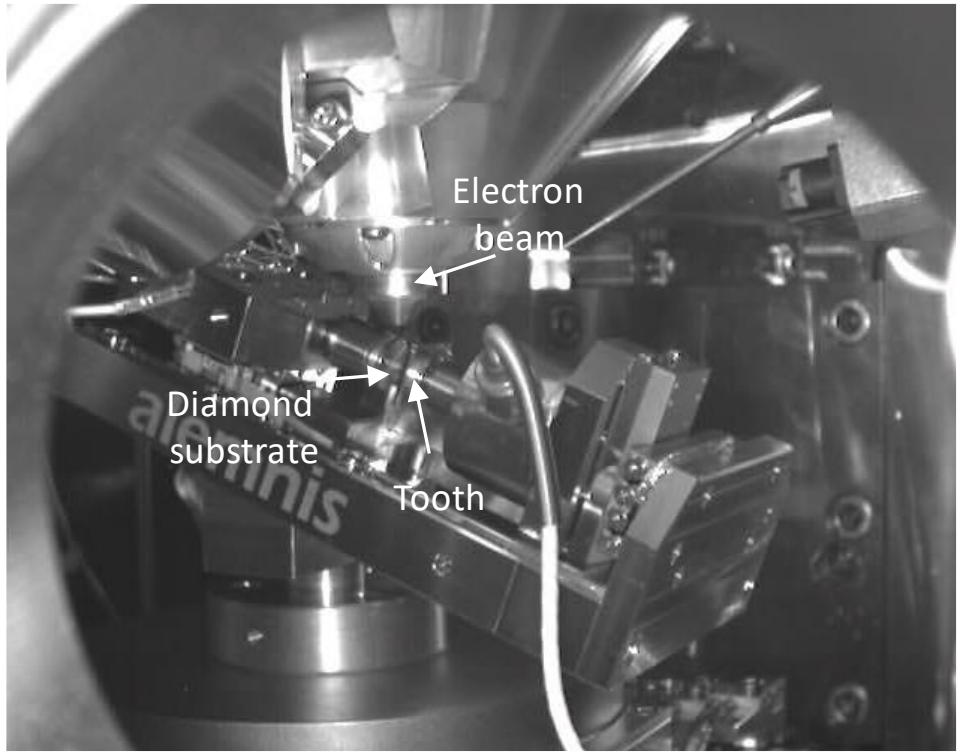


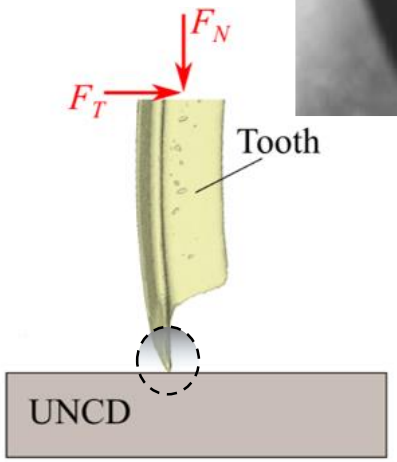
Killian et al., Adv. Funct. Mater., 21, 2011

Espinosa H.D., Zaheri A., Restrepo D., Nguyen H., Daly M., Frank M., **McKittrick J.**, *Matter, 2019*

In Situ SEM Wear Experiments

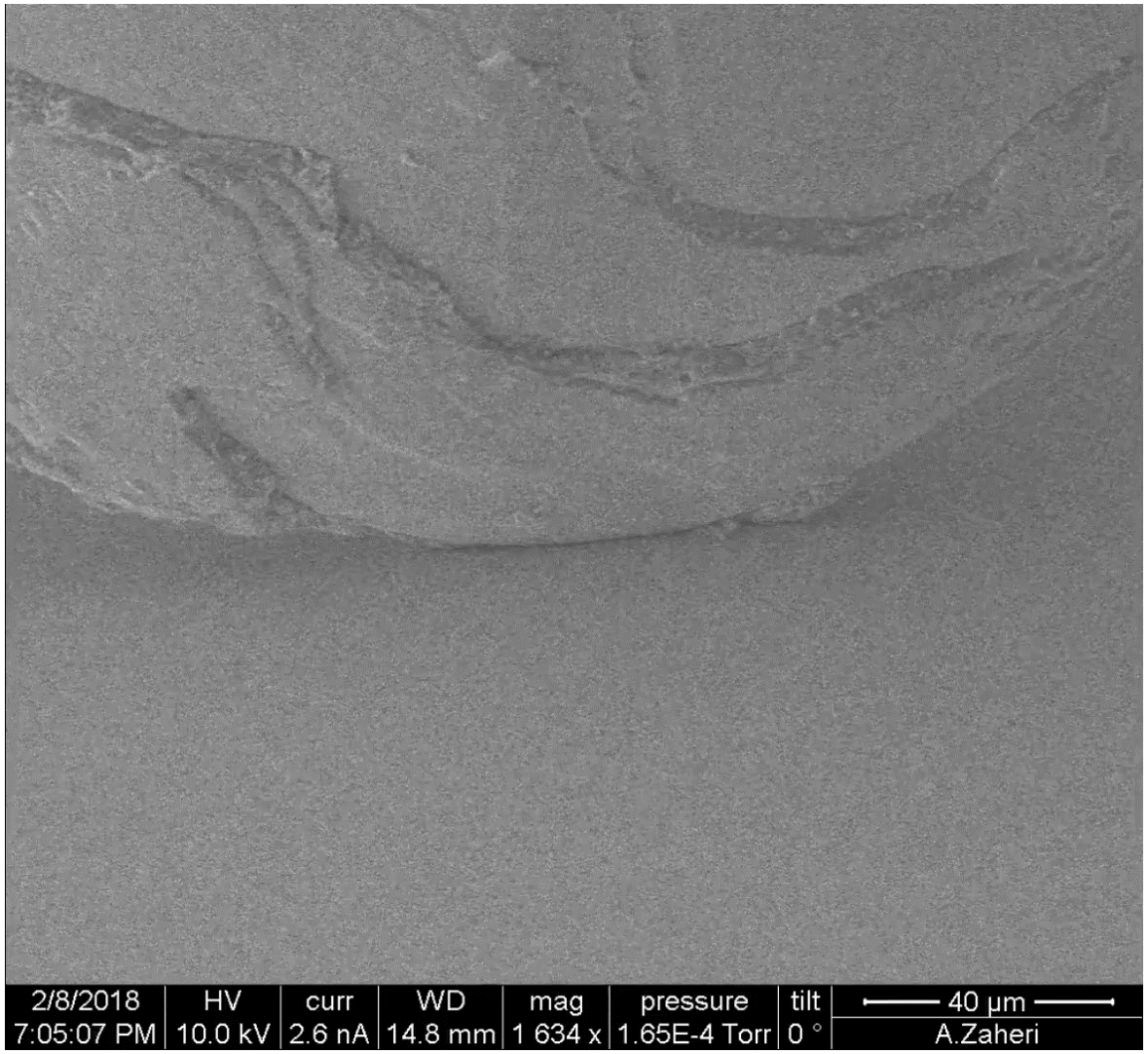
N. Alderete





Dimensional Analysis:

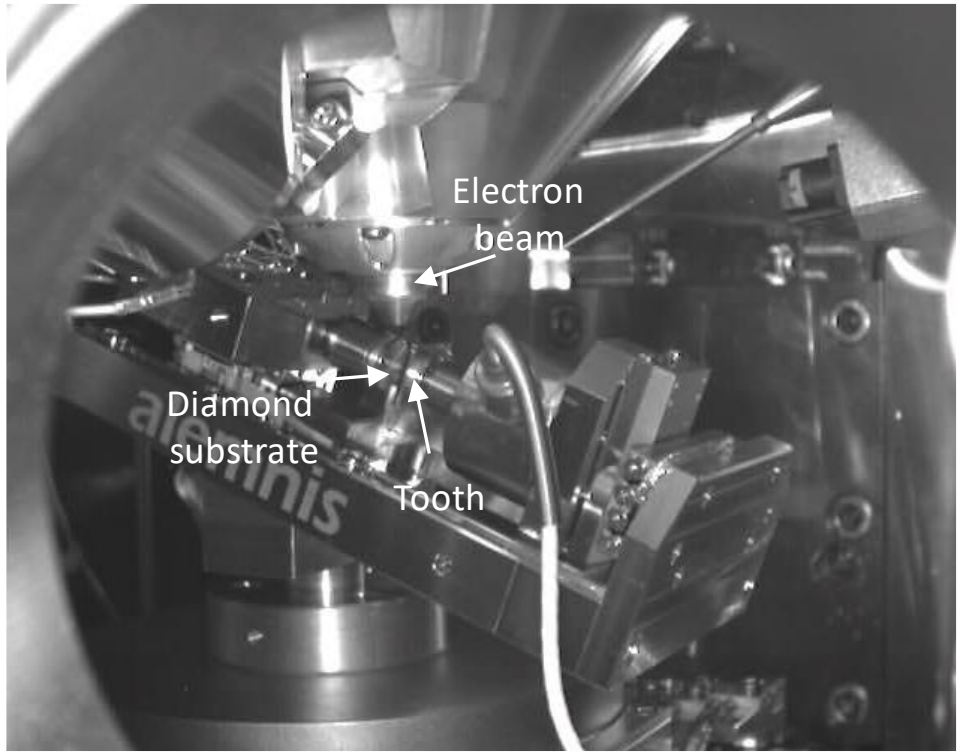
$$\frac{w^i}{a^i} = F\left(\mu, \frac{F_N}{a^2 H_{tooth}}, \frac{L}{a}\right)$$

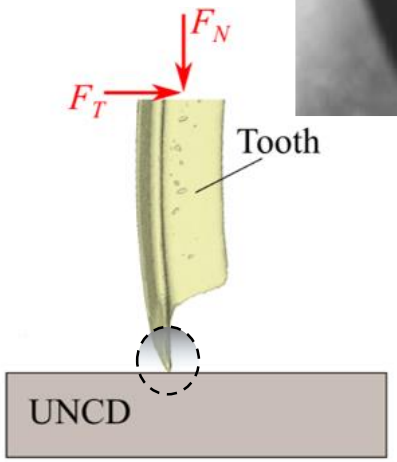


Espinosa H.D., Zaheri A., Restrepo D., Nguyen H., Daly M., Frank M., McKittrick J., *Matter,* 2019
Alderete, N., Zheri, A., Espinosa H.D., *Exp. Mechanics*, 2019

In Situ SEM Wear Experiments

N. Alderete





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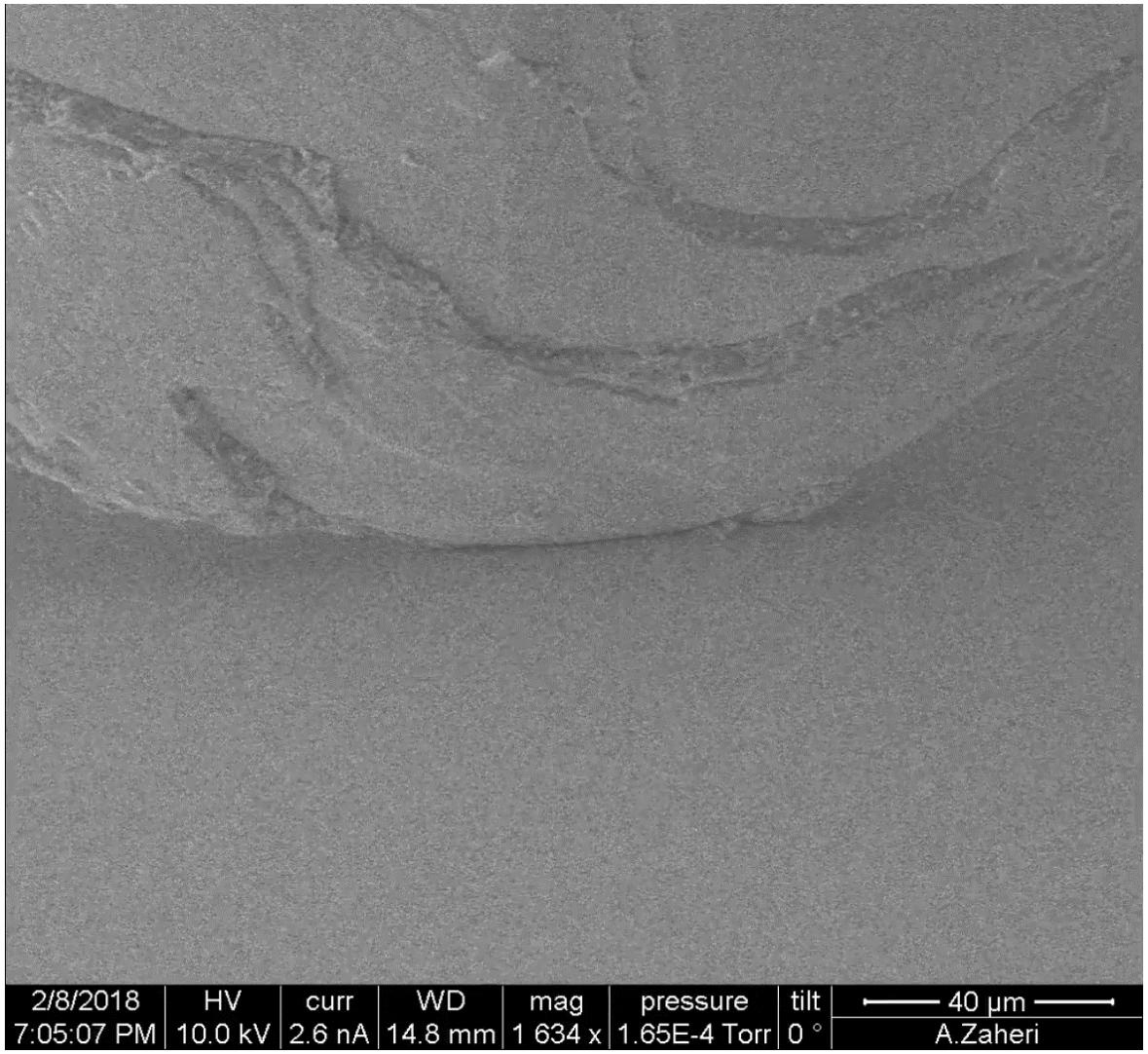
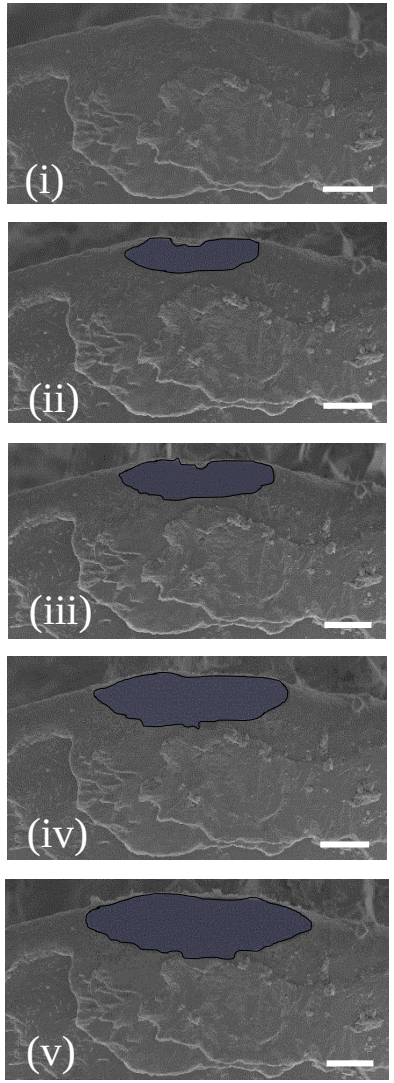


First 3D visualization of wear and plate chipping

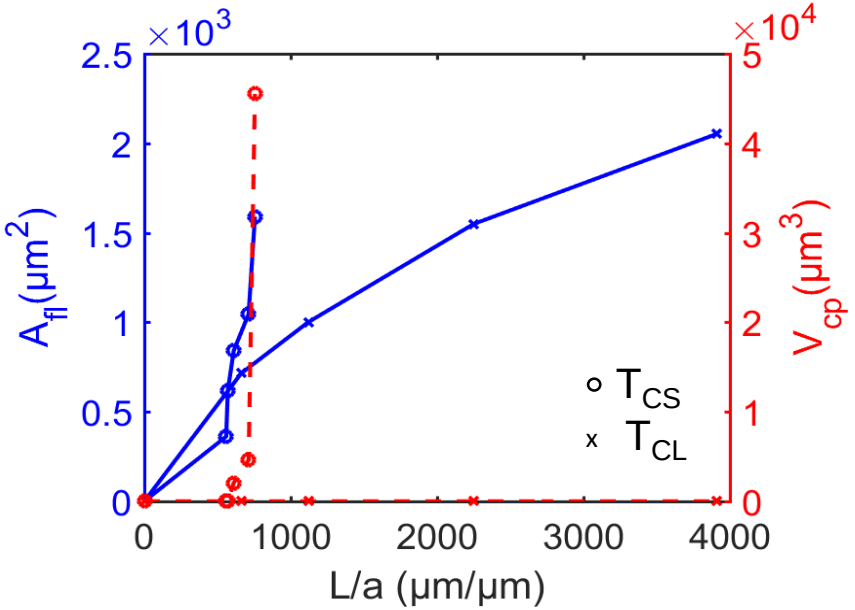
Espinosa H.D., Zaheri A., Restrepo D., Nguyen H., Daly M., Frank M., McKittrick J., *Matter,* 2019
Alderete, N., Zheri, A., Espinosa H.D., *Exp. Mechanics*, 2019

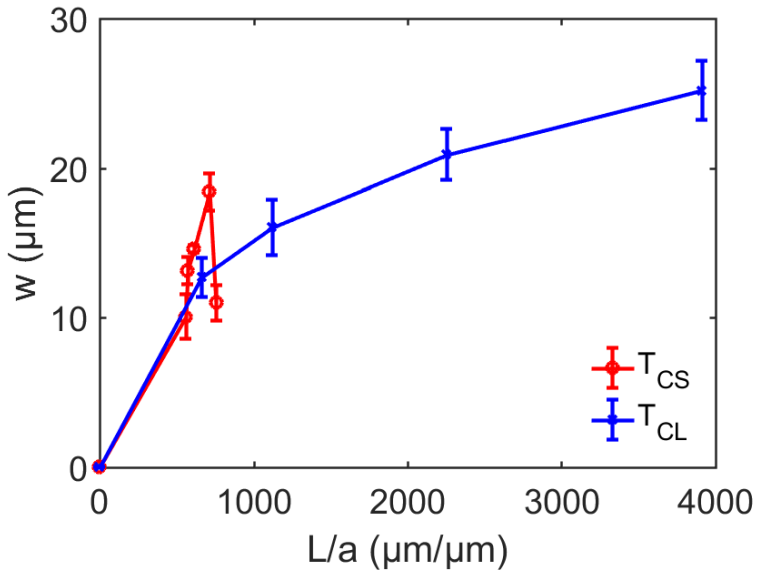
Regimes for Self-sharpening

Constant Load (CL)

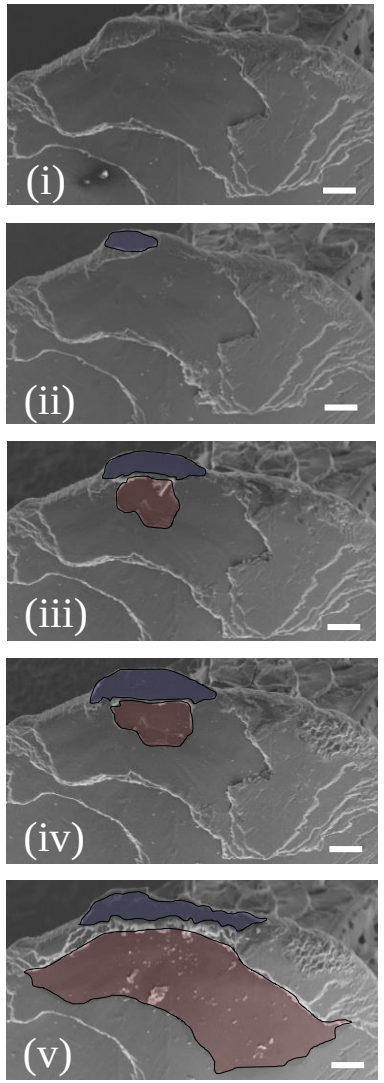


Scale bars: 20μm



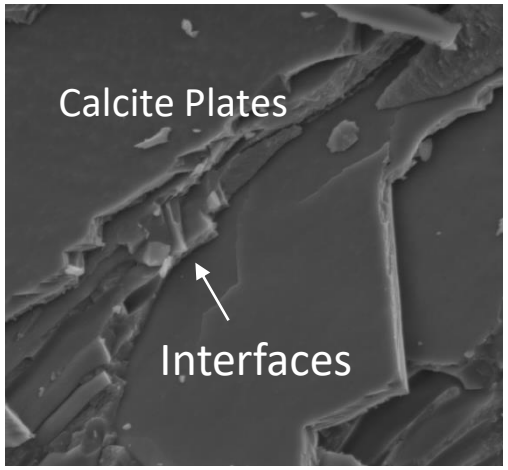


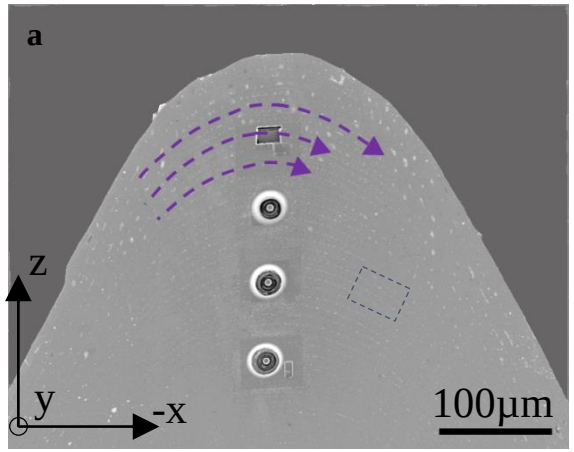
Constant Stress (CS)

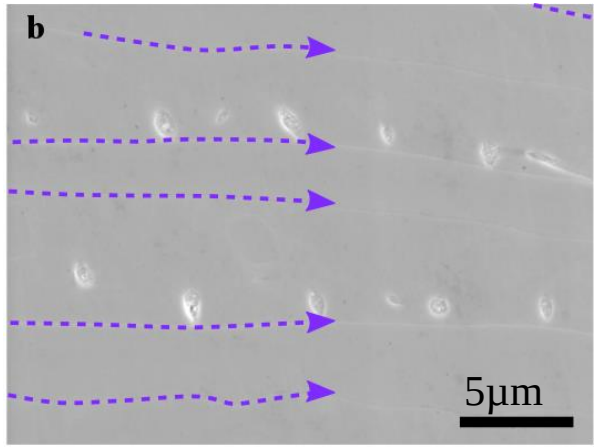


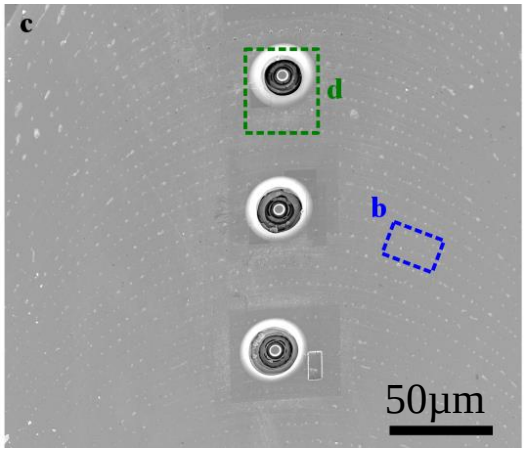
Scale bars: 20μm

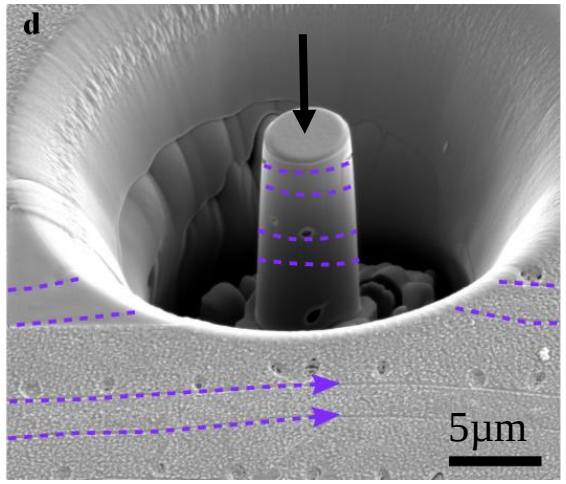
Strength of Interfaces Between Plates Nanopillar Compression

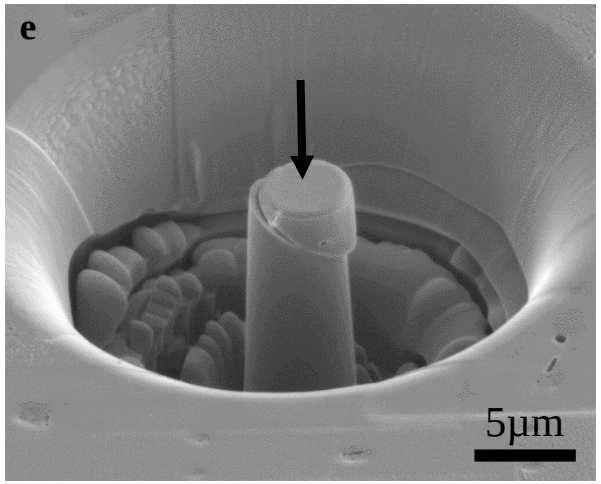


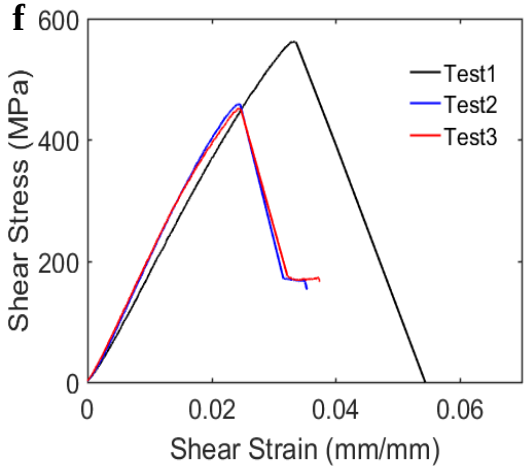




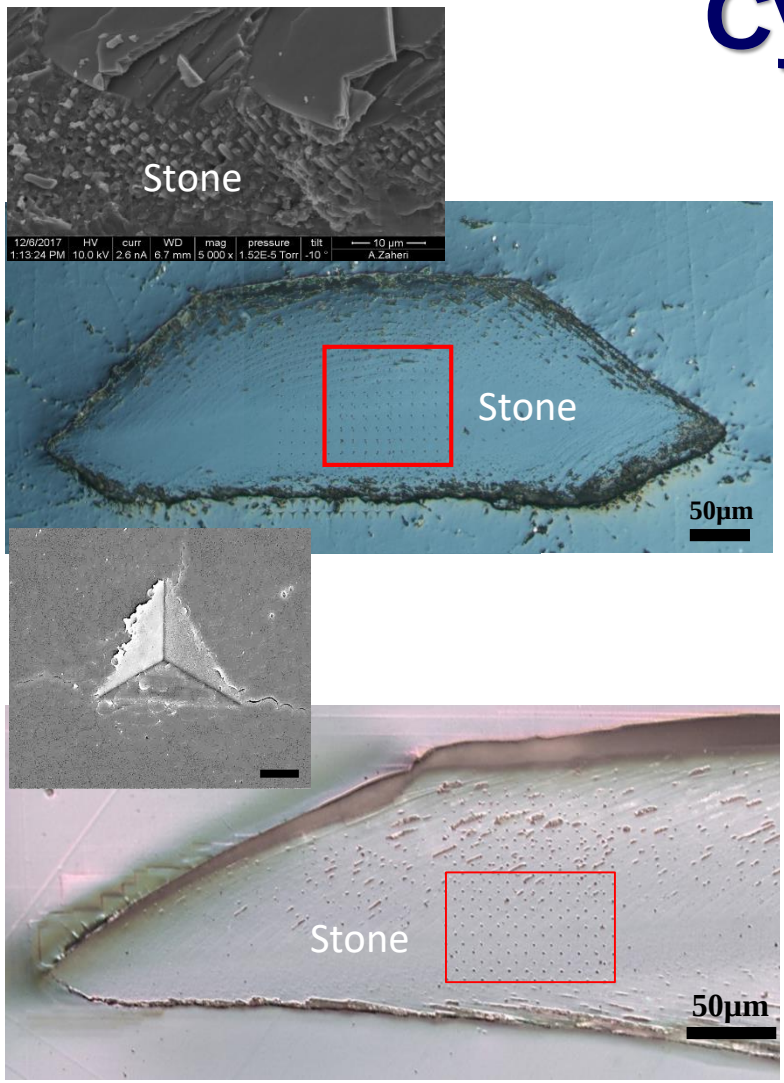


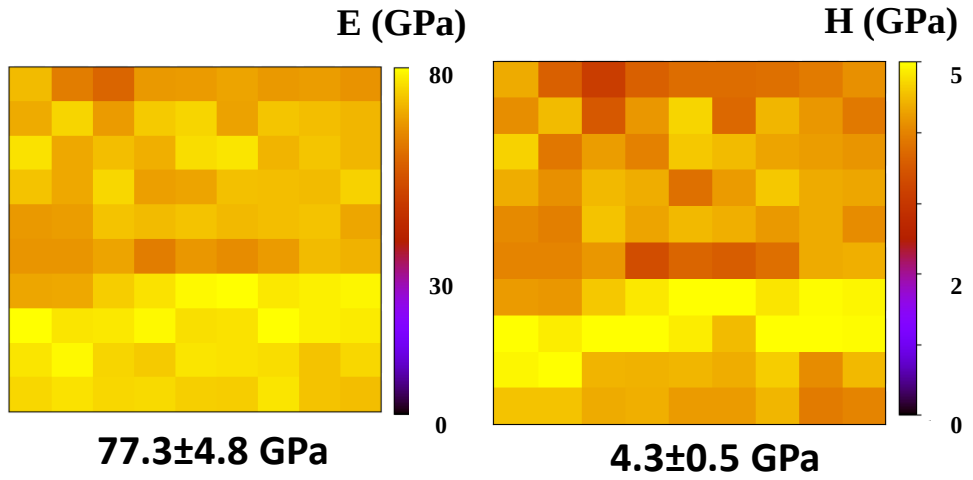


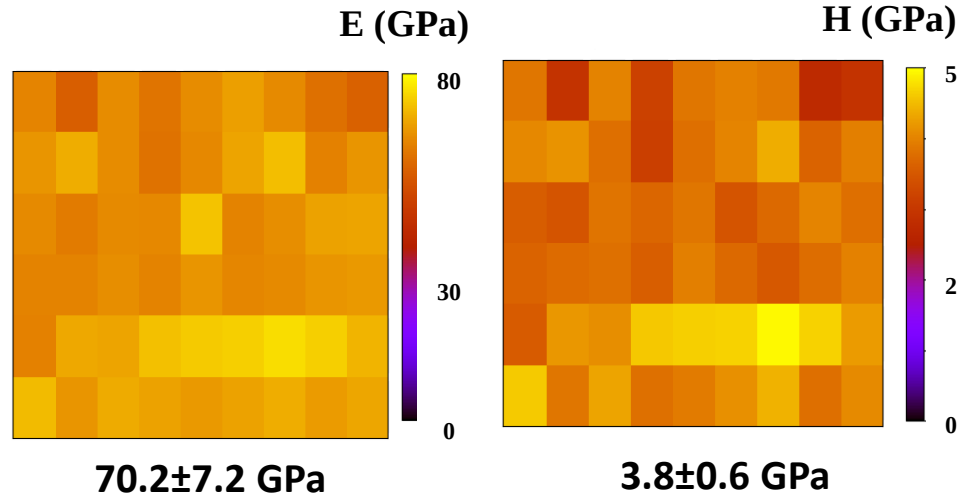


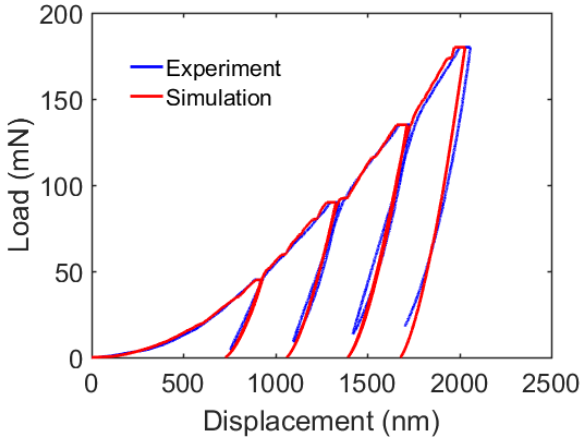


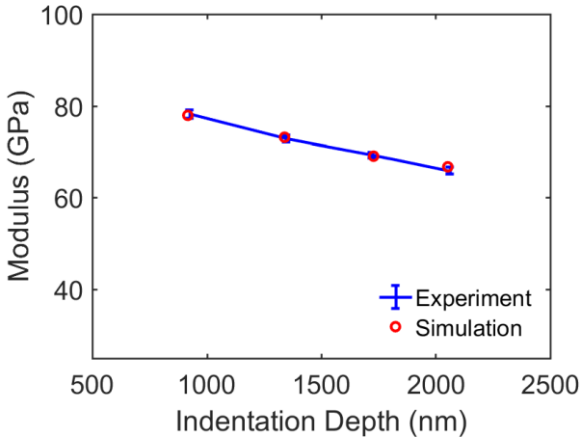
Identification of Stone Constitutive Behavior - Cyclic Nanoindentation







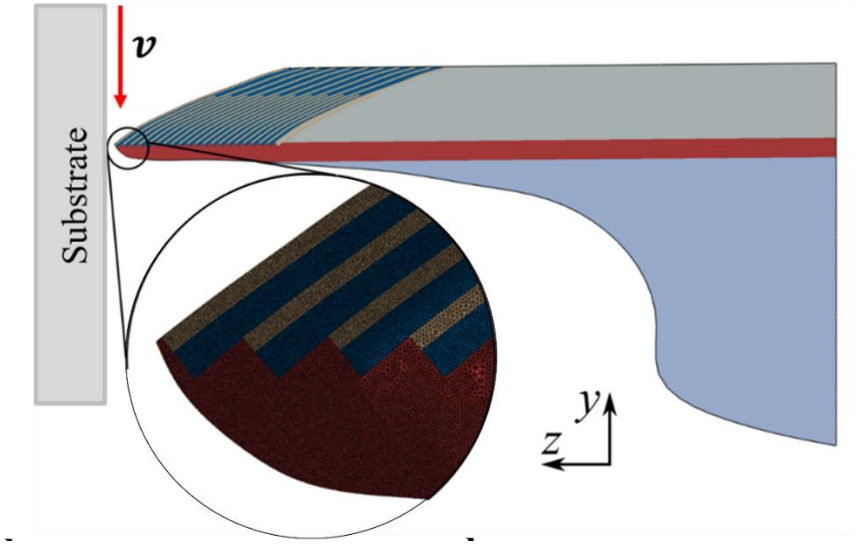


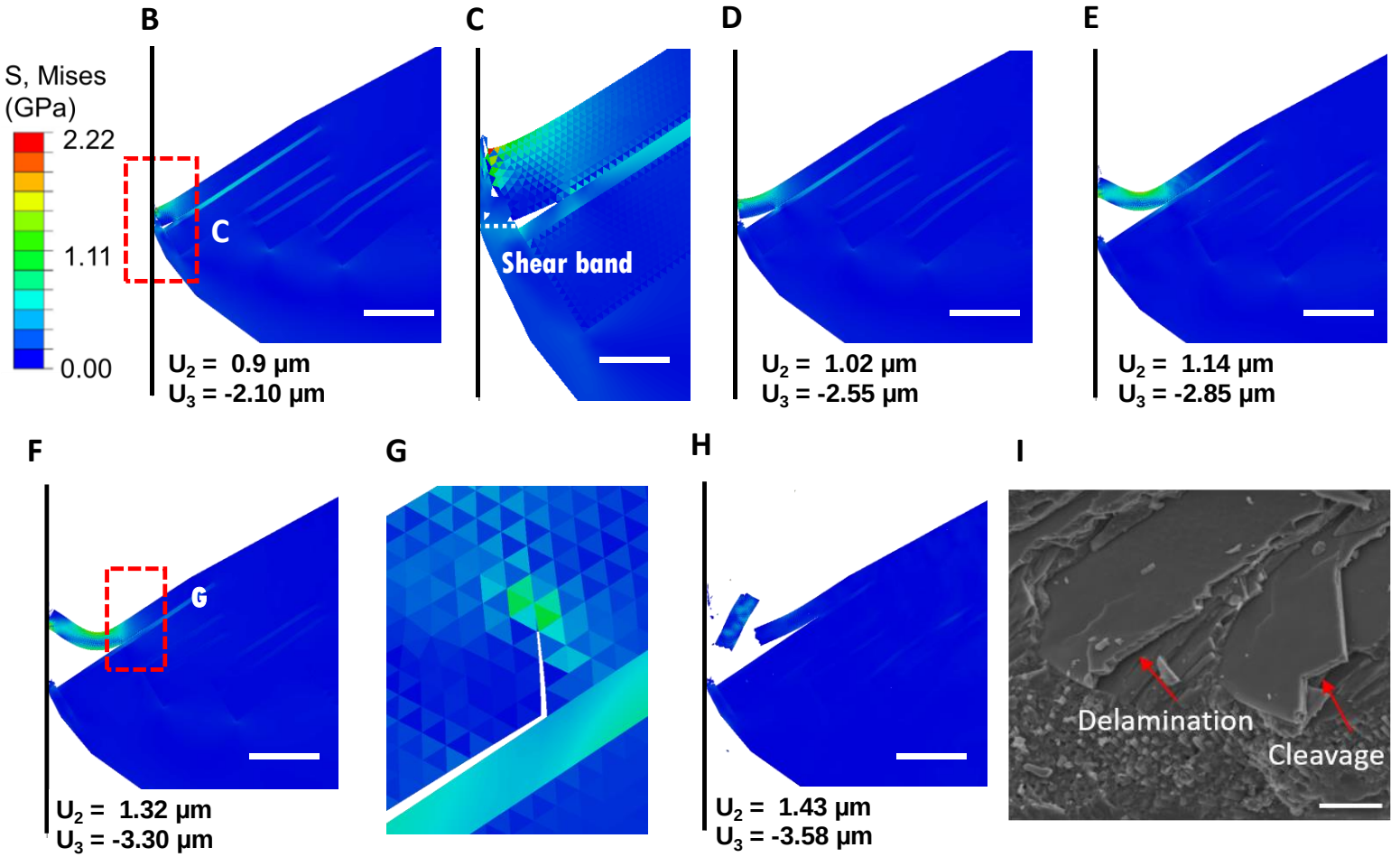


Stone undergoes plasticity and strain softening due to damage

Espinosa H.D., Zaheri A., Restrepo D., Nguyen H., Daly M., Frank M., McKittrick J., *Matter, 2019*

Nonlinear 2D FEA Model





- Stone modeled as a elasto-plastic material with damage (model parametrization based on nanoindentation experiments)
- Interfaces between calcite plates modeled with cohesive laws (strength determined from micropillar compression tests)
- Calcite plates modeled with anisotropic elasticity
- Plate cleavage modeled using cohesive elements

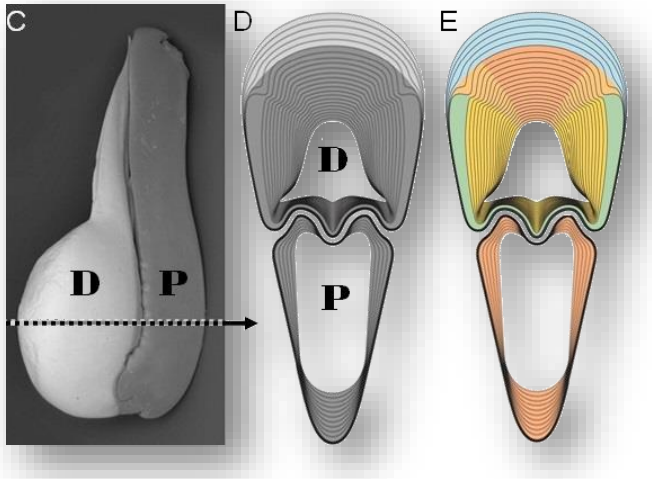
Espinosa H.D., Zaheri A., Restrepo D., Nguyen H., Daly M., Frank M., McKittrick J., *Matter, 2019*

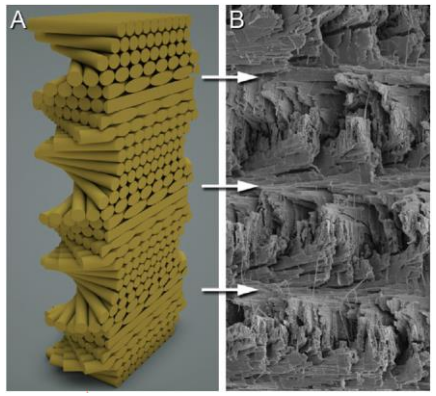
Natural Materials as Discovery Platforms

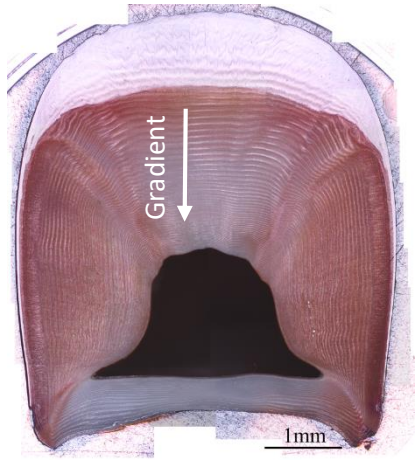
Opportunities

Acoustic Bandgaps in Bouligand Structures

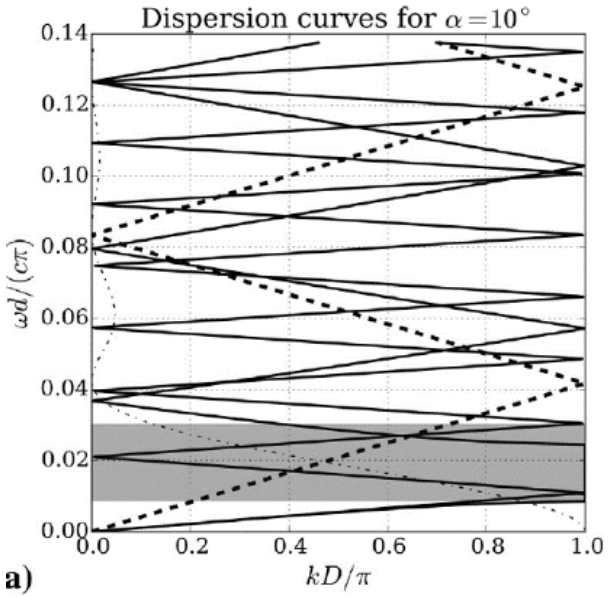
The mantis shrimp





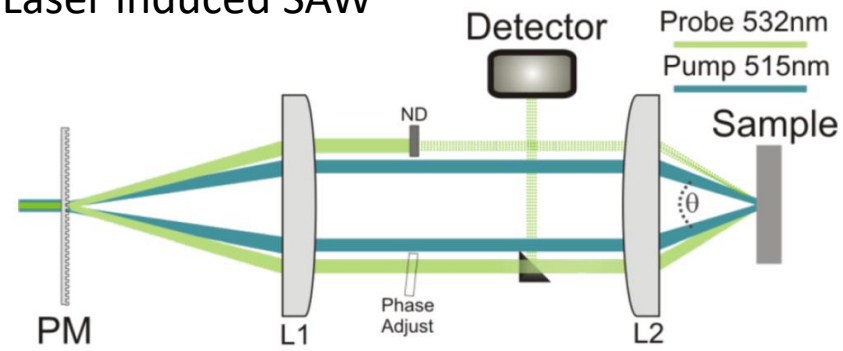


Bloch analysis



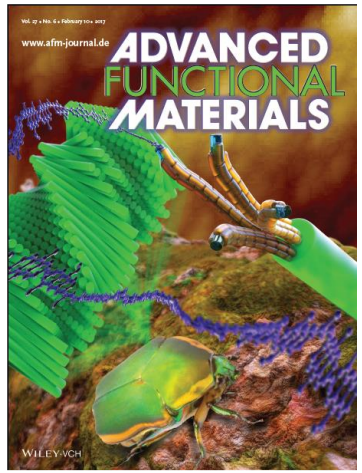
Pablo Zavattieri

Laser induced SAW

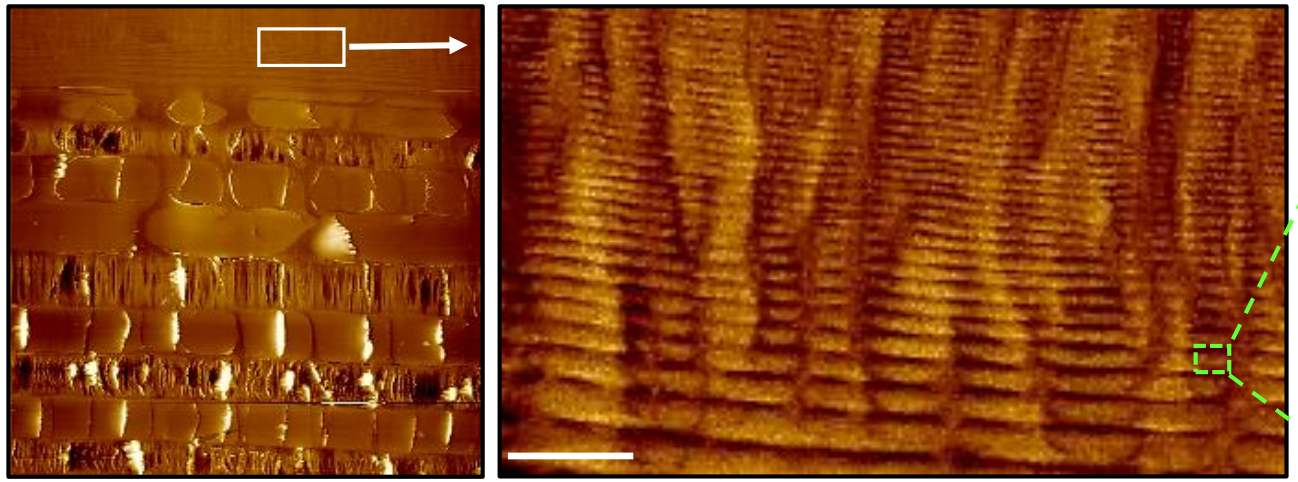


N. Guarin-Zapata, J. Gomez, N. Yaraghi, D. Kisailus, and **P. D. Zavattieri**, "Shear wave filtering in naturally-occurring Bouligand structures," *Acta Biomaterialia,* 2015

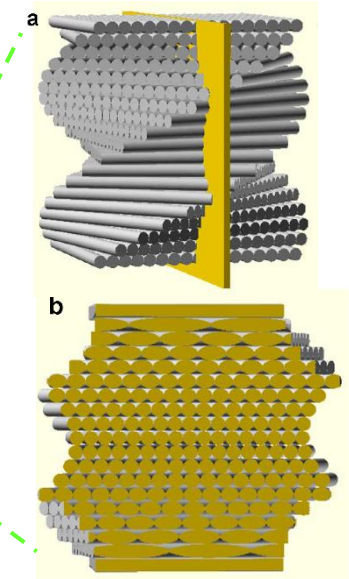
Morphological Gradients and Nanoscale Properties

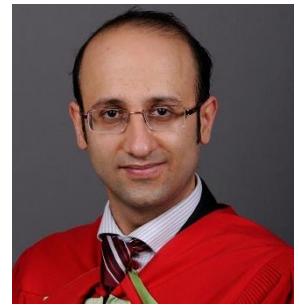


The Beetle Elytra



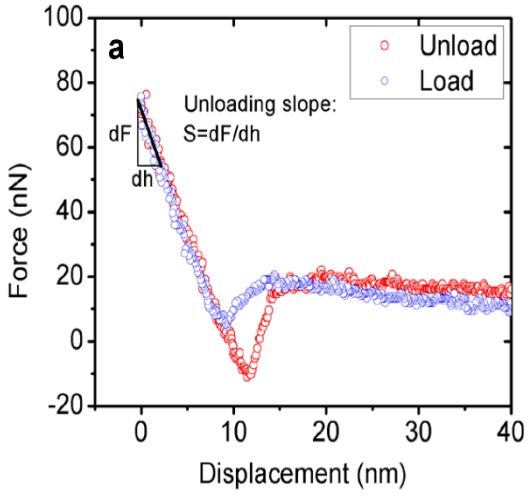
Helicoidal chitin nanofibers

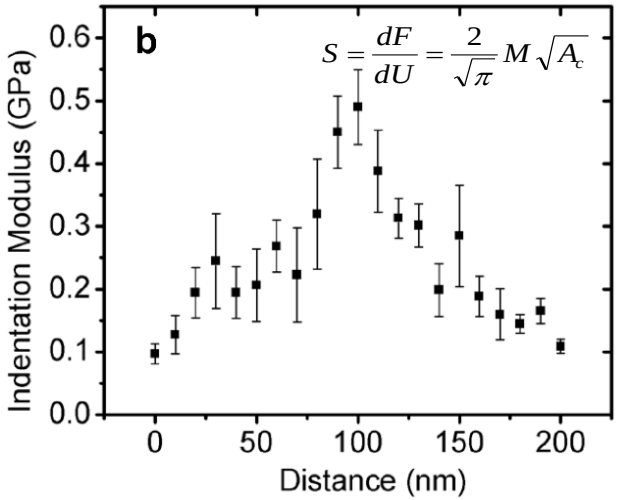




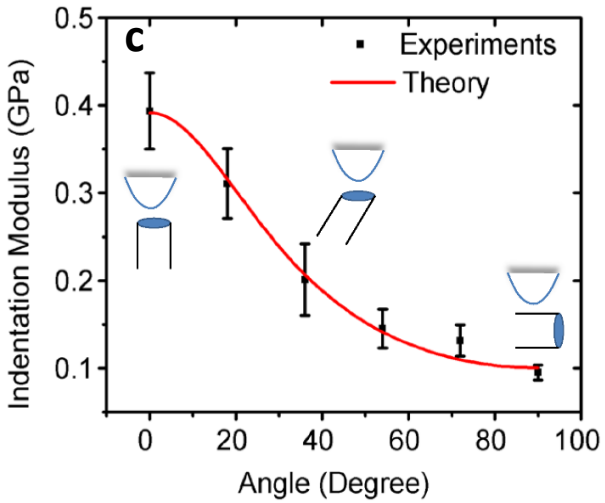
M. Asgari

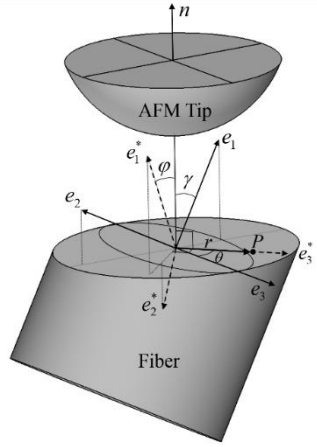
Atomic Force Microscopy Indentation





Contact Mechanics Analysis





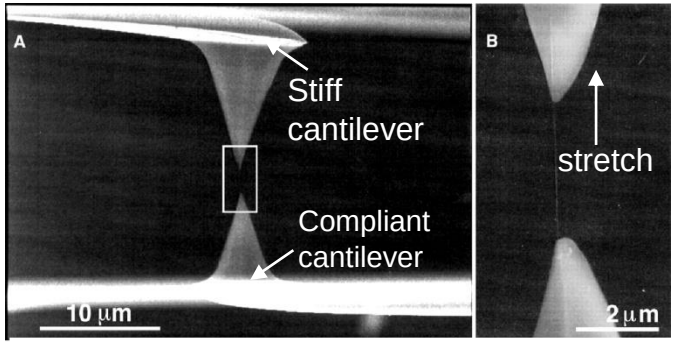
$$\nu_{xy} = \nu_{xz} = 0.3$$
$$E_x = 710MPa$$
$$E_z = 70MPa$$
$$G_{yz} = 90MPa$$

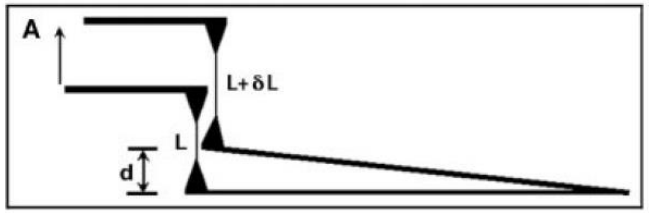
R. Yang, A. Zaheri, W. Gao, C. Hayashi, H.D. Espinosa, *Advanced Functional Materials*, 2017

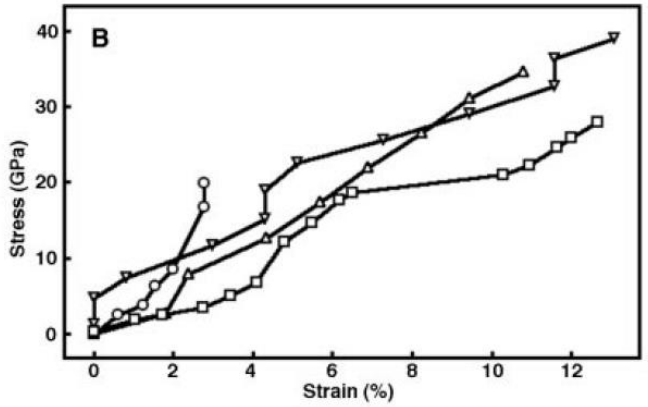
Outline

- A fascinating wave propagation experiment to investigate failure initiation and kinetics in ceramics
- Mesoscale stochastic models to bridge atomistic processes and continuum measurements
- Functionality driven evolution, role of hierarchies in natural ceramics … and inferring material design rules
- **Seeing the invisible**: failure in low dimensional materials

A Story: When Experiments and QM Predictions Do Not Agree







Yu, Ruoff, et al., *Science* (2000)

Arc discharge CNTs

E~ 270-350 GPa

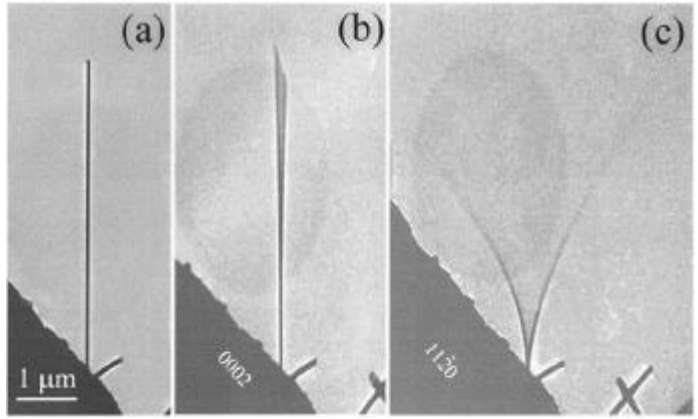
σ_f ~11-30 GPa

QM Predictions

E~ 1 TPa

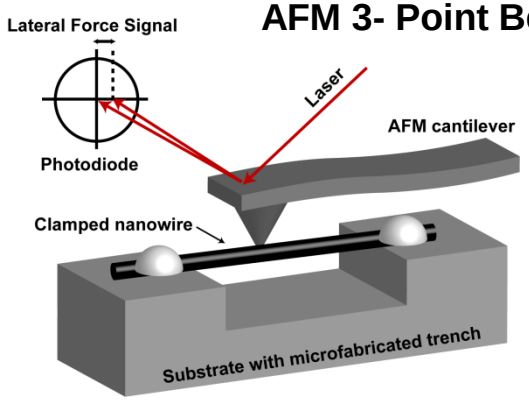
σ_f ~110 GPa

Dynamic Resonance



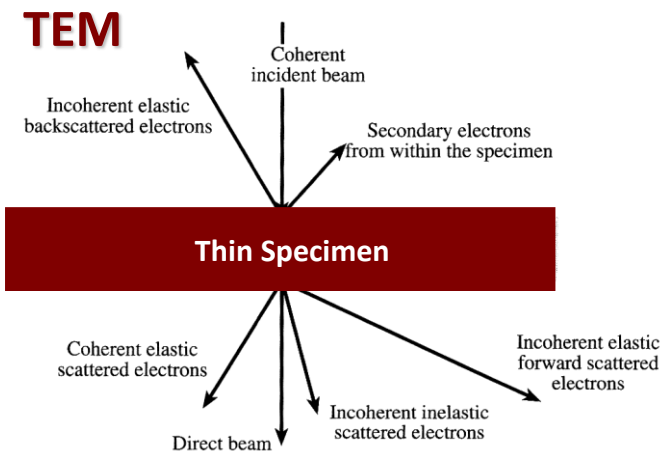
Chen et al., *Phys. Rev. Lett.*, (2006)

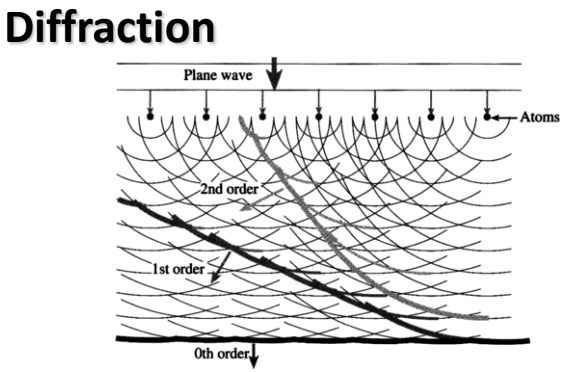
AFM 3- Point Bending

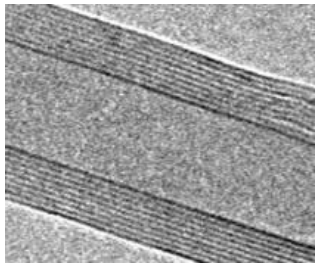


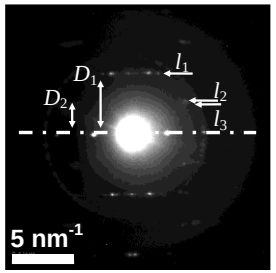
Ni et al., *Nanotechnology,* (2006)

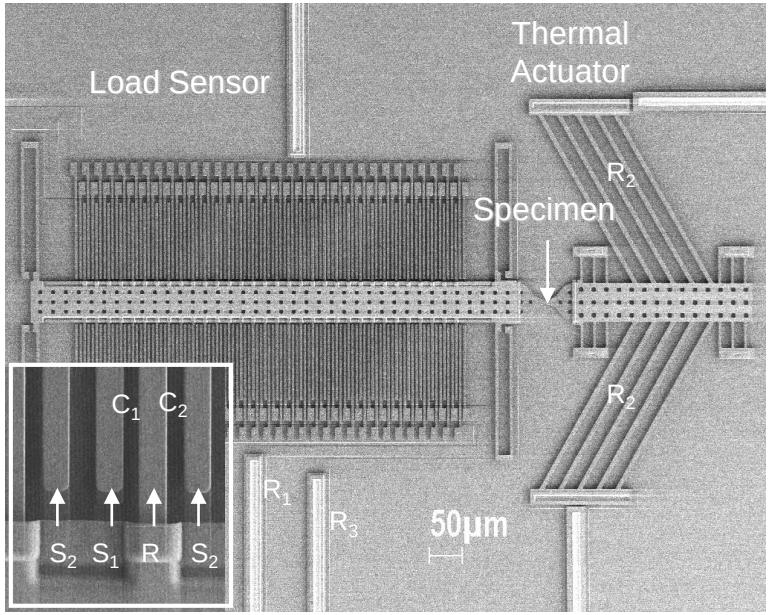
In situ TEM MEMS-Based Nanoscale Material Testing

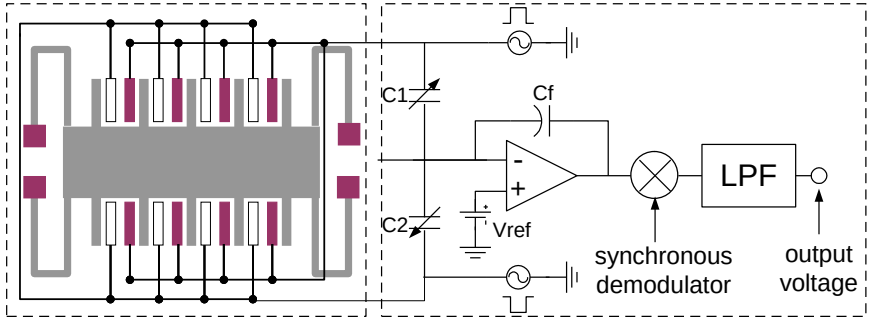




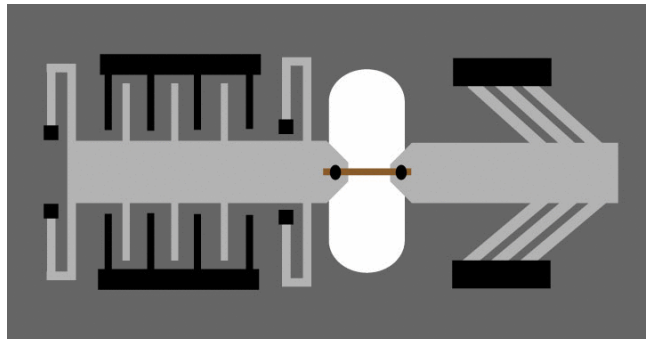


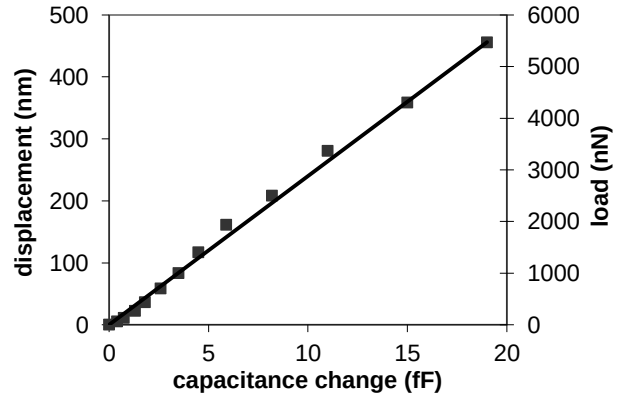






$$V_{sense} = \frac{V_o}{C_0}\Delta C \sim \frac{2N\varepsilon A V_o}{C_0 d_0^2}\Delta d$$





Load = displacement × stiffness

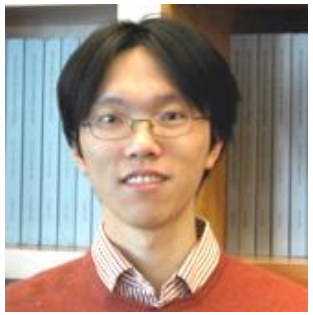
Device Resolution

Capacitance change: 0.05 fF
Displacement: 1 nm
Load: 12 nN

Zhu, Moldovan and Espinosa, *Appl. Phys. Lett.*, 86, **2005**
Espinosa et al., *JMEMS*, Vol. 16, **2007**

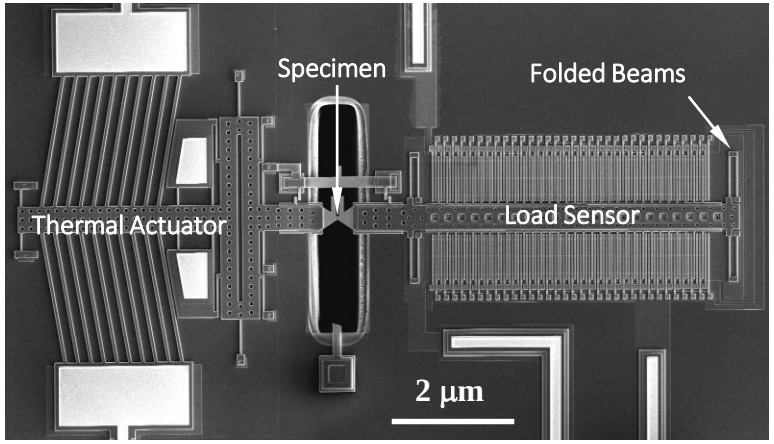
In-situ TEM Experiments using MEMS

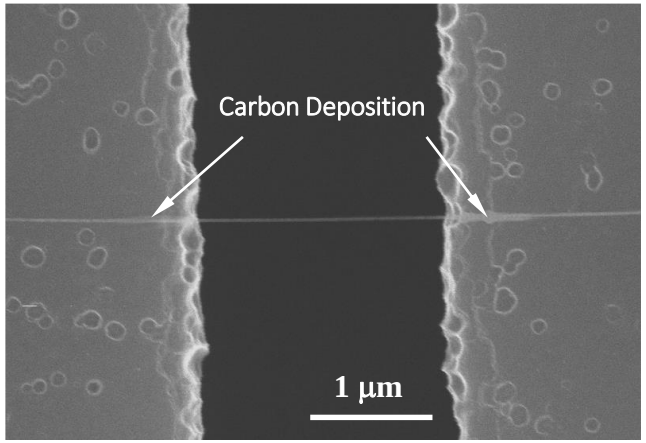
- Well-defined loading condition – pure axial loading
- Electronic measurement of load
- Simultaneous observation of atomic structures and defects

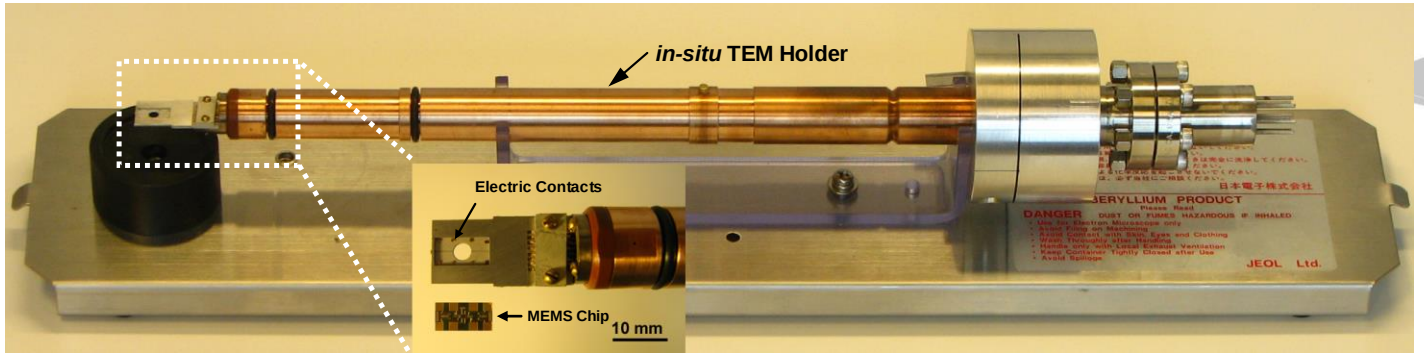


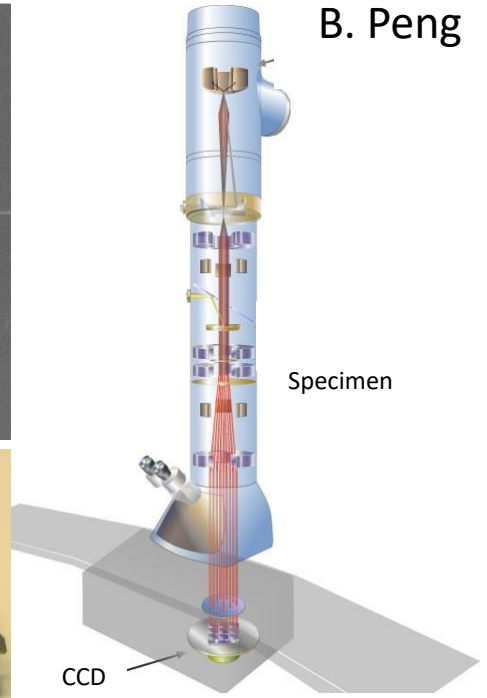
B. Peng

Yong Zhu



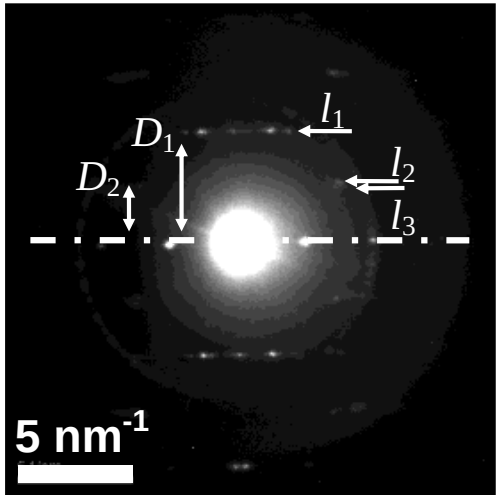


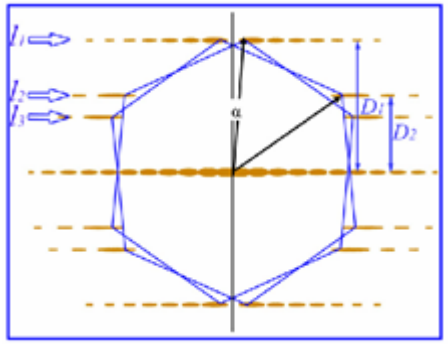




Zhu and H.D. Espinosa, *PNAS*, **2005**

Tube Chirality and Diameter



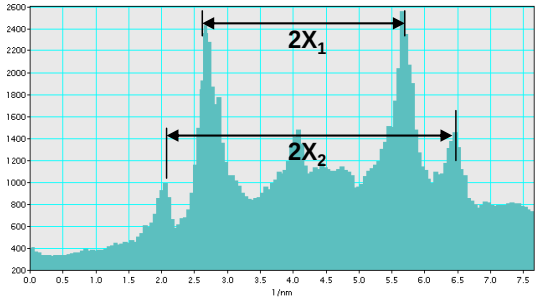


$$I_{l1}(R) \propto \left|J_v(\pi dR)\right|^2$$

$$I_{l2}(R) \propto \left|J_u(\pi dR)\right|^2$$

$$I_{l3}(R) \propto \left|J_{u+v}(\pi dR)\right|^2$$

Bessel function



Line 1

$$\frac{X_2}{X_1} = 1.47 \rightarrow v = 8$$

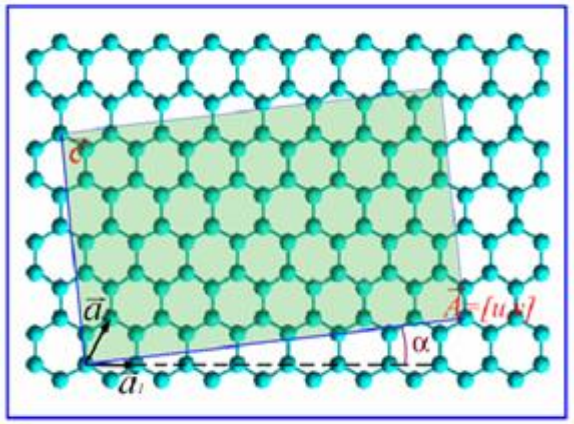
$$\frac{v}{u} = \frac{(2D_2 - D_1)}{(2D_1 - D_2)} = 0.0434$$

$$d = A/\pi$$

$$\rightarrow u = 184, v = 8,$$

$$\rightarrow \alpha = 2.111^o, d = 14.72nm$$

L.C. Qin, *Reports on Progress in Phys*. 69, 2006

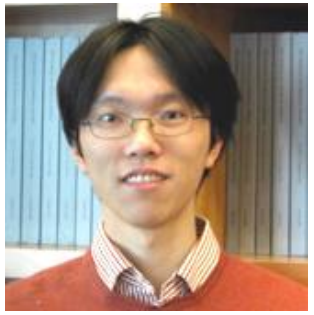


$$\vec{A} = (u, v) = u\vec{a}_1 + v\vec{a}_2$$

$$A = a_0(u^2 + v^2 + uv)^{1/2}$$

$$\alpha = \tan^{-1}\left(\frac{2D_2 - D_1}{\sqrt{3}D_1}\right)$$

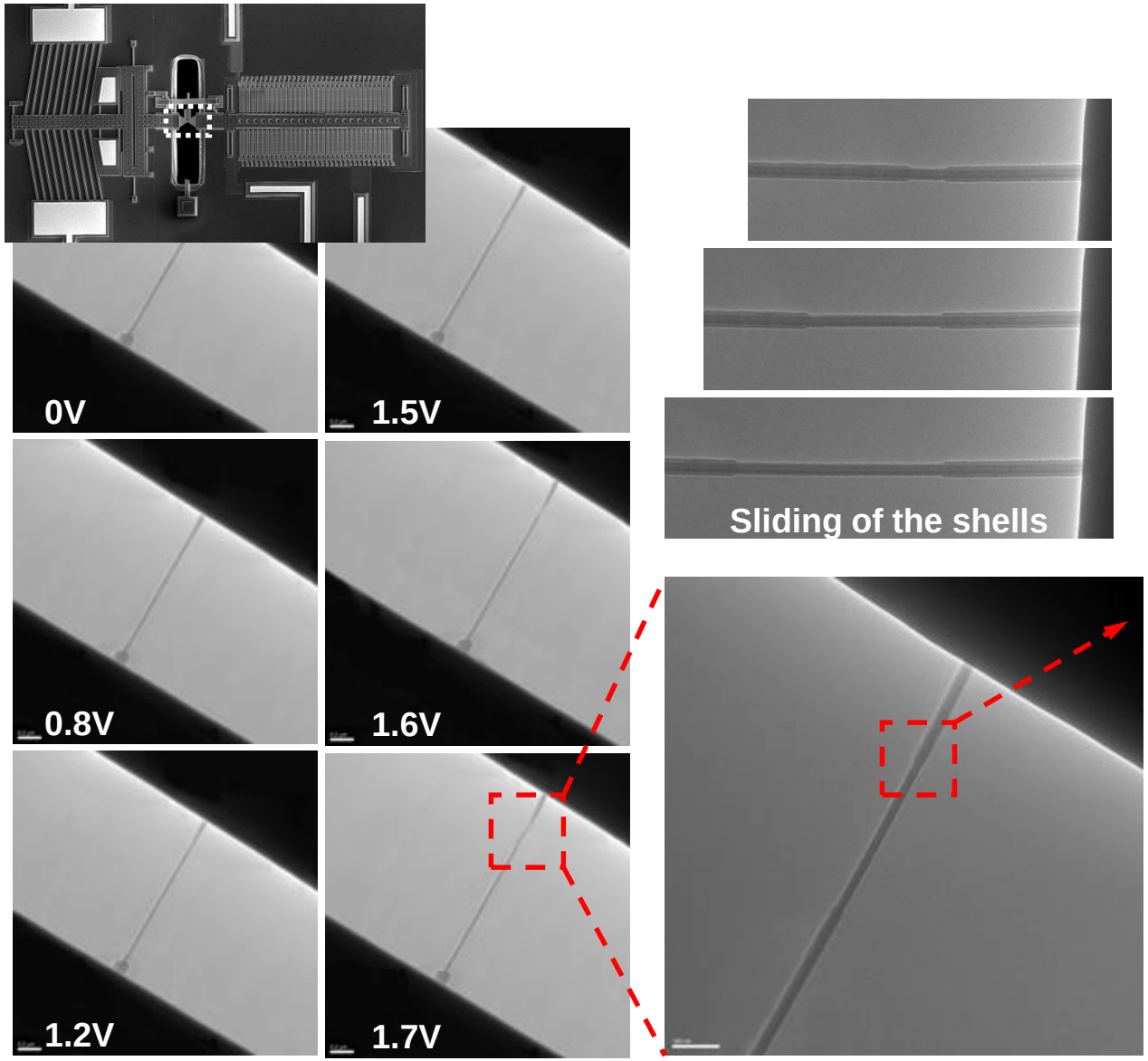
$d = 14.72\ nm$

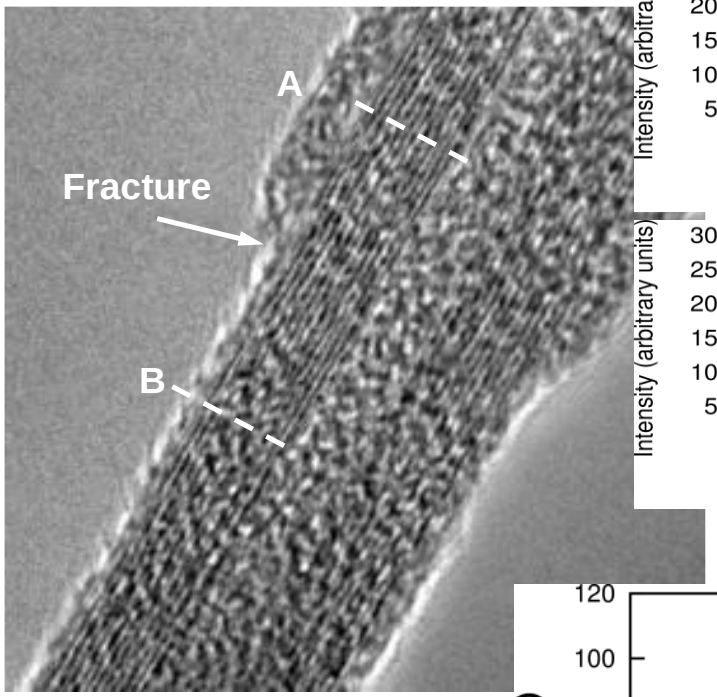


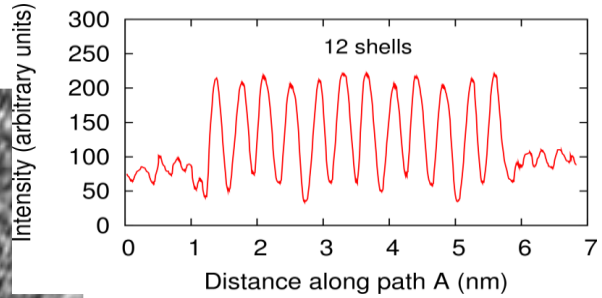
B. Peng

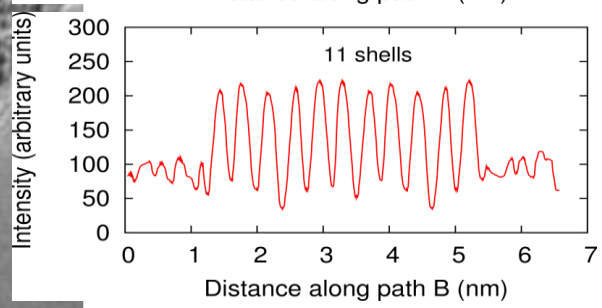
Peng, Espinosa, et al., *Nature Nanotechnology*, **2008**

Stretching and Atomic Imaging of a MWCNT

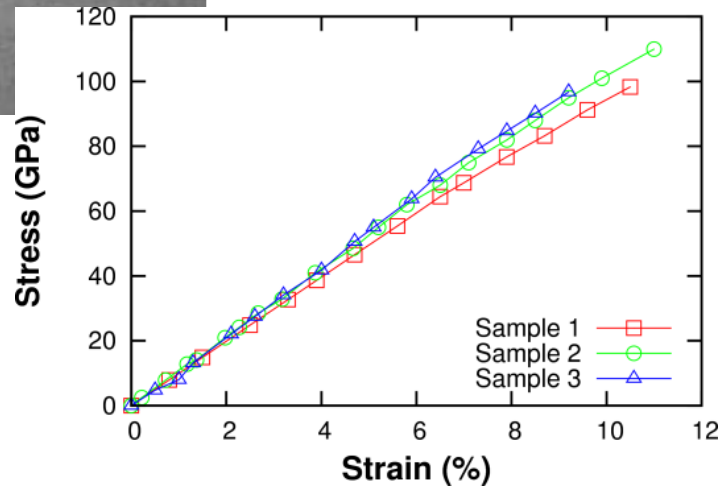








Experiments
E=1 TPa ;
σ_f ~100 GPa



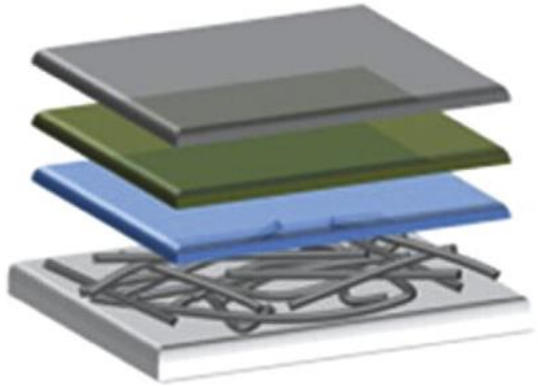
Theory
QM Predictions: E=1 TPa ; σ_f ~110 GPa

Peng, Espinosa, et al., *Nature Nanotechnology*, **2008**

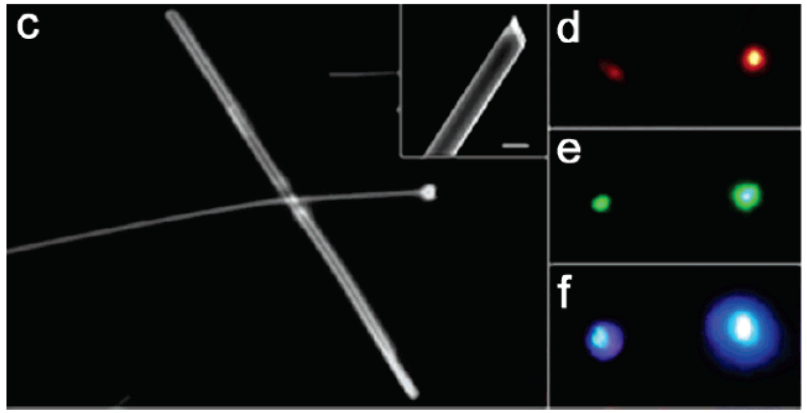
Metallic and Semiconducting Nanowires

http://www.cambrios.com/

AgNWs/PET film-based organic solar cells



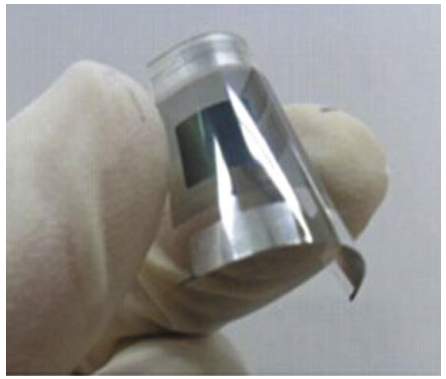
Integrated Nanophotonics (GaN)



Sirbuly, *et al., J. Phys. Chem. B,* 109, 2005

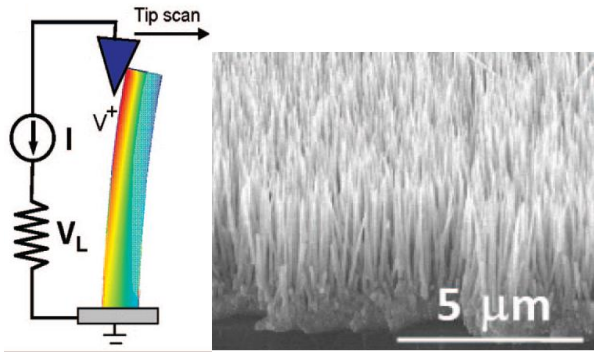
Transparent electrode applications:

- Touch screens
- Solar cells
- Transparent heaters
- Stretchable RF antennas
- Transparent EM shields
- Strain sensors



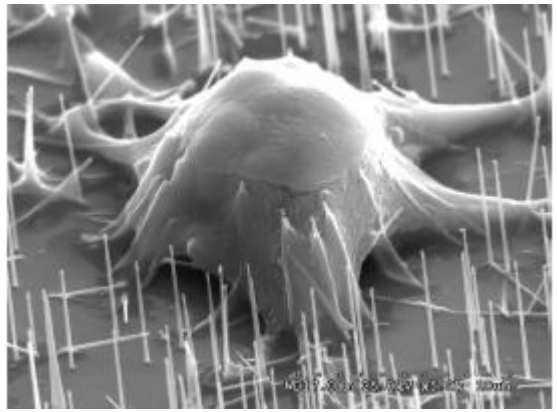
W. He and C. Ye, *JMST*, 215

Energy Harvesting (ZnO)



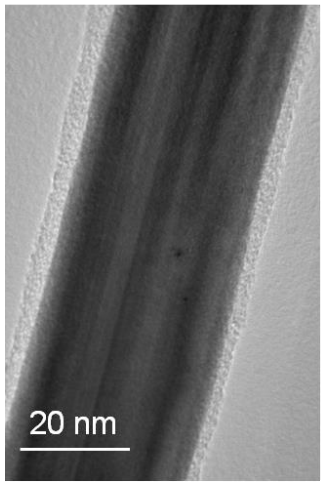
Lu, Wang, *et al., Nano. Letts.,* 2009

Cell Studies

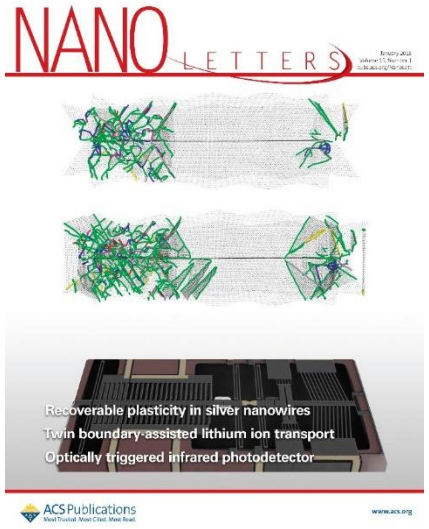


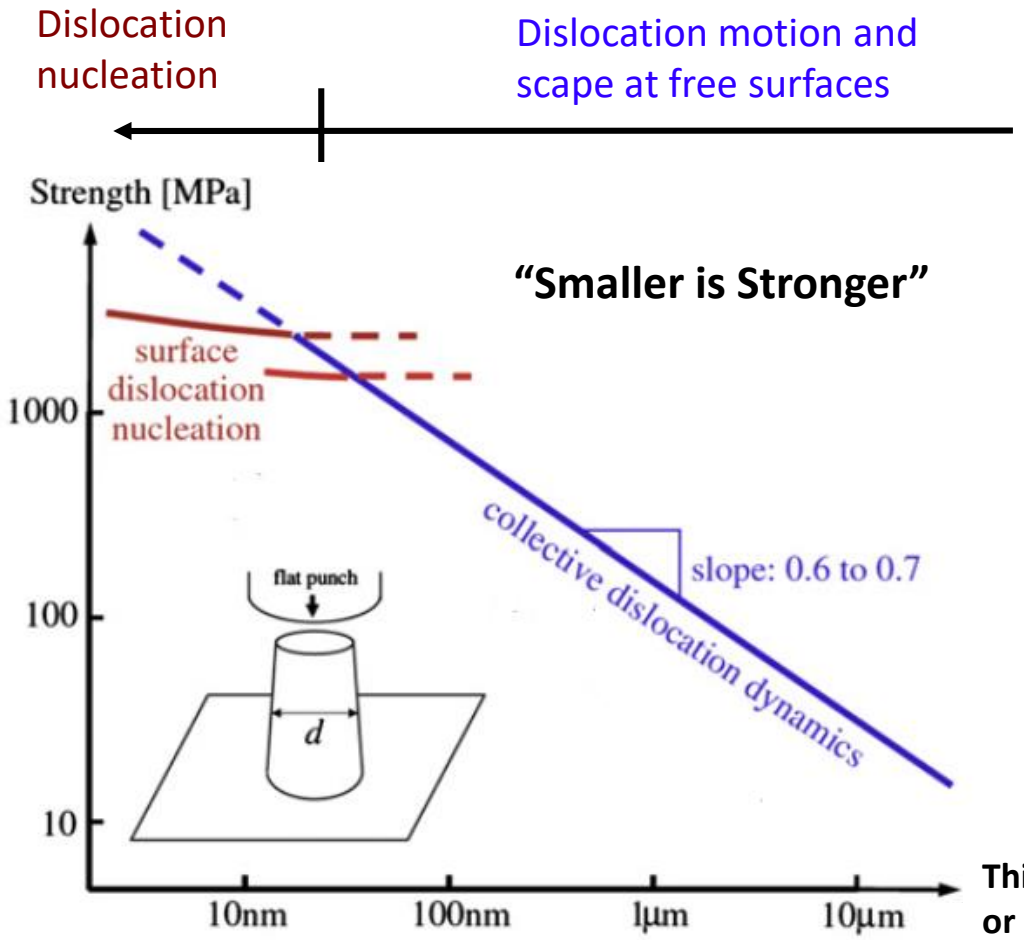
P.D. Yang et al., *Nano Lett.,* 2010

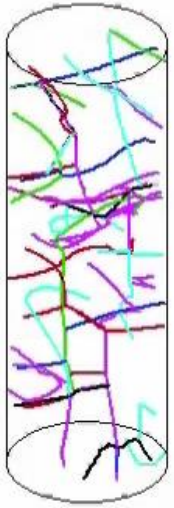
Plasticity at Small Scales– Transition from Dislocation Dynamics to Dislocation Nucleation



Dislocation free



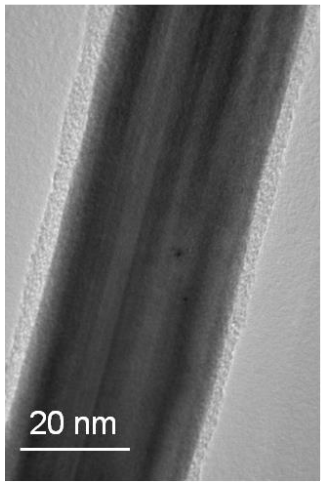




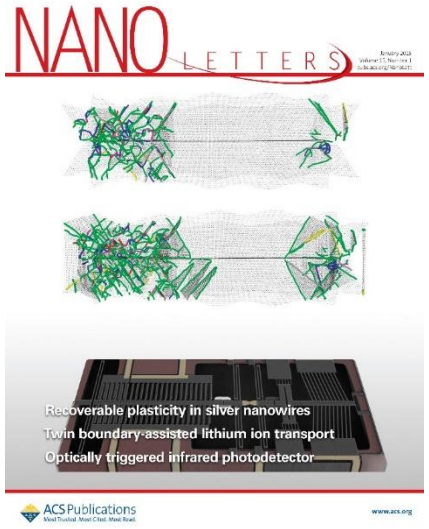
Internal dislocation structure

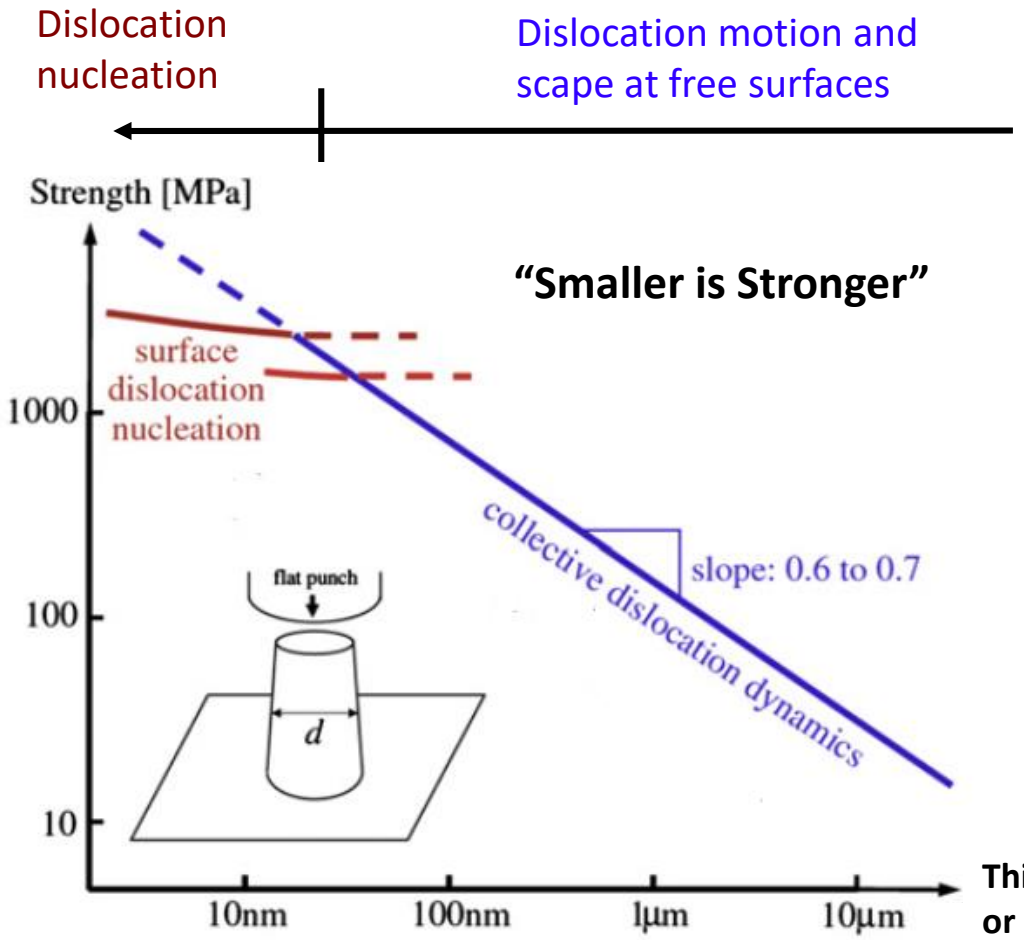
Nix, Greer et al., *Acta Mater.,* **2005**
Espinosa, Berbenni, Panico, Schwarz, PNAS, **2005**
Tang, Schwarz, Espinosa, *PRL,* **2007**

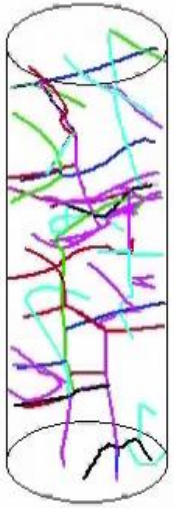
Plasticity at Small Scales– Transition from Dislocation Dynamics to Dislocation Nucleation



Dislocation free



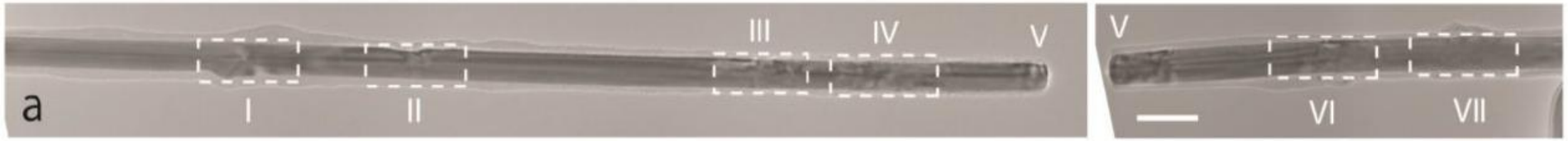




Internal dislocation structure

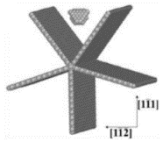
Nix, Greer et al., *Acta Mater.,* **2005**
Espinosa, Berbenni, Panico, Schwarz, PNAS, **2005**
Tang, Schwarz, Espinosa, *PRL,* **2007**

Plasticity in Ag Penta-twinned Nanowires



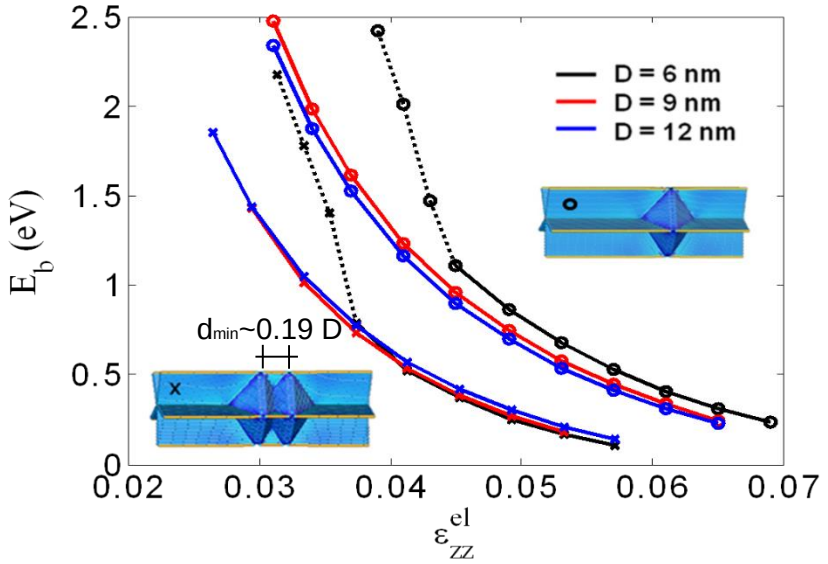
Tobin Filleter

Surface nucleation of stacking faults

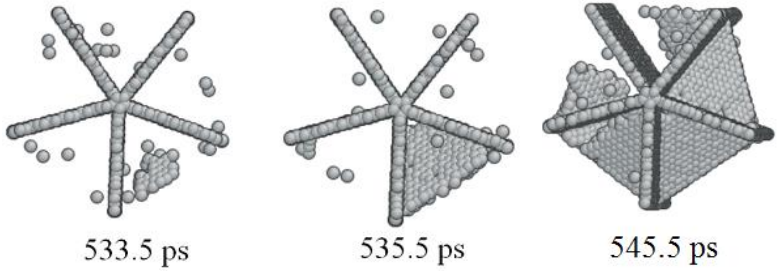


Energy Barrier Calculations

1st and 2nd Stacking Fault Decahedrons



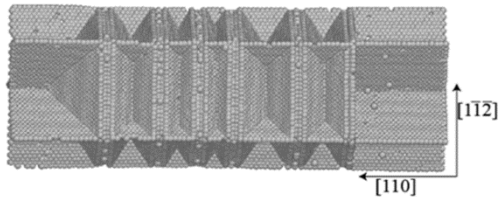
MD predictions



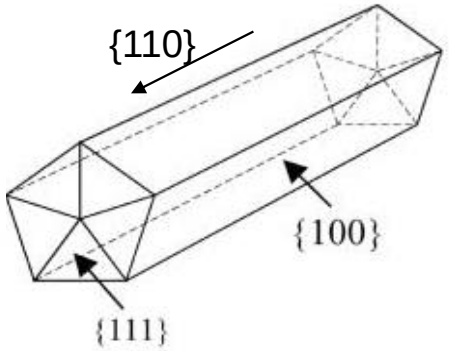
Surface nucleation of Shockley partial

Sweeps entire domain

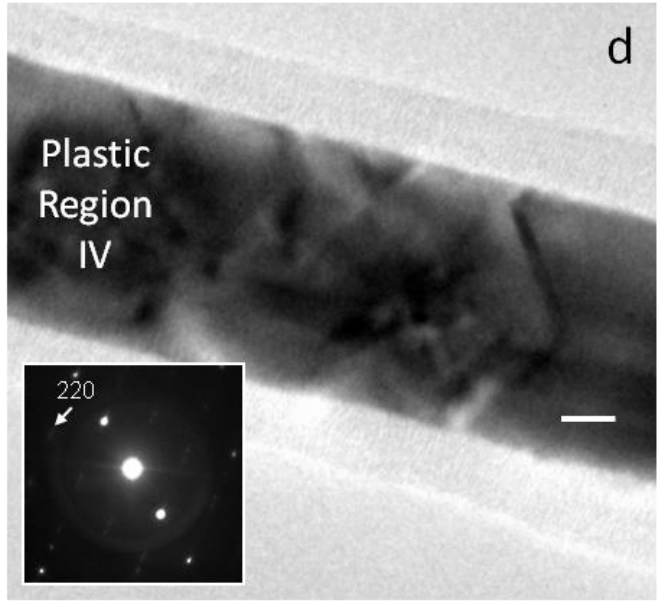
Sweeps over 5 domains



Stacking Fault Decahedrons (SFD)



In situ TEM Experiments

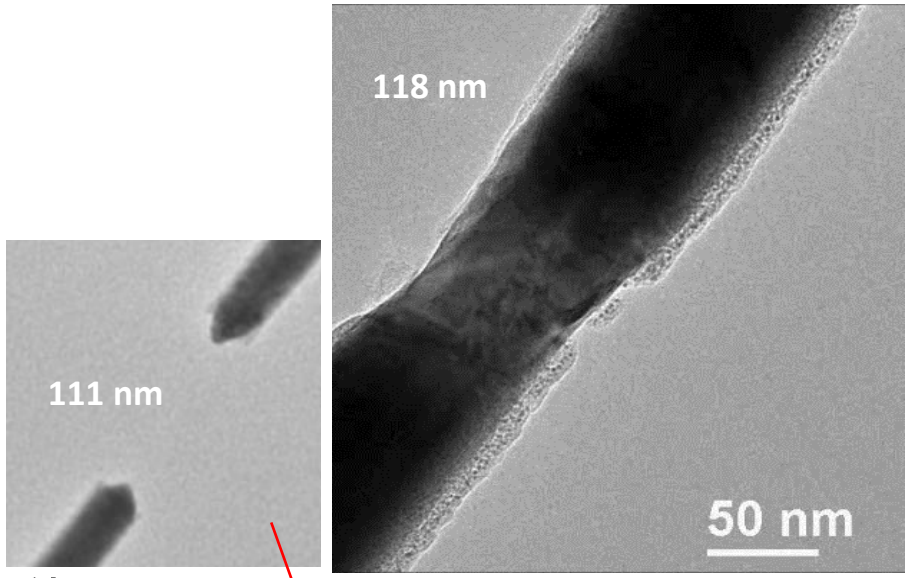


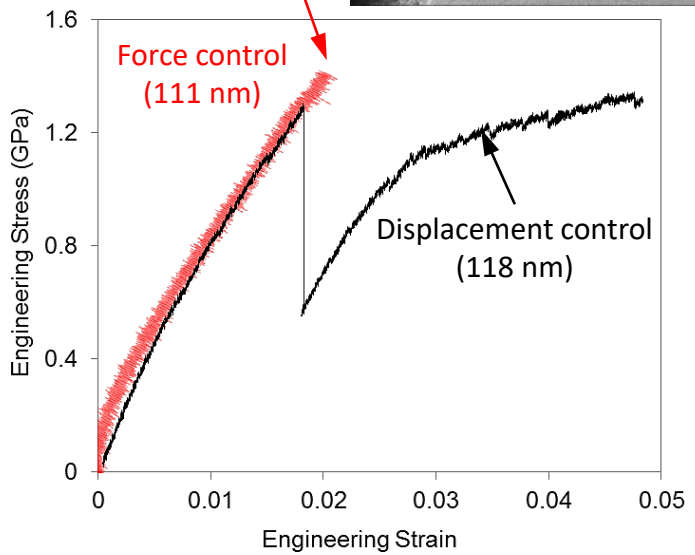
In collaboration with Wei Cai

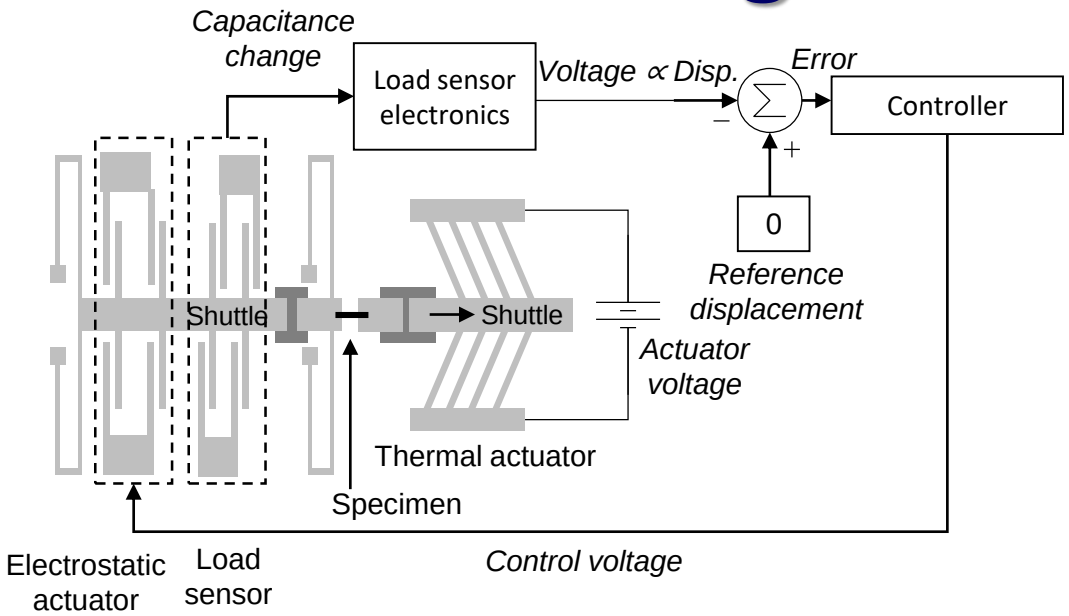
"Nucleation-Controlled Plasticity in Penta-Twinned Silver Nanowires, " by T. Filleter, W. Cai, Espinosa, et al., *Small*, **2012**

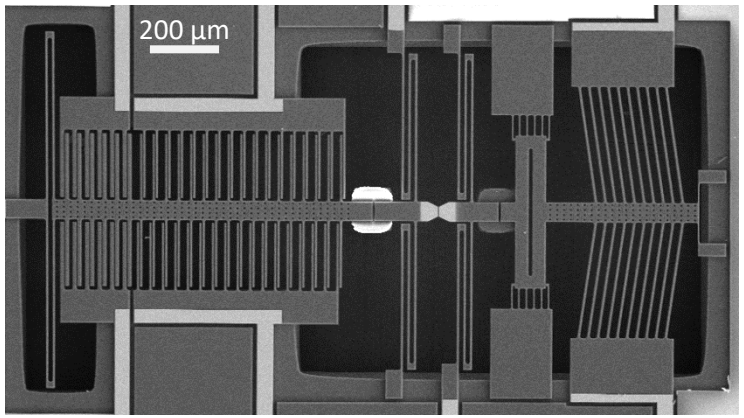
Capturing Necking Instabilities Displacement-Controlled *in situ* Testing

R. Bernal





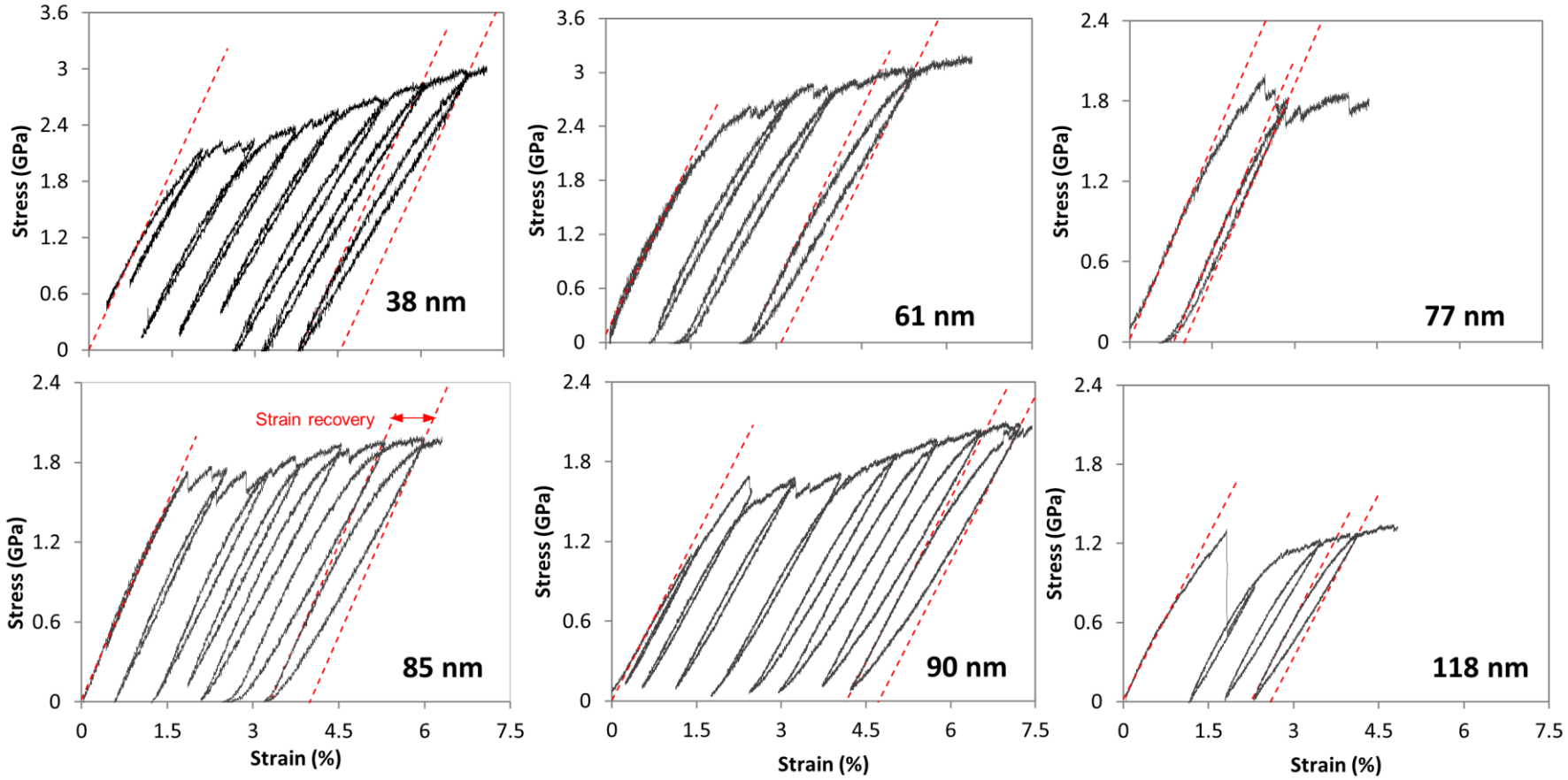


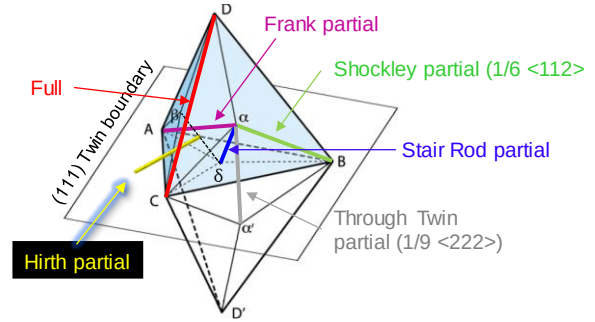


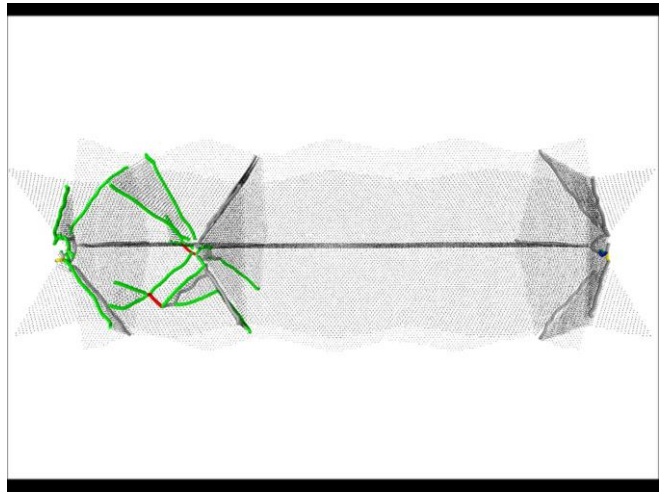
MEMS with feedback control

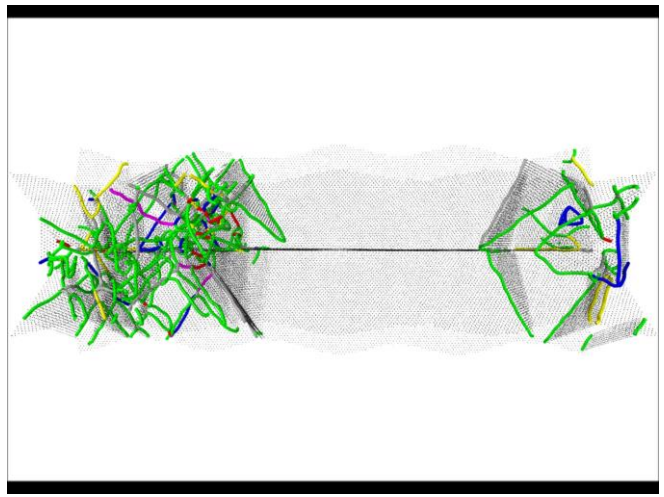
Bernal, Pantano, Espinosa et al. *Meccanica*, **2014**

Plasticity is Size Dependent and Partially Reversible





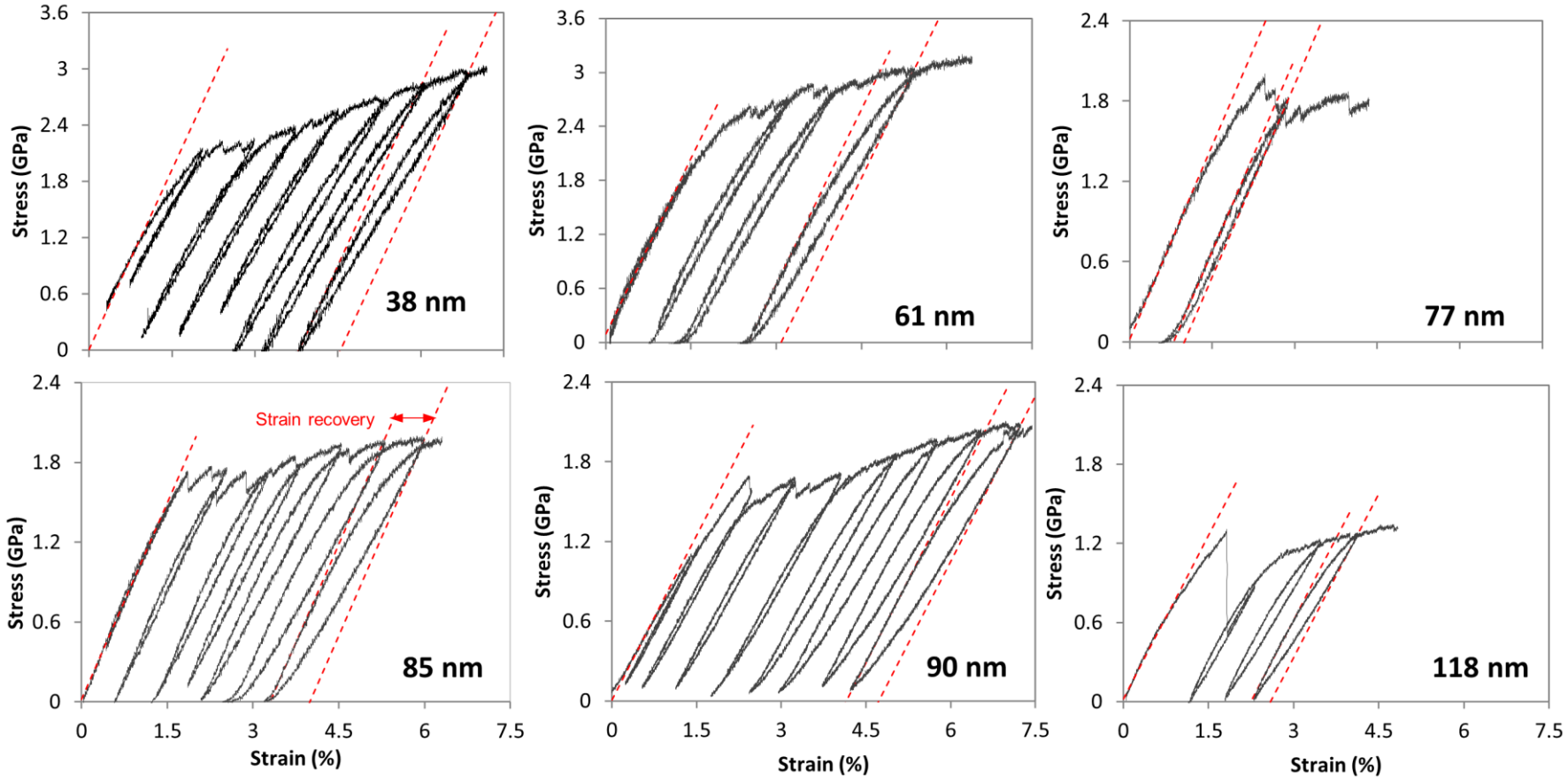


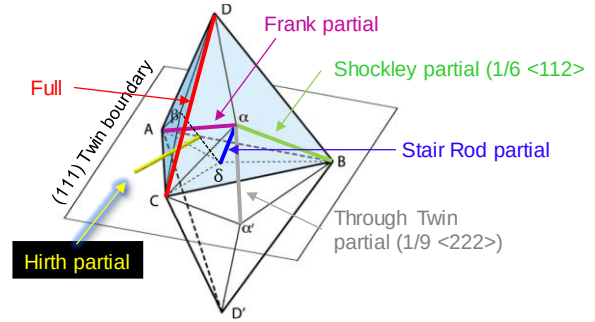


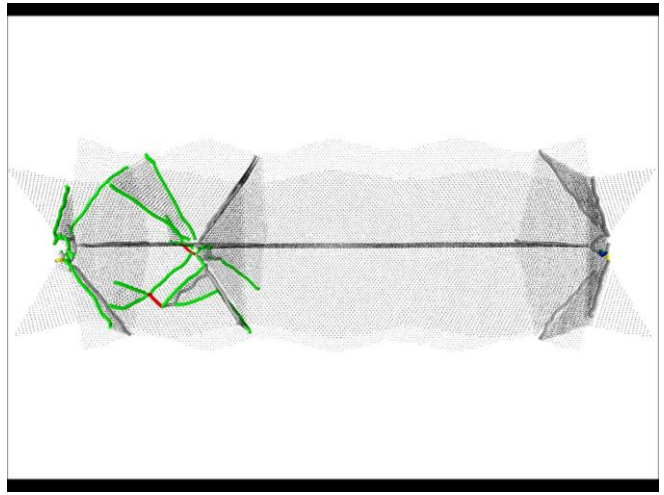
- Size dependent yield stress
- Reversed plastic flow upon unloading. Strong Intrinsic Bauschinger Effect!

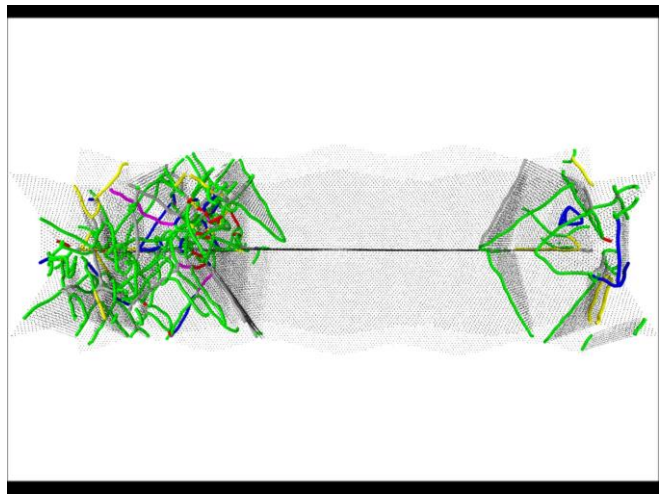
Bernal, Cai, Espinosa et al., *Nano Letters,* **2014**

Plasticity is Size Dependent and Partially Reversible









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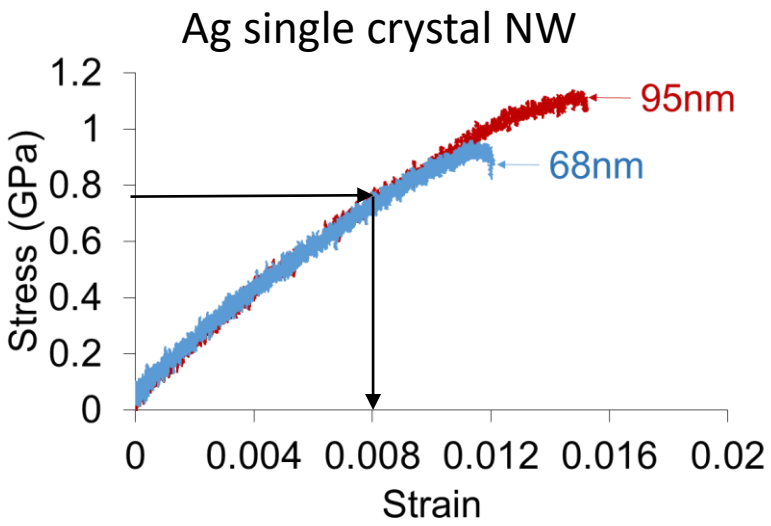
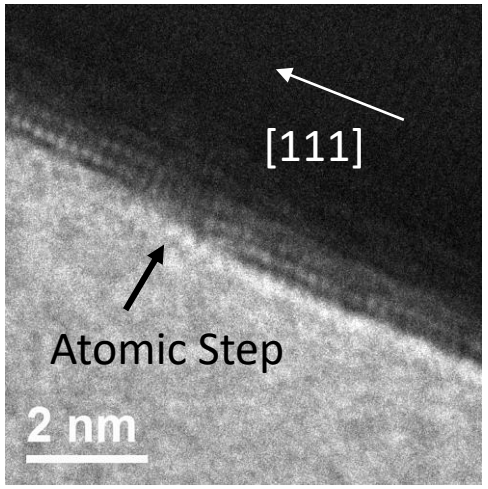
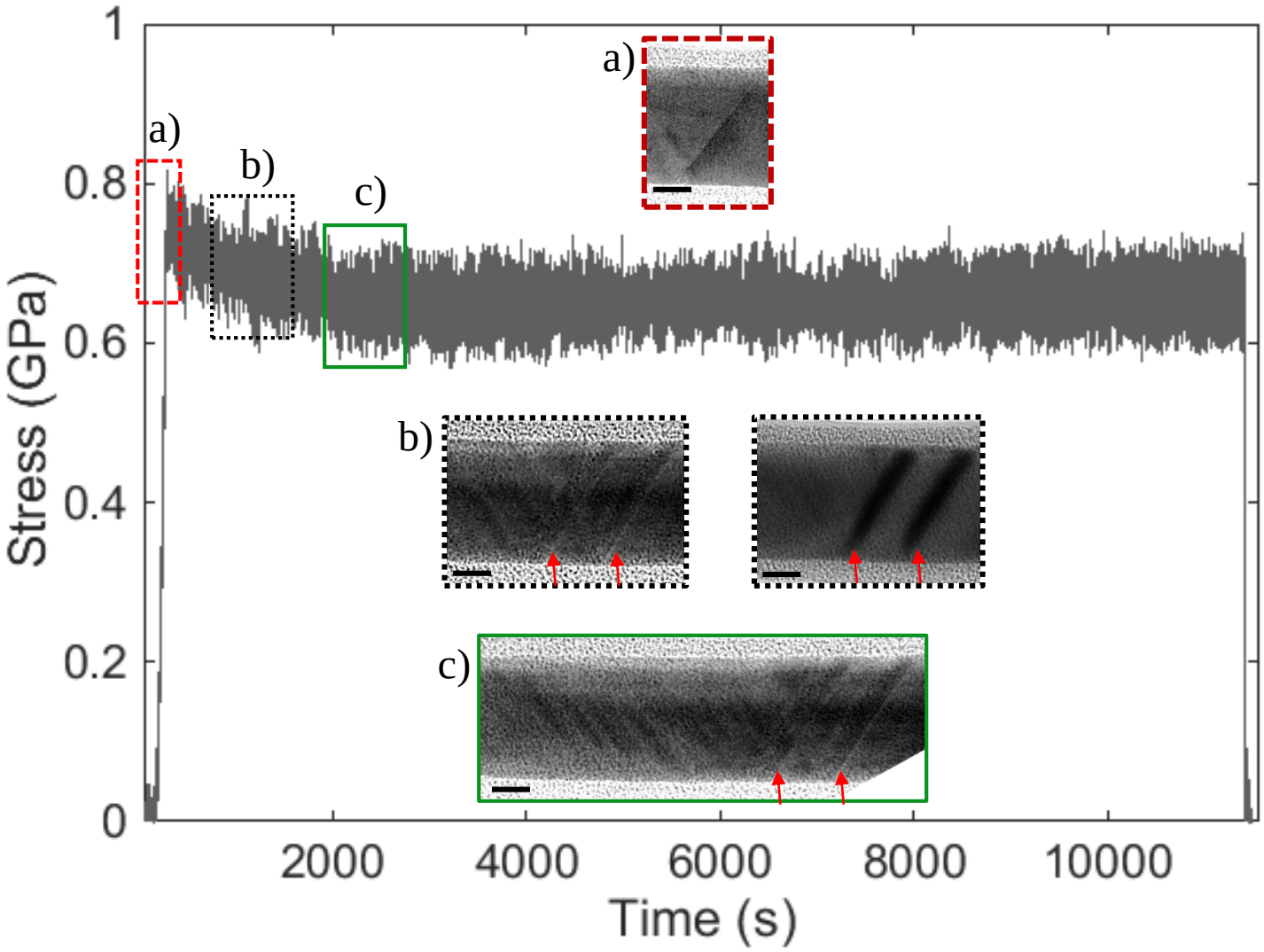
Bernal, Cai, Espinosa et al., *Nano Letters,* **2014**

Reliability Issues – Time Dependent Necking Instabilities

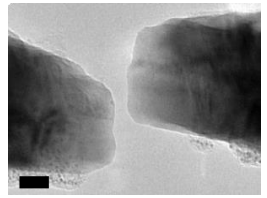






Raj

★ Abrupt Failure 3.5 hs

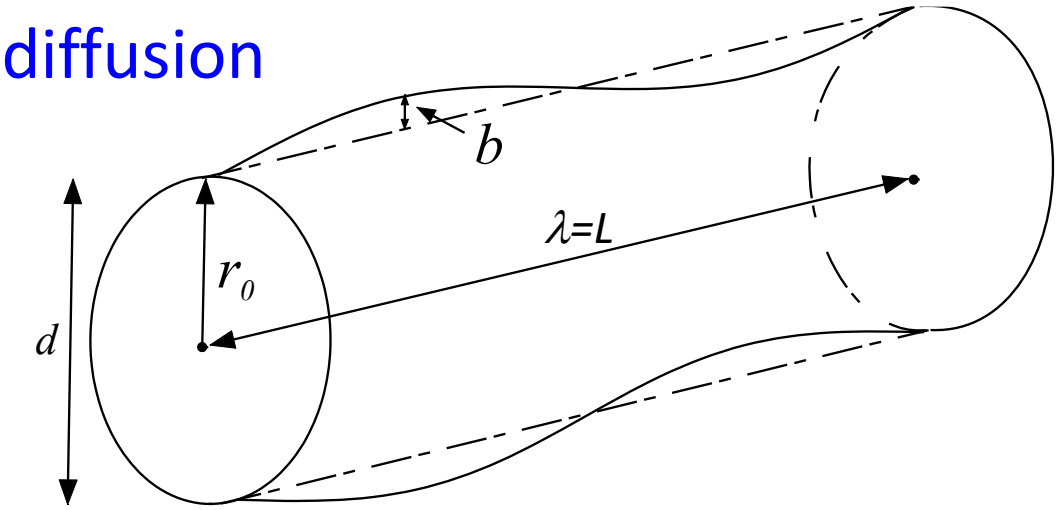


In collaboration with Wei Cai

R. Ramachandramoorthy, W. Cai, H.D. Espinosa, *ACS Nano*, 2016

Balancing Energies

NW with surface roughening via atomic diffusion



$\lambda=L$

$d=2r_0$

$k=\frac{2\pi}{L}$

$$W_e=\frac{1}{2}F\Delta$$

$$\Delta=\frac{F}{E}\int_0^L\frac{dx}{A(x)}\approx\frac{F}{E}\int_0^L\frac{dx}{\pi(r_0+b\sin kx)^2}$$

Taylor expansion and integration

$$\Delta=\frac{FL}{E\pi}\left(r_0^2+\frac{3}{2}b^2r_0^2\right)$$

Using the approximation: $\frac{1}{1+x}\approx 1-x$

$$F=\frac{E\pi r_0^2\Delta}{L}\left(1-\frac{3}{2}b^2\right)$$

Then,

$$W_e=\frac{1}{2}\frac{E\pi r_0^2\Delta^2}{L}\left(1-\frac{3}{2}b^2\right)$$

with $\Delta W_e=-\frac{3E\pi r_0^2b^2\Delta^2}{4L}$

The change in energy with change in surface area is:

$$\Delta W_s=2\gamma\pi r_0\int_0^L\left(\sqrt{1+(bk\cos kx)^2}-1\right)dx$$

Taylor expansion and integration

$$\Delta W_s=\frac{\gamma\pi r_0b^2k^2L}{2}$$

$$\Delta W_s=\Delta W_e$$

$$\varepsilon_c=\frac{2\pi}{\sqrt{3}\lambda}\sqrt{\frac{\gamma d}{E}}$$

From time scaling, i.e., λ^2/D, where D is the diffusivity:

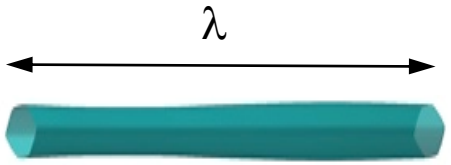
$$t=\frac{4\pi^2\gamma d}{3ED\varepsilon^2}$$

For silver: $\gamma=1\ \mathrm{J/m^2}$, $E=83\ \mathrm{GPa}$ and $D=10^{-17}\ \mathrm{m^2/s}$. For λ=400nm, d=100 nm, and ε=1%

$t\approx 4$ hours

Phase Field Model of Surface Diffusion

Faceted NW with a non-uniform cross-section

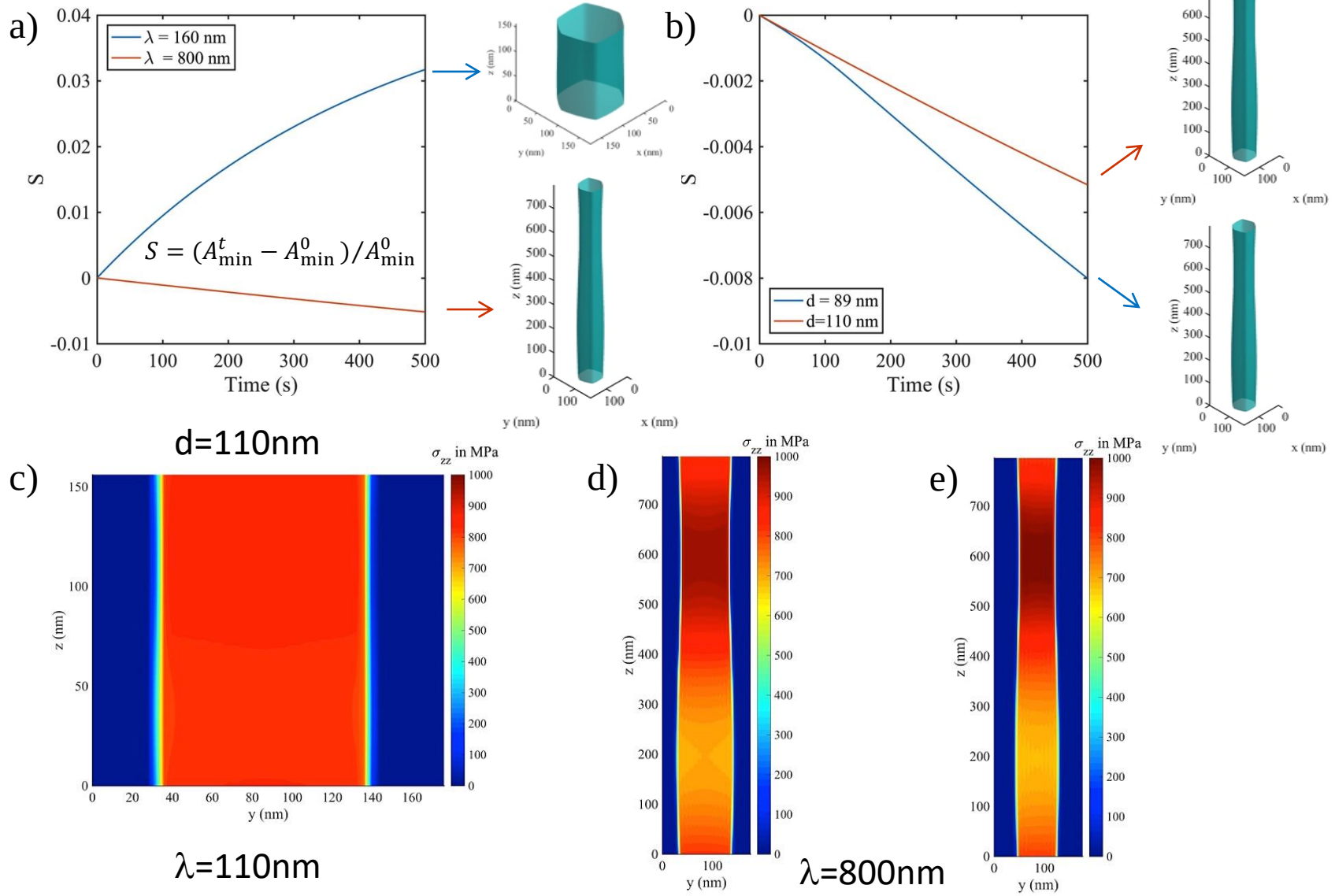


$$F[\emptyset(\boldsymbol{x})+u(\boldsymbol{x})]=F^{in}(\emptyset(\boldsymbol{x}),\nabla\emptyset(\boldsymbol{x}))+F^{el}(\nabla u(\boldsymbol{x}),\emptyset(\boldsymbol{x}))$$

$$u_i(\boldsymbol{x},t+\Delta t)=u_i(\boldsymbol{x},t)-\frac{\delta F^{el}}{\delta u_i}\Delta t;$$

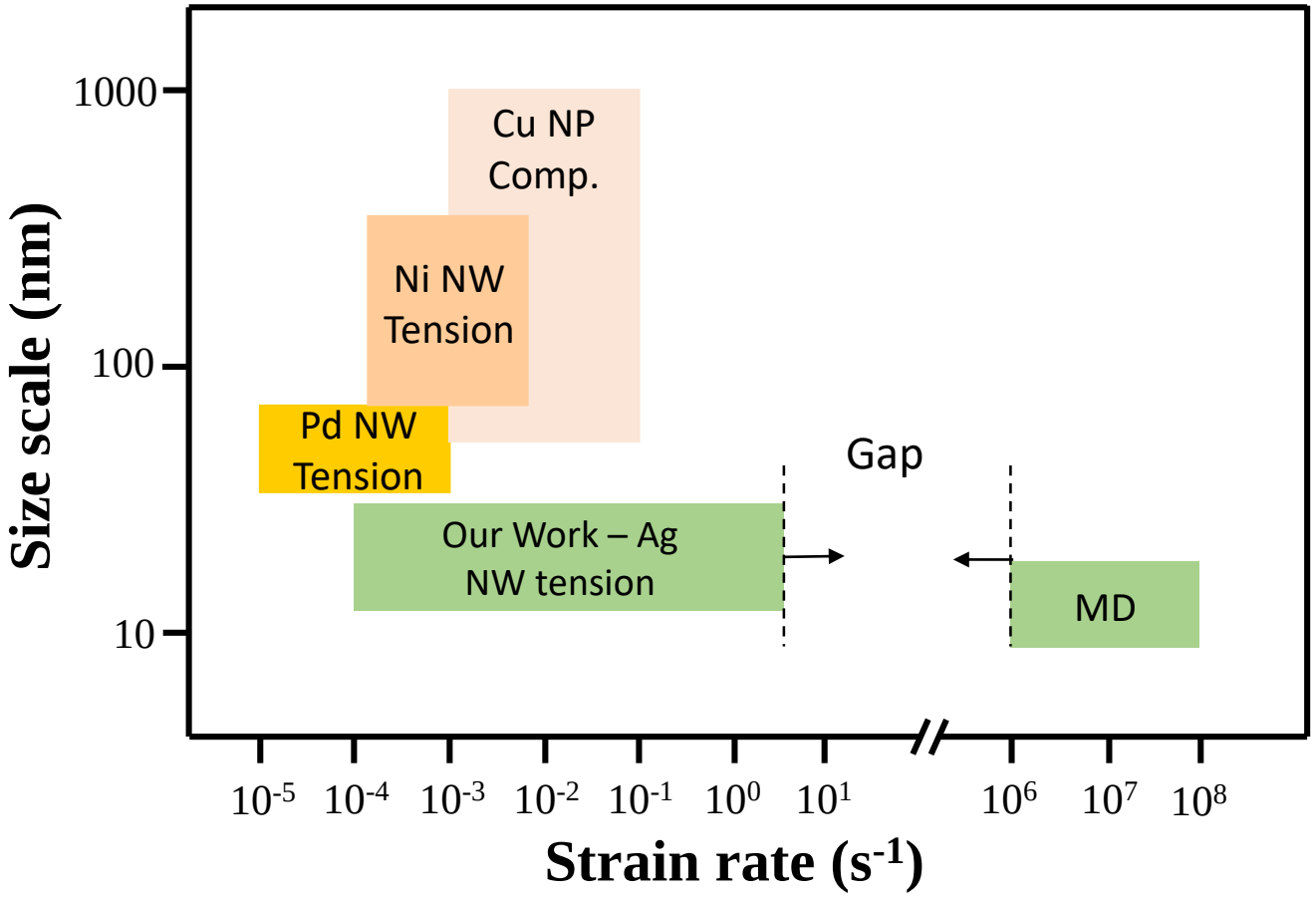
$$\frac{d\emptyset}{dt}=K\nabla^2\left(\frac{\delta F^{in}}{\delta\emptyset}+\frac{\delta F^{el}}{\delta\emptyset}\right)$$

Cahn-Hilliard

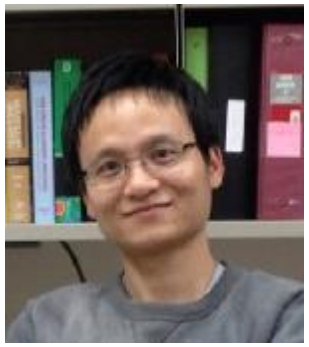


R. Ramachandramoorthy, **W. Cai**, H.D. Espinosa, *ACS Nano*, 2016

Strain Rate Effects – Closing the Gap with MD

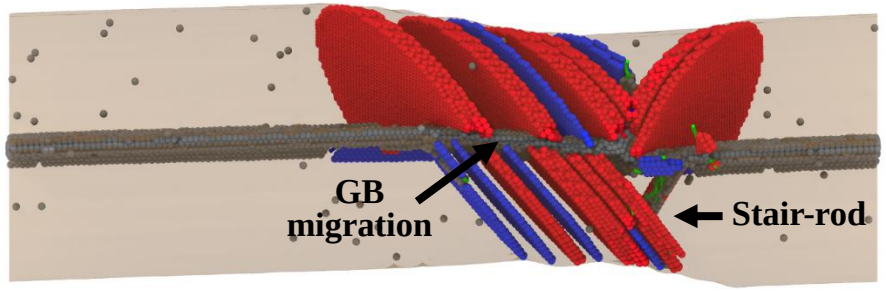


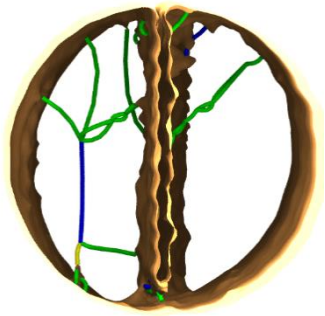
Brittle to Ductile Transition – Rate Effects

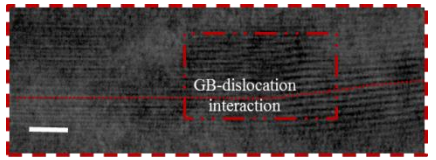


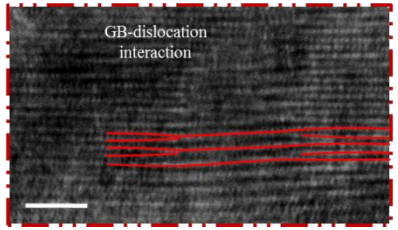
Wei Gao

Bi-crystal Ag Nanowires

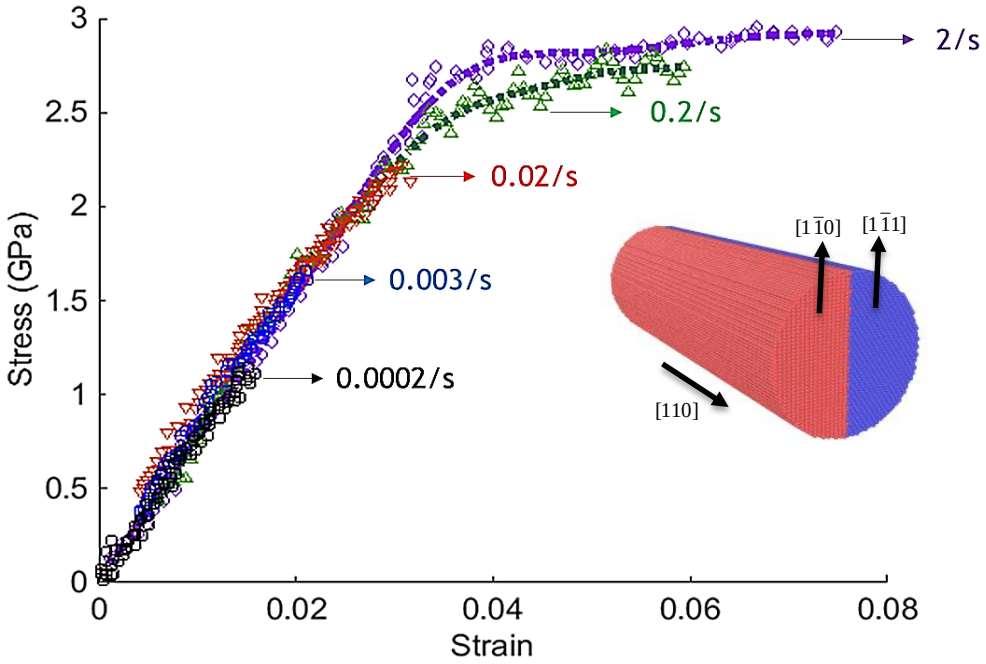


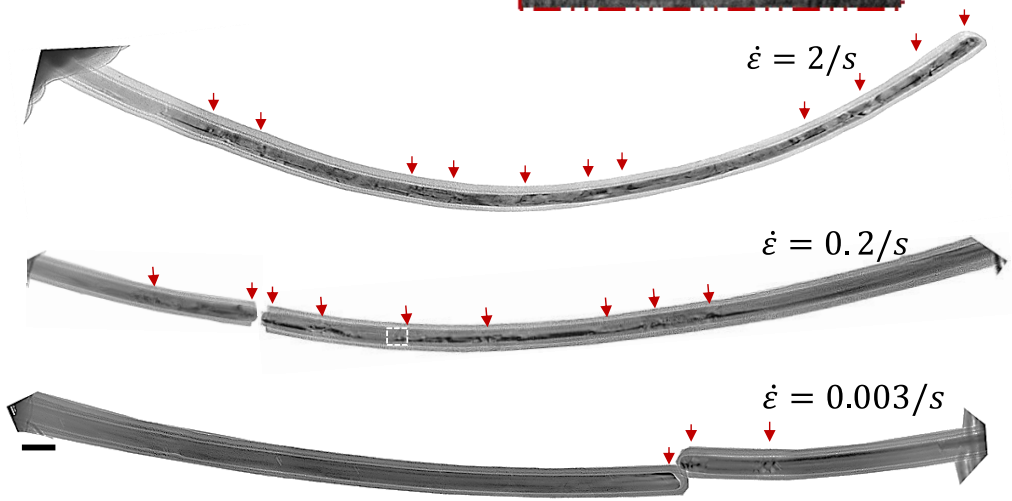






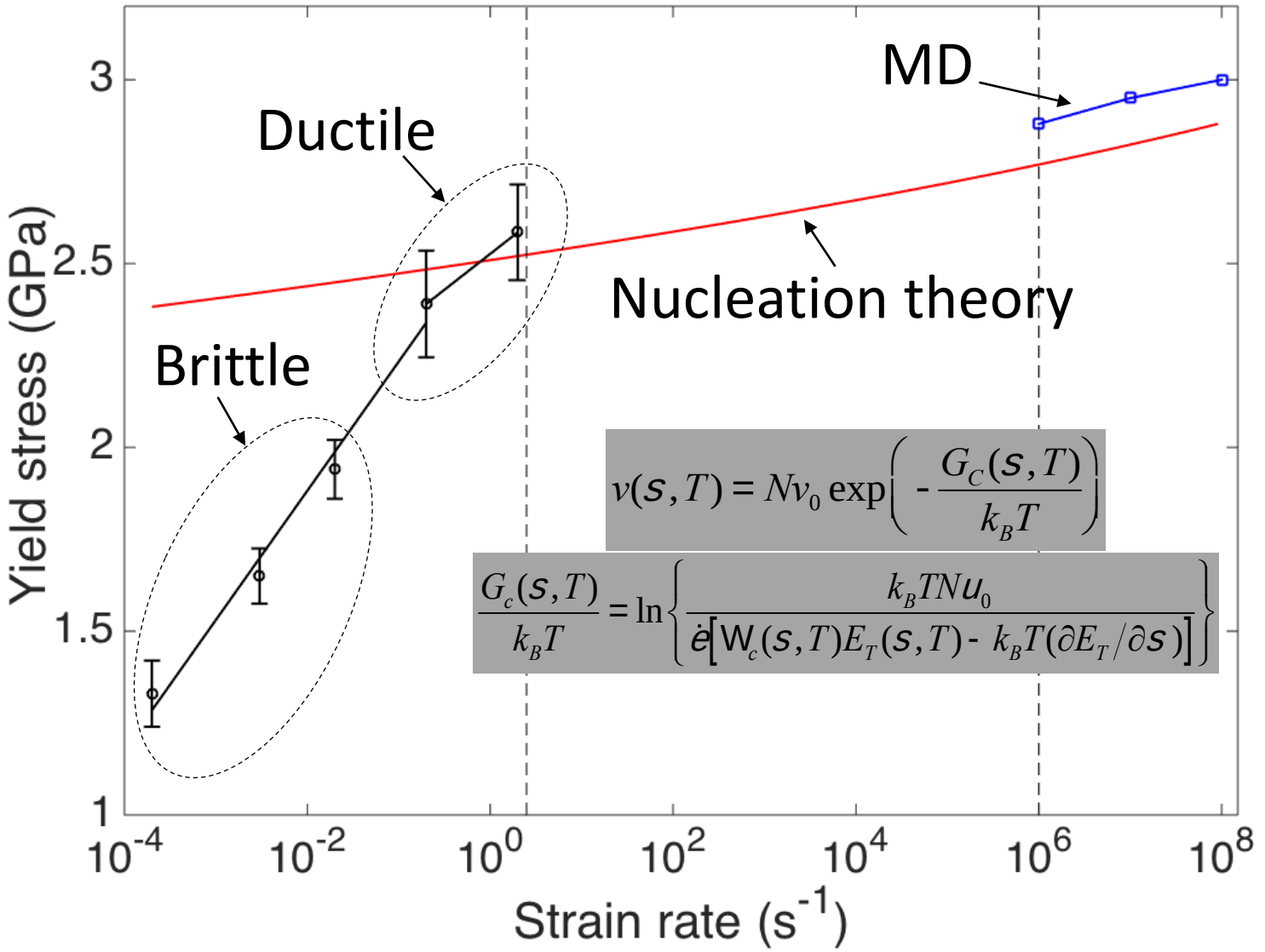
Raj





Ramachandramoorthy, Gao, Bernal, Espinosa, *Nano Letters,* **2015**

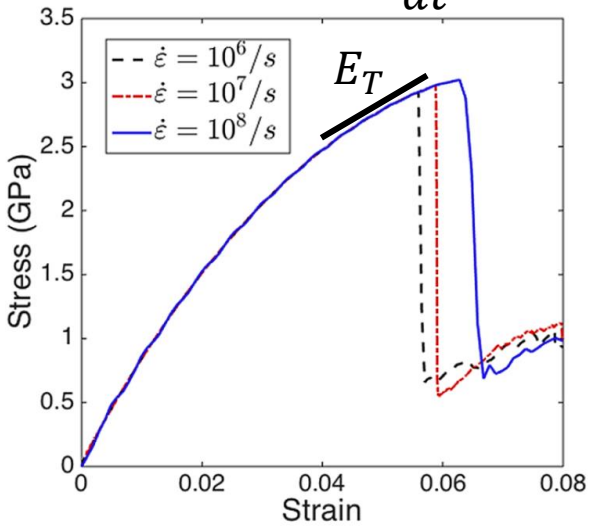
Nucleation Theory Predictions



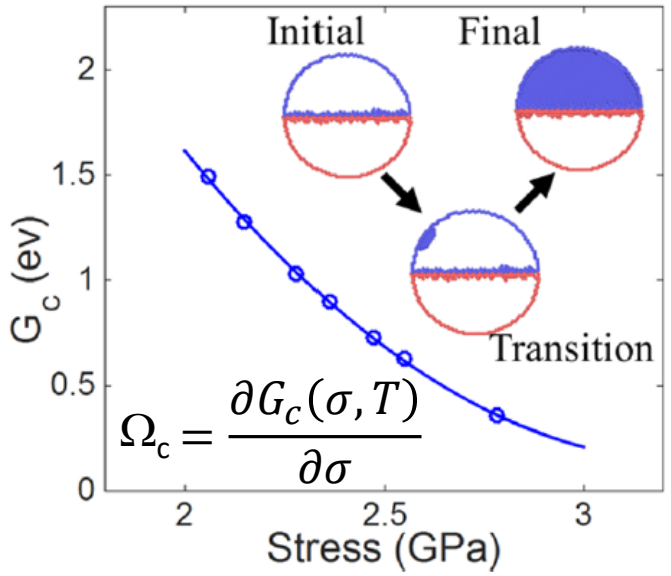
$$v(s,T) = Nv_0 \exp\left(-\frac{G_C(s,T)}{k_B T}\right)$$

$$\frac{G_c(s,T)}{k_B T} = \ln\left\{\frac{k_B T N u_0}{\dot{e}\left[W_c(s,T)E_T(s,T) - k_B T(\partial E_T/\partial s)\right]}\right\}$$

$$\frac{d\sigma}{dt} = E_T(\sigma,T)\frac{d\varepsilon}{dt}$$



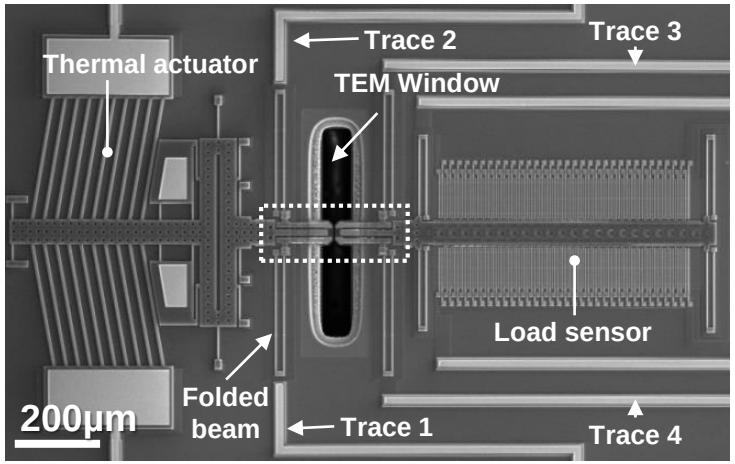
$G_c(\sigma,T)$: *Gibbs free energy*

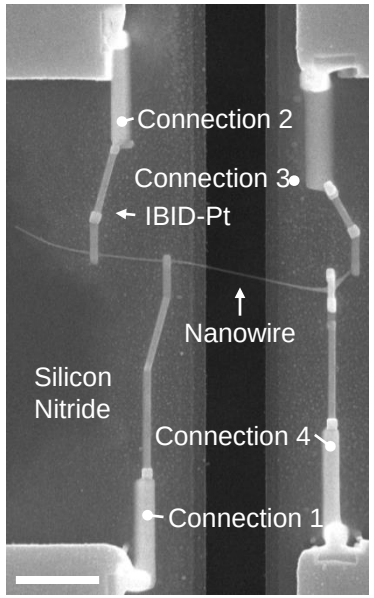


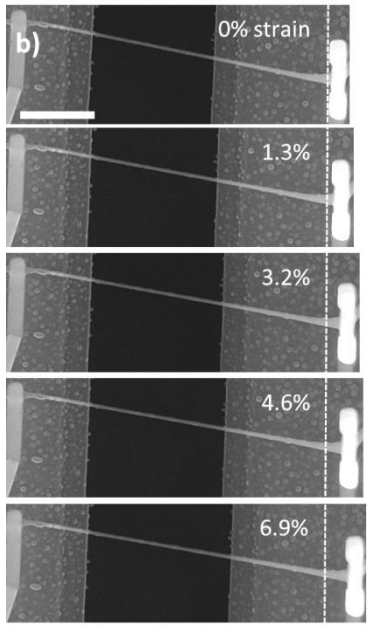
R. Ramachandramoorthy, W. Gao, R. Bernal, Espinosa, *Nano Letters,* **2015**
T. Zhu et al., PRL, **2008**

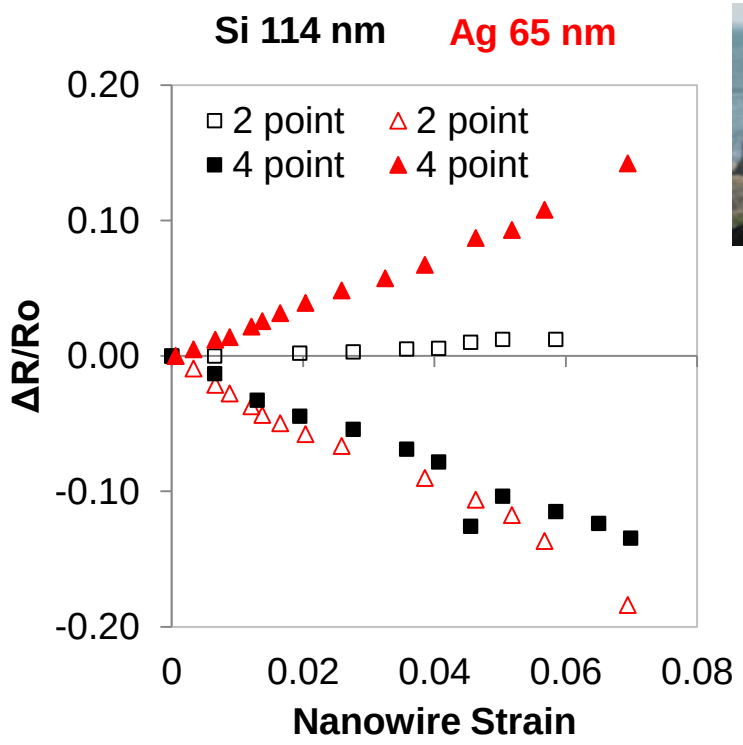
Nanoscale Measurements of Coupled Fields

Giant Piezoresistivity in Si NWs?









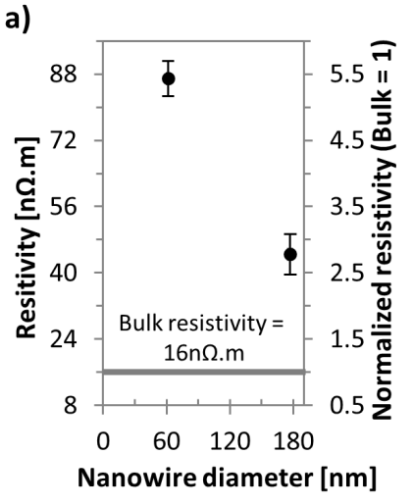
R. Bernal

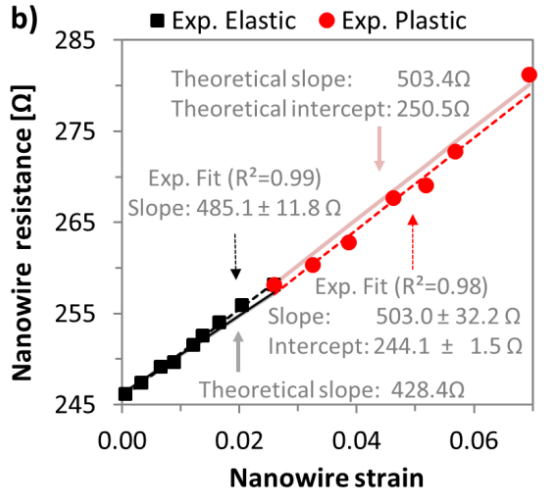
n-type Si NWs
Strains in excess of 7%
π_1 Si NW 4 times π_1 bulk

$$\frac{\Delta R}{R_0}(\varepsilon) = (1+2\nu)\varepsilon + \frac{\Delta\rho}{\rho_0}$$

$$\frac{\Delta\rho}{\rho_0} = \pi_1\sigma + \ldots$$

Si NW Diameter (nm)	Piezoresistive Coefficient (x 10^{-11} Pa)
114	-2.1
141	-3.7
Bulk [111]	-7.53



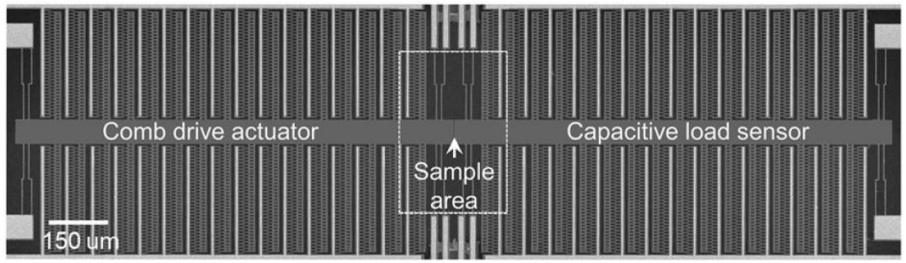


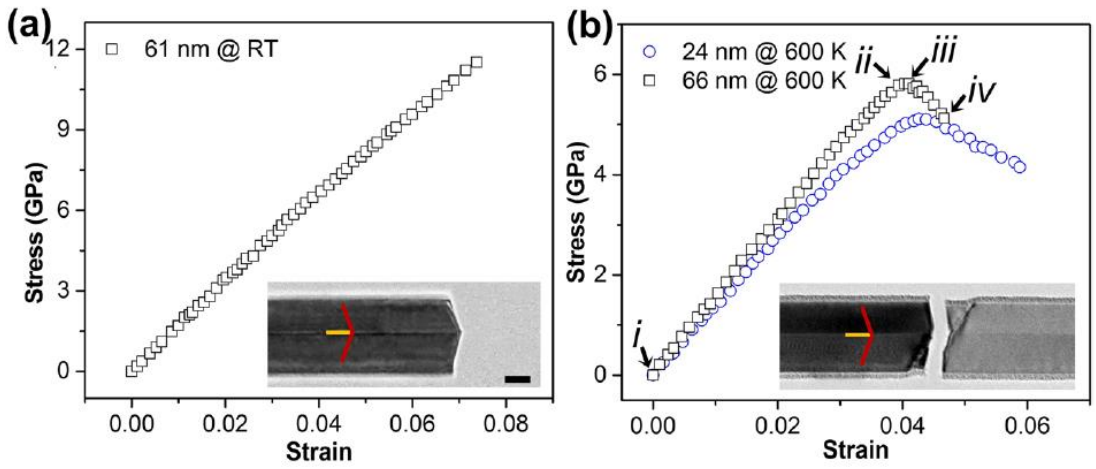
Bernal, Espinosa et al. *Small,* **2013**

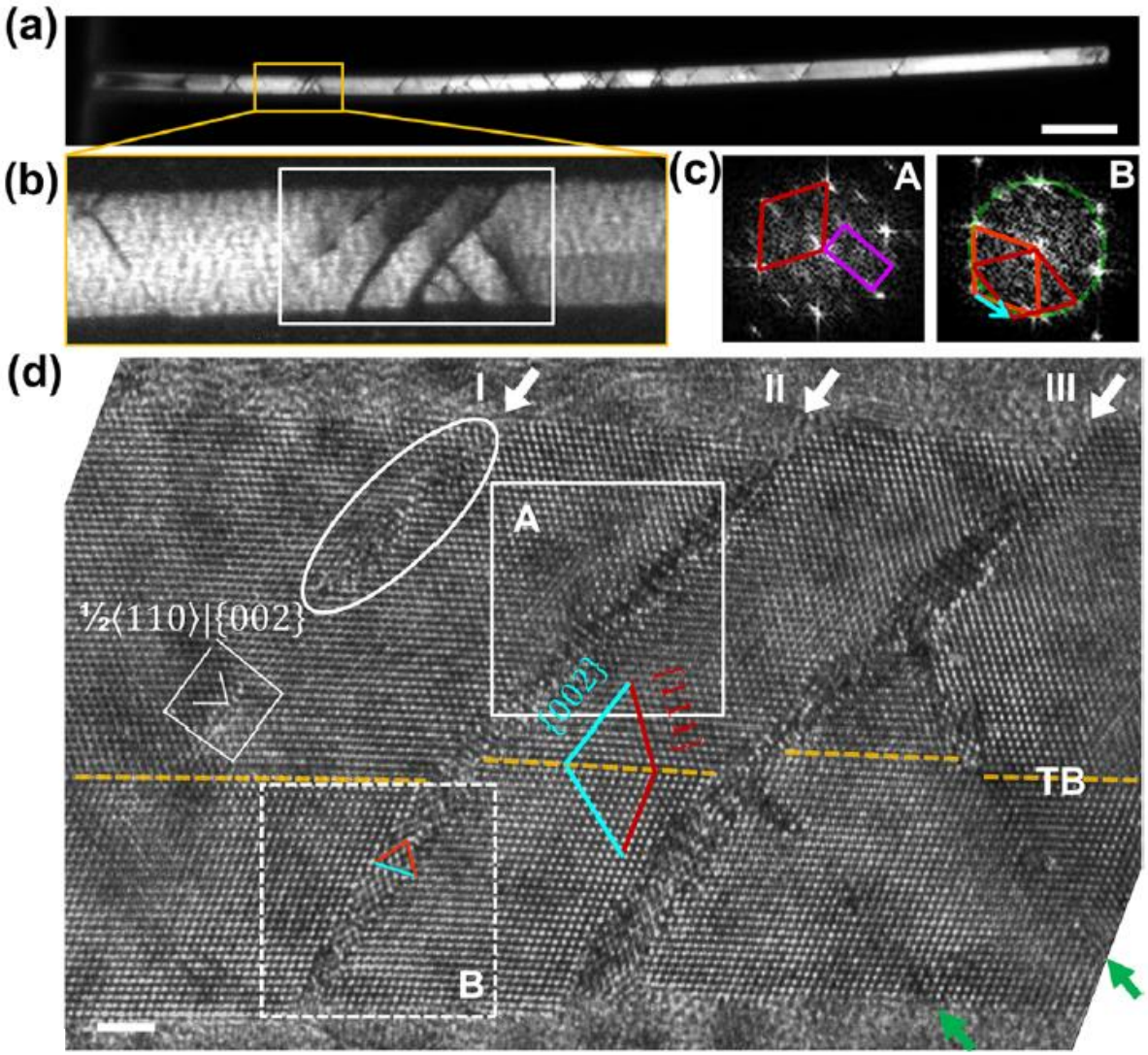
Brittle to Ductile Transition - Temperature Effects

Yong Zhu

MEMS with heating elements



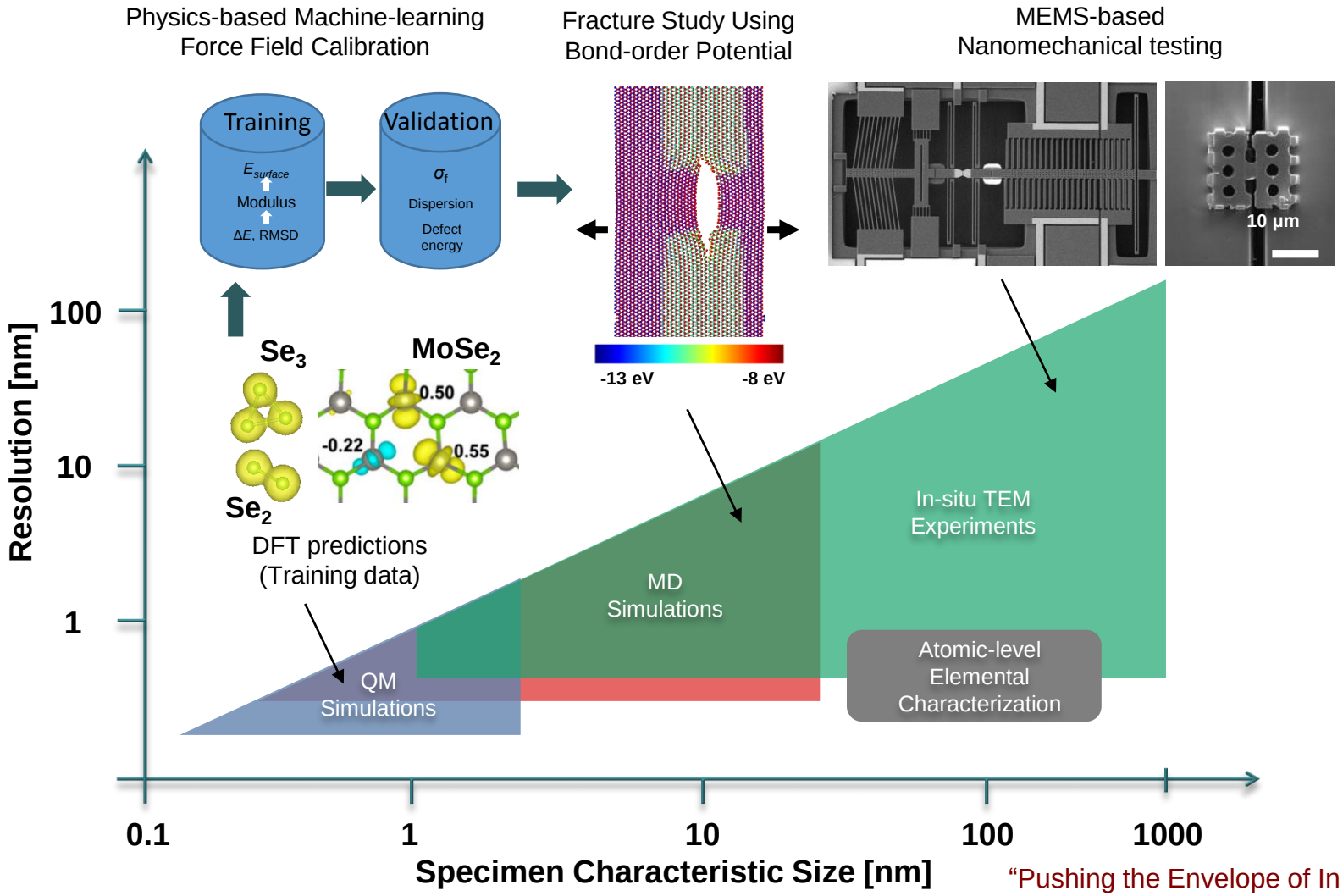


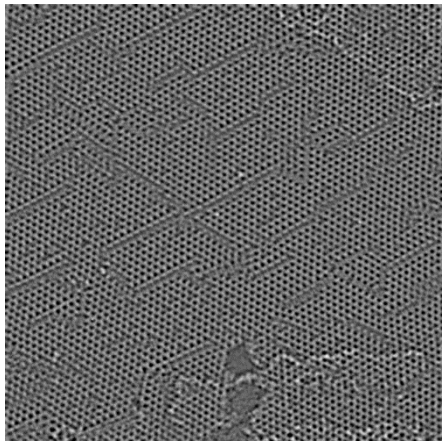


Cheng, T. Zhu, Y. Zhu, et al., *Nano Letters*, 19, 2019

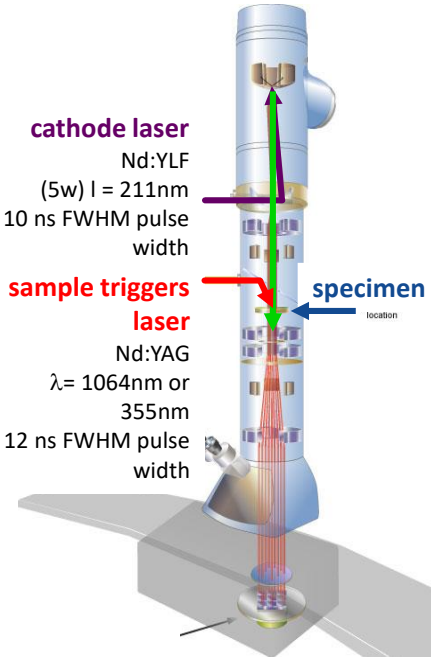
Opportunities

- Resolving time scales: ultra fast microscopy
- Fracture of 2D materials; Force fields parametrized using ML





Wang, Shanshan, et al. *ACS Nano* (2016).



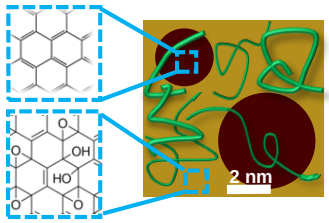
Ultra Fast - TEM

"Pushing the Envelope of In Situ Transmission Electron Microscopy," Ramachandramoorthy, Bernal, Espinosa, *ACS Nano,* **2015**

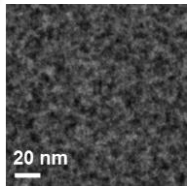
Using Chemistry to Toughen 2D Materials

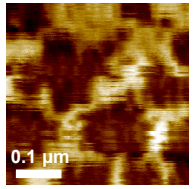
Case Study: GO-PVA Nanolaminates

H. Nguyen



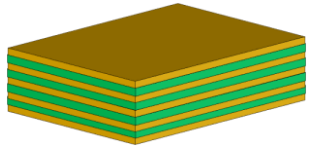
Functional Groups Mediated Hierarchical Assembly

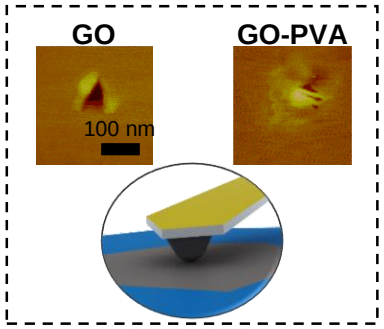


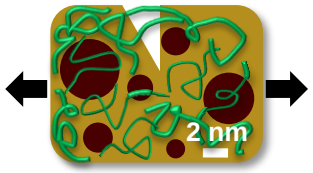


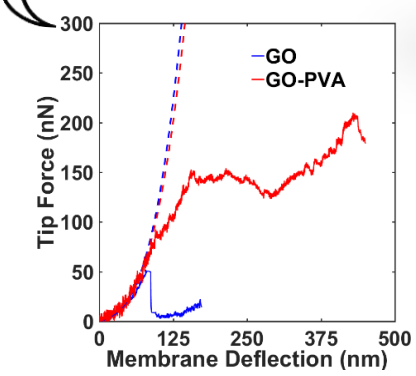
AFM Membrane Deflection

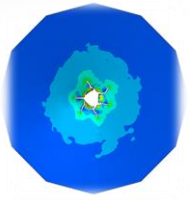
Architectural-defect-free nanocomposites



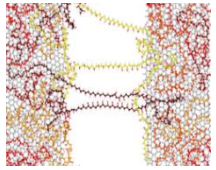




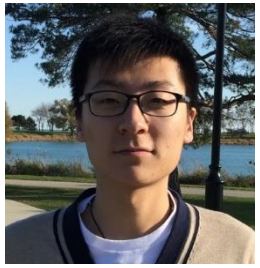




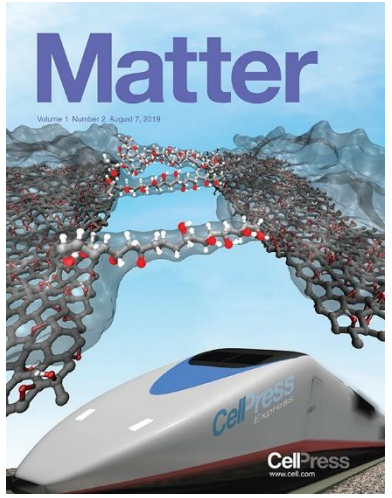
Piercing Resistance



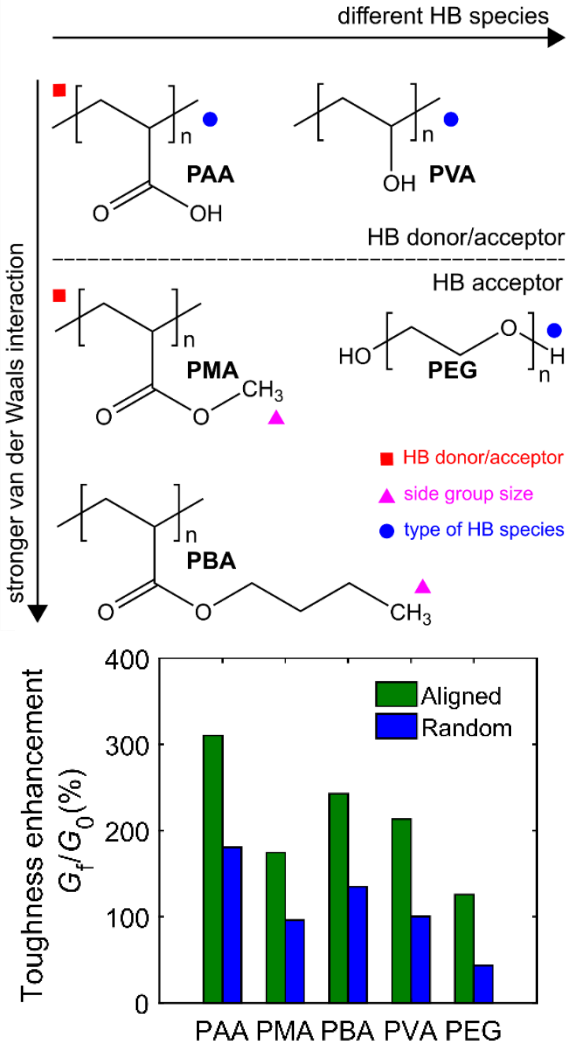
Polymer Crack Bridging



Xu Zhang



Computational exploration of polymer chemistry



Soler-Crespo, Espinosa, et al. "Atomically Thin Polymer Layer Enhances Toughness of Graphene Oxide Monolayers," *Matter* (2019).

Zhang, Xu, Espinosa et al., *Nanoscale,* 11, 2019

Acknowledgements

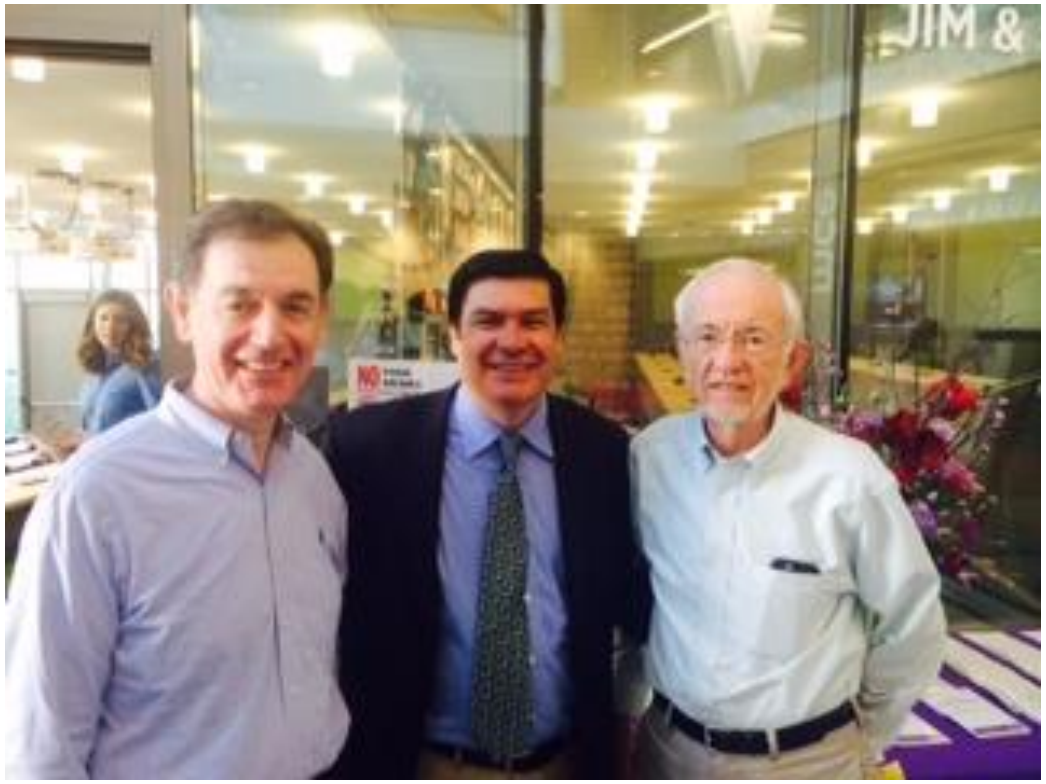


Photo: *James K. Knowles* Lecture, Caltech, 2016

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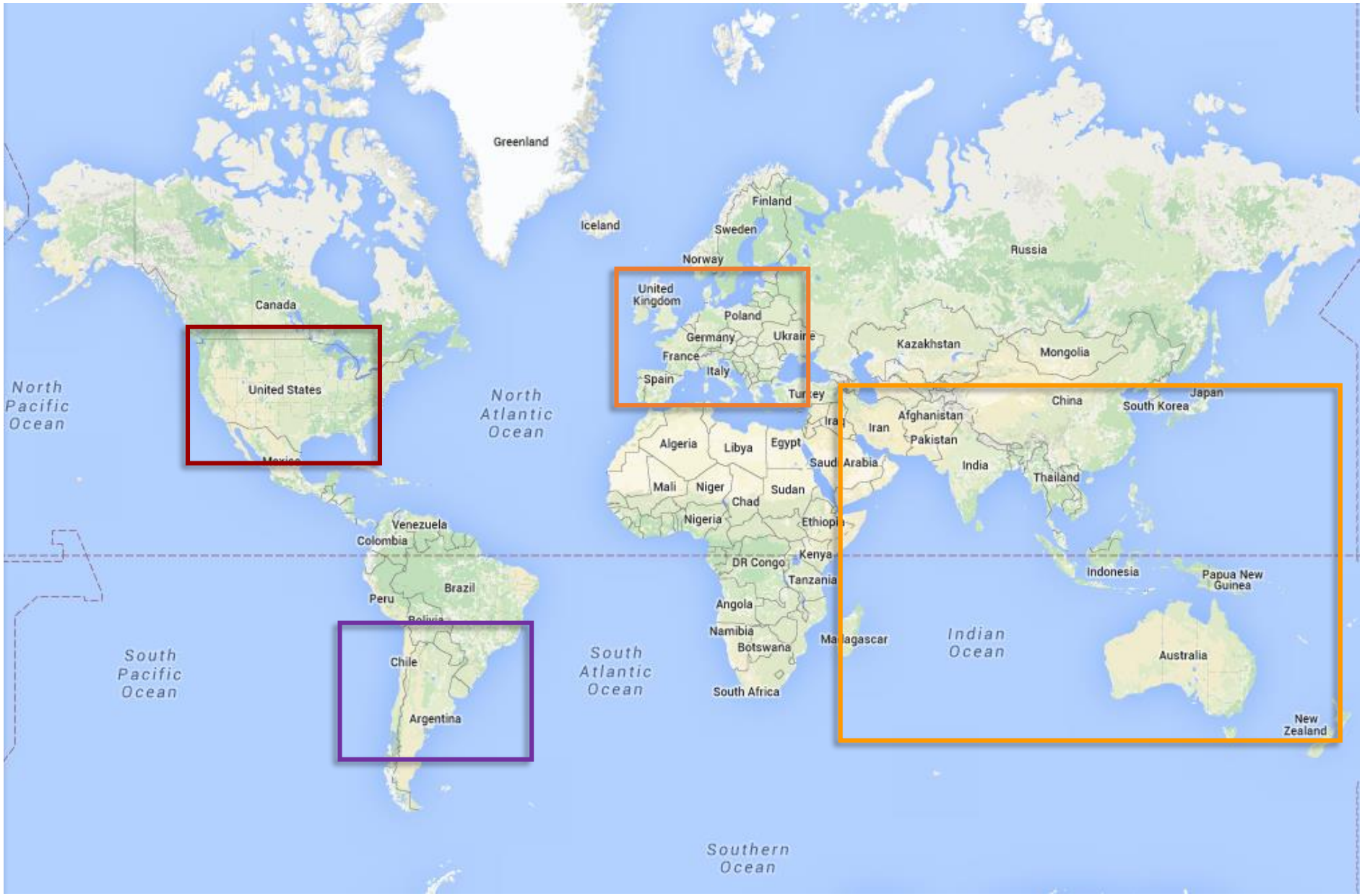

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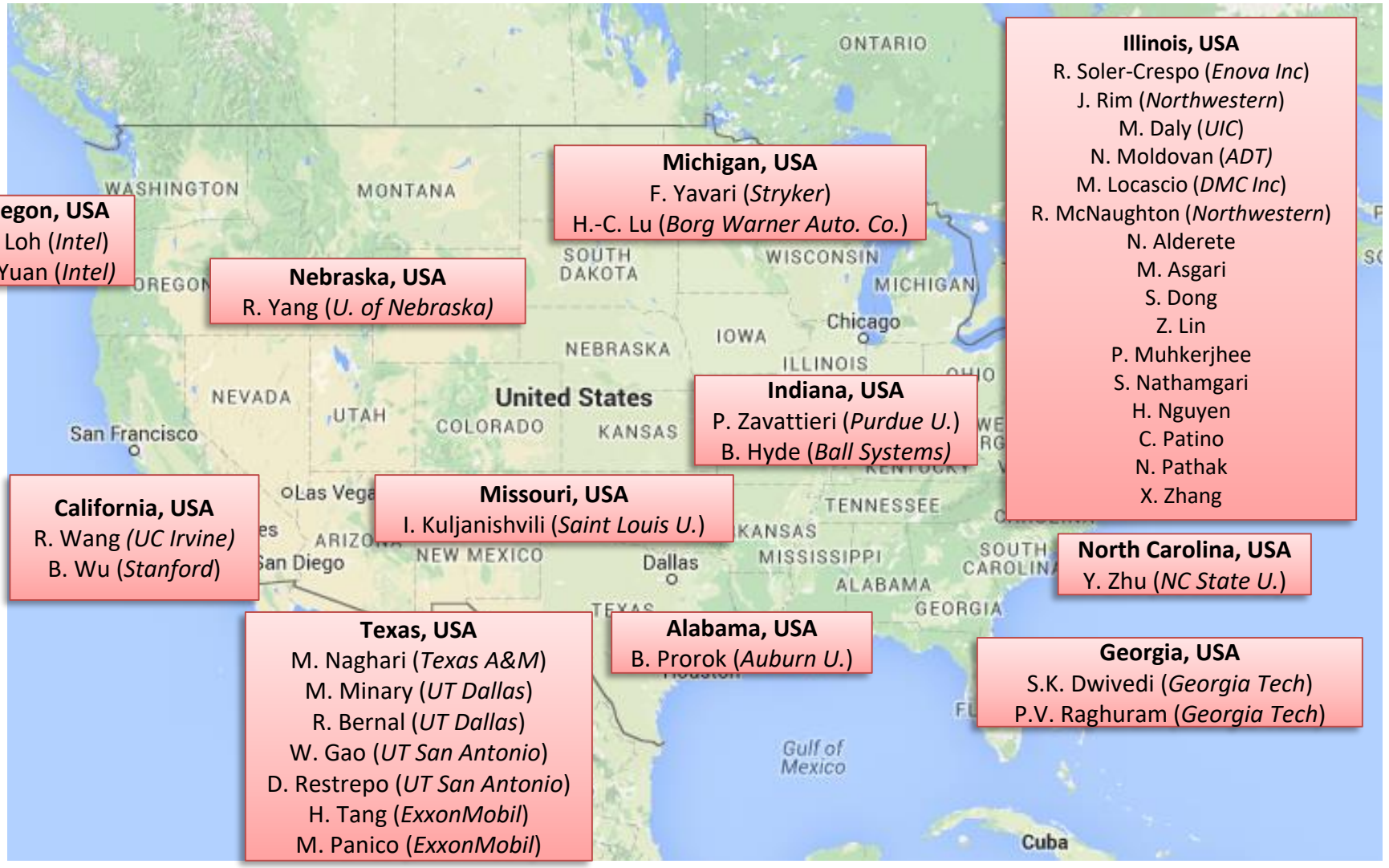

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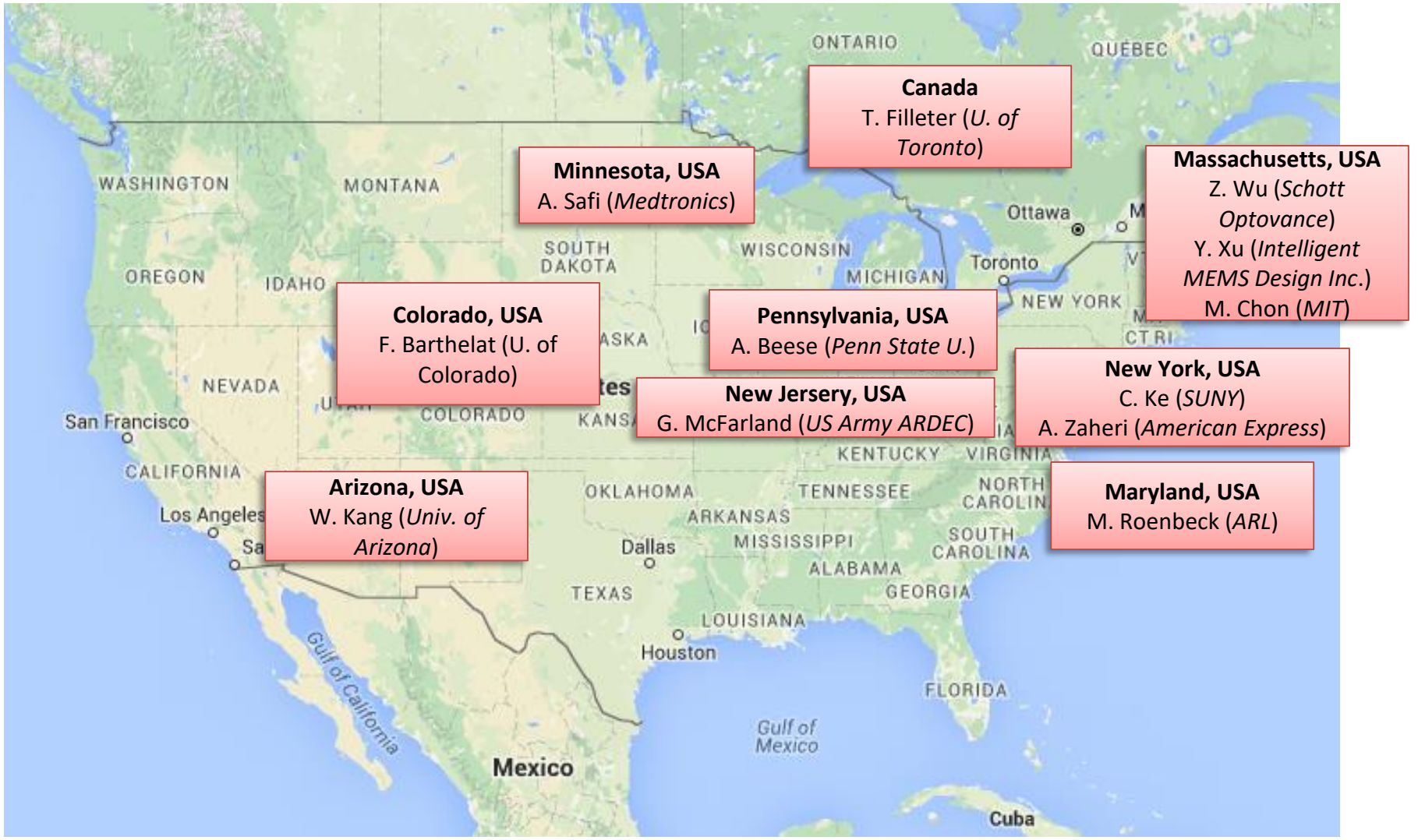

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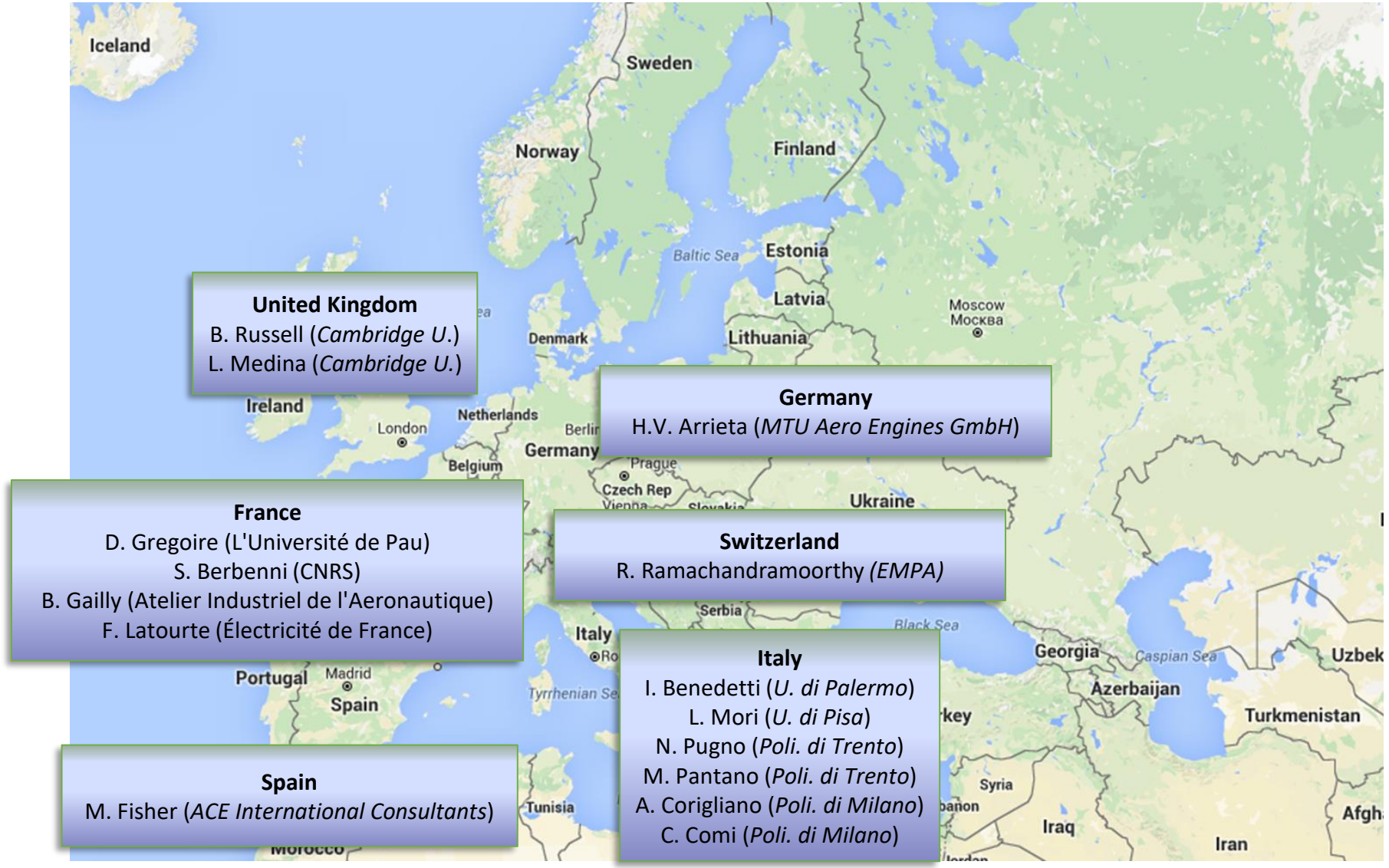

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Thanks!