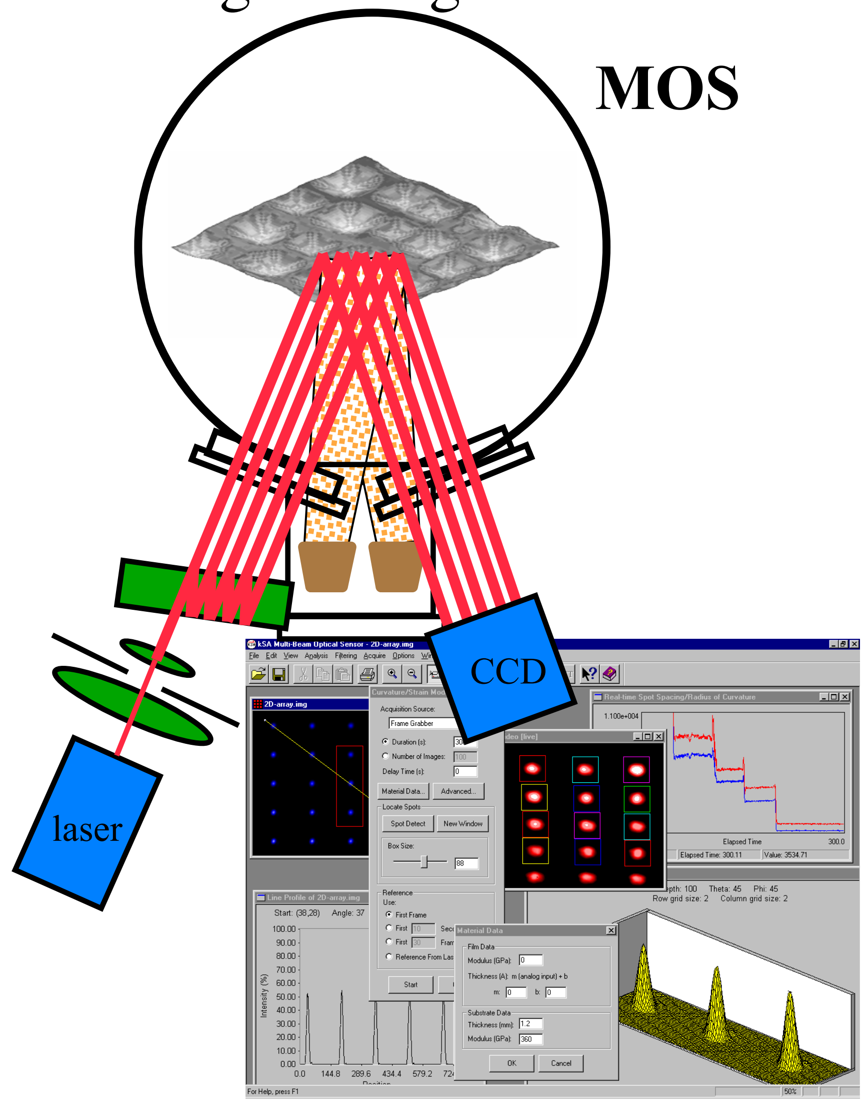
Measurement of stress evolution in thin films using real-time *in situ* wafer curvature (k-Space MOS)

Eric Chason
Brown University, School of Engineering

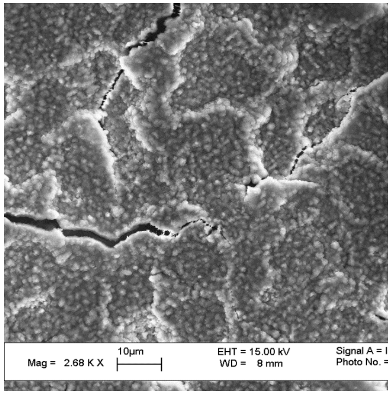
- Intro to k-Space MOS (multi-beam optical sensor)
    - theory
    - capabilities
    - analysis
- Examples
    - polycrystalline films
        - steady-state stress
          Scaling with D/RL
        - stress vs thickness
    - sputtering
    - tin whisker formation
    - battery materials



Stress in thin films is a generic problem

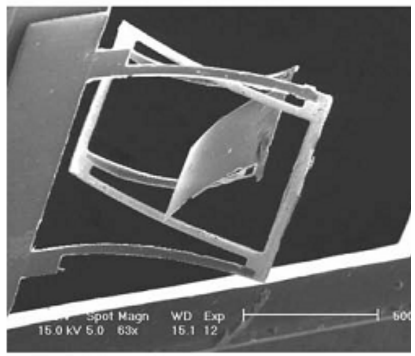
Leads to decreased performance, deformation, failure

Cracks in Sn-Li electrodes



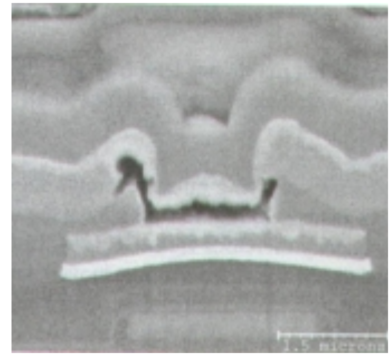
Chao and Guduru. , MRS, 2012

Deformation in Ni MEMS devices



Matovic et al. , J. of Mech. Eng. Sci.2006

Stress voiding/ electromigration



Sn Whiskers

NASA website: http:// nepp.nasa.gov/whisker

Thin Solid Films review, 2012

Contents lists available at SciVerse ScienceDirect

Thin Solid Films

journal homepage: www.elsevier.com/locate/tsf

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Critical review

A kinetic analysis of residual stress evolution in polycrystalline thin films

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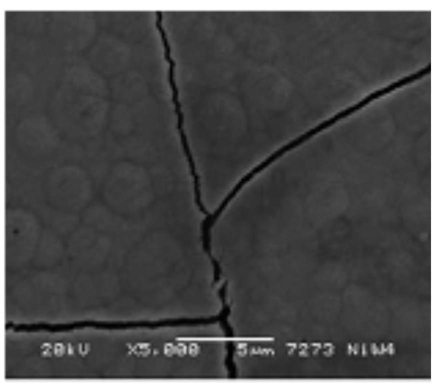
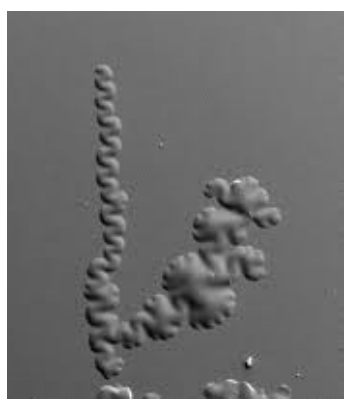
Stress in electro-plated NiW



Mizushima et al. , Electrochimica Acta. 2006

Diamond on Si delamination



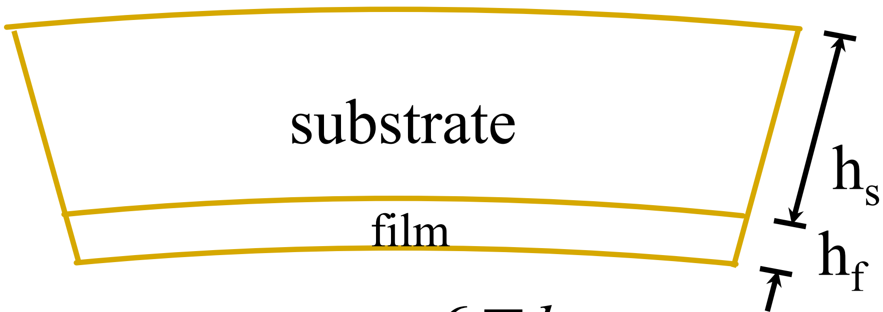
diamond.kist.re.kr/DLC/ mwmoon/gallery.htm

Want to:
understand stress,
control stress,
predict stress

→ **First need to measure it**

Measure thin film stress via wafer curvature

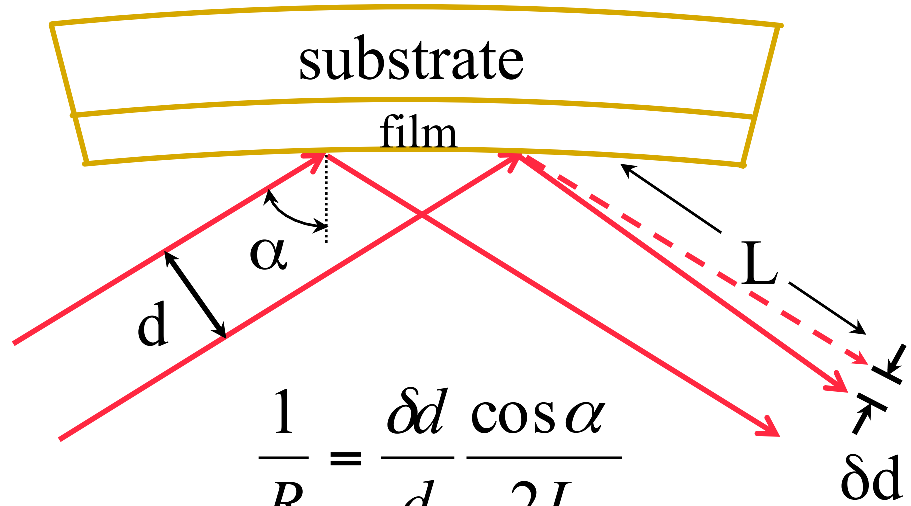
Stressed film bends the substrate
Stoney's equation



$$\kappa = \frac{1}{R} = \frac{6\bar{\sigma}h_f}{M_s h_s^2}$$

For multiple layers: $$\frac{1}{R} = \frac{6}{M_s h_s^2}\sum_i \bar{\sigma}_i h_i$$

MOSS (multi-bean optical sensor)
Curved surface deflects
array of parallel beams

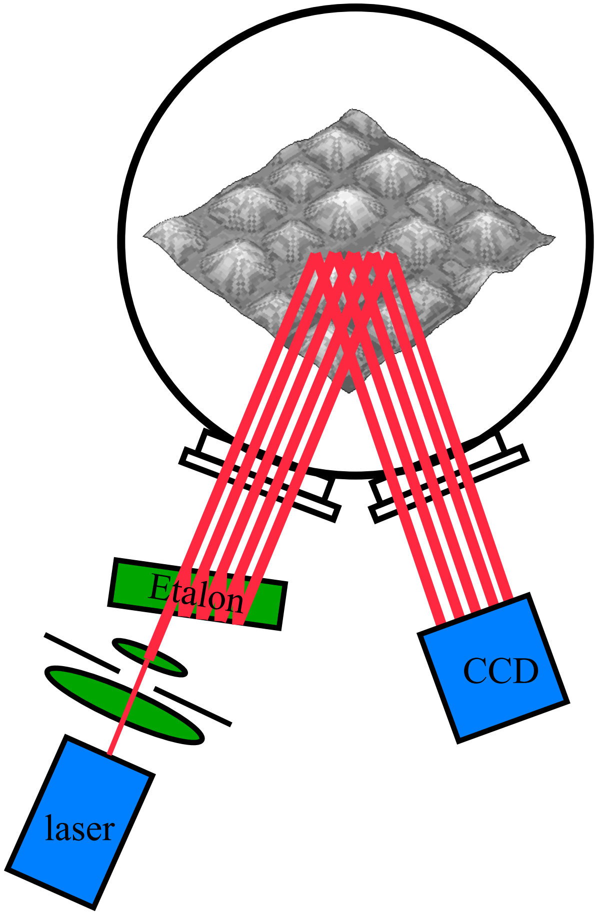


$$\frac{1}{R} = \frac{\delta d}{d}\frac{\cos\alpha}{2L}$$

Curvature measures <u>product</u> of average stress x thickness

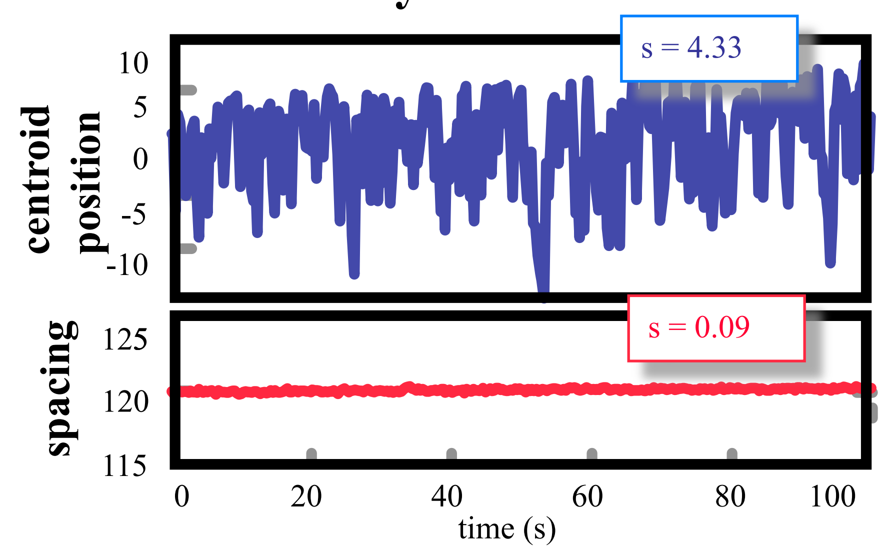
$$\bar{\sigma} h_f = \frac{\delta d}{d}\frac{M_s h_s^2 \cos\alpha}{12L}$$

Multibeam approach (MOSS): easy to implement/robust



- System requirements:
  - Ports to measure specular reflection
  - Reflective surface (backside ok)
- Measurement technique
  - Etalon produces array of parallel beams
  - CCD measures change in beam spacing (δd/d) $\Rightarrow \frac{1}{R} = \frac{\delta d}{d}\frac{\cos\alpha}{2L}$

Multi-beam technique reduces sensitivity to vibration



Measure difference between beams not absolute position

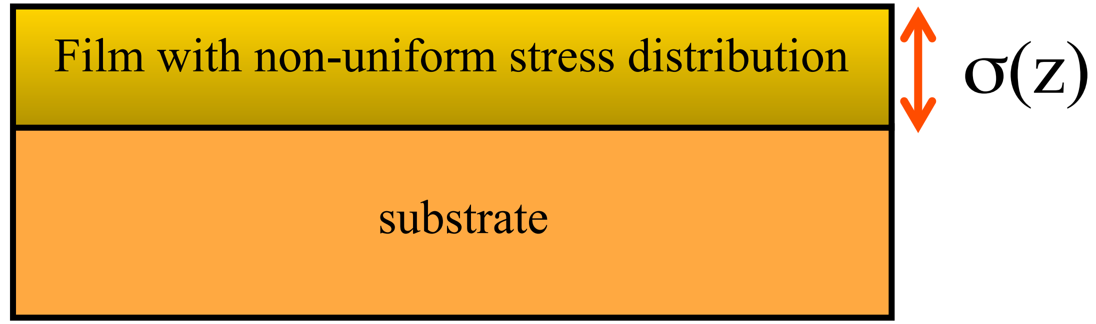
- Simple, stable optics (aligned outside processing chamber)
- in situ, real-time, high sensitivity
- R> 20 kM, σh_f < 1 GPa-Å
- Can see 0.1 ML Ge on Si(001)

Interpreting curvature measurements:

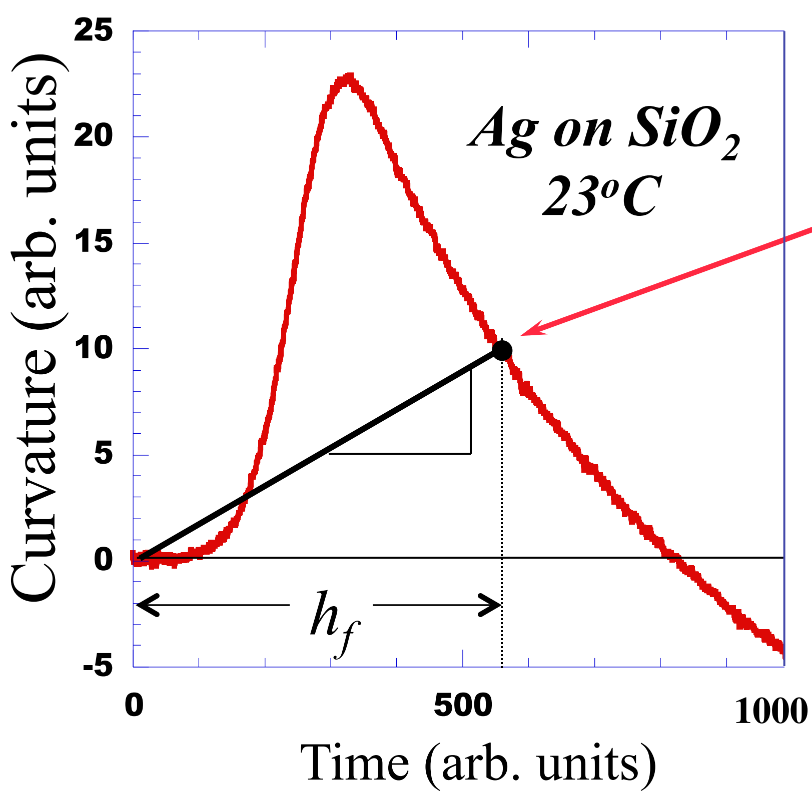
How does curvature relate to evolving stress distribution?

Average stress:

$$\bar{\sigma} = \frac{1}{h_f}\int_0^{h_f}\sigma(z)\,dz$$



Curvature vs. time



$$\bar{\sigma} = \frac{M_s h_s^2}{6}\frac{\kappa}{h_f}$$

-Thickness changes with time (deposition)

- Curvature changes as film grows over time

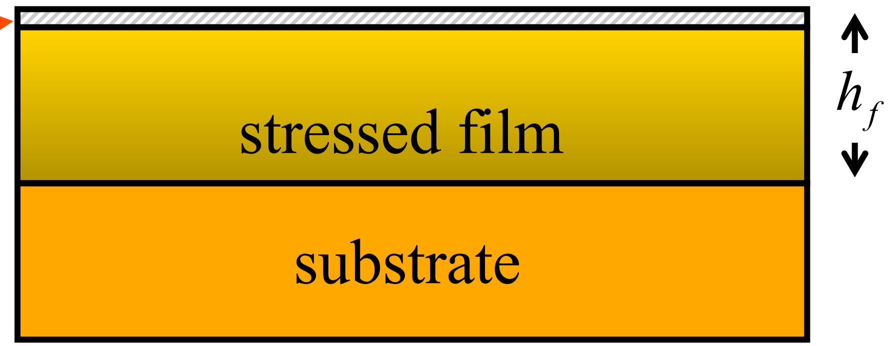
- Shows stress is not uniform throughout film

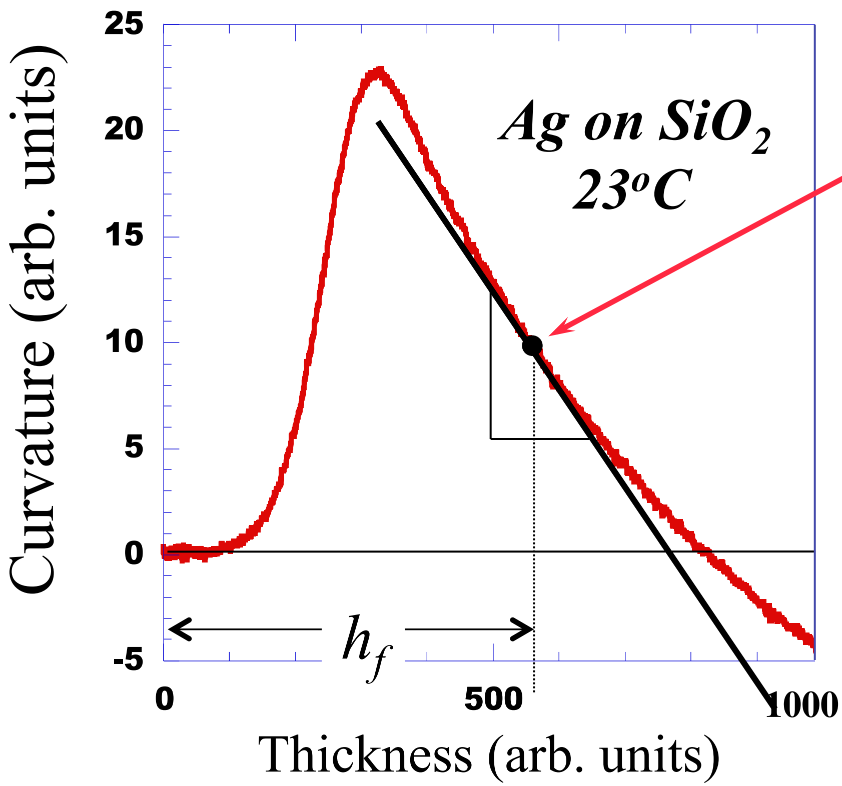
***Stress distribution*:**

Study change of curvature with time

$$\frac{d\kappa}{dt} \propto \boxed{\sigma(h_f,t)\frac{\partial h}{\partial t}} + \int_0^{h_f} \frac{\partial \sigma(z,t)}{\partial t} dz$$

(1) Stress in new layers at the surface, $\sigma(h_f,t)$





$$\frac{d\kappa}{dh} = \frac{d\kappa / dt}{dh / dt} \propto \sigma(h_f,t)$$

-<u>Incremental stress</u> proportional to slope of κ vs h

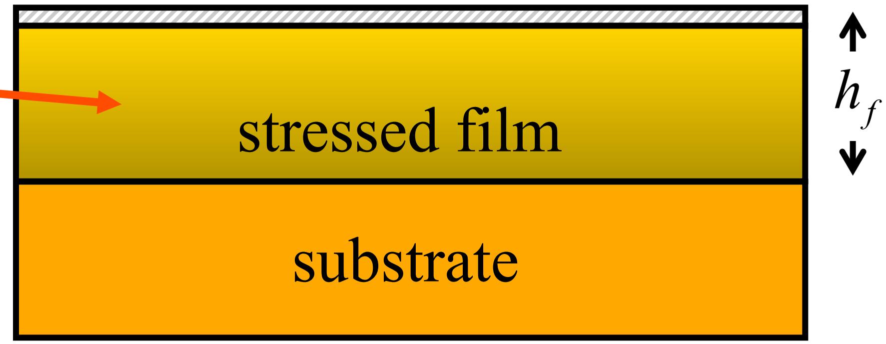
- But only if stress not changing in rest of film

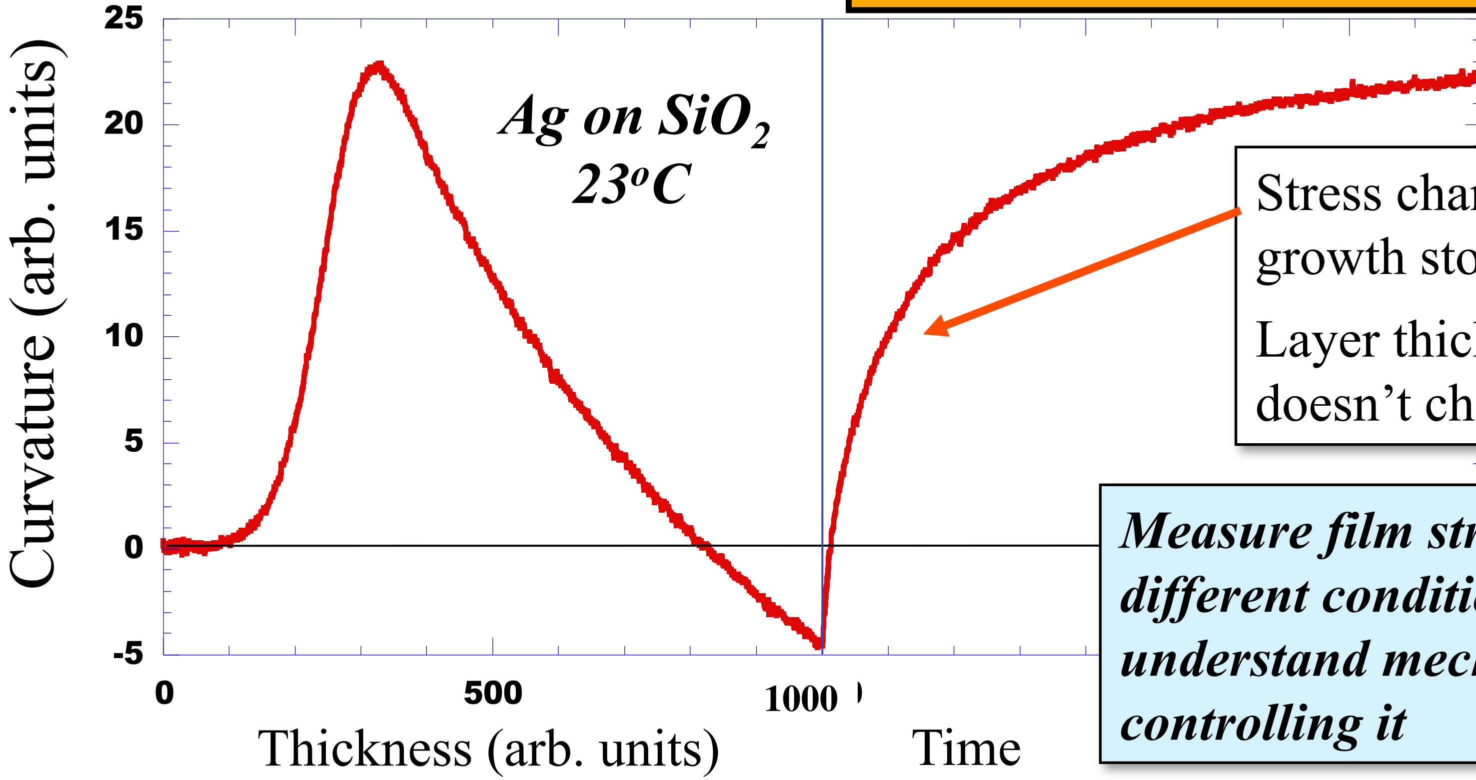
Stress distribution:

Study change of curvature with time

$$\frac{d\kappa}{dt} \propto \sigma(h_f, t)\frac{\partial h}{\partial t} + \int_0^{h_f} \frac{\partial \sigma(z,t)}{\partial t}\,dz$$

(2) Change in stress of existing layers





Stress changes when growth stopped

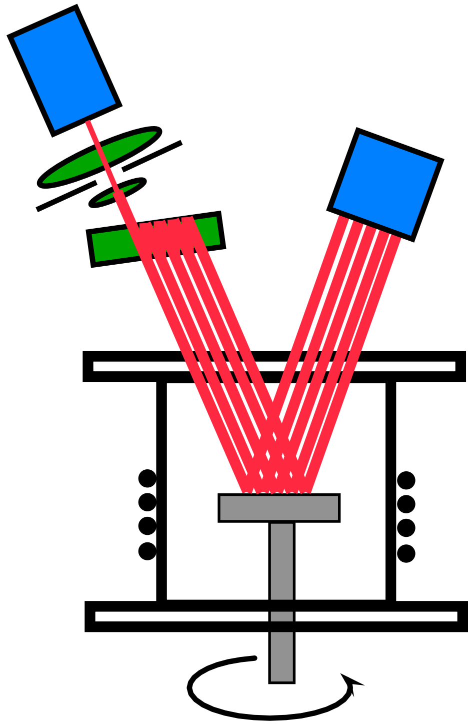
Layer thickness doesn't change

Measure film stress under different conditions to understand mechanisms controlling it

MOS can be implemented on many platforms

Deposition techniques

- *CVD*
- *sputtering*
- *PVD*
- *MBE*
- *PLD*
- *electrodeposition*



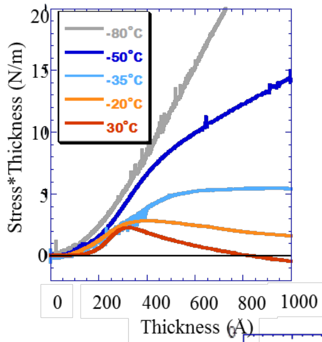
MOS on GaN rotating disk CVD reactor

(Hearne et al)

Materials systems

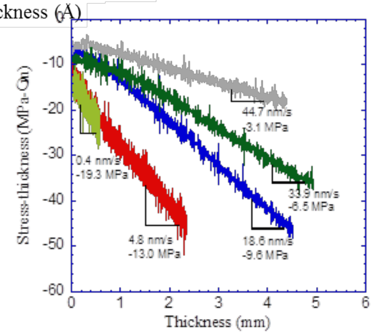
- *heteroepitaxy (SiGe/Si, InGaAs/GaAs)*
- *optoelectronics (GaN, AlGaN, GaSb)*
- *hard coatings (DLC, a-C)*
- *oxides (TiO_2, CeO_2)*
- *polycrystalline metals*

Examples from stress evolution studies



1. Residual stress in polycrystalline films

- *Electrodeposition/evaporation*
- *Dependence on growth conditions, material*
- *Evolution with film thickness*



2. Sputter deposition

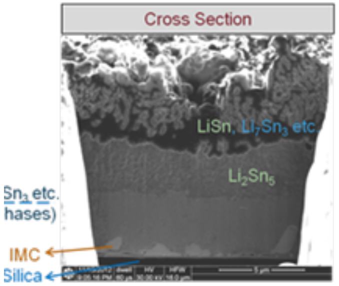
- *Effect of processing parameters (surface roughness)*

3. Mechanical properties of Sn films

- *stress leads to whiskers*
- *enhance stress relaxation*

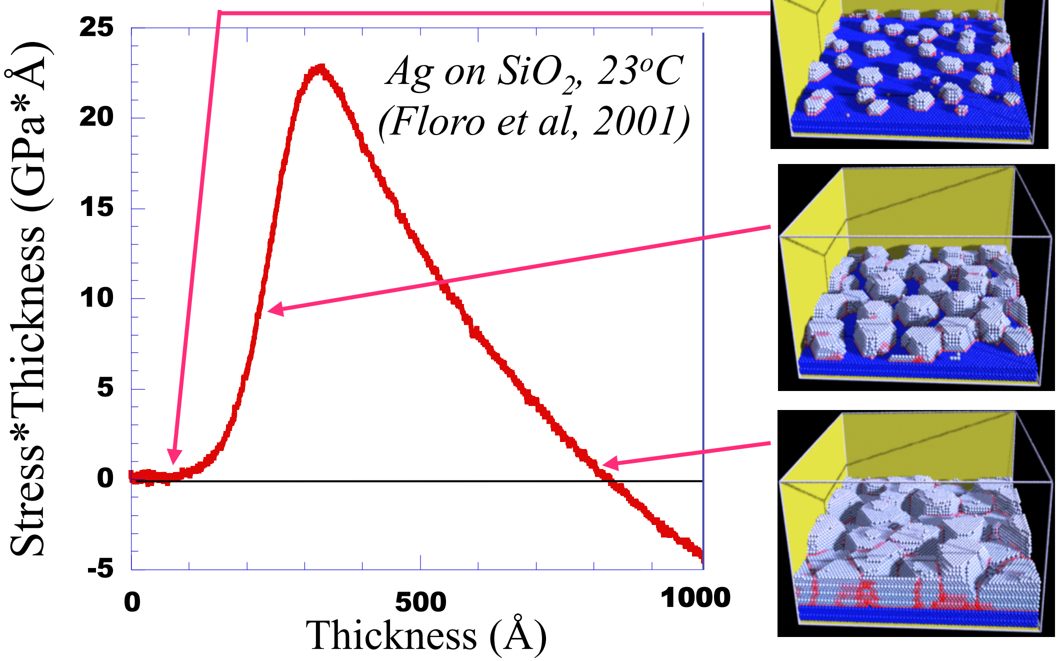
4. Strain in battery materials

- *large volume changes*
- *associated with phase changes*



Features of stress evolution in polycrystalline films

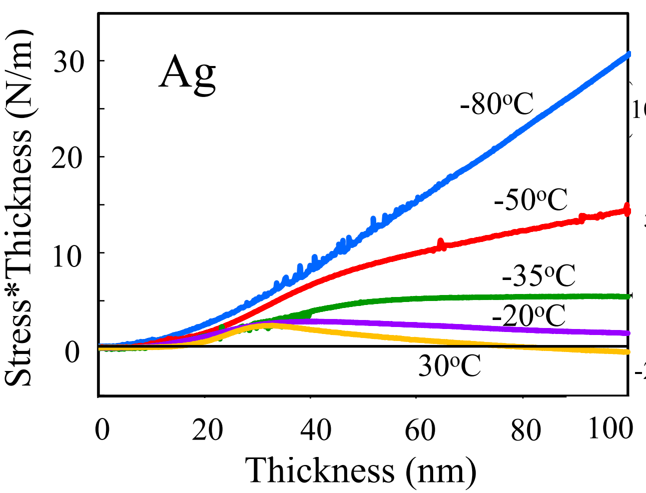
Stress changes with microstructure

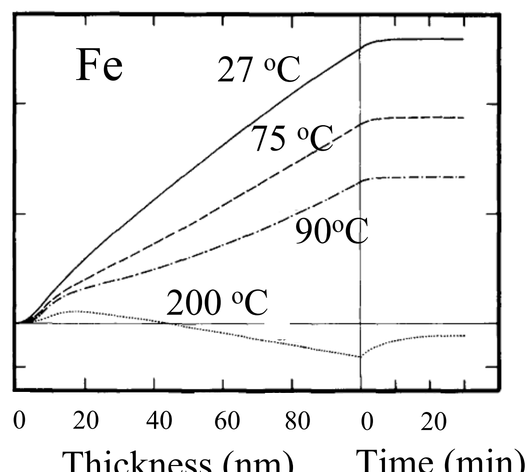


Stages of film/stress evolution:

- Nucleation
  - *Compressive or no stress*
- Coalescence
  - *Tensile rise*
- Continuous film
  - *Steady-state compressive (for high atomic mobility)*

Stress depends on kinetics (temperature, material, deposition rate)





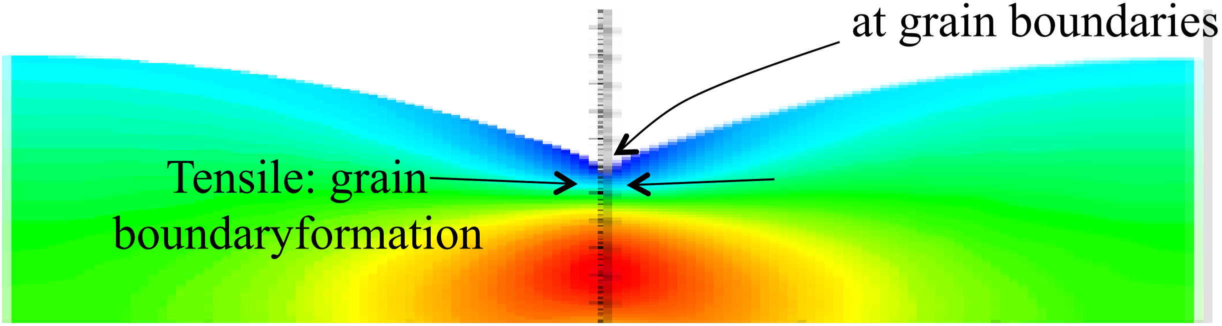
Ag on SiO_2 *(Chason, Hearne, JAP 2013)*

Fe on MgF_2 *(Thurner and Abermann, TSF, 1990)*

- Lower T, same growth rate:
  → more tensile
- At 30 °C:
  Fe: tensile, Ag: compressive

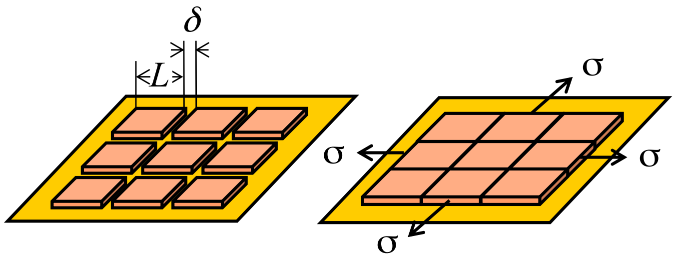
Simple model for stress evolution in polcrystalline films

Consider stress as balance between different generation/relaxation mechanisms occurring at triple junction (top of grain boundary)



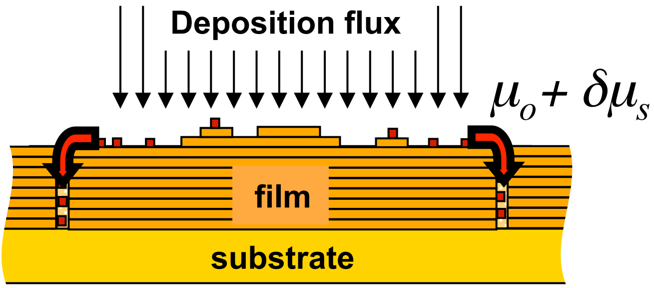
- Tensile → grain boundary formation

$$\sigma_T \propto \left(\frac{\gamma E}{L}\right)^{\frac{1}{2}}$$



- Compressive
  - → insert atoms into grain boundary (driven by surface supersaturation)

$$\sigma_C = \delta\mu_s/\Omega$$

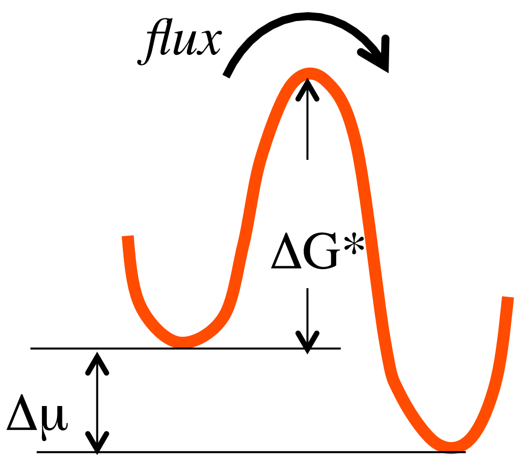


- Mediated by kinetic processes on surface:
  - Growth rate *R*, diffusivity *D*, grain size *L*

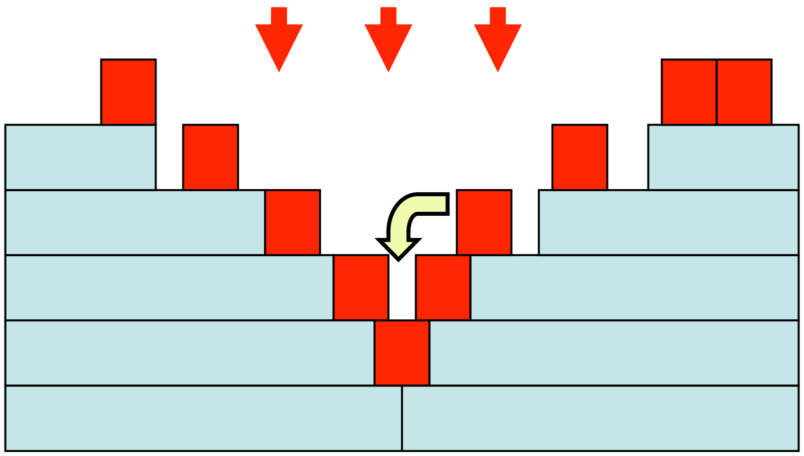
Write equations for evolution of stress

Δμ drives atoms into or out of gb

1) $\Delta\mu = \delta\mu_S + \sigma_{tj}\Omega$



Combine stress as grain boundary forms (tensile) with stress as atoms are inserted into it (compressive)



2) $$\frac{\partial N_{tj}}{\partial t} \cong 4C_s \frac{D}{a^2}\frac{\Delta\mu}{kT}$$

3) $$Induced\ stress: \sigma_{tj} = \sigma_T - M_f \frac{N_{tj}\cdot a}{L}$$

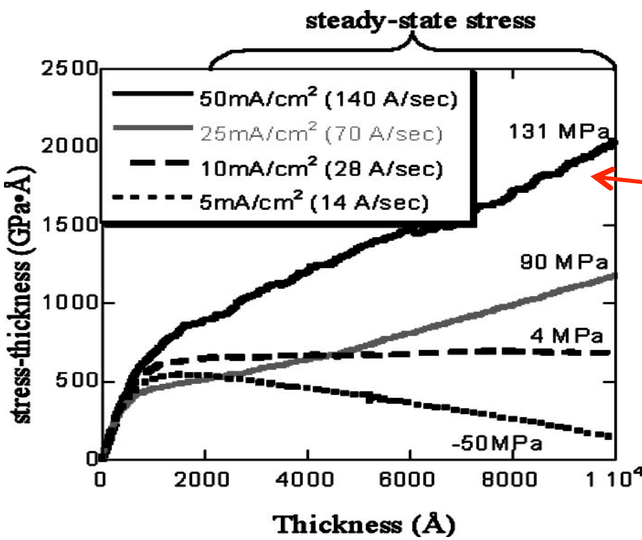
Master equation for stress evolution at triple junction:

$$\frac{\partial \sigma_{tj}}{\partial t} = -\frac{4C_s M_f}{akT}\frac{D}{L}\cdot(\sigma_{tj}\Omega + \delta\mu_S)$$

$$\sigma_{tj} = \sigma_C + (\sigma_T - \sigma_C)\cdot e^{-\Delta t_{tj}/\tau}\ where\ \Delta t_{tj} = a/\dot{h}_{gb}$$

Rate of growth of grain boundary

Steady state stress: dependence on growth rate

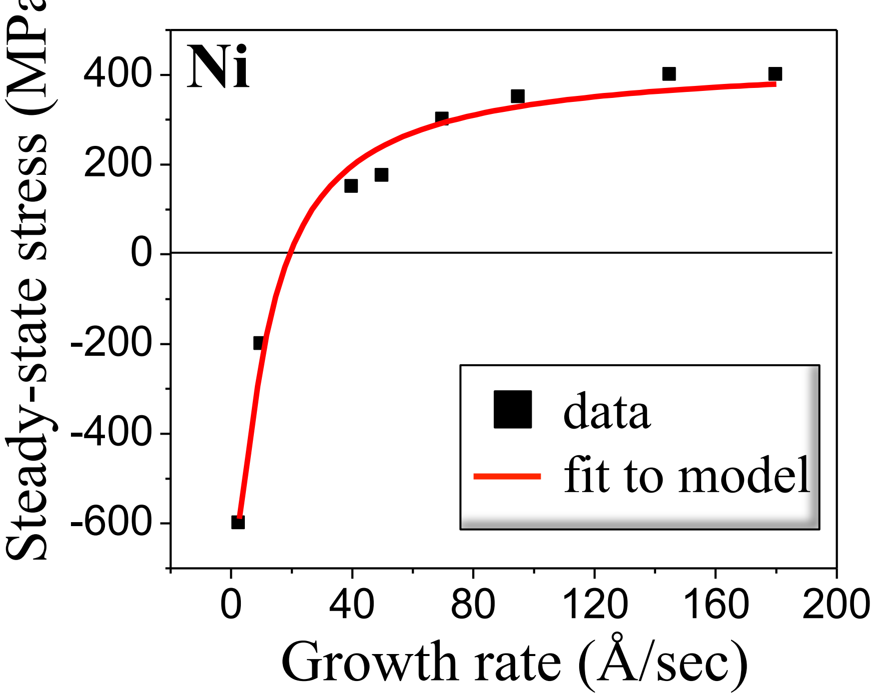


Electrodeposited Ni on Au, *Hearne et al, JAP* ***97*** *(2005)*

Stress reaches steady-state (constant slope)
- Different σ_{SS} for each growth rate

Model prediction:

$$\sigma_{SS} = \sigma_C + (\sigma_T - \sigma_C) \cdot e^{-\alpha D / RL}$$



Grain size remains ~1 μm

Key parameter: $\dfrac{D}{RL}$

$\dfrac{D}{RL} \ll 1$ — Low D, high R → tensile

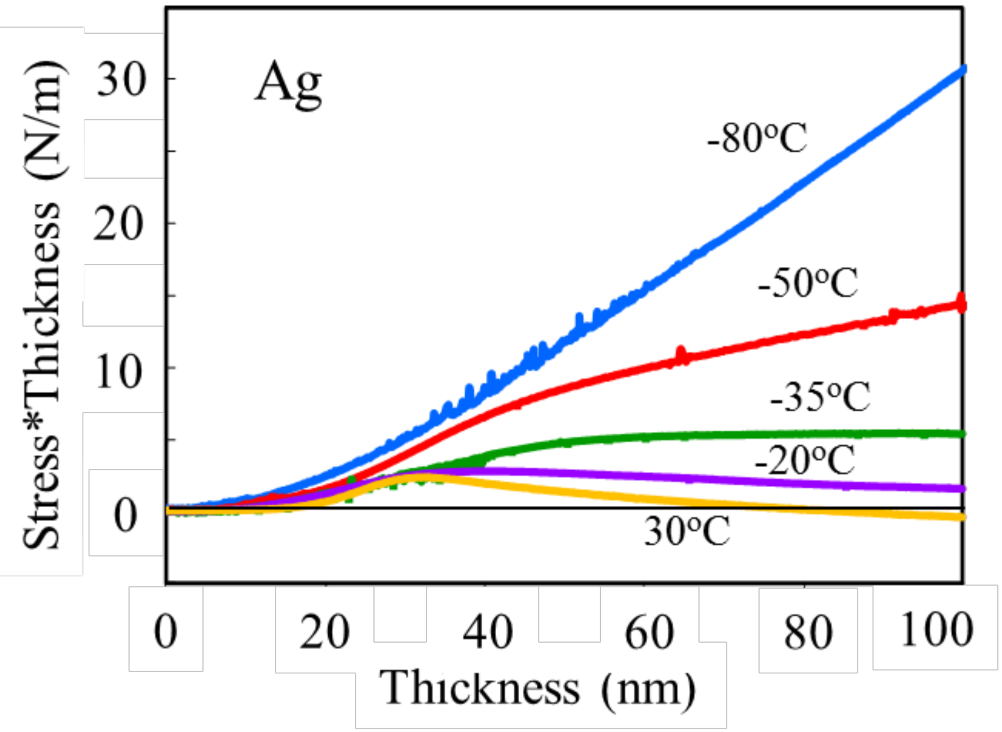
$\dfrac{D}{RL} \gg 1$ — High D, low R → compressive

→ Determines growth rate for stress-free films

Stress vs thickness: effect of coalescence of islands

Data: Stress changes with thickness
Depends on temperature

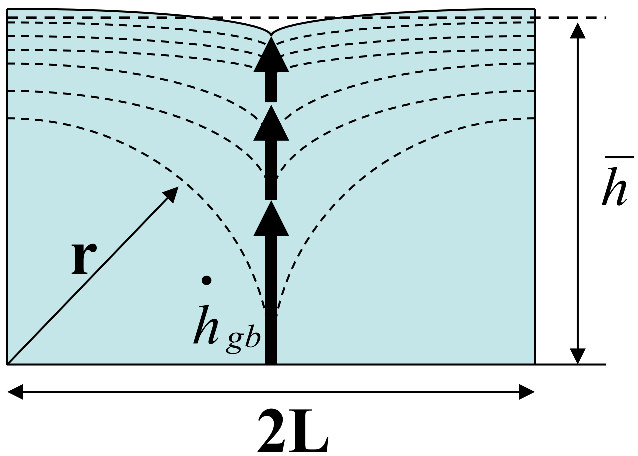
PVD Ag on SiO_2, (Hearne)



Model:

$$\sigma_i = \sigma_C + (\sigma_T - \sigma_C) \cdot \exp\left(-\frac{\alpha D}{L \frac{\partial h_{gb}}{\partial t}}\right)$$

- $\partial h_{gb}/\partial t$ changes during coalescence
- Model islands as cylindrical caps to calculate grain boundary velocity



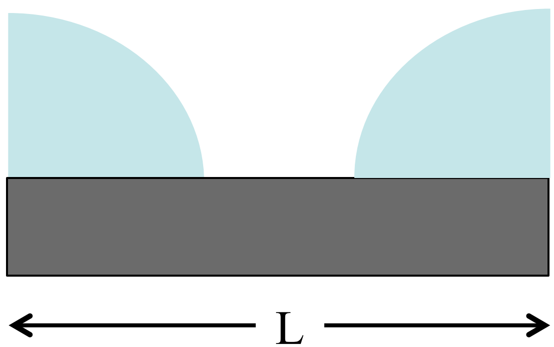
Grain boundary growth rate changes as film grows

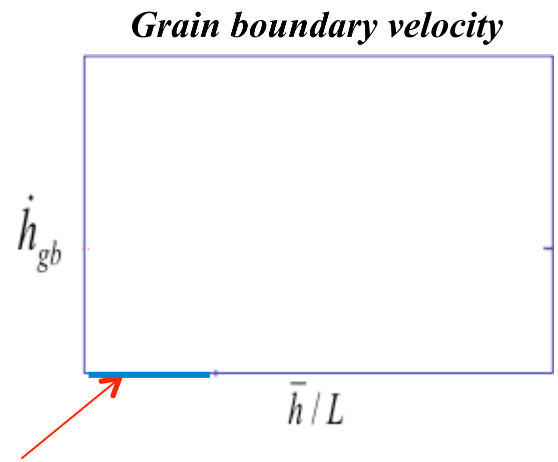
Stress changes with grain boundary velocity

Consider process of coalescence

- Calculate how $\partial h_{gb}/\partial t$ changes during coalescence
- Model islands as cylindrical caps
- Initial spacing is L

Grain boundary velocity changes as islands grow



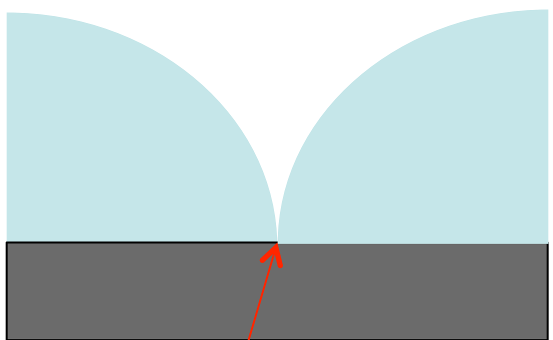


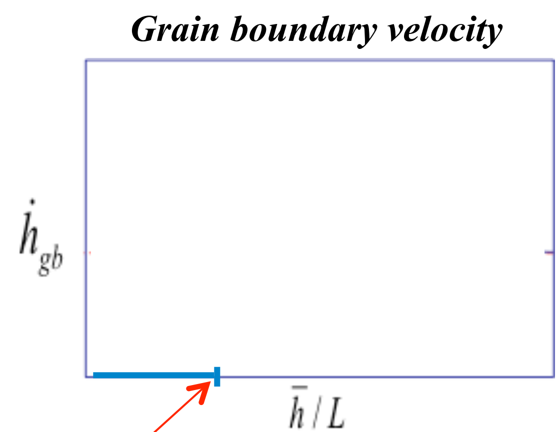
Before coalescence, $\partial h_{gb}/\partial t = 0$

(no grain boundary)

Consider process of coalescence

- Calculate how $\partial h_{gb}/\partial t$ changes during coalescence
- Model islands as cylindrical caps

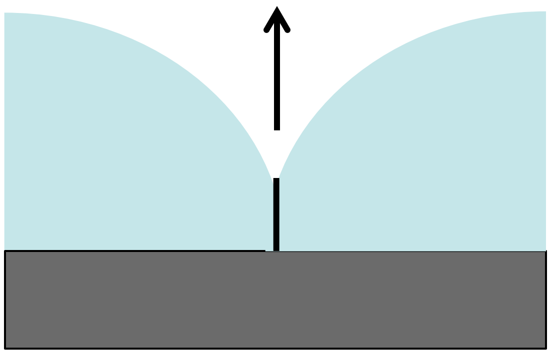


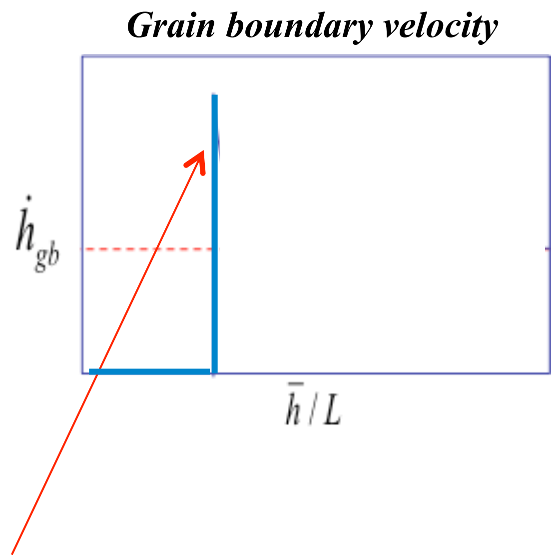


Grain boundary forms at onset of coalescence

Consider process of coalescence

- Calculate how $\partial h_{gb}/\partial t$ changes during coalescence
- Model islands as cylindrical caps

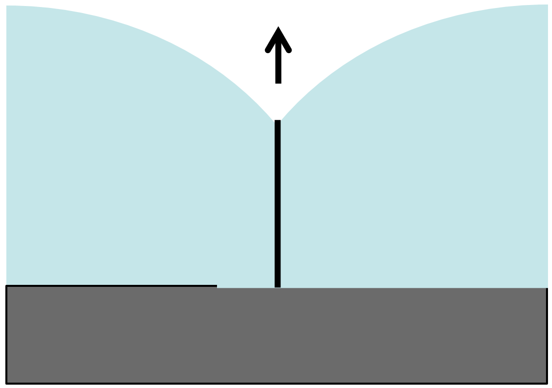


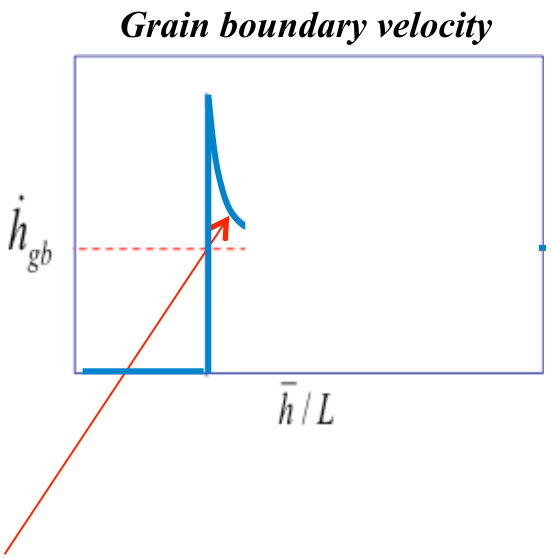


Grain boundary grows rapidly at first

Consider process of coalescence

- Calculate how $\partial h_{gb}/\partial t$ changes during coalescence
- Model islands as cylindrical caps

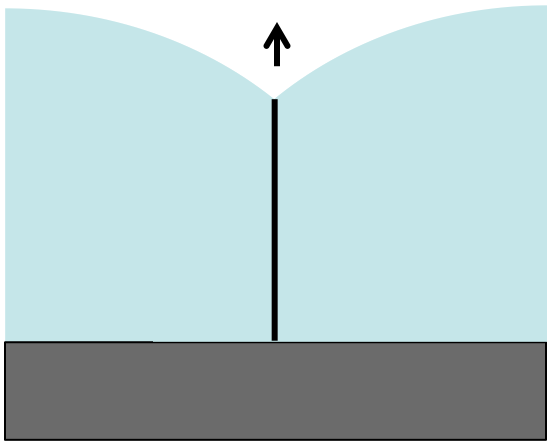


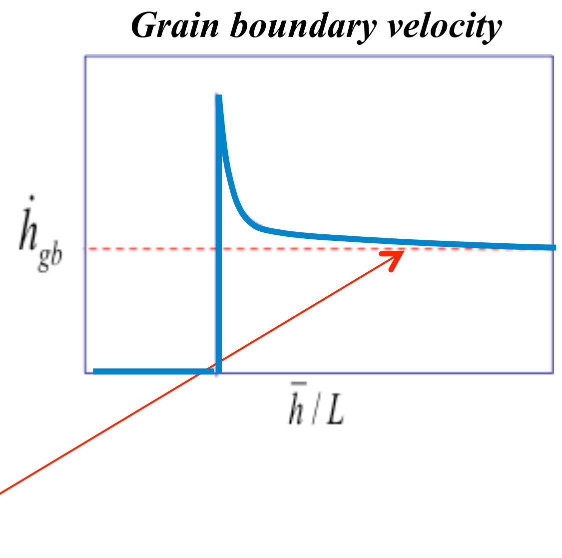


Slows down as film gets thicker

Consider process of coalescence

- Calculate how $\partial h_{gb}/\partial t$ changes during coalescence
- Model islands as cylindrical caps





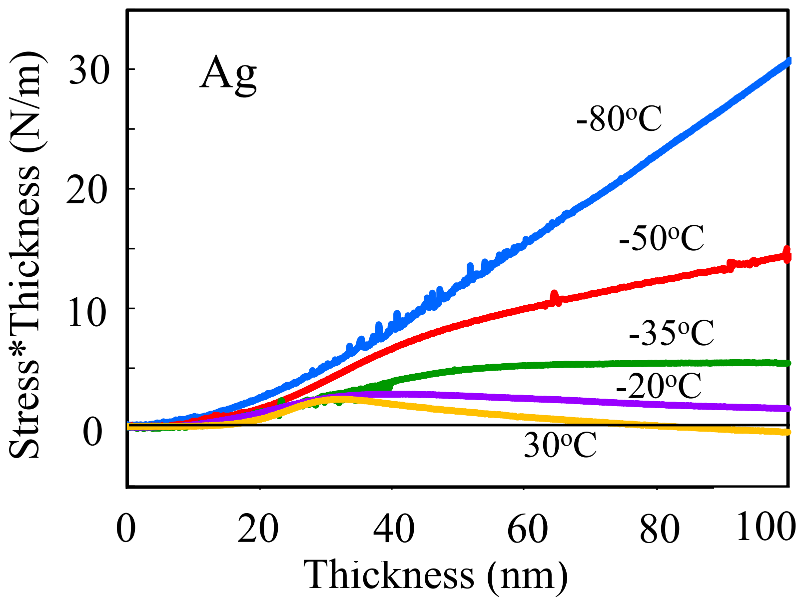
$\dot{h}_{gb}$ approaches average growth rate (*R*) as film gets thicker (steady-state)

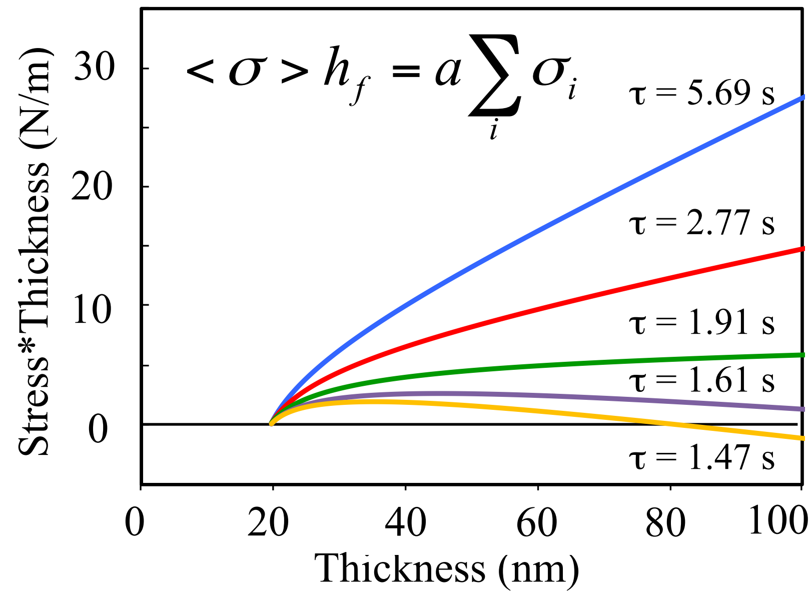
Model fits Ag on SiO_2 data

Change atomic mobility (D) at constant R, L

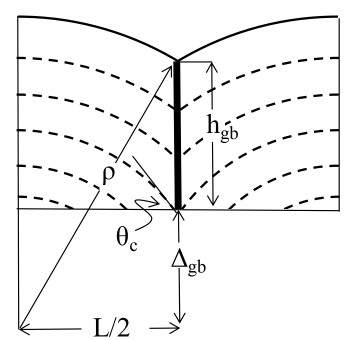
PVD Ag on SiO_2, (Hearne)

Model: $\sigma_i = \sigma_C + (\sigma_T - \sigma_C)\cdot \exp(-\frac{\alpha D}{L\,\dot{h}_{gb}})$





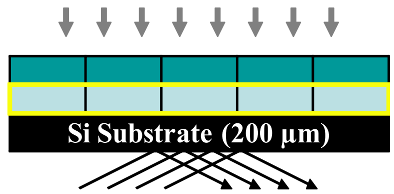
- Islands are cylindrical caps, contact angle ~68 deg,
- Fitting parameters: σ_c, σ_T , τ
  Use same σ_T (442 MPa) and σ_C (-359 MPa) for all temperatures
- τ different for each *T* (proportional to 1/*D*)



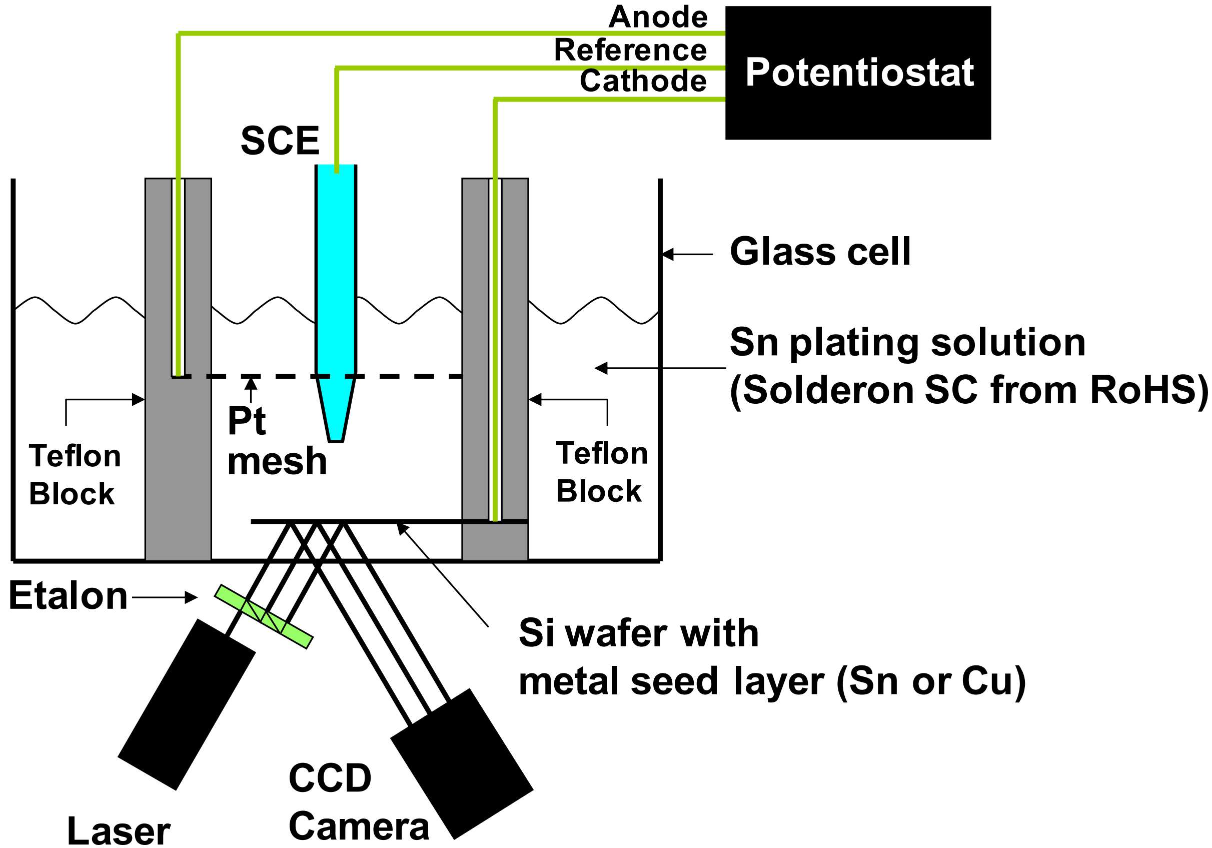
→ Grain boundary model captures change with thickness, temperature

Role of grain boundary in high mobility material (Sn)

Monitor stress during electrochemical deposition



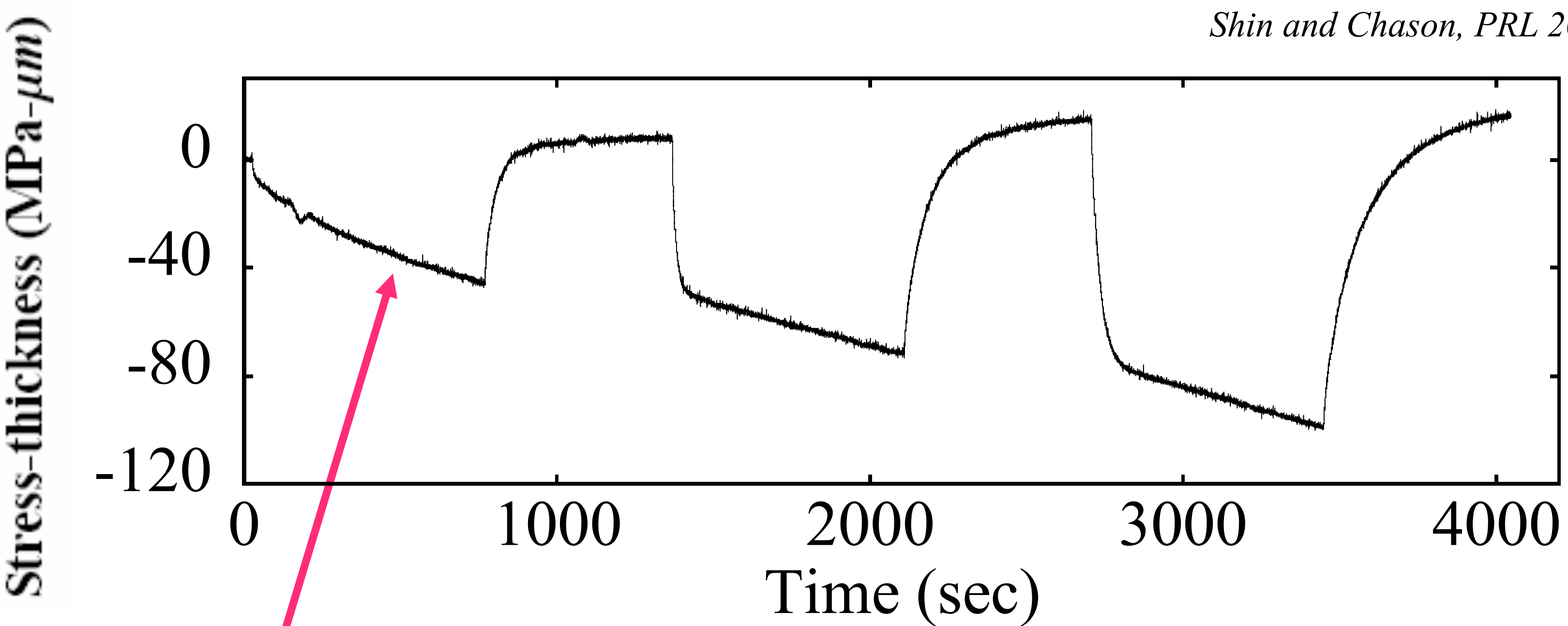
1) Evaporate seed layer of Sn (1 µm)
2) Electrodeposit Sn film at constant voltage



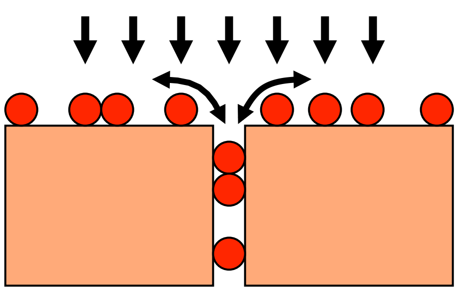
Look at effect of growth interrupts

Stress behavior during interrupt & regrowth

Shin and Chason, PRL 2009

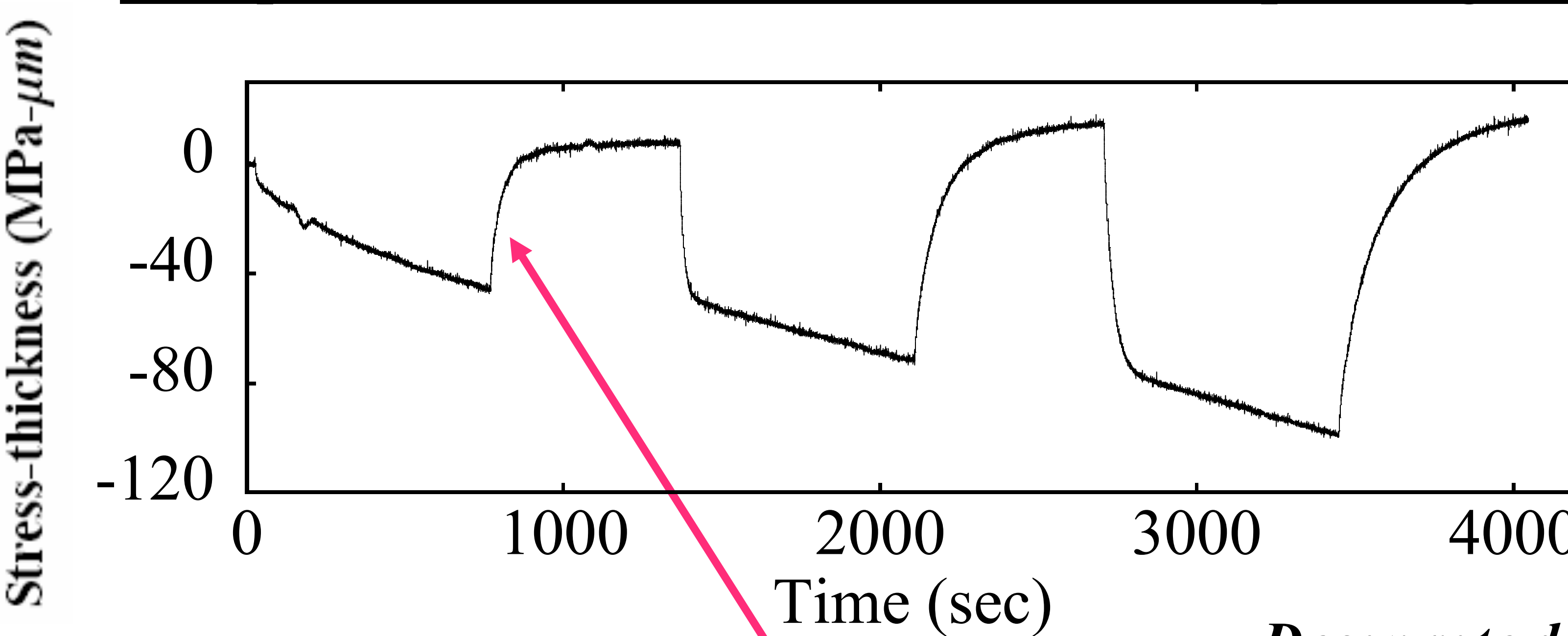


Steady state

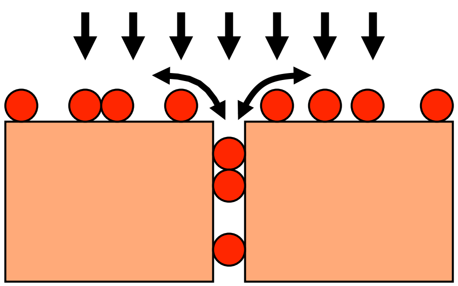


$$\sigma_{SS} = \frac{\sigma_T + (\alpha D / RL)\,\sigma_C}{1 + (\alpha D / RL)}$$

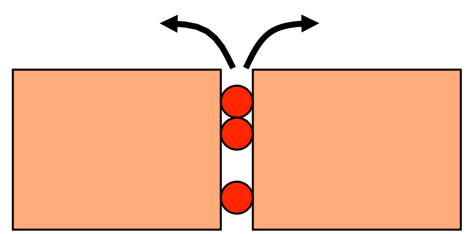
Interpretation of stress behavior at interrupt & regrowth



Steady state



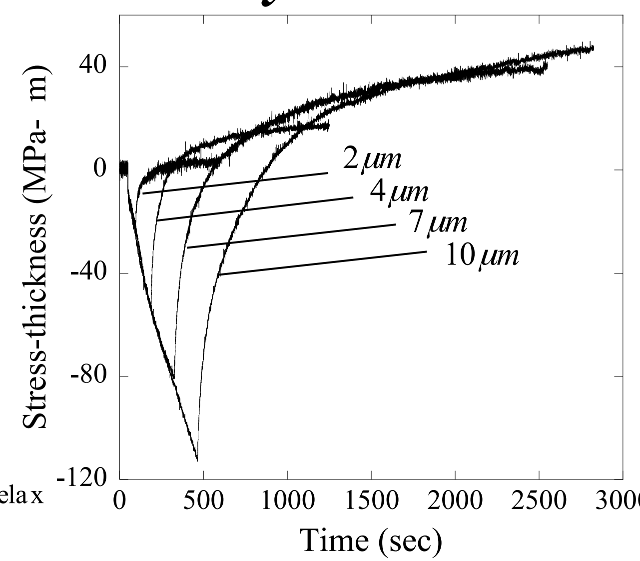
Interrupt



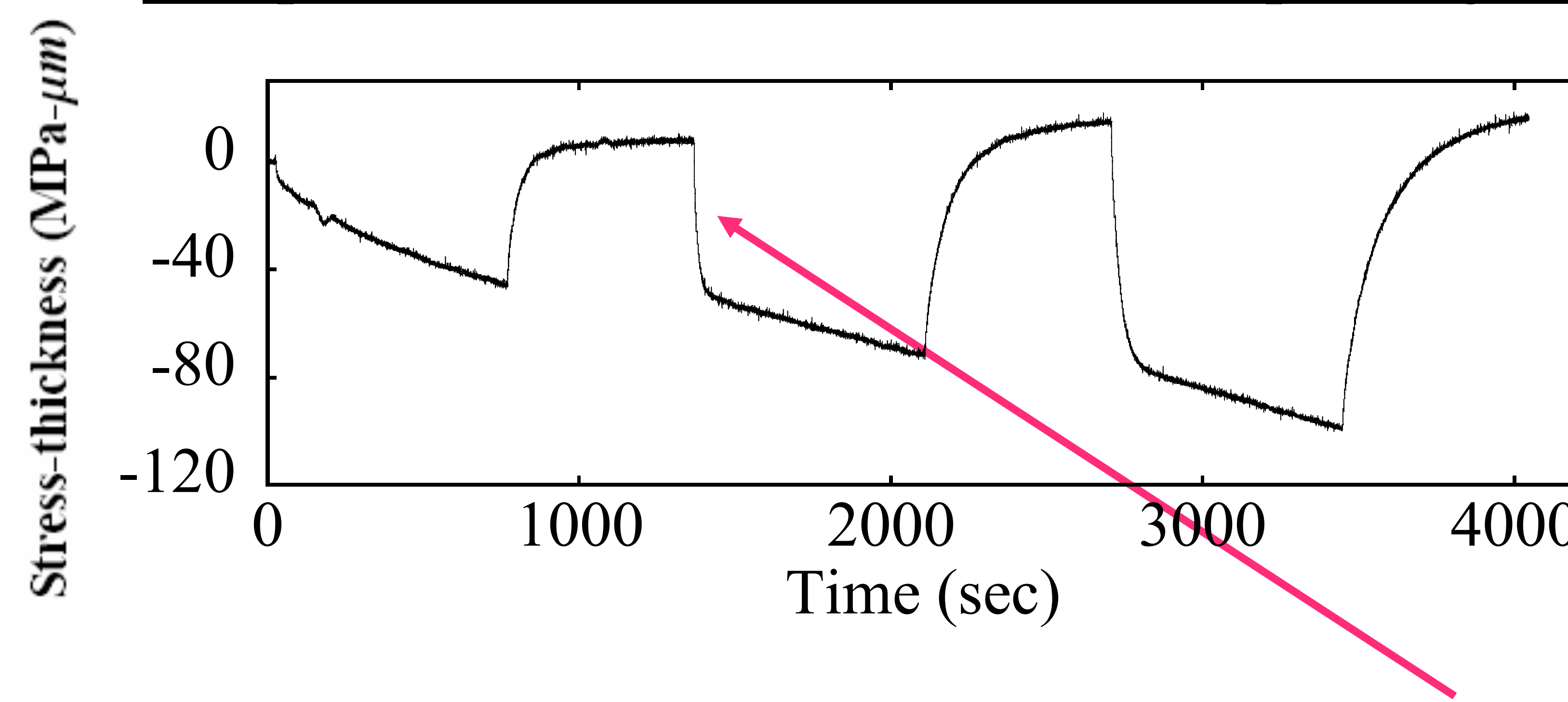
$$\delta\mu_S = 0 \quad R = 0$$

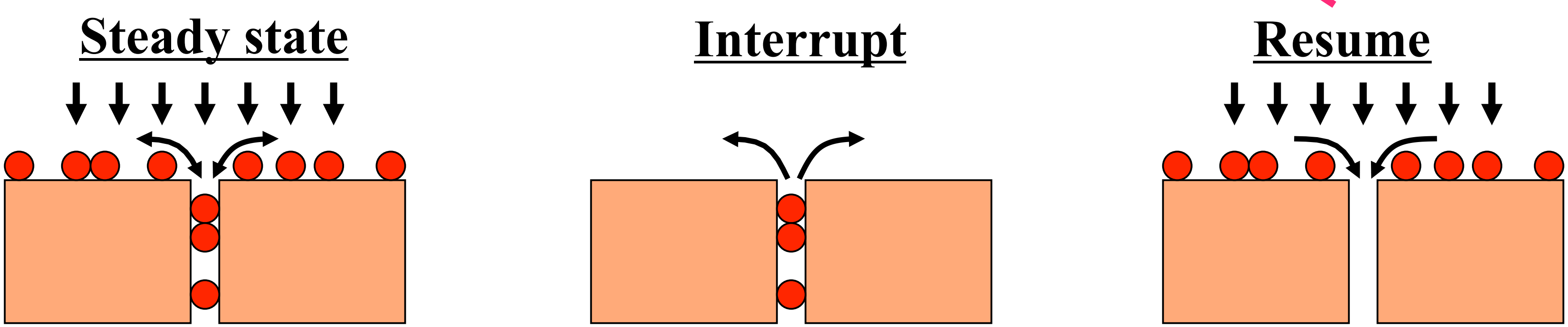
$$\sigma = \sigma_f + (\sigma_i - \sigma_f)e^{-t/\tau_{\text{relax}}}$$

Decay rate depends on layer thickness



Interpretation of stress behavior at interrupt & regrowth



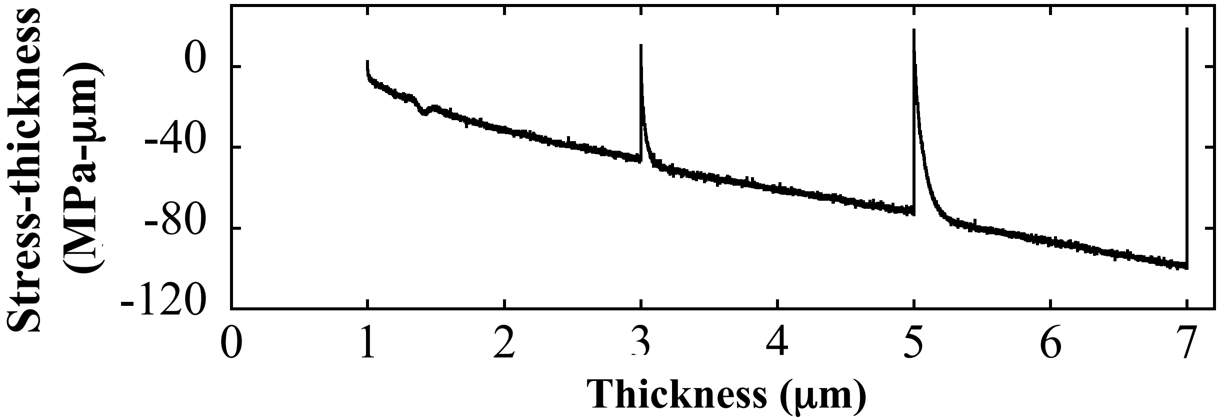


$$\sigma_{SS} = \frac{\sigma_T + (\alpha D / RL)\,\sigma_C}{1 + (\alpha D / RL)}$$

Stress measurements in electrodeposited Sn

Stress-thickness vs thickness

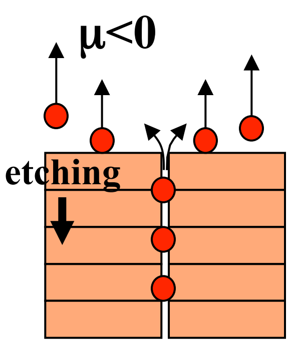
- same slope after interrupts

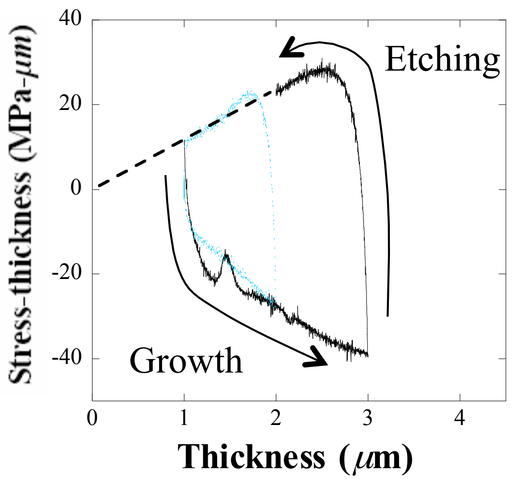


Stress is independent of layer thickness:

→ confirms role of grain boundary in stress evolution

Stress during etching





Equivalence between growth and etching:

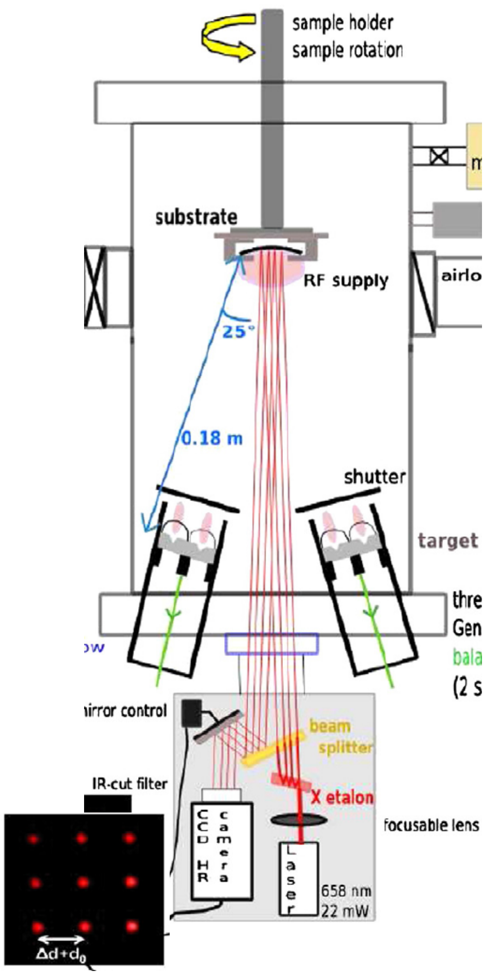
- negative chemical potential on surface induces tensile stress in film

→ confirms role of surface chemical potential in stress evolution

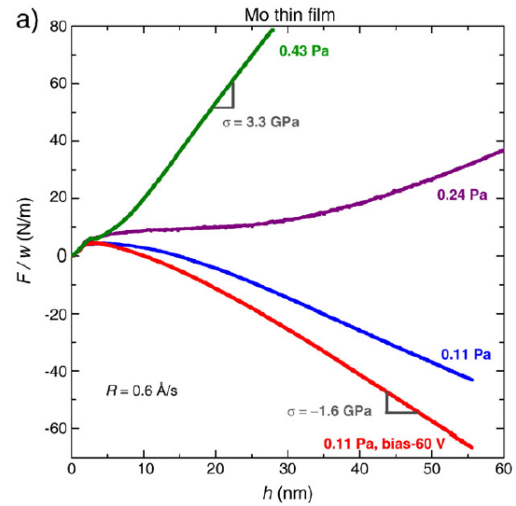
Stress evolution during sputter deposition

Additional parameters: ion energy, gas pressure

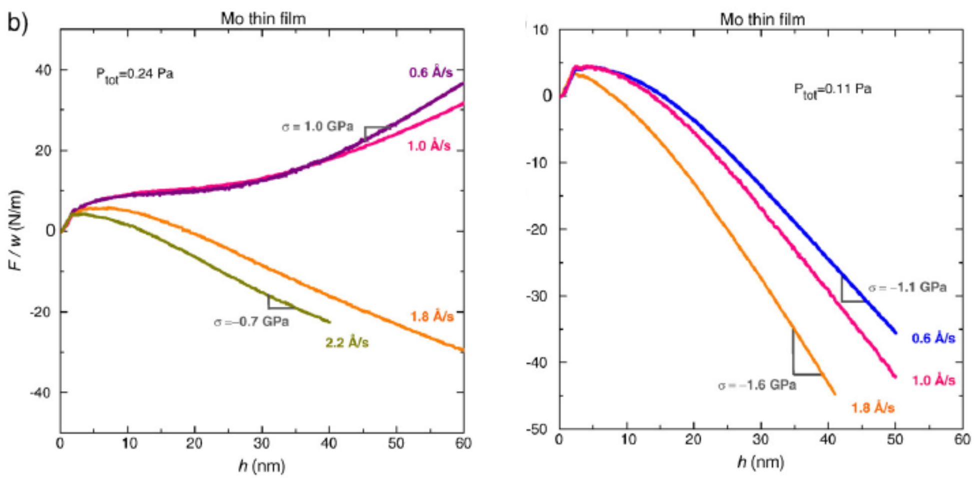
MOSS with magnetron sputtering sources
(Mo films, Fillon, Abadias, et al. TSF 2010)



Lower pressure → more energetic particles
Stress becomes more compressive



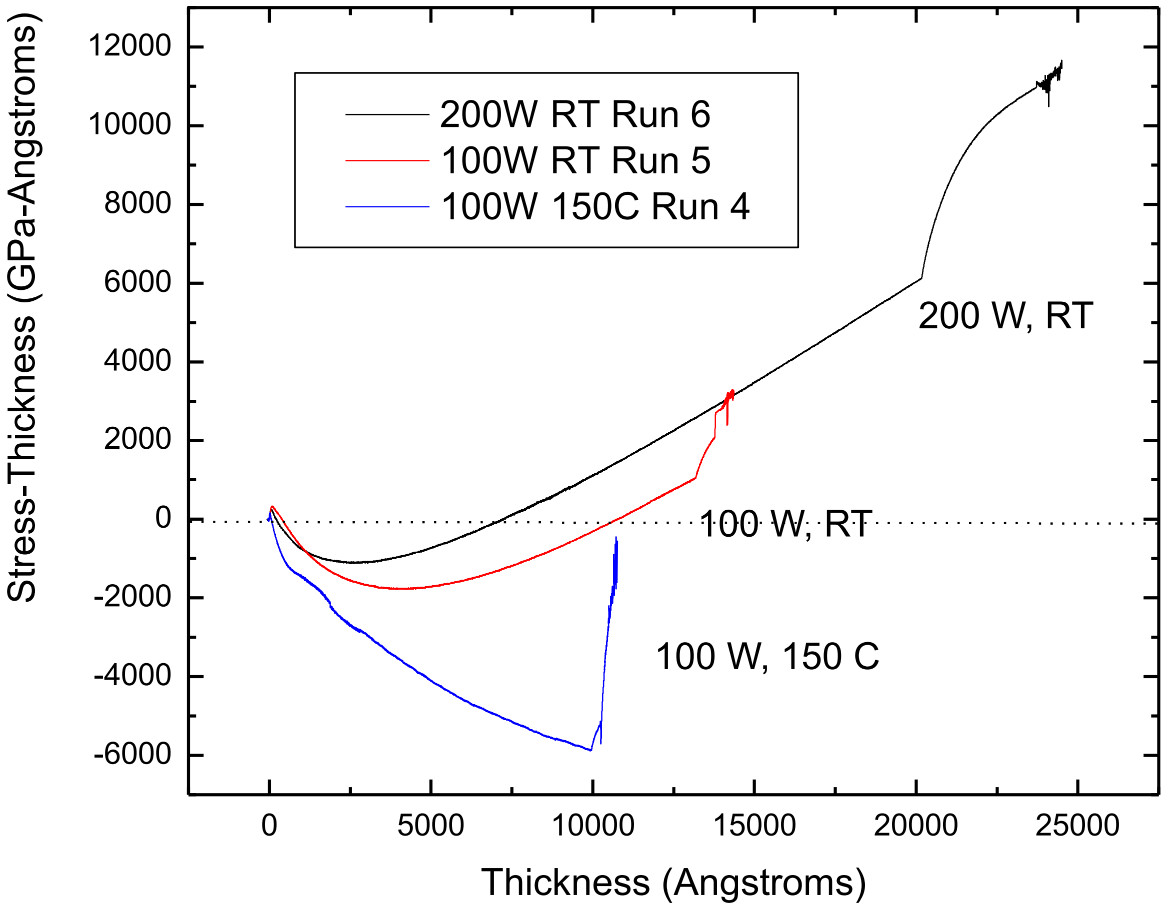
Dependence on growth rate different than evaporation
Don't know grain size or grain evolution

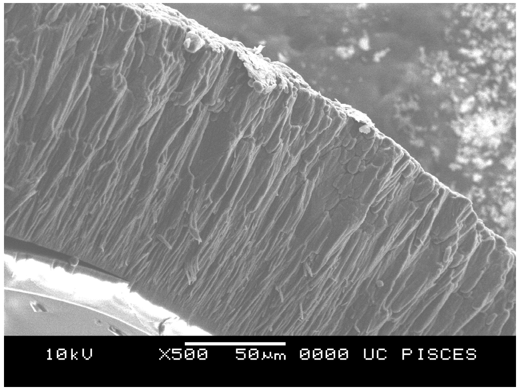


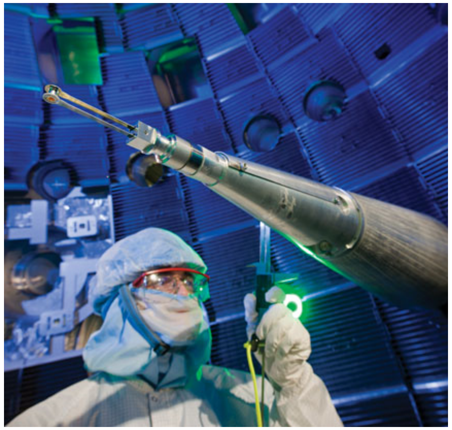
Stress evolution during sputter deposition (LLNL)

Be targets for NIF: need films with low stress (thick > 100 μm)

Higher growth rate (power) → more tensile
Higher T → more compressive

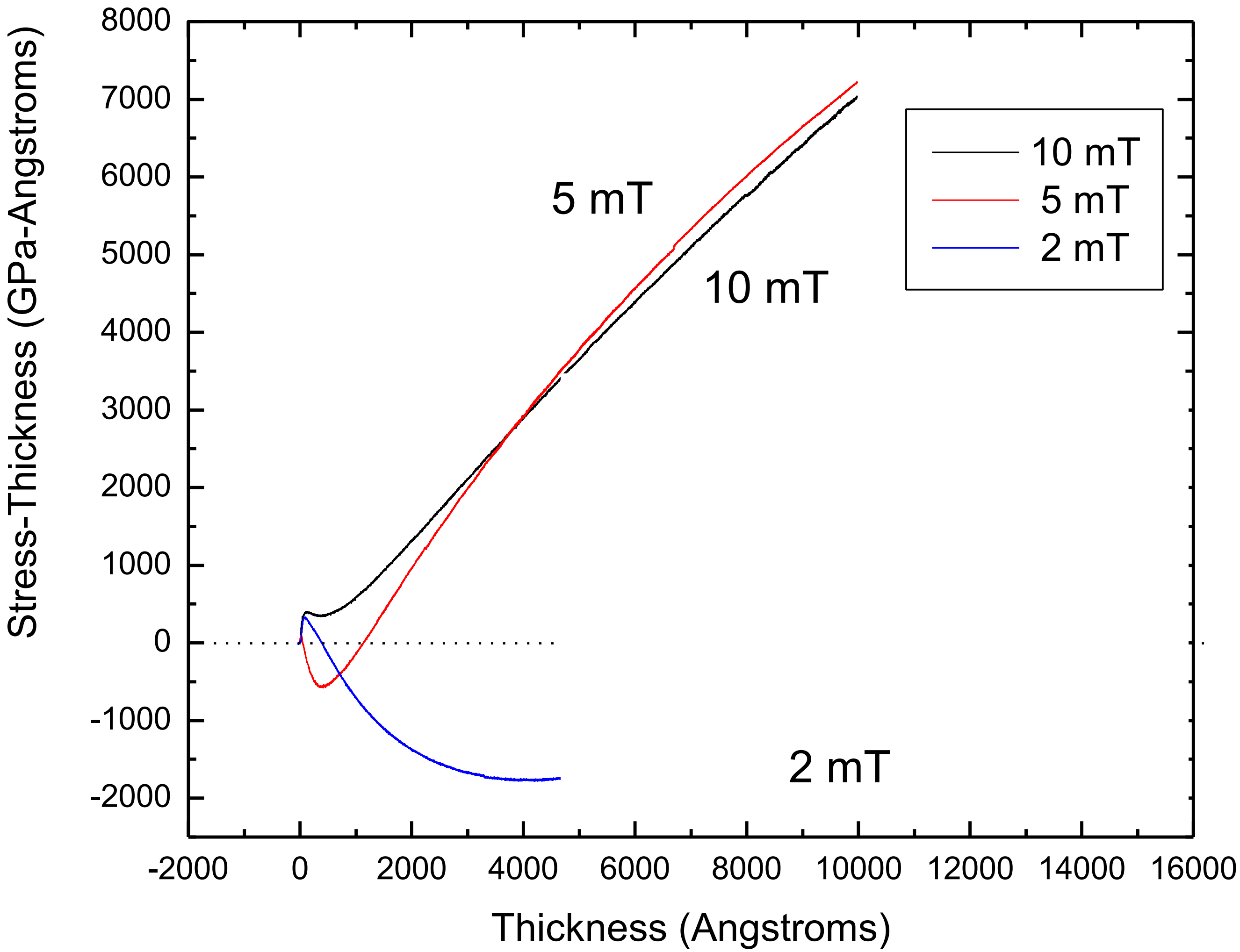






Be sputtering results (LLNL): effect of pressure

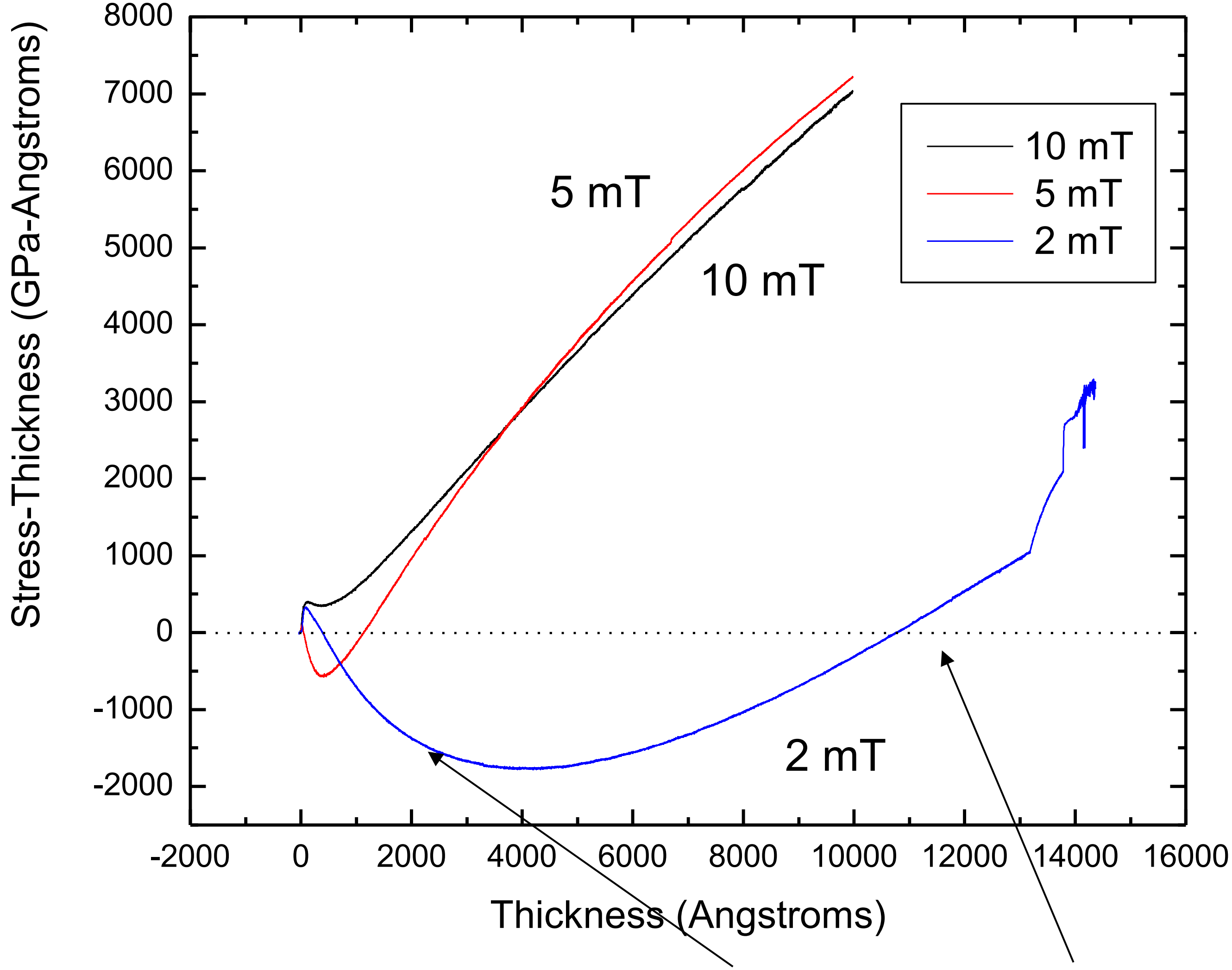
Lower pressure → more compressive initially



Why? Lower pressure means less scattering, more energetic incident particles: implant into surface, produce higher density

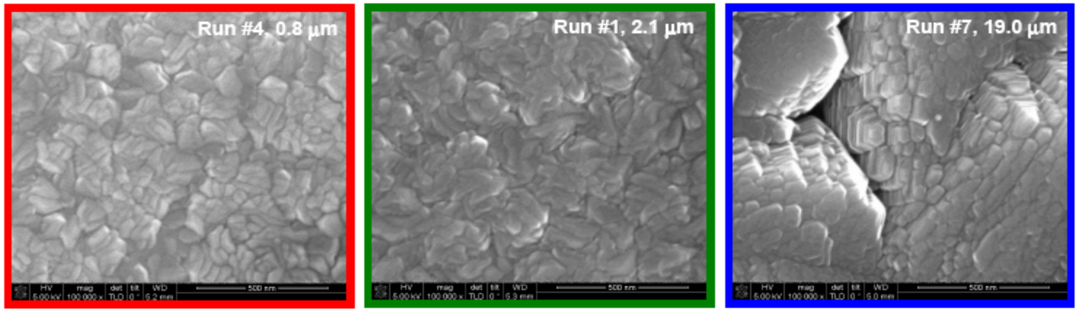
Be sputtering results

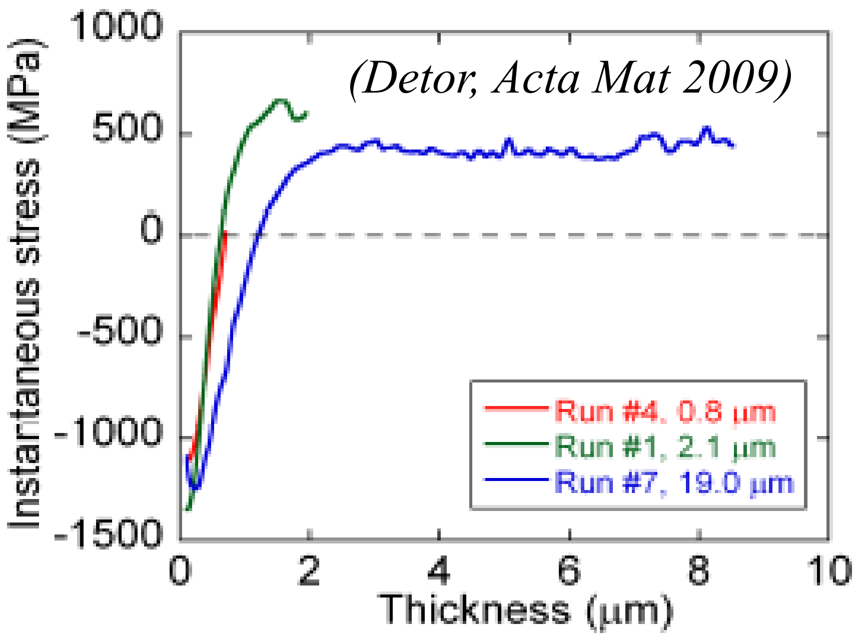
Lower pressure → more compressive initially



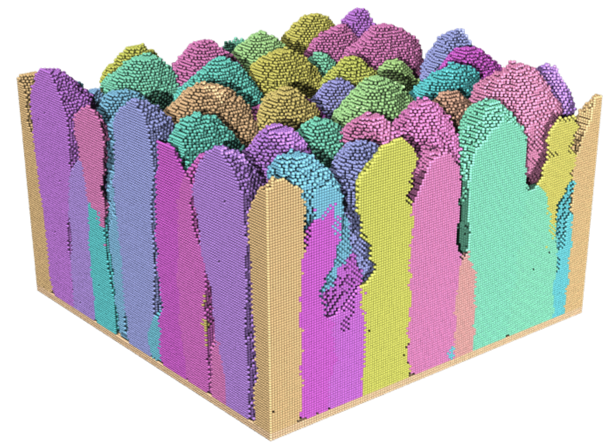
BUT: Incremental stress changes from compressive to tensile as layer gets thicker → kept same temperature, growth rate

Reason: Stress change correlated with rougher surface morphology

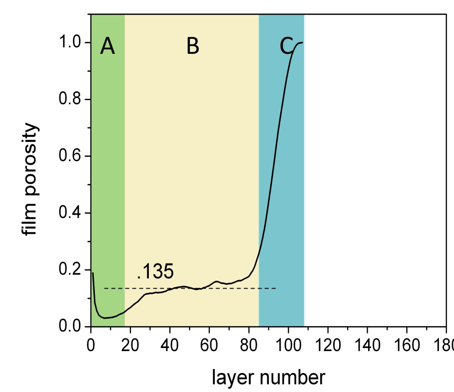




Film structure: roughening instability
(Zepeda-Ruiz, APL 2010)



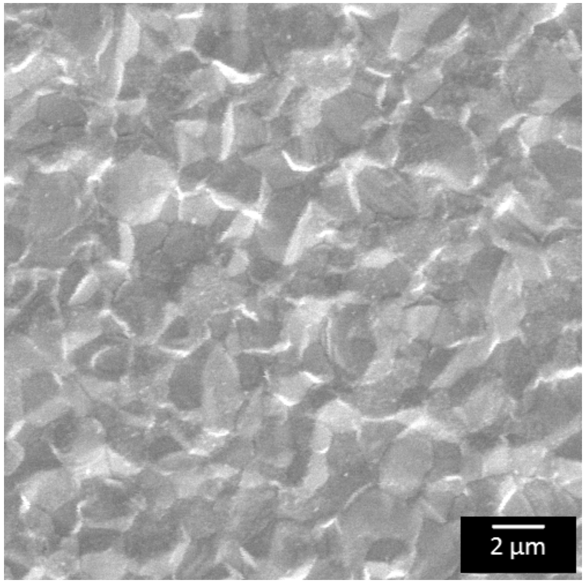
Porosity vs depth



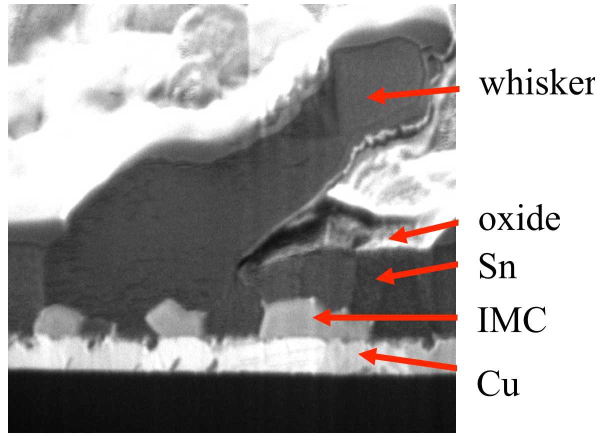
Greater roughness → Turns off compressive stress generation
Film becomes tensile

Sn whisker growth: driven by stress from IMC (intermetallic) formation

Whiskers form in Pb-free Sn coatings on Cu – cause systems failure (satellites, pacemakers)

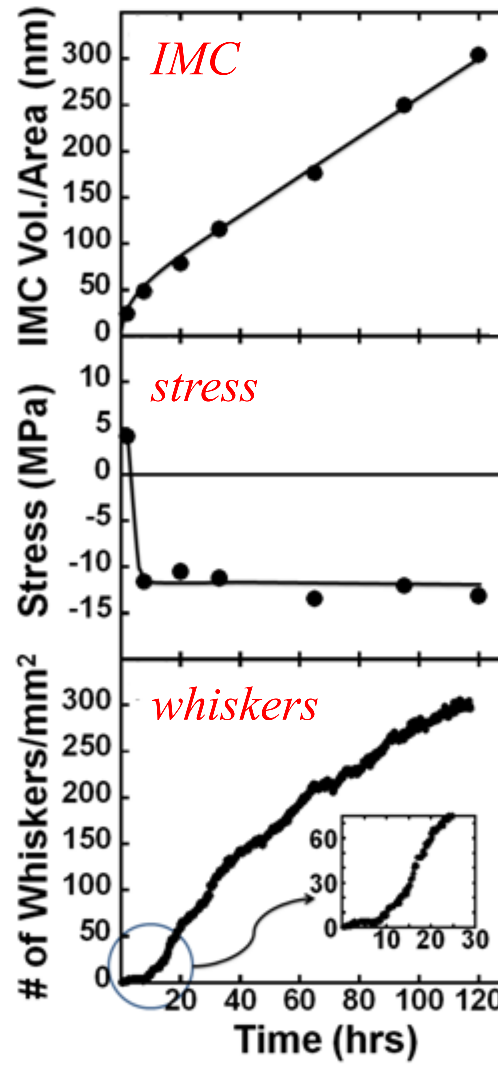


IMC forms at Cu-Sn interface



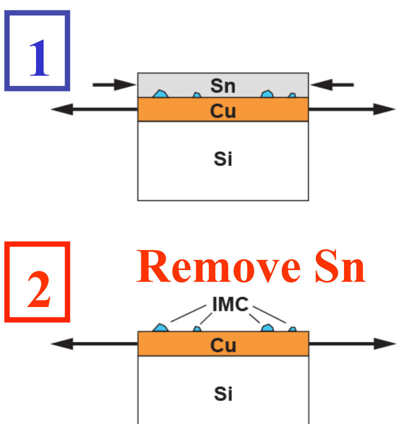
Correlate IMC/stress/whiskering

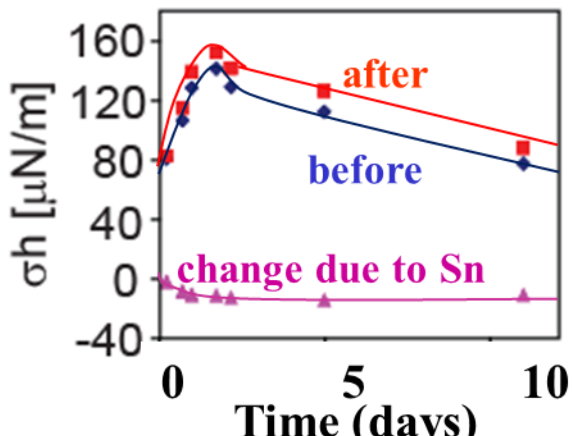
(Chason & Jadhav, APL 2009)



Measure stress evolution with MOSS

Wafer curvature measures *total* force exerted by film.





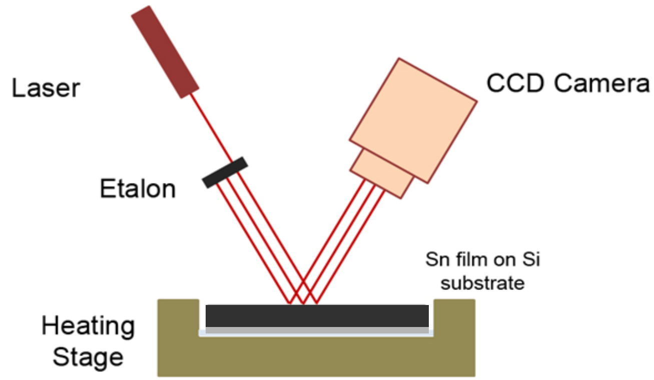
Remove Sn layer – change in curvature gives stress in Sn

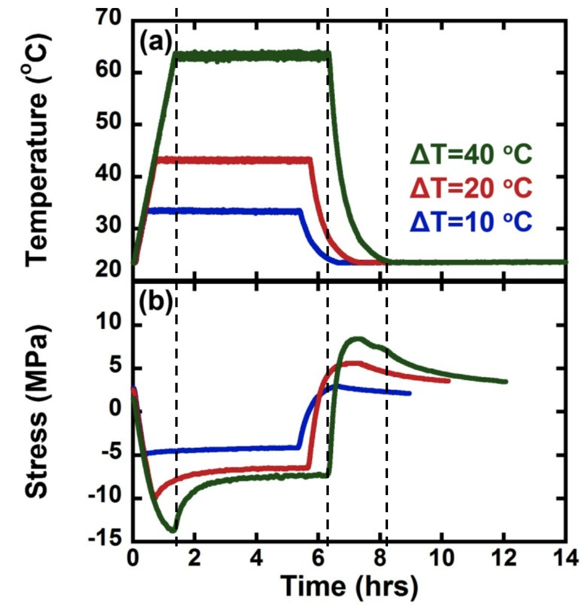
Reduce whiskering by enhancing stress relaxation

- Measure mechanical properties of layers:
  Sn and Sn alloys

- Find coatings that have low stress even after IMC grows

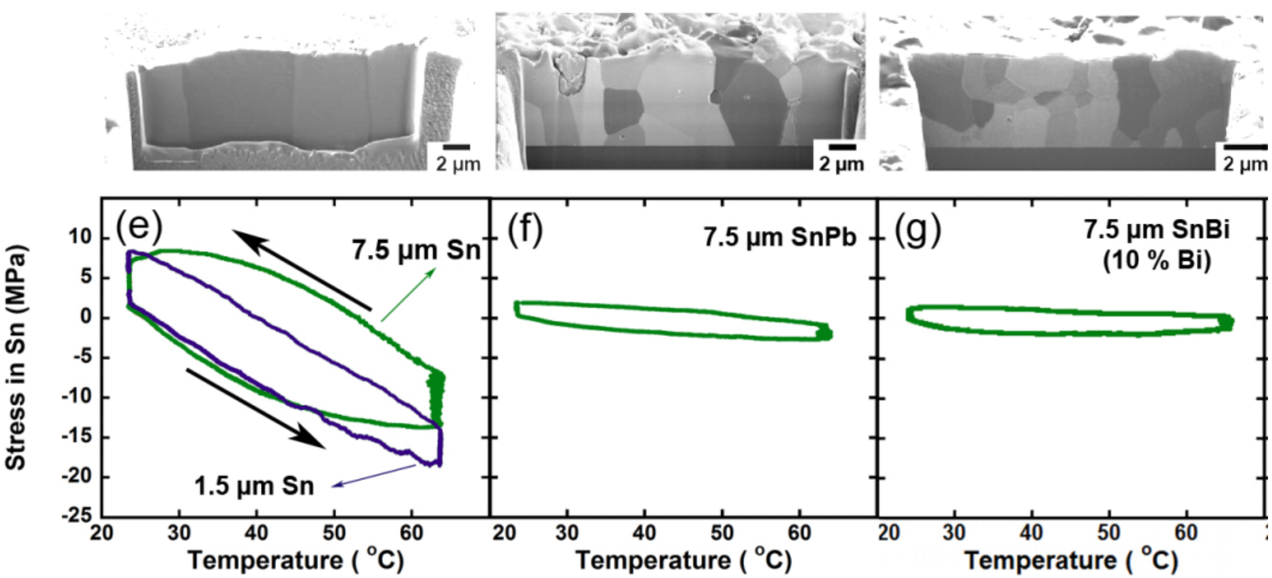
Use thermal expansion mismatch to create strain





Measure stress vs. strain for different films

Pure Sn | **Sn- 10%Pb** | **Sn-10% Bi**

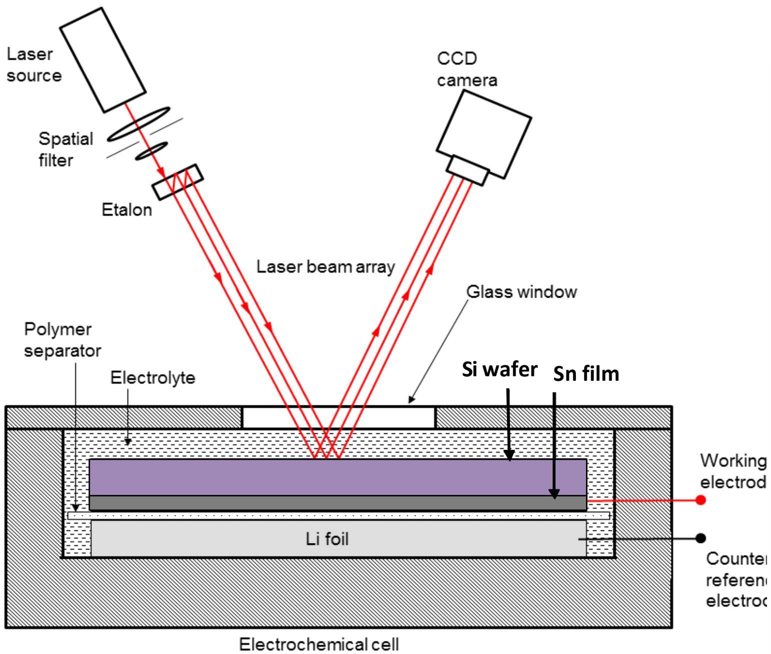


- These results agree with conclusions from whisker studies

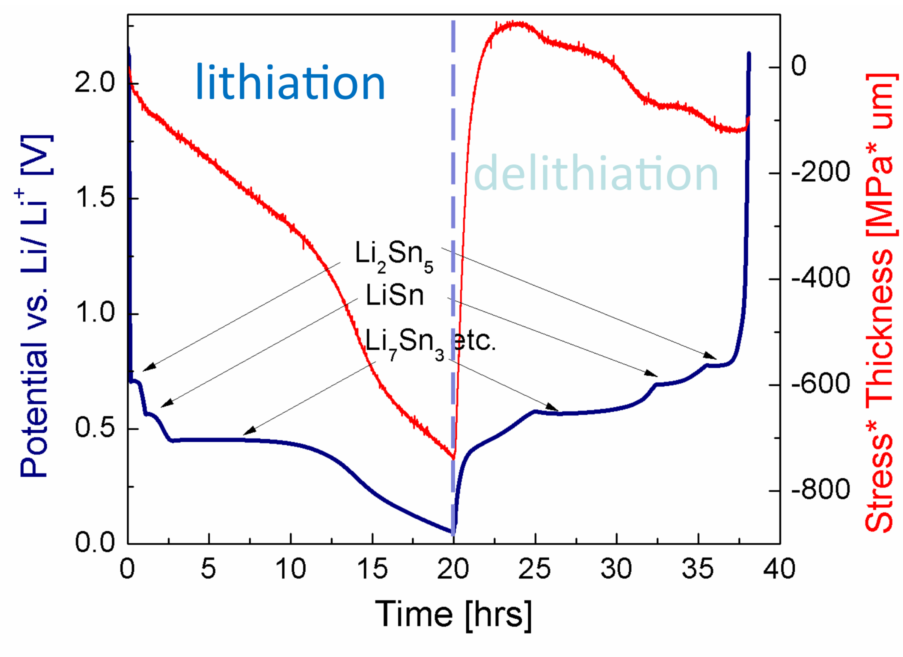
- More relaxation with
  - **larger grain size**
  - **thickness**
  - **horizontal grain boundaries**

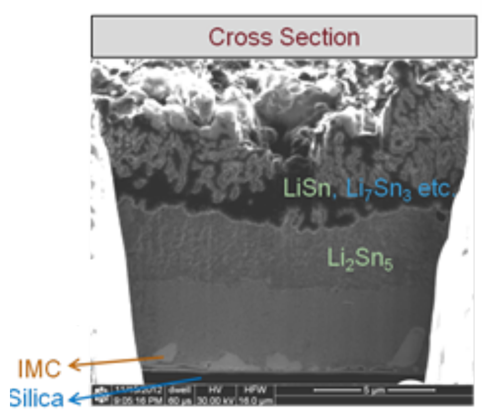
Stress evolution during charging/discharging of batteries (lithiation of Sn anode)

Put MOSS on electrochemical cell



Simultaneously measure C-V and stress-thickness





Measure stress associated with phase changes

Need to know layer thicknesses to interpret MOSS data

(Chen, Guduru 2013)

Summary

- Multi-beam wafer curvature (MOS) enables stress evolution to be monitored in real-time
    - useable on wide variety of platforms
    - sensitive, robust, easy to interpret

- Stress dynamics provide more information than single stress measurement
    - Key for
        - modeling
        - understanding sources of stress
        - controlling stress (optimizing processing conditions)
- Frontiers
    - Understanding multi-component materials
    - Energetic particle effects

Take home point:

In situ monitoring useful for understanding stress evolution

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