The Origin and Control of Residual Stress in Polycrystalline Films for Applications in Microsystems

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Outline:
• Overview of Residual Stress in Polycrystalline Films
  - Extrinsic Stress
  - Intrinsic Stress
• Stress Evolution During Evaporative Deposition of Polycrystalline Films

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Polycrystalline Si MEMS Accelerometer

(NovaSensor) (Senturia, Microsystems Design, KAP)
Buckling

\[ \sigma_0 \approx -\frac{\pi^2}{3} \frac{EH^2}{L^2} \]

\( E \sim 100\text{GPa} \)
\( H \sim 1\mu\text{m} \)
\( L \sim 300\mu\text{m} \)

\[ \Rightarrow \quad \sigma_0 \sim 4\text{MPa} \]

(Nunan et al, Vacuum Technology and Coating, Jan. 2001, p.27: Analog Devices)
Compressive stress: buckling

Tensile stress: low compliance

Stress gradients: beam curling

Residual Stresses in Thin Films

**Residual**: stress at the time of application (after processing)

**Extrinsic**: generated by external forces, e.g. differential thermal expansion during heating and cooling

**Intrinsic**: generated during film formation, e.g.
- coalescence (all)
- shot peening (sputter deposition)
- trapped gas (sputter deposition, CVD)
- other
Extrinsic Stress: Thermal Stress

\[
\sigma_f = \int_{T_0}^{T} \tilde{E}_f (\alpha_f - \alpha_s) \, dT
\]

\[
\sigma_f \approx \tilde{E}_f \left( \alpha_f - \alpha_s \right) \Delta T
\]

\(\sigma_f\) = biaxial stress in the film  
\(\tilde{E}_f\) = biaxial modulus (depends on texture) of the film  
\(\alpha_f\) = thermal expansion coefficient of the film  
\(\alpha_s\) = thermal expansion coefficient of the substrate  
\(\Delta T\) = difference between actual temperature and the zero stress temperature
<table>
<thead>
<tr>
<th>Material</th>
<th>Thermal Expansion Coefficient (dL/dT) per K near room temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>23.6 x 10^{-6}</td>
</tr>
<tr>
<td>Cu</td>
<td>16 x 10^{-6}</td>
</tr>
<tr>
<td>W</td>
<td>4.3 x 10^{-6}</td>
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<tr>
<td>Al_{2}O_{3}</td>
<td>5 x 10^{-6}</td>
</tr>
<tr>
<td>Borosilicate Glass</td>
<td>1.5 x 10^{-6}</td>
</tr>
<tr>
<td>Fused Silica</td>
<td>0.3 x 10^{-6}</td>
</tr>
<tr>
<td>Si</td>
<td>2.6 x 10^{-6}</td>
</tr>
<tr>
<td>GaAs</td>
<td>6.9 x 10^{-6}</td>
</tr>
<tr>
<td>Ge</td>
<td>0.5 x 10^{-6}</td>
</tr>
</tbody>
</table>
Experimental Characterization of Stress Evolution During Post-Deposition Thermal Treatments

\[ \sigma_f = \frac{1}{6} \frac{h_s^2 E_s}{h_f} \frac{1}{R} \]

Stoney’s equation

\[ \sigma_f \approx \bar{E}_f (\alpha_f - \alpha_s) \Delta T \]

Thermal Stress of Tungsten on Si

\[ \Delta \sigma = 70\text{-}300 = -230\text{MPa}, \Delta T = 450\text{-}30 = 420^\circ C \]
Ag on oxidized silicon.

Stress-Temperature Curves

- First cycle is different
- Following cycles are similar

Generic $\sigma$ vs. $T$ curves for fcc metals with clean, unpassivated surfaces

- Elastic
- Elasto-plastic
- Plastic

Yielding and grain growth
Capped Films: Stress-Temperature Curves

Expected result for dislocation-mediated plasticity:
\[ \sigma_{\text{flow}}(h) \uparrow, \ h \downarrow \]

M.J. Kobrinsky and C.V. Thompson
Appl. Phys. Lett. 73, 2429, 1998
Dependence of the Yield Stress on Crystallographic Texture Grain Size and Film Thickness

\[ \sigma_y \approx \left( \frac{W_d \sin \phi}{b \cos \lambda \cos \phi} \right) \left( \frac{2}{d \sin \phi} + 1 \right) \]

(C.V. Thompson, J. Mater. Res. V.8, p.237, 1993.)
Uncapped Films: Stress-Temperature Curves

![Graph showing stress vs. temperature for different film thicknesses.]

Unexpected result:
\[ \sigma_{\text{flow}}(h) \uparrow \downarrow, \ h \downarrow \]
\[ \Rightarrow \text{diffusive creep} \]

M.J. Kobrinsky and C.V. Thompson
Diffusional Creep

**GB diffusion limited:**

**Surface diffusion limited:**

\[ \dot{\varepsilon} \propto \frac{\delta_{gb}D_{gb}}{dh^2} (\sigma - \sigma_{th}) \]

\[ \dot{\varepsilon} \propto \frac{\delta_{s}D_{surf}}{d^2h} (\sigma - \sigma_{th}) \]

Sliding affects proportionality constants.

\( d \sim h \)
Inelastic Regimes in Uncapped Ag Films

The graph shows the stress (MPa) as a function of temperature (°C) for different film thicknesses (h = 240 nm, h = 375 nm, h = 475 nm). The data indicates two distinct regimes:

- **Dislocation-mediated plasticity**:
  - The graph demonstrates how stress decreases with increasing temperature, indicating plastic deformation caused by dislocations.

- **Diffusional creep**:
  - At higher temperatures, the stress levels off, indicating creep behavior due to diffusional processes.

The graph highlights the transition between these two regimes, with different symbols and colors representing the film thicknesses.
Effect of TaN Capping Layer on Cu Lines

- Capping Layer Suppresses Diffusive Creep
- Capped Lines Elastically Accommodate Stress Over a Very Broad Stress/Temperature Range

Residual Stresses in Thin Films

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Stress In Sputter Deposited Films

Stress in Polycrystalline Silicon Films Deposited via CVD

Stress vs. Thickness for Films Deposited via Evaporative Deposition

Type 1: e.g., Ti, W, Ta, TiN, Cr at room temperature

Type 2: Al, Cu, Au, Ag at Room Temperature

~ 1 GPa tensile

~ 100 to 200 MPa compressive

Stress state depends on material and final film thickness.

after R. Abermann, Vacuum 41, 1279 (1990)
Experimental results - Fe on SiO$_2$ at Different Substrate Temperatures

Stress Evolution
During Evaporative Deposition of Polycrystalline Films
**In-situ Stress Monitoring: Cantilever Curvature**

- **Sample**
- **UHV system**
- **Monoatomic flux**
- **E-beam source**

$h_{\text{beam}} \gg h_{\text{film}}$

$h_{\text{film}}\sigma_{\text{film}} = \kappa \frac{E_{\text{beam}}}{(1 - \nu_{\text{beam}})} h_{\text{beam}}^2$

- Measure tip deflection $\Rightarrow$ determine $\kappa$ $\Rightarrow$ determine $h_{\text{film}}\sigma_{\text{film}}$
Displacement Measurements

Laser reflection system

- Displacement resolution <1nm

Capacitance system

- Displacement resolution <1nm
- Bandwidth ~ 1 kHz

(C. Friesen, Ph.D. Thesis, M.S.E., M.I.T.)
MEMS-Based In-Situ Stress Measurement

Micromachined Piezoresistive Microcantilever

In-Situ Stress Measurement During Deposition of a Cu Film

Stress vs. Thickness for Films Deposited via Evaporative Deposition

Type 1: e.g., Ti, W, Ta, TiN, Cr at room temperature

Type 2: Al, Cu, Au, Ag at Room Temperature

~1 GPa tensile

e.g. Ti, W, Ta, TiN, Cr at R.T. (Si at 600°C < T_{dep}/T_m < 1100°C)
e.g. Al, Cu, Au, Ag at R.T. (Si at 1100°C < T_{dep}/T_m)
Stress-Thickness Evolution During Volmer-Weber (Cu)

Cu on borosilicate glass, UHV evaporation at 1A/s and room temperature
Nucleation and Growth Leading to Formation of a Polycrystalline Film
Tensile Stress Generation Due to Island Coalescence

Surface Energy

Grain Boundary Energy

\[ \sigma = \left[ \frac{2\gamma_s - \gamma_{gb}}{r} \frac{E}{1-\nu} \right]^{1/2} \]

Finite Element Method (FEM) Analysis of Island Coalescence

Output from FEM calculation:
• strain energy
• average stress in island

Equilibrium value of $z_0/r = 0.2$ for coalescence of 10 nm Ag island.

Comparison of Models

Coalescence of Ag islands with $90^0$ contact angle with substrate

Stress due to island coalescence mechanism affected by:
- contact angle with substrate
- degree of traction with substrate

a force balance requires: \( \gamma_s = \gamma_i + \gamma_f \cos(\theta) \)

models so far have assumed \( \theta = 90^0 \) (e.g. \( \gamma_s = \gamma_i \))

if \( \gamma_s > \gamma_i \) then \( \theta < 90^0 \)
Influence of Contact Angle on Island-Coalescence Stress

The Tensile Stress Due to “Boundary Zipping” Depends on:

• The island size at coalescence \( r \), which depends on the adatom mobility, the island nucleation rate, and the island growth rate

  \[
  \text{Adatom Mobility } \downarrow \Rightarrow r \downarrow \Rightarrow \sigma_{\text{zip}} \uparrow
  \]

  \[
  \text{Substrate Temperature } \downarrow \Rightarrow r \downarrow \Rightarrow \sigma_{\text{zip}} \uparrow
  \]

  \[
  \text{Deposition Flux } \uparrow \Rightarrow r \downarrow \Rightarrow \sigma_{\text{zip}} \uparrow
  \]

(C.V. Thompson, J. Mater Res., V14, p3164, 1999.)

• The film surface energy, \( \gamma_s \).

• The film-substrate surface energy, \( \gamma_i \) (e.g. the choice of substrate).
Stress-Thickness Evolution During Volmer-Weber (Cu)

Cu on borosilicate glass, UHV evaporation at 1A/s and room temperature
Origin of Pre-Coalescence Compressive Stress?

\[ f = \text{surface stress} = \gamma + \frac{\partial \gamma}{\partial \epsilon} \]

When islands are small, Laplace pressure leads to high stress and a lattice parameter smaller than equilibrium, which is "locked in".


or

A compressive surface stress leads to a force that causes curvature (at all R).


but surface stresses are usually tensile (positive)!
Post-Coalescence Reversible Relaxation of Compressive Stress During a Growth Interruption

Pre-Coalescence Reversible Relaxation of Compressive Stress

Cu on borosilicate glass, UHV evaporation at 1A/s and room temperature.

Homoepitaxial Growth of Cu/Cu(111) and Ag/Ag(111)

- Reversible stress-thickness phenomenology quantitatively similar to that observed in polycrystalline films.

Reversible Stress Change Phenomenology Observed in:

• Volmer-Weber systems

Pre-coalescence    Coalescence    Post-coalescence

• Homoepitaxial systems
Some Definitions and Quantities to Note:

- **Initial instantaneous stress**: \( \sigma^+_{ss} \)
- **Reversible stress**: \( \sigma^-_{ss} \)
- **Change**: \( \Delta(\sigma-h)_{rlx} \)
- **Reversible change**: \( \frac{\partial(\sigma h)}{\partial h} \)_{init}
- **Time**: \( t_{rlx} \)
- **Time/Thickness**: \( t_{rec} \)
Effect of Pre-Interruption Deposition Flux in the Post-Coalescence Regime

Pre-interruption growth flux \( \Rightarrow \Delta (\sigma h)_{\text{rec}} \uparrow \)

Effect of Pre-Interruption Deposition Flux in Pre-Coalescence and Coalescence Regimes

\[ F_{\text{dep}} \uparrow \Rightarrow \Delta (\sigma h)_{\text{rec}} \uparrow \]


0.1 nm/s
\[ \Delta (\sigma h)_{\text{rec}} \sim 30 \text{ N/m} \]

0.1 nm/s
\[ \Delta (\sigma h)_{\text{rec}} \sim 30 \text{ N/m} \]

0.02 nm/s
\[ \Delta (\sigma h)_{\text{rec}} \sim 20 \text{ N/m} \]

0.1 nm/s
\[ \Delta (\sigma h)_{\text{rec}} \sim 30 \text{ N/m} \]
Instantaneous Stress \[d(\sigma - h)/dh\] vs. Thickness

\[
\left[ \frac{\partial (\sigma h)}{\partial h} \right]_{\text{init}}
\]

\(~ 1\text{GPa (}< 0.1 \text{ monolayer)}\)

Instantaneous Stress: $\partial(\sigma h)/\partial h$ when growth is resumed

Only perturbation to surface is increased adatom population.

can be accurately measured at short times using capacitive curvature measurements (1kHz)
Displacement Field Due to Adatom

- Interaction forces tend to displace nearest neighbors in surface radially outward

- Treat adatom as point force --- Lau & Kohn

\[ u(r) = \frac{1 - \nu^2}{\pi E} \frac{A}{r^2} \]

- The displacement field is generally compressive for isotropic surfaces.
Thermodynamic Treatment of Homo-Adatom Adsorption

Assuming B is small (a simple bond order argument $B \sim 0.04 \text{ eV/adatom}$), a simple relation makes a connection between experiment and the force-dipole magnitude $A$:

$$f' (\rho, A, B) = \gamma' (\rho, B) + \frac{\partial \gamma'}{\partial \varepsilon} (\rho, A),$$

chemical component of adatom/surface interaction

$$u(r) = \frac{1 - v^2}{\pi E} \frac{A}{r^2}$$

Post-coalescence data for VW growth of polycrystalline films:

\[ A = 0.668 \text{ eV} \pm 0.079 \text{ eV} \]

Epitaxial growth:

\[ A = 0.710 \pm 0.096 \]

( EAM calculations: \[ A = 0.572 \text{ eV} \])
Reversibly Relaxed Stress Change:

- Observed in all stages of Volmer-Weber (polycrystalline film) and homoepitaxial film growth.

- The component of the stress that reversibly relaxes during a growth interruption decreases with decreasing pre-interruption growth.

- The stress builds up faster when deposition is resumed, than it relaxes during an interruption. (Kinetically asymmetric, with a large initial instantaneous stress when growth is resumed.)

Model:

- Excess adatom population and kinetic roughening during growth lead to reversibly relaxed compressive surface stress. (Friesen and Thompson, Spaepen)
Reflection High Energy Electron Diffraction (RHEED) During Homoepitaxial Growth

- Flat surfaces give ‘streaky’ patterns, atomically rough surfaces give ‘spotty’ patterns.
- Therefore, monitor spot length during growth interruption.
RHEED data correlates quite well to stress-thickness data

- Both Ag/Ag(111) & Cu/Cu(111) growth at R.T. results in 3D/multilayer mode (kinetic roughening).

- Excess surface defects (adatoms, ledges, 2D islands) lead to compressive surface stress.

- Growth interruption allows relaxation of surface to flat, low stress state.
Model for the Origin of Residual Compressive Stress:
- Non-equilibrium, compressive *surface* stress develops during growth.
- The lattice structure adopted by the growing film is in equilibrium with the steady state surface state, leading to a net compressive residual stress.
Summary:

- Differential thermal expansion leads to residual stresses whose magnitudes are strongly affected by deformation, and therefore by dimension and encapsulation, of films.

- Intrinsic stresses can be highly tensile or compressive, depending on the film and substrate materials, the deposition technique, and the deposition conditions.

- Tensile intrinsic stresses in polycrystalline films are often associated with the island coalescence process.

- Compressive stresses are associated with shot peening, trapped gas, and the surface state during deposition.

- Residual stress can be controlled in predictable ways through control of deposition conditions, especially the deposition temperature and deposition rate.