Heterogeneous Stress Relaxation in Thin Films:
Whiskers, Hillocks, and Beyond

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Tin Film on Si Substrate

(a) Graph showing temperature variation with different ΔT values for 1.4 μm Sn film.

(b) Graph showing temperature variation with different ΔT values for 8 μm Sn film.

SEM image of tin film on Si substrate, scale bar 50 μm.
Sn-Cu intermetallic growth creates 10-20 MPa compressive stress in Sn films. Room Temperature = 60% $T_{\text{melting}}$

**Elastic**

**Plastic ( = permanent deformation)**

- Coble creep
- Nabarro-Herring creep
- Dislocation glide
- Delamination
- Cracks
- Hillocks
- Whiskers

- Dewetting
- Holes
- Coarsening
- Grain boundary sliding
- Grain boundary migration
- Grain boundary grooving

...
Film Heterogeneity
Evidence of Texture – FIB top view

- Ion channeling in focused ion beam imaging
- Contrast corresponds to grain orientation
- Anisotropy – elastic, plastic, diffusion, grain boundary properties - depends on crystal structure
- Effect depends on texture
Tin Whisker Formation in Electronic Circuits

Tin-Plated Connector Pins after 10 years
Courtesy of NASA - Goddard Space Flight Center

Photo from: http://nepp.nasa.gov/whisker/index.html
Understanding Whisker Growth

Whisker Morphology – Growth Processes

Microstructure of the System

- Tin film with a three-dimensional microstructure
- Grain shape and crystallographic orientation relationship
- Dependent on deposition conditions, electrolyte, substrate ...
- Stress relief mechanisms depending on film composition, creep, grain structure, IMC, ...

1/10,000 grains
1/100,000 grains ...

How do whiskers or hillocks “nucleate”?
Whisker Morphology – Surface Grain

Heterogeneous stress relaxation in thin films

- Simple model for whisker and hillock growth and shrinkage – grain boundary sliding limited diffusional creep
- “Nucleation” of whiskers – Laue/ALS
- Thermal cycling of “bicrystals” – orientation and geometry dependence – Laue/ALS
- In-situ measurements during thermal cycling
- Scientific questions needing answers
Sn-Cu intermetallic growth creates 10-20 MPa compressive stress in Sn films.

Room Temperature = 60% $T_{\text{melting}}$

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...
Accretion stress = $\sigma$  Sliding friction = $\beta$

For cylinder of radius $r$ and height $h$ in a film of thickness $t$

$$\sigma \pi r^2 > 2\pi r h \beta$$

For cylindrical grains of radius $r$ and height = film thickness $t$

$$\sigma \pi r^2 > 2\pi r t \beta$$

$$r > 2 \beta t / \sigma$$

Whisker Growth: GB Sliding

Straight whisker growing normal to film surface

Whisker Growth: GB Sliding

- Localized Coble creep → atomic flux to surface grain

Whisker Growth: GB Sliding

- GB are faceted (accretion and sliding components)
- Flux at accretion planes

Whisker Growth: GB Sliding

- GB are faceted (accretion and sliding components)
- Flux at accretion planes causes shear at the GB

![Diagram of whisker growth showing GB sliding and flux at accretion planes](image-url)
Whisker Growth: GB Sliding

- GB are faceted (accretion and sliding components)
- Flux at accretion planes causes shear at the GB
- Overcomes sliding friction ($\beta$)

Whisker Growth: GB Sliding

- GB are faceted (accretion and sliding components)
- Flux at accretion planes causes shear at the GB
- Overcomes sliding friction ($\beta$) $\rightarrow$ GB sliding

Sliding (Out-of-plane Growth)

Preferential accretion planes

Flux

Sliding

Flux

Sliding-induced Shear

Fricition $\beta$

Straight whisker growing normal to film surface
Whisker Growth: GB Sliding

- GB are faceted (accretion and sliding components)
- Flux at accretion planes causes shear at the GB
- Overcomes sliding friction (\(\beta\)) → GB sliding
Cylindrical Grain in a Thin Film with a Capping Oxide Layer

Accretion stress = $\sigma$  
Sliding friction = $\beta$  
Oxide fracture stress = $\sigma_{\text{oxide}}$

For cylinder of radius $r$ and height $h$ in a film of thickness $t$ capped by an oxide of thickness $x$

$\sigma \pi r^2 > 2\pi r h \beta + 2\pi r x \sigma_{\text{oxide}}$
Grain Subsidence: GB Sliding

- GB are faceted (accretion and sliding components)
- Flux at accretion planes causes shear at the GB
- Overcomes sliding friction (β) → GB sliding
- What if the film stress becomes tensile?
Grain Subsidence: GB Sliding

- GB are faceted (accretion and sliding components)
- Flux at accretion planes causes **shear** at the GB
- Overcomes **sliding friction** ($\beta$)
- **What if the film stress becomes tensile?**

[Diagram showing GB sliding with flux, shear, and sliding planes.]
Grain Subsidence: GB Sliding

- GB are faceted (accretion and sliding components)
- Flux at accretion planes causes shear at the GB
- Overcomes sliding friction ($\beta$)
Evolution of a sunken grain at (a) 2 cycles, (b) 5 cycles, and (c) 20 cycles.

Thermally Cycled Defects: Grain Subsidence
Microstructure Dependent Stress States – Elastic Strain Energy Density (calculated)

<table>
<thead>
<tr>
<th>Texture Orientation</th>
<th>(001)</th>
<th>(111)</th>
<th>(110)</th>
<th>(100)</th>
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<tr>
<td>Skeleton</td>
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<td><img src="b" alt="Image" /></td>
<td><img src="c" alt="Image" /></td>
<td><img src="d" alt="Image" /></td>
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<tr>
<td>Elastic Stress</td>
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<td><img src="g" alt="Image" /></td>
<td><img src="h" alt="Image" /></td>
<td><img src="i" alt="Image" /></td>
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<tr>
<td>Thermal Cycling</td>
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<td><img src="k" alt="Image" /></td>
<td><img src="l" alt="Image" /></td>
<td><img src="m" alt="Image" /></td>
</tr>
</tbody>
</table>

Polychromatic x-ray synchrotron micro-diffraction
- Advanced Light Source (ALS), beamline 12.3.2
- Martin Kunz, Tamura et al. 2009
- 1µm × 1µm beam size
- 2 µm (before thermal cycling) & 1µm (after thermal cycling) step sizes

Laue diffraction – Crystallographic grain orientations, elastic deviatoric strains, average peak-widths indexed using the XMAS software (Tamura, MacDowell et al. 2003).
Relative geometrically necessary dislocation (GND) densities estimated from the average peak-width, and elastic strain energy densities (ESEDs) were calculated by assuming a zero stress normal to the sample plane (σ_{zz} = 0) (Tamura, MacDowell et al. 2003; Sarobol, Chen et al. 2013)

Purdue graduate students
Pylin Sarobol – Sandia
Ying Wang – Dow
Wei-Hsun Chen - Cymer

Local Grain Orientations Around Defects

Sn+0.7wt%Cu

20µm x 20µm areas around a whisker on electrodeposited SnCu film

Out-of-Plane Strain Distribution

- Synchrotron micro-diffraction gives grain orientation and strain.
- OOF2 simulation used the measured grain orientations to calculate elastic strain. All grains assumed to be columnar with the same height. 2% uniform biaxial compressive strain applied to the film. Out-of-plane elastic strain due to grain misorientations calculated.
- Very HIGH strain predicted by simulation but very LOW strain measured at the whisker location → in actual sample, whisker grain grew out of the film plane and the strain was locally relaxed.

Whisker Growth from Solidified Film

- Larger grained samples by solidification
  - Melting and solidification of Sn-Ag-Cu solder on Cu
  - Thermally cycled -55°C to 85°C for 1500 cycles

- Defects much smaller (~5 μm) than grains formed during solidification (~1 mm)

- Large grain size advantageous for studying defect formation relative to the adjacent grains

Linear Defect Density (defects/mm)

Since the grains are large and the grain boundaries apparent, the behavior of the boundaries can be characterized by optical microscopy, SEM, EBSD, FIB, microdiffraction (ALS), FEA,…
Four Types of Behavior

None

Defects

Sliding

Defects + Sliding
Four Types of Behavior

None

Defects

Sliding

Defects + Sliding
Before and After Thermal Cycling:

SEM, EBSD, microdiffraction at ALS (single crystal and multi-crystal analysis)

After only: FIB
Evolution of a Microstructure during Thermal Cycling
How Metal Films Handle Stress

- Simple model for whisker and hillock growth and shrinkage – grain boundary sliding limited diffusional creep
- “Nucleation” of whiskers -
- Thermal cycling of “bicrystals” – orientation and geometry dependence
- In-situ measurements during thermal cycling
  
  Appearing and disappearing defects
- Unanswered questions
In-situ UHV Studies of Microstructural Evolution during Thermal Cycling

- Two Sn electrolytes - 5 µm Sn electroplated on 0.2 µm PVD Sn on tungsten sheet (99.95 mass% purity), 0.25 mm thick
- Substrate preparation:
  - Tungsten coupons polished to a 1 µm diamond finish
  - Cleaned and etched with 10% potassium hydroxide in water
  - Placed in electron beam (e-beam) evaporation deposition chamber operating with a base pressure in the low $10^{-6}$ Pa ($10^{-8}$ torr) range
  - Etched with an ionized argon beam for a final in-situ cleaning of the surface.
  - Deposition of a seed layer, 0.2 µm thick, of high-purity Sn (99.99 mass%) at low $10^{-4}$ Pa
- In-situ heating and cooling stage in UHV SEM – base pressure $10^{-9}$Pa
  - Temperature calibrated
In-situ UHV Studies of Microstructural Evolution during Thermal Cycling: Film 1
In-situ UHV Studies of Microstructural Evolution during Thermal Cycling: Film 2

Thermal strain = volumetric strain = $10^{-3}$
In-situ UHV Studies of Microstructural Evolution during Thermal Cycling

Behavior depends on the film and the oxide
Many more local changes observed

Wafer curvature measurements not enough ...
Observation after cycling is not enough ...
Hillock Formation and Grain Growth – 500 nm Gold on Silicon

<table>
<thead>
<tr>
<th>Accelerating Voltage</th>
<th>Working Distance</th>
<th>Magnification</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 kV</td>
<td>9.9 mm</td>
<td>5000 x</td>
</tr>
</tbody>
</table>

5 μm
Grain Growth and Dewetting in Ag Films
- 50 nm Ag on Si
- AFM after initial SEM observation and annealing

Contamination layer from initial SEM imaging

M. Kammer, 2014
Ph.D. Thesis
How Metal Films Handle Stress

- Simple model for whisker and hillock growth and shrinkage – grain boundary sliding limited diffusional creep
- “Nucleation” of whiskers -
- Thermal cycling of “bicrystals” – orientation and geometry dependence
- In-situ measurements during thermal cycling

**Appearing and disappearing defects**

- Requires 3D, in-situ imaging experiments with sufficient spatial and temporal resolution
  - APS, NSRRC, others …
  - Your suggestions welcome
The mechanism of initial de-wetting and detachment of thin Au films on YSZ

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Fig. 11. SE SEM micrograph of a 200-nm thick Au film on YSZ annealed at 700 °C for 27 h. The large holes are the result of coalescence of smaller holes.

Fig. 7. (a) BSE SEM micrograph of a small hillock in the Au film, after annealing at 700 °C for 10 min and (b) cross-section SEM micrograph of two hillocks in the Au film.
High resolution TEM image of as-electrodeposited tin film

- electron-beam Pt protective layer
- thin carbon layer
- (6.8±0.5)nm Sn<sub>x</sub>O<sub>y</sub> layer
- Sn grain
Scanning TEM image as-electrodeposited tin film

- Thin carbon layer shows strong contrast with Sn$_x$O$_y$ layer
- Electron-beam Pt protective layer
- Sn grain
- (6.8±0.5)nm Sn$_x$O$_y$ layer
- 20 nm scale bar