Interfacial Mechanical Integrity of Layered Interconnect Stacks: Experiment Characterization By Nano-Indentation

Ashraf -F. Bastawros
Department of Aerospace Engineering, Iowa State University


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Outline

• Motivation

• Ultrathin film mechanical property
  - Layered Films
  - Interfaces

• Adhesive and cohesive measurements

• Summary
Motivation

ITRS-2007 Grand Challenge: Reliability: Improved test methodologies and modeling of Cu/Low-K interface reliability

Needs:

- **Back End Processing:**
  New experimental methods to assess the mechanical integrity of stacks with multiple interconnect layers

- **Die/Package Interface: Metrology**
  a) *Thermo-mechanical measurements:*
    - Material properties,
    - Detect onset of failure (*sub-um cracking and delaminations on packaged units*),
    - Identify failure mechanisms
    - Model verification for boundary conditions failure modes
  
  b) *Failure analysis:*
    - New techniques for probing the interface region, for blurred interfaces
Motivation

Develop testing technique to

- Mechanical properties of ultrathin films (modulus, flow stress)
- Detect the onset of failure (delamination and/or film fracture)
- Measure the cohesive and interfacial fracture energies.

AMD's 9 Layer Metal Stack

www.iue.tuwien.ac.at/phd/wessner/node27.html
Motivation

3D Integration - TSV (through Silicon Via)

ChipPAC, Courtesy of IMEC)
http://www.flipchips.com/tutorial71.html

- Deposited oxide thickness: 1.9-2.0μm on top side
- Sidewall oxide thickness at top sidewall is 1.3-1.4μm
- Sidewall thickness in middle is 0.7-0.8μm
- Thickness of oxide on the via-base is 0.35-0.45μm

TSV: Plasma deposited SiO₂

Ranganathan, et. al, IEEE/ECTC 2008 Proceedings, pp. 859-865
1. Measuring Film Mechanical Properties

Layered Stack
(Film on a substrate)
1. Measuring Film Mechanical Properties

Combination of nano-scratch and nano indentation to measure

- Thickness
- Flow Stress
- Modulus

Thickness, Flow Stress, Modulus
Nano-Scratch Elementary Model

Assume:
- No pile-up or sink-in
- Statically admissible state of stress (SASS)

\[ P_o \approx \xi \sigma_y \]

\[ F_n \approx \pi \xi \sigma_{ys} \left( R(h-t) + \frac{\sigma_{yf}}{\sigma_{ys}} Rt \right) \]

\[ F_t \approx \frac{4\sqrt{2}R}{3} \xi \sigma_{ys} \left( (h-t)^{3/2} + \frac{\sigma_{yf}}{\sigma_{ys}} \left( h^{3/2} - (h-t)^{3/2} \right) \right) \]
Nano scratch: Oxide formation in copper (0.6 wt% NH₄OH)

- Electroplated copper
- Planarization
- Chemical exposure then mechanical scratch

Ref. scratch depth < Ch. Exp. scratch depth

softening
Nano scratch: Oxide formation in copper (0.6 wt% NH₄OH)

![Graph showing % change in h vs. Normal force (µN) for different exposure times (90, 300, 600 seconds).]

<table>
<thead>
<tr>
<th>Exposure Time (s)</th>
<th>90</th>
<th>300</th>
<th>600</th>
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<tbody>
<tr>
<td>Film thickness, t (nm)</td>
<td>5.0±0.2</td>
<td>5.7±0.6</td>
<td>7.3±0.8</td>
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<tr>
<td>Film strength $\sigma_{yf}/\sigma_{ys}$</td>
<td>0.52</td>
<td>0.48</td>
<td>0.45</td>
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Angle resolved XPS ---
Chawla et al (1992)

- XPS signal intensity ratio with take off angle from Cu surface
- High angle measurements (@ 80° are used to fit 2-layer model
**Comparison: XPS- vs. Nano-scratch**

**XPS- Prediction**

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<tr>
<td>1&lt;sup&gt;st&lt;/sup&gt; layer (nm)</td>
<td>0.8</td>
<td>0.6</td>
<td>0.5</td>
</tr>
<tr>
<td>2&lt;sup&gt;nd&lt;/sup&gt; layer (nm)</td>
<td>4.0</td>
<td>5.2</td>
<td>6.6</td>
</tr>
<tr>
<td>Film thickness, t (nm)</td>
<td>4.8</td>
<td>5.8</td>
<td>7.1</td>
</tr>
</tbody>
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**Nano-scratch Prediction**

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Dynamic Stiffness Measurements (DSM)

- **Quasi-Static load**
- **Dynamic load**
  - Amplitude = 50% of static load
  - $f = 10\text{Hz}$
DSM-Convoluted Stiffness Measurements

(a) 0 seconds
Constant

(b) 90 seconds
20-25nm
$h/t \sim 4-5$

(c) 300 seconds
28-33nm
$h/t \sim 4-5$

(d) 600 seconds
32-38nm
$h/t \sim 4-5$
DSM-FEM Calibration

- Axisymmetric Rigid indenter, on elastoplastic, strain hardening film/substrate
- Changed $\frac{E_{yf}}{E_{ys}}$ between 0.1-1.0
- Used Oliver and Pharr, 1992 contact modulus definition
- Generated calibration master curve
FEM Master Calibration Curve

For the tested film system \( E_{yf} / E_{ys} \approx 0.2 \)
2. Measuring Film Mechanical Properties

Perpendicular Interface

- Deposited oxide thickness: 1.9-2.0μm on top side
- Sidewall oxide thickness at top sidewall is 1.3-1.4μm
- Sidewall thickness in middle is 0.7-0.8μm
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Measuring Film Mechanical Properties

- Perform a line of indent across the interface
- Estimate the apparent interface thickness, W
- Correlate W with t utilizing FEM calibration, for a given material parameter set
- Provide an empirical fit for the FEM correlations
FEM Calibration of Interface Measurements

![Diagram showing hardness (H/σ_y) vs position (x/R) for different ratios of Young's modulus (E_i/E_2) and stress (σ_{y1}/σ_{y2})](image)

- **Position (x/R)**: -4.0, -2.0, 0.0, 2.0, 4.0
- **Hardness (H/σ_y)**: 6.6, 6.4, 6.2, 6.0, 5.8, 5.6, 5.4

- **(a) x=-5T**: Material I, Material II
- **(b) x=-2T**: Material I, Material II
- **(c) x=0**: Material I, Material II
- **(d) x=2T**: Material I, Material II
- **(e) x=5T**: Material I, Material II

- **σ_{y1}/σ_{y2} = 1.0**
- **E_i/E_2 = 3.0**
- **T/R = 1.0**

---

**Material I**

**Material II**

---

**σ_{x}/σ_y**

- +4.0%
- +3.0%
- +2.0%
- +1.0%
- +0.5%
- +1.5%
- +2.0%
Representation of the FEM Results for “T-W” Correlation

Measured by Nano indentation

Estimated Interface Thickness

Material Parameters

\[ \frac{W - \xi T}{R} = A \left( \frac{E_1 \sigma_{y1} h_2}{E_2 \sigma_{y2} h_1} - 1 \right)^{\alpha} \]

- Measured by Nano indentation
- Estimated Interface Thickness
- Material Parameters

\[ A = 1.9365 \]
\[ \alpha = 0.1267 \]
\[ \xi = 0.26 \]
3. Measuring Film Adhesive and Cohesive Fracture Toughness in Ultrathin Films

Failure due to poor interfacial adhesion
Testing for Adhesive and cohesive Strength

**Many Conventional Methods:**
- Four-point bend and double cantilever test
- Tape peel and stud pull test
- Blister test
- Superlayer test

**Limitations:**
- Difficulty in sample preparation, especially for ultrathin films
- Difficulty in gripping to match with boundary condition for modeling
- Limited instrumentation resolution
- Stochastic variation at the submicron length scale
Conventional: Film Fracture & Delamination by Nanoindentation

- Test adhesion (weak interface) or film fracture (brittle)
- Reduce Substrate effect

\[ \Gamma_c = \Gamma(P_{\text{max}}, c) \]

Blister formation, W-superlayer
(Volinsky et al, 2002)

Radial Cracking of ta-C
(Jungk et al, 2006)
Combined Nano-Indentation/
Nano-AE Fracture Measurements
Combined Nano-Indentation/
Nano-AE Fracture Measurements

- No sample preparation
- No superlayer
- Mixed film fracture and delamination
- Deal with pile up!
Combined Nano-Indentation/
Nano-AE Fracture Measurements

![Load-Displacement Curve (cube corner tip)](image)

- **O (50nm)**
- **AE-events**
- **Displacement excursion**
Analysis: Film Fracture and Delamination

AE-Energy for different events; channel cracks have the highest AE-energy emission.

- Total fracture energy from displacement excursion
- Cohesive/Adhesive Energy partition from AE-energy ratio
Combined Nano-Indentation/Nano-AE Fracture Measurements

Crack Length Measurements:
- AFM or SEM measurements of channel cracks
- AFM, measure of delamination front
Test Results for SiC\textsubscript{x}N\textsubscript{y}

Tested two sets of “Nitrogen-doped silicon carbide, SiC\textsubscript{x}N\textsubscript{y}” thin films on Si (Courtesy of IBM, 40nm nominal thickness) with two different residual stress states, tensile and compressive.

<table>
<thead>
<tr>
<th>Wafer Type</th>
<th>Thickness A°</th>
<th>Residual stress</th>
<th>Modulus GPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wafer-1 96SJG4</td>
<td>452.0</td>
<td>430MPa (tensile)</td>
<td>85.3+/-6</td>
</tr>
<tr>
<td>Wafer-2 08SJA0</td>
<td>404.7</td>
<td>-460MPa (compressive)</td>
<td>93.2+/-2.6</td>
</tr>
</tbody>
</table>
Total Crack Length vs. Final Load

Utilize average measurements
Delamination Size vs. Final Load

Utilize average measurements

- Wafer 1
  - 96SJG4 (+σ)

- Wafer 2
  - 08SJA0 (-σ)

Extent of delamination

Final Load

δ (nm)

Load (μN)
Displacement jump vs. excursion Load

Excursion Load vs. Load (µN)

- Wafer 1
  - Jump-I
  - Jump-II

- Wafer 2
  - Jump-I
  - Jump-II

- 96SJG4 (±σ)
- 08SJA0 (±σ)
Test Results for SiC\textsubscript{x}Ny

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* Preliminary estimates, does not account for residual stresses

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<tr>
<th>Film Type</th>
<th>Film Toughness* ( \Gamma\textsubscript{film} ) (kJ/m\textsuperscript{2})</th>
<th>Fracture Toughness* ( K\textsubscript{c} ) (MPa m)</th>
<th>Interface Adhesion* ( \Gamma\textsubscript{interface} ) (J/m\textsuperscript{2})</th>
</tr>
</thead>
<tbody>
<tr>
<td>96SJG4</td>
<td>1.21</td>
<td>7.40</td>
<td>0.42</td>
</tr>
<tr>
<td>Wafer-1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>08SJA0</td>
<td>0.71</td>
<td>5.37</td>
<td>0.35</td>
</tr>
<tr>
<td>Wafer-2</td>
<td></td>
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Summary

- Approximate, yet Simple experimental approach based on nano-scratch and DSM nano-indentation measurements to measure:
  - Film mechanical properties
  - Adhesion and cohesion energies
- No specimen preparation is required.
- At the nano-scale, hybrid experimental/FEM is required.
- Further work is needed to examine other conditions of hard film on soft substrate and excessive sink-in or pile-up.