COMBINED NANOINDENTATION AND AFAM FOR MECHANICAL CHARACTERIZATION OF ULTRA LOW-K THIN FILMS

André Clausner¹ (andre.clausner@ikts.fraunhofer.de), Ehrenfried Zschech¹, Martin Gall¹, Matthias Kraatz¹, Malgorzata Kopycinska-Mueller¹, Yvonne Standke¹, Uwe Mühle¹, Elham Moayedi¹, Kong Boon Yeap², Khashayar Pakbaz³, and Sukesh Mahajan³

¹ Fraunhofer IKTS-MD, Germany; ² GLOBALFOUNDRIES, USA; ³ SBA Materials, USA
AGENDA

- Introduction, materials
- Nano-indentation of ULK film
- Combined Nano-indentation and AFAM
- Drawing conclusions on the pore topology using mechanical data
- Take home messages
Ultra Low-k nano-porous materials in nano-electronics

- Decreasing on-chip interconnect pitch (including inter-layer dielectrics dimensions) in nano-electronic products → higher signal delay, power loss, …
- Need of dielectric materials with ultra low k-values (ULKs)
  - Nano-porous organosilicate glasses (OSGs) for k-values below 3,0
Fabrication of organosilicate glass UKL thin films

Samples of building block in the OSG network [1].

Application: sol-gel processes using spin coating and final curing or CVD deposition.

OSG chemistry can include porogens for insertion of controlled porosity.

Mechanical strength of nano-porous OSG ULKs

- Reliability issues caused by crack propagation (CPI, thermo-mech. stresses)
- Gradients in the ULK film can lead to electrical failure even if mean k-value is OK
- Mechanical characterization of the ULK films is important (E, Gradients)

Criterion for crack propagation:
\[ \frac{\pi \sigma^2 a}{E} \geq 2\gamma \]

Low E leads to easy crack propagation.

Decrease of E with higher p.

Elastic modulus of porous OSGs

Crack propagation in a multilevel interconnect.
Motivation: high elastic modulus at a given $k$-value

- Optimizing the chemical structure and/or the pore topology
- One example: producing an ordered pore structure:

Self assembly sol gel process (SBA materials): Triblock co-polymer, removed by thermal or UV curing.

Techniques needed for the measurement of the mechanical properties of nano-porous thin OSG films

→ Nanoindentation and AFAM

<table>
<thead>
<tr>
<th>Sample</th>
<th>Target $k$</th>
<th>Actual $k$</th>
<th>Porosity, $p$ (%)</th>
<th>Thickness (nm)</th>
<th>Elastic Modulus, $E$ (GPa)</th>
<th>Cure process</th>
</tr>
</thead>
<tbody>
<tr>
<td>SA-OSG1</td>
<td>2.4</td>
<td>1.80</td>
<td>49</td>
<td>600</td>
<td>3.1 ± 0.1</td>
<td>Thermal</td>
</tr>
<tr>
<td>SA-OSG2</td>
<td>2.0</td>
<td>2.08</td>
<td>42</td>
<td>650</td>
<td>3.8 ± 0.2</td>
<td>Thermal</td>
</tr>
<tr>
<td>SA-OSG3</td>
<td>2.2</td>
<td>2.21</td>
<td>30</td>
<td>716</td>
<td>5.5 ± 0.3</td>
<td>Thermal</td>
</tr>
<tr>
<td>SA-OSG4</td>
<td>2.2</td>
<td>2.25</td>
<td>31</td>
<td>620</td>
<td>6.5 ± 0.3</td>
<td>Thermal</td>
</tr>
<tr>
<td>SA-OSG5</td>
<td>2.4</td>
<td>2.37</td>
<td>27</td>
<td>627</td>
<td>6.9 ± 0.4</td>
<td>Thermal</td>
</tr>
<tr>
<td>SA-OSG6</td>
<td>2.4</td>
<td>2.41</td>
<td>24</td>
<td>693</td>
<td>7.3 ± 0.3</td>
<td>Thermal</td>
</tr>
<tr>
<td>CVD-OSG1</td>
<td>2.4</td>
<td>N.A.</td>
<td>25</td>
<td>530</td>
<td>3.7 ± 0.3</td>
<td>UV &amp; Thermal</td>
</tr>
<tr>
<td>CVD-OSG2</td>
<td>2.7</td>
<td>N.A.</td>
<td>12</td>
<td>660</td>
<td>6.6 ± 0.7</td>
<td>Thermal</td>
</tr>
<tr>
<td>CVD-OSG3</td>
<td>3.0</td>
<td>N.A.</td>
<td>0</td>
<td>520</td>
<td>13.0 ± 1.2</td>
<td>Thermal</td>
</tr>
</tbody>
</table>

TABLE 1. Sample description and experimental results for SA-OSG and CVD films.
Samples: OSG thin film samples from SBA materials

First set of SBA SA-OSG ULKs

<table>
<thead>
<tr>
<th>Sample</th>
<th>K</th>
<th>Porosity p</th>
<th>Film thickness (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3</td>
<td>0,000</td>
<td>582</td>
</tr>
<tr>
<td>2</td>
<td>2,88</td>
<td>0,051</td>
<td>607</td>
</tr>
<tr>
<td>3</td>
<td>2,87</td>
<td>0,055</td>
<td>553</td>
</tr>
<tr>
<td>4</td>
<td>2,39</td>
<td>0,258</td>
<td>564</td>
</tr>
<tr>
<td>5</td>
<td>2,27</td>
<td>0,309</td>
<td>512</td>
</tr>
<tr>
<td>6</td>
<td>2,25</td>
<td>0,318</td>
<td>491</td>
</tr>
<tr>
<td>7</td>
<td>2,19</td>
<td>0,343</td>
<td>490</td>
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<tr>
<td>8</td>
<td>2,05</td>
<td>0,403</td>
<td>492</td>
</tr>
<tr>
<td>9</td>
<td>1,92</td>
<td>0,458</td>
<td>490</td>
</tr>
<tr>
<td>10</td>
<td>1,91</td>
<td>0,462</td>
<td>470</td>
</tr>
<tr>
<td>11</td>
<td>1,82</td>
<td>0,500</td>
<td>504</td>
</tr>
</tbody>
</table>

Second set of SBA SA-OSG ULKs

<table>
<thead>
<tr>
<th>Sample</th>
<th>k</th>
<th>Porosity p</th>
<th>Film thickness (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2</td>
<td>0,4</td>
<td>511,14</td>
</tr>
<tr>
<td>2</td>
<td>2,2</td>
<td>0,31</td>
<td>502,38</td>
</tr>
<tr>
<td>3</td>
<td>2,3</td>
<td>0,28</td>
<td>468,87</td>
</tr>
<tr>
<td>4</td>
<td>2,4</td>
<td>0,23</td>
<td>321,09</td>
</tr>
<tr>
<td>5</td>
<td>2,5</td>
<td>0,19</td>
<td>393,14</td>
</tr>
<tr>
<td>6</td>
<td>2,6</td>
<td>0,14</td>
<td>219,92</td>
</tr>
<tr>
<td>7</td>
<td>3</td>
<td>0</td>
<td>335,93</td>
</tr>
</tbody>
</table>

- SBA Spin-on OSG ULKs featuring a self-assembly process of the porogen
Nano-indentation

A schematic of the Hysitron nanoindentation system.

- Hardness $H$
- Elastic modulus $E$

Elastic-plastic contact with Berkovich tips

$$S = \frac{dF}{dh} = \beta \frac{2}{\sqrt{\pi}} \sqrt{A_c}$$

Hysitron TI-950
Elastic-plastic nanoindentation on thin films

- **Red**: yield zone

**Thin film**

**Substrate**

**Hardness H**
- Mainly determined by the yield zone
- Local property
- Low Substrate influence

**Elastic modulus E**
- Mainly determined by the elastic field outside the yield zone
- More a global property
- High substrate influence
Indentation hardness and modulus of thin films

- Hardness is ruled by the yield zone
- Yield zone should not reach substrate
  - Safe contact: $h_c < 1/10 \times h_{film}$ (Buckle rule)
- For $E$, 10% rule is not appropriate
- Elastic fields outreach much further
  - Indentation depths < 1% of $h_{film}$ needed

Yield zone radius vs. contact depth

- $a_Y$ always well below $10 \times h_c$

Film vs. substrate modulus

- Elastic measurement with spherical indenter
## Indentation hardness H for the first sample set

### Hardness gradients vs. porosity p

- **Tip rounding influence**
- **Substrate influence at higher indentation depths**

#### Sample data:
- Sample 3, p=0%, k=2.88
- Sample 6, p=32%, k=2.25
- Sample 10, p=42%, k=1.92
- Sample 11, p=50%, k=1.82

#### Graph:
- Normalized hardness H vs. contact depth h_c/nm
- Porosity p dependent surface gradient
- Harder and maybe denser top layer

### Hardness increase at the surface vs. p

- **Porosity dependent surface gradient**
- **Harder and maybe denser top layer**
Indentation hardness $H$ for the second sample set

Hardness gradients vs. porosity $p$

- Sample 1, $p=0.4$, $k=2$
- Sample 2, $p=0.31$, $k=2.2$
- Sample 4, $p=0.23$, $k=2.4$
- Sample 7, $p=0$, $k=3.0$

Substrate influence

Surface gradient

Hardness increase at the surface vs. $p$

Porosity dependent surface gradient

Harder and maybe denser top layer
Nano-indentation results for the Elastic modulus $E$

**First sample set**

- Sample 1, $p=0\%$, $k=3.0$
- Sample 4, $p=26\%$, $k=2.4$
- Sample 6, $p=32\%$, $k=2.25$
- Sample 11, $p=50\%$, $k=1.82$

**Second sample set**

- Sample 1, $p=0.4$, $k=2$
- Sample 2, $p=0.31$, $k=2.2$
- Sample 5, $p=0.19$, $k=2.5$
- Sample 7, $p=0$, $k=3.0$

- Forces are too high to significantly surpass substrate influence
- Surface gradient not visible
- Need for a higher resolution $E$ measurement $\rightarrow$ AFAM
AFAM principle

AFM contact mode

AFAM – add on
Contact resonance frequencies of an AFM cantilever

\[ f_n = F_i(k^*, k_c, L_1, L) \]

Dynamic behavior of the cantilever

Tip-sample contact stiffness

Contact mechanics

\[ k^* = 2aE^* \]
Elastic contact on thin films

Main aspects for the substrate influence on the E measurements

- Tip radius → the bigger the tip radius the deeper reaches the elastic field
- Contact force → the bigger the contact force the deeper the elastic field

Low forces and sharp tips for the E-gradient measurements
Surface gradients for the elastic modulus become visible via AFAM!

AFAM studies intensified for the second sample set
Combined AFAM and nano-indentation: Second sample set

- Surface gradients for the elastic modulus become visible with sharp tips
- Film modulus becomes visible for round tips
Surface gradient in the elastic modulus, AFAM results

First sample set

Second sample set

➢ AFAM also shows porosity dependent surface gradient
Comparison of AFAM and nano-indentation results

First sample set

Second sample set

- AFAM shows very comparable results to nano-indentation
OSG pore topology and elastic modulus

Random overlapping spherical pores
Hashin-Shtrikman upper bound
Ordered non-overlapping

Random overlapping spherical solids

OSG pore topology and elastic modulus

Random overlapping spherical pores

Hashin-Shtrikman upper bound

Ordered non-overlapping

Normalized elastic modulus, $E/E_s$

Porosity, $p$

Take home messages

- AFAM and nano-indentation complement each other well for the mechanical characterization of porous thin films.

- From mechanical data of porous thin films, conclusions about the pore-topology can be drawn.
Thank you for your attention!

- You are invited to work with us on challenging topics in a city that is:
  - One of most important *European Centers of Microelectronics*
  - a *Center of Materials Science and Engineering*
  - and full of history and culture, with a high quality of life and an excellent surrounding.