EUV-photoresist: characterization tools and techniques at NIST

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Outline

• Resist outgassing
  – Total outgas by pressure rise
  – Analysis by Gas Chromatography/Mass Spectroscopy (GC/MS)
• Dose-to-Clear ($E_0$) measurement
• EUV interference lithography (EUV-IL)
• Summary
Resist outgas: rates and analysis

Unique expertise available at NIST

Electron and Optical Physics Division
• SURF III EUV radiation source
• Extensive UHV experience
• Familiarity with EUVL community and issues
• Unique GC/MS instrument outfitted with cryofocus sample injection

Process Measurements Division
• Absolute pressure measurement

Analytical Chemistry Division
• GC/MS expertise

Polymers Division
• Photoresist expertise

Center for Nanoscale Science and Technology (CNST)
• Photoresist pre- and post-exposure processing expertise
Outgassing at SURF III

Data from several runs made under varying conditions (blue, green, and black are full intensity)
## Data Analysis (8 runs)

<table>
<thead>
<tr>
<th>post-exposure time (min)</th>
<th>average ((10^{14} \text{ cm}^{-2}))</th>
<th>Std. dev. ((10^{14} \text{ cm}^{-2}))</th>
<th>Std. dev.</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>2.77</td>
<td>0.20</td>
<td>7.2%</td>
</tr>
<tr>
<td>5</td>
<td>2.99</td>
<td>0.13</td>
<td>4.3%</td>
</tr>
<tr>
<td>10</td>
<td>3.11</td>
<td>0.06</td>
<td>1.9%</td>
</tr>
<tr>
<td>20</td>
<td>3.20</td>
<td>0.18</td>
<td>5.6%</td>
</tr>
<tr>
<td>30</td>
<td>3.11</td>
<td>--</td>
<td>--</td>
</tr>
</tbody>
</table>
GC/MS with cryofocus

Heated sample tube

Sample introduction

Analysis mode

6-way valve

Cryofocus

GC column

LN reservoir

N₂

He

GC column temp.

50°C

250°C

Time (min.)

0

15

-2
GC/MS analysis

1 - CO$_2$
2 - isobutene
3 - water
4 - acetone
5 - CS$_2$
6 - toluene
7 - tert-butylbenzene
Resist Outgassing Summary and further work

- Mechanical measurement of absolute pressure rise provides stable, repeatable measurements of area outgassing.
- Cryotrapping and subsequent GC/MS with cryofocus are relatively unbiased, reliable techniques for analysis of outgas components.
- Plan to bake out chamber into cryotrap to investigate non-volatiles.
- Plans to repeat measurements on pulsed source and different resists to get direct comparison between pulsed and synchrotron sources.
Resist Dose-to-clear measurements

• EUV resist sensitivities previously tied to a measurement of EUV-2D resist done on Sandia 10x microstepper ~ 10 years ago

• Recent absolute measurements at LBNL indicate 1.9x smaller $E_0$ for MET-1K than value based on Sandia EUV-2D measurement

• Independent second absolute determination of $E_0$ for EUV resist by NIST to confirm surprising large difference
Experimental Strategy

1. Existing beamline on SURF III modified specifically for this measurement; top hat uniformity better than 4% in central 2mm; absolute flux measured with a calibrated photodiode with 1% uncertainty.

2. Environmental control:
   a. Wafers loaded into beamline in glove bag with continuous nitrogen flow;
   b. Wafer transported in airtight mask containers to CNST Nanofab for PEB and processing in Amine-free clean room;
   c. PEB delay of ~ 20 minutes.

3. Processing done at CNST with state-of-the-art equipment.

CCD image of Illumination spot
Measure incoming EUV light with monitor photodiode and correlate with SURF III beam current. Monitor diode subsequently compared to diode measurements behind exposure aperture.
Experiment design

Expose wafer for a given time while recording SURF III beam current. Repeat 15 times to create exposure ring. Check flux relationship to beam current periodically with monitor diode.
NIST Results
(Resist B)

E₀ Dose to clear value

<table>
<thead>
<tr>
<th>Sample</th>
<th>E₀ (mJ/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>E01</td>
<td>5.72</td>
</tr>
<tr>
<td>E02</td>
<td>6.33</td>
</tr>
<tr>
<td>E04</td>
<td>5.75</td>
</tr>
<tr>
<td>Avg.</td>
<td>5.9</td>
</tr>
</tbody>
</table>

Measurements confirmed by Intel NanoSpec device

PEB Delay ~ 20min

NIST NanoSpec limit 10nm
Intel $E_0$ results based on assumed value for MET-1K
Resist Dose-to-Clear Summary

• NIST has made measurement of $E_0$ of a commercial EUV resist

• Resulting measurements @ NIST show $E_0 \sim 2\times$ smaller than measurements @ Intel based on assumed value for sensitivity of “standard” resist

• NIST measurements support LBNL’s previously announced $E_0$ data (2/08)

• NIST currently setup to continue work on other resists if industry deems it necessary; PEB delay of $\sim 20$ minutes can be shortened if necessary

• NIST investigating adding improved thickness measurements for resist thicknesses below 10 nm
EUV Interference Lithography

Goals:
• Add resist patterning on the nanoscale level to the set of EUV metrology tools at NIST
• Provide nanoscale test bed for NIST and industrial developers and users of EUV resists
• Prove applicability of cascaded grating design to EUV-IL and provide experience to optimize performance of such systems
• Expand capabilities of cascaded grating EUV-IL to include two-dimensional patterning
EUV Interference Lithography

Cascaded grating design

300-second time for exposure with 100 mJ/cm² resist
EUV Interference Lithography

Interferometer testbed construction complete. Stability tests underway.

Dedicated beamline design for implementation at SURF III
Summary

• Resist Outgassing:
  – Mechanical measurement of absolute pressure rise provides stable, repeatable measurements of area outgassing rate
  – Cryotrapping and subsequent GC/MS with cryofocus provide reliable analyses of outgas components

• Dose-to-clear:
  – NIST has made an absolute measurement of $E_0$ of an EUV resist
  – NIST currently setup to continue work on other resists as needed

• EUV-IL:
  – NIST has constructed interferometer testbed and have a design for EUV-IL dedicated beamline
  – Planned demonstration experiments on existing beamline to begin shortly.