Semiconductor Related Programs at NIST

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Outline

• Brief Introduction to NIST
• Areas of Work
• Applications
• Summary
National Institute of Standards and Technology

NIST strengthens the U.S. economy and improves the quality of life by working with industry to develop and apply technology, measurements, and standards.

NIST Assets Include:
- National measurement standards
- 3,200 employees
- $720 million annual budget
- 1,200 industrial partners
- 2,000 field agents
- 1,600 guest researchers
- $1.6 billion R&D partnerships with industry
- Baldrige Quality Award
NIST Major Programs

NIST Laboratories
Nation’s ultimate reference point for measurements and standards to support industry, science, health care, safety, defense.

Baldrige National Quality Program
Annual Baldrige awards in manufacturing, service, small business, education, and health care promote business excellence.

Advanced Technology Program
R&D partnerships with private sector to develop broadly beneficial new technologies.

Manufacturing Extension Partnership
Nationwide network of extension centers assisting the Nation’s 385,000 smaller manufacturers in all 50 states and Puerto Rico.
NIST FY2001 Budget

- **Advanced Technology Program**: $145.4M
- **NIST Laboratories**: $306.7M
- **Manufacturing Extension Partnership**: $104.9M
- **Other Agency**: $82.9M
- **Fees**: $39.8M
- **Construction**: $34.8M
- **Baldrige National Quality Program**: $5.2M

*Projected*
• Measurements and standards are critical enablers of the technology infrastructure

• Serve the American people through support of all major economic and scientific activities including:

• **Health care**
  • Standards for clinical tests, radiation diagnostics and treatment, many others.

• **Manufacturing**
  • Semiconductors, automotive, discrete parts, many others.
  • Dimensional standards, chemical and physical properties data, many others.

• **Information technology & telecommunications**
  • Standards and tests for computer security, interoperability, software performance, communications infrastructure, many others
NIST Measurements & Standards for Manufacturing

NIST support for the entire lithography process to manufacture microelectronic devices

- Light Scattering
- Optical Properties
- Index of Refraction of Gases
- Index of Refraction of Materials
- Laser Wavelength Standards
- Mask Properties
- Laser Power Measurements

12/20/2001 ERC Teleseminar
Areas of Work

• Lithography Metrology
• Critical Dimension and Overlay Metrology
• Thin Film and Shallow Junction Metrology
• Interconnect and Packaging Metrology
• Wafer Characterization and Process Metrology
• Test Metrology
Areas of Work (2)

- Lithography Metrology
  - 3 Projects
- Critical Dimension and Overlay Metrology
  - 6 Projects
- Thin Film and Shallow Junction Metrology
  - 3 Projects
Areas of Work (3)

- Interconnect and Packaging Metrology
  - 8 Projects
- Wafer Characterization and Process Metrology
  - 10 Projects
- Test Metrology
  - 2 Projects
Time Base of Programs

- **Short-term** (2002 – 2004): 40 projects
- **Long-term** (2009 – 2012): 5 projects
Applications
Intrinsic Birefringence of CaF$_2$
Birefringence in Cubic Crystals

I. Stress-Induced Birefringence

grown-in or externally applied (mounts, gravity, etc.)
• variable magnitude and orientation (sample-to-sample and within sample)
• weak dispersion visible-UV (NIST-SEMATECH 157 Review 11/00)
• can in principle be reduced to any desired value

II. Intrinsic Birefringence

due to symmetry breaking effect of finite q of photon at short λ
preliminary measurements in CaF$_2$ (above 157nm and 193nm target values)
• magnitude and orientation fixed by crystal (no sample dep., uniform)
• strong dispersion $\sim 1/\lambda^2$
• CANNOT be reduced! (inherent property of crystal)
(but since fully predictable and symmetric, can be corrected for in principle)
Has been measured in, e.g., Si$^1$ and GaAs$^2$

Implications

1) Intrinsic birefringence $\Delta n(157 \text{ nm}) \approx 6.5 \times 10^{-7}$ (6.5 nm/cm)
   • exceeds birefringence target value for 157 nm lithography (1 nm/cm)
     (1st Int. Symp. On 157 nm Lithography, May 2000)
2) Intrinsic birefringence $\Delta n(193\text{ nm}) = 3.6 \times 10^{-7}$ (3.6 nm/cm)
   • may exceed birefringence requirements of 193 nm lithography
3) $\Delta n = 0$ for [111] direction (lens orientation)
   • but [110] only $\theta = \cos^{-1}(2/3)^{1/2} = 35.26^\circ$ away
   • concern for high NA systems
4) Good news: effect completely predictable and symmetric
   • thus can correct for in principle
5) Need to know the full angle dependence of the effect
   • fortunately this is completely determined by symmetry alone
Birefringence Results for CaF$_2$

Measurements of Birefringence of CaF$_2$ in the UV

<table>
<thead>
<tr>
<th>Wavelength (nm)</th>
<th>Line Source</th>
<th>$10^7(n_{[110]} - n_{[001]})$</th>
</tr>
</thead>
<tbody>
<tr>
<td>365.062</td>
<td>Hg I</td>
<td>$-0.19 \pm 0.08$</td>
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<tr>
<td>253.652</td>
<td>Hg I</td>
<td>$-1.2 \pm 0.1$</td>
</tr>
<tr>
<td>193.09</td>
<td>CI</td>
<td>$-3.6 \pm 0.2$</td>
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<tr>
<td>184.95</td>
<td>CI</td>
<td>$-4.5 \pm 0.3$</td>
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<tr>
<td>175.19</td>
<td>CI</td>
<td>$-5.2 \pm 0.2$</td>
</tr>
<tr>
<td>165.72</td>
<td>CI</td>
<td>$-5.8 \pm 0.2$</td>
</tr>
<tr>
<td>156.10</td>
<td>CI</td>
<td>$-6.5 \pm 0.4$</td>
</tr>
</tbody>
</table>

$q \parallel [001] \rightarrow \Delta n = 0$

$q \parallel [111] \rightarrow \Delta n = 0$
Electroplating of Cu
Electrolytic Copper
On-Chip Metallization

Physical Vapor Deposition (PVD)
Chemical Vapor Deposition (CVD)
Electrodeposition
“superconformal deposition”
The Problem: Copper Metallization – 5-10 Years Out
ITRS --> several interconnect roadblocks at 70 nm node (2008)

Input: 1999 Advanced Metallization Conf.
Panos Andricacos (IBM)
Richard Alkire (Illinois)
Alan West (Columbia)
Tom Ritzdorf (Semitool)

Why NIST? Expertise
- electrochemistry
- surface characterization
- electron microscopy
- lithography

Environment
- fosters collaboration
- 3+ years out

Broad Application
- fundamental electrodeposition issues
- measurement opportunities
- MEMS/NEMS

Superconformal Deposition
extendibility beyond 100 nm (?)
lack of fundamental understanding

12/20/2001 ERC Teleseminar
Cl-PEG-MPSA $\rightarrow$ Hysteresis in Cu Kinetics

Copper Deposition
Current-Voltage Measurement

Cl-PEG-MPSA
Competitive Adsorption
Cu$^{2+} + 2e^- \rightarrow Cu$

12/20/2001 ERC Teleseminar
All data needed for the model obtained directly from hysteresis measurements on planar electrodes

Local time-dependent surface coverage:

\[
\frac{d\theta}{dt} = k_{\text{eff}} C_i (1-\theta) + \frac{i\Omega}{2F} \kappa \theta
\]

\( \kappa = 0 \) on flat electrodes,

\[
\frac{d\theta}{dt} = k_{\text{eff}} C_i (1-\theta) = \frac{D_{\text{MPSA}} (C_{\text{MPSA}} - C_i)}{\Gamma} \frac{\delta}{\Gamma}
\]

\( D_{\text{MPSA}} = \) MPSA diffusion coefficient
\( C_{\text{MPSA}} = \) MPSA bulk concentration
\( \Gamma = \) maximum MPSA coverage
\( \delta = \) diffusion layer thickness

Electrochemical kinetics for Cu deposition:

\[
i(\theta) = i_o(\theta) \left(1 - \frac{i(\theta)}{i_L}\right) \exp \left(\frac{-\alpha(\theta) F}{RT} \eta\right)
\]
Simulation: MPSA Concentration

- 0.0 µmol/L MPSA
- 0.5 µmol/L MPSA, Low θ, Insufficient Acceleration
- 5.0 µmol/L MPSA, Optimum θ, Superconformal Growth
- 40 µmol/L MPSA, Uniform Saturation, Conformal Growth
Accomplishments/Impact

Developed a cross-laboratory program on measurements and modeling for superconformal copper deposition (MSEL, EEEL, CSTL)
  • developed the first non-proprietary bath that yields superfill down to dimensions of 60 nm and aspect ratios of 3:1
  • demonstrated that inhibition alone is *not* sufficient to ensure superfill in direct contradiction to current thinking and models

New time saving measurement, on *planar substrates*, for determining superfill efficacy
  • demonstrated one-to-one correlation between I-V hysteresis, resistance drop (recrystallization rate) and superfill efficacy of electrolytes
  • incorporated into control software by ECI Technology, a leading supplier of analytical plating and processing tools, to complement their CVS (cyclic voltammetric stripping) quality control technology.

Theory predicts processing windows for superconformal deposition
  • alternative to the transport-limited inhibition model
  • provide the theoretical underpinning for filling nanometer scale features
Moisture Calibration
Low Concentration Humidity Standards

- Developed Standards for Low-Moisture Concentrations in Gases - Low-Frost Point Generator (LFPG)
  - High Precision Moisture Generator
    - Humidity levels:
      5 nmol/mol to ~ 5,000 µmol/mol
    - Operating temperature: 173 to 273 K
      (±2.5 mK stability at 173 K)
  - Planned improvements - 200 ppt
    (153K or -120° C operating temp.)
- Calibrated laser absorption hygrometer based on Wavelength Modulation Spectroscopy (WMS) between 5 and 2500 nmol/mol
- Incorporated WMS system as a check standard into the LFPG system as a null detector between LFPG and other humidity generators, e.g., permeation tube generators
Thermodynamic Properties of Reactive Gases
• Measurements of the speed of sound in reactive gases yield values for thermodynamic properties of the gases.

• These properties had only been estimated in the past due to the difficulties associated with making the measurements directly.
<table>
<thead>
<tr>
<th>Chemical</th>
<th>Formula</th>
<th>Formula</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>allene</td>
<td>C₃H₄</td>
<td>phosgene</td>
<td>COCl₂</td>
</tr>
<tr>
<td>arsenic trifluoride</td>
<td>AsF₃</td>
<td>phosphorous trifluoride</td>
<td>PF₃</td>
</tr>
<tr>
<td>arsenic</td>
<td>AsH₃</td>
<td>phosphorous pentafluoride</td>
<td>PF₅</td>
</tr>
<tr>
<td>trimethyl arsine</td>
<td>As(CH₃)₃</td>
<td>phosphine</td>
<td>PH₃</td>
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<tr>
<td>diborane</td>
<td>B₂H₆</td>
<td>sulfur dioxide</td>
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<td>pentaborane</td>
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<td>stibine</td>
<td>SbH₃</td>
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<td>boron trichloride</td>
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<td>silane</td>
<td>SiH₄</td>
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<td>Si₂H₆</td>
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<td>Cl₂</td>
<td>silicon tetrafluoride</td>
<td>SiF₄</td>
</tr>
<tr>
<td>chlorine trifluoride</td>
<td>ClF₃</td>
<td>titanium tetrachloride</td>
<td>TiCl₄</td>
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<td>ethylene oxide</td>
<td>C₂H₄O</td>
<td>tungsten hexafluoride</td>
<td>WF₆</td>
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<td>vinyl fluoride</td>
<td>C₂H₃F</td>
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<td>Ga(CH₃)₃</td>
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<td>nitrous oxide</td>
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<td>trimethyl indium</td>
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<tr>
<td>nitrogen trifluoride</td>
<td>NF₃</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Summary

- NIST has an extensive set of programs in support of the semiconductor industry
- These programs address issues across all the important technological areas needed by the industry
- NIST collaborates intensely with ISMT, companies, and universities
- More information can be obtained by calling us (301-975-4400) or going to www.nist.gov or www.eeel.nist.gov/omp