3D Interferometry and Nanomechanics, Electronics, N95 Face Masks and More

Kurt Rubin

Acknowledgements
Oskar Amster, Kelly Barry, Bernhard Botters, Menno Bouman, Nannan Ehmke, Jim Elman, Ben Garland, Randy Geels, Warren Jolley, Professor Grigoris Kaltsas, Yujie Meng, Jeff Reichert, Rayner Schelwald, Rae Zeng

2/16/21
Characterization Needs for Devices and Materials are Diverse

Labs

- **Stylus Profiler**
  - Alpha-Step®, Tencor™

- **Optical**
  - Zeta
  - Profilm3D®

- **Thin Film**
  - Filmetrics®

- **Nanomechanics**
  - Nano Indenter®

- **Electrical**
  - R50 Sheet Resistance
  - Eddy Current, 4-point probe

Fabs

- **HRP®**
  - Stylus-based
  - Automated High Resolution Profilers

- **Candela®**
  - Optical-based
  - Defect Inspection Systems

**Technology Scaling**
- Connect R&D ↔ Manufacturing
- Measurement technology in Lab tools scales to automated Fab tools
Flexible Electronics

- Characterizing 3D topography using optical interferometry and color
### Flexible & Printed Electronics

#### 2020 Organic and Printed Electronics Applications Roadmap

<table>
<thead>
<tr>
<th>Year</th>
<th>Display</th>
<th>Solar</th>
<th>Device</th>
<th>System</th>
</tr>
</thead>
</table>
| 2020       | Foldable displays for phones, reflective PFD                            | OPV objects; portable chargers, OPV-K210 products                     | Printed devices: memory, RFID antennas, primary battery, active backplane, passive electric elements, Sensors: glass, moisture, temperature, humidity, printed phone case integrated antenna, thin flexible Si chips | Integrated smart systems: Smart label sensors (humidity, temperature), Sensors for blood analysis, NFC labels, Hybrid systems (printed components + flexible ICs, HAMs (sensors)) |}

**Attributes**
- Very low production cost
- Enable new applications
- Synergistic with conventional IC

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*A New Frontier of Printed Electronics: Flexible Hybrid Electronics, Adv. Mater. 2020, 32, 1905279*
Inkjet-Printed Flexible Thermogenerators

Composite mixture to tune properties

1. PEDOT:PSS (conducting polymer mixture)
2. Carbon-Quantum-Dot (nanoparticle)

Carbon Quantum Dots (CQD)

Inkjet Print

Thermoelectric Response

Optical Interferometry + True Color Imaging

- **History**
  - 1717 - Newton analyzes interference between surfaces
  - 1919 - Michaelson-Morley creates interferometer
  - Past century - nano to astronomy (LIGO) innovations

- **3D optical interferometry**
  - Precisely measure topography
    - White-light (WLI), phase-shifting (PSI) and composite (WLI + PSI) interferometry
      ⇒ Ångstrom to mm feature heights

- **Topography + Color**
  - Complementary information about geometry and material
  - Understand interplay: topography, geometry, fabrication, materials, and performance

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3D Optical Interferometry with True Color Visualization Advances Understanding of Flexible Electronics

Kurt A. Rubin, Rayner Schelwald, Dimitris Barmpakos, Apostolos Segkos, Christos Tsamis, and Grigoris Kaltsas

*Laser Focus World, Volume 56, Issue 09, Sept. 2020, pp. 19-22*
3D Interferometric Optical Profiler

- Interference of light reflected from sample and reference mirror
- Topography-dependent interference patterns converted to 3D topographic data

- White-light interferometry (WLI)
- Phase-shift interferometry (PSI)
- Composite-interferometry (WLI + PSI)
- Total Focus™ = Topography + Color
- Stitching
Prototype Printed Flexible Electronic Devices

- Built entirely by multilayer inkjet printing

- Electrical contacts
  - Sintered 100nm Ag nanoparticles
  - 80μm-diameter drops

- Conductor traces
  - PEDOT:PSS + Carbon Quantum Dots
    - Organic PEDOT:PSS
      - Class of electrically conducting polymers
    - Carbon Quantum Dots (CQDs)
      - Enhance tunability of electrical conductivity and optical response of PEDOT:PSS
Two-pass Inkjet Conductor Trace and Electrical Contact

- White light interferometry (WLI) to measure heights
- Multiple FOV stitched together form a single 3D dataset (topographic + color)
- Color enables differentiating materials

<table>
<thead>
<tr>
<th>Feature</th>
<th>Roughness [µm]</th>
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</thead>
<tbody>
<tr>
<td>Overlap</td>
<td>0.20</td>
</tr>
<tr>
<td>Ag Contact</td>
<td>0.73</td>
</tr>
</tbody>
</table>

![Graph showing height, distance, and overlap](image)
Inkjet Conductor Trace

- Position-dependent color variation of PEDOT:PSS + CQD from thickness variation
  - Optical interference between semitransparent PEDOT:PSS layer and substrate
- Composite WLI + PSI interferometry
  - Higher vertical resolution than WLI (~10x)
  - Good for characterizing smoother surfaces (e.g. polyamide)

<table>
<thead>
<tr>
<th>Feature</th>
<th>Conductor Thickness [µm]</th>
<th>ISO 25178 Roughness Sq [µm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Substrate (polyimide)</td>
<td>N/A</td>
<td>0.028</td>
</tr>
<tr>
<td>1 pass 0wt% CQD</td>
<td>0.317</td>
<td>0.058</td>
</tr>
<tr>
<td>2 pass 50wt% CQD</td>
<td>0.970</td>
<td>0.158</td>
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</table>
“Flexible Electronics” is a Steadily Growing Cross-Disciplinary Field

Flexible Electronics Publications

~2 decades exponential growth

Source: Dimensions

Breakdown of 2020 Publications

<table>
<thead>
<tr>
<th>Category</th>
<th># Publications</th>
<th>#Citations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Engineering</td>
<td>5,610</td>
<td>13,944</td>
</tr>
<tr>
<td>Materials Engineering</td>
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<td>11,491</td>
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<tr>
<td>Chemical Sciences</td>
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<td>7,162</td>
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<tr>
<td>Physical Chemistry</td>
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<td>5,161</td>
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<tr>
<td>Macromolecular and Materials Chemistry</td>
<td>785</td>
<td>1,252</td>
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<tr>
<td>Electrical and Electronic Engineering</td>
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<td>1,640</td>
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<tr>
<td>Technology</td>
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<tr>
<td>Physical Sciences</td>
<td>577</td>
<td>813</td>
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<tr>
<td>Biomedical Engineering</td>
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<td>1,088</td>
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<tr>
<td>Nanotechnology</td>
<td>285</td>
<td>846</td>
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<tr>
<td>Information and Computing Sciences</td>
<td>249</td>
<td>436</td>
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<tr>
<td>Inorganic Chemistry</td>
<td>224</td>
<td>798</td>
</tr>
<tr>
<td>...</td>
<td>...</td>
<td>...</td>
</tr>
</tbody>
</table>
Optical Characterization of Thin Films

- Wavelength-dependent Optical Reflectance Spectroscopy
- Polymer, silicon, carbon, 2D materials
Thickness Dynamics of Polymer Films Floating on a Liquid Surface

- Accurate glass transition + diffusion kinetics measurements
  - Floating film on liquid minimize substrate-polymer interactions and mechanical constraints

- Spectral reflectance measurement
  - Short time measurement (<< 1 sec) ⇒ reduced sensitivity to thermally-induced film motion
  - Correct material and physics optical model + wide spectral range (200 < λ <1100 nm) ⇒ accurate film thickness

![Diagram of Polystyrene (PS) film and measurement system](image)

<table>
<thead>
<tr>
<th>Time Region</th>
<th>Temperature Region</th>
</tr>
</thead>
<tbody>
<tr>
<td>8nm</td>
<td>37nm</td>
</tr>
<tr>
<td>68nm</td>
<td>100nm</td>
</tr>
</tbody>
</table>
| PS polymer film thickens and narrows above $T_{\text{glass}}$ due to diffusion

Lu et al, Macromolecules 2009, 42, 9111–9117
Measure Thickness of Wrinkled Nanoporous Silicon Membrane

- Nanoporous membrane for separating small molecules
- Membrane thickness affects device performance and yield
- Spectroscopic ellipsometry proved difficult to extend to this type of sample – required a witness sample

Solution: Spectral reflectance measurement + model fit

- Nanoporous silicon membrane after removal from substrate
- Sample from Simpore
- Filmetrics® F40-UV film thickness measurement system
- Well-defined small measurement size (<40mm)
- Fit model to measured wavelength-dependent spectra
- Determine thickness and Real and Imaginary index of refraction
Measure Thickness of Ultra-Thin Carbon Films

Reflectance Spectroscopy is Applicable Over a Wide Thickness Range

Thickness Measurement Rule of Thumb
• Thicker material \(\Rightarrow\) measure with larger \(\lambda\) and higher spectral resolution
• Thinner material \(\Rightarrow\) measure with smaller \(\lambda\)

Measured spectra
Model Fit
33 x 33\(\mu\)m measurement area (tunable)
Optical Micro-Spectroscopy for Characterizing Ultra-Thin Films

- Two dimensional (2D) materials
  - Consist of single or small number of atomic layers
- Properties differ from 3D due to reduced dimensionality
  - Layer-dependent optical band gap
- Potential applications
  - Nanoelectronics, sensing, spintronics, optoelectronics, Qubits, …
- Examples
  - Graphene, hexagonal boron nitride, layered transition metal dichalcogenides, …
- Reflectance spectroscopy
  - Detect single atomic layers
  - Obtain material-unique fingerprints
  - Quantify number of layers in Van-der-Waals heterostructures

- >2000 2D materials have been discovered
- Stack layers of different materials to tune optical, electrical, and mechanical properties
Optical Microspectroscopy

Characterize 2D materials and heterostructures deposited via exfoliation, CVD, and laser direct writing

Characterize 2D Materials with Optical Microspectroscopy

Large area Optical Image

Spectral Reflectance Wavelength and Amplitude Calculation Matches to Measurement

Graphene (Gr) + hexagonal Boron Nitride (hBN) heterostructures on SiO₂

N95 Mask

- Topography characterization by 3D optical profiling
- Nanoindentation characterization of microfiber mechanical properties
Cross-section of Coronavirus (colorized)

Research Background & Motivation

Serious PPE shortages worldwide

Serious concerns about N95 decontamination & reuse
## Mask vs. Respirator

<table>
<thead>
<tr>
<th>Masks (Surgical)</th>
<th>Respirator (N95)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Loose fitting, Covering the nose and mouth</td>
<td>Tight fitting, Creating facial seal</td>
</tr>
<tr>
<td>One way protection</td>
<td>Two way protection</td>
</tr>
<tr>
<td>No safety rating assigned</td>
<td>Specifications and standard</td>
</tr>
<tr>
<td>80% particles blocked</td>
<td>95% + particles blocked</td>
</tr>
<tr>
<td>Three layer design (Melt-blown material)</td>
<td>Three layer design (Melt-blown material)</td>
</tr>
</tbody>
</table>

![Mask image](image1)

![Respirator image](image2)

https://fastlifehacks.com/n95-vs-ffp/
N95 RRFs Decontamination Recommendations by FDA & CDC

Vaporous Hydrogen Peroxide

Moist Heat

Today’s Discussion

UVGI (ultraviolet germicidal irradiation)
Meltblown Microfibers + Corona Electrostatic Charging $\implies$ N95 Respirator

Meltblowing process

Corona Electrostatic Charging

Charging N95 fibers Invented by Peter Tsai

Courtesy of Prof. Peter Tsai
UVGI Sterilization

- Uses ultraviolet C (short-range ultraviolet, $\lambda = 100$-280nm) light
- High doses of UV-C lights required to sterilize
- UV radiation can degrade polymers
- Some damage to N95 observed at high UV-C doses

UVC dose $\propto \frac{\text{UV bulb power} \times \text{Exposure time}}{4 \times \pi \times \text{UV bulb distance}^2}$
Measurement of Polypropylene Microfiber Geometries

- Fibers have very rich topography
- Prefer non-contact measurement technique to minimize disturbing fibers

Measurement Approach
- 3D topographic data
  - CGSI (Confocal Grid Structured Illumination)
- No surface charging effect on samples
- Fast measurement

Zeta-20 Optical Profiler
Topography of Meltblown fibers after UV exposure
N95 Respirator Meltblown Layer Topography versus UV Dose

Dose = 0 J/cm²

Dose = 1 J/cm²

Dose = 7 J/cm²

Dose = 13 J/cm²

Dose = 19 J/cm²

Dose = 31 J/cm²
N95 Microfiber Width Analysis at Different UVC Dosages

Fiber transition after 0 min UV-C exposure

Fiber transition after 155 min UV-C exposure

38W 254nm UV-C

Average Fiber Width (µm)

Y = -6.1ln(x) + 14
R² = 0.95

UVC Dose (J/cm²)
Measure Mechanical Properties at Small Scale by Nanoindentation

1. **Indent**
   - Load/Unload

2. **Measure during indentation**
   1. Load applied
   2. Depth tip penetrates
   3. Stiffness

3. **Extract material’s mechanical properties**
   1. Hardness (H)
   2. Modulus (E)

**Continuous stiffness measurement**
- Oscillate probe during indentation
- Measures hardness and modulus as a function of depth or load

W.C. Oliver, G.M. Pharr J. Mater. Res. 7 (1992)

- Nano Indenter® G200X
- Diamond Berkovich Indenter
- NanoVision scanning probe microscope
Meltblown Microfiber Response to Nanoindentation

Typical nanoindentation load and displacement curves

(a)

(b)
Fiber Mechanical Property Changes After Ultraviolet Light Exposure

Each UV exposure dose started with fresh polypropylene microfibers.
Nanoindentation Response of Single Melt-Blown Non-Woven Fiber

Fiber protrudes ~600nm high above epoxy

Before Nanoindentation

After Nanoindentation

Inelastic response
Residual indentation after Load / Unload cycle

16 μm X 16 μm scanned area

Load (mN)

0.1 mN

0.3 mN

0.5 mN

Depth (nm)

0.1 mN

0.3 mN

0.5 mN

Residual Indent Depth [nm]

-57

-107

-147

Distance Along Fiber [μm]
Effect of UV Irradiation on N95 Mask Fibers

- Characterization of N95 masks at the individual fiber level enables understanding of process-induced changes
  - Nanoindentation quantifies mechanical response
  - 3D Optical profiling via structured illumination quantifies geometric response

- Over-exposure to UVC radiation during decontamination processes causes loss of strength of the polypropylene microfiber layer
  - Young’s modulus, E, and hardness H both decrease with increase of UVC dose
  - Average fiber width decreases as a function of exposure time
Turning Innovation into Products ⇒ Automated Optical Inspection

Candela

Defect Map

Zeta

Optical Mapping and Characterization of Defects

SiC TED

GaN Downfall

SiC TED

GaN Downfall
Characterization Needs for Devices and Materials are Diverse

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  - Defect Inspection Systems

Technology Scaling
- Connect R&D ↔ Manufacturing
- Measurement technology in Lab tools scales to automated Fab tools
Characterization techniques discussed today

▪ Wavelength-dependent micro-spectroscopy of films
▪ Interferometry, structured illumination, and color for determining 3D topography
▪ Nanoindentation to determine local mechanical properties

Thank you!

Questions?

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https://www.kla-tencor.com/products/instruments