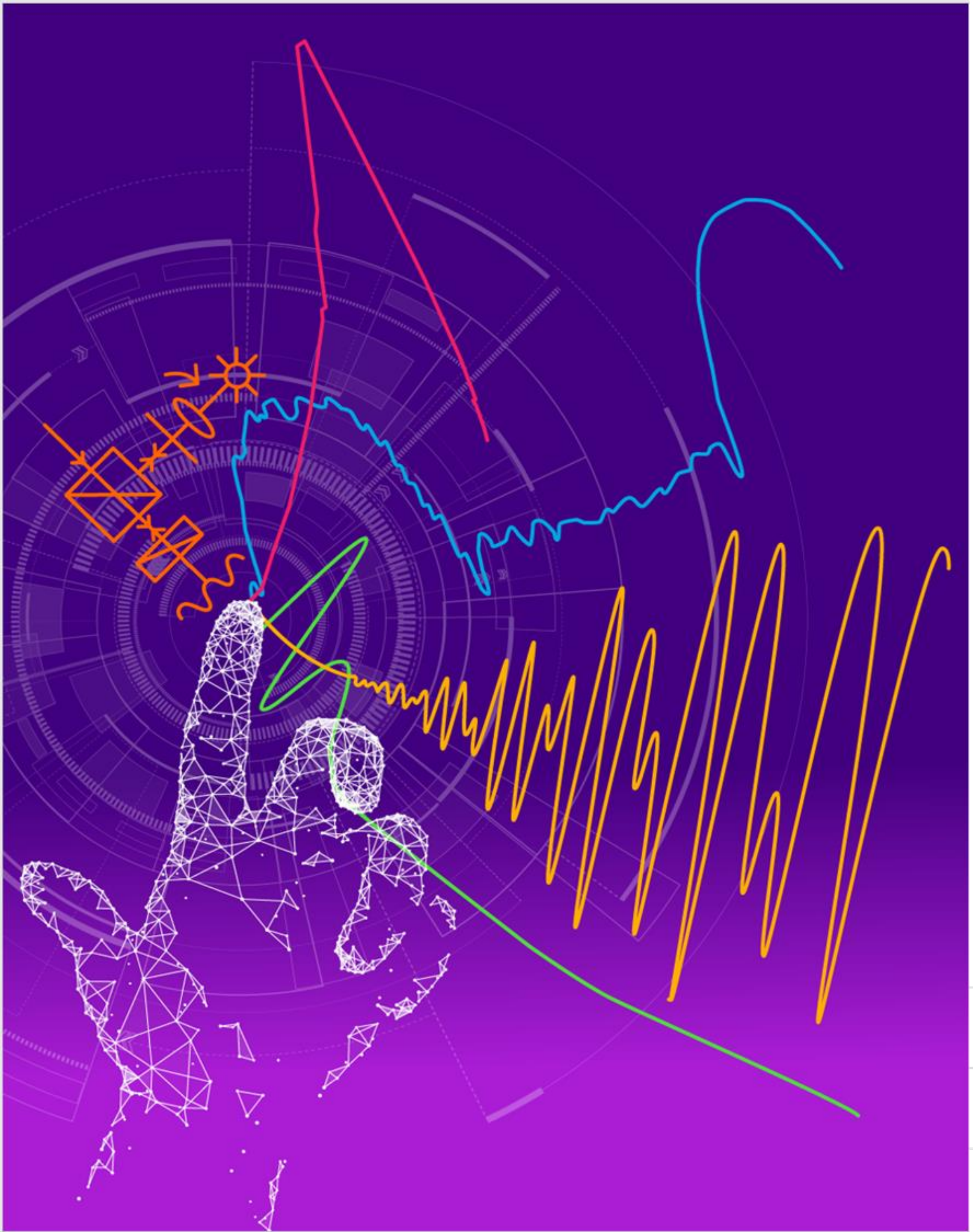


3D Interferometry and Nanomechanics, Electronics, N95 Face Masks and More

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

Techniques Illustrated in Today's Discussion

Stylus Profiler

Alpha-Step®, Tencor™

Optical

Zeta

Profilm3D®

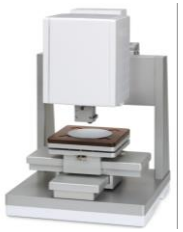
Thin Film

Filmetrics®

Nanomechanics

Nano Indenter®

Electrical



R50 Sheet Resistance
Eddy Current, 4-point probe

Fabs

HRP®

Stylus-based

Optical-based

Automated High Resolution Profilers

Candela®

Defect Inspection Systems

Technology Scaling

- Connect R&D ⇔ Manufacturing
- Measurement technology in Lab tools scales to automated Fab tools

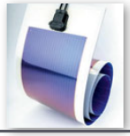
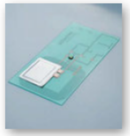
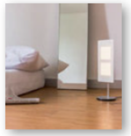
KLA INSTRUMENTS

Flexible Electronics

- Characterizing 3D topography using optical interferometry and color

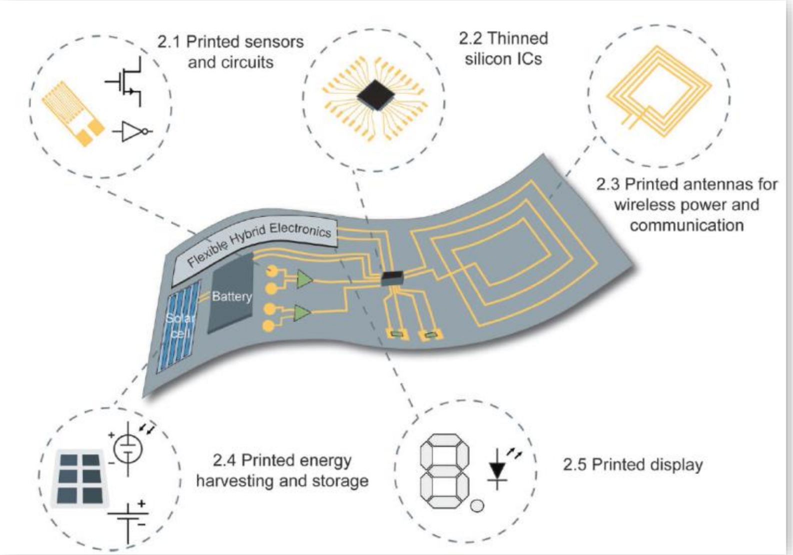
Flexible & Printed Electronics

2020 Organic and Printed Electronics Applications Roadmap

		2020	2021-23	2024-26	2027+	
Display		Foldable displays for phones; Reflective EPD	large flexible OLED-Displays; rollable TV; curved display for automotive interior	In-mold electronic (IME) Displays;	Flexible QD-Displays; flexible µLED-Displays	**Flexible & OLED Displays**
Solar	**OPV**	OPV objects; portable chargers; OPV-R2R products	Opaque OPV for BIPV; Large area OPV foil; OPV power supply	Semitransparent OPV for BIPV; OPV for autonomous sensors	Color and shape on demand; OPV on "all" surfaces (e.g. wallpaper, mobile devices) combined with thin film battery	
Device		Printed devices: memory, RFID antenna, primary battery, active backplane, piezoelectric elements; Sensors: glucose, pressure, temperature, humidity; printed phone case integrated antenna; thin flexible Si-chips	light sensor; stretchable conductors / resistors; 3D touch sensors; OTFT backplanes for low energy displays and OPD; 3D & large area flexible electronics; active touch sensors	Printed secondary ion battery; printed super caps; gesture sensors	Complex stretchable electronics; Printed complex logic;	**Electronics & Components**
System	**Integrated Smart Systems**	Smart label sensors (humidity, temperature); Sensors for blood analysis; NFC labels; Hybrid systems (printed components + flexible ICs); HMIs (sensors)	Ambient monitoring (e.g. humidity); sensors embedded in molded parts (automotive); on-skin human monitoring patches for sports; ambient intelligence (connected); Sleep disturbance monitoring;	On-skin human monitoring patches in clinical environment; Single article tagging (food)	Smart labels with geo localization; Breath analyzer for medical prevention	
Lighting		Flexible white OLED modules; rigid red OLEDs for automotive applications	Flexible red OLEDs (segmented) for automotive applications; transparent OLEDs; OLEDs for interior lighting of automotive	3D OLEDs; OLED signage; OLED for medical applications	OLED for aircraft and railway interior application	**OLED Lighting**

oe-a Roadmap Summary 8th Edition

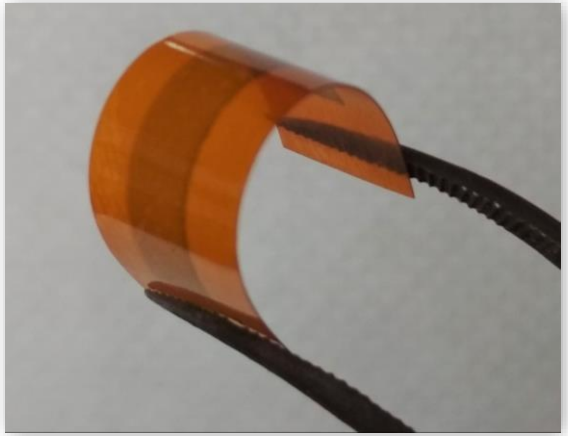
- Attributes
  - Very low production cost
  - Enable new applications
  - Synergistic with conventional IC



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

KLA INSTRUMENTS

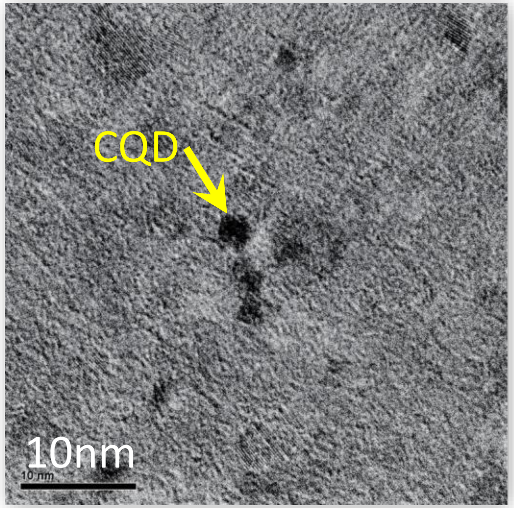
Inkjet-Printed Flexible Thermogenerators



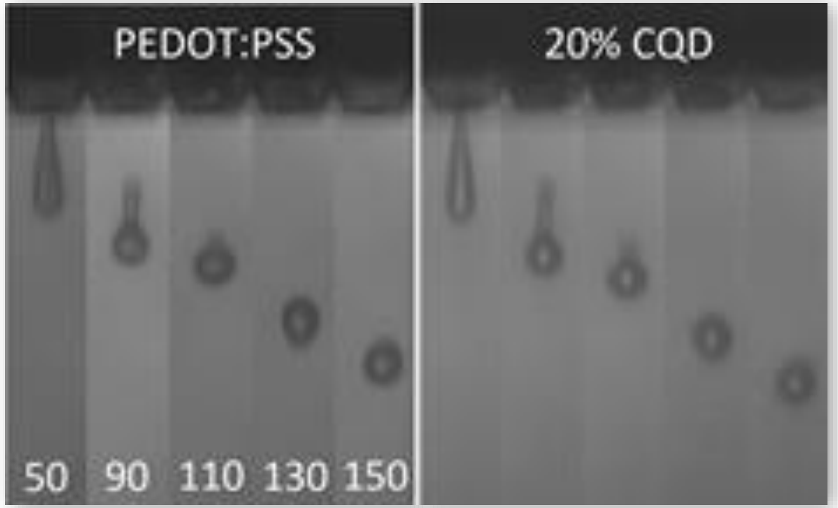
Composite mixture to tune properties

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

Carbon Quantum Dots (CQD)

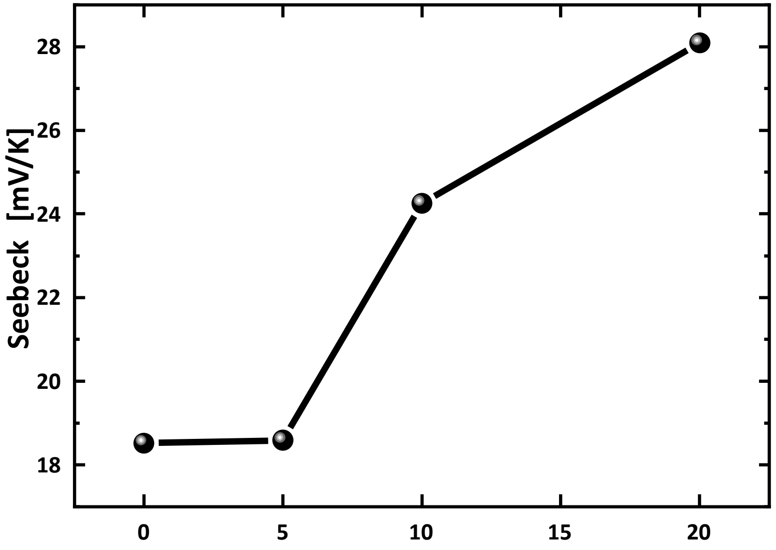


Inkjet Print



Time [μs]

Thermoelectric Response



Barmpakos, D., Segkos, A., Tsamis, C., & Kaltsas, G. (2019, June). Enhancement Of PEDOT: PSS Seebeck Coefficient Using Carbon-Quantum-Dot-Based Nanocomposite Materials: Application To Inkjet Printing On Flexible Substrate. In *2019 20th International Conference on Solid-State Sensors, Actuators and Microsystems & Eurosensors XXXIII (TRANSDUCERS & EUROSENSORS XXXIII)* (pp. 2515-2518). IEEE.

KLA INSTRUMENTS

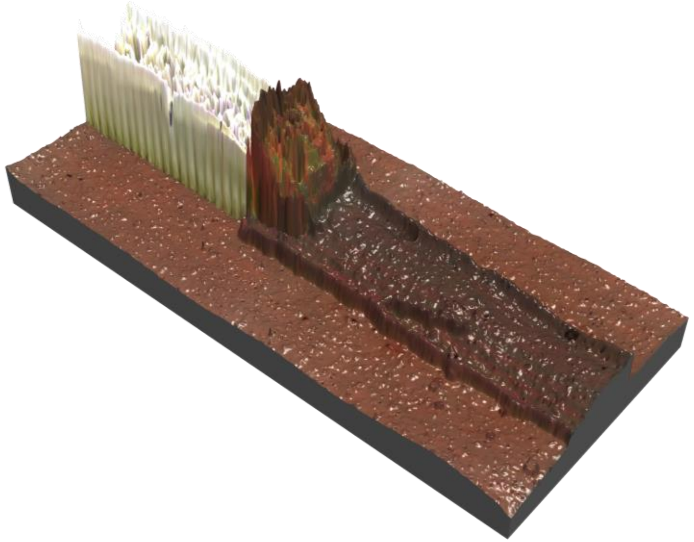
Optical Interferometry + True Color Imaging

- 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



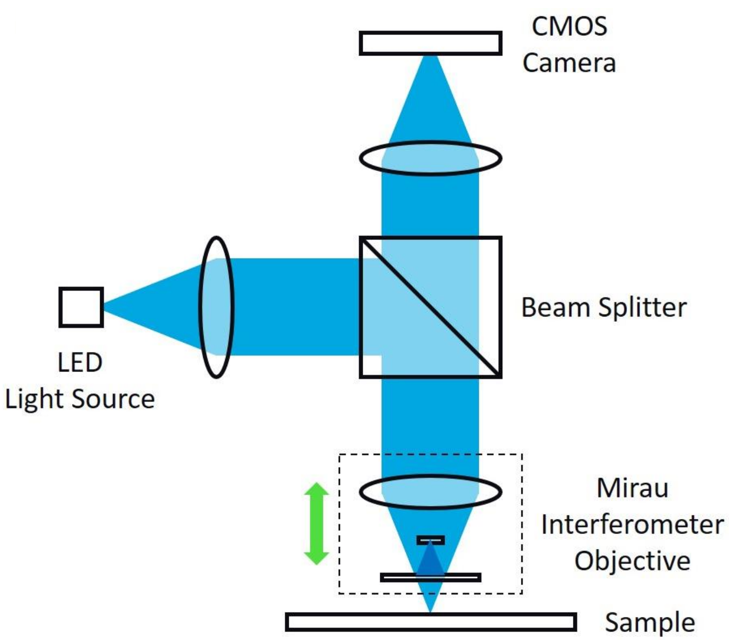
3D Optical Interferometry with True Color Visualization Advances Understanding of Flexible Electronics

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]

KLA INSTRUMENTS

3D Interferometric Optical Profiler



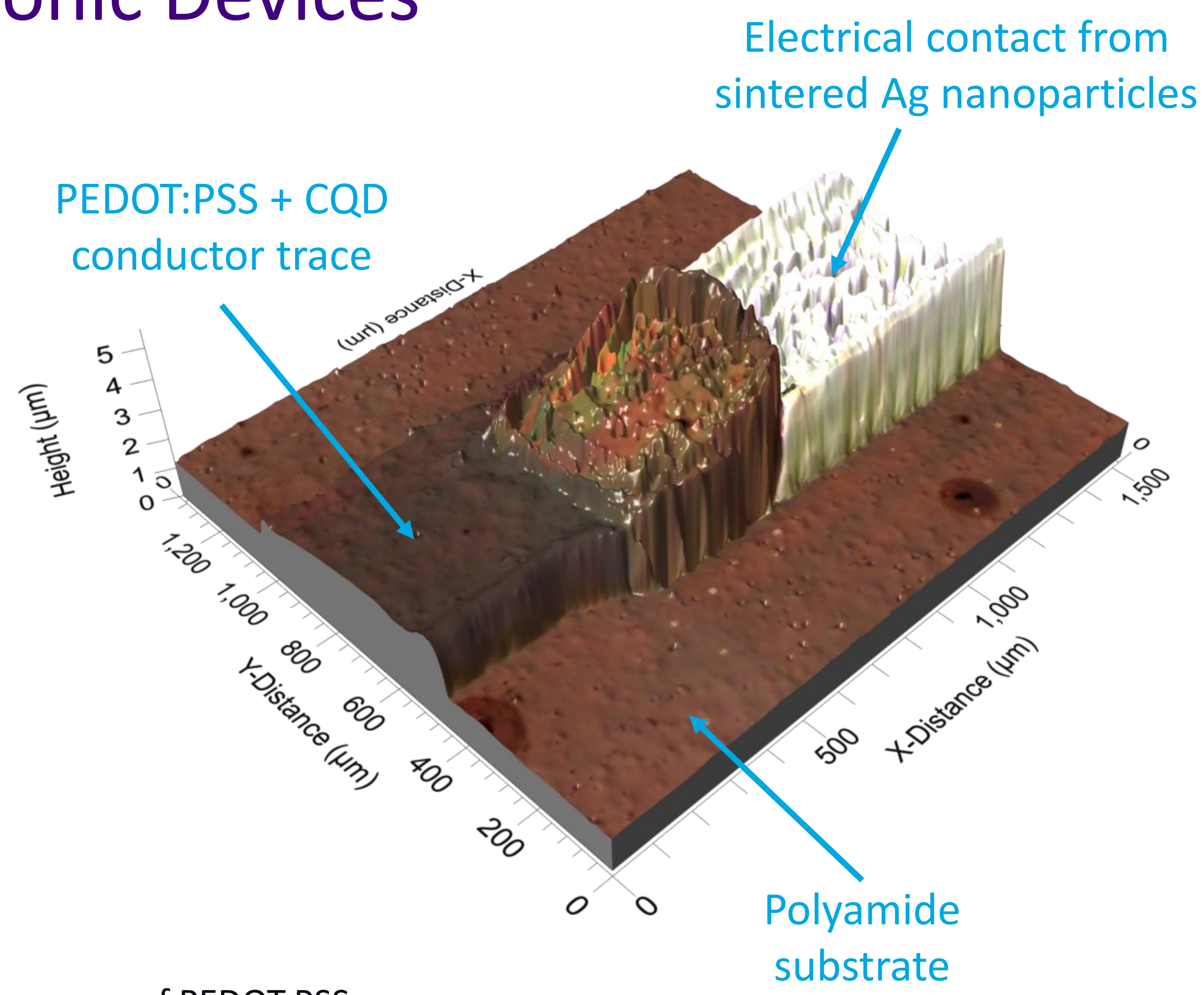
- White-light interferometry (WLI)
- Phase-shift interferometry (PSI)
- Composite-interferometry (WLI + PSI)
- Total Focus™ = Topography + Color
- Stitching

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

KLA INSTRUMENTS

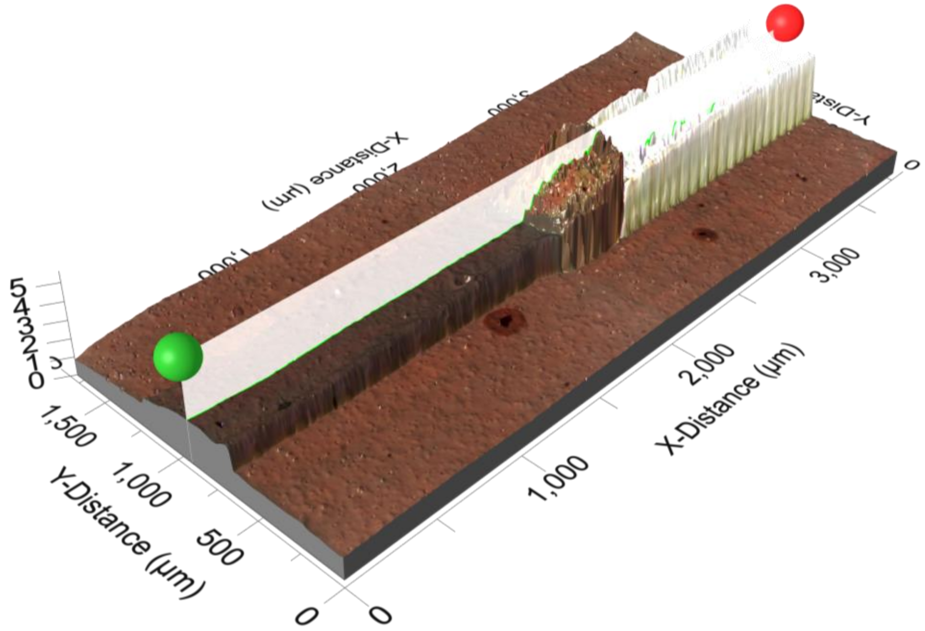
Prototype Printed Flexible Electronic Devices

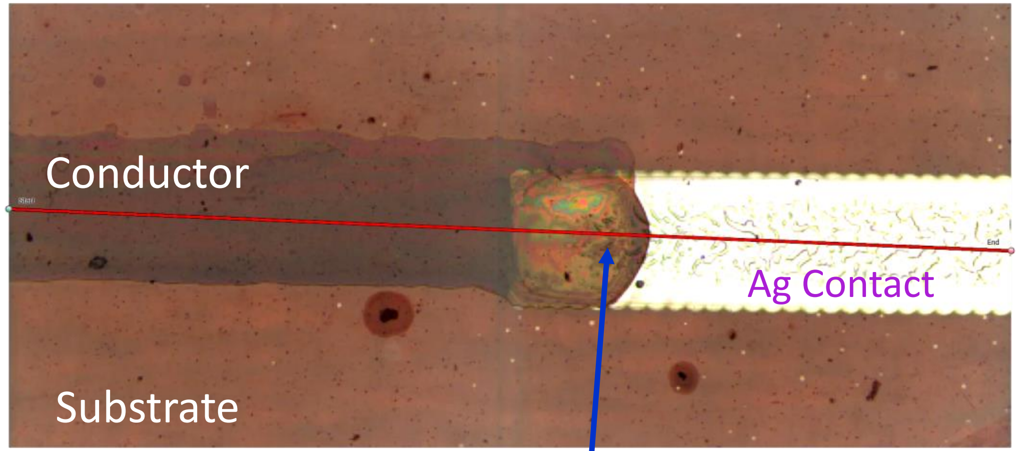
- Built entirely by multilayer inkjet printing
- Electrical contacts
  - Sintered 100nm Ag nanoparticles
  - 80µm-diameter drops
- Conductor traces
  - PEDOT:PSS + Carbon Quantum Dots
    - Organic PEDOT:PSS
      - Class of electrically conducting polymers
    - Carbon Quantum Dots (CQDs)
      - Enhance tunability of electrical conductivity and optical response of PEDOT:PSS

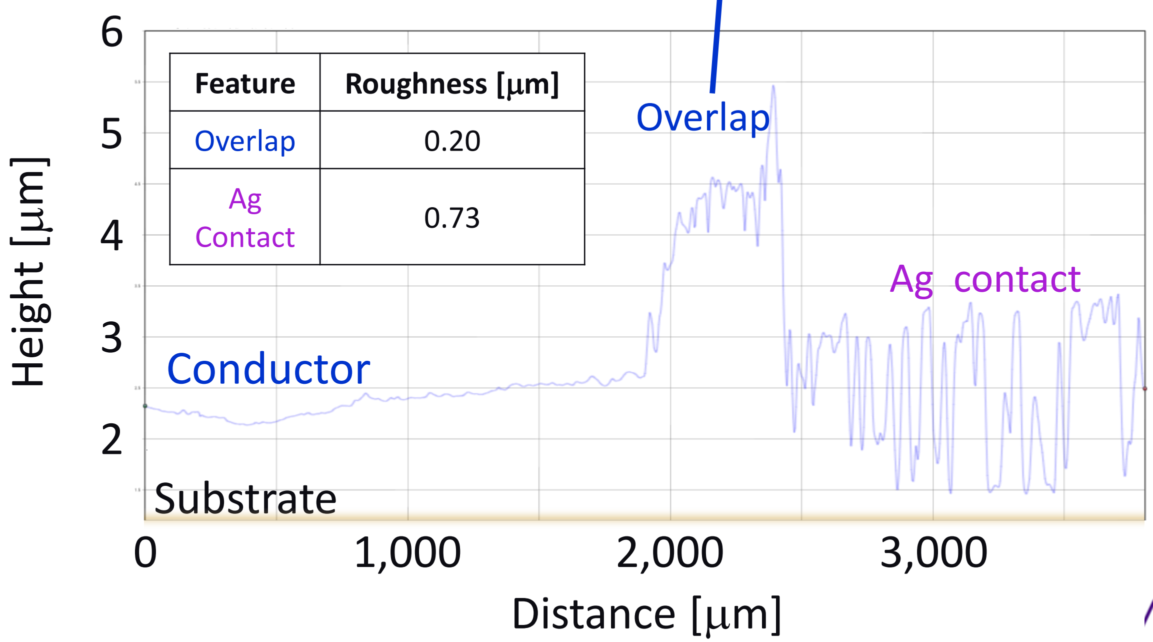


KLA INSTRUMENTS

Two-pass Inkjet Conductor Trace and Electrical Contact





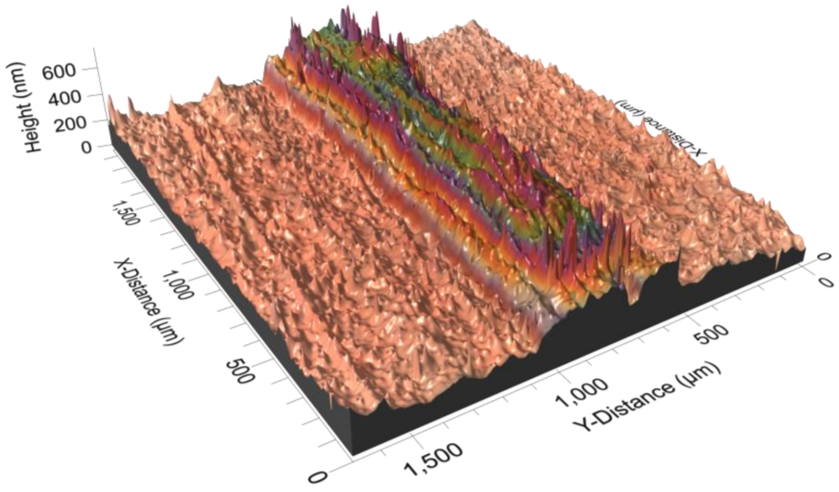


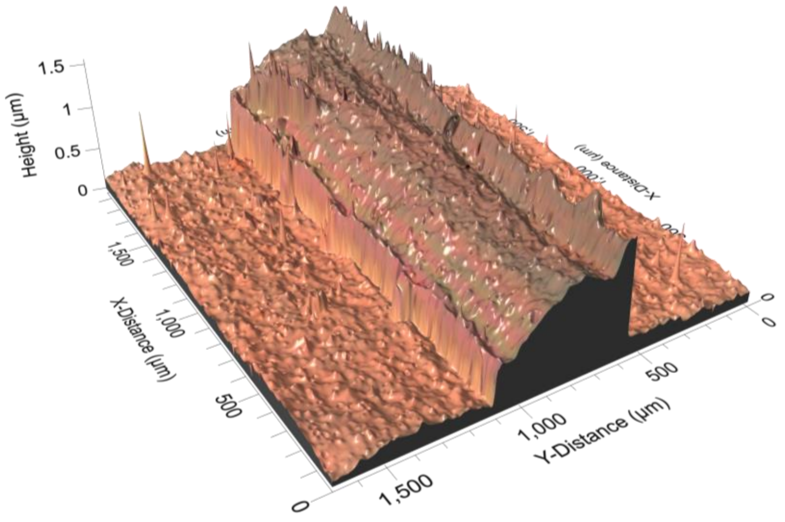
Feature	Roughness [µm]
Overlap	0.20
Ag Contact	0.73

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

INSTRUMENTS

Inkjet Conductor Trace



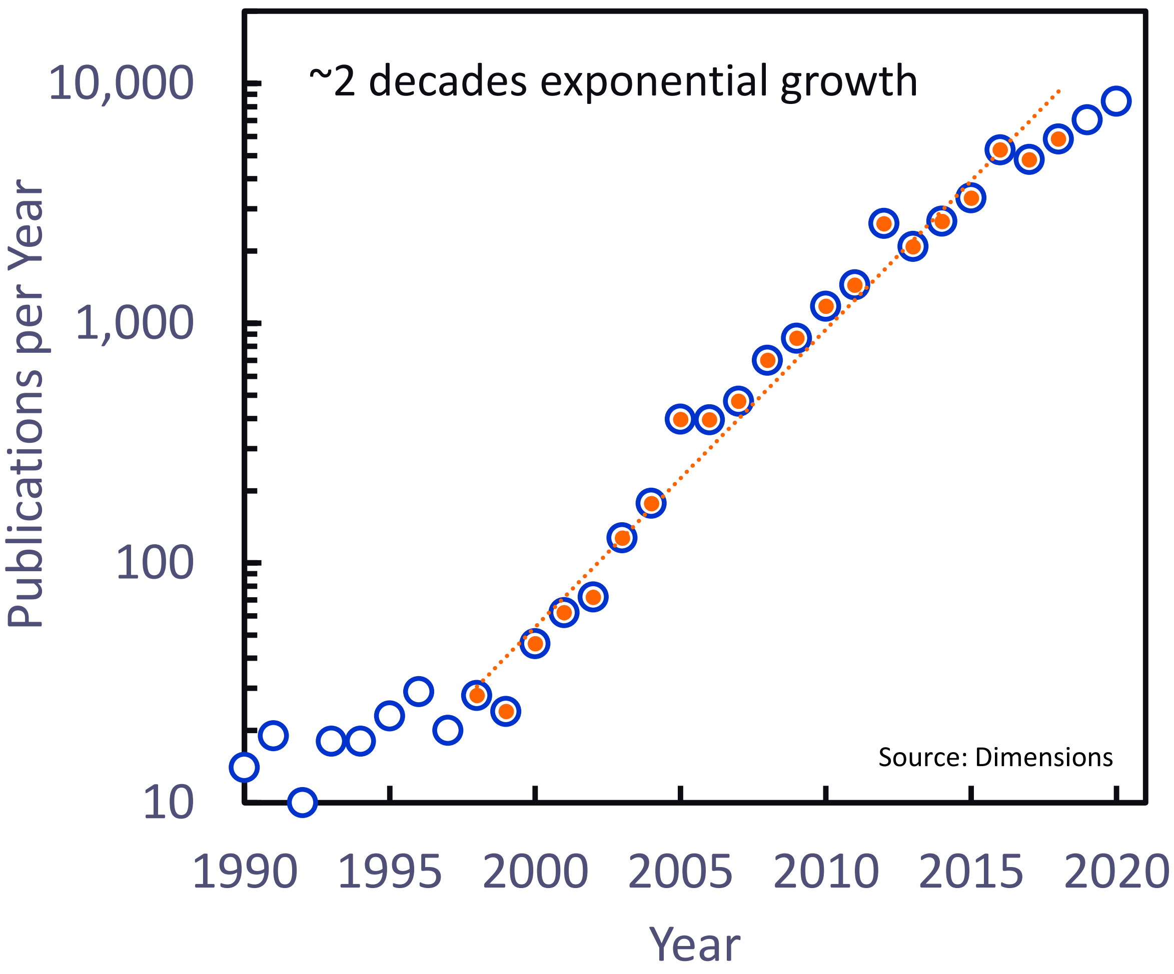


Feature	Conductor Thickness [µm]	ISO 25178 Roughness Sq [µm]
Substrate (polyimide)	N/A	0.028
1 pass 0wt% CQD	0.317	0.058
2 pass 50wt% CQD	0.970	0.158

- 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)

“Flexible Electronics” is a Steadily Growing Cross-Disciplinary Field

Flexible Electronics Publications



Breakdown of 2020 Publications

Category	# Publications	#Citations
Engineering	5,610	13,944
Materials Engineering	4,519	11, 491
Chemical Sciences	2,643	7,162
Physical Chemistry	1,816	5,161
Macromolecular and Materials Chemistry	785	1,252
Electrical and Electronic Engineering	728	1,640
Technology	620	1,151
Physical Sciences	577	813
Biomedical Engineering	411	1,088
Nanotechnology	285	846
Information and Computing Sciences	249	436
Inorganic Chemistry	224	798
…	…	…

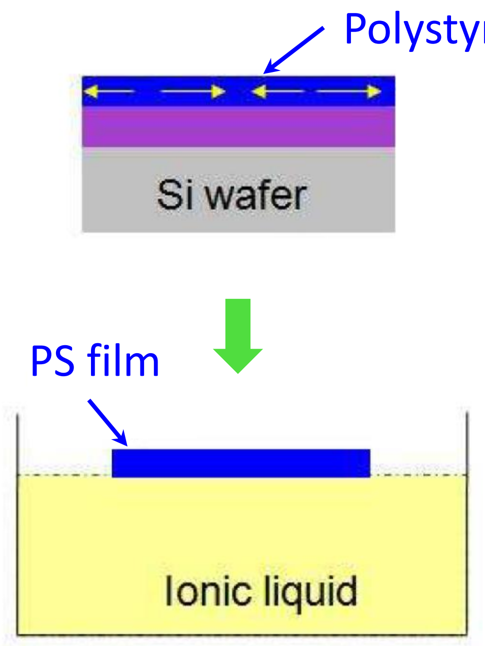
KLA INSTRUMENTS

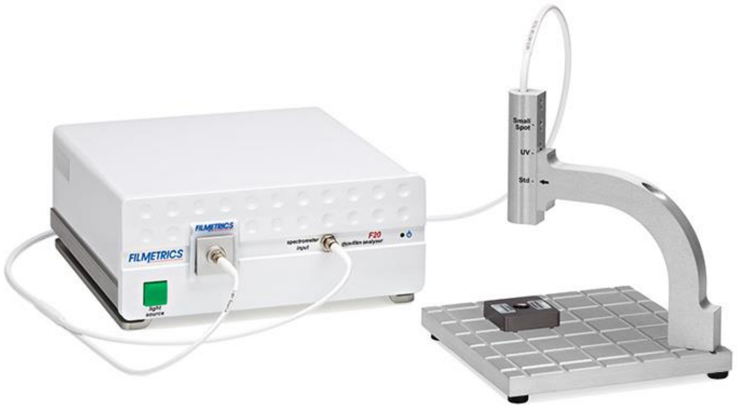
Optical Characterization of Thin Films

- Wavelength-dependent Optical Reflectance Spectroscopy
- Polymer, silicon, carbon, 2D materials

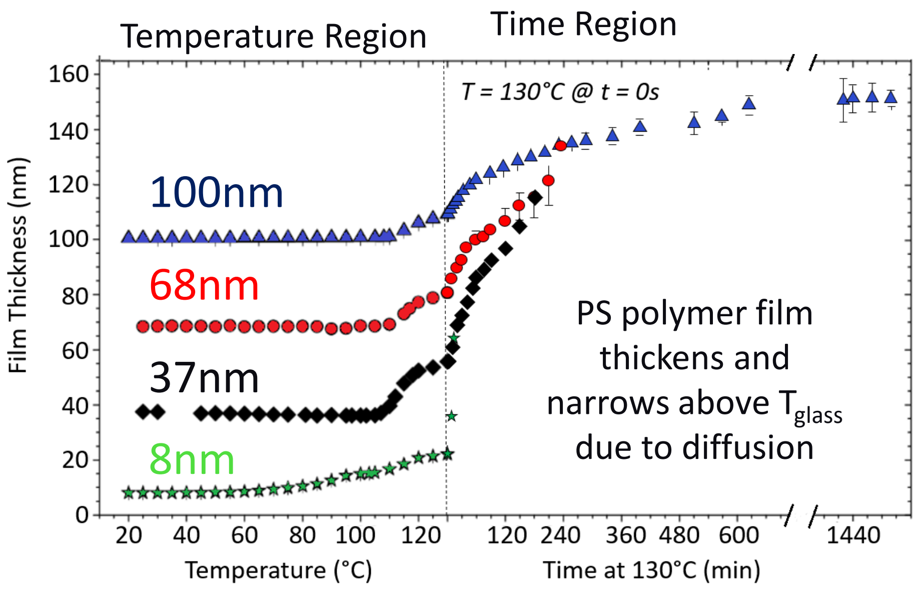
Thickness Dynamics of Polymer Films Floating on a Liquid Surface

- 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





Filmetrics® F20-UV
film thickness measurement system

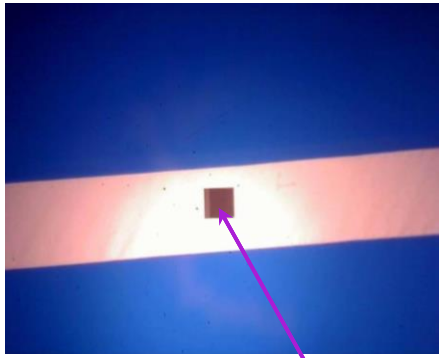


Lu et al, Macromolecules 2009, 42, 9111–9117

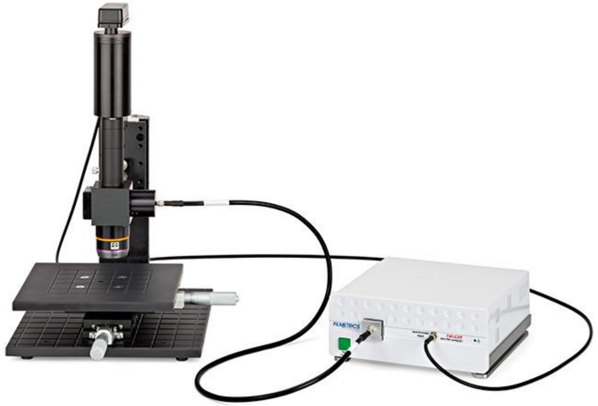
Measure Thickness of Wrinkled Nanoporous Silicon Membrane

- 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



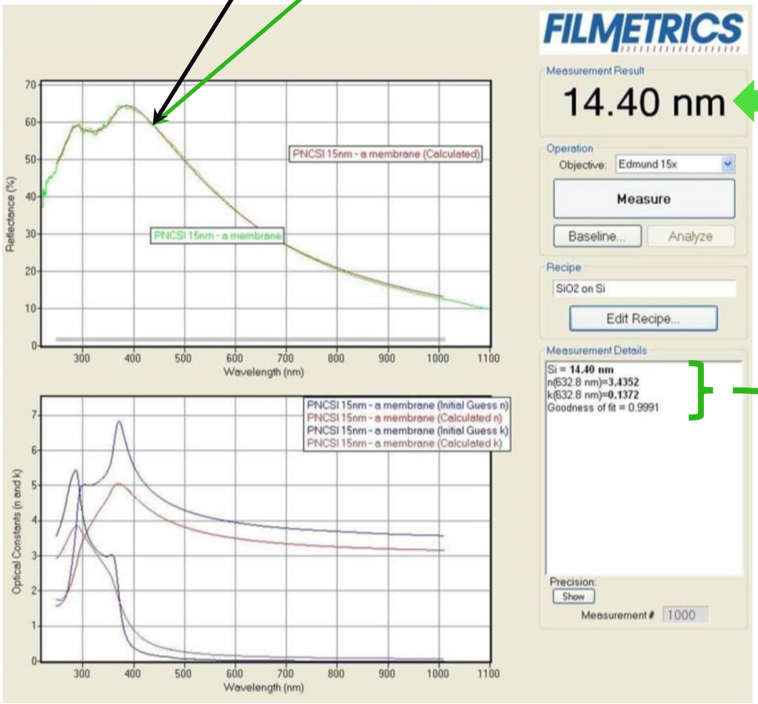
- Nanoporous silicon membrane after removal from substrate
- Sample from Simpore



- Filmetrics® F40-UV film thickness measurement system
- Well-defined small measurement size (<40mm)

Measured spectra

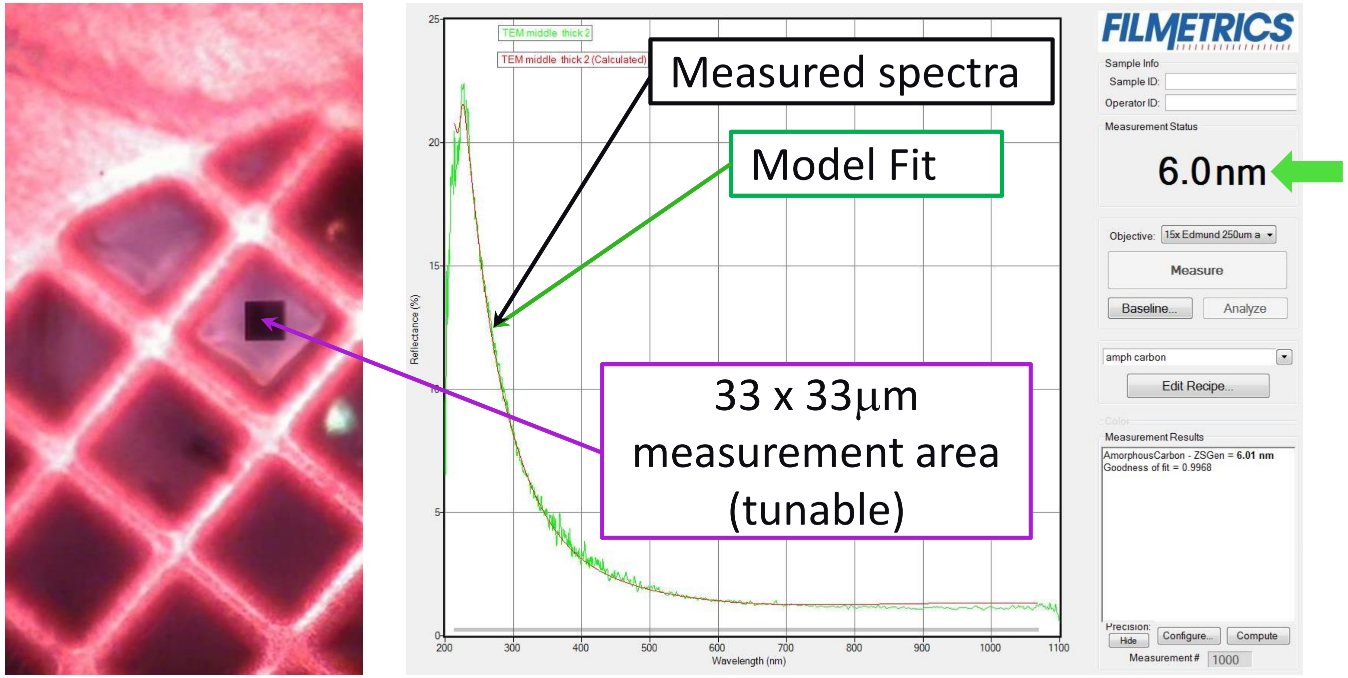
Model Fit



- Fit model to measured wavelength-dependent spectra
- Determine thickness and Real and Imaginary index of refraction

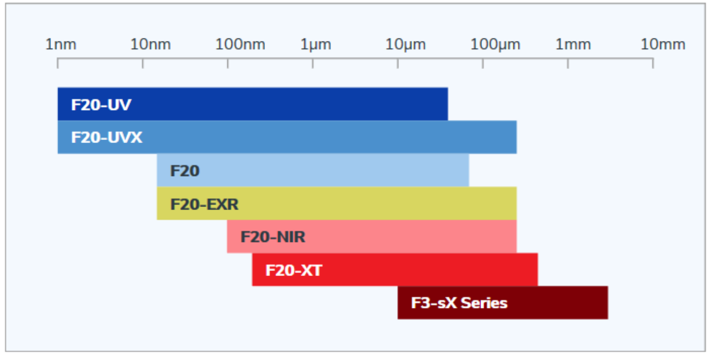
KLA INSTRUMENTS

Measure Thickness of Ultra-Thin Carbon Films



Reflectance Spectroscopy is Applicable Over a Wide Thickness Range

Thickness Range*



Model	Thickness Range*	Wavelength Range
F20	15nm - 70µm	380-1050nm
F20-EXR	15nm - 250µm	380-1700nm
F20-NIR	100nm - 250µm	950-1700nm
F20-UV	1nm - 40µm	190-1100nm
F20-UVX	1nm - 250µm	190-1700nm
F20-XT	0.2nm - 450µm	1440-1690nm
F3-sX Series	10µm- 3mm	960-1580nm

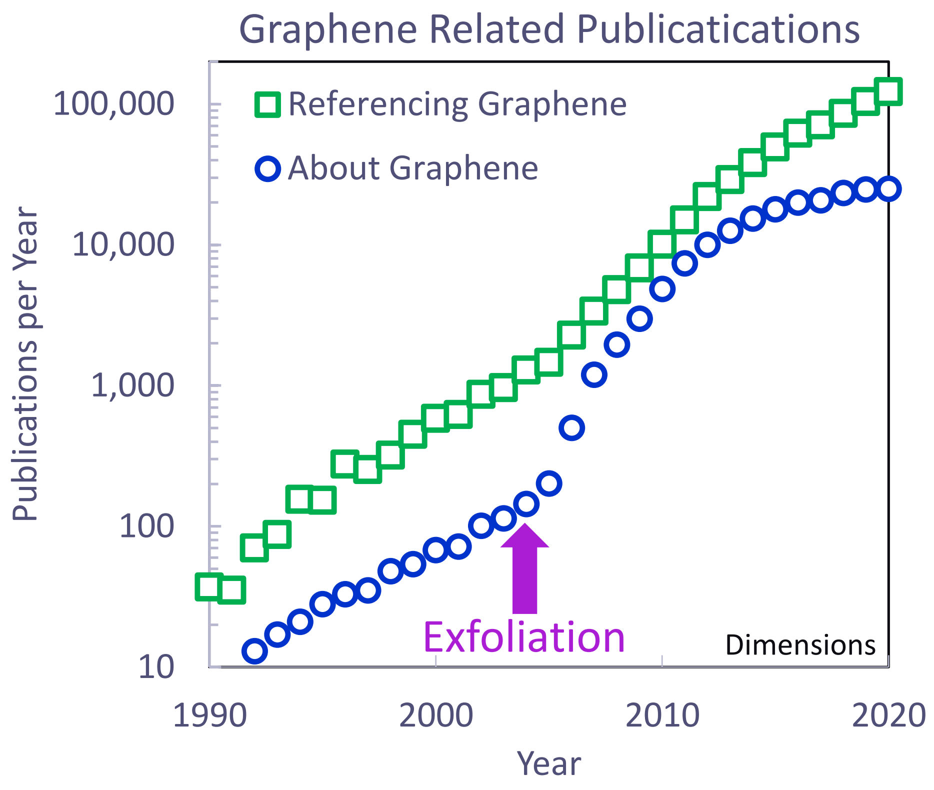
*film stack dependent

Thickness Measurement Rule of Thumb

- Thicker material $\Rightarrow$ measure with larger λ and higher spectral resolution
- Thinner material $\Rightarrow$ measure with smaller λ

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



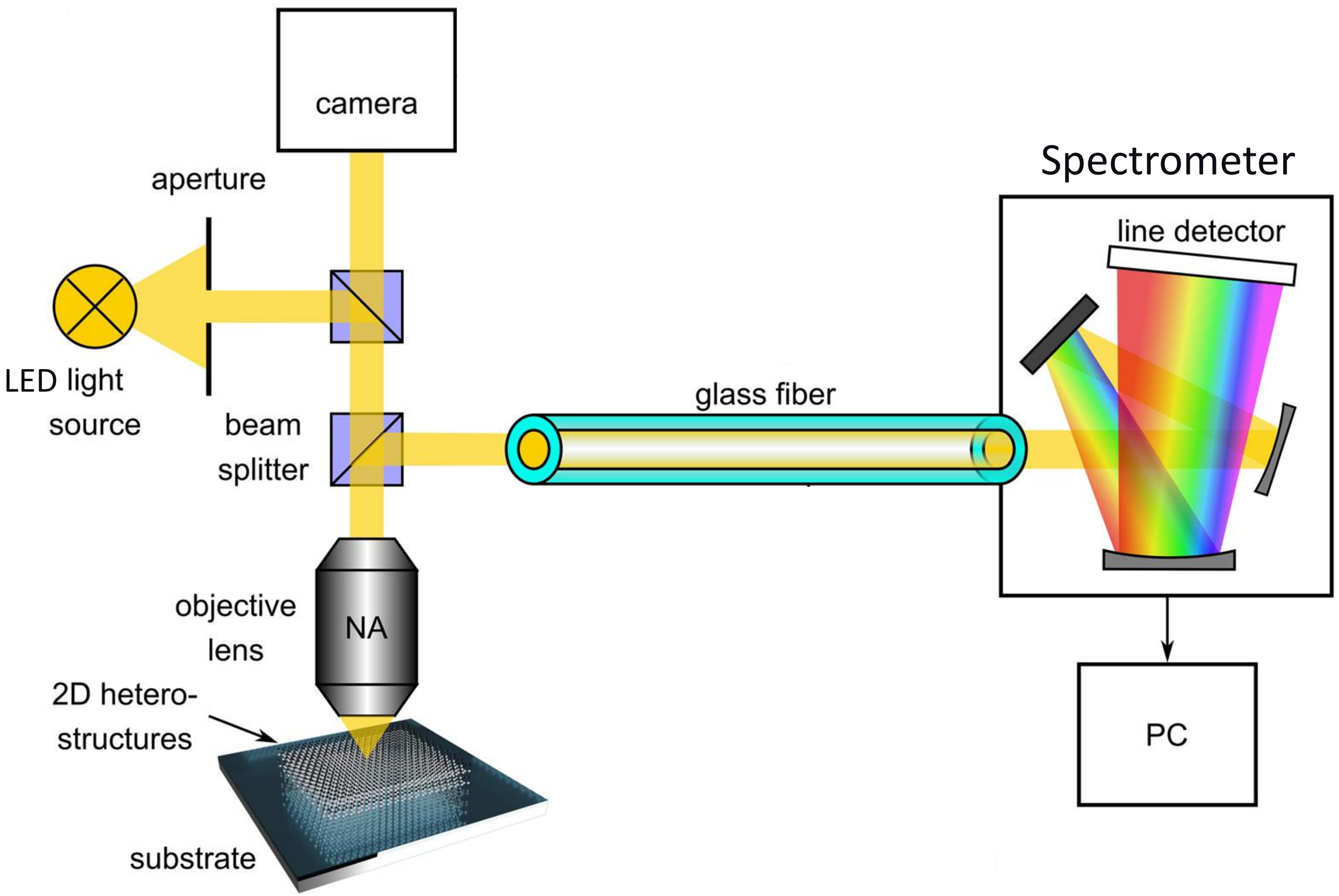
- >2000 2D materials have been discovered
- Stack layers of different materials to tune optical, electrical, and mechanical properties

Hutzler et al, Highly accurate determination of heterogeneously stacked Van-der-Waals materials by optical microspectroscopy. *Sci Rep* **10,** 13676 (2020)

KLA INSTRUMENTS

Optical Microspectroscopy

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



Hutzler et. al., Highly accurate determination of heterogeneously stacked Van-der-Waals materials by optical microspectroscopy. *Sci Rep* **10,** 13676 (2020)

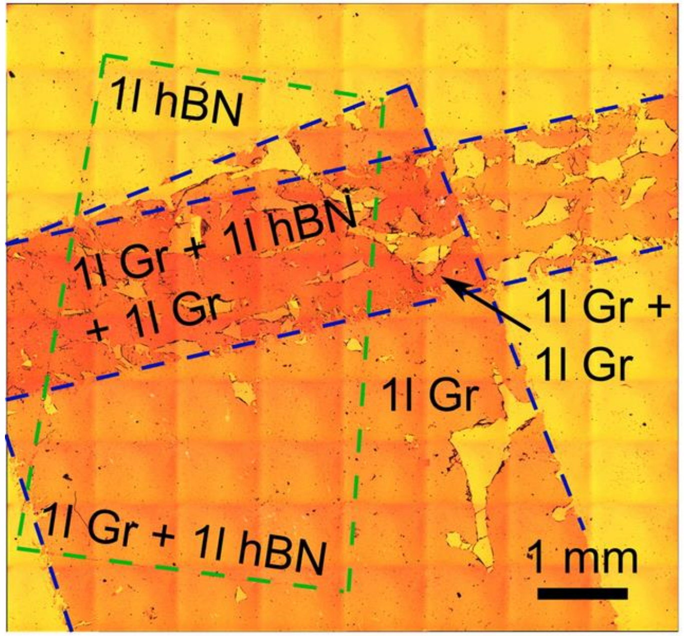
Zeta 300 3D optical profiler

- Film thickness measurement option
- 0.4 to 0.9 NA objectives

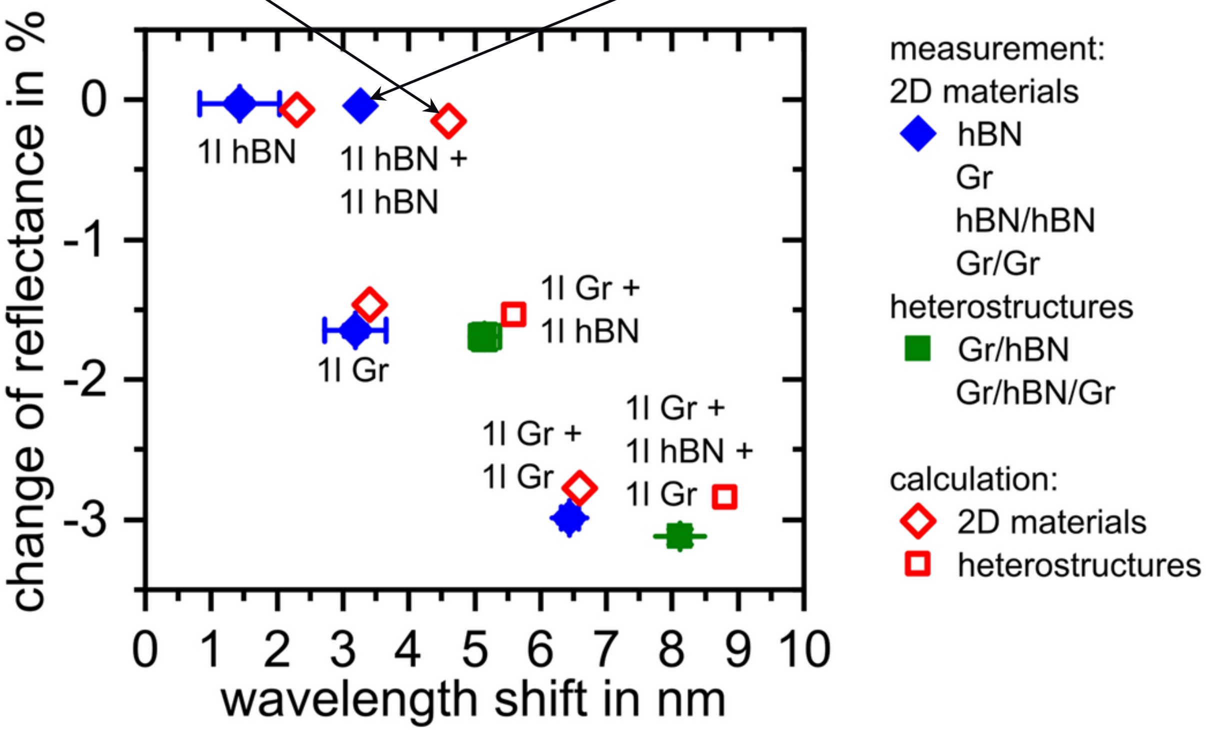
KLA INSTRUMENTS

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 SiO_2

Hutzler et al, Highly accurate determination of heterogeneously stacked Van-der-Waals materials by optical microspectroscopy. *Sci Rep* **10,** 13676 (2020)

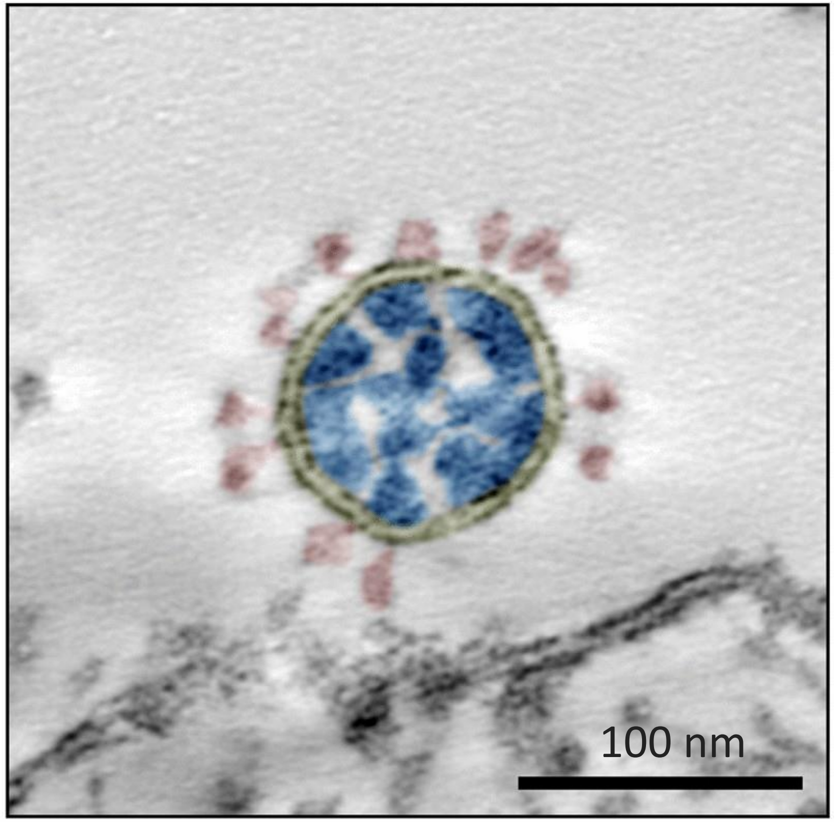
KLA INSTRUMENTS

N95 Mask

- Topography characterization by 3D optical profiling
- Nanoindentation characterization of microfiber mechanical properties

N95 Respirator Microfiber Response to Ultraviolet Exposure

Cross-section of Coronavirus (colorized)



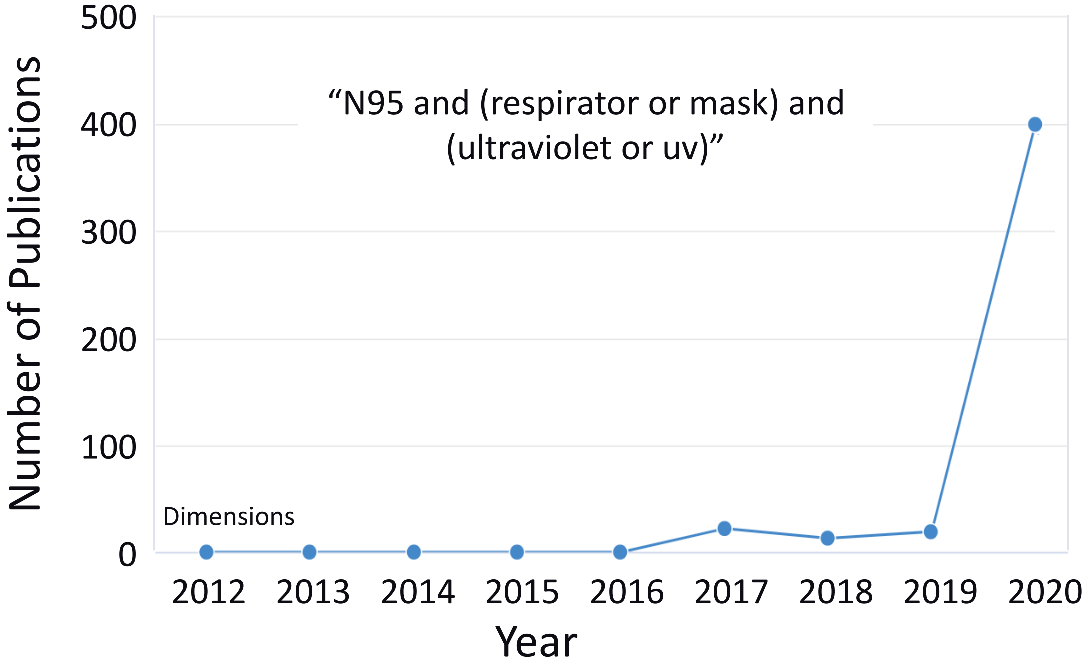
[Laue, M., Kauter, A., Hoffmann, T. et al. Morphometry of SARS-CoV and SARS-CoV-2 particles in ultrathin plastic sections of infected Vero cell cultures. Sci Rep 11, 3515 (2021)]

MRS Advances
MRS MATERIALS RESEARCH SOCIETY

Effect of Ultraviolet C Disinfection Treatment on the Nanomechanical and Topographic Properties of N95 Respirator Filtration Microfibers

Yujie Meng [a1], Rae Zeng [a2], Kurt Rubin [a2] and Kelly Barry [a2]

DOI: https://doi.org/10.1557/adv.2020.347 Published online by Cambridge University Press: 21 September 2020



KLA INSTRUMENTS

Research Background & Motivation

https://www.mercurynews.com/wp-content/uploads/2020/03/SJM-L-SHORTAGE-0320-2.jpg?w=862

Serious PPE shortages worldwide

https://acsh.assetsadobe.com/is/image//content/dam/cen/98/web/4/WEB/20200427lnp2-main.jpg/?$responsive$&wid=700&qlt=90,0&resMode=sharp2

Serious concerns about N95 decontamination & reuse

Mask vs. Respirator

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

A N95 respirator mask. (Robin Lubbock/WBUR)

N95 RRFs Decontamination Recommendations by FDA & CDC

Vaporous Hydrogen Peroxide

https://images.wsj.net/im-170613?width=620&size=1.5

Moist Heat

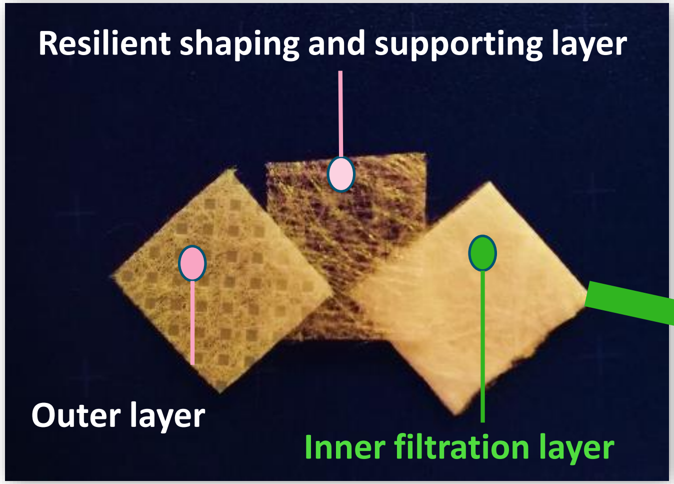
https://x9b1f1ulw2g31537u1gl95q1-wpengine.netdna-ssl.com/wp-content/uploads/2020/04/n95-in-oven-2.jpg

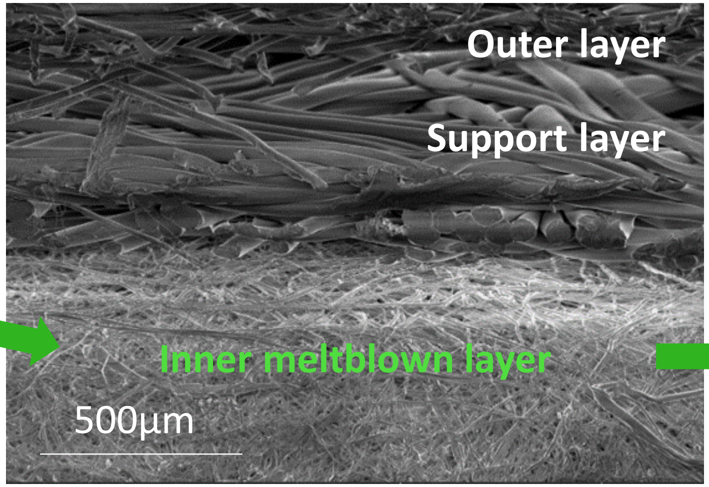
Today's Discussion

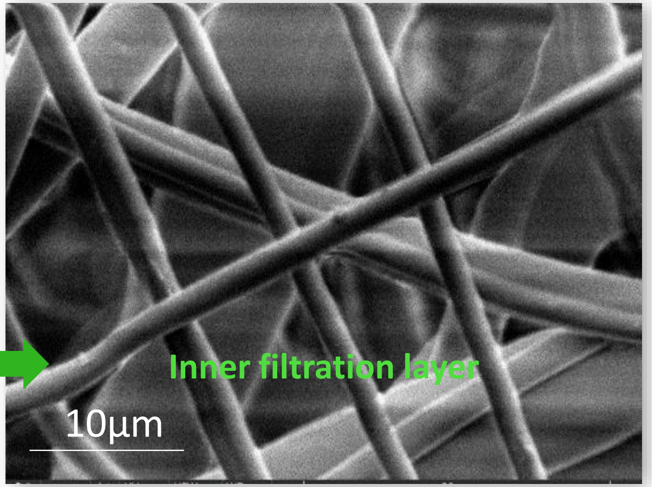
UVGI (ultraviolet germicidal irradiation)

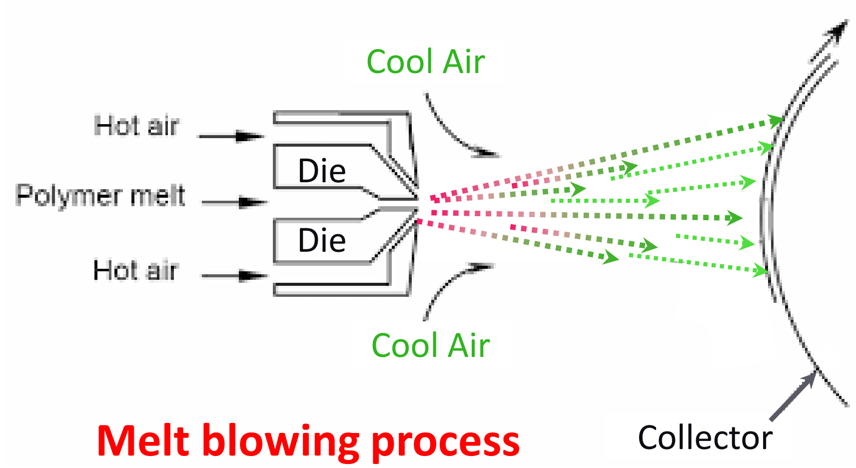
https://cdn.mos.cms.futurecdn.net/CxbAU4fC7jayV6Qd2ixDaH.jpg

Meltblown Microfibers + Corona Electrostatic Charging ⇒ N95 Respirator



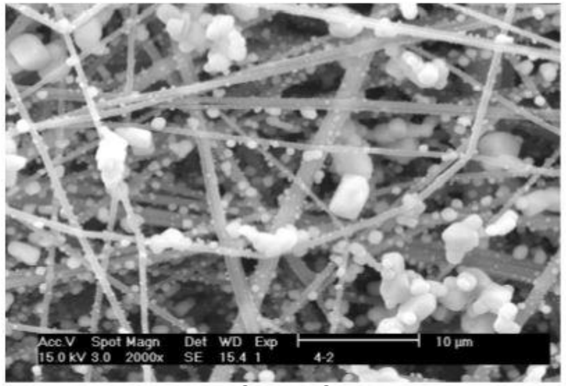






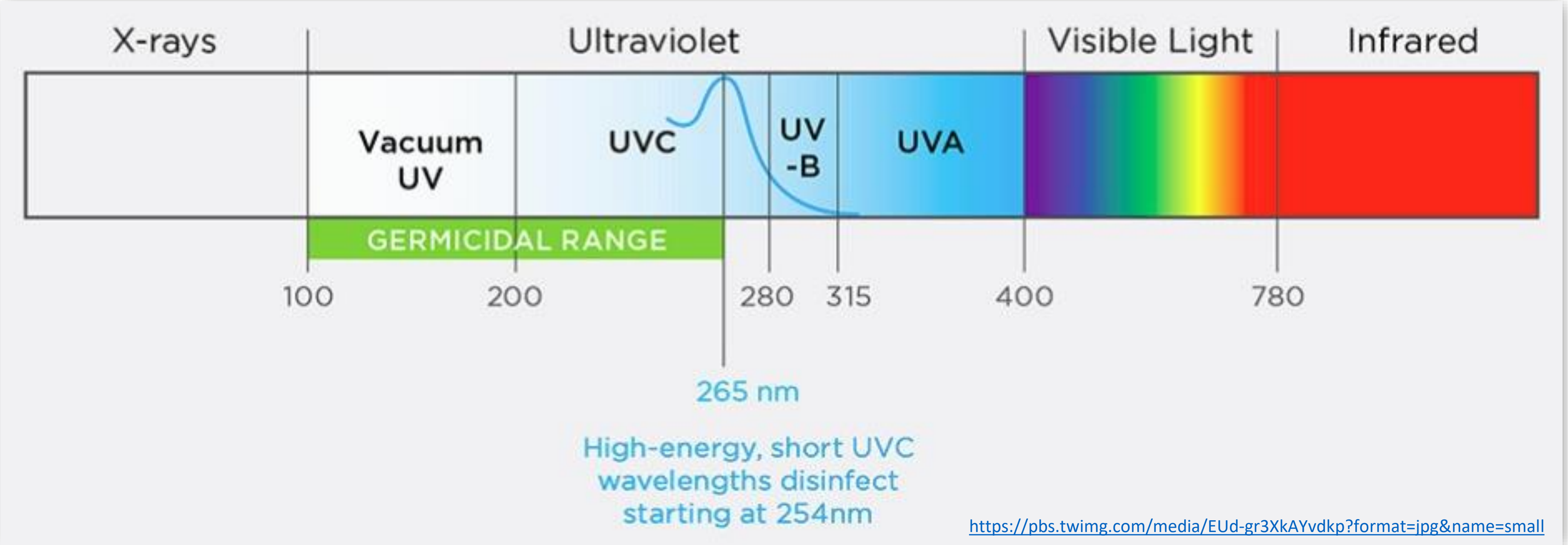
Corona Electrostatic Charging

Charging N95 fibers
Invented by Peter Tsai



Courtesy of Prof. Peter Tsai

UVGI Sterilization



https://pbs.twimg.com/media/EUd-gr3XkAYvdkp?format=jpg&name=small

- Uses ultraviolet C (short-range ultraviolet, λ = 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

$$\text{UVC dose} \propto \frac{\text{UV bulb power} * \text{Exposure time}}{4 * \pi * \text{UV bulb distance}^2}$$

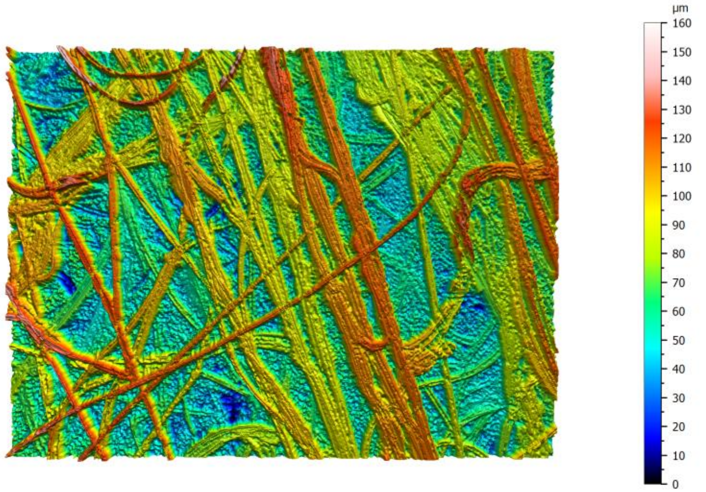
KLA INSTRUMENTS

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

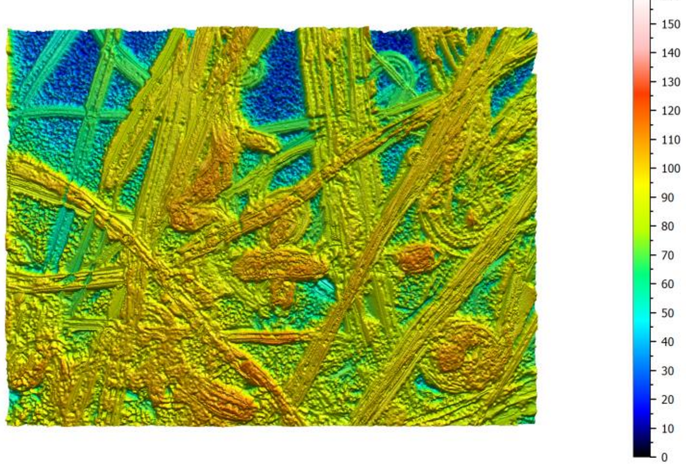
Zeta-20 Optical Profiler



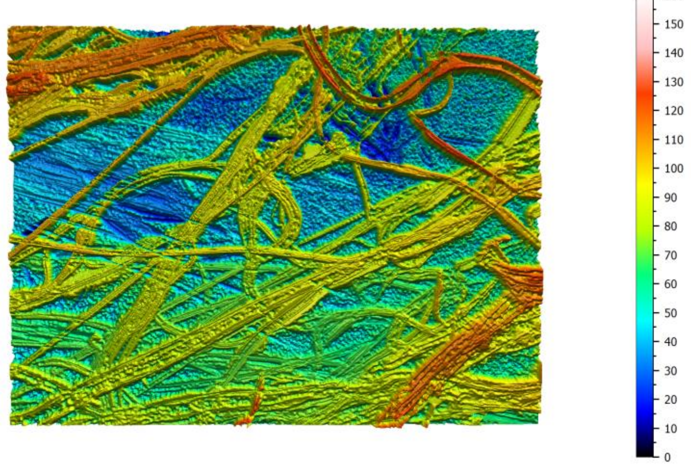
Topography of Meltblown fibers after UV exposure

N95 Respirator Meltblown Layer Topography versus UV Dose

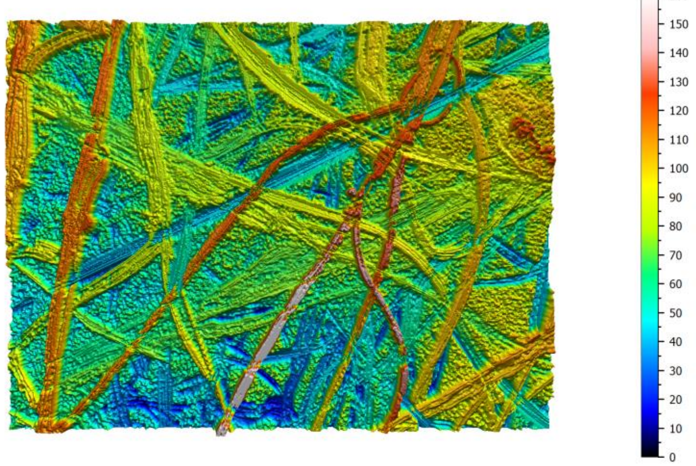
Dose = 0 J/cm²



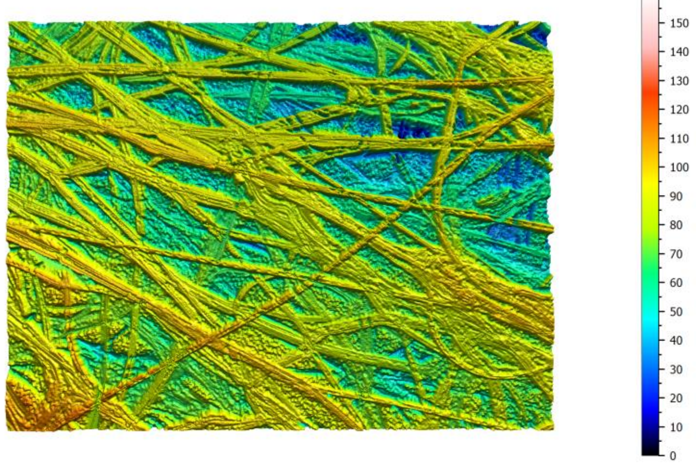
Dose = 1 J/cm²



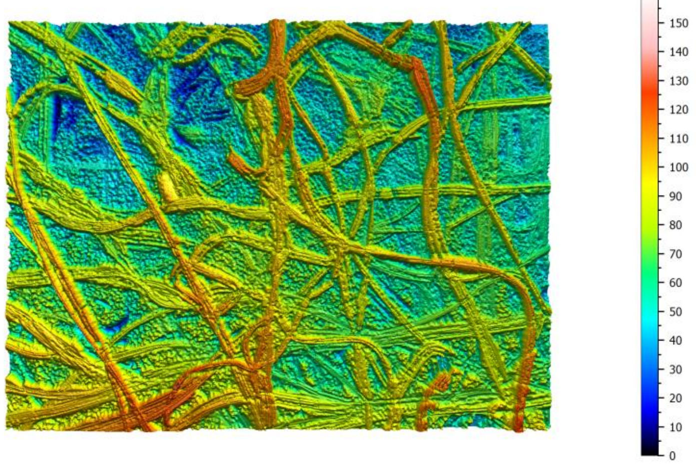
Dose = 7 J/cm²



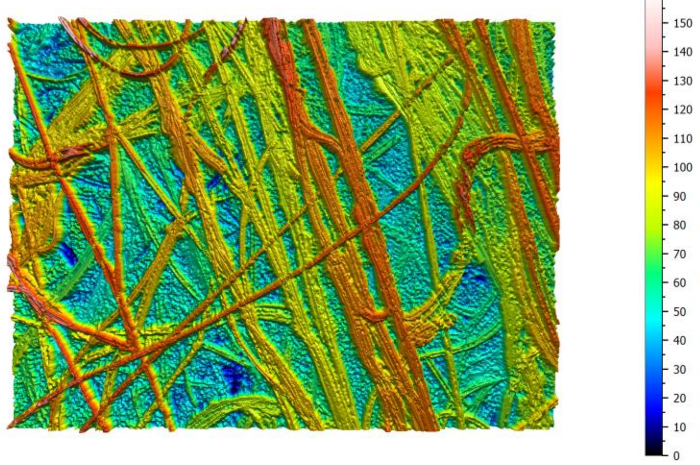
Dose = 13 J/cm²



Dose = 19 J/cm²

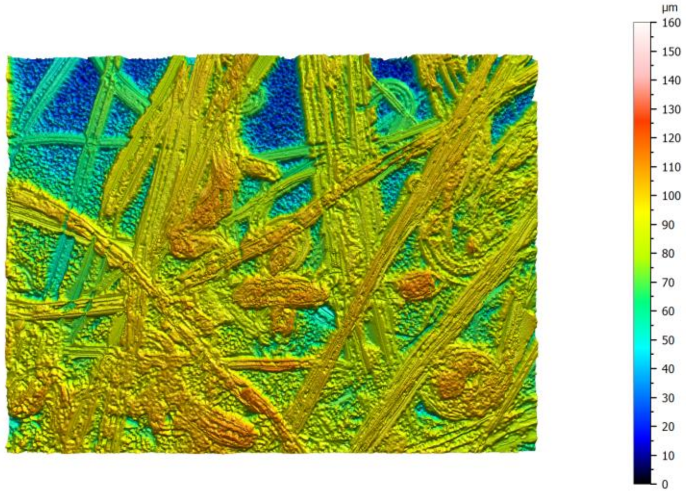


Dose = 31 J/cm²

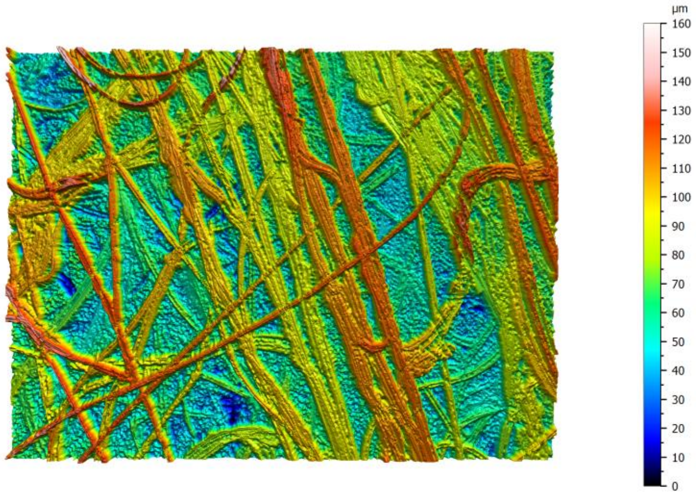


N95 Microfiber Width Analysis at Different UVC Dosages

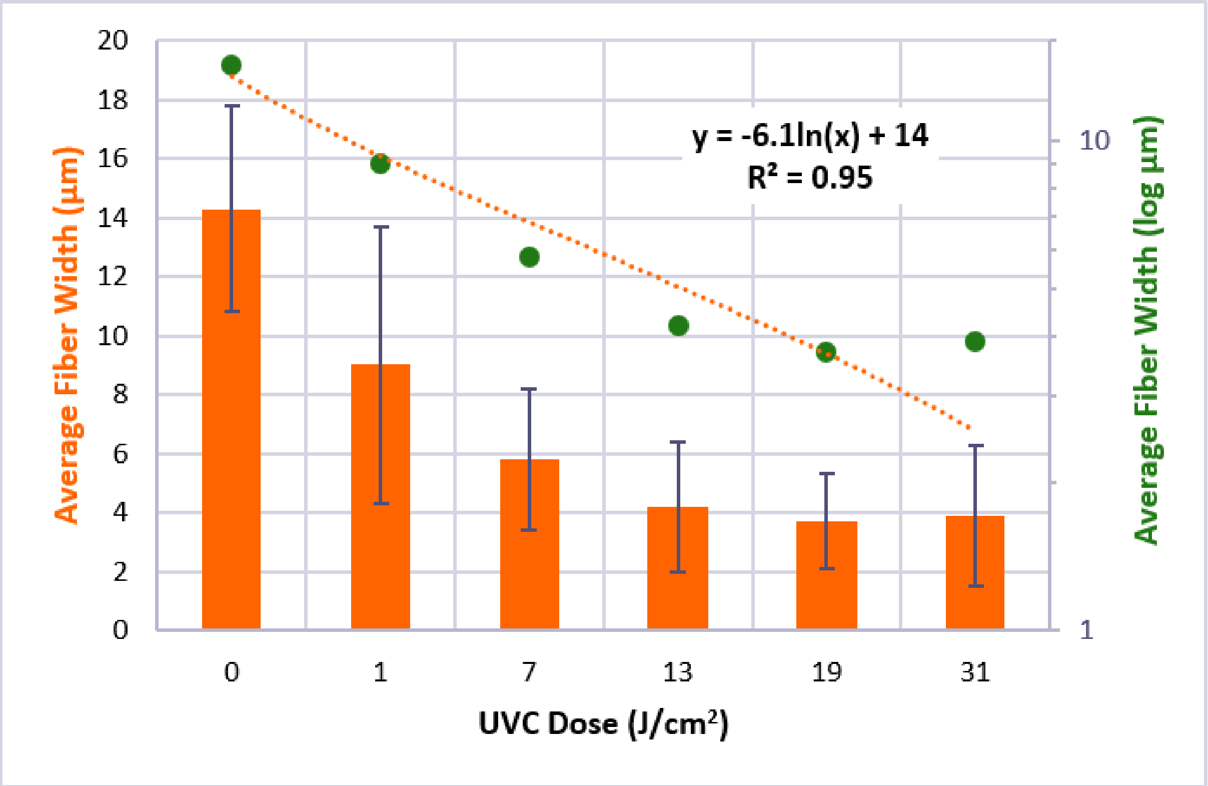
Fiber transition after 0 min UV-C exposure



Fiber transition after 155 min UV-C exposure



38W 254nm UV-C

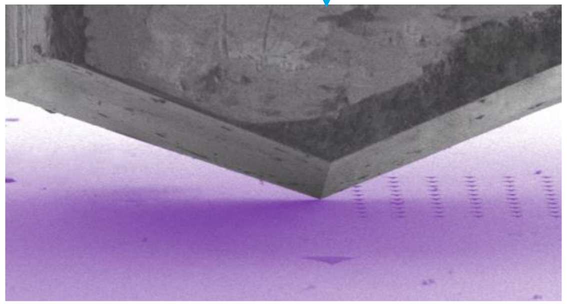


KLA INSTRUMENTS

Measure Mechanical Properties at Small Scale by Nanoindentation

1. Indent

Load/Unload

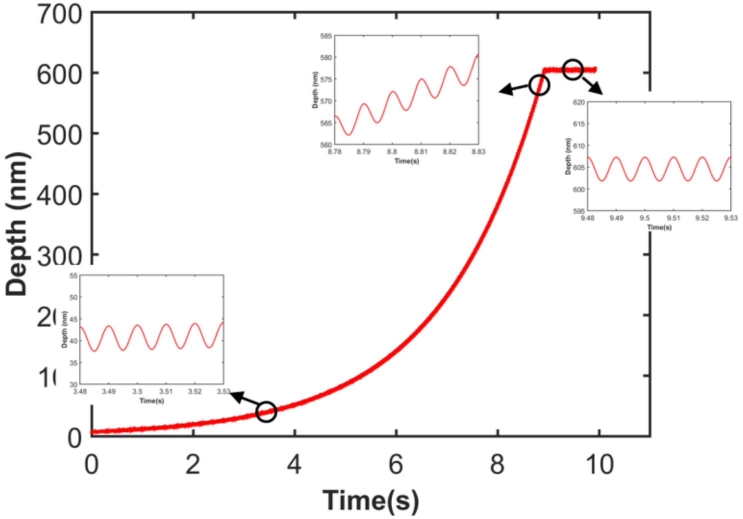


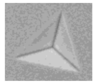
2. Measure during indentation
   1. Load applied
   2. Depth tip penetrates
   3. Stiffness
3. Extract material's mechanical properties
   1. Hardness (H)
   2. Modulus (E)

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

Continuous stiffness measurement

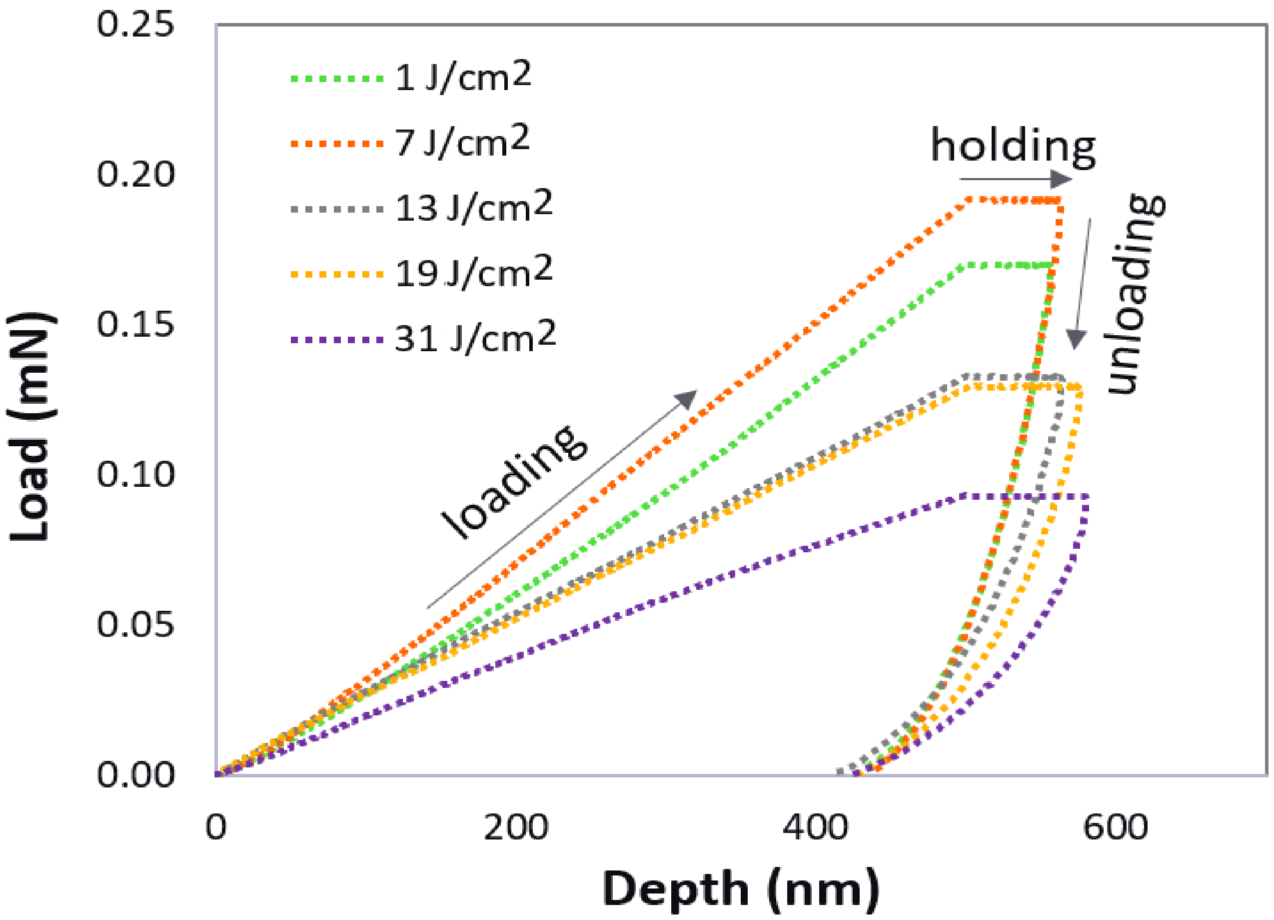
- Oscillate probe during indentation
- Measures hardness and modulus as a function of depth or load



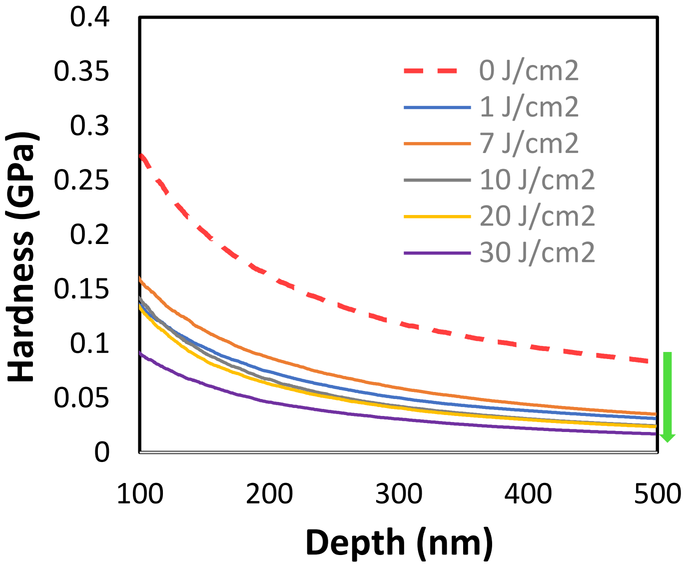
- Nano Indenter® G200X
- Diamond Berkovich Indenter 
- NanoVision scanning probe microscope

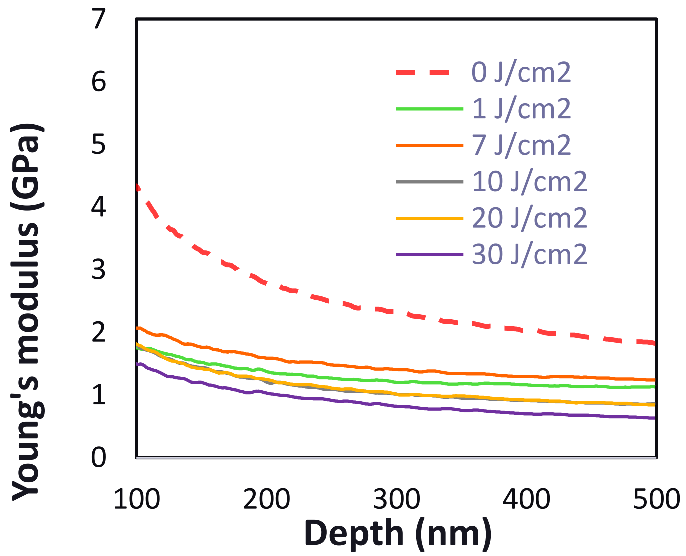
KLA INSTRUMENTS

Meltblown Microfiber Response to Nanoindentation

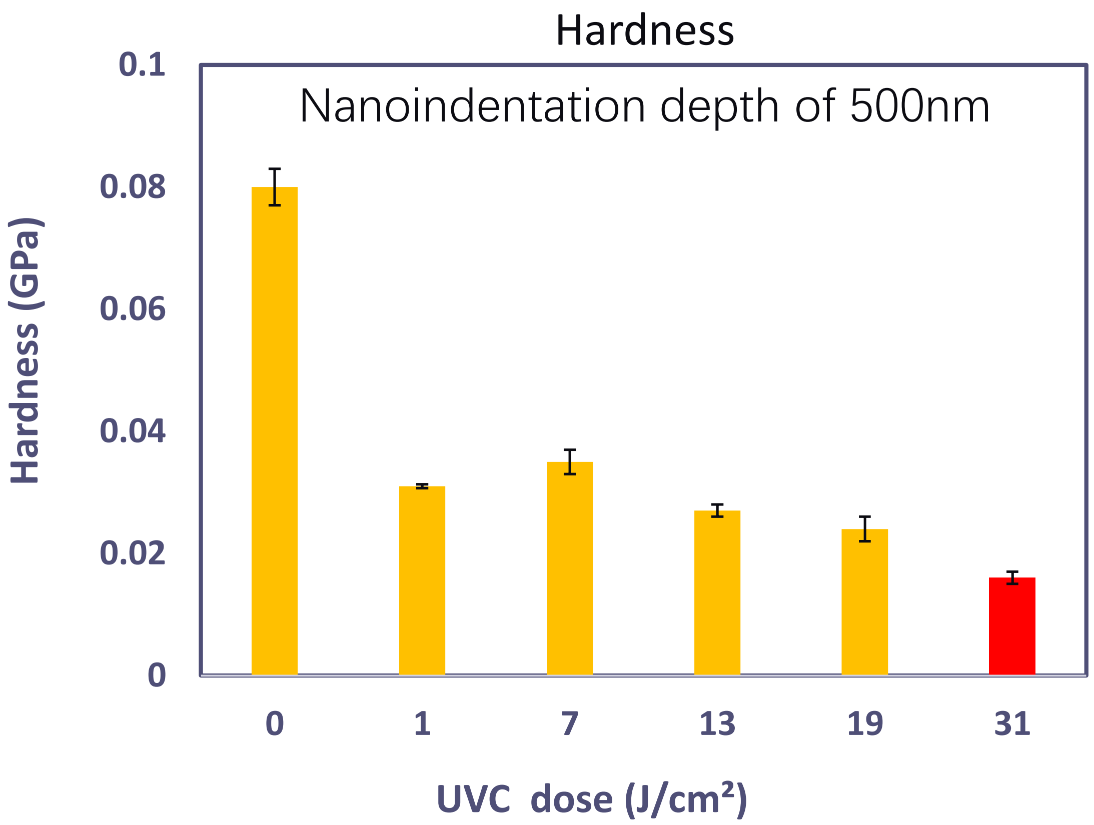


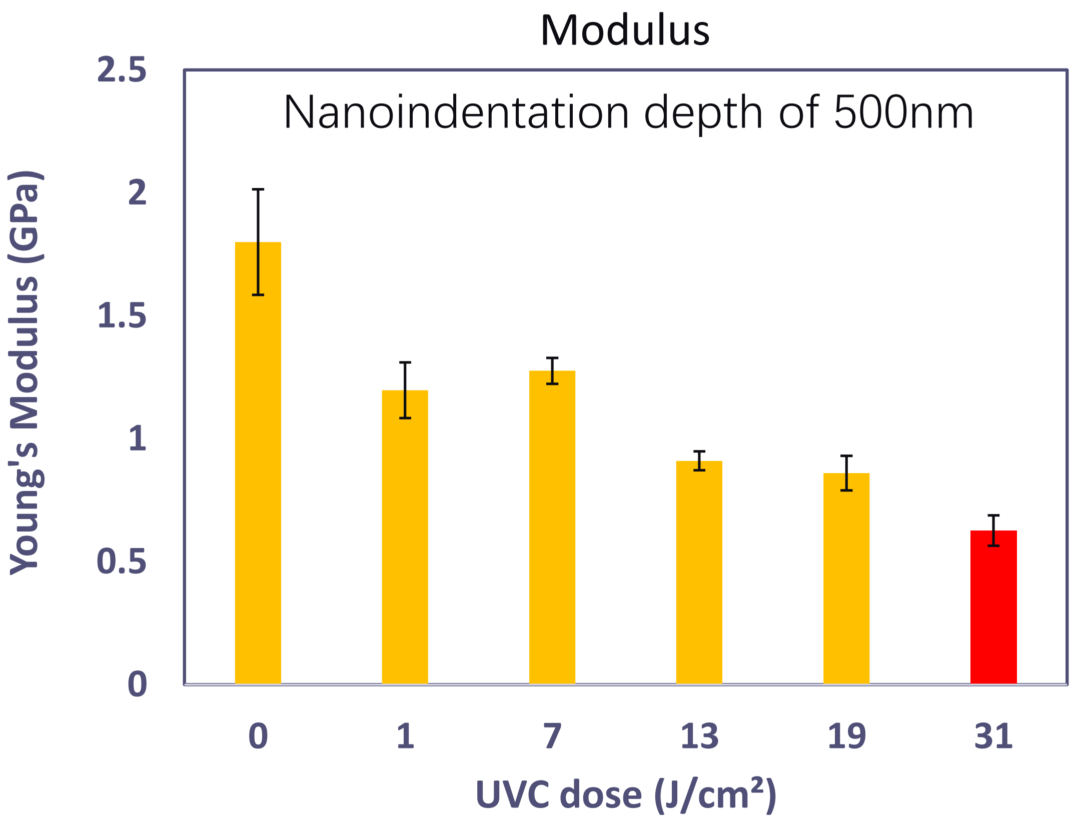
Typical nanoindentation load and displacement curves





Fiber Mechanical Property Changes After Ultraviolet Light Exposure





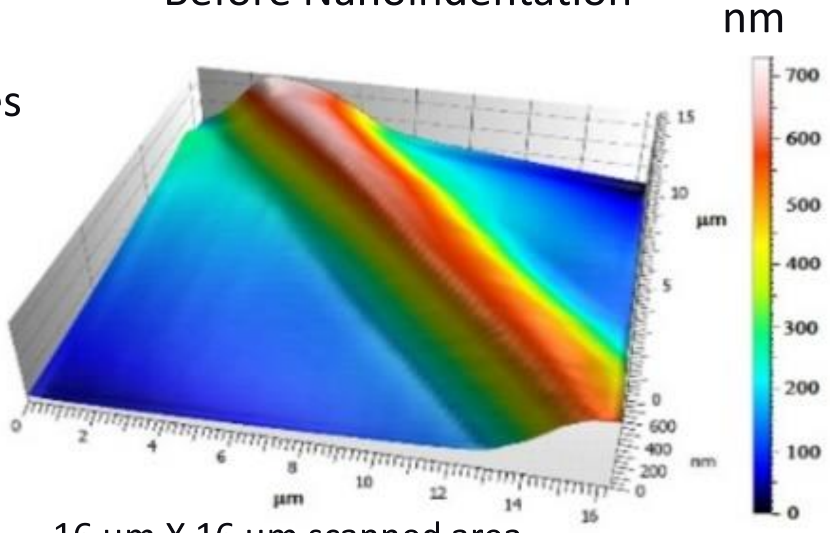
Each UV exposure dose started with fresh polypropylene microfibers

KLA INSTRUMENTS

Nanoindentation Response of Single Melt-Blown Non-Woven Fiber

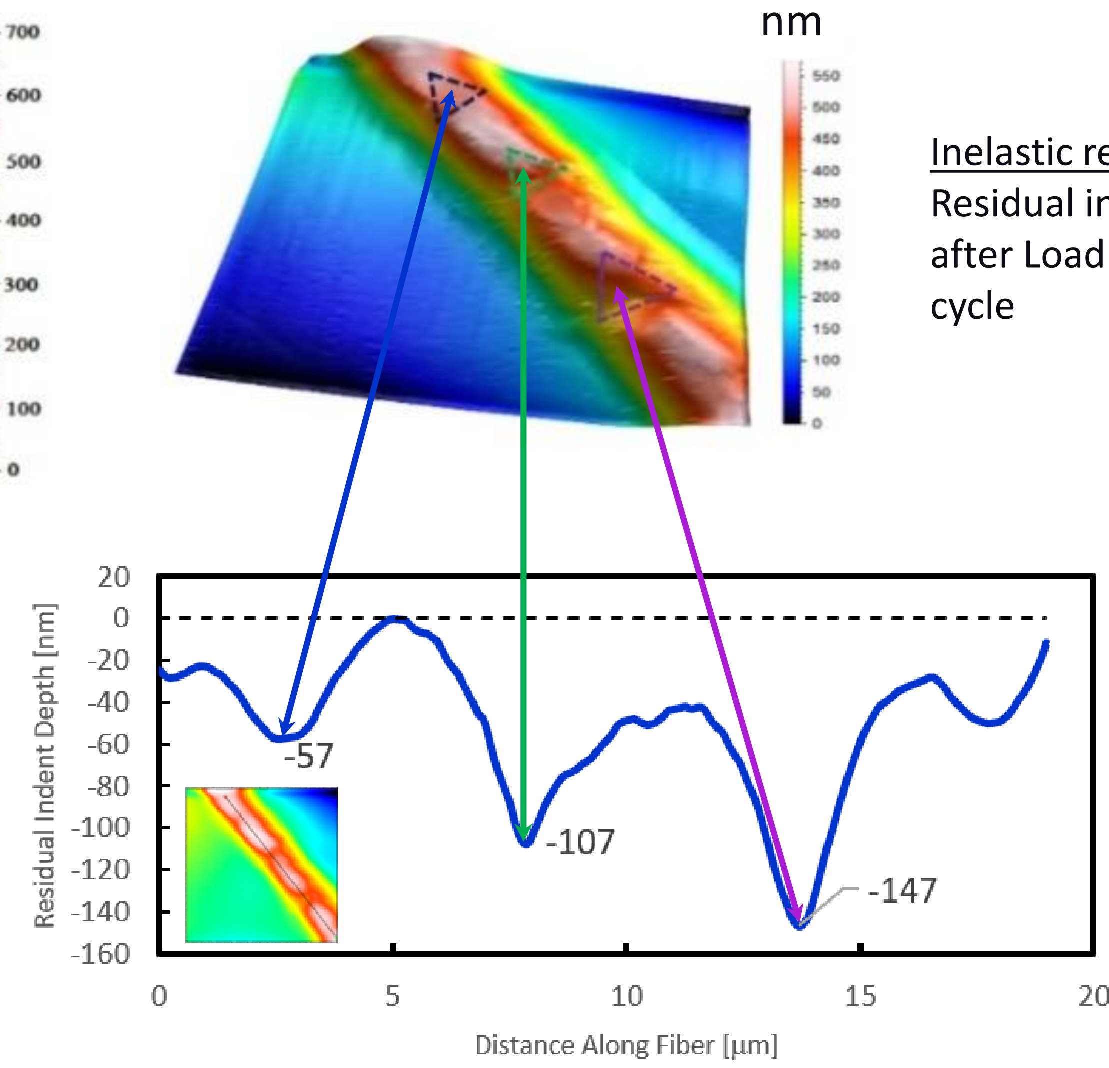
Before Nanoindentation

Fiber protrudes ~600nm high above epoxy



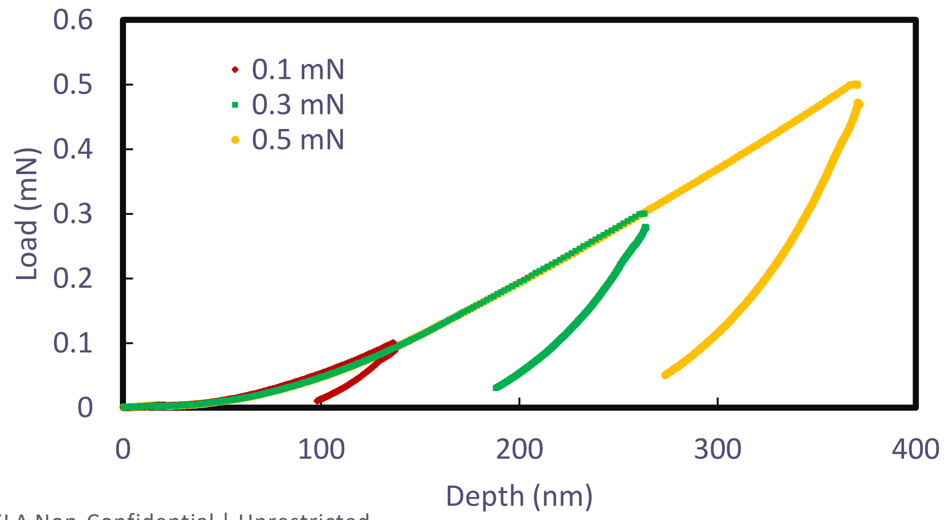
16 µm X 16 µm scanned area

After Nanoindentation



<u>Inelastic response</u>

Residual indentation after Load / Unload cycle



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

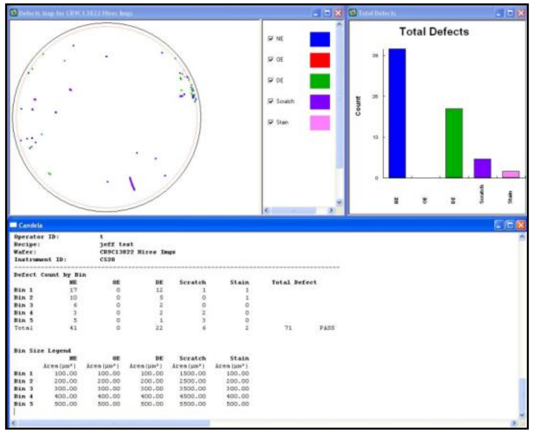
- Over-exposure to UVC radiation during decontamination processes causes loss of strength of the polypropylene microfiber layer
  - Young’s modulus, E, and hardness H both decrease with increase of UVC dose
  - Average fiber width decreases as a function of exposure time

KLA INSTRUMENTS

Turning Innovation into Products ⇒ Automated Optical Inspection

Candela

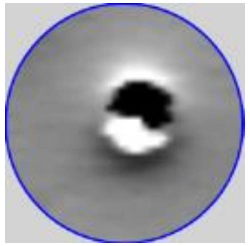
Defect Map



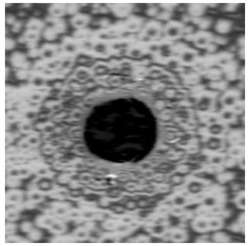
Zeta

Optical Mapping and Characterization of Defects

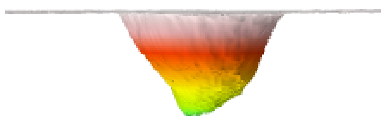
SiC TED



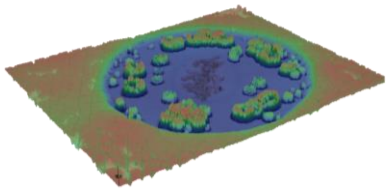
GaN Downfall



SiC TED



GaN Downfall

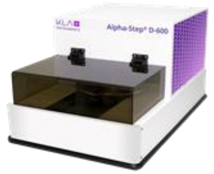


Characterization Needs for Devices and Materials are Diverse

Labs

Techniques Illustrated in Today's Discussion

Stylus Profiler



Alpha-Step®, Tencor™

Optical

Zeta

Profilm3D®

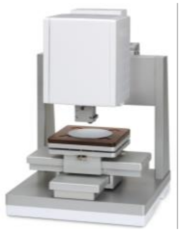
Thin Film

Filmetrics®

Nanomechanics

Nano Indenter®

Electrical



R50 Sheet Resistance
Eddy Current, 4-point probe

Fabs

HRP®

Stylus-based

Optical-based

Automated High Resolution Profilers

Candela®

Defect Inspection Systems

Technology Scaling

- Connect R&D ⇔ Manufacturing
- Measurement technology in Lab tools scales to automated Fab tools

KLA INSTRUMENTS

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