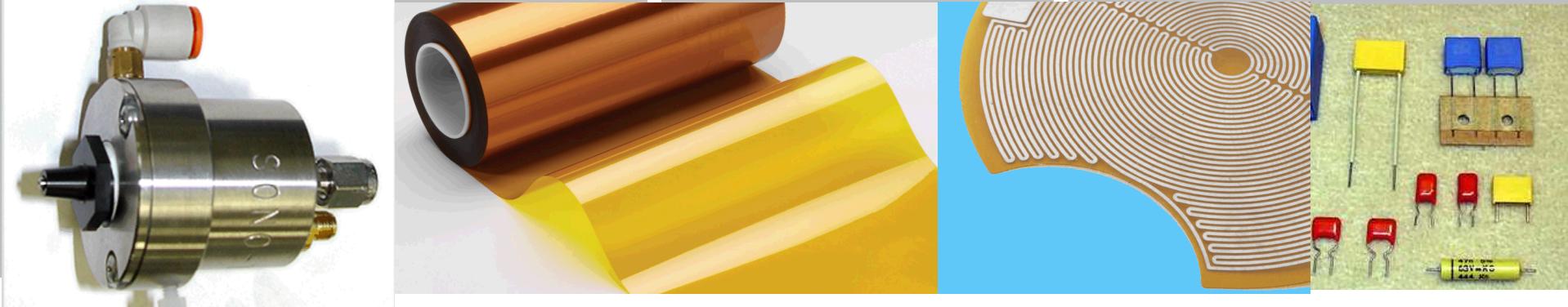


Exceptional service in the national interest



Understanding Sources of Defects in Polyimide Films Using Aerosol Based Printing

Judith M. Lavin, David M. Keicher, Shaun R. Whetten, Penny B. Moore, Seethambal S. Mani, Leah Appelhans



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Outline

- **Polyimide Applications & Problems Caused by Defects in Polyimides**
- **Direct Write System – Additive Manufacturing**
- **Aerosol Based Printing of Polyimide using Sono-Tek Spray Nozzle**
- **Comparison of Aerosol Based Printing to Traditional Approach**
- **Process Conditions Studied to Understand Source of Defects**
- **Experimental Results**
- **Conclusion**

Properties & Applications of Polyimides

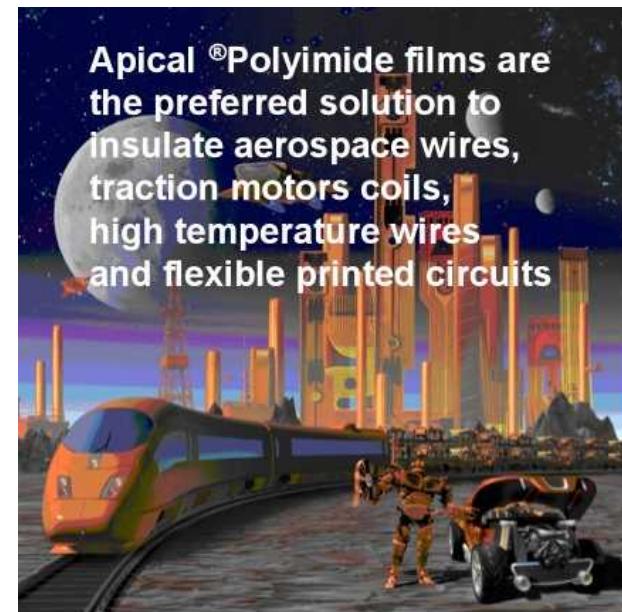
- Widely used in industry
 - Lightweight
 - Flexible
 - Resistant to heat and chemical
 - Robust physical and mechanical properties over a range of temperatures
- Commercially available polyimides
 - DuPont -Kapton
 - Apical - PI films
 - Dunmore
 - Norton Films



Dunmore- Scratch Resistant Films for consumer and electronic touch screen applications



Kapton is used in applications such as the solar array and for thermal management in the United States space program



Parameters of Concern in Developing Polyimide Films

- Bulk density/uniformity of film
- Topology
- Defect density

Effects of Sub Optimal Polyimide Films:

a. Physical

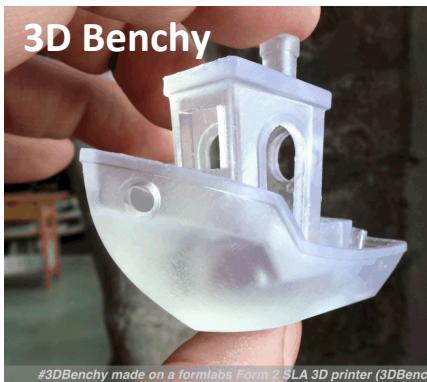
- Crazing & Cracking
- Roughness
- De-wetting
- Delamination
- Bubbles

b. Performance (in dealing with electronic components (capacitors))

- Decreased dielectric breakdown strength
 - Permittivity contrasts
 - Field enhancements

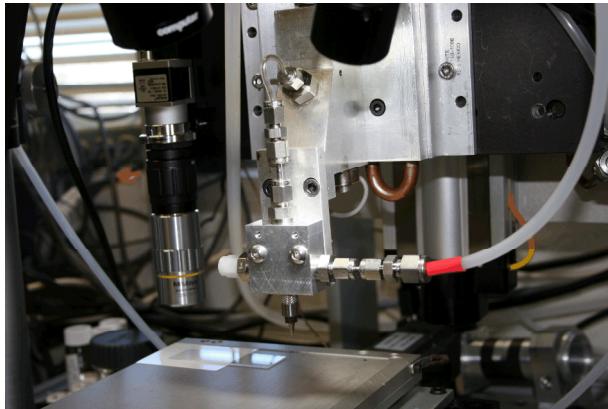
Advantages of Direct Write AM in Films Deposition

- Zero assembly requirements
- Minimal waste
- Minimal to zero design constraints
- Greater innovation
 - New materials
 - New processing approaches
 - Unique component configurations
 - Rapid flexibility and adaptability in production



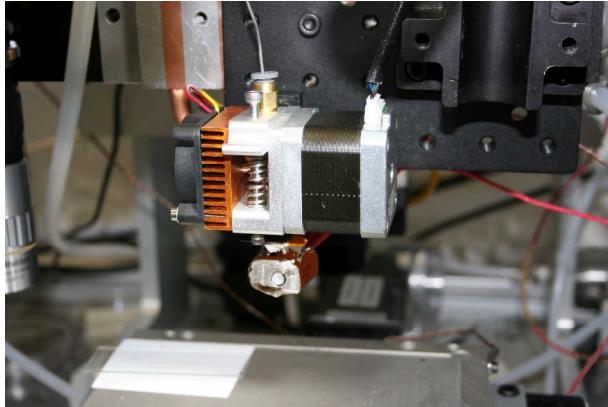
DuPont-3D Anti Counterfeiting Film
for Labels or Packaging

Direct Write Additive Manufacturing Modular System



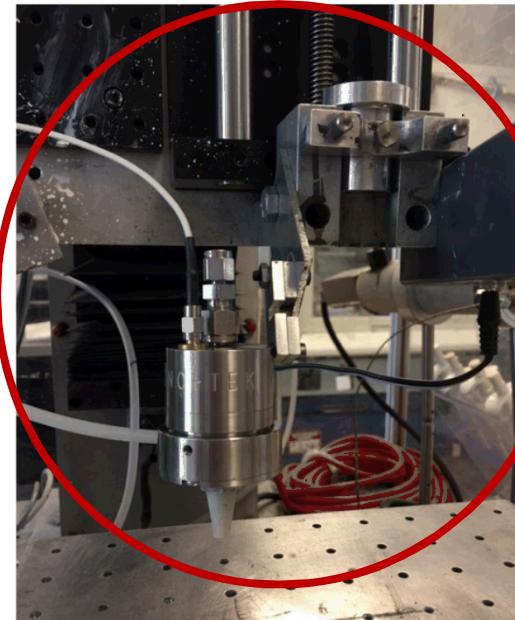
MicroJet Print Head

- 0.5-3 micron droplet
- Viscosity up to 100cP



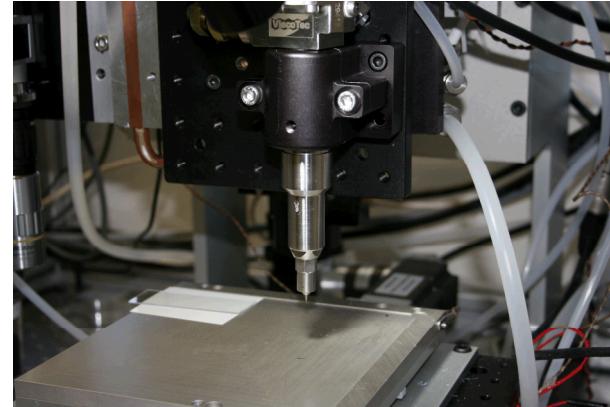
Thermal Extruder

- 200 microns-mm
- Any filament based material



SonoTek Ultrasonic Aerosol Nozzle

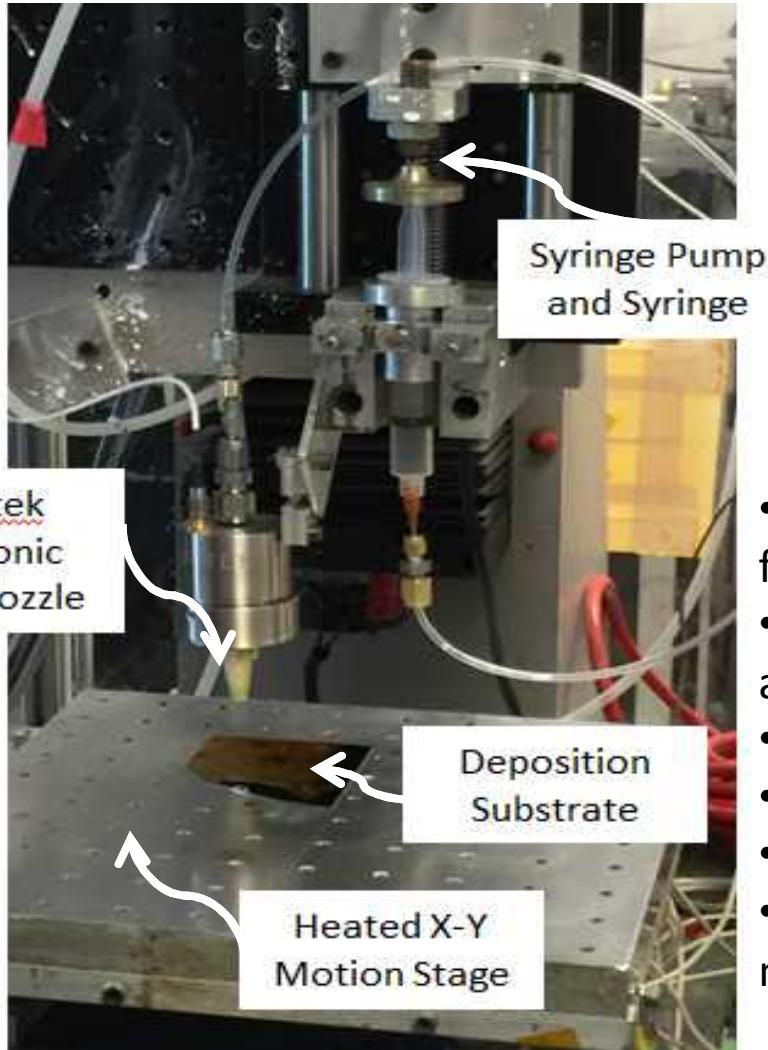
- 30-40 microns
- Viscosity up to 1000cP



Progressive Cavity Pump

- Viscosity up to 10^6 cP
- Print slurries of polymers, metals, ceramics

Spray Dispense Films Using Sono-Tek Ultrasonic Aerosol Spray Head



- Sono-tek spray head uses ultrasonic frequency to atomize material
- Compressed air (~1psi) is used to carry the aerosol & entrains the spray at the tip of the nozzle
- Material is deposited in aerosol form
- Droplet size is dictated by resonant frequency
- Nozzle deposits 30-40 micron droplets
- Decreased waste, controllable spray, uniform micron thick coating, planar & 3D substrates

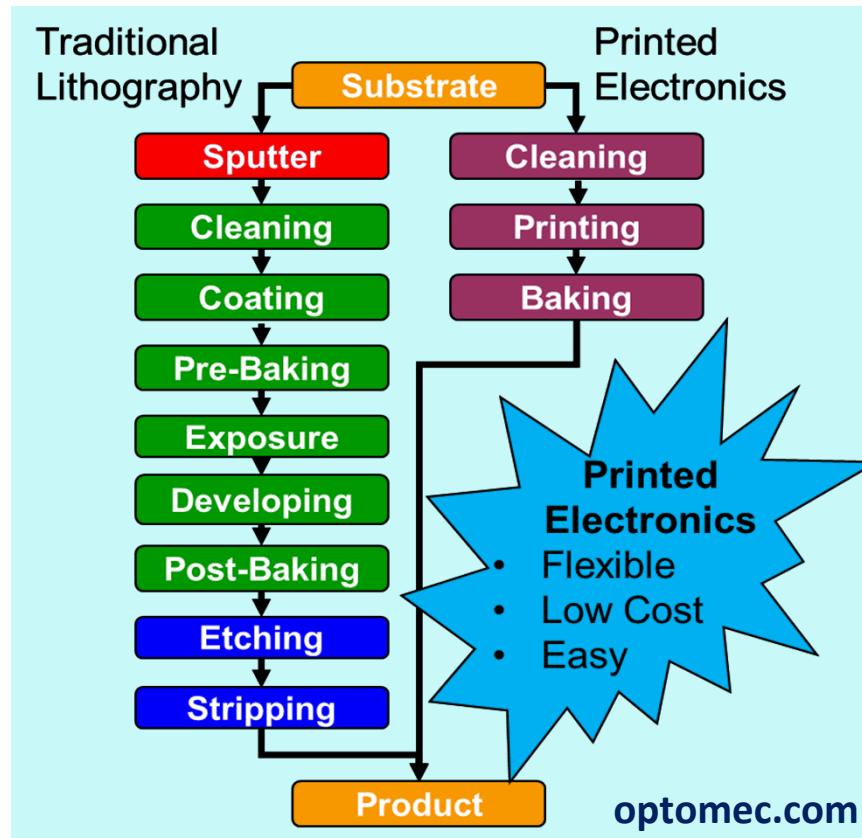
Comparison of Aerosol Based Printing to Traditional Approach

■ Traditional Approach for Printed Electronics

- Higher capital equipment cost
- Many processing steps
- Not easily customized nor adaptable

- Direct Write Printed Approach

- Lower capital equipment cost
- Fewer processing steps
- Easily customized and adaptable



Process Conditions Investigated to Understand Source of Defects

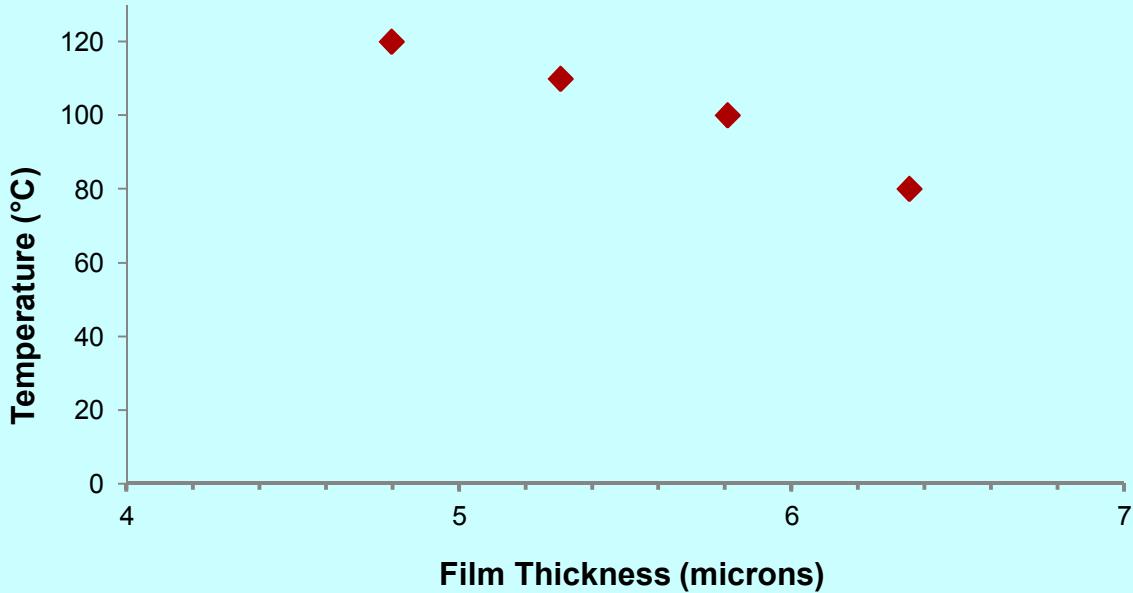
- Substrate temperature
- Table speed
- Nozzle distance from substrate
- Cure temperature

Process conditions are considered unfavorable when they result in uneven topology or surface roughness because these characteristics contribute to poor dielectric performance → here we include topology in this study

- All experiments were carried out on gold coated silicon wafers
- Sigma Aldrich Polyamic Acid that is converted to Polyimide on heating to form Films

Film Thickness as Function of Substrate Temperature

Deposition Temperature (°C) vs. Film Thickness (μm)



Deposition Parameters

- Table speed 20mm/sec
- Nozzle 1" above substrate
- Platen temperatures:
 - 80C
 - 100C
 - 110C
 - 120C
- Results are post cure

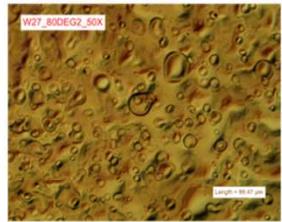
Increasing substrate temperature:

- decrease in film thickness
 - Faster evaporation kinetics at higher substrate temperature?
 - Volume dispensed?
 - Film density?

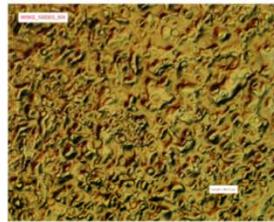
Surface Roughness as Function of Substrate Temperature

High Resolution Images of PI Films Deposited at Varying Platen Temperature

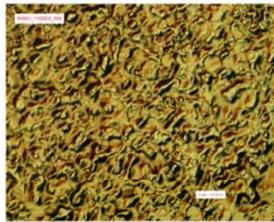
80 °C



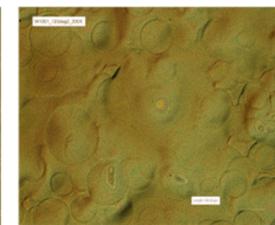
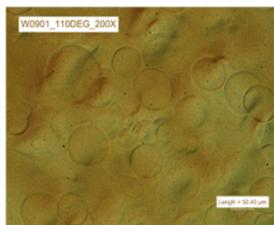
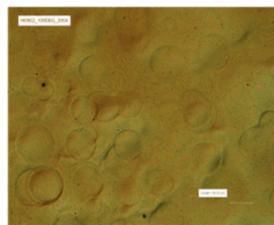
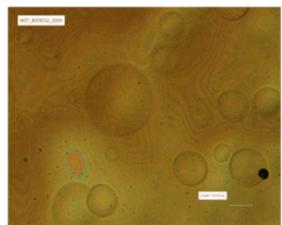
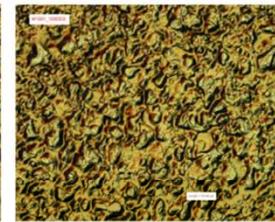
100 °C



110 °C



120 °C



Upper micrograph at 50X and lower micrograph at 200X.

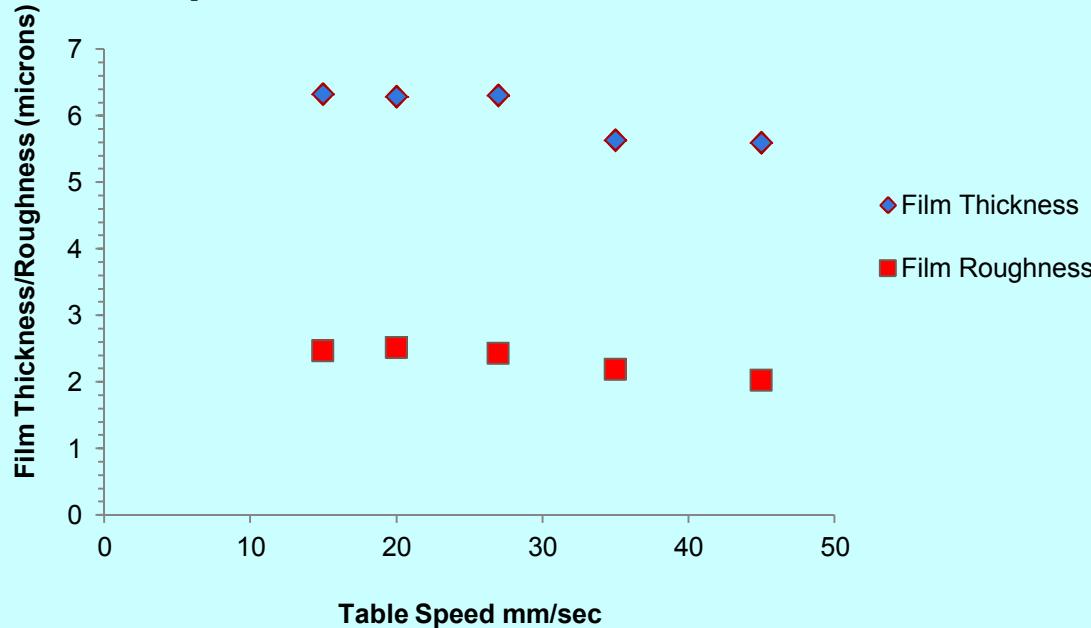
Increasing substrate temperature:

- increase in surface roughness/topology

Deposition Temperature (°C)	Film Thickness (μm)	Film Roughness (Rq, μm)
80	6.36	2.08
100	5.81	2.29
110	5.30	2.36
120	4.79	2.39

Film Thickness and Roughness as a Function of Table Speed

Film Properties as Function of Table Speed



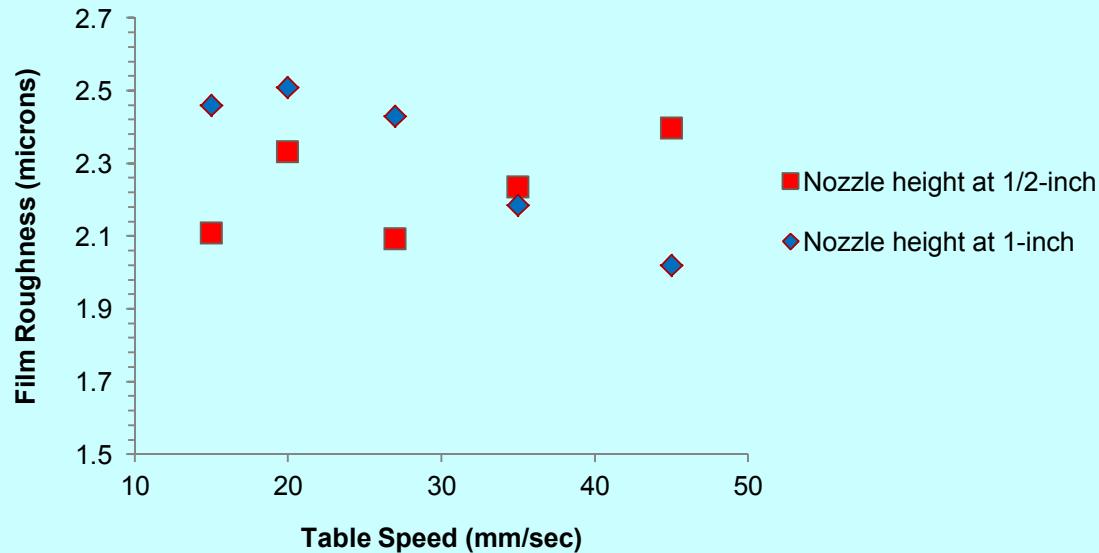
Deposition Parameters

- Platen at 95C
- Nozzle 1" above substrate
- Table speeds:
 - 15mm/sec
 - 20mm/sec
 - 27mm/sec
 - 35mm/sec
 - 45mm/sec

- Increasing table speed – decrease in film thickness
- Increasing table speed - subtle decrease in film roughness

Nozzle Distance from Substrate

Film Roughness vs Table Speed with Varing Nozzle Height



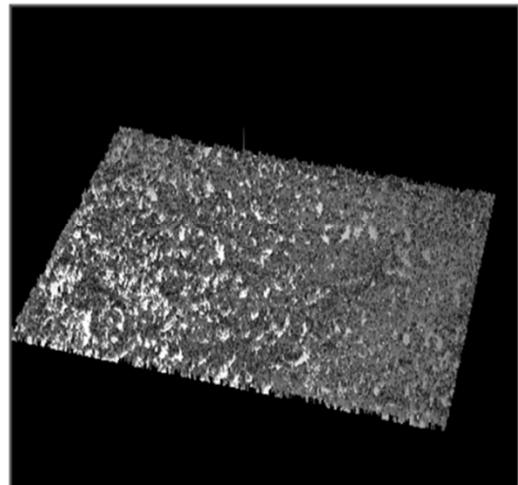
Deposition Parameters

- Platen at 95C
- ■ Nozzle $\frac{1}{2}$ " above substrate
- ♦ Nozzle 1" above substrate
- Table speeds:
 - 15mm/sec
 - 20mmsec
 - 27mm/sec
 - 35mm/sec
 - 45mm/sec

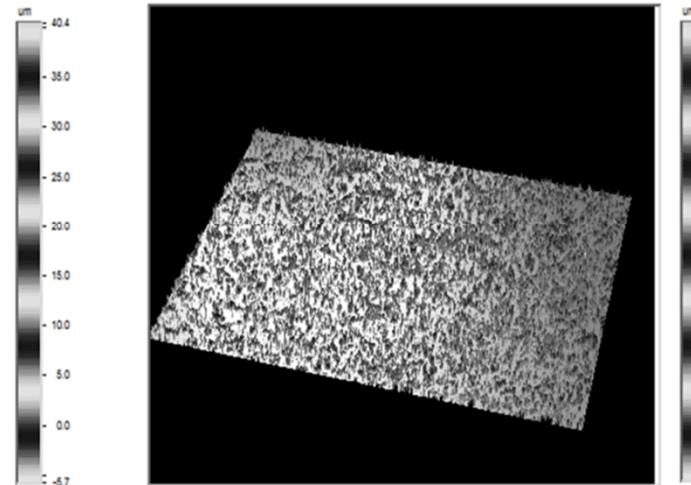
- Close to substrate – film uniformity increase with increasing table speed
- Further from substrate – film uniformity decreases with increasing table speed

Nozzle Distance from Substrate

3-D Side View of PI Film Showing Surface Roughness and Defects



Smooth Film Surface



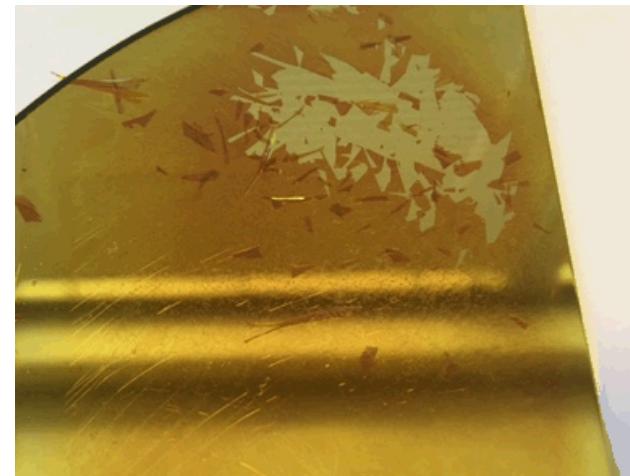
Rough Film Surface

- 3D side view images of PI film deposited on gold coated silicon wafers showing surface roughness
- Higher contrast features are indicative of greater surface roughness

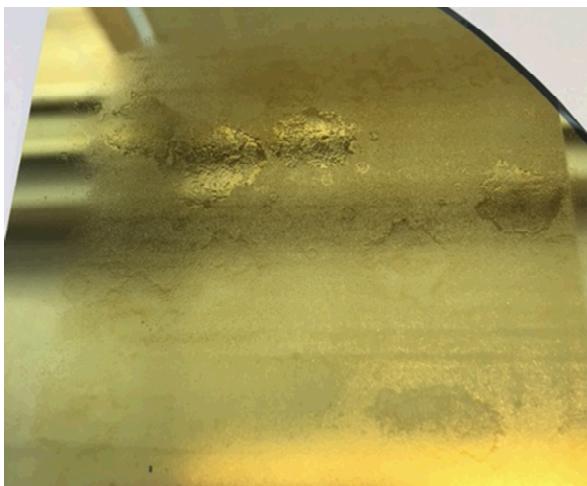
Results – Cure Temperature



Crazing: thermal shocking by cooling too rapidly



Cracking: curing at too high a temperature

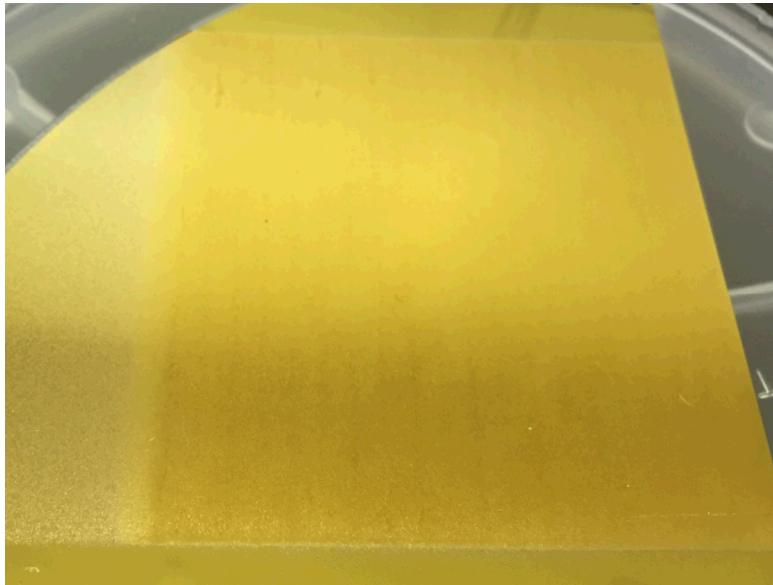


De-wetting:



Bubbles: curing too high temperature without a step-cure profile (using syringe deposition method) 15

Results – Optimal Conditions to Minimize Defects



Optimal Deposition Parameters

- Platen temperature of 80 °C
- Table speed of 45mm/sec
- Nozzle-height 1" above substrate

Optimal Cure Conditions

- Step-cure profile temperature
- 125 °C for 3 hours followed by
- Gradual ramping of 5 °C/min to 225 °C
- 225 °C for 12 hours 12 hours
- Gradual cooling to ambient temperature

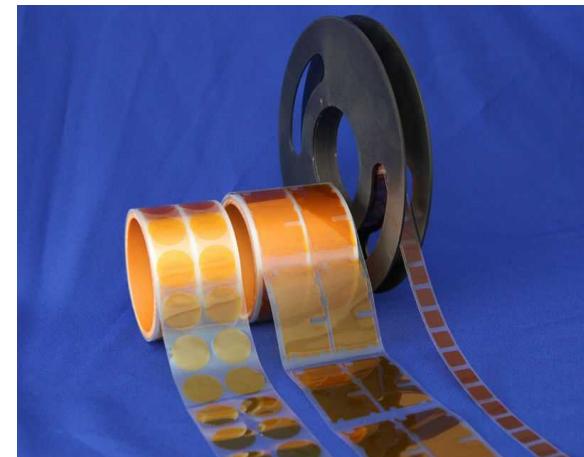
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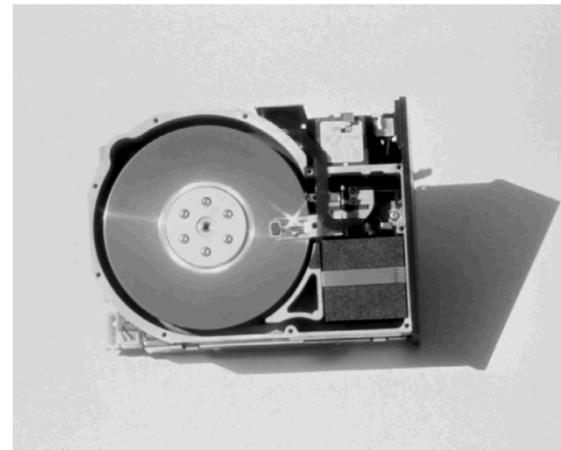


3D anti counterfeiting film
Film Integrates Clearly Visible, Unique 3D Features Directly on Labels or Packaging



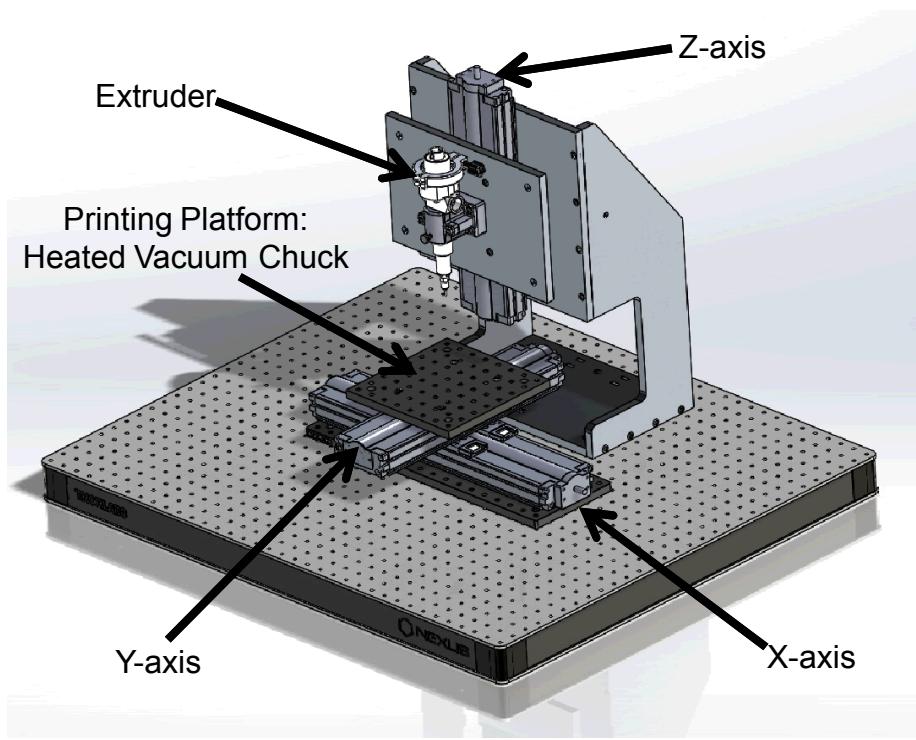
NORTON-High performance capacitors are needed to survive high temperature environments, particularly in military aircraft. Our CHEMFILM® Type C film is the thinnest pure PTFE film. This ultra-thin film (as low as 5 microns!) is often metalized and rolled creating a spiral of alternating dielectric and conductive layers. The thin material together with high temperature capabilities make ideal, compact high performance capacitors for the most extreme environments.

Kapton® is used in numerous electronic applications, including hard disk drives.



2D Solutions -polymer insulators

Direct Write Additive Manufacturing



- **Modular system – Sono-Tek Spray Nozzle; Syringe Dispense; PCP, Extruder; MicroJet**
- **Process camera module**
- **3+-axis motion control**
- **Heated vacuum plate**
- **Process vision module with manual yaw, pitch, and adjustable focus**
- **Substrate alignment module with 1000X magnification**