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Molecules to Manufacturing:
Expanding the Polymeric Materials Toolbox

In-Situ Processing Measurements

DREAMS
Design, Research, and Education for Additive Manufacturing Systems
http://www.me.vt.edu/dreams

Macromolecules Innovation Institute
At the intersection of science, engineering, and society

Christopher B. Williams
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Associate Director, Macromolecules & Interfaces Institute
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VT Additive Manufacturing Faculty

AM Processes | Advanced Materials | Process Modeling | Intelligent Manufacturing

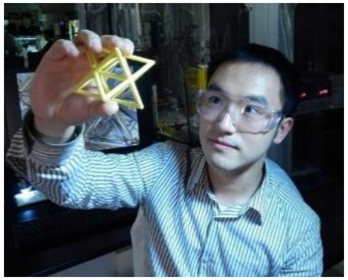

Chris Williams (ME)


Tim Long (Chem)


Donald Baird (ChemE)


David Dillard (BEAM)

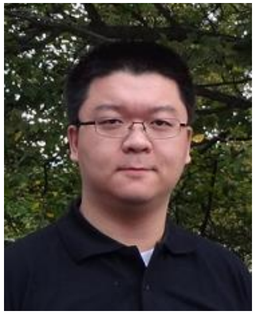

James Kong (ISE)


Rayne Zheng (ME)


Michael Bortner (ChemE)


Shashank Priya (ME)


Scott Case (BEAM)


Ran Jin (ISE)


Blake Johnson (ISE)


Hang Yu (MSE)


Carolina Tallon (MSE)


Steve McKnight (NCR)

2015 College of Engineering Team Hire

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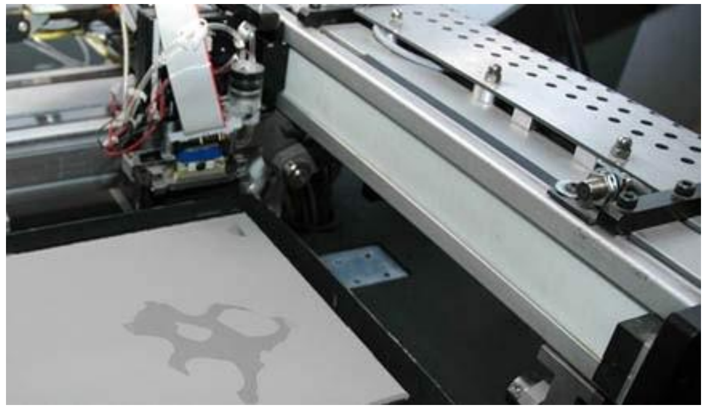
DREAMS Lab: Facilities

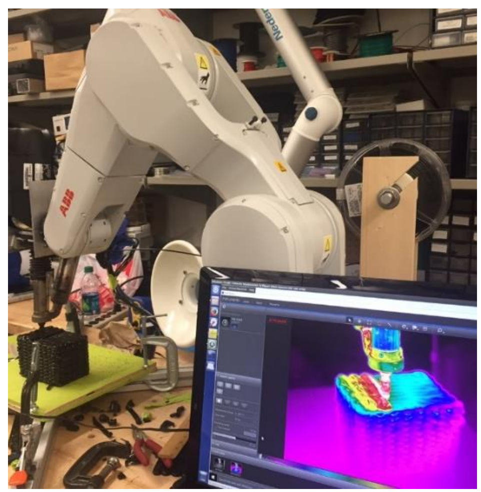
Extrusion



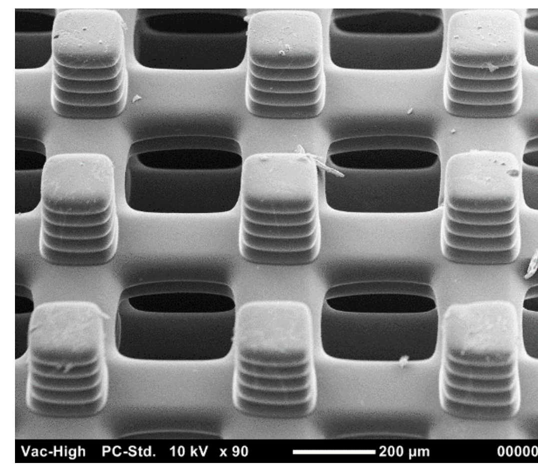
Metal/Ceramic/Sand Binder Jetting

Multi-Material Jetting

Polymer Powder Bed Fusion



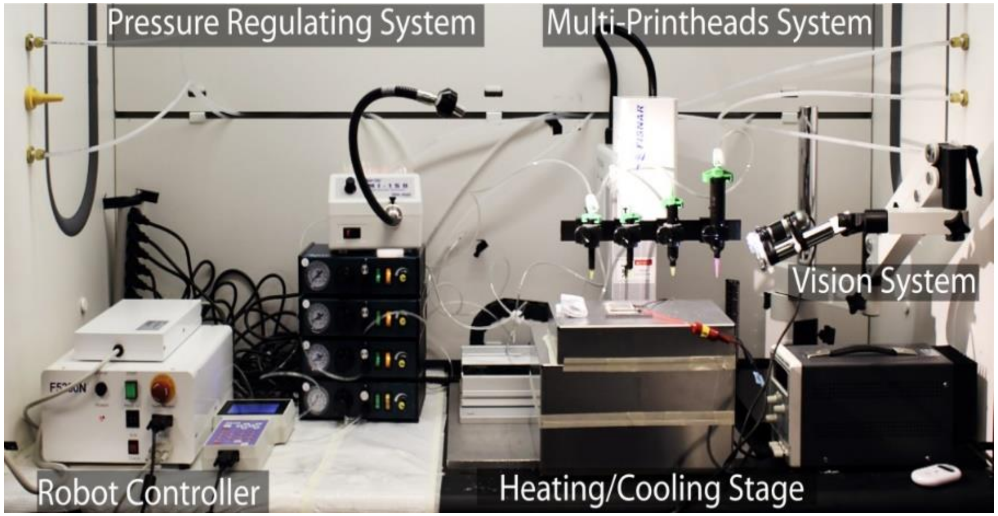
6 DoF Extrusion



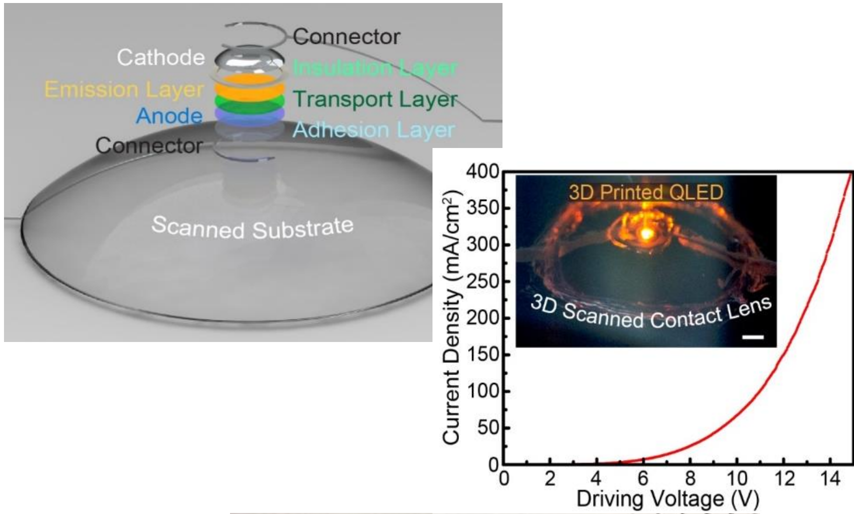
Mask Projection Vat Photopolymerization

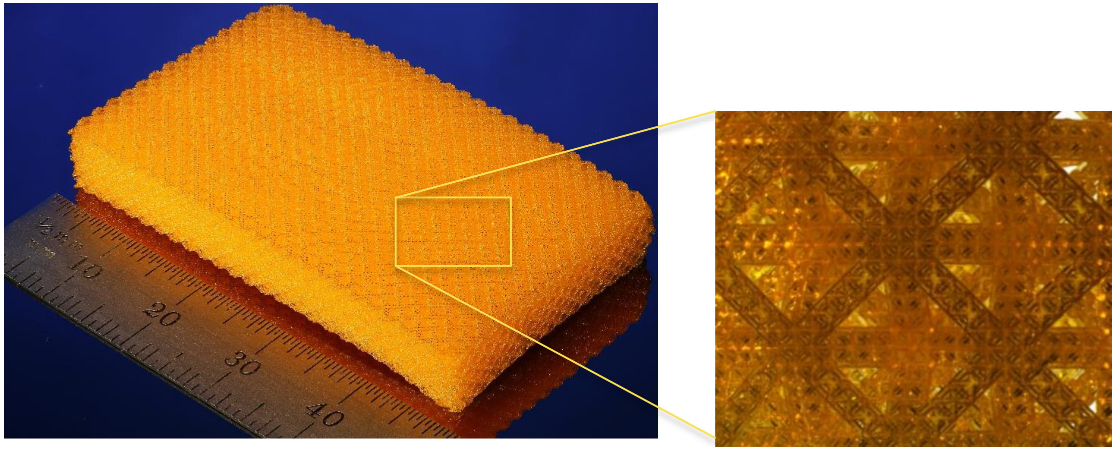
DREAMS

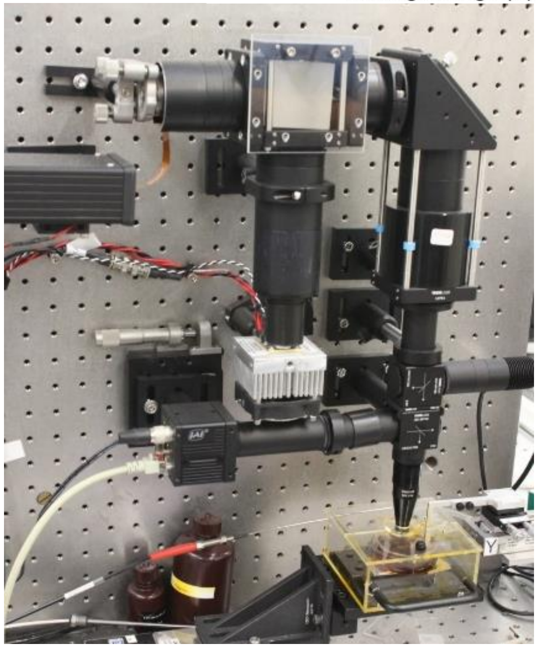
Additive Manufacturing Research Facilities



Multi-Material Precision Extrusion (B. Johnson)







Mask Projection Vat Photopolymerization (R. Zheng)

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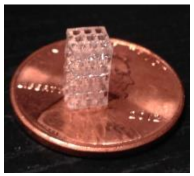
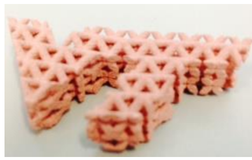
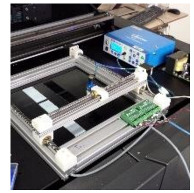
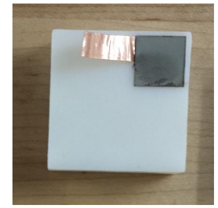
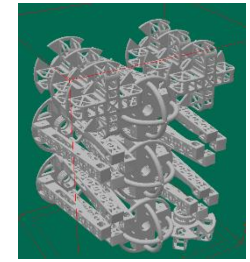
Research Across the AM Process











Advanced Materials for AM

AM Processes

Quality Assurance

Application

Design for AM

In-Situ Monitoring

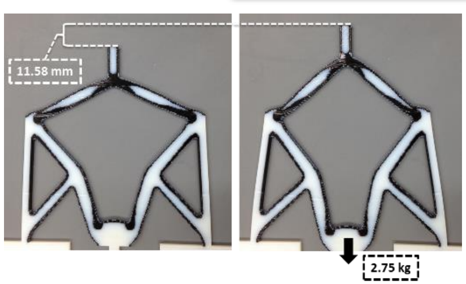
Cyber-Physical Security

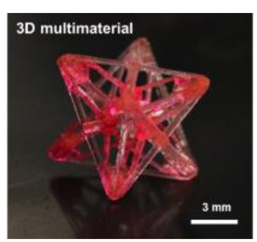
Workforce Development

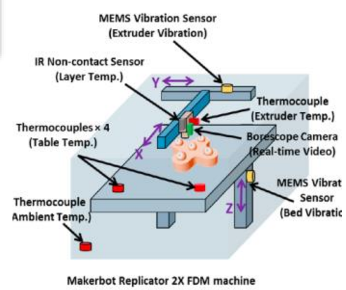
Decision Support

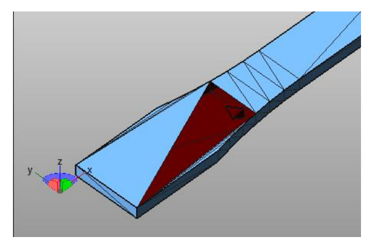
Design Optimization

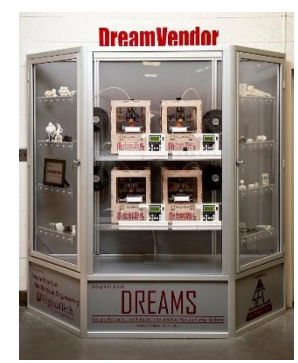
Metamaterial Design





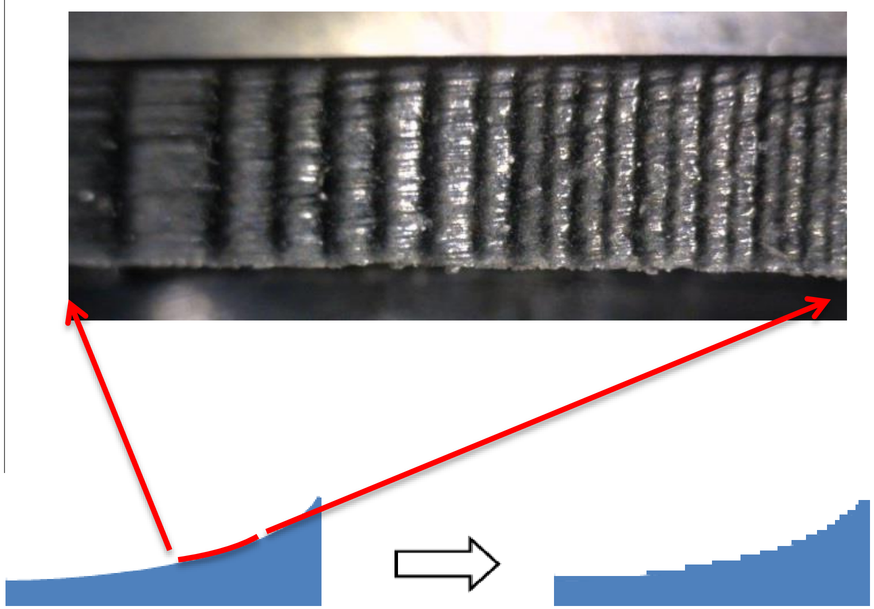






VT Additive Manufacturing Faculty

AM Processes	Advanced Materials	Process Modeling	Intelligent Manufacturing
 Chris Williams (ME)	 Tim Long (Chem)	 David Dillard (BEAM)	 James Kong (ISE)



Moore & Williams, *Rapid Prototyping Journal*, 2014

VT Additive Manufacturing Faculty

AM Processes

Chris Williams (ME)

Advanced Materials

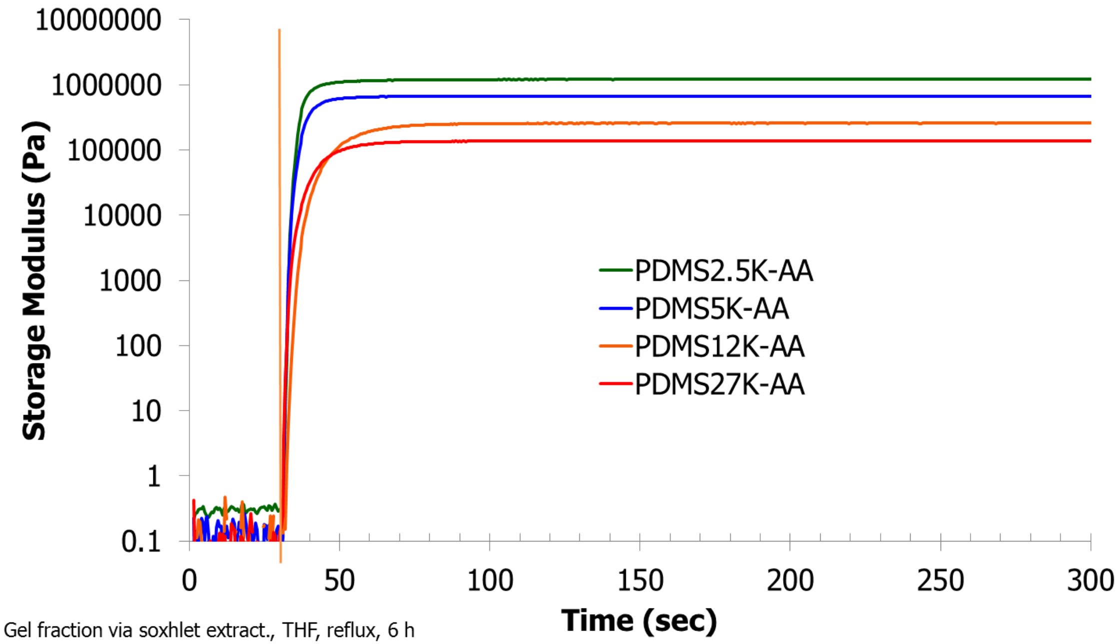
Tim Long (Chem)

Process Modeling

David Dillard (BEAM)

Intelligent Manufacturing

James Kong (ISE)



Gel fraction via soxhlet extract., THF, reflux, 6 h

TA DHR-2 with UV Curing Accessory, 20mm parallel plate, 500 µm gap, 1 Hz, 0.3 % strain, N_2

2 wt % DMPA, 7.0 mW/cm^2, 30 s delay

Schultz, Lambert, Chartrain, …, Williams & Long, *ACS Macro Lett.*, 2014

VT Additive Manufacturing Faculty

AM Processes

Chris Williams (ME)

Advanced Materials

Tim Long (Chem)

Process Modeling

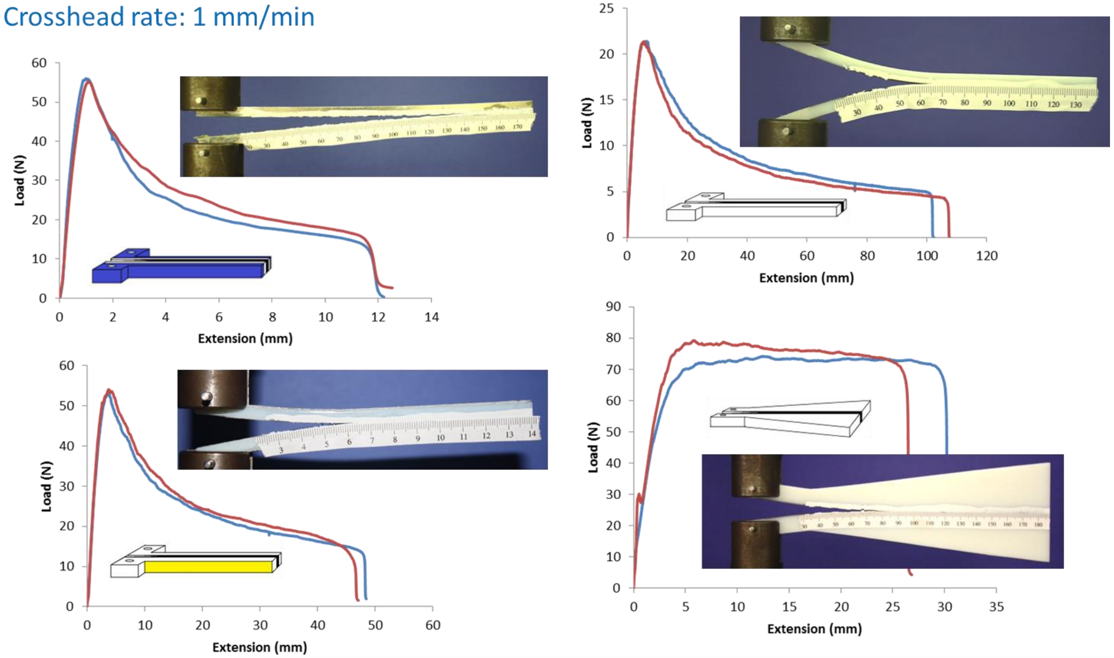
David Dillard (BEAM)

Intelligent Manufacturing

James Kong (ISE)

Determination of the mode I adhesive fracture energy, GIC, of structural adhesives using the double cantilever beam (DCB) and tapered double cantilever beam (TDCB) specimens, in BS 7991:2001. 2001.

Crosshead rate: 1 mm/min



Vu, Bass, Meisel, Orler, Williams & Dillard, *SFF*, 2014

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AM Processes

Chris Williams (ME)

Advanced Materials

Tim Long (Chem)

Process Modeling

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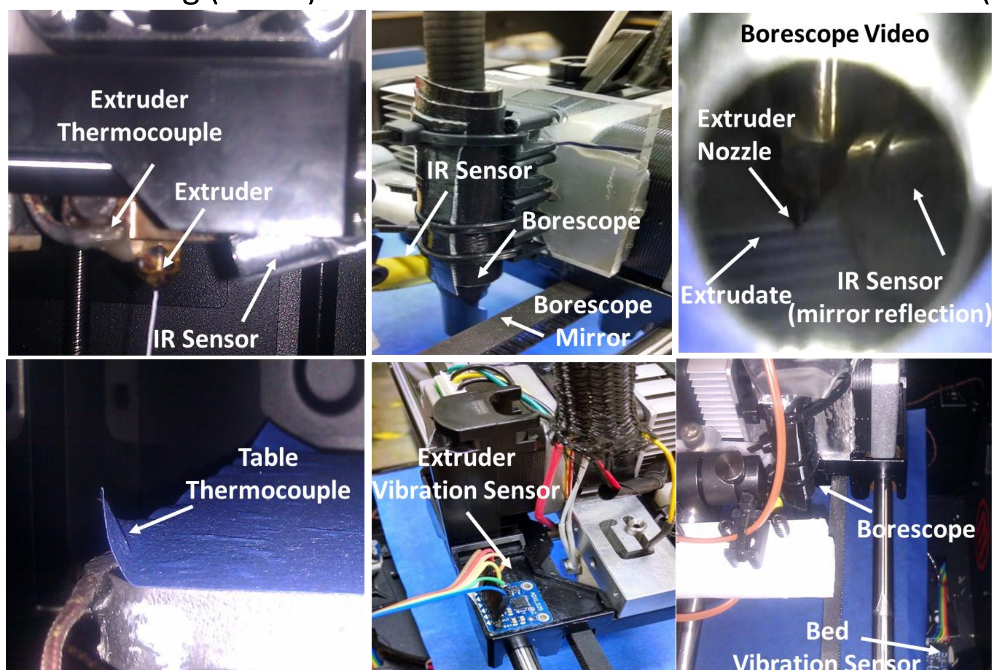
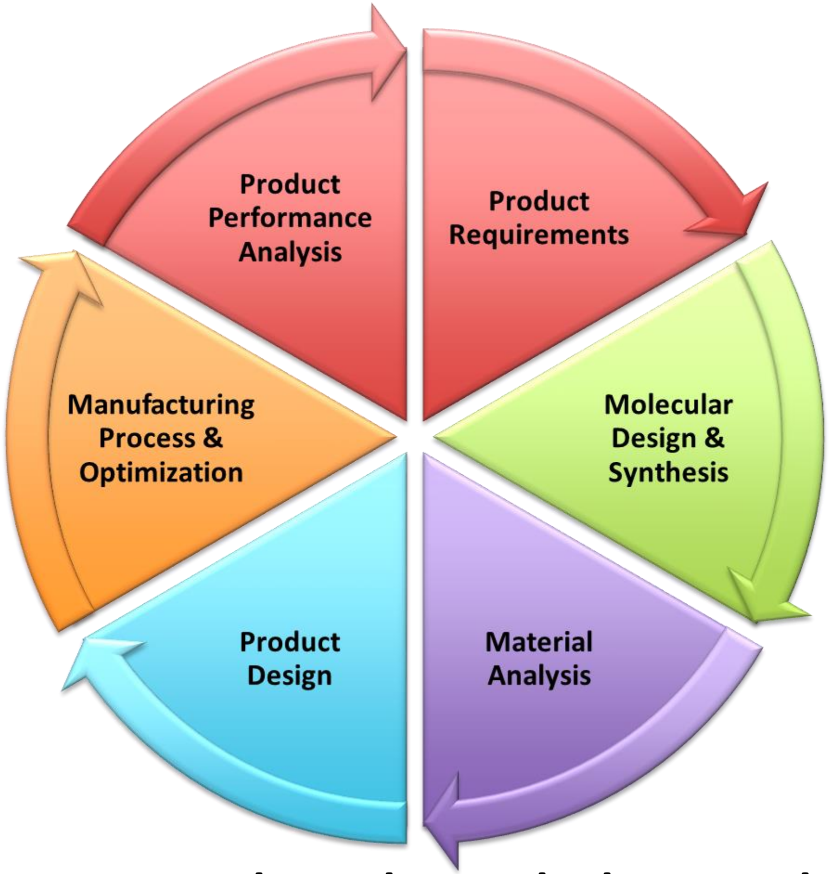


Rao, Liu, Roberson, Kong & Williams, *ASME Trans Journal of Manufacturing Science and Engineering* , 2015

From Molecules to Manufacturing

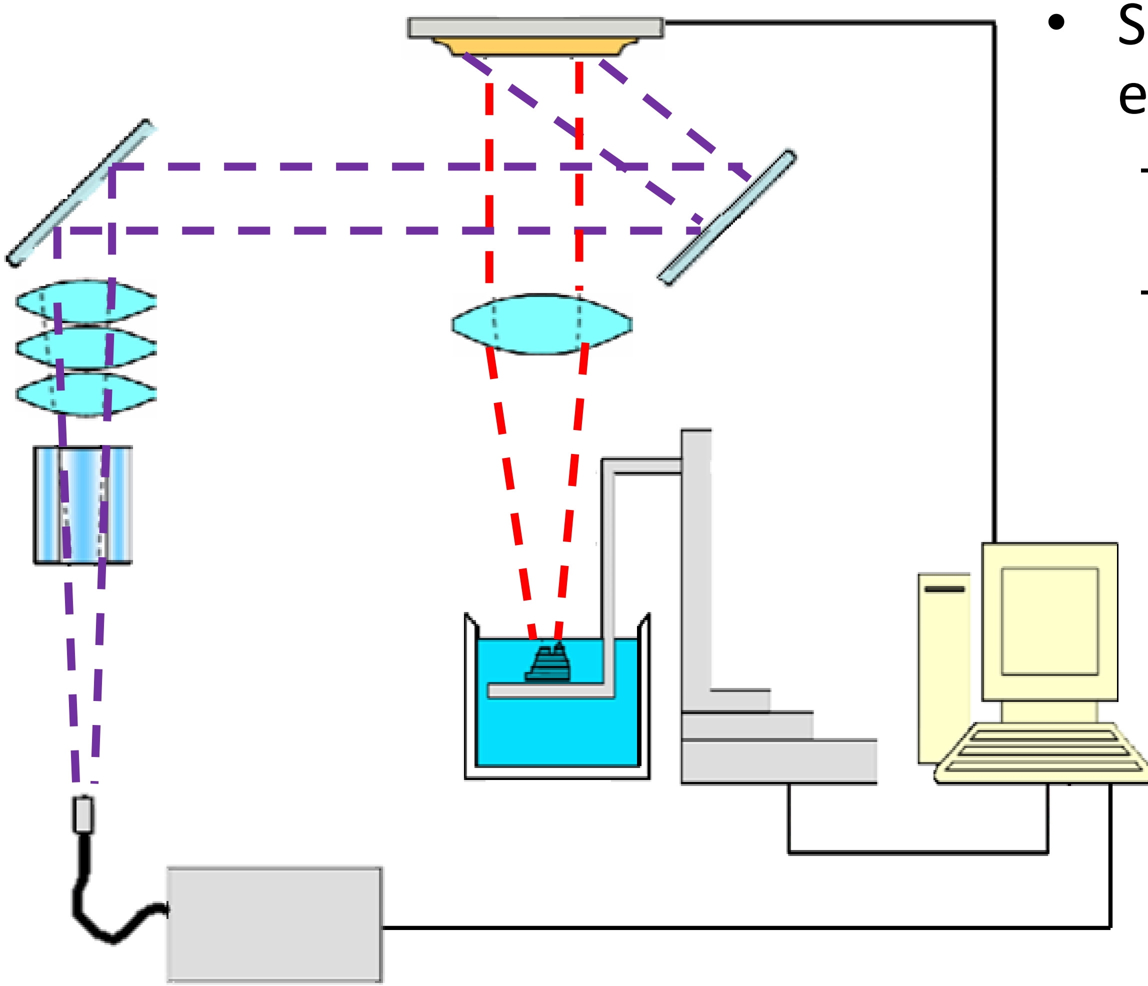


Opportunity to realize breakthrough products via ***concurrent design*** of ***polymer chemistry***, ***part geometry***, and ***manufacturing process***.

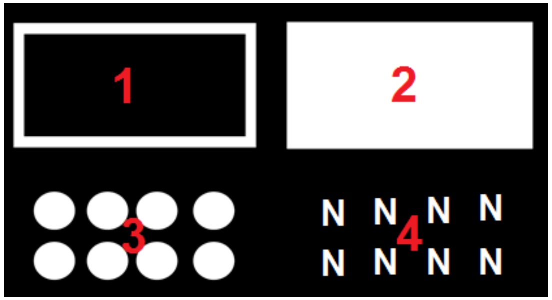
To achieve this goal, we need …

- **Pre-process measurements**
  - Materials screening methodologies
  - What makes a material printable?
  - Map characterization of raw material to process parameters
- **In-situ process measurements**
  - Process-structure-property relationships
  - How do process parameters affect the printed material?
  - What is the quality of the printing part’s shape & composition?
- **Post-process measurements**
  - Validate quality of final part shape and material
  - What are the properties of the printed part?

Projection Stereolithography



- Selective UV light exposure via DMD
  - White areas are exposed to UV light and cured
  - Black areas remain uncured liquid resin



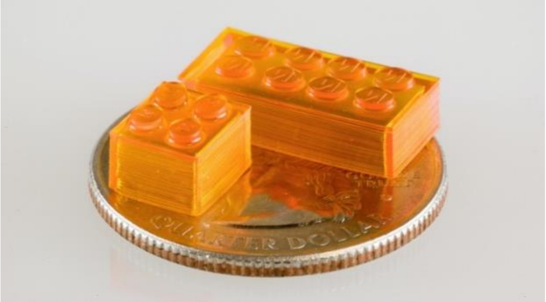
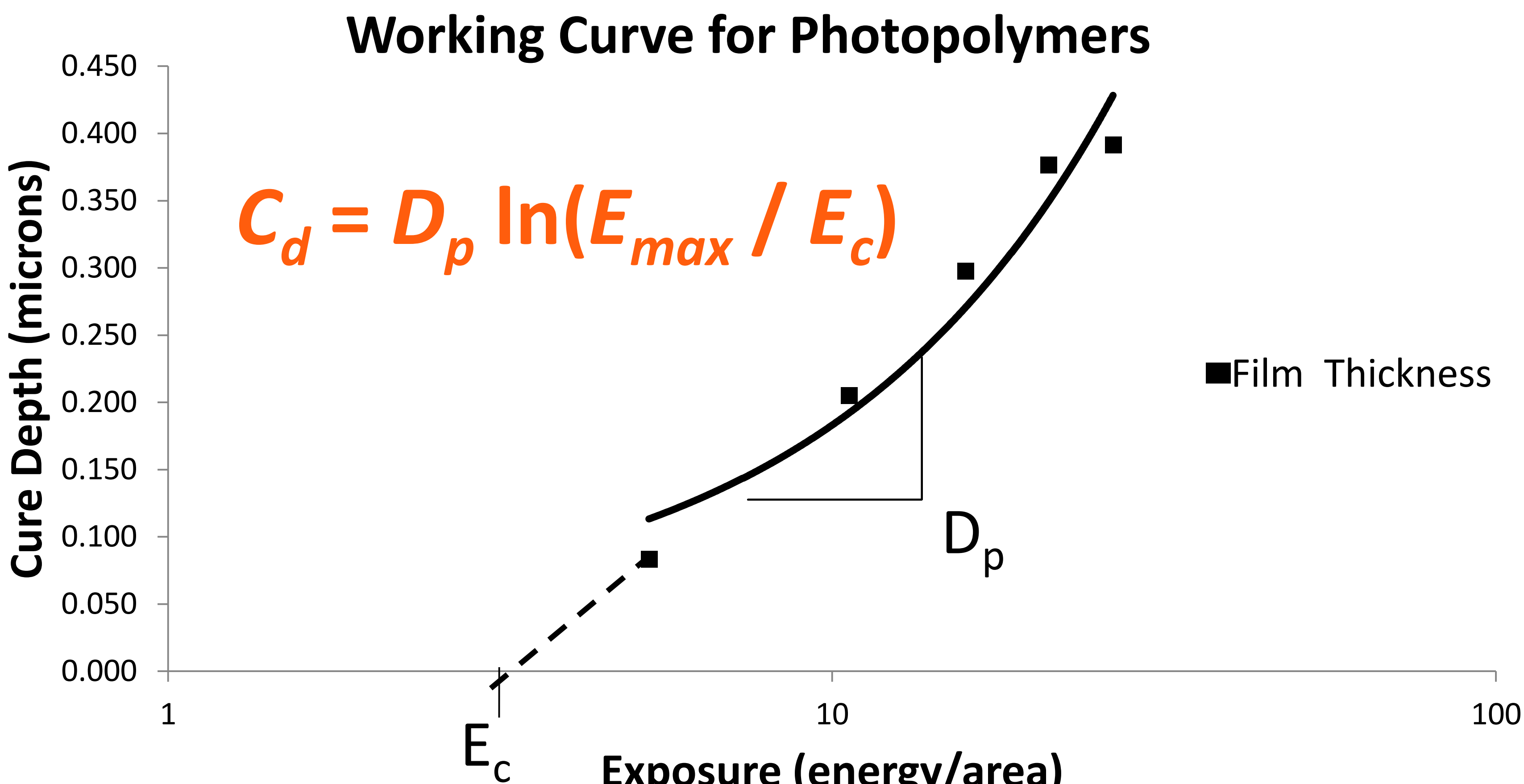


Image: https://nano-cemms.illinois.edu/materials/3d_printing_full

DREAMS

Relating Material Properties to Process Parameters



Jacobs, F. (1992). *Rapid Prototyping & Manufacturing: Fundamentals of StereoLithography* (First ed.), Society of Manufacturing Engineers.

DREAMS

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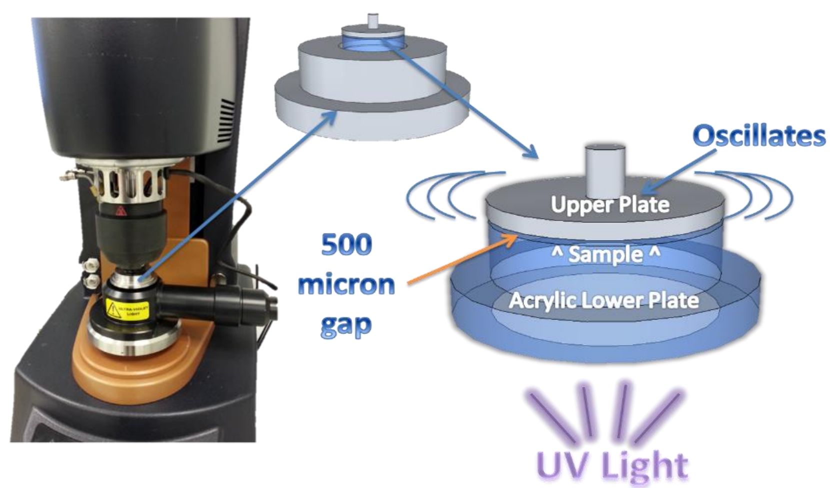
A photocuring accessory offers rheological characterization of UV-curable polymers

UV Curing Accessory

- High-pressure mercury light source for UV radiation
- UV wavelengths in the range of 320 to 500 nm
- UV-curing accessory with light guide, reflecting mirror assembly, and collimator



DREAMS

TA Instruments-Discovery Hybrid Accessories
http://www.tainstruments.com

Photorheology demonstrates decreasing photocured plateau modulus with increasing PDMS molecular weight

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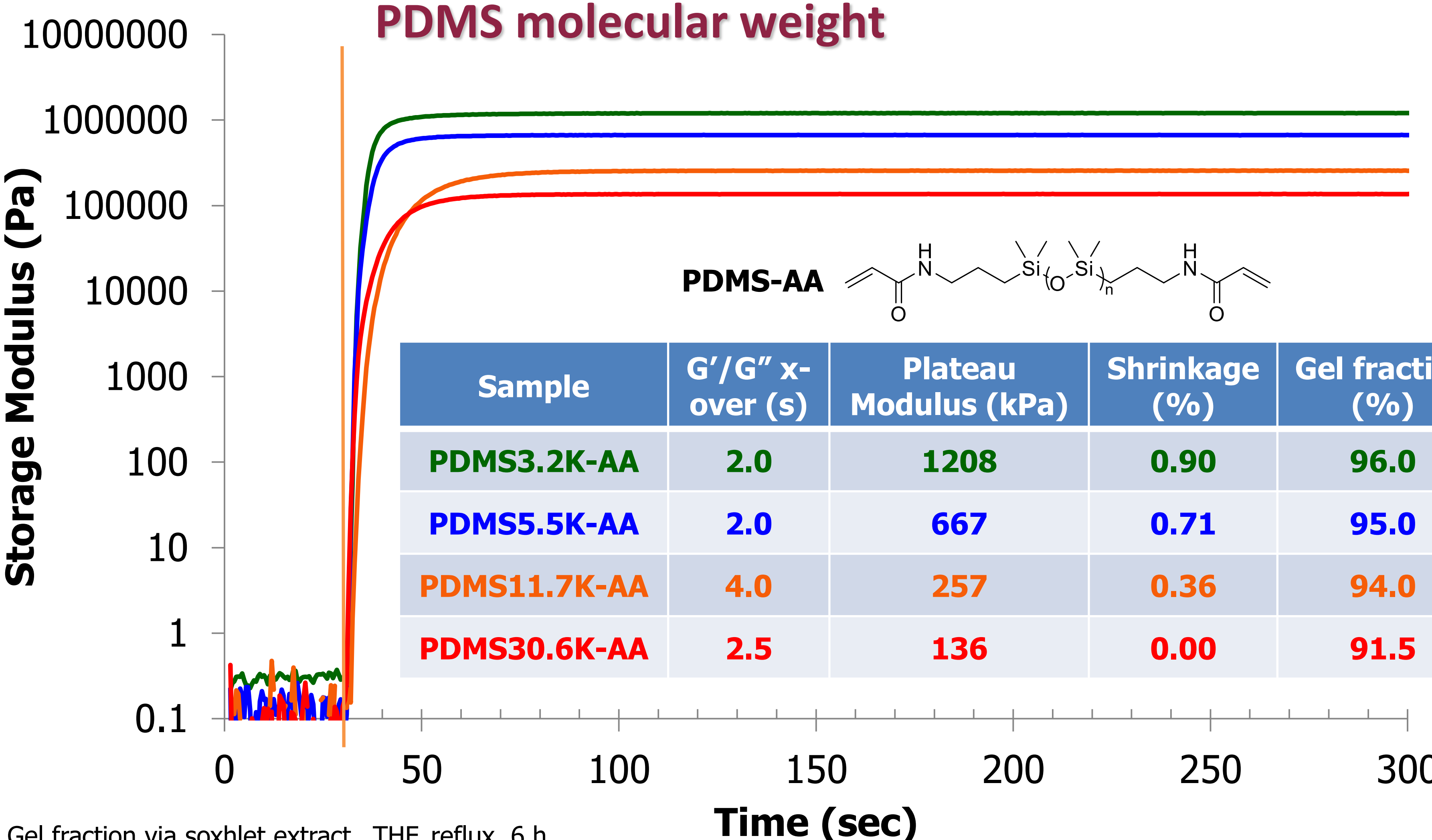

PDMS-AA

Sample	G′/G″ x-over (s)	Plateau Modulus (kPa)	Shrinkage (%)	Gel fraction (%)
PDMS3.2K-AA	2.0	1208	0.90	96.0
PDMS5.5K-AA	2.0	667	0.71	95.0
PDMS11.7K-AA	4.0	257	0.36	94.0
PDMS30.6K-AA	2.5	136	0.00	91.5

Gel fraction via soxhlet extract., THF, reflux, 6 h

TA DHR-2 with UV Curing Accessory, 20mm parallel plate, 500 μm gap, 1 Hz, 0.3 % strain, N_2

2 wt % DMPA, 7.0 mW/cm^2, 30 s delay

DREAMS

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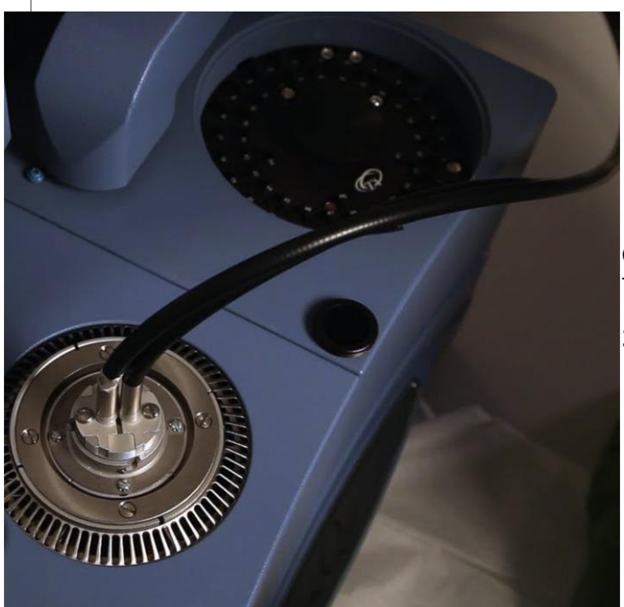
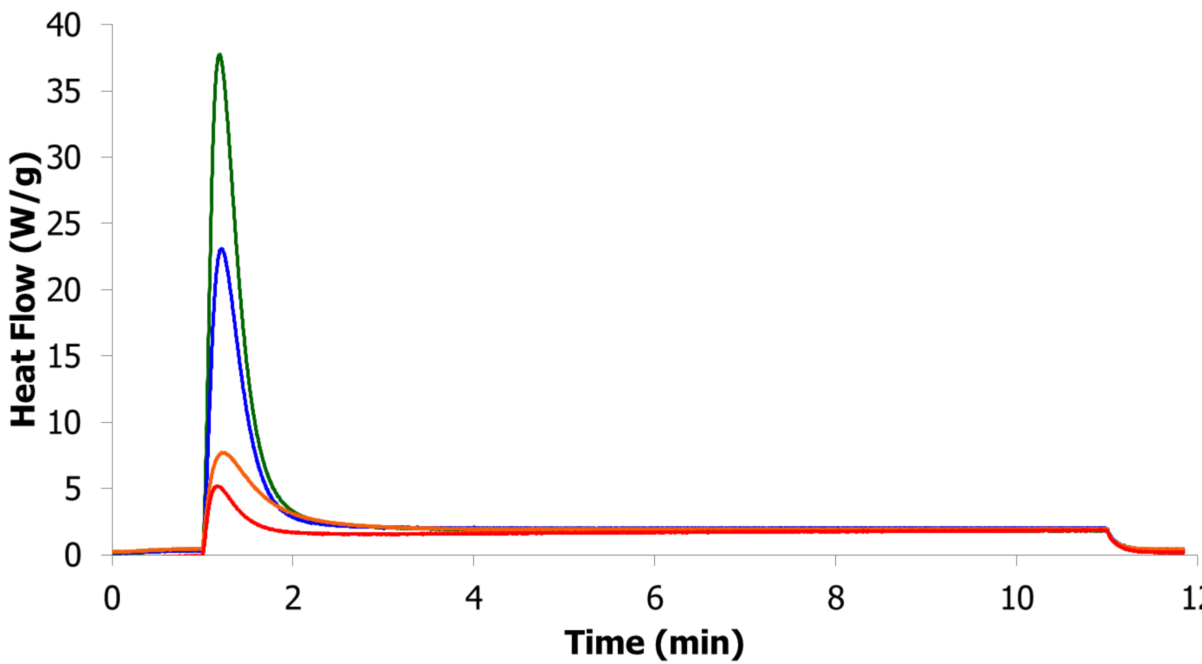
A photocuring accessory offers calorimetric characterization of UV-curable polymers





Q_A for acrylate homopolymerization = 86.0 kJ/mol
TA Q2000 DSC with photocuring accessory, 25 °C, N_2, n = 3

2 wt % DMPA, 5.0 mW/cm^2, light on at 1 min, off at 11 min

DREAMS

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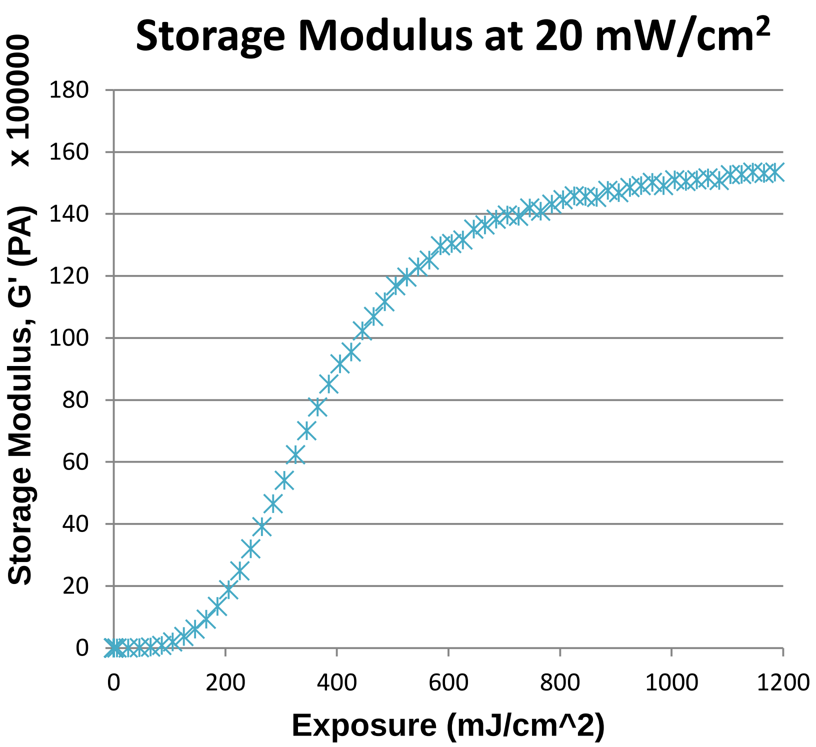
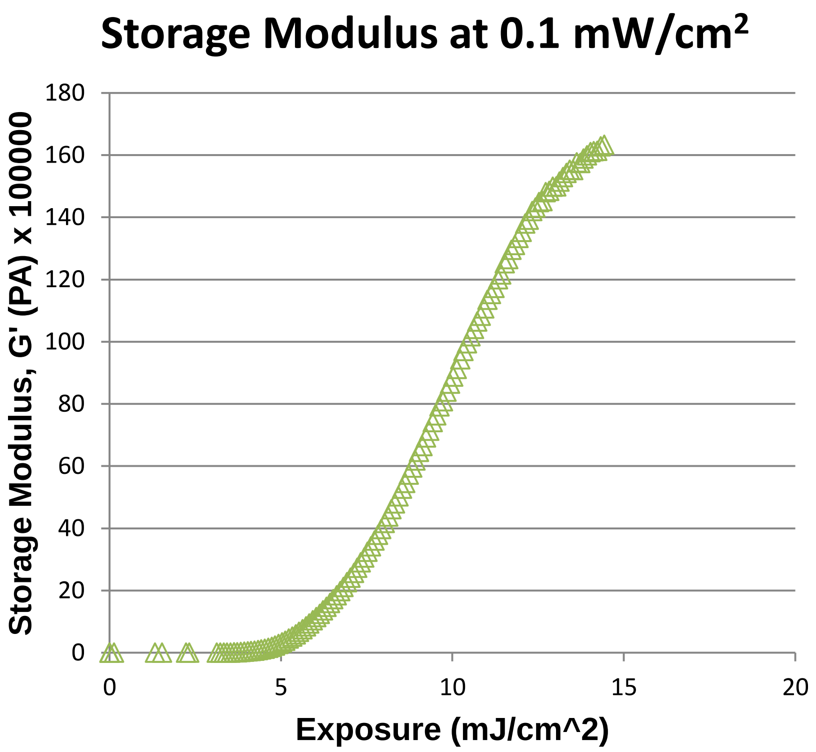
Critical Exposure & Intensity

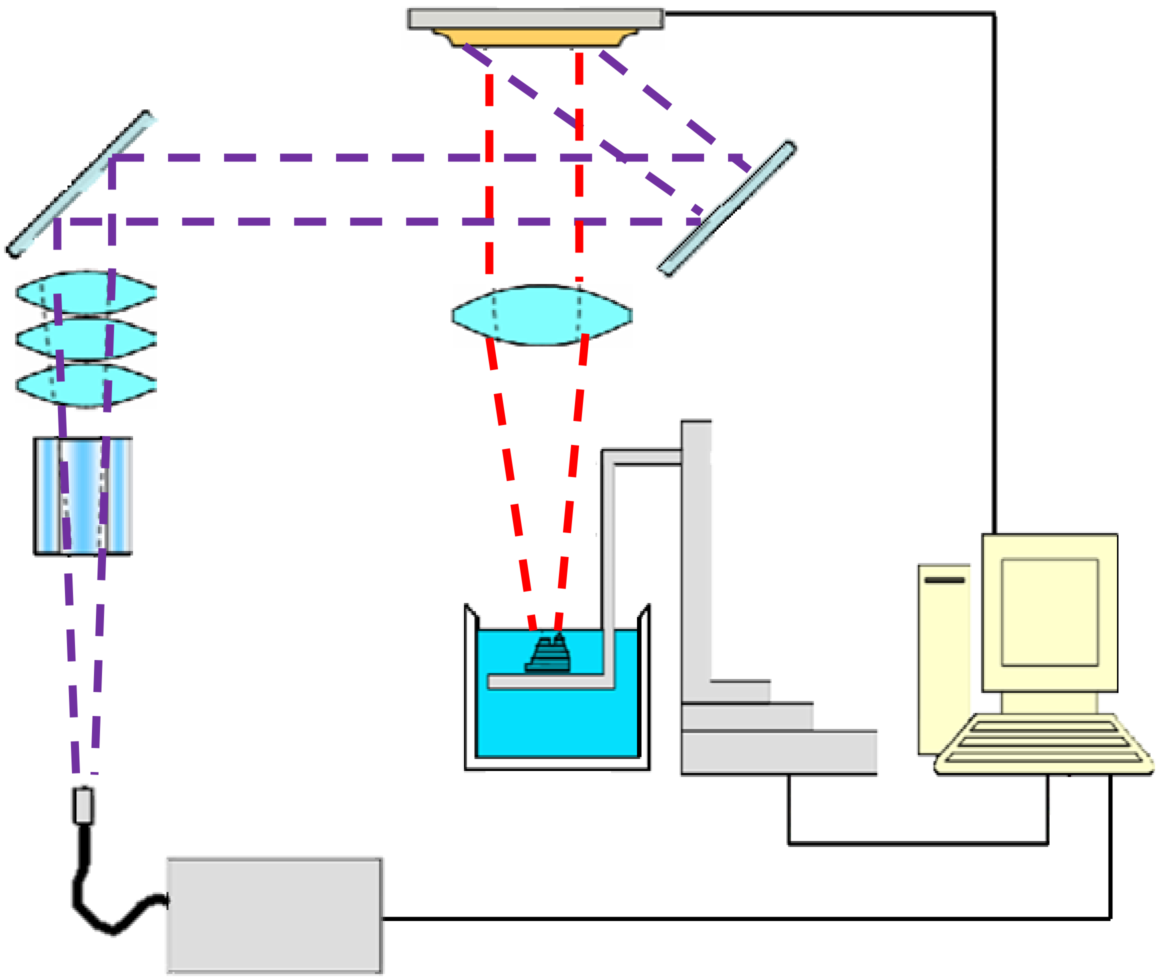




- Photorheometry was used to compare critical exposures of polymer (with 2wt% QDs) cured at different intensities.
- Less total energy is needed for lower intensities

DREAMS

Relevant Process Variables

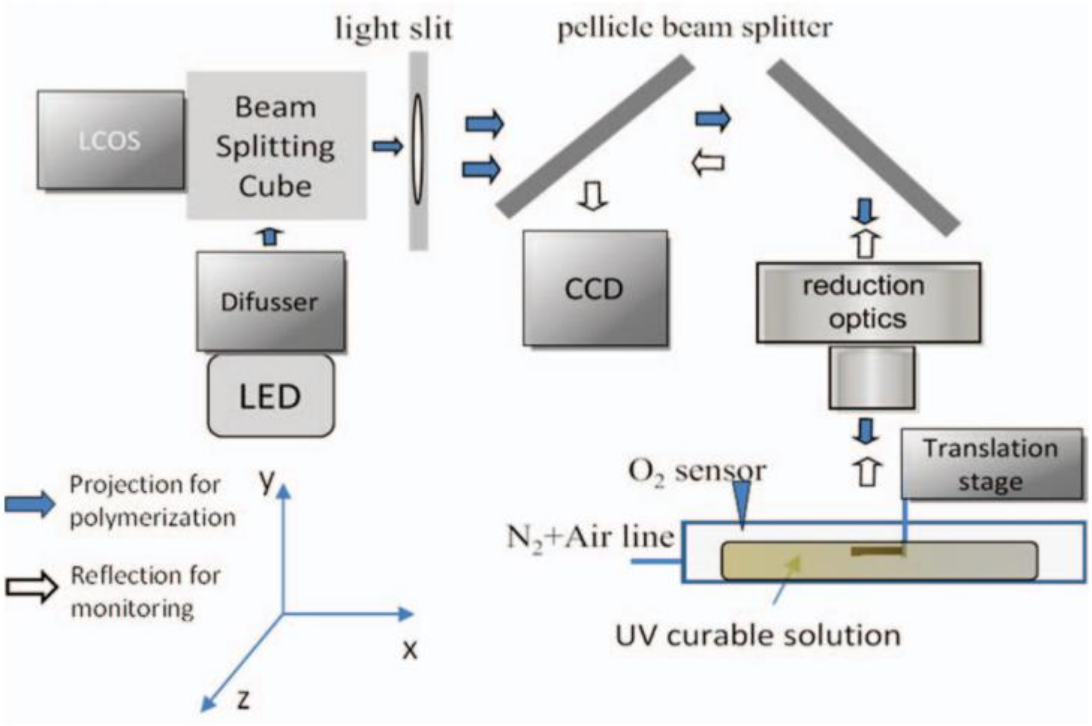


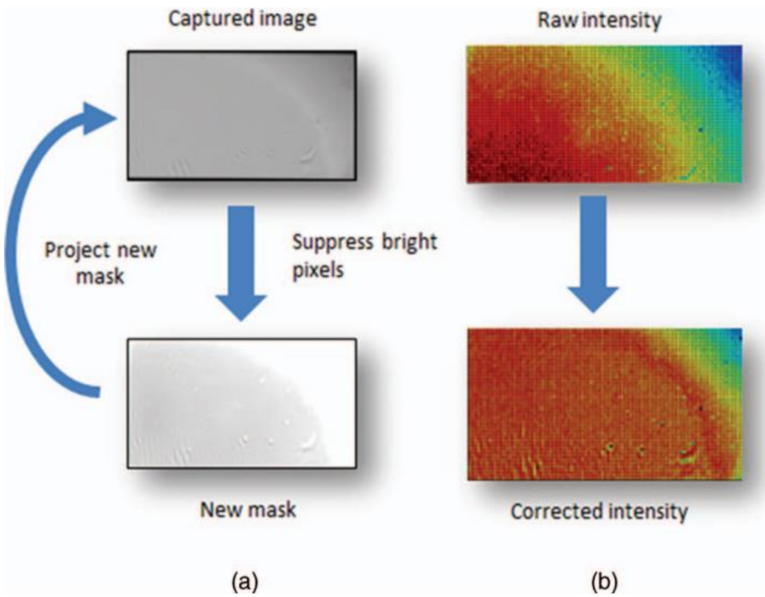
- Exposure
- Wavelength
- Intensity
- Oxygen
- Temperature
- Humidity
- Material composition
  - Photo initiator
  - Photo absorber
  - Fillers
  - Age

DREAMS

In-situ Measurements for Photopolymerization

UV Intensity Correction via CCD



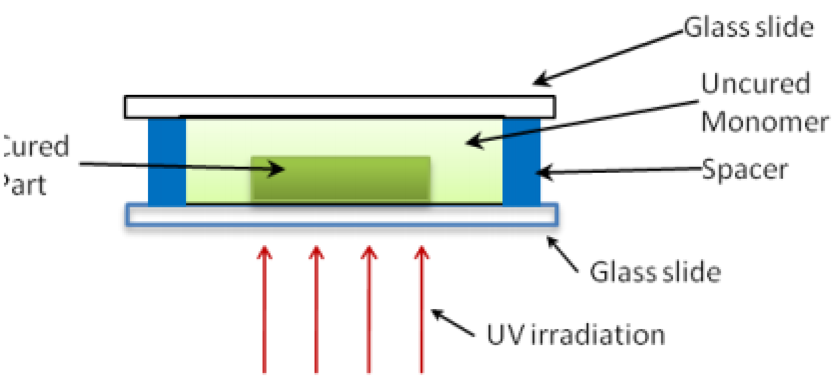


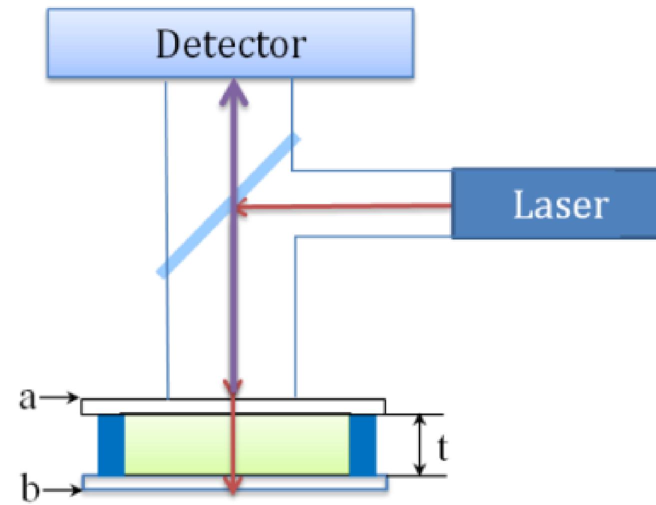
Zheng et al., *Review of Scientific Instrument*, 2012

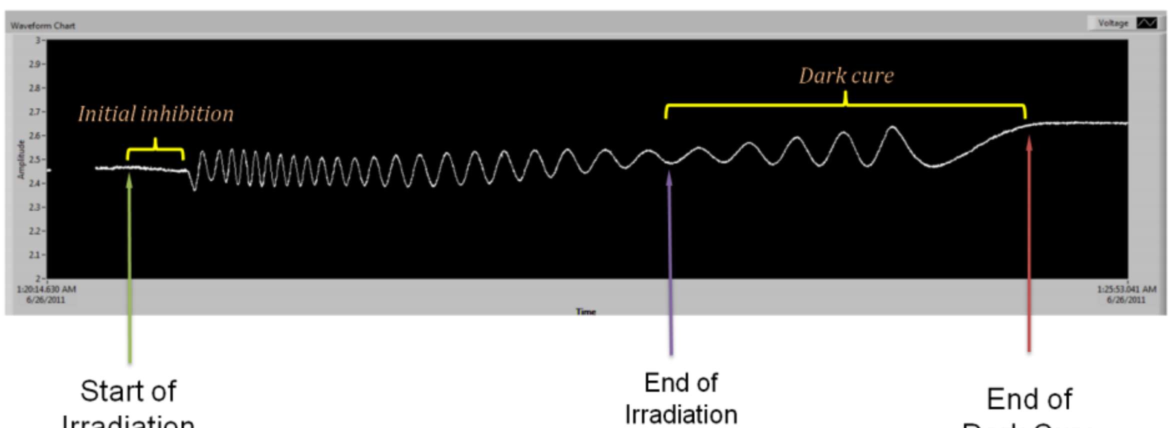
DREAMS

In-situ Measurements for Photopolymerization

In-situ Interferometry



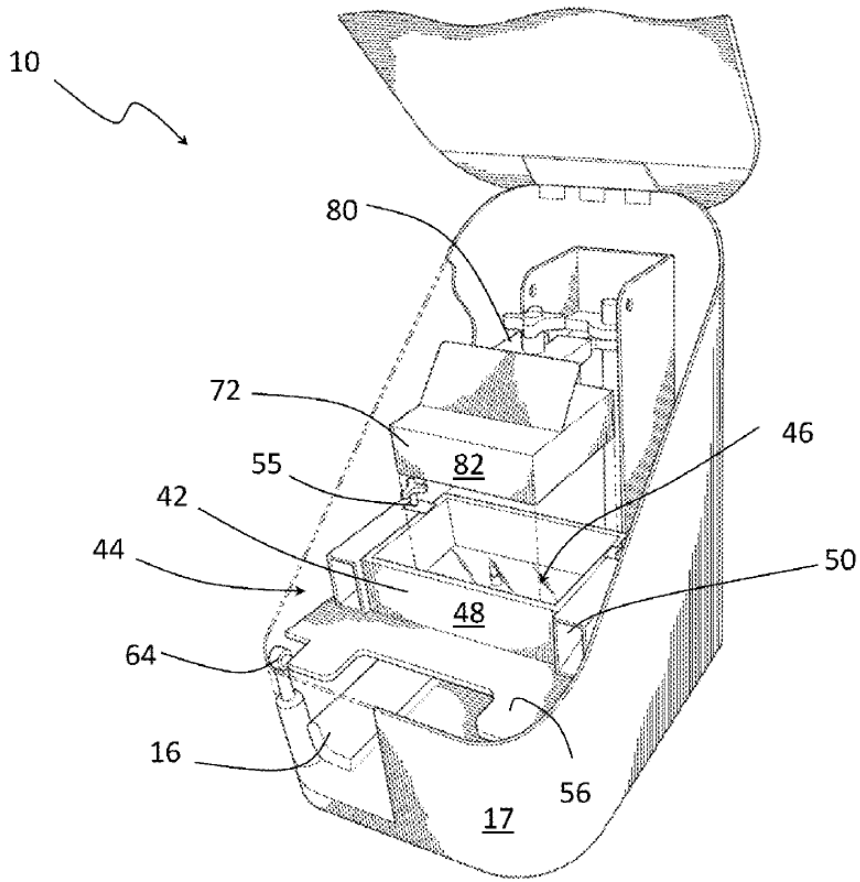




Jarawala, Schwerzel, Rosen, *SFF*, 2011
Jones, Kwatra, Jariwala, Rosen, *SFF*, 2013

In-situ Measurements for Photopolymerization

In-situ Interferometry + Closed Loop Control



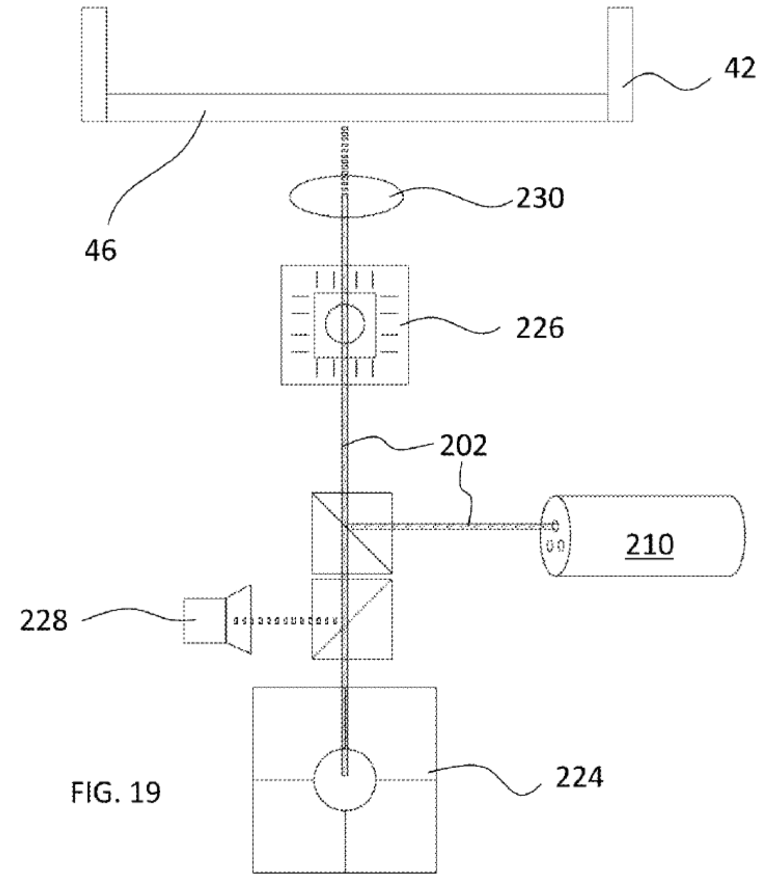
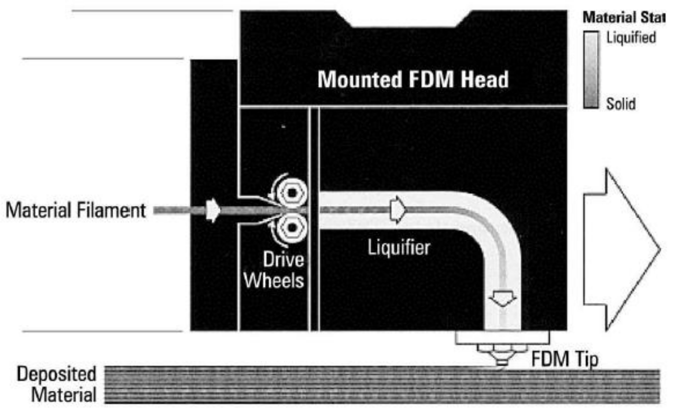


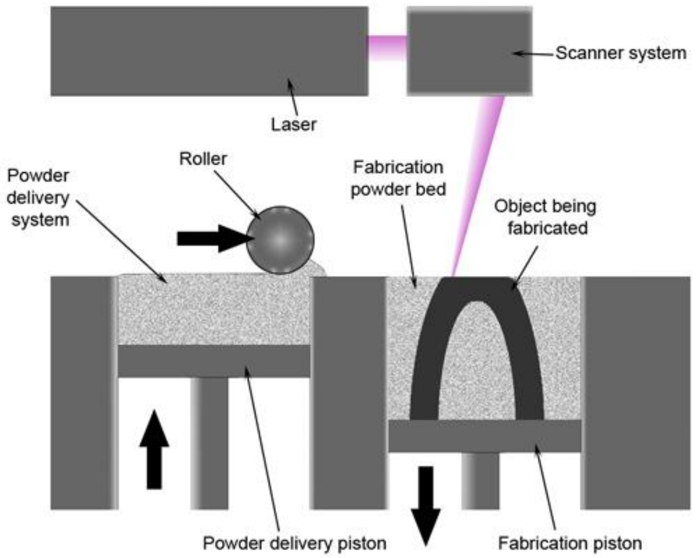
FIG. 19

Old World Labs; US 2015/0183168

Complexity of Polymer AM

Figure 1 Fused deposition modeling process





- **Material**
  - Rheology (shear & temp)
  - Stiffness
  - Degradation (recycling)
- **Environment**
  - Temperature
  - Humidity
  - Heat transfer
- **Print**
  - Melt temperature
  - Shear
  - Toolpath
- **Part**
  - Heat transfer from shape & toolpath
  - Location

DREAMS

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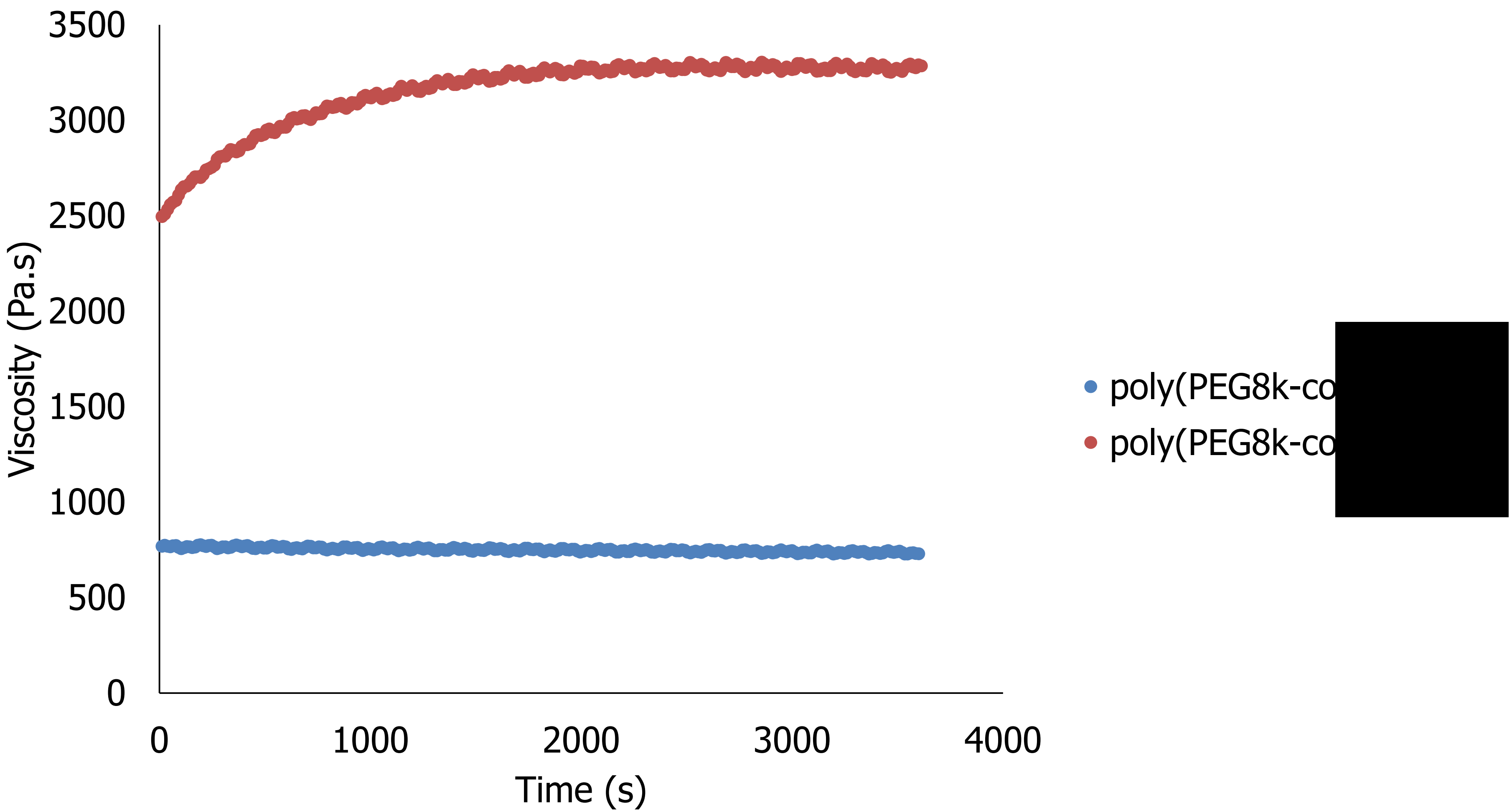
Rheology can be affected by thermal history, humidity, time



Ares G2 rheometer, 1 % strain, 1 rad/s, Na: 77 °C, Ca: 120 °C

DREAMS

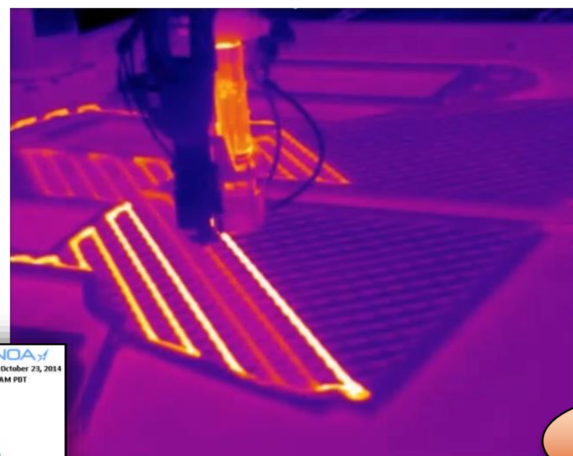
Process & Materials

Big Area Additive Manufacturing

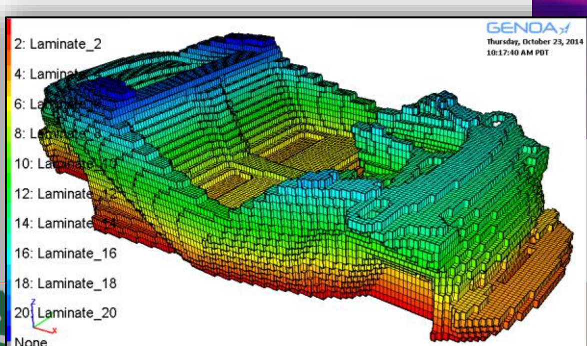
Printability

Structural Integrity

Analyze



Model



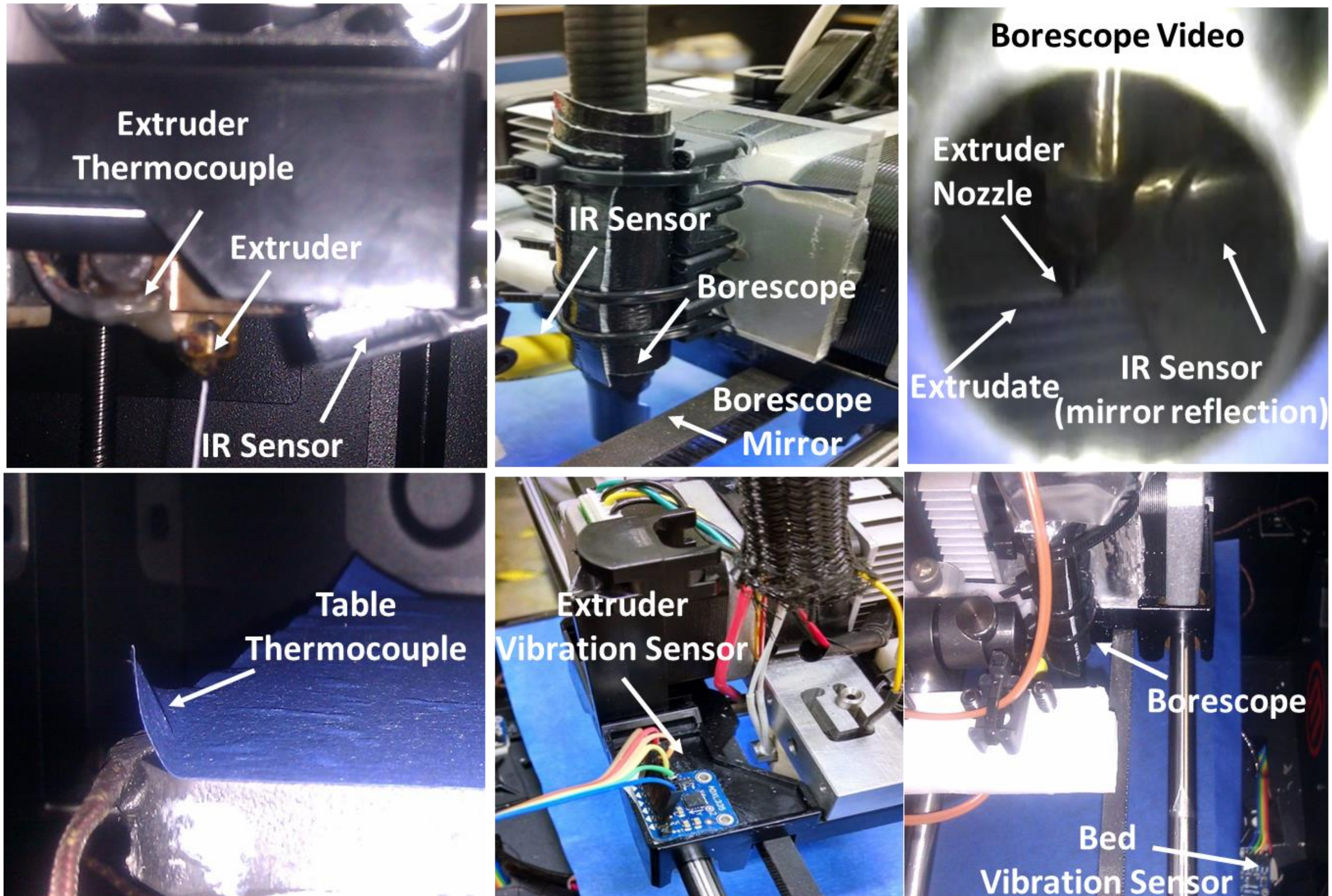
Material Development

Talagain et al., *SAMPE Journal*, 2015

Heterogeneous Sensing + Data Fusion

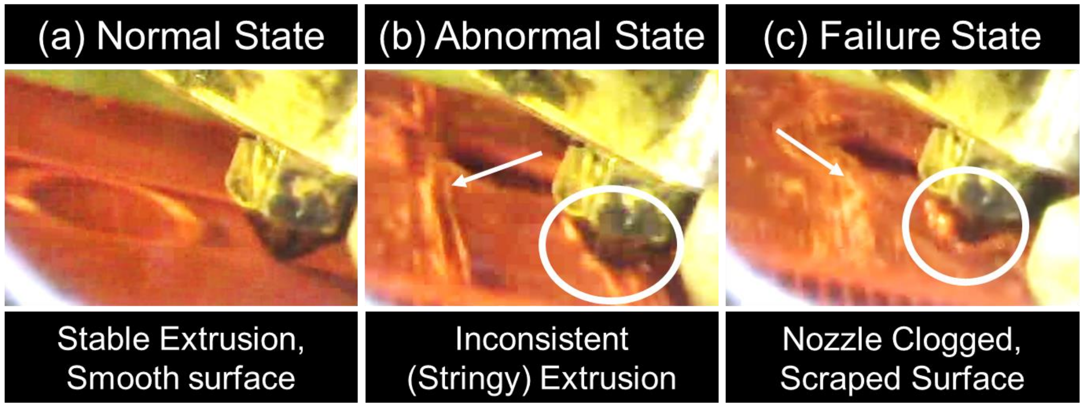
13 sensors are installed on the machine.

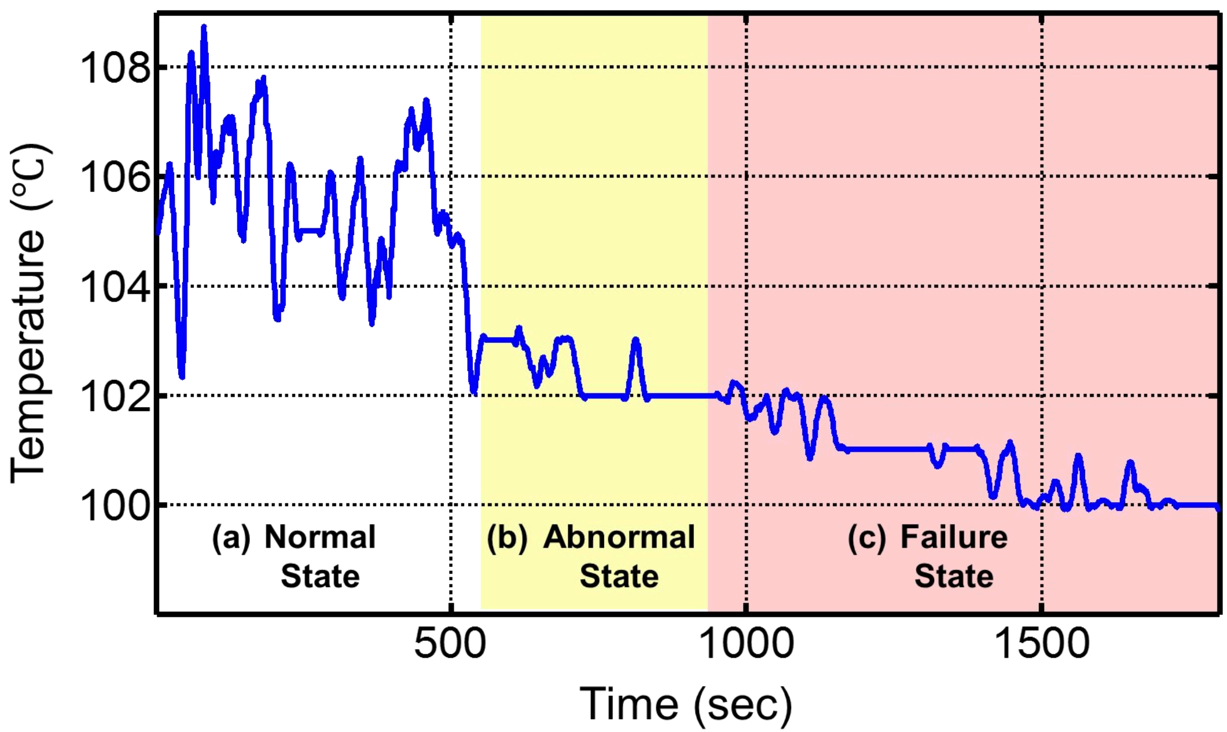
Which are useful? Can we use all of them? Or should we use a few of them?

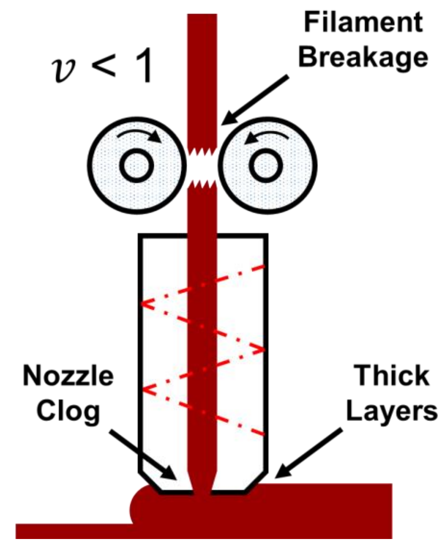


Rao, Liu, Roberson, Kong & Williams, *ASME Trans Journal of Manufacturing Science & Engineering*, 2015

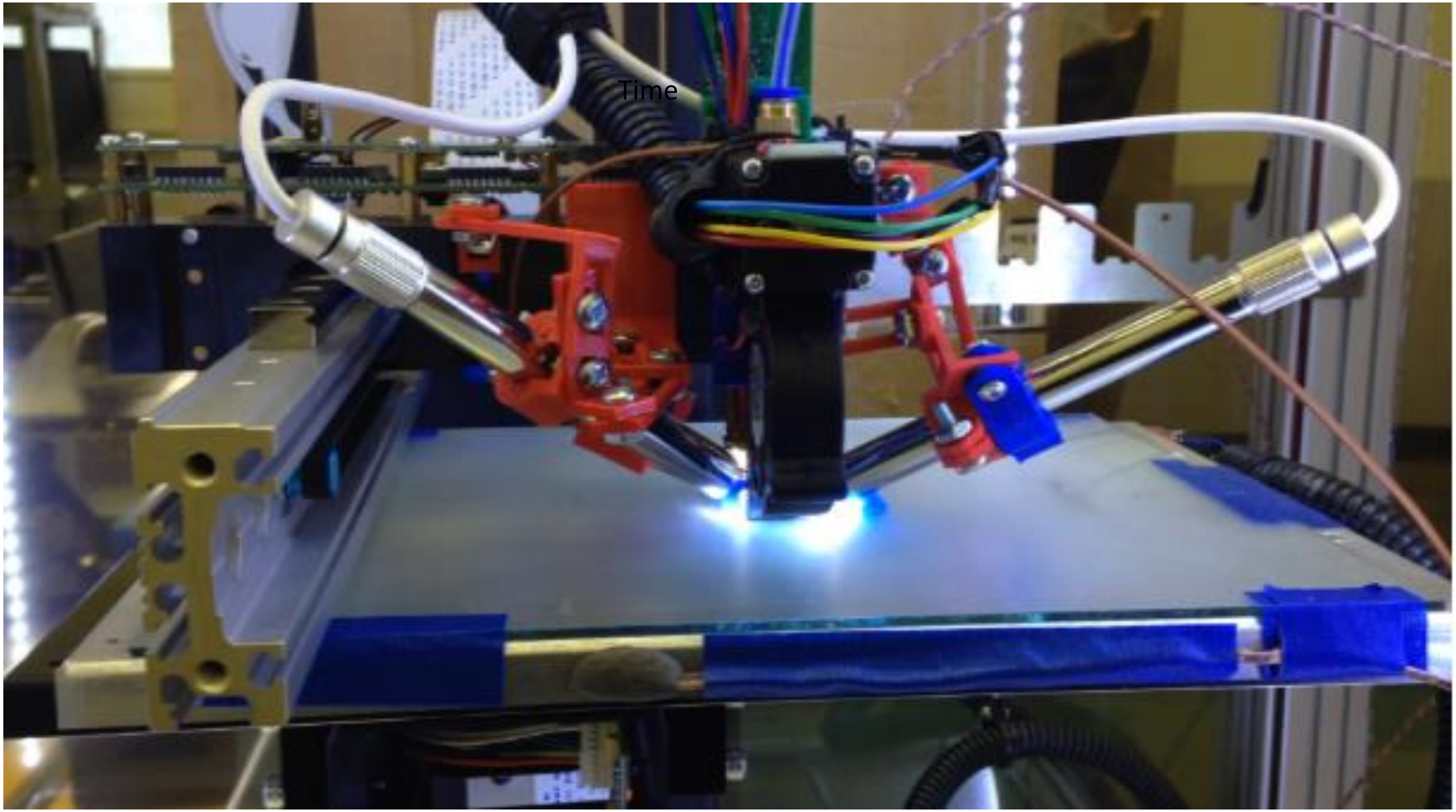
Monitoring Extruder Temperature





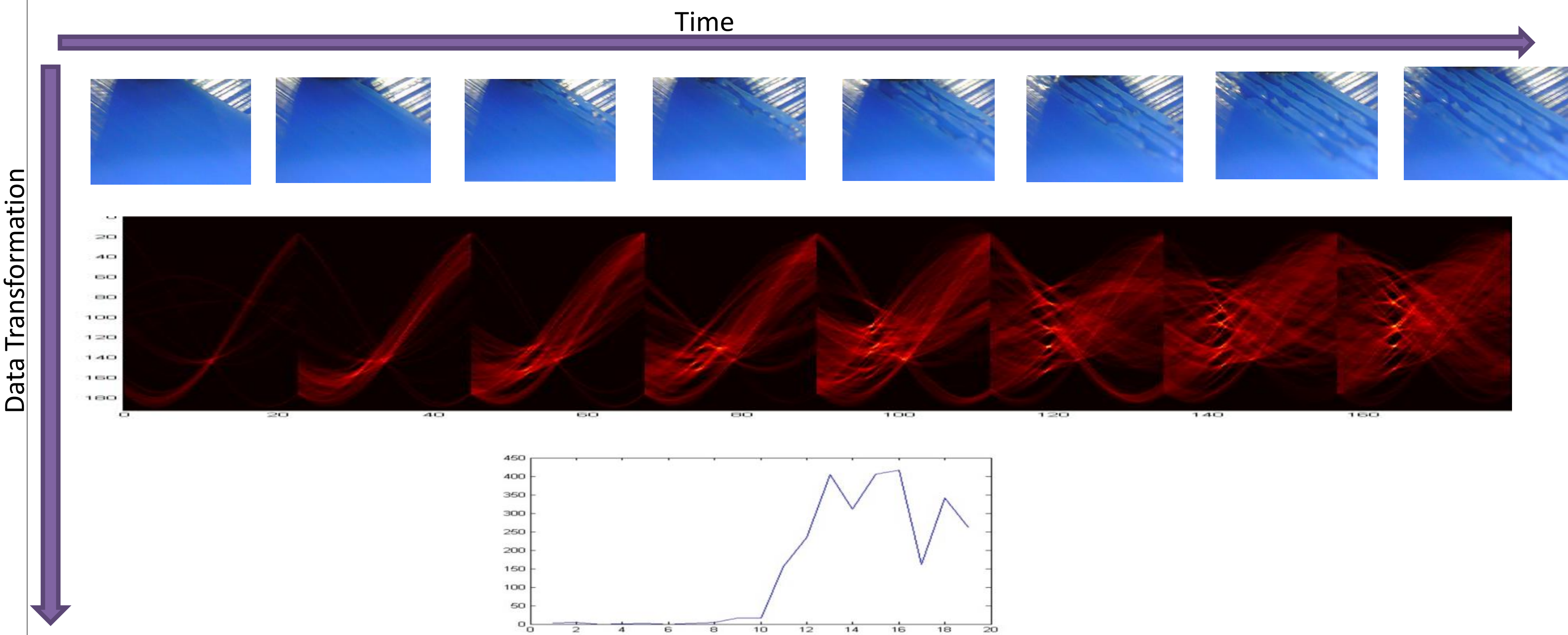


Monitoring Video



Digital Microscope based Machine Vision System

Monitoring Video



Digital Microscope based Machine Vision System

Virginia Tech
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Looking ahead…

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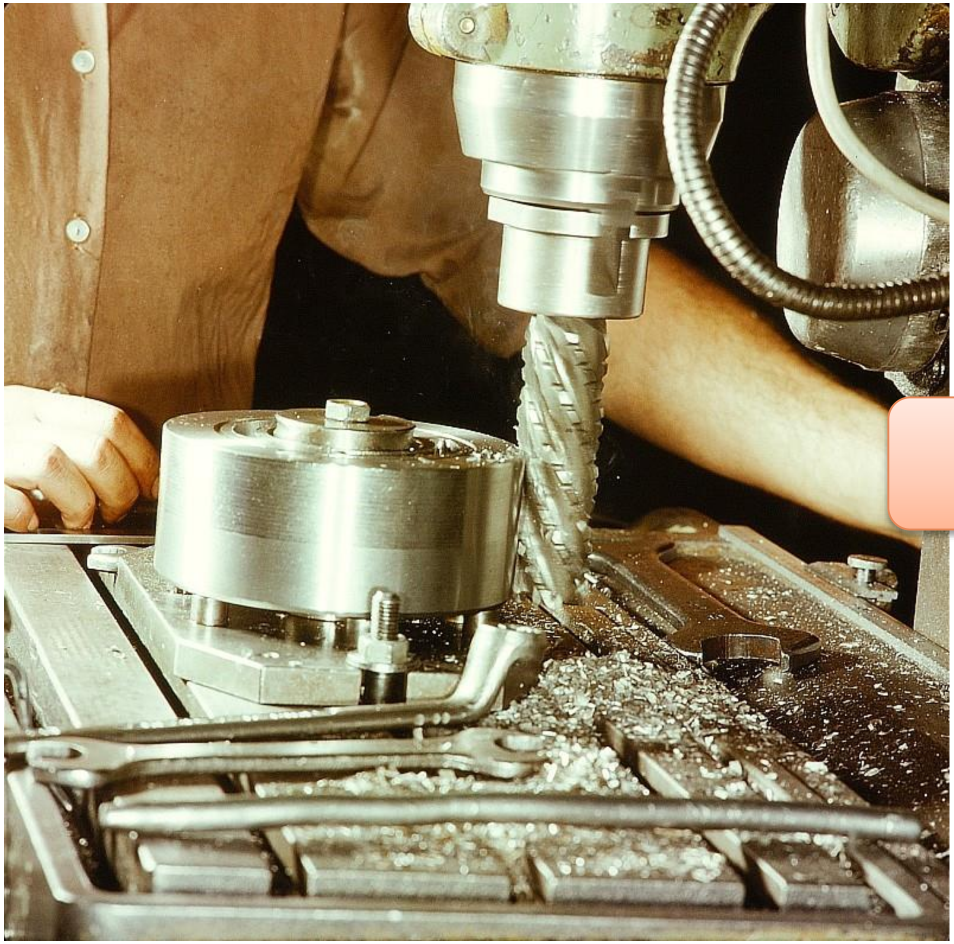


vs.

AM enables a designer to specify material location and material properties on a voxel-by-voxel basis.

DREAMS

General AM Embedding Process

1. Design channels
2. Pause build
   (& remove support)
3. Embed component
4. Anchor component
5. Resume build

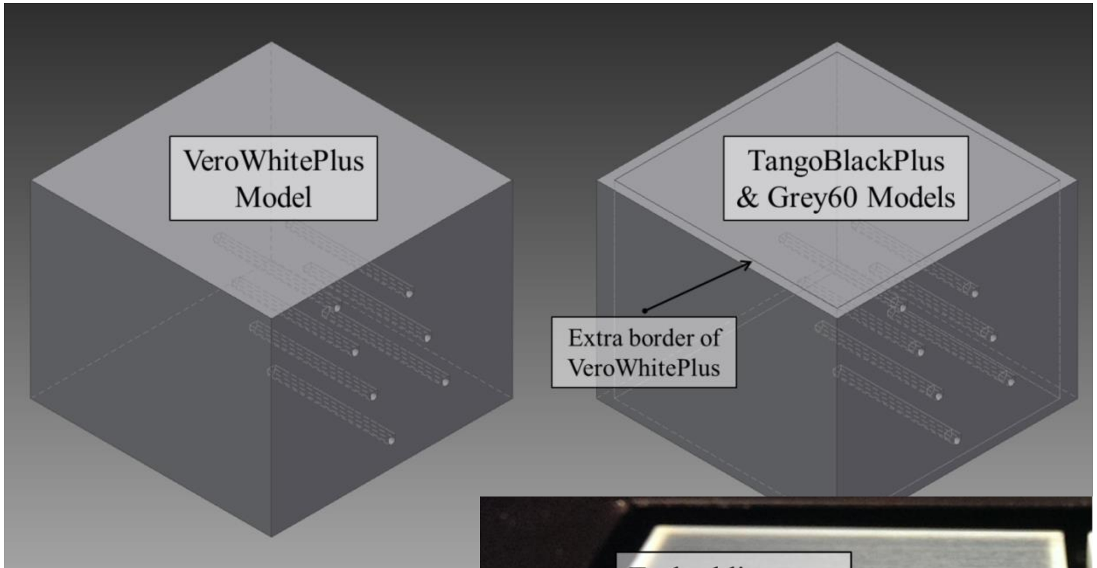
Meisel, Elliott, Williams, 2014, *Journal of Intelligent Systems & Structures*

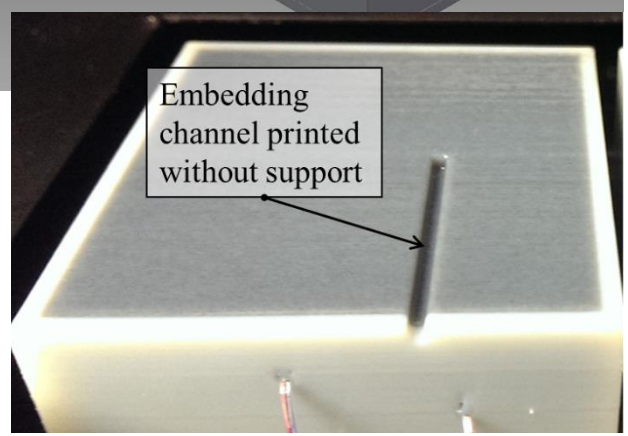
DREAMS

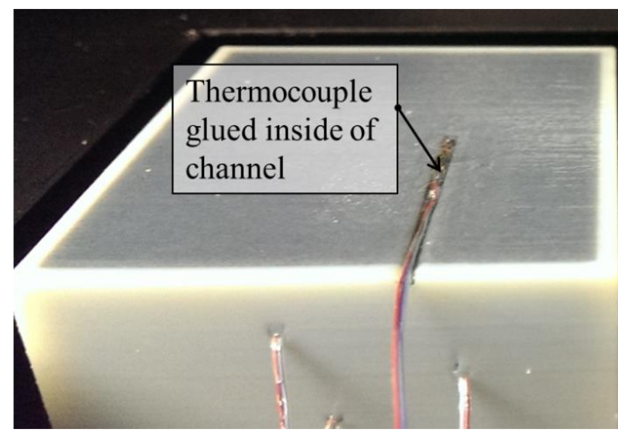
Stiltner, Elliott & Williams, 2011, *Int'l Solid Freeform Fabrication Symposium*

Embedded Thermocouples

CAD models - channels for thermocouples



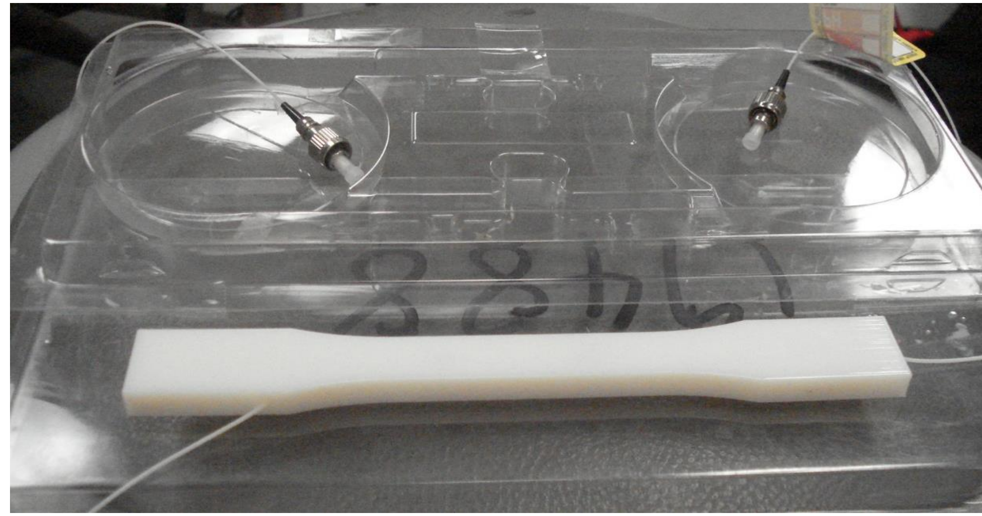


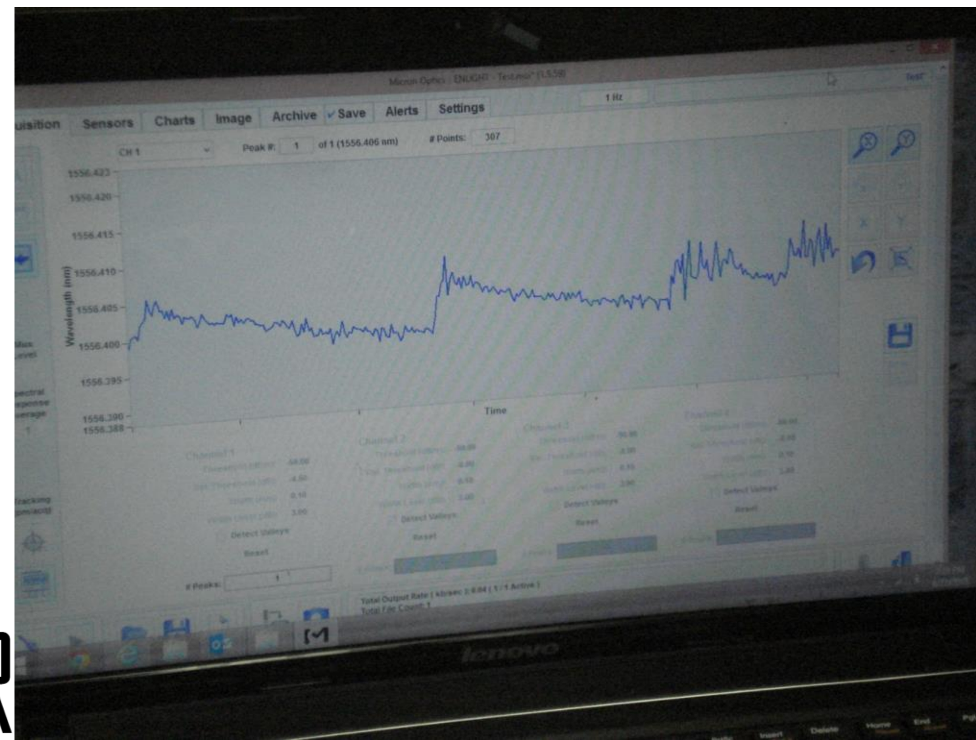


Thermocouples embedded during build process

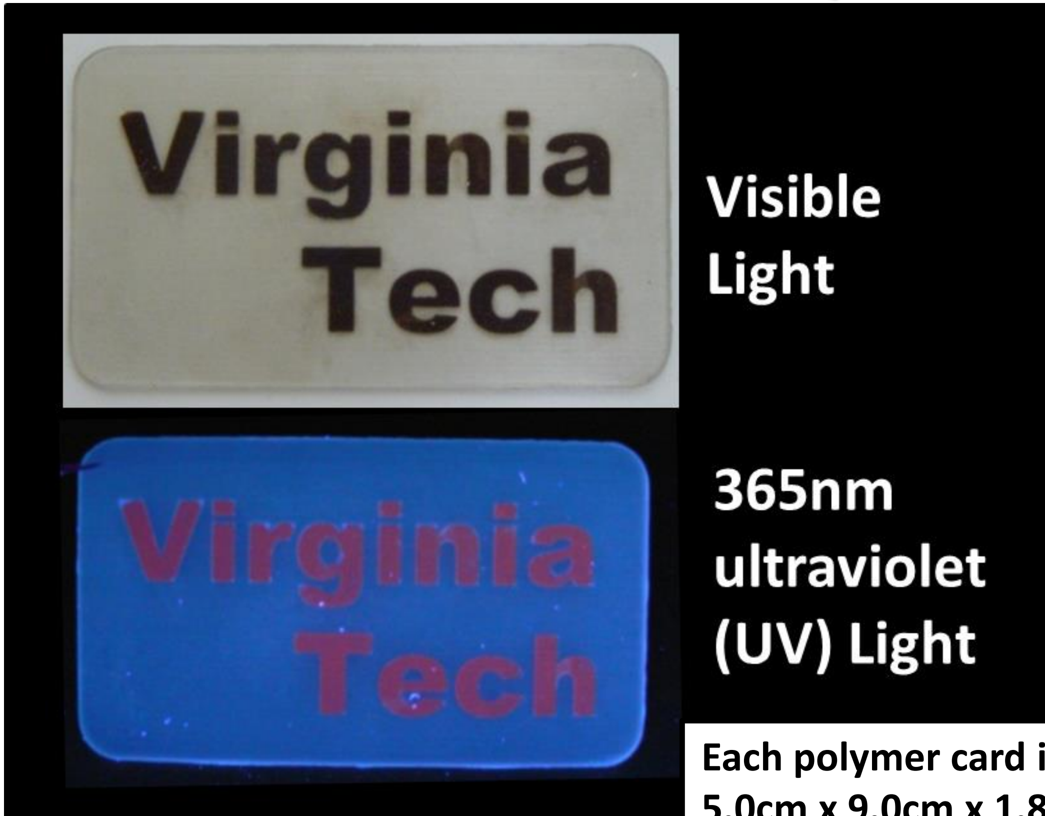
DREAMS

Embedded Fiber Sensor

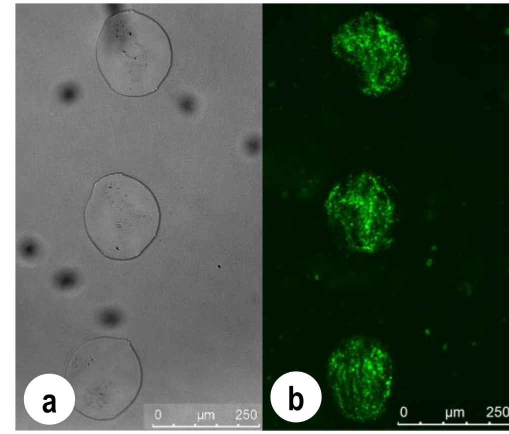




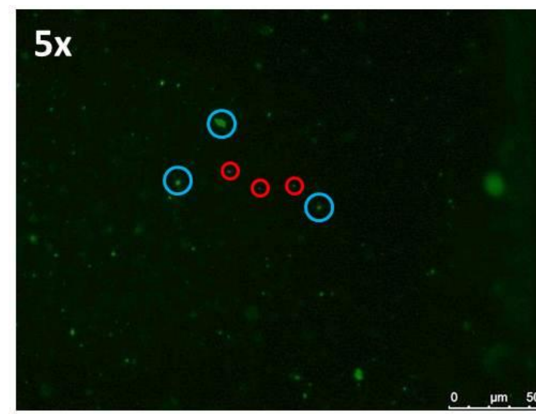
Material as a Sensor: Jettable Quantum Dot Photoplymer



Each polymer card is 5.0cm x 9.0cm x 1.8mm



Images of jetted QD Nanoink in (a) visible and (b) UV light



Fluorescent microscope images of QD nanoparticles in 3D Printed part.

Ivanova et al., *Add Mfg*, 2014; Elliot et al., *Adv Eng Mat,* 2013

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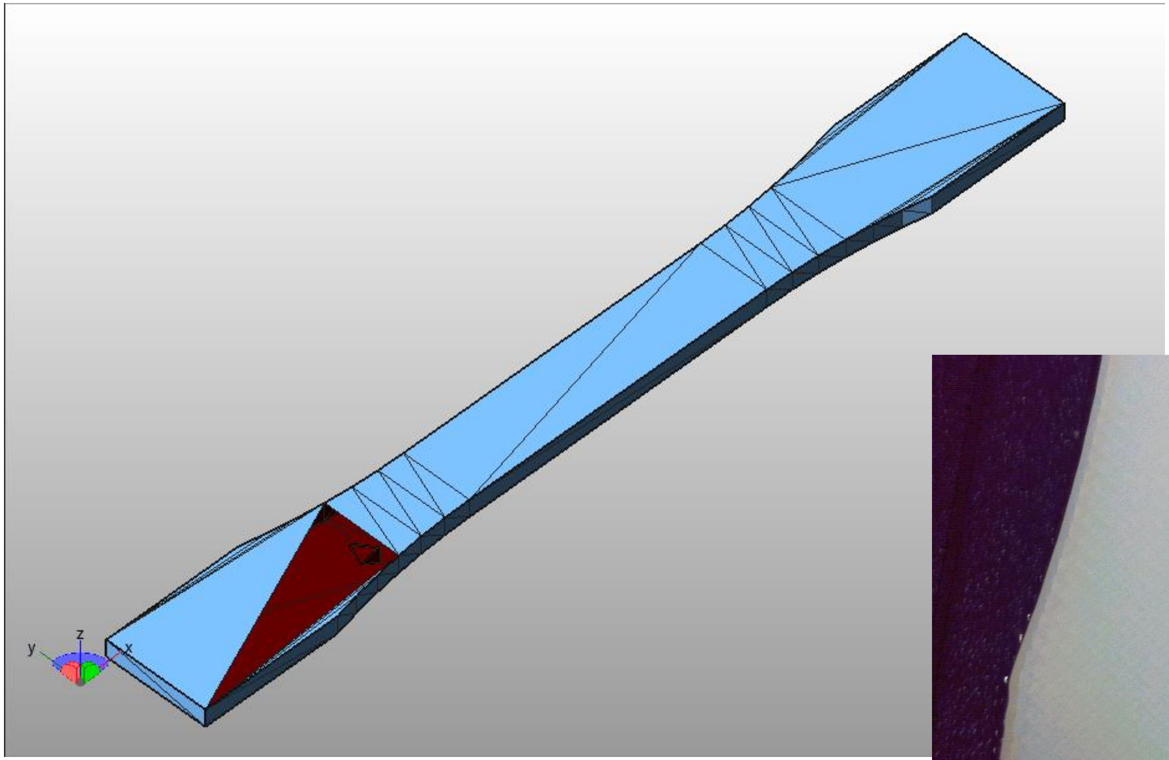
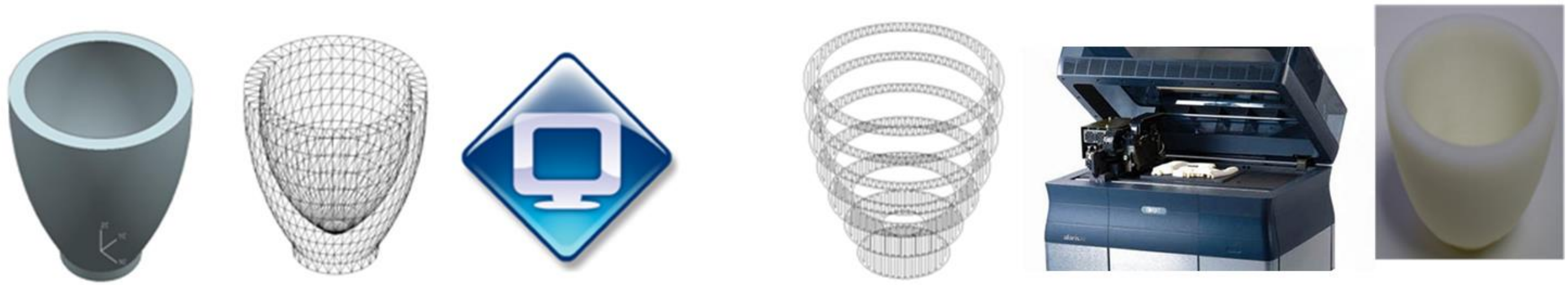
AM Cyber-Physical Security

STL as an Attack Vector

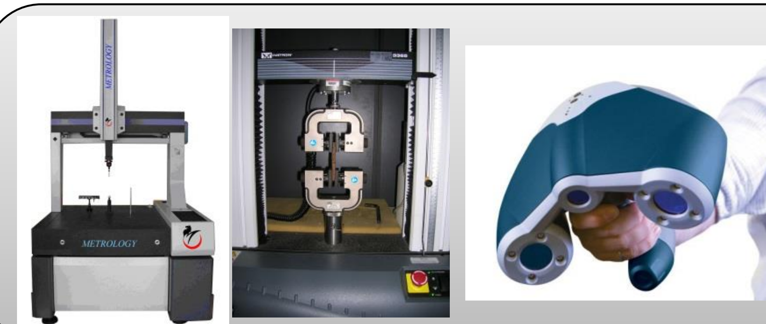


DREA

Security of Quality Control



3D Cad Model | .STL File | Slicing Software | Layer Slices & Tool Path | 3D Printer | 3D Object



Quality Control

- **Replace / modify firmware**
- **Machine tampering / modification**
- **Alter recorded data sets**

(Wells & Camelio)

DREAMS

We must consider the cyber security of our in-situ measurements.

Side channel measurements are as important as closed-loop control.

DREAMS

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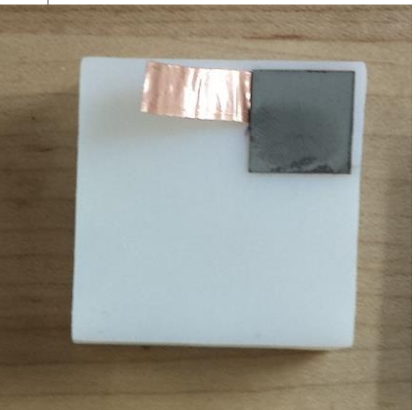
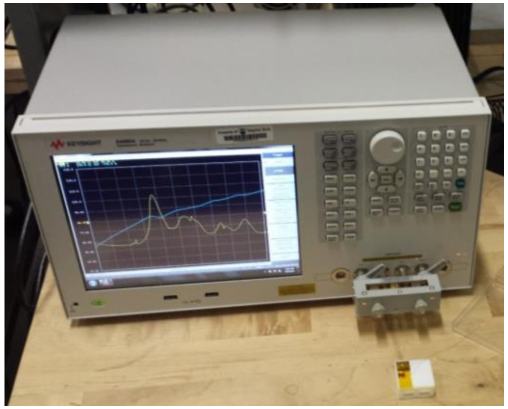
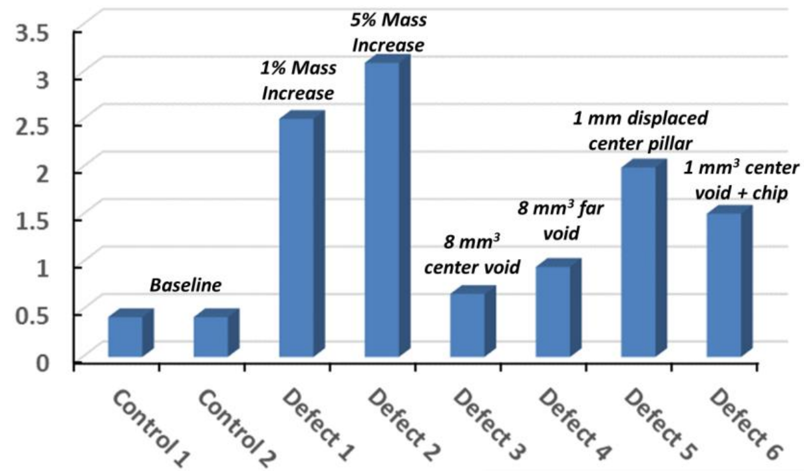
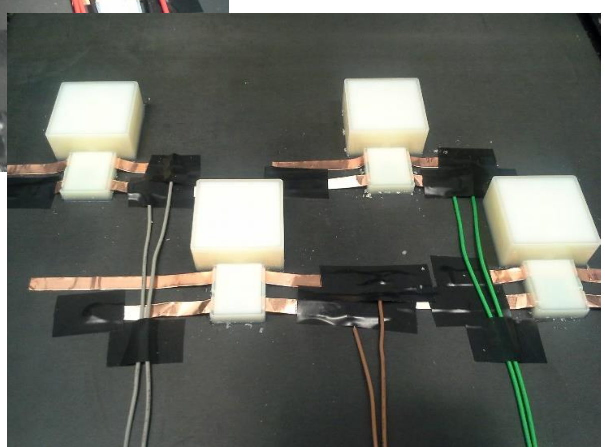
In-Situ Impedance-based Monitoring

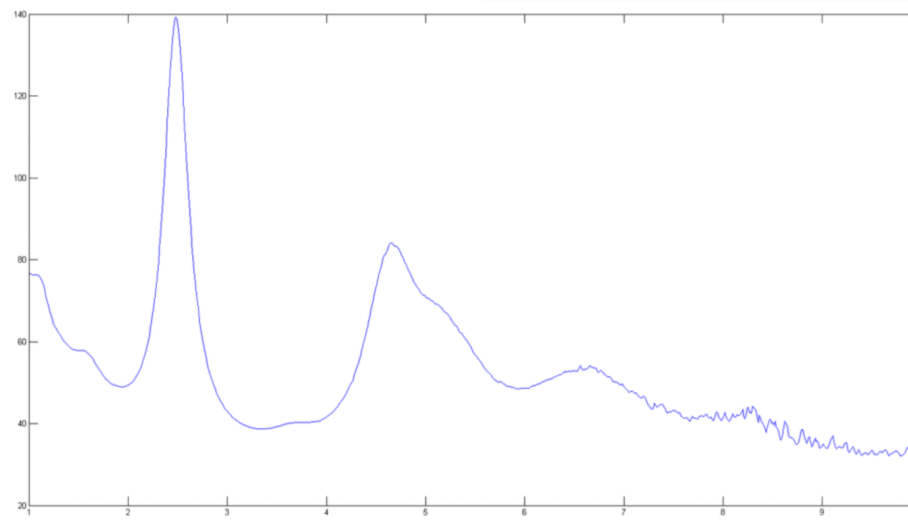






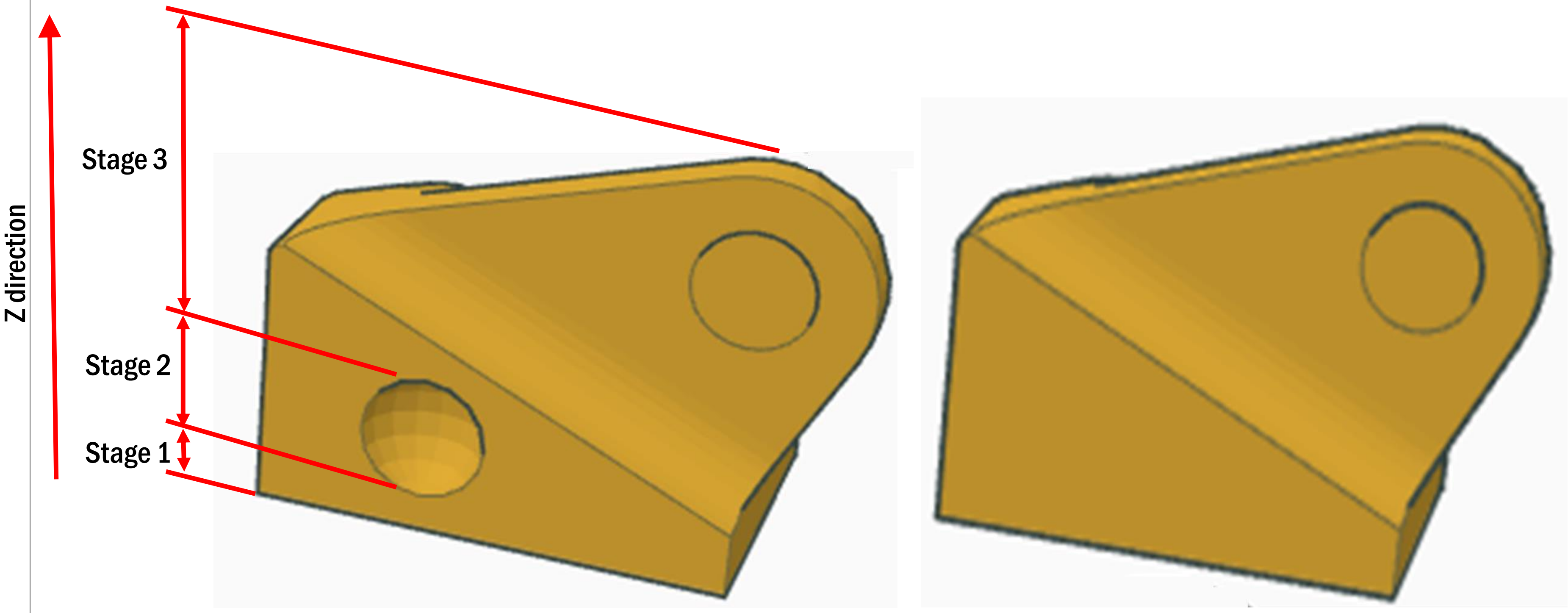
Albakri, Sturm, Williams & Tarazaga, SFF 2015





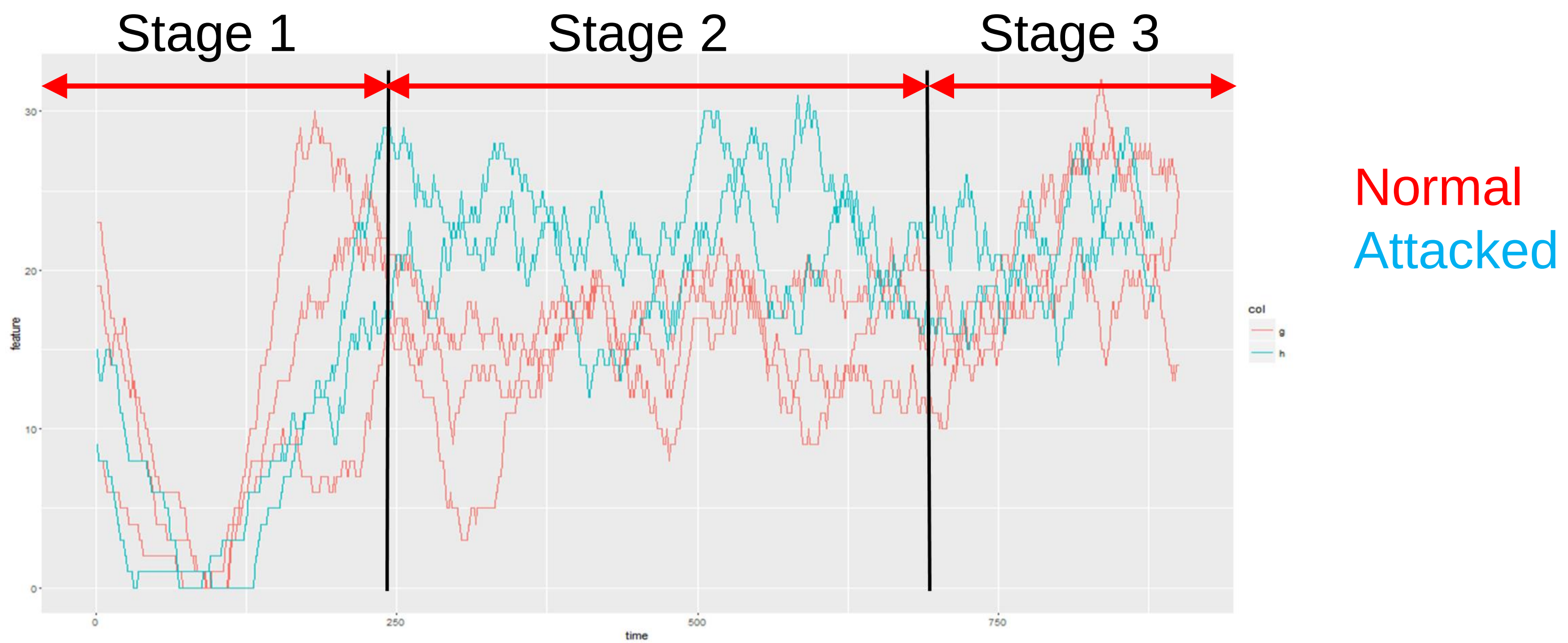
Sturm, Albakri, Tarazaga & Williams, SFF 2016

Online Side Channel Measurements



Attached part (L) and normal part (R)
G codes are different in stage 2; but the same in stages (1) & (3)

Online Side Channel Measurements



- Accelerometer (XYZ) mounted close to extruder
- Spectral graph theory based sensor fusion
- Significant overlap of signal features between normal and attacked parts in stages (1) and (3), not separable
- Separable signal features between normal and attacked parts in stage (2)

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Acknowledgements

Collaborators

- Dr. James Kong
- Dr. Timothy Long
- Dr. David Dillard
- Dr. Jules White
- Callie Zawaski
- Justin Sirrine
- Allison Pekkanen
- Lindsey Bass
- Ivan Vu
- Dr. Amelia Elliott (ORNL)
- Dr. Jacob Moore (PSU-Altoona)
- Dr. Nicholas Meisel (PSU)
- Dr. Prahalad Rao (UNebraska)

Sponsors

- Industrial Sponsors
- National Science Foundation
  - #1436592 (PI: Kong)
  - #146804 (PI: Camelio)
  - *Any opinions, findings, and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the National Science Foundation.*
- Virginia Center for Innovation Technology
- VT Institute for Critical Technology & Applied Science

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DREAMS

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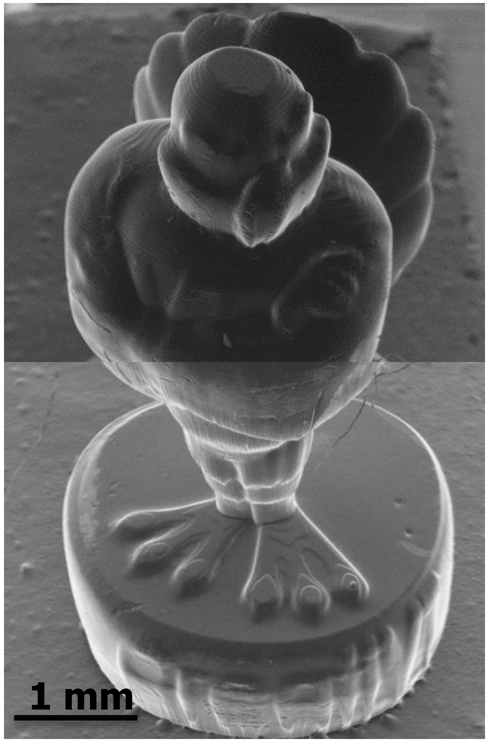
Molecules to Manufacturing:
Expanding the Polymeric Materials Toolbox

In-Situ Processing Measurements



Thank you.

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cbwill@vt.edu

DREAMS