Molecules to Manufacturing: Expanding the Polymeric Materials Toolbox

In-Situ Processing Measurements

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VT Additive Manufacturing Faculty

AM Processes
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- Rayne Zheng (ME)
- Blake Johnson (ISE)

Advanced Materials
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Intelligent Manufacturing
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- Scott Case (BEAM)
- James Kong (ISE)
- Ran Jin (ISE)

2015 College of Engineering Team Hire
Additive Manufacturing Research Facilities

Multi-Material Precision Extrusion (B. Johnson)

Mask Projection Vat
Photopolymerization (R. Zheng)
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

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

Determination of the mode I adhesive fracture energy, $G_{IC}$, of structural adhesives using the double cantilever beam (DCB) and tapered double cantilever beam (TDDB) specimens, in BS 7991:2001. 2001.
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
Relating Material Properties to Process Parameters

Working Curve for Photopolymers

\[ C_d = D_p \ln\left(\frac{E_{max}}{E_c}\right) \]

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
Photorheology demonstrates decreasing photocured plateau modulus with increasing PDMS molecular weight.

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, N₂
2 wt % DMPA, 7.0 mW/cm², 30 s delay
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/cm², light on at 1 min, off at 11 min
• Photorheometry was used to compare critical exposures of polymer (with 2wt% QDs) cured at different intensities.
• Less total energy is needed for lower intensities
Relevant Process Variables

- Exposure
- Wavelength
- Intensity
- Oxygen
- Temperature
- Humidity
- Material composition
  - Photo initiator
  - Photo absorber
  - Fillers
  - Age
In-situ Measurements for Photopolymerization

UV Intensity Correction via CCD

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

Old World Labs; US 2015/0183168
Complexity of Polymer AM

- **Material**
  - Rheology (shear & temp)
  - Stiffness
  - Degradation (recycling)
- **Environment**
  - Temperature
  - Humidity
  - Heat transfer
- **Print**
  - Melt temperature
  - Shear
  - Toolpath
- **Part**
  - Heat transfer from shape & toolpath
  - Location
Rheology can be affected by thermal history, humidity, time.

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

Big Area Additive Manufacturing

Material Development

Printability

Structural Integrity

Analyze

Model

Talagoin 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?
Monitoring Extruder Temperature

(a) Normal State
Stable Extrusion, Smooth surface

(b) Abnormal State
Inconsistent (Stringy) Extrusion

(c) Failure State
Nozzle Clogged, Scraped Surface

Time (sec)

Temperature (°C)

(a) Normal State
(b) Abnormal State
(c) Failure State

Filament Breakage
Nozzle Clog
Thick Layers

Z. Kong (Virginia Tech)
Monitoring Video

Digital Microscope based Machine Vision System

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Monitoring Video

Digital Microscope based Machine Vision System

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

Stiltner, Elliott & Williams, 2011, *Int’l Solid Freeform Fabrication Symposium*
Embedded Thermocouples

CAD models - channels for thermocouples

VeroWhitePlus Model

TangoBlackPlus & Grey60 Models

Extra border of VeroWhitePlus

Embedding channel printed without support

Thermocouple glued inside of channel

Thermocouples embedded during build process
Embedded Fiber Sensor
Material as a Sensor: Jettable Quantum Dot Photopolymer

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

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

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

Ivanova et al., Add Mfg, 2014; Elliot et al., Adv Eng Mat, 2013
AM Cyber-Physical Security

Design & PLM

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

File Transfer

Quality Control
STL as an Attack Vector
Security of Quality Control

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

(Wells & Camelio)
We must consider the cyber security of our in-situ measurements.

Side channel measurements are as important as closed-loop control.
In-Situ Impedance-based Monitoring

Albakri, Sturm, Williams & Tarazaga, SFF 2015

Sturm, Albakri, Tarazaga & Williams, SFF 2016
Attached part (L) and normal part (R)
G codes are different in stage 2; but the same in stages (1) & (3)

Z. Kong & J. Camelio (Virginia Tech)
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)

Z. Kong & J. Camelio (Virginia Tech)
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Thank you.

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