NIST Perspectives on Additive Manufacturing for Civil Infrastructure Design and Construction

Scott Z. Jones    Chiara Ferraris

NSF Workshop on Additive Manufacturing for Civil Infrastructure Design and Construction

July 13-14, 2017

scott.jones@nist.gov
National Institute of Standards and Technology

- Provide measurement “toolbox” for the nation
  - Provide solutions to measurement problems
  - Try to assure that the necessary measurements and quality are available to meet the nations most significant needs
- From the assumed and expected to the extraordinary
- Absolute correctness of results is paramount to NIST Labs
Concrete is an additively manufactured material.

- Formwork erected to make final shape of structure
- Automated methods can eliminate formwork or integrate it into final structure

Traditional formwork construction. Building a structure to build a structure

Concentric rings of cement paste added layer by layer
Additive Manufacturing in Construction
Two methods

Extrusion Systems
- Gantry crane or robotic arm
- Material pumped through piping to nozzle
- Material mixed continuously or batched as required

Powder Bed Systems
- Powder bed – like an ink jet printer
- Adhesive deposited in layers
- Resolution: print small features
- Generates waste material

NIST Paste Printer
Bam Group and Universe Architecture
Challenges in Concrete AM

Cement “buildability” window

Print quality of cement structures changes with time

A low yield stress, low viscosity material cannot support layers
Challenges in Concrete AM

Inter-layer bond: Cold joints and defects are possible.

Cross sections of the structures built after initial setting time contain defects and apparent cold joints, while the cross section of structures just prior to initial setting...
Challenges in Concrete AM

Material Curing: printed specimens allowed to cure in laboratory environment experienced cracking as a result of evaporation.
Why is Metrology Important?

International Bureau of Weights and Measures (BIPM): Metrology is the “science of measurement, embracing both experimental and theoretical determinations at any level of uncertainty in any field of science and technology.”

- Prescriptive to performance-based specifications
- Assess the strength of a AM wall
- Maximum shear stress on layer at $\frac{\pi}{4}$

\[
\sigma_n = \mathbf{n} \cdot \sigma = |\sigma| \cos^2(\theta), \\
\sigma_t = \mathbf{t} \cdot \sigma = |\sigma| \sin(\theta) \cos(\theta)
\]
Why is Metrology Important?

International Bureau of Weights and Measures (BIPM): Metrology is the “science of measurement, embracing both experimental and theoretical determinations at any level of uncertainty in any field of science and technology.”

**Objective** is to relate rheology to printability

- Rotational → Yield Stress
- Oscillatory Strain sweep → Stress at $G' = G''$
- SAOS and LAOS → $G' = G''$

![Parallel Plate Geometry](image)

Oscillation at Frequency
$f = \frac{\omega}{2\pi}$

Shear Strain:
$\gamma(t) = \gamma_A \sin(\omega t)$

Shear Stress:
$\tau(t) = \tau_A \sin(\omega t + \delta)$

- Steady onset of yield stress
- Rapid onset of yield stress

Initial Set 75 min

Flow Stress, $\tau_f$: $m_1 = 0.085 \text{ Pa/min}$
$\beta_1 = 29.016 \text{ Pa}$
$RSE = 7.823$

Initial Set 69 min
$\tau_f = m_2 \tau + \beta$
$m_2 = 4.255 \text{ Pa/min}$
$\beta_2 = -257.026 \text{ Pa}$
$RSE = 28.927$

![Stress vs. Time](image)
The NIST Approach
Measurement Science for Concrete AM.

**Objective:** Develop measurement science tools (metrologies, standards, and guidance documents) for quantitatively evaluating the critical material properties and ensuring the desired field performance of cement-based additive manufacturing.

How do we ensure a process or material is suitable for AM?

- **Artifact for print quality**
  - Verify machine or material performance

https://www.nist.gov/el/intelligent-systems-division-73500/production-systems-group/nist-additive-manufacturing-test
The NIST Approach
Measurement Science for Concrete AM.

**Objective:** Develop measurement science tools (metrologies, standards, and guidance documents) for quantitatively evaluating the critical material properties and ensuring the desired field performance of cement-based additive manufacturing.

How do we ensure a process or material is suitable for AM?

- **Artifact for print quality**
  - Verify machine or material performance

- **Traditional concrete test for AM**
  - Compressive strength
**Objective:** Develop measurement science tools (metrologies, standards, and guidance documents) for quantitatively evaluating the critical material properties and ensuring the desired field performance of cement-based additive manufacturing.

How do we ensure a process or material is suitable for AM?

- **Artifact for print quality**
  - Verify machine or material performance
- **Traditional concrete test for AM**
  - Compressive strength
  - Slump test

Extrude a cylinder of a known height, $H$, and diameter, $\varnothing d_{nozzle}$ over a grid (Green circle). Measure diameter after a prescribed time (Red circle).
**Objective:** Develop measurement science tools (metrologies, standards, and guidance documents) for quantitatively evaluating the critical material properties and ensuring the desired field performance of cement-based additive manufacturing.

How do we ensure a process or material is suitable for AM?

- **Artifact for print quality**
  - Verify machine or material performance

- **Traditional concrete test for AM**
  - Compressive strength
  - Slump test
  - Setting time measurements

---

**Chart:**

- **Y-axis:** Normalized Penetration Depth
- **X-axis:** Time (min)

- Data points indicate a decrease in penetration depth over time, suggesting the material's performance characteristics.
**Objective:** Develop measurement science tools (metrologies, standards, and guidance documents) for quantitatively evaluating the critical material properties and ensuring the desired field performance of cement-based additive manufacturing.

How do we ensure a process or material is suitable for AM?

- **Artifact for print quality**
  - Verify machine or material performance

- **Traditional concrete test for AM**
  - Compressive strength
  - Slump test
  - Setting time measurements

\[
\tau_f = m t + \beta
\]

- Initial Set
  - \( m_1 = 0.085 \text{ Pa/min} \)
  - \( \beta_1 = 29.016 \text{ Pa} \)
  - \( \text{RSE} = 7.823 \)

- Steady on-set of yield stress
  - \( m_2 = 4.255 \text{ Pa/min} \)
  - \( \beta_2 = -257.026 \text{ Pa} \)
  - \( \text{RSE} = 28.927 \)

- Rapid onset of yield stress
Objective: Develop measurement science tools (metrologies, standards, and guidance documents) for quantitatively evaluating the critical material properties and ensuring the desired field performance of cement-based additive manufacturing.

How do we ensure a process or material is suitable for AM?

- Artifact for print quality
  - Verify machine or material performance

- Traditional concrete test for AM
  - Compressive strength
  - Slump test
  - Setting time measurements

- In-line process measurements
  - Capillary rheometry

- Machine learning to predict cement/concrete performance
  - CCRL data set
NIST Cement AM Facilities
Cement Paste Printer

• Nozzle Designs
  – Adjustable diameters
  – Capillary rheometer
  – Simultaneous paste and polymer printer

• In-line mixing of set accelerator
  – Control cement hydration to initiate setting

• Progressive Cavity pump
  – Mix cement and water as needed

• Data Acquisition
  – Pump Pressure – strain gauges
  – Material Flow Rate

• Ultrasonic Detection of Setting
  – Air-coupled ultrasonic transducers.
  – Detect reflection of Lamb wave as setting takes place.
NIST Cement AM Facilities
Mortar Printer

- Explore methods of extending yield stress range
  - Admixtures before extrusion
  - Actively mix (grout pump)

- Bigger picture: large scale printing
  - Predict printability on the small scale
  - Small batch mixing and micro rheometers

- Investigate more parameters
  - Other rheological properties
  - Printing parameters

RoboPrinter at NIST – Large scale cement and mortar 3D printer in development (3 m × 3 m × 3 m cube!)
Lab to Commercialization

• Continue studies to understand relationship between material properties, machine settings, print quality, and print performance.
  – Control onset of initial set.
  – Material delivery.

• Codes and Standards
  – Measuring compressive strength, rheology, and other material properties.
  – Performance-based specification of materials!

• In-line and in-situ measurements of material properties – NDT/NDE
  – Cold joint and flaw detection.
  – Strength build up.

• Machine design
  – Nozzle design – influence on print quality.

• What about reinforcements?
  – Fibers – orientation and effectiveness.
  – Parallel printing – incorporate other AM techniques to create reinforcement.
NIST Perspectives on Additive Manufacturing for Civil Infrastructure Design and Construction

Scott Z. Jones  Chiara Ferraris

NSF Workshop on Additive Manufacturing for Civil Infrastructure Design and Construction

July 13-14, 2017

scott.jones@nist.gov
Preliminary Work: Our approach has been to combine conductivity with rheological measurements to assess the transition of cement pastes during reaction. This allows us to investigate how factors like water content and the addition of minerals such as calcite affect the rate of this transition. Properties such as cement strength and porosity can be assessed by these measurements and may be useful for applications such as 3-D printing where in-situ measurements of these properties are essential for process control.

An example curing curve is shown in Figure 2 where we monitor the viscoelastic properties of the cement as a function of time as well as the AC conductivity. As is evident from the data, immediately after mixing the cement begins to set and after 24 hours of curing time, it achieves its final strength. In the initial curing stages, a rapid increase in conductivity is associated with the dissolution of Ca\(^{+2}\) ions from the pores in the cement and over the course of the first hour we observed a reduction in conductivity consistent with the conversion free Ca\(^{+2}\) to form C-S-H. For the sample in Figure 2a, after around 4 hours of curing, there is a drastic decrease in the conductivity as the pores between the cement grains are closed off and the ions are confined to interstitial spaces within the solid. This time-scale of this transition corresponds to the same time where the cement has reached 90% of its final strength.

To further characterize this system, we have performed preliminary small angle neutron scattering (SANS) studies on a cement as it cures in 50 vol % D\(_2\)O (3.9 \(\times\) \(10^{-6}\) Å\(^2\)) corresponding to the match point for CaCO\(_3\). This composition provides sufficient contrast such that the Ca\(_3\)S phase is visible but its contribution to the scattering signal minimized to reduce multiple scattering. We acquired a scattering profile every hour over the first 10 hours of curing and plotted the data as a Porod plot IQ\(^4\) vs Q in Figure 2b. The scattering profiles show an increase in intensity at high-Q at the expense of interfacial area at low-Q and an isobestic point at Q = 0.0055 Å\(^{-1}\) corresponding to a length scale of ~100 nm. We attribute the reduction at low-Q to the dissolution of C\(_3\)S to Ca\(^{+2}\) and the increase at high-Q to the production of C-S-H globules. Further, the time-scales of these processes are consistent with those observed in Figure 1a. The slow decrease in conductivity shows that the production of C-S-H is constantly occurring and its only when a critical amount of material

- Small angle oscillatory shear measurements in parallel with conductivity and neutron scattering.
- SAOS and conductivity measure setting
- Neutron scattering links two measurements to microstructure development
Cement/Limestone Mixtures

Previous NIST work: Using limestone powder to minimize paste content


- Fine limestone powders act as an accelerator
- Filling gaps in cement PSD improves rheology
- Obtain maximum packing according to Brouwers

![Graph showing cumulative heat release and cement paste mixtures]

25:50:25 blend of 2.2 µm:cement:16 µm falls close to ideal packing. Limestone acts to accelerate mixture, offsetting delay due to HRWRA
Cement/Limestone Mixtures

Previous NIST work: Using limestone powder to minimize paste content


- Fine limestone powders act as an accelerator
- Filling gaps in cement PSD improves rheology
- Obtain maximum packing according to Brouwers
- Increasing packing increases shear stress. Good for AM?

Figure 9. Heat flow (left) and cumulative heat release (right) for the cement-limestone blend pastes in phase two of the paste study. Ratios in legend indicate fine limestone:cement:coarse limestone proportions on a volume basis.

Figure 10. Shear stress vs. shear rate for parallel (serrated) plate rheological measurements for the ternary blend cement pastes. Typical relative error for viscosity is about 7%.

Canadian study that compared the performance of limestone powders with median particle diameters of 3 µm and 17 µm and found basically equivalent performance [22]. Due to the requisite high dosage of HRWRA that is envisioned for the low w/s mixtures being developed in this study, it was decided to maintain about a 5% proportion of the fine limestone (i.e., 10:90 fine:coarse in the 50% limestone, 50% cement blend) to help offset the anticipated retardation produced by the HRWRA. The initial overlap of the w/c=0.4 control and 5:50:45 paste heat flow curves in Figure 9 is consistent with this hypothesis. However, if some delay in the setting times, such as 1 h to 2 h, were not an issue for the field application, it is possible that acceptably performing cement/limestone powder blends could be engineered using only the 16 µm limestone powder or even the 19.2 µm one, for example, likely minimizing both costs and materials storage.

Adjusting fine:coarse limestone can change rheology.
Evaluating Print Quality

- A challenging test print: a tall thin wall (6.5 mm x 38 mm x 74 mm, 24 layers)
- Prints separated by 5-10 minute intervals until extrusion was impossible for our machine
- Prints were evaluated based on a ‘printability’ rubric

Print quality changes with time

$\begin{align*}
t &= 0 \text{ min} \\
t &\geq 60 \text{ min}
\end{align*}$

Printability Rubric

<table>
<thead>
<tr>
<th>Property</th>
<th>Quantification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Extrudability</td>
<td>Good pumping = 1</td>
</tr>
<tr>
<td></td>
<td>Jamming = 0</td>
</tr>
<tr>
<td>Buildability</td>
<td># of Layers</td>
</tr>
<tr>
<td>Early Strength</td>
<td>Standing = 0.5</td>
</tr>
<tr>
<td></td>
<td>Falling over = 0</td>
</tr>
<tr>
<td>Print Quality</td>
<td>Cold Joints</td>
</tr>
<tr>
<td></td>
<td>Cracks/Defects</td>
</tr>
</tbody>
</table>
Relating Rheology to Printability

Mixture Design:
- $w/s = 0.22$
- 50:50 mass ratio of Limestone Powder:Cement
- Polycarboxylate-based “High Range Water Reducer”
  - 4.5 mL/kg powder
- Three particle sizes ($D_{50}$)
  - 50:50 blend 2.2 µm:16 µm
  - 2.2 µm
  - 16 µm

- Yield stress is important for printing, but not the only factor
- Each material has an ideal yield stress range for printing (650-900 Pa for this print)
Setting Time Measurements

Need for new test method?

- Oscillatory Rheometry: 35 mm serrated parallel plate with 0.6 mm gap.
- Constant Oscillation: $\gamma = 1.0$ mm/mm at $f = 1.0$ 1/s
- Setting time defined at point $G' = G''$

![Graph showing time measurements and setting time definition.](image)
Setting Time Measurements

Need for new test method?

- Rotational Rheometry: 35 mm serrated parallel plate with 0.6 mm gap.
- Rotation deforms material in one direction.
- Large displacements break down structure - may prevent setting.
Setting Time Measurements

Need for new test method?

- Oscillatory Rheometry: 35 mm serrated parallel plate with 0.6 mm gap.
- Strain Sweep at $f = 1.0$ 1/s
- Plot stress at $\gamma = 1.0$ mm/mm