Mechanical Response and Micro-CT Characterization of 3D Printed Cement Paste Elements with Controlled Architecture

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Direct Ink Writing (DIW) Is a Method of Patterning Materials

DIW:

- **Layer-by-Layer** Patterning Materials in 3 Dimensions
- Extrusion-based fabrication method using **computer-controlled** translation stage (gantry)
- Used for polymer melts and **colloidal gels** & slurries such as cement paste
- For Cement Paste: Re-configure printer assembly and processing parameters
- Work with materials such as Silicone and Chocolate to **integrate** and **parameterize**.
- Ink Development, flow processes, rheology, extrudability, shape-holding

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**Fused Filament Fabrication (FFF)**

- **Stepper motor-driven** extrusion system (Syringe and Plunger)
- **Silicone**
- **Chocolate**
- Nozzle Dia.: **1.36 mm**
- Layer Height: **1.00 mm**
DIW Can do More:

It allows to develop prototypes for:

- Evaluation of intertwined mechanisms between: Processing-Structure- Properties / Performance

- Achieving novel material-structural systems: “Architectured Materials”

- Combination of materials and space
- DIW comes at the cost of the “weak interface”
- Engineered to have new properties
- Properties not offered by material or structure alone

Hypothesis: DIW can enhance the mechanical response of brittle hcp materials via design
Representative Designs of Architectures

Honeycomb
Sandwich panel prism
Grid
Compliant

Scale bars: 10 mm

Bouligand (\(\gamma = 2^\circ\))
Bouligand (\(\gamma = 45^\circ\))
Cellular
Bouligand (\(\gamma = 8^\circ\))
Solid

\(\gamma\)?

Design &
Fabricate with a variety of \(\gamma\) and infill percentage:

\(\gamma = 8^\circ\)
Hydrated Cement Paste (HCP) can be made Compliant by Design

- 1st, Cyclic linear L-D response prior to contact
- 2ndary slope
- Two discrete moduli

Scale bars: 3.0 mm
Role of the “Weak interface”

➢ No significant differences between strength of cast and printed specimens

Weak interfaces can be utilized to control the crack path and to improve fracture properties

➢ At 0° and 45°: Horizontal crack deflection
➢ Secondary micro-cracking advanced at interface

➢ At 90°: Clear cleavage at Interface
➢ No micro-Cracking
HCP with Bouligand architecture can increase toughness, when combined with “Weak interface”

- Enhanced WOF (for cellular high $\gamma$, and solid small $\gamma$)
- Higher inelastic deformation
- W/o sacrificing the strength
- Outperform in strength compared to cast hcp counterparts (i.e., theoretical strength-porosity curve for hcp)

**Discs:**
55 mm Dia.
8 mm Ht.
15 mm
Micro-CT of 8° pitch angle solid Bouligand architectures (post-fracture):

Small pitch angle Bouligand architecture promoted damage mechanism such as:

- Interfacial cracking and micro-cracking
- Crack twisting

Therefore it allows for:

- Controlled fracture and crack growth at interface
- Enhanced energy dissipation and toughness
- Enhanced damage and flaw tolerance

**We can Infer:** Bio-inspired Bouligand Architectures + “Weak Interfaces” promote interfacial damage and allow for enhance the mechanical response
Competing mechanisms between small and large pitch angles

- Larger $\gamma$ (e.g., $45^\circ$) allow crack growth in materials as opposed to
  - smaller $\gamma$ (e.g., $8^\circ$) that promote interfacial damage mechanisms

- **Open question**: The role of interfacial strength in this trade-off?
Incorporation of the “weak interface” in favor of enhanced performance in architectured cement-based materials:

We have used DIW:

- To combine several architectures (such as Honeycomb or Bouligand) in order to explore the processing-structure-property relationship in hcp.

Combined effects of architecture and interfacial porosity on mech. performance:

- Improvement of performance characteristic
- Promotion of unique damage mechanisms, such as spread of interfacial cracking and micro-cracking
- Promotion of toughening mechanisms
- Increase in fracture resistance, resulting in quasi-brittle and flaw-tolerant behaviors in brittle hcp elements; without sacrificing the strength.

This could be one approach in 3D-printing that allow new of designing materials and structures.
Characterization of the Interface
What are the characteristics of the Core vs. Interfaces?

A lab-based X-ray Micro-CT can be used to evaluate the processing-induced heterogeneities:

Lamellar Architecture

4X scan (4.04 µm pixel size) 0.4X scan (32.26 µm pixel size)
Microstructural Features (0.4X): Pores / Re-arrangements / White Regions

- Macro-pores
- Micro-pores
- Re-arrangement
- White regions
- Homog. Core vs. Porous Interfacial Regions (IRs)
Microstructural Features: Micro-Channels and Re-arrangement – 4X Scan

Top View

Side View

- Filaments
- Re-arrangement causing triangular micro-channels

Moini et al., RILEM, ETH. 2018
How about the cast specimen?

**Cast:**

- Randomly Distributed Pores in cast

**3D-printed hcp:**

- Patterned Pore Network in 3D-printed layered specimen

- Volumetric Segmentation

VS.
Micro-CT characterization of 3DP hcp:

- Revealed 4 microstructural features in lamellar architecture as follow:
  
  I,II) Macropores, and micropores at (IRs)
  
  III) Re-arrangement of filaments
  
  IV) Accumulation of anhydrous cement grains near the macro-pores (white regions)

- A porous interface/network was characterized.

- Pore network (at both macro and micro scale) appeared to align with filaments orientation in the lamellar architecture → control of pore architecture

- These features are processing-induced heterogeneities & depend on processing and environmental conditions; They can result in anisotropic properties.

- Lab-based Micro-CT is a useful tool for non-destructive evaluation of microstructure and porosity.
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Jeff Youngblood (Purdue), Joe Biernacki (Tennessee Tech), Jan Olek (Purdue) M. Reza Moini (PhD student, Purdue), Florence Sanchez (Vanderbilt), Pablo Zavattieri (Purdue)

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Back-up slides
Microstructural Features (4X): Pores/Re-arrangements/White regions

- Micro-channels
- Connectivity at IRs
- Re-arrangement
- White regions
  Represent accumulation of anhydrous grains near the pores
Microstructural Features: **Micro- and Macro-Pores** at IRs –0.4X Scan

- **Micro-Pores:** at vertical and horiz. Planes (IRs)
  - H1
  - H2

- **Macro-Pores:** at vertical planes
  - (Side view)
There is a huge need for advanced manufacturing in construction

United Nation Goals for Sustainable Development:
- Resilient infrastructure
- Safe and sustainable human settlement
- Sustainable use of terrestrial ecosystem

National Academy of Engineers Grand Challenges for Engineers:
- Restore and improve urban infrastructure

Productivity Improvement In Construction:
- Flat or declining

http://107.22.164.43/millennium/challeng.html
http://engineeringchallenges.org/File.aspx?id=11574&v=34765dff
Rudimentary 3D printing of cement is been done before

**World wide efforts on 3D printing concrete:**

- Universities (Delf, Dresden, ETH, IFFSTAR, …)
- US Army, Private and public sector within US, China, UAE, …

*Images with links to various 3D printing projects*
Direct Ink Writing (DIW) Is a Method of Patterning Materials

**DIW:**
- Patterning materials in 3 dimension
- Fabrication method with computer-controlled translation stage
- No need for tooling, dies, or lithography mask
- Capability for multi-material deposition of gels colloids and slurries
Printing Platform for DIW of Cement Paste

A Fused Filament Fabrication (FFF) Printer is merged with an extruder system and is modified to serve DIW of cement paste

- Ultimaker FFD printer to achieve high resolution at reasonable cost
- Integrated Discovery digital syringe pump and connected with a tube
- Worked with Silicone and Chocolate to integrate and parameterize.

- Processing parameters were optimized for cement paste

Nozzle Dia.: 1.36 mm
Layer Height: 1.00 mm
Printing Parameters?

- **Slicer (Interpreter):** Converts a digital 3D model into printing instructions for 3D printer.

- **Input:** STL file (Geometry of printed object) and several **printing parameters**,
  - Nozzle Size
  - Extrusion multiplier
  - Print speed (F)
  - Infill %
  - Layer height
  - Extrusion width
  - Movement speed
  - Bed Temperature, Fan, etc.
  - ……

- **Output:** G-code (machine-readable **toolpath** commands)
  - 5-Axis commands: Coordinates (X, Y, Z), E, F
Cement formulation has a large effect on print quality

**Ink Formulation:**
- Cement Paste (Not extrudable)
- Cement Paste + HRWRA
- Cement Paste + HRWRA + VMA

**Challenges at small scale:**
- Suitable viscosity for high shear
- Suitable yield stress upon extrusion (Shape holding)
- Segregation/bleeding
- Suitable printing parameters in Slicer
Can Design via DIW allow control of the mechanical response?

Hypothesis for Hardened Cement-Based Materials:

- **Mechanical response** of architectured 3D printed solid/cellular cement-based materials is influenced by their **architecture**.

**Fundamental Behavior under Flexure and Tension:**

- Uni-axial flexural strength (3PB/4PB) → **Prisms**
  Suitable for characterizing Interfacial properties

- Bi-axial flexural strength (Ball on 3 Balls) – **B3B** → **Solid and Architectured Discs**
  Suitable for characterizing architectured materials

Scale bars: 5.0 mm
Now we can 3D-print cement—What are we going to do with it?

“Architectured Materials”:

- Combination of **materials** and **space**
- Materials engineered to have **new properties**
- Properties not offered by material or structure alone


http://vcg.isti.cnr.it/Publications/2015/PZMPCZ15/

Gladman et al. Nature materials, 2016, 4D-P


[https://doi.org/10.1007/978-3-319-99519-9_16](https://doi.org/10.1007/978-3-319-99519-9_16)
Damage-Property-Microstructure Relationship (in Compression)

- Characteristics of a Microstructure of Patterned elements: **Helicoidal Architecture**

- IR affect damage propagation and allow

- Damage **delocalization** and control of crack propagation path

- That could cause **anisotropic** mech. prop.
**Damage-Property-Microstructure Relationship (in Compression)**

**Properties-Microstructural Architecture**

- Layer-wised lamellar 3D-printed cement-based elements have anisotropic compressive strength properties.

![Stress-Strain Response](image)

- Architecture can be used to significantly enhance WOF with incorporation of Bouligand architectures.

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**Sp. C.S. - 3 days (N.cm/gr)**

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**Sp. WOF - 3 days (N.cm^4/gr)**

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Damage-Property-Microstructure Relationship (in Compression)

**Damage-microstructural Architecture Interaction:**

- 3D printed cement-based elements present interfacial cracking and damage mechanisms that are different from the cast counterparts.

- These mechanisms can result in lower, higher, or similar compressive strength and WOF depending on the interaction between cracking and architecture (and testing direction).

- A cracking-microstructure interaction exist through which interfacial damage (crack and micro-crack) is promoted.
Other Interesting Findings:
- Strength development of materials over time can result in significant differences in mechanical properties of 3D-printed elements compared to cast counterparts.

- This may be due to the evolution of Materials Strength/Interfacial Strength.
Damage-Property-Microstructure Relationship (in Compression)

Properties-Microstructural Architecture

• The interfacial porosity (4X) in 3D-printed layered (lamellar) cement paste (3%) can be higher than the porosity in randomly distributed pores in cast elements (1%).

• It is hyp. that morphology of the architecture of the porosity can be as critical as the total amount of porosity to the strength-property relationship.
Damage-Property-Microstructure Relationship (in Compression)

**Properties-Microstructural Architecture**

- Microstructural architecture (i.e., 4 adjacent filament in 2 layers) of a 3-day-old cement paste was found to have a 25-25-50% proportion for the three pores-hydrated-unhydrated phases.

- This includes interfacial and microstructural porosity.

- The interfacial porosity was found to be interconnected, forming a continuous pore network.

![Segmented Image](image)

![Pore Frequency (Y) vs. Volume (X)](image)
Microstructural Architecture

Microstructural architecture can be controlled via design and DIW to investigate intertwined mechanisms:

- (Micro)structure: Characteristics of intra- and inter-filaments (interfaces)
- Processing (i.e., extrusion)
- Properties (fresh and hardened)
Interface is porous, but how weak is its strength?

Using 3PB:

- Although the interfaces were identified weak and porous.
- No significant difference was found between interfacial strength and bulk materials strength when structure was collectively tested in 3PB.
...by controlling crack path during failure
...by controlling crack path during failure

(a.1) 3D-0.4X

(a.2) 3D-0.4X, 4X ROI

(b1) XY-0.4X

(b2) YZ-0.4X

Typical Micro-cracking at the IRs near main crack

(c) XZ-4X

Hairline micro-cracks

(d) XY-4X

Micro-cracking

Main crack
...by controlling crack path during failure
Key Factors in 3D-printing cementitious materials

- Paste Rheology/Ink Development
- Chemistry
- Sample Outcome
- Hydration

Key Factors in Objective I. & II

Flow Rate
Speed (XY)
Nozzle Size

Viscosity
Yield ...

Print Quality
Height Stability
Width Consistency

Hydr. Phases

Processing Parameters

Objective I. & II

- Print Quality
- Height Stability
- Width Consistency

Objective I. & II

- Flow Rate
- Speed (XY)
- Nozzle Size