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

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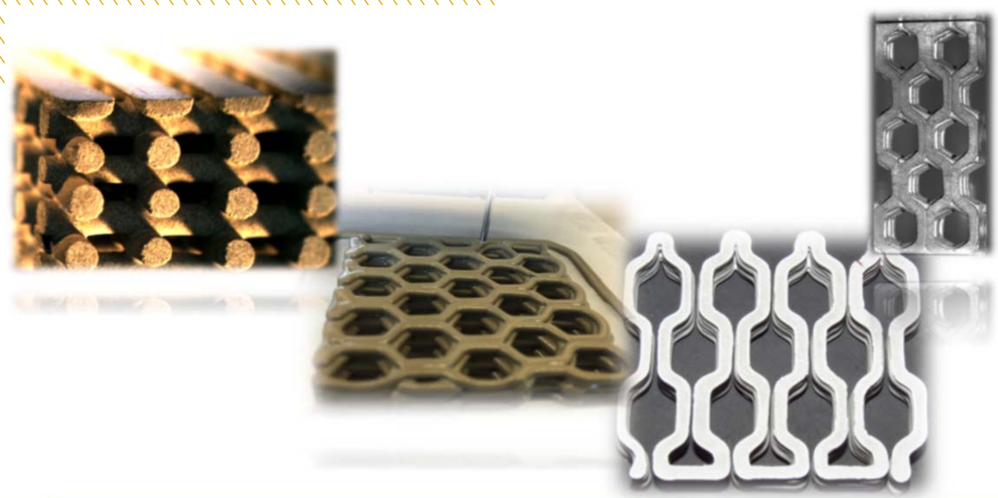


Support:
NSF CMMI 1562927

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**

Fused Filament Fabrication (FFF) 3D Printer

Stepper motor-driven extrusion system (Syringe and Plunger)

Chocolate **Silicone**

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"**

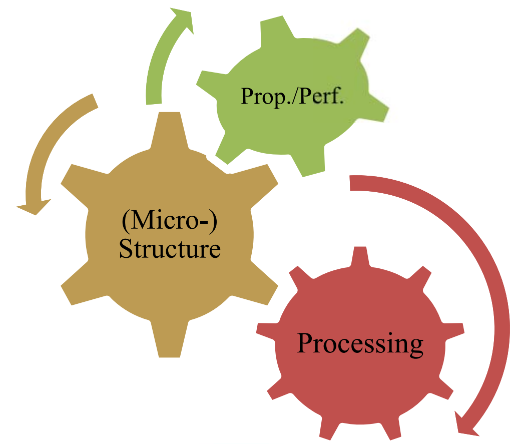
- **Engineered** to have **new properties**

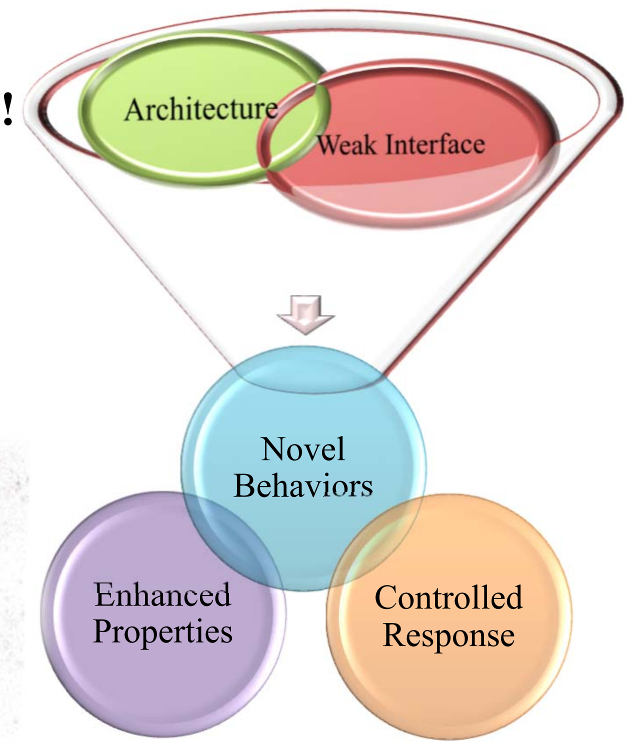
- **Sometimes we may embrace! Can we control its property!**

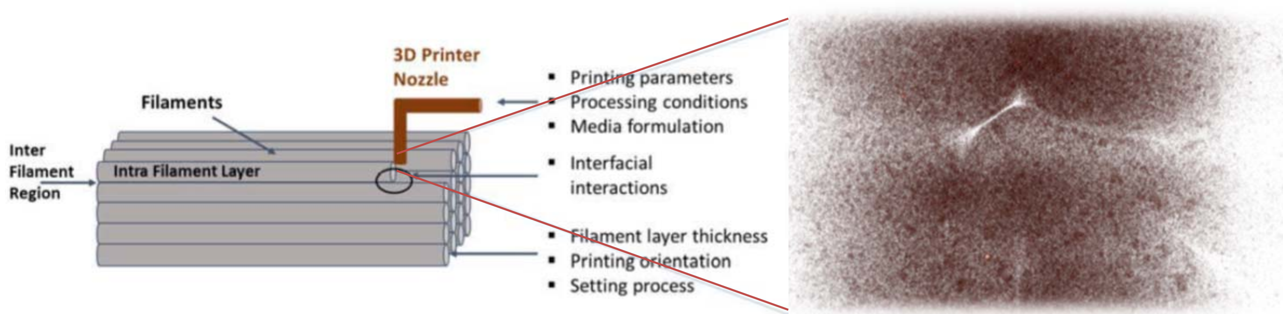
- **Properties** not offered by material or structure alone

- Architecture + Interface "weak" characteristics →

  Hypothesis: DIW can enhance the **mechanical response of brittle hcp materials via design**

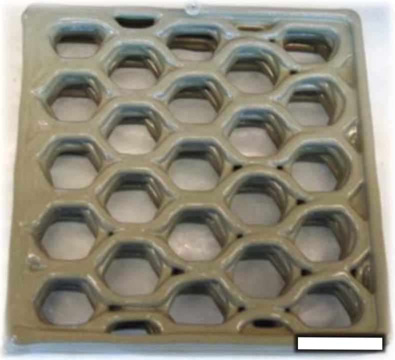




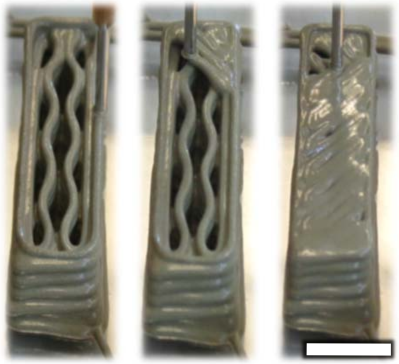


Representative Designs of Architectures

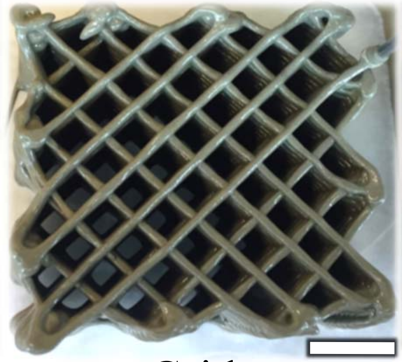
Scale bars: 10 mm



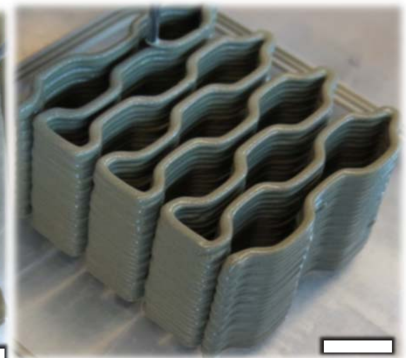
Honeycomb



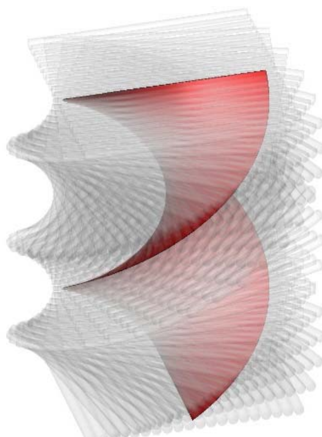
Sandwich panel prism



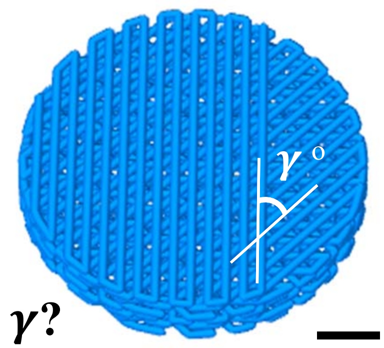
Grid



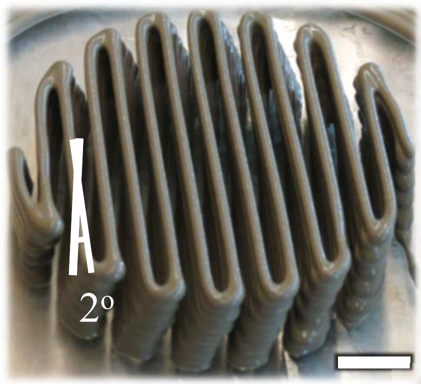
Compliant



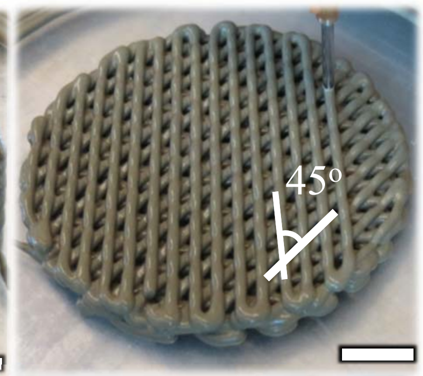
Bouligand
(Helicoidal)
Architecture



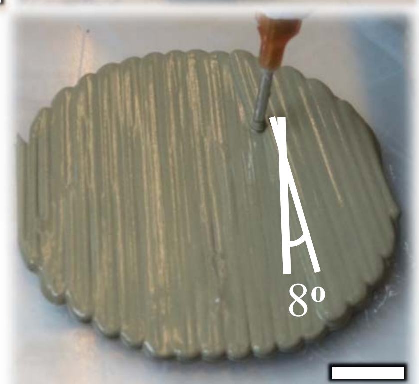
Design
&
Fabricate with a variety of γ and infill percentage :



Bouligand (γ =2º)



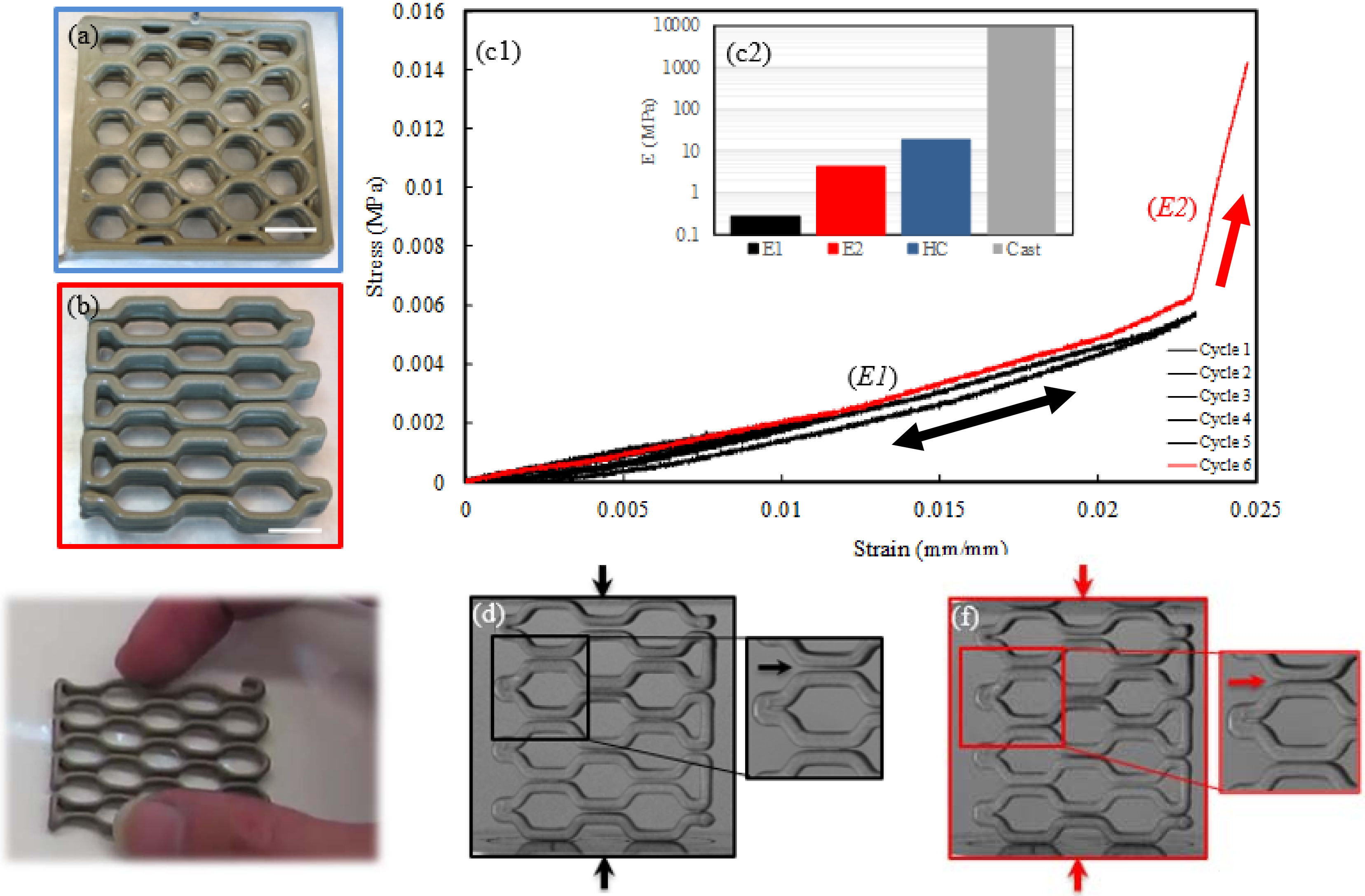
Bouligand
(γ = 45º)
Cellular



Bouligand
(γ = 8º)
Solid

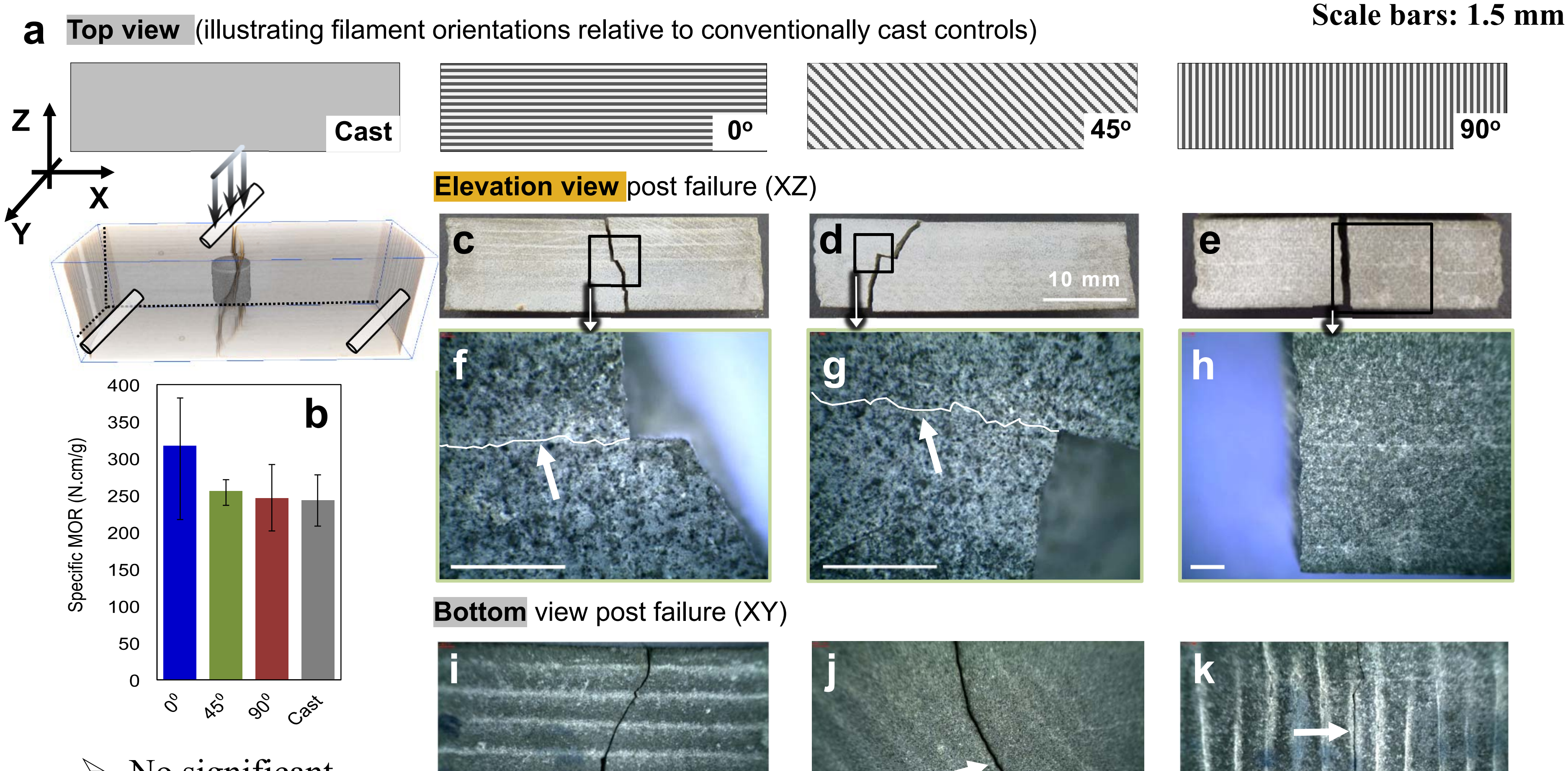
Hydrated Cement Paste (HCP) can be made Compliant by <u>Design</u>

Scale bars: 3.0 mm



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

Role of the "Weak interface"

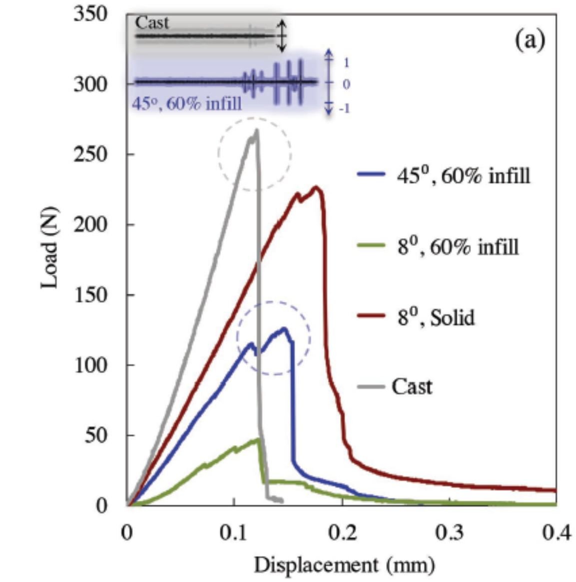


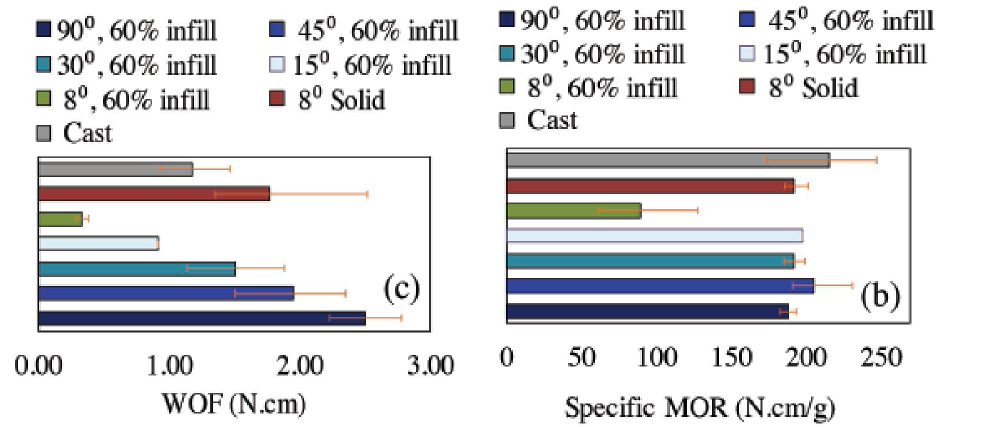
- No significant differences between strength of cast and printed specimens

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

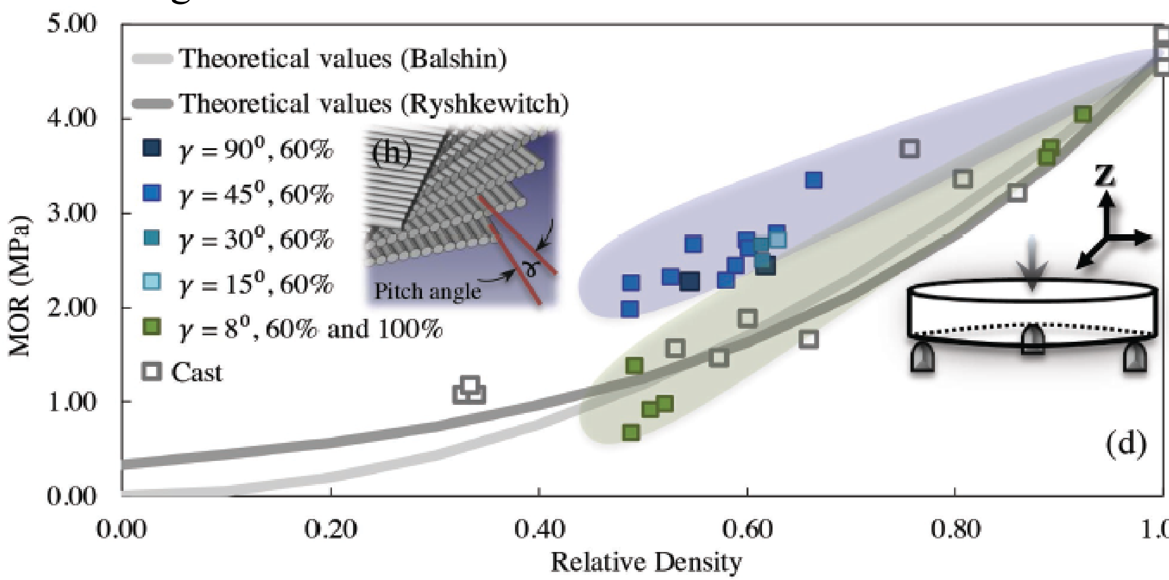
- At 0° and 45° : Horizontal crack deflection Secondary micro-cracking advanced at interface
- At 90° : Clear cleavage at Interface No micro-Cracking

HCP with Bouligand architecture can increase toughness, when combined with "Weak interface"



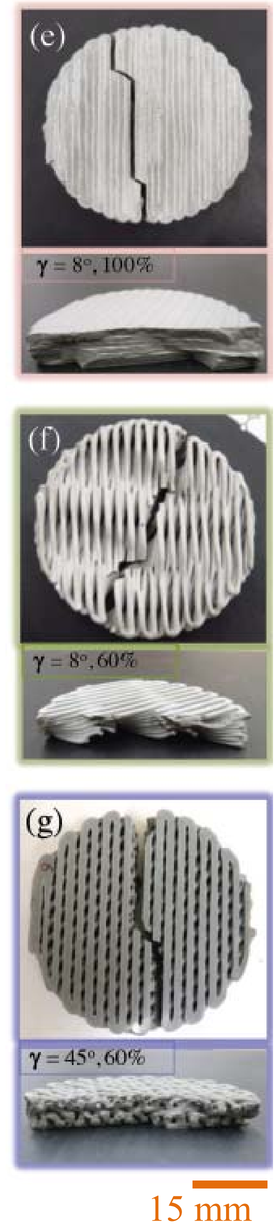


- W/o sacrificing the strength
- Enhanced WOF
  (for cellular high γ, and solid small γ)
- Higher inelastic deformation



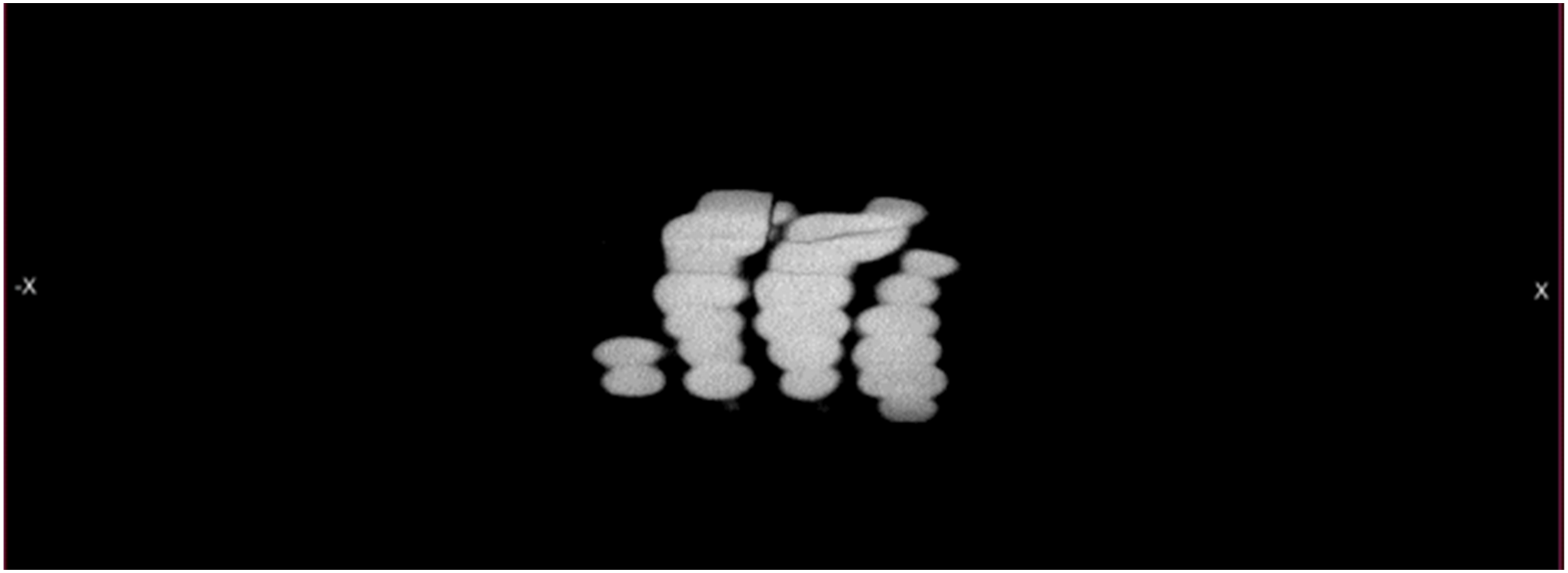
- Outperform in strength compared to cast hcp counterparts

  (i.e., theoretical strength-porosity curve for hcp)



Discs:
55 mm Dia.
8 mm Ht.

Micro-CT of 8° pitch angle solid Bouligand architectures (post-fracture):



Small pitch angle Bouligand architecture promoted damage mechanism such as:

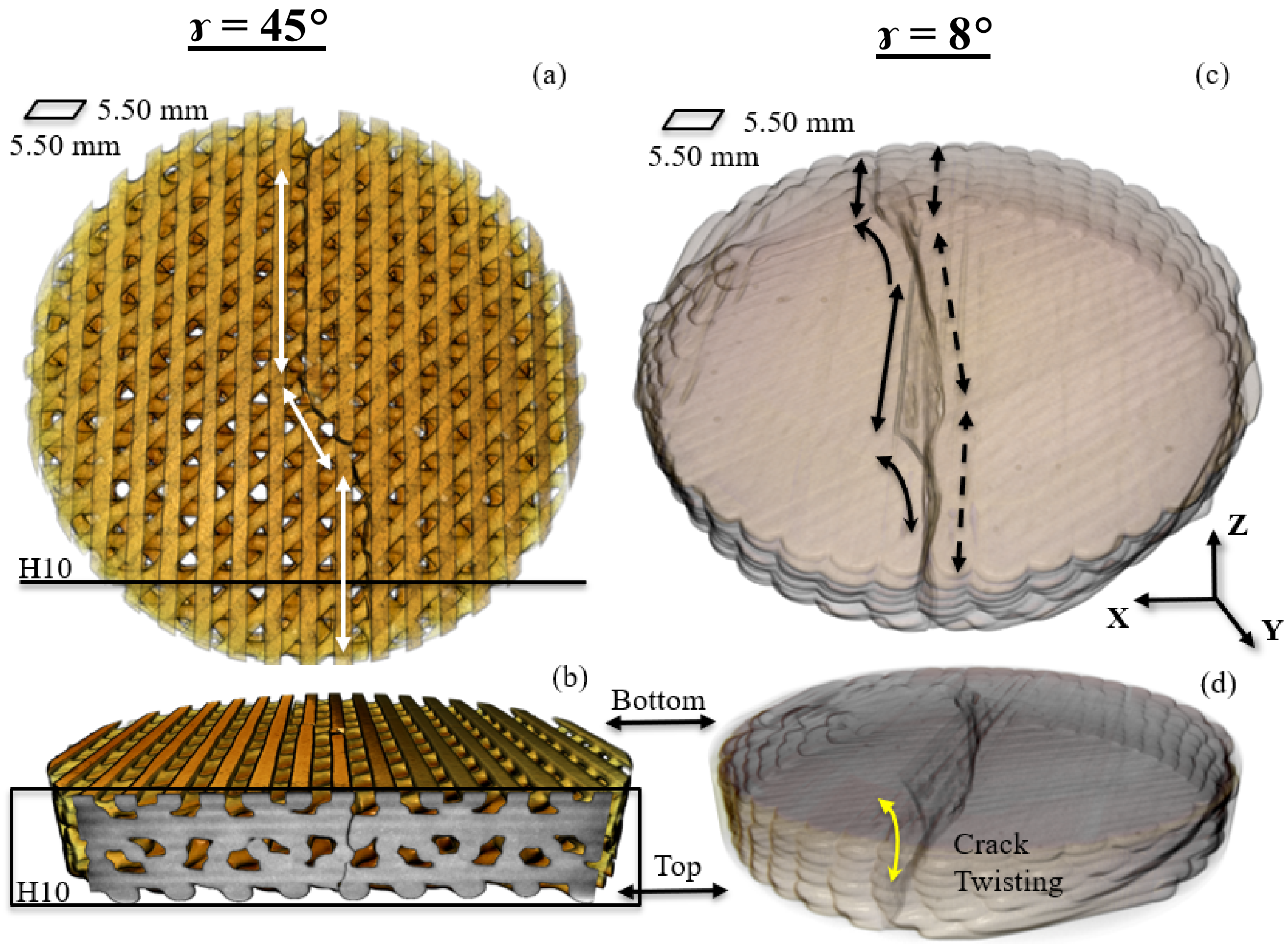
- Interfacial cracking and micro-cracking
- Crack twisting

Therefore it allows for:

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

<u>We can Infer:</u> Bio-inspired Bouligand Architectures + "Weak Interfaces" promote interfacial damage and allow for enhance the mechanical response

Competing mechanisms between small and large pitch angles

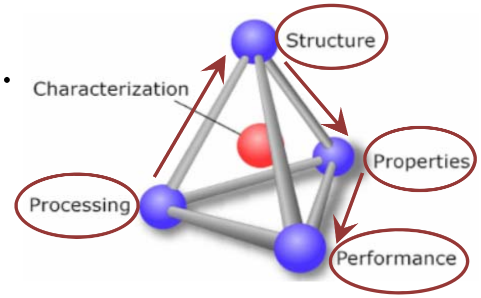


- ➢ Larger ɤ (e.g., 45°) allow crack growth in materials as opposed to
  - ➢ smaller ɤ (e.g., 8°) that promote interfacial damage mechanisms
- ➢ **<u>Open question</u>: The role of <u>interfacial strength</u> in this trade-off ?**

Incorporation of the “weak interface” in favor of enhanced performance in architectured cement-based materials:

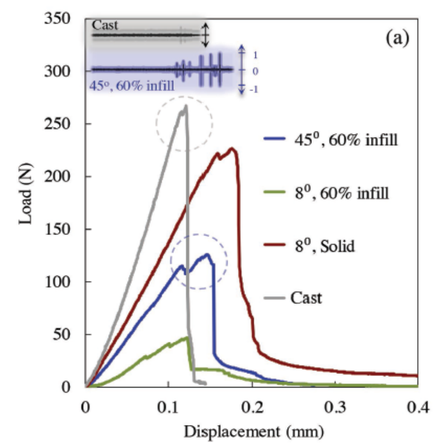
We have used DIW :

- 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.



Moini et. al., Adv. Mater. 2018

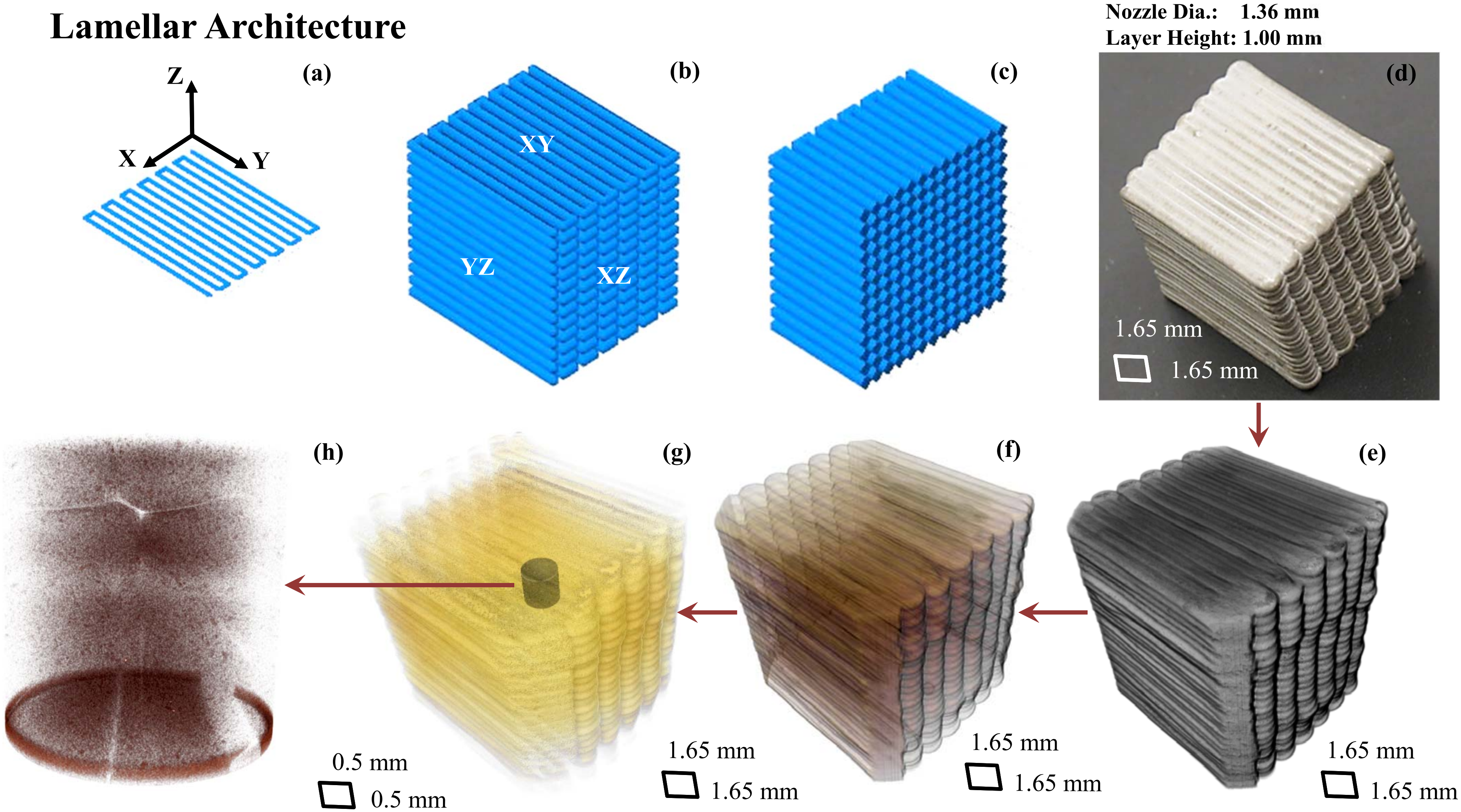
This could be one **approach** in 3D-printing that allow **new of designing materials and structures**

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

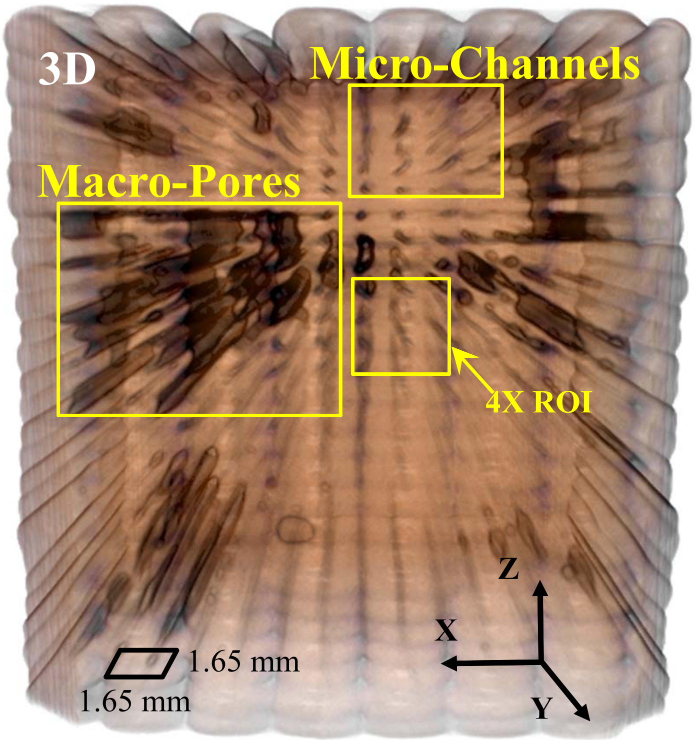


4X scan (4.04 µm pixel size)

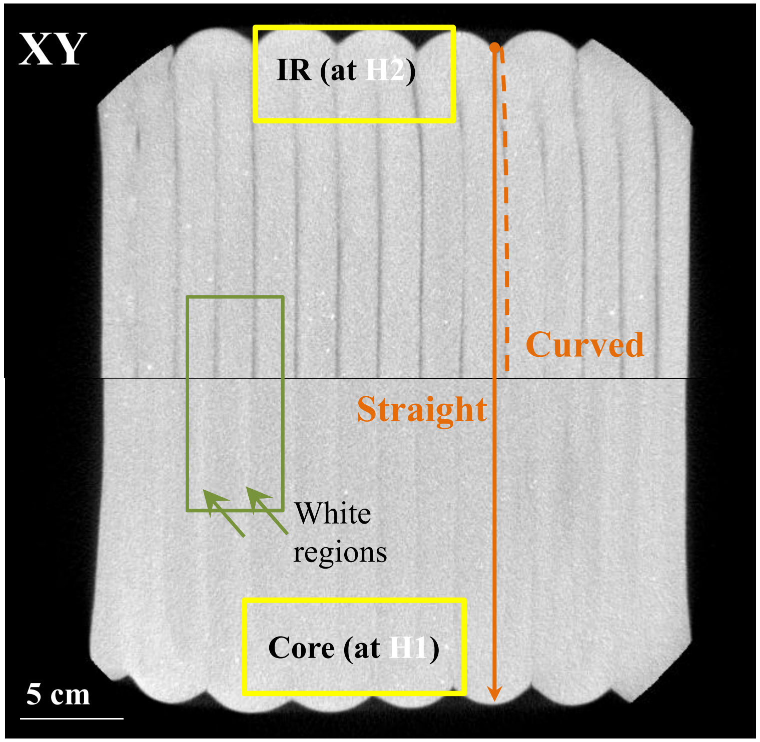
0.4X scan (32.26 µm pixel size)

Microstructural Features (0.4X): Pores / Re-arrangements / White Regions

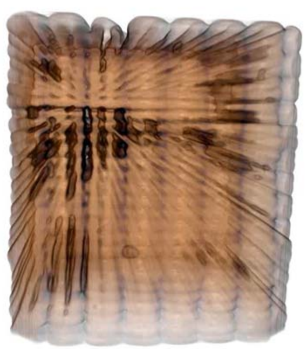
Side View

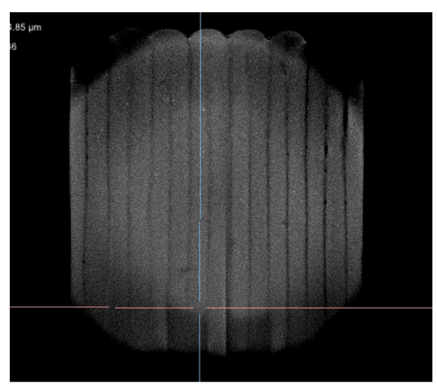


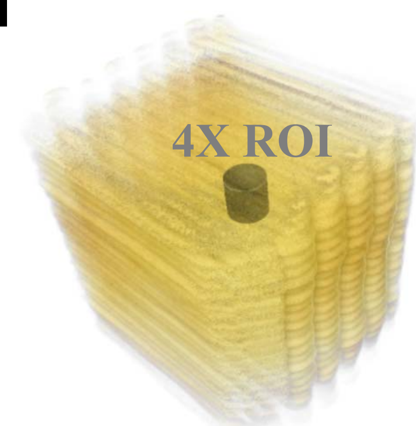
Top View



- Macro-pores
- Micro-pores
- Re-arrangement
- White regions
- Homog. Core vs. Porous Interfacial Regions (IRs)

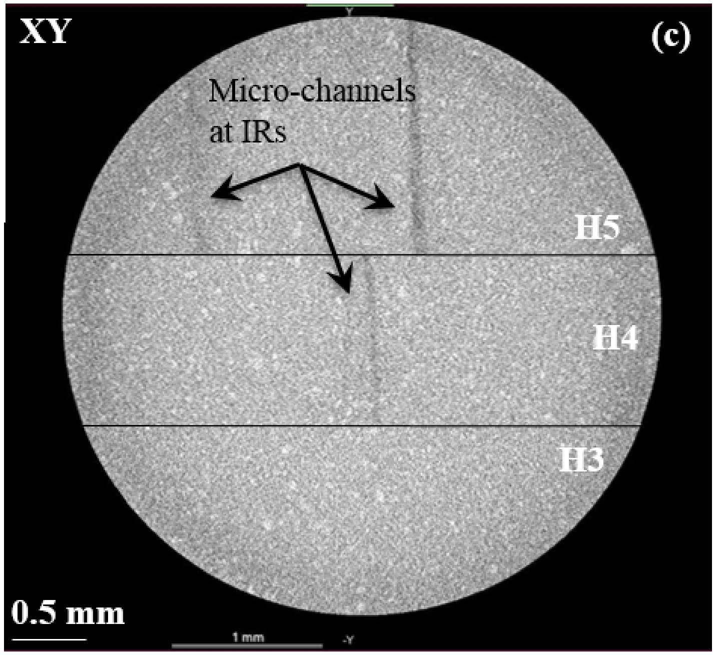




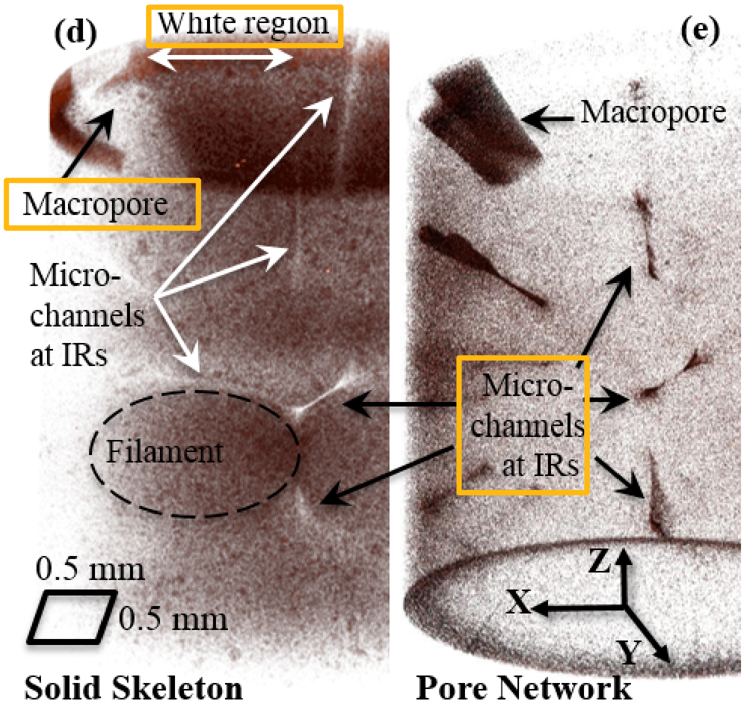


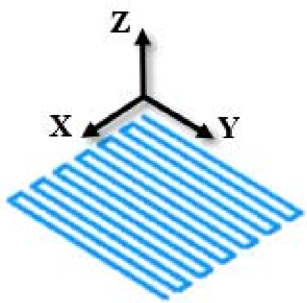
Microstructural Features: Micro-Channels and Re-arrangement – 4X Scan

Top View



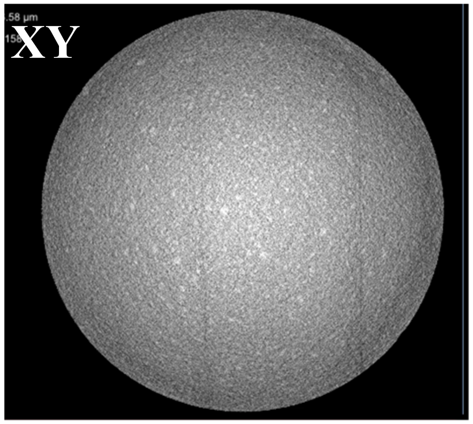
Side View

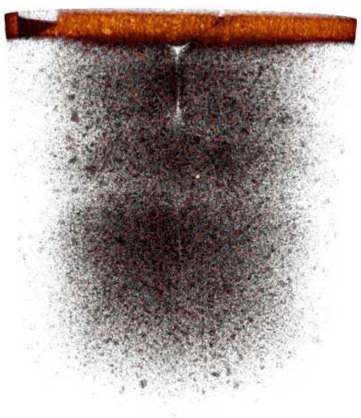


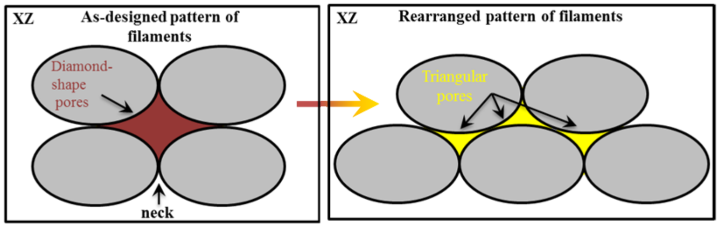


- Filaments Re-arrangement

causing triangular micro-channels



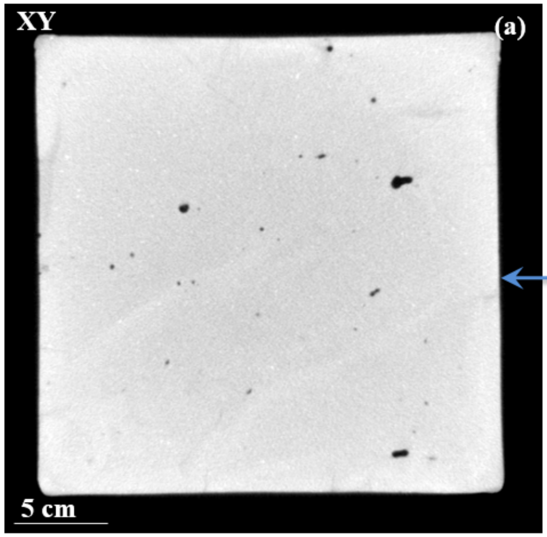


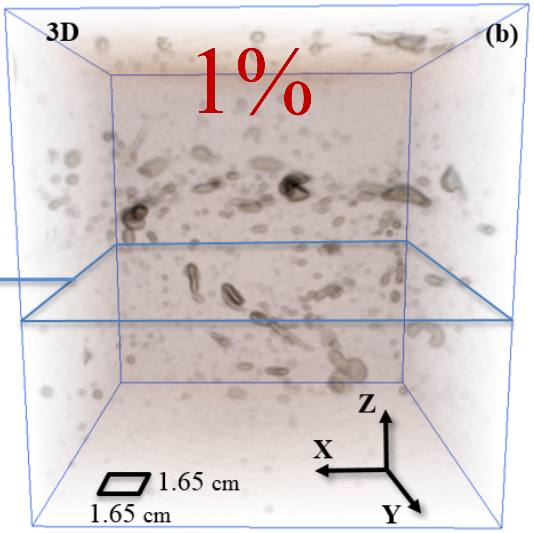


Moini et. al., RILEM, ETH. 2018

How about the cast specimen?

Cast:

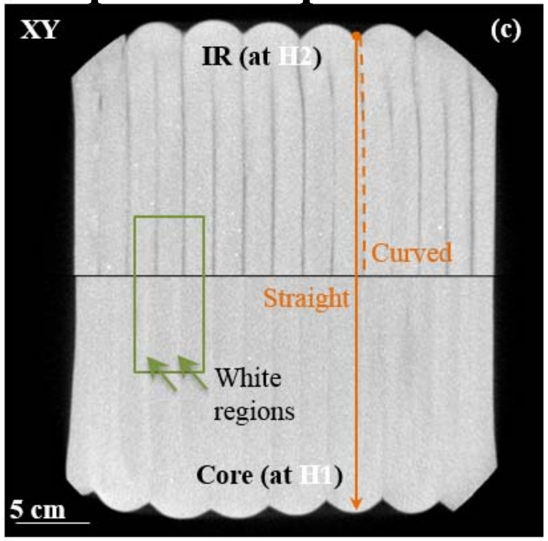


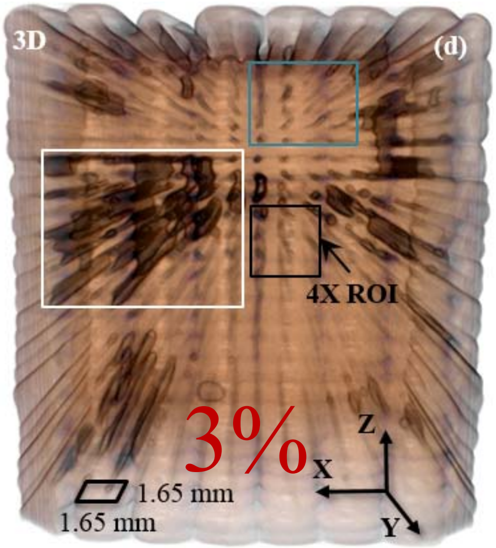


- Randomly Distributed Pores in cast

VS.

3D-printed hcp :

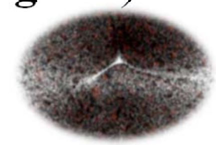




- Patterned Pore Network in 3D-printed layered specimen
- Volumetric Segmentation

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 mircostructure and porosity.

Acknowledgment

<u>Support:</u>

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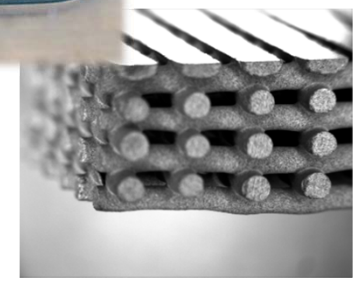
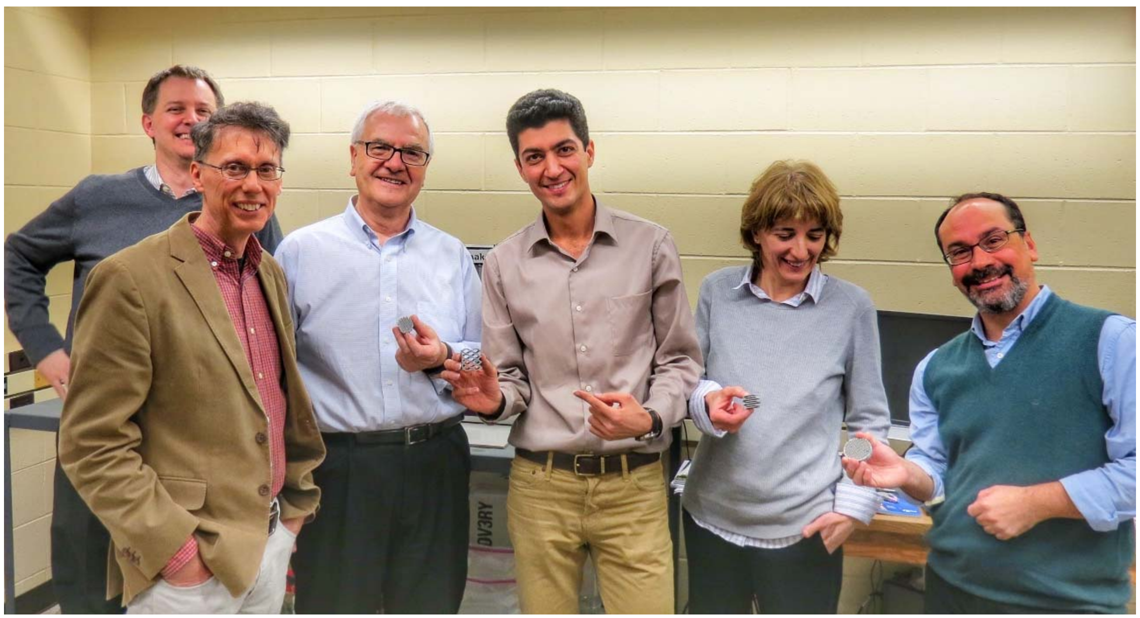
Purdue College of Engineering





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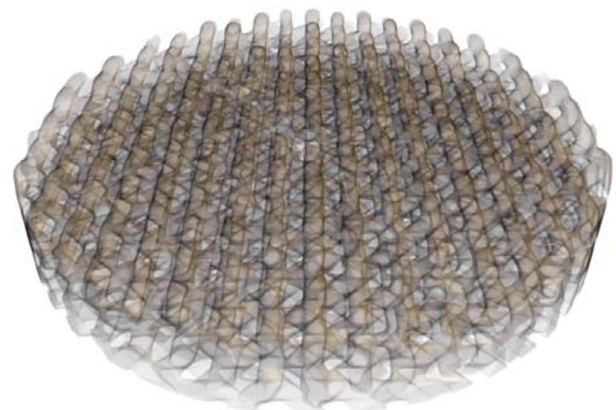
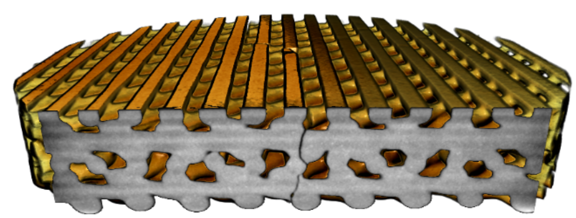
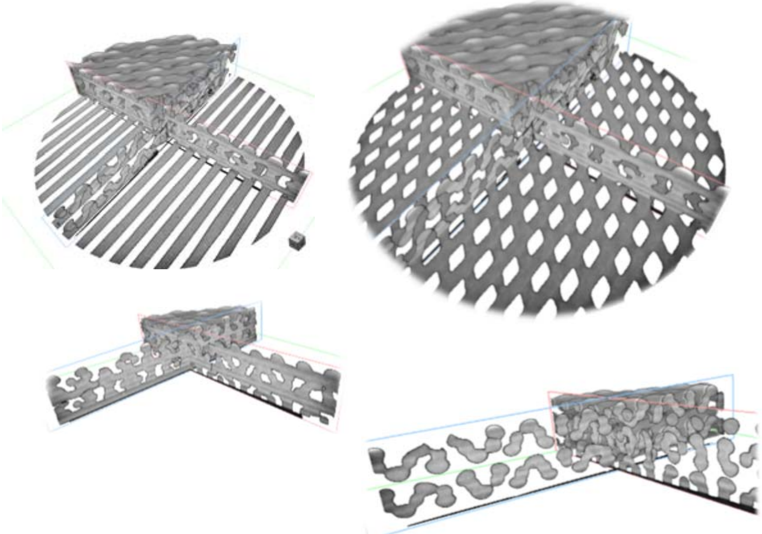


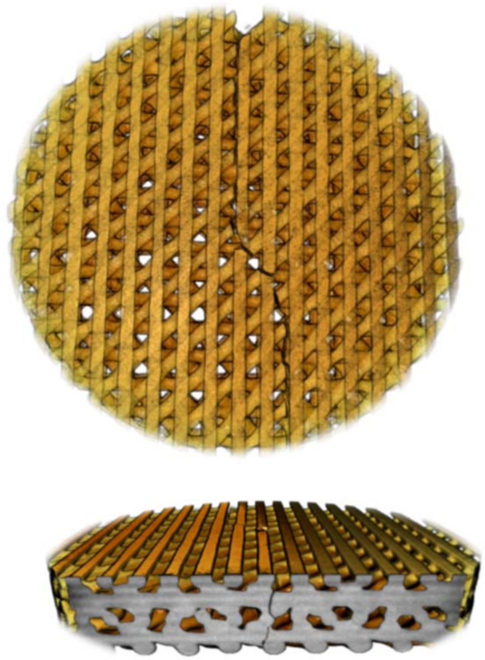


Contact: Mmoini@purdue.edu

Thank you!

Questions?





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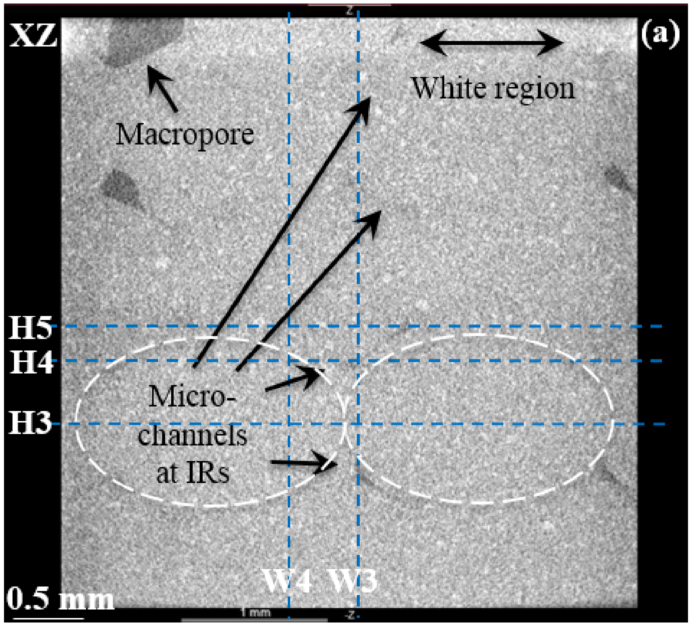
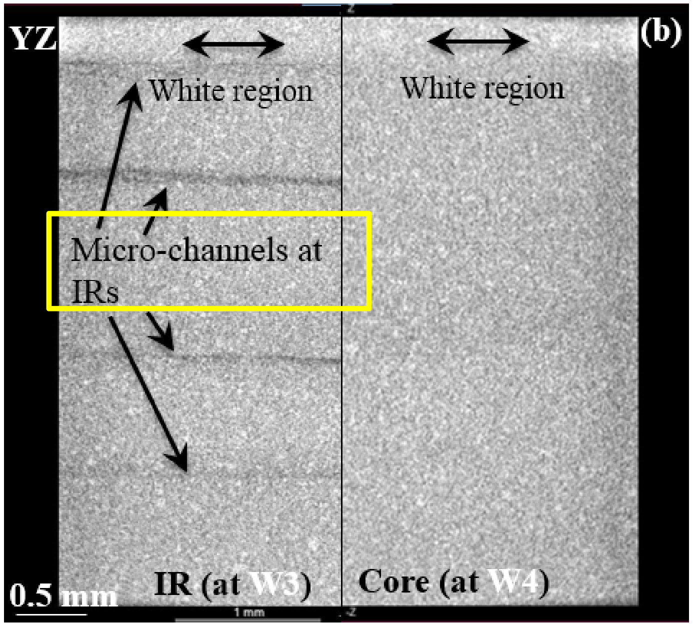
Back-up slides

Microstructural Features (4X): <u>Pores/Re-arrangements/White regions</u>

Side View



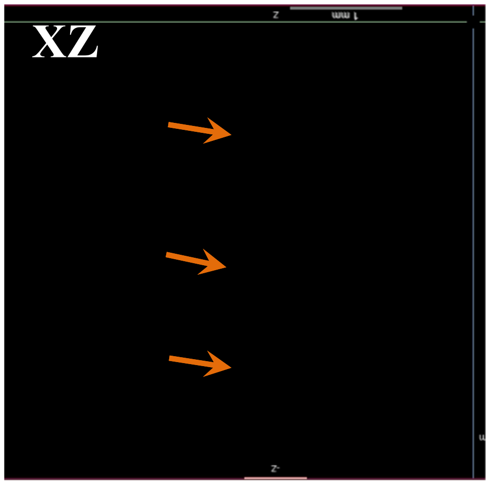


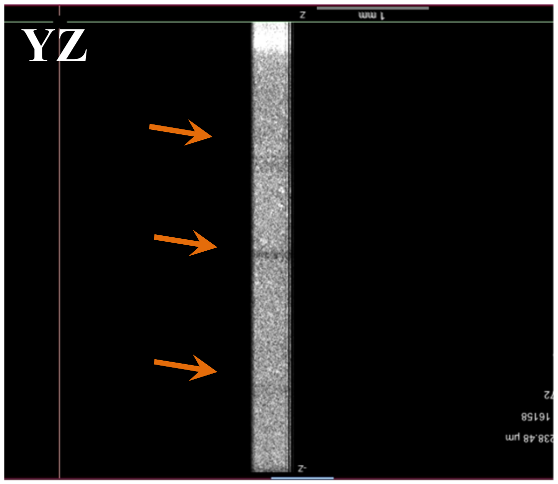
- <u>Micro-channels</u>

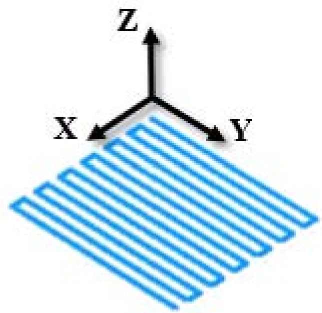
  Connectivity at IRs

- <u>Re-arrangement</u>
- <u>White regions</u>

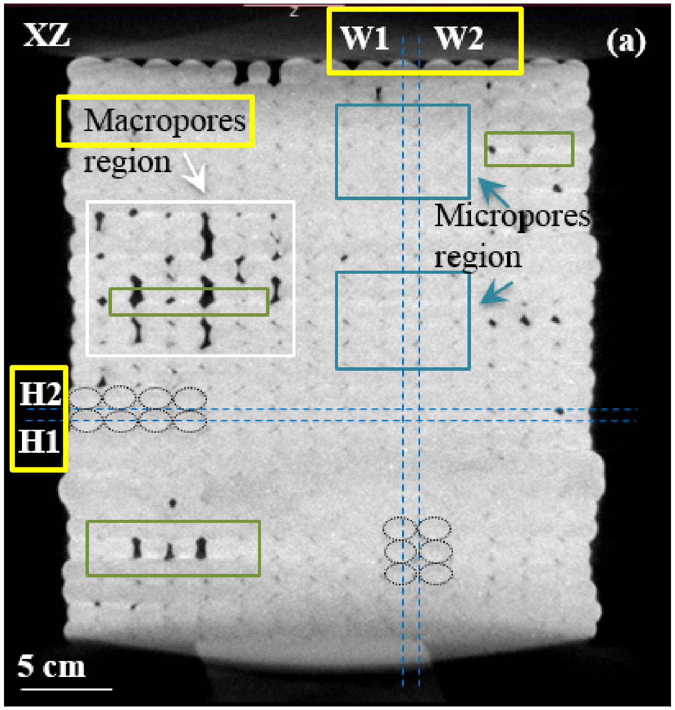
  Represent accumulation of anhydrous grains near the pores

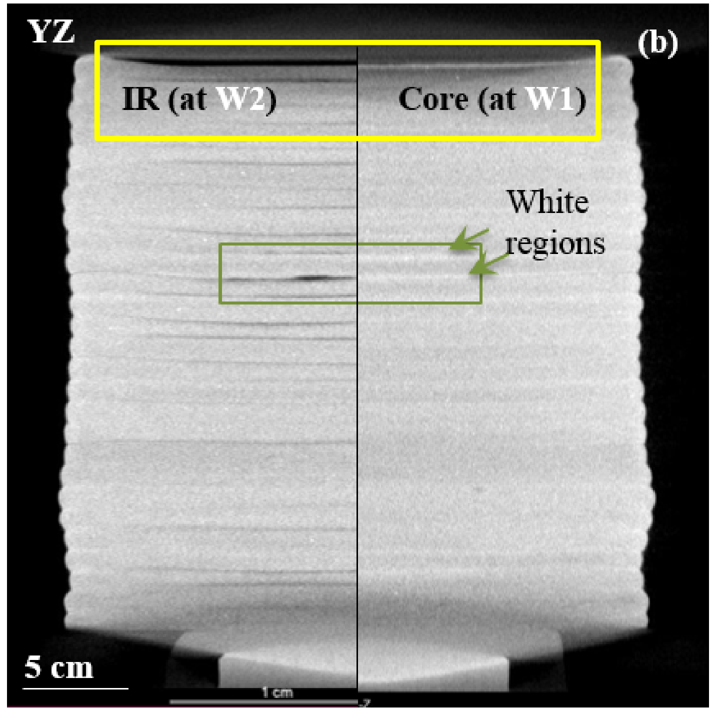


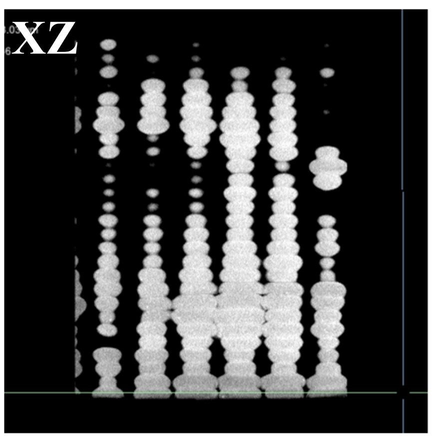




Microstructural Features: Micro- and Macro-Pores at IRs –0.4X Scan

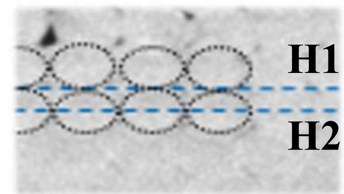






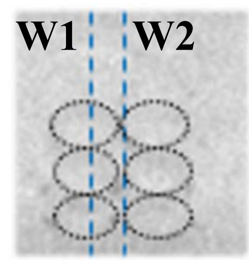
- **Micro-Pores:**

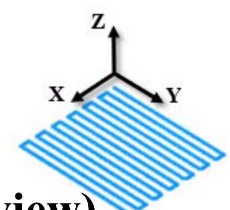
  at vertical and horiz. Planes (IRs)



- **Macro-Pores:**

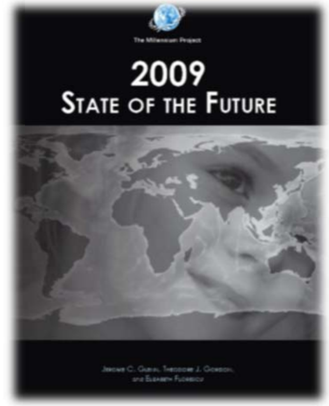
  at vertical planes





(Side view)

There is a huge need for advanced manufacturing in construction



<u>United Nation Goals for Sustainable Development</u>:

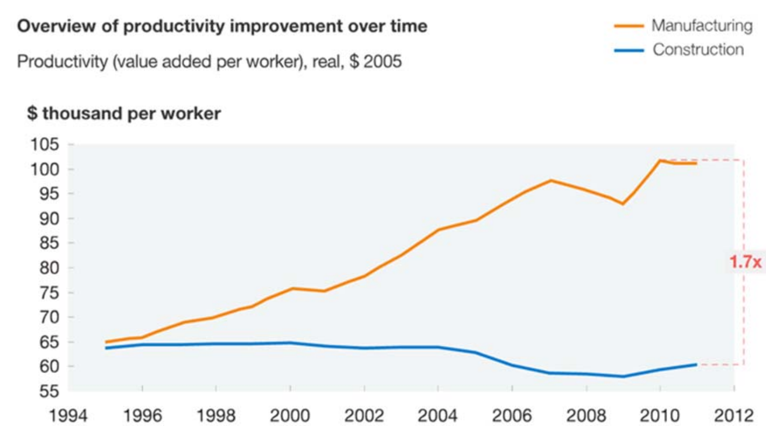
- Resilient infrastructure
- Safe and sustainable human settlement
- Sustainable use of terrestrial ecosystem

<u>National Academy of Engineers Grand Challenges for Engineers:</u>

- Restore and improve urban infrastructure

<u>Productivity Improvement In Construction</u>:

- Flat or declining



Source: Expert interviews; IHS Global Insight (Belgium, France, Germany, Italy, Spain, United Kingdom, United States); World Input-Output Database

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

<u>World wide efforts on 3D printing *concrete*</u>:

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

http://www.totalkustom.com/home.html

www.erdc.usace.army.mil

www.TUe.nl/3DConcretePrinting

www.genesisdimensions.com/

http://www.gizmag.com/china-winsun-3d-printed-house/31757/

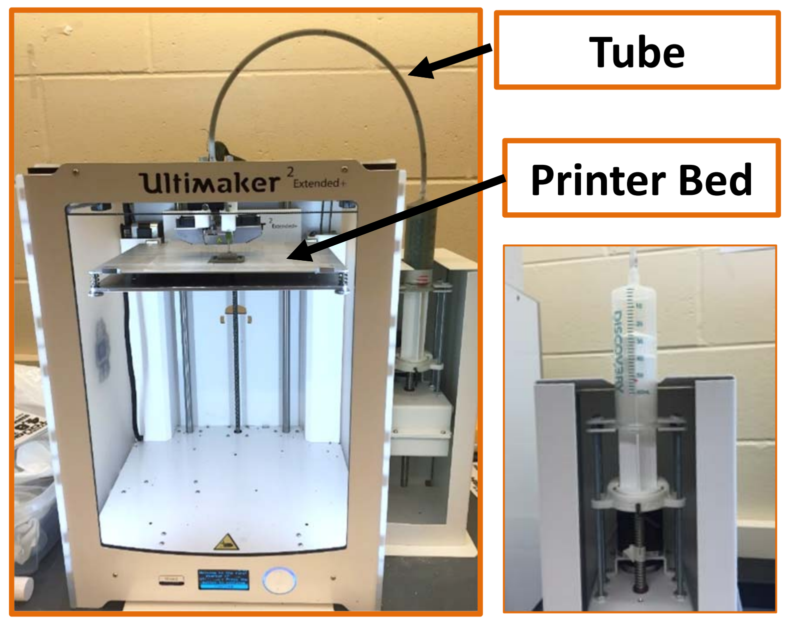
http://www.washingtonpost.com/news/innovations/wp/2015/02/05/yes-that-3d-printed-mansion-is-safe-to-live-in/

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

- **DIW**
  - Robocasting → Colloidal Gel
  - Micropen Writing → Colloidal Fluid
  - Fused Deposition → Polymer Melt
- **Photo-curable Inkjet**
  - Photo-polymer Resin
    - SLA
    - Polyjet
    - Two-photon
- **SLS/SLM**
  - Metal
  - Ceramic
  - Plastic
  - Glass
  - (Powders)

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

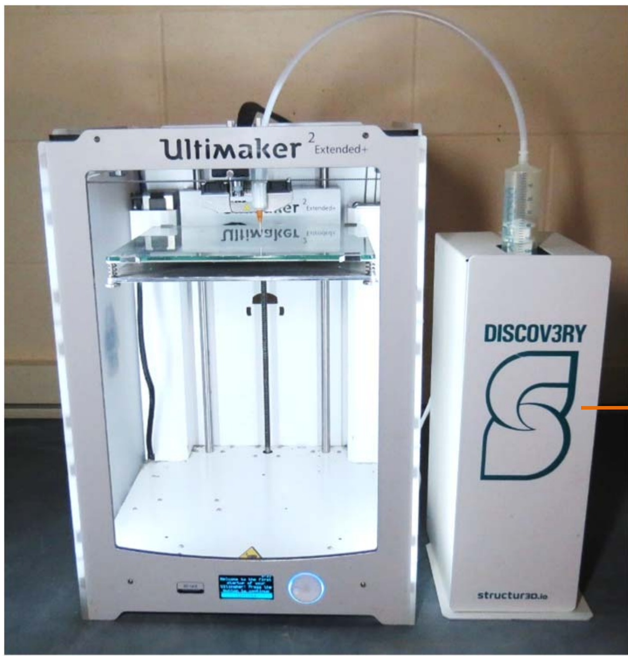


Printing Platform for DIW of Cement Paste

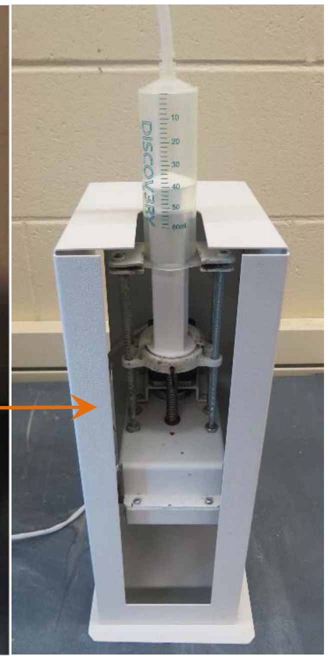
A Fused Filament Fabrication (FFF) Printer is merged with an extruder system and is modified to serve DIW of cement paste

- 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.

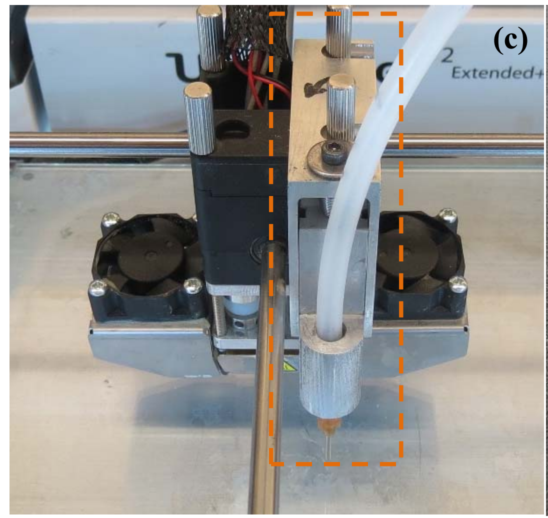
Nozzle Dia.: 1.36 mm
Layer Height: 1.00 mm

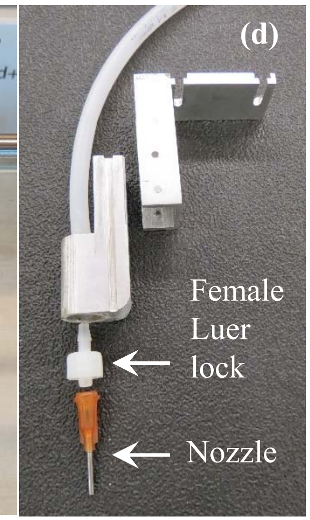


Fused Filament Fabrication (FFF) 3D Printer

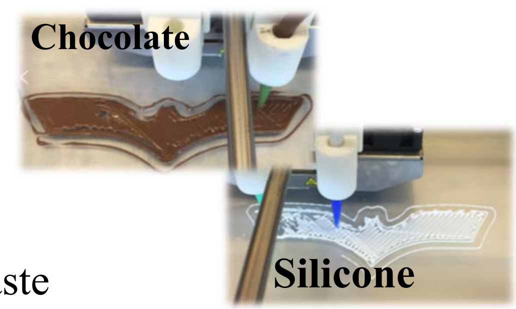


Stepper motor-driven extrusion system (Syringe and Plunger)





Nozzle holder assembly

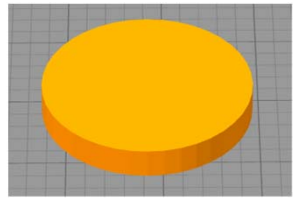


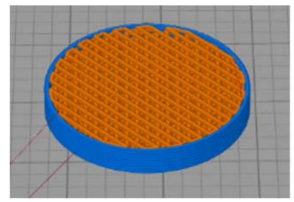
Connection to tube

- <u>Processing parameters</u> were optimized for cement paste

Printing Parameters ?

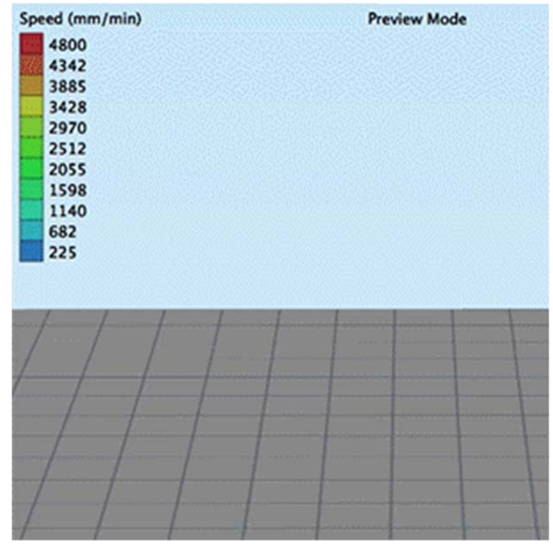
- <u>**Slicer (Interpreter)**</u>:
  Converts a digital 3D model into printing instructions for 3D printer



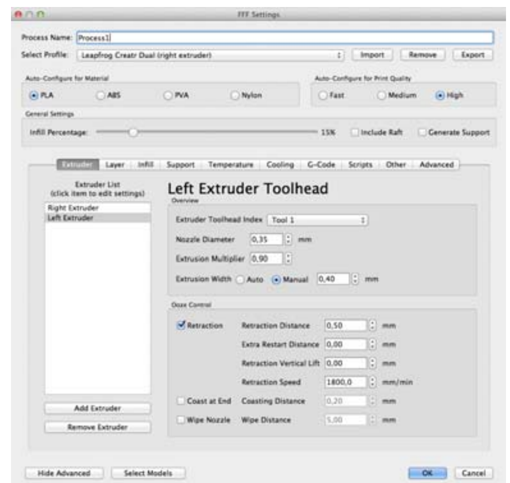


- **Input: STL file** (Geometry of printed object) and several **printing parameters**,

printing parameters:



  - ➢ Nozzle Size
  - ➢ Extrusion multiplier
  - ➢ Print speed (F)
  - ➢ Infill %
  - ➢ Layer height
  - ➢ Extrusion width
  - ➢ Movement speed
  - ➢ Bed Temperature, Fan, etc.
  - ➢ ……



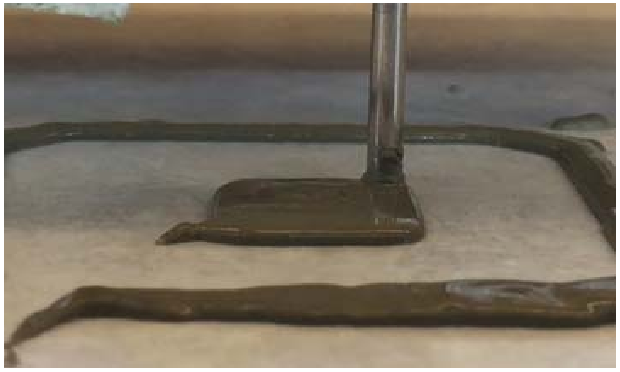
- **Output: G-code** (machine-readable **toolpath** commands)
  - ➢ **5-Axis** commands: Coordinates (X, Y, Z), E, F

Cement formulation has a large effect on print quality

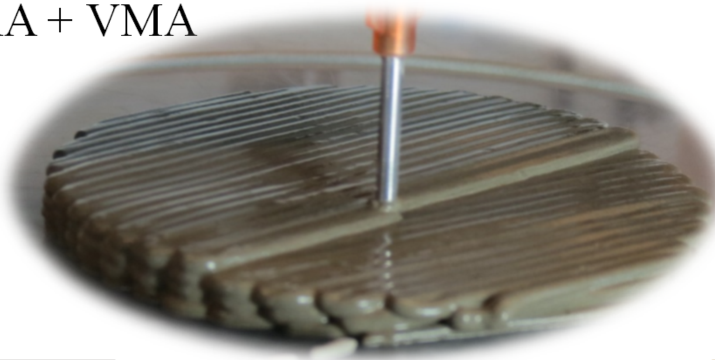
Ink Formulation:

- Cement Paste
  (Not extrudable)

- Cement Paste + HRWRA



- Cement Paste + HRWRA + VMA



<u>Challenges at small scale</u>:

- 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.**

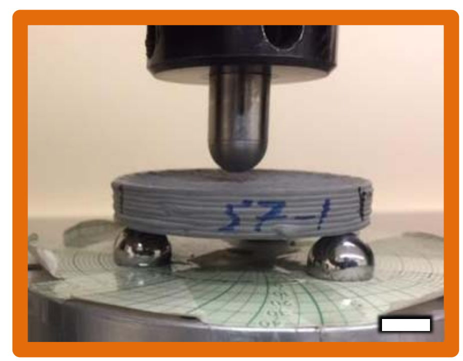
Fundamental Behavior under Flexure and Tension:

- 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

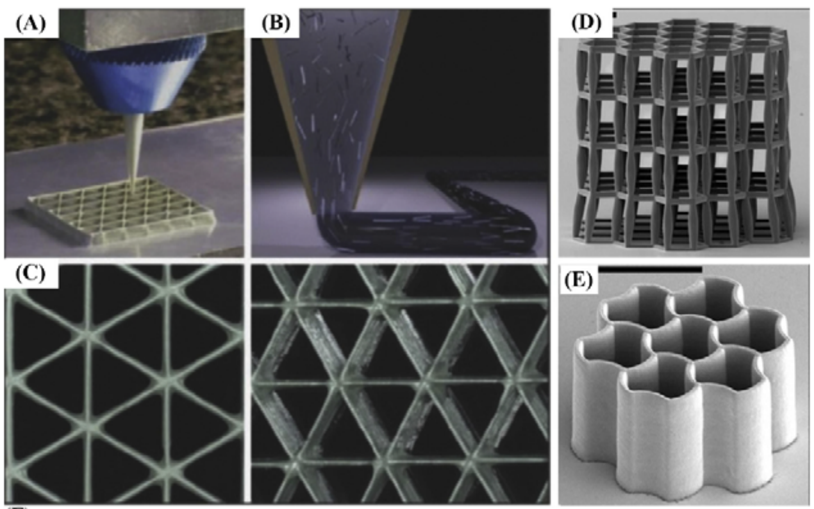


Scale bars: 5.0 mm

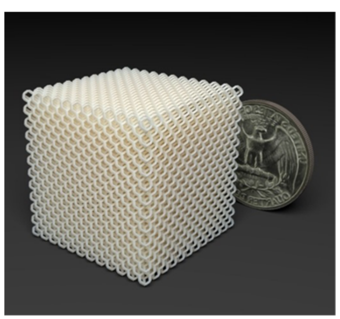
Now we can 3D-print cement- What are we going to do with it?

"**Architectured Materials**":

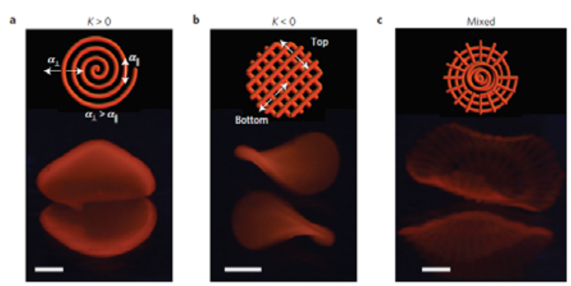
- Combination of **materials** and **space**
- Materials engineered to have **new properties**
- Properties not offered by material or structure alone



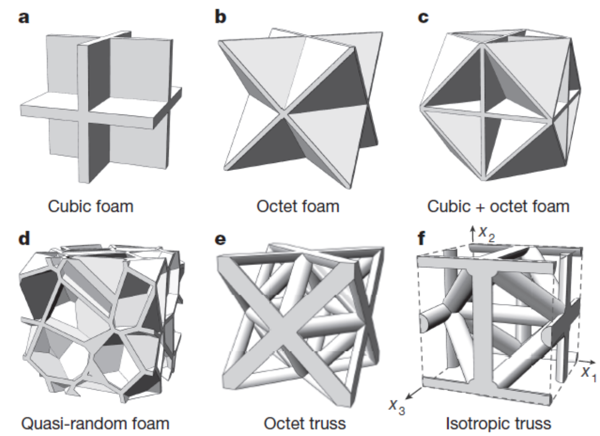
Zheng, et al, Progress in Materials Science, 2015. HC.

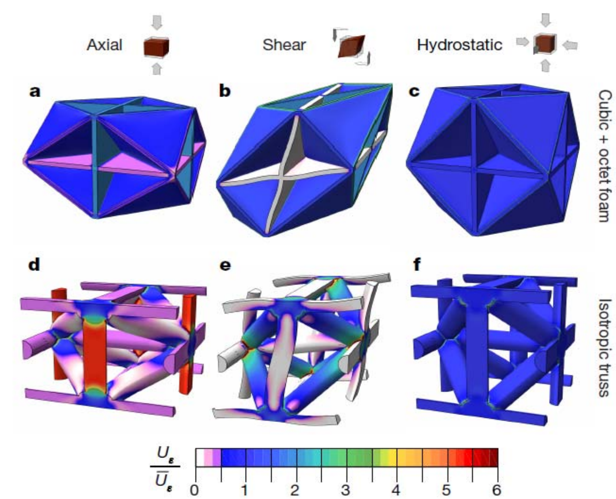


http://vcg.isti.cnr.it/Publications/2015/PZMPCZ15/



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

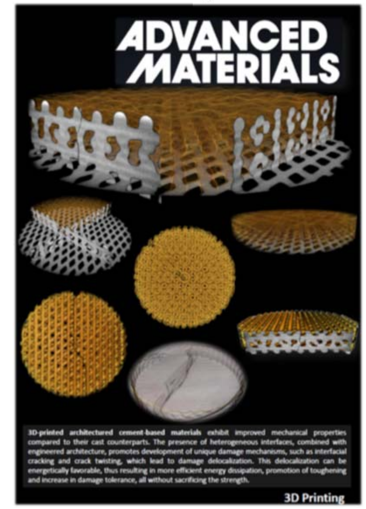




Berger et al. Nature Letter, 2017. , Mech. Metam.

Further Info:

“Frontispiece”

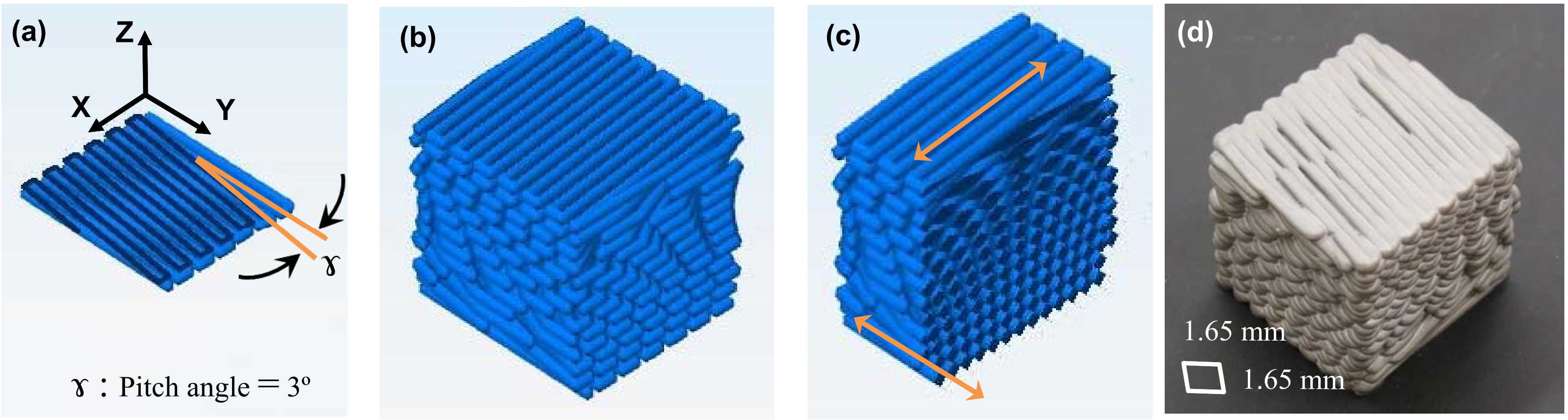


- Mohamadreza Moini, Jan Olek, Jeffrey Youngblood, Bryan Magee, Pablo D. Zavattieri, ***“Additive Manufacturing and Performance of Architectured Cement-based Materials”***, *Advanced Materials,* **2018**. https://doi.org/10.1002/adma.201802123.

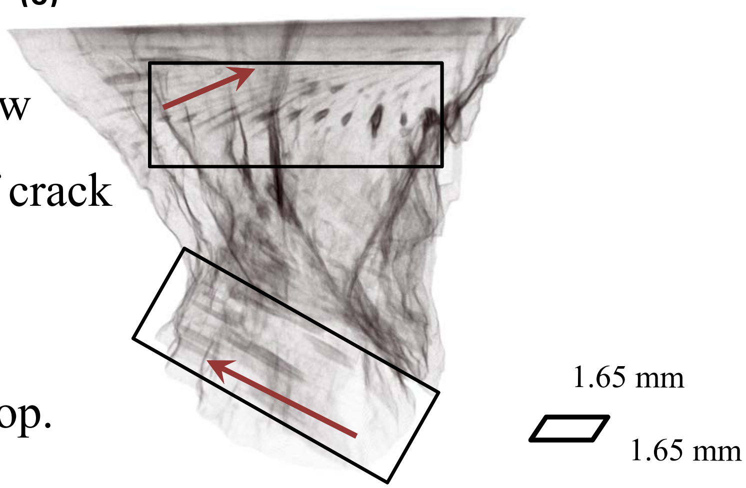
- Mohamadreza Moini, Jan Olek, Bryan Magee, Pablo Zavattieri, Jeffrey Youngblood, ***“Additive Manufacturing and Characteristics of Architectured Cement-based Materials via X-ray Micro-Computed Tomography”***, 1st RILEM International Conference on Concrete And Digital Fabrication, ETH, Zurich, Springer proceeding, **2018.** 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**



(e)

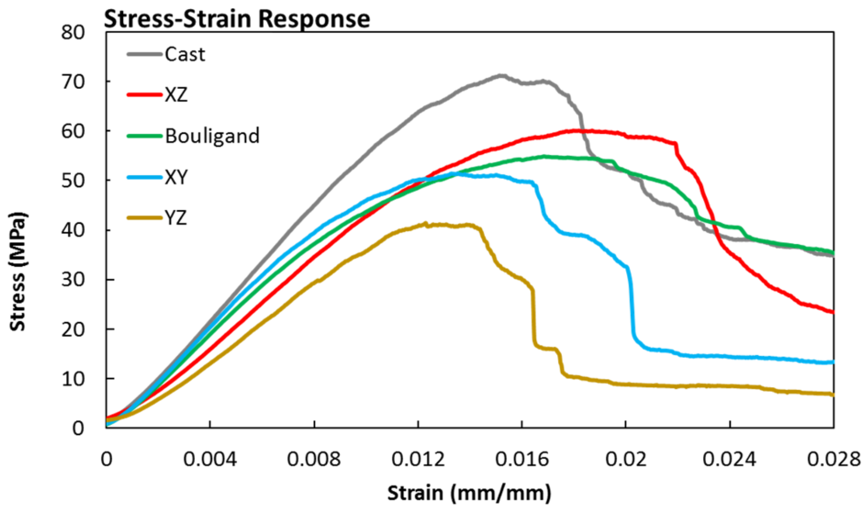


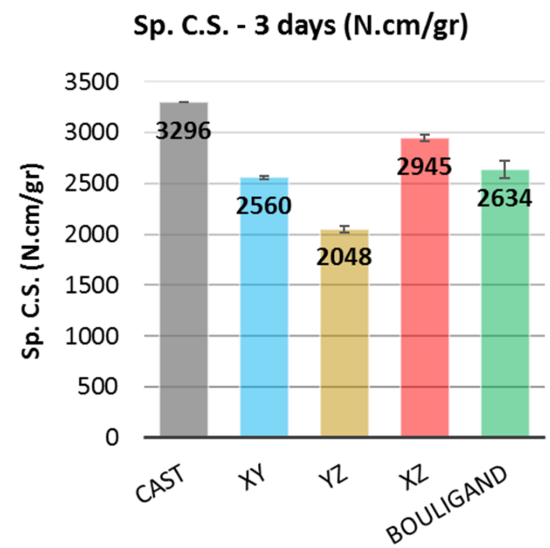
- ➢ IR affect damage propagation and allow
- ➢ Damage **delocalization** and control of crack propagation path
- ➢ That could cause **anisotropic** mech. prop.

Damage-Property-Microstructure Relationship (in Compression)

Properties-Microstructural Architecture

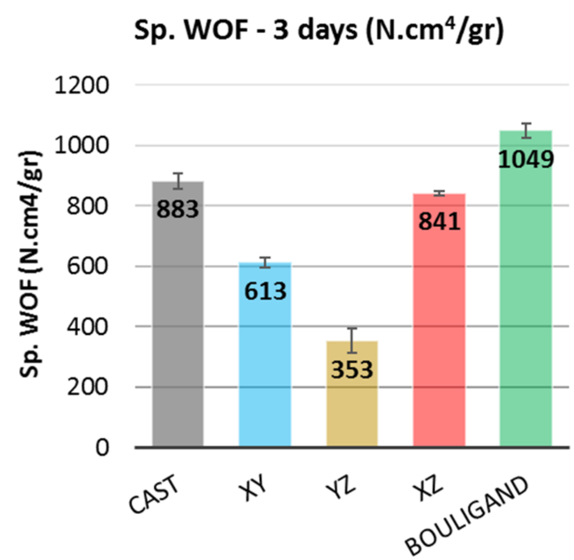
- Layer-wised lamellar 3D-printed cement-based elements have anisotropic compressive strength properties





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

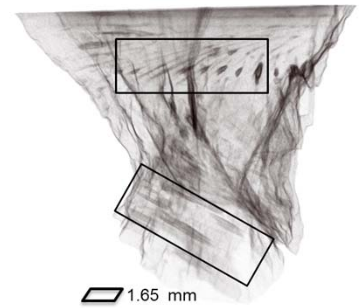
	Cast	XY	YZ	XZ	Bouligand
		Sp. WOF.			
Cast		Sig.	Sig.	**Non Sig.**	**Sig.**
Lamellar - XY	Sig.		Sig.	Sig.	Sig.
Lamellar - YZ	Sig.	Sig.		Sig.	Sig.
Lamellar - XZ	**Non Sig.**	Sig.	Sig.		Sig.
Bouligand - XY	**Non Sig.**	**Non Sig.**	Sig.	Non Sig.	
	Sp. C.S.				



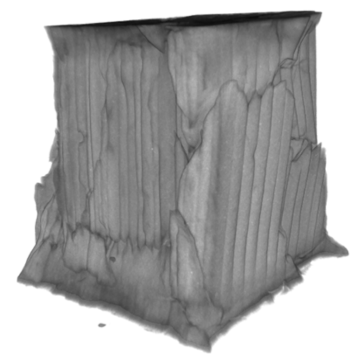
Damage-Property-Microstructure Relationship (in Compression)

Damage-microstructural Architecture Interaction:

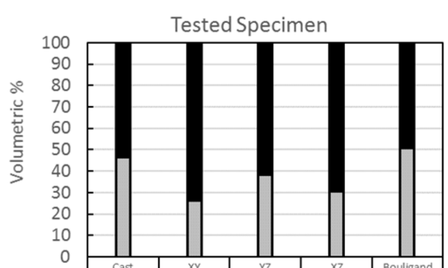
- 3D printed cement-based elements present <u>interfacial cracking</u> and <u>damage mechanisms</u> that are different from the cast counterparts.



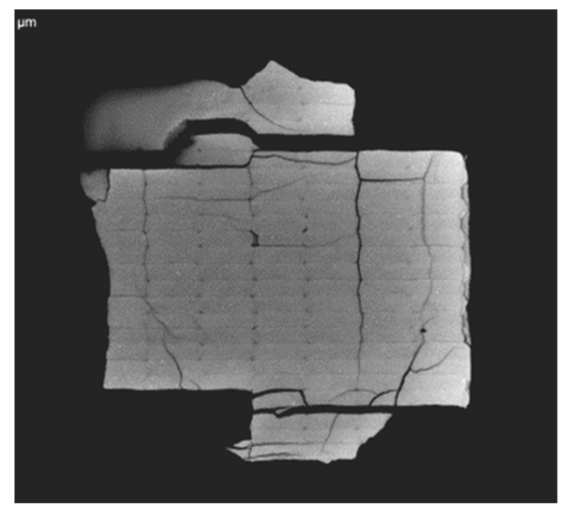
- These mechanisms can result in lower, higher, or similar compressive strength and WOF depending on the interaction between cracking and <u>architecture</u> (and <u>testing direction).</u>



- A cracking-microstructure interaction exist through which <u>interfacial damage (crack and micro-crack)</u> is promoted.



	Cast	XY	YZ	XZ	Bouligand
Int.	53	74	62	70	49
Damaged Ext.	47	26	38	30	51

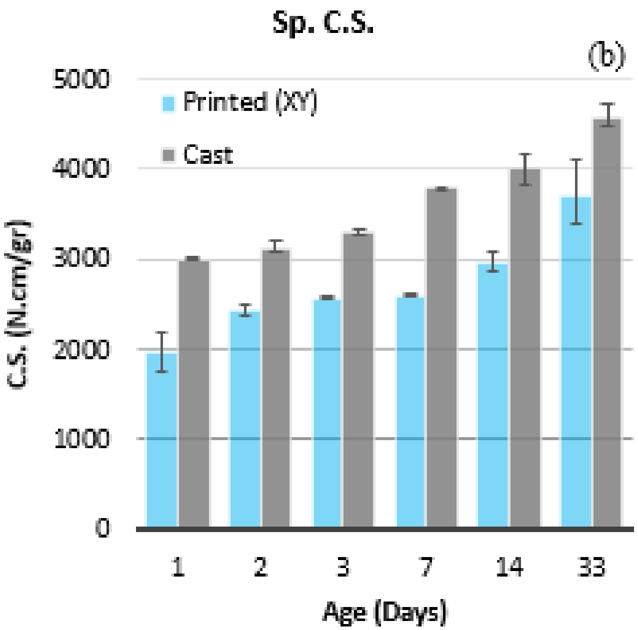


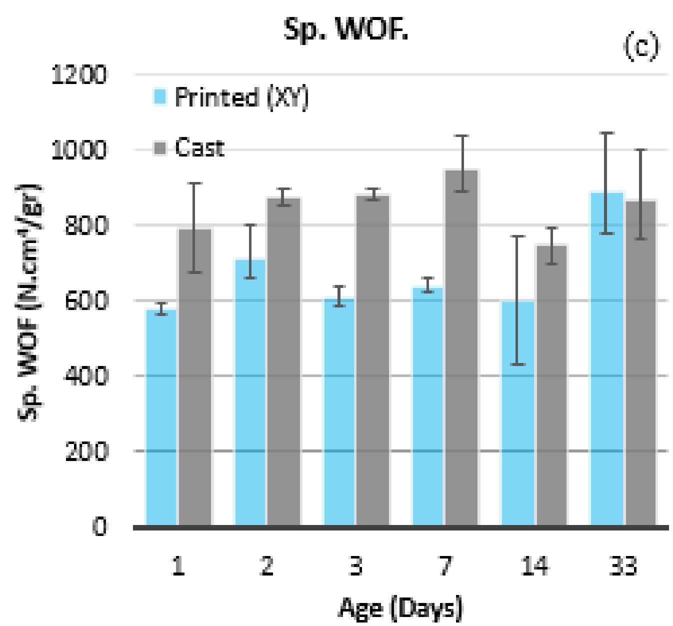
Damage-Property-Microstructure Relationship (in Compression)

Other Interesting Findings:

- Strength development of materials over time can result in significant different in mechanical properties of 3D-printed elements compared to cast counterparts.

- This may be due to the evolution of *Materials Strength/Interfacial Strength*.





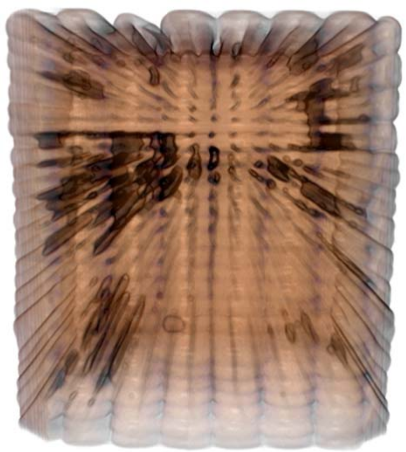
Age		1	2	3	7	14	33
Difference between C & P	Sp. C.S.	Non-Sig	Sig.	Sig.	Sig.	Sig.	Non-Sig
	Sp. WOF.	Non-Sig	Sig.	Sig.	Non-Sig	Non-Sig	Non-Sig

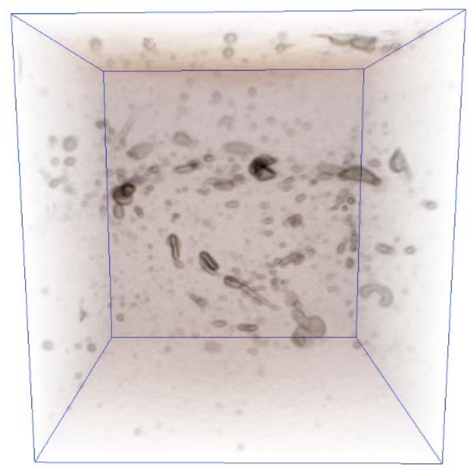
Damage-Property-Microstructure Relationship (in Compression)

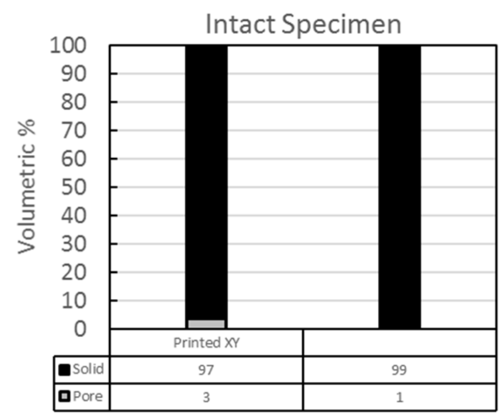
Properties-Microstructural Architecture

- 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 <u>morphology of the architecture of the porosity</u> can be as critical as the <u>total amount of porosity</u> to the strength-property relationship.



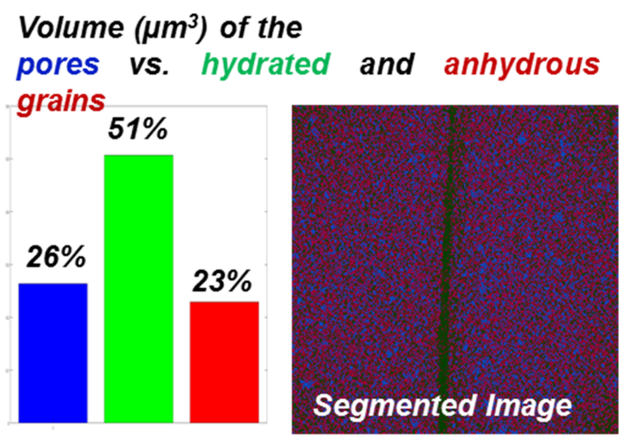




Damage-Property-Microstructure Relationship (in Compression)

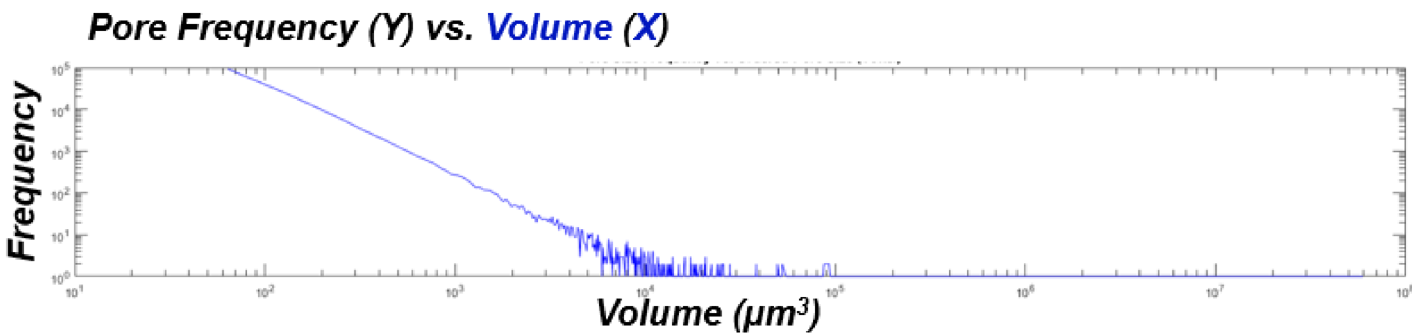
Properties-Microstructural Architecture

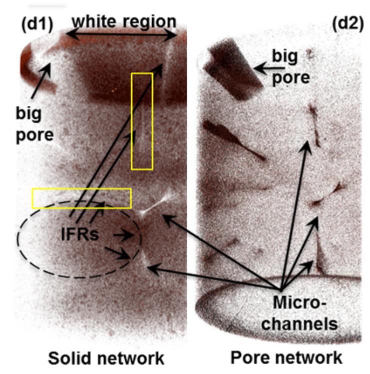
- Microstructural architecture (i.e., 4 adjacent filament in 2 layers) of a 3-day-old cement paste was found to have a 25-25-50% proportion
- for the three pores-hydrated-unhydrated phases



- This includes interfacial and microstructural porosity

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

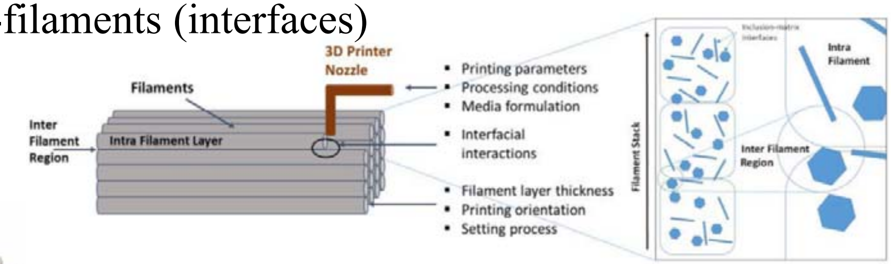


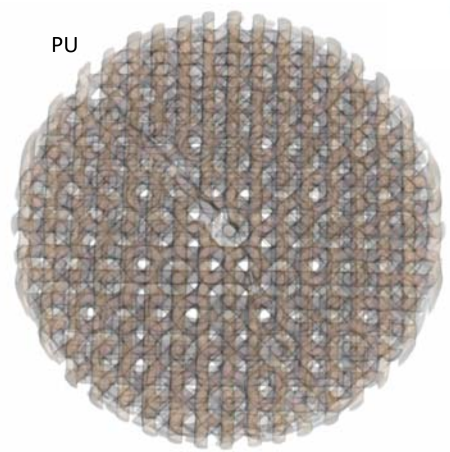


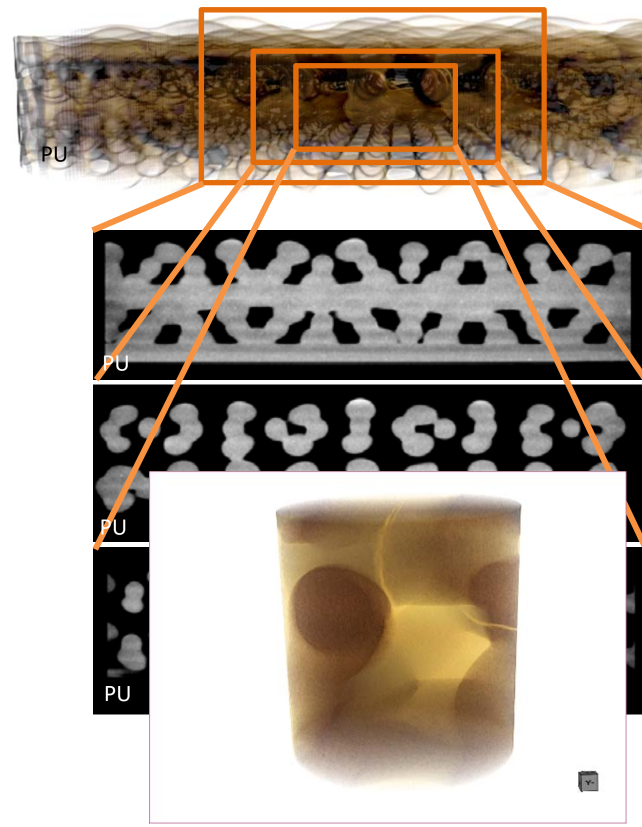
Microstructural Architecture

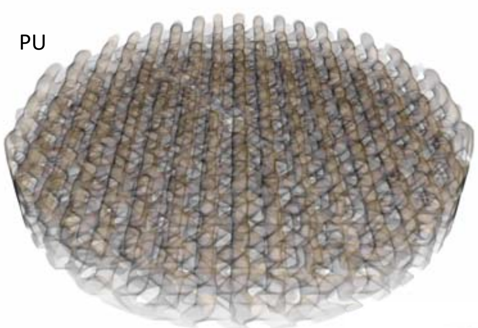
Microstructural architecture can be controlled via design and DIW to investigate intertwined mechanisms

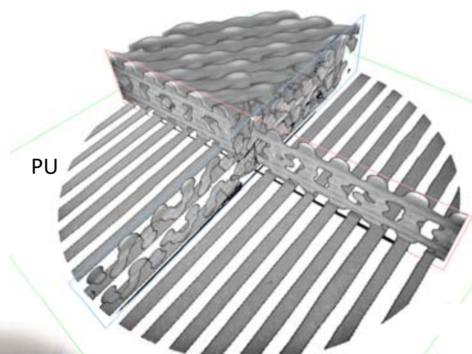
- (Micro)structure : Characteristics of intra- and inter-filaments (interfaces)
- Processing (i.e., extrusion)
- Properties (fresh and hardened)

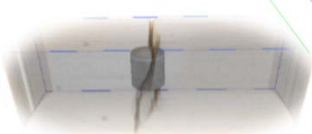






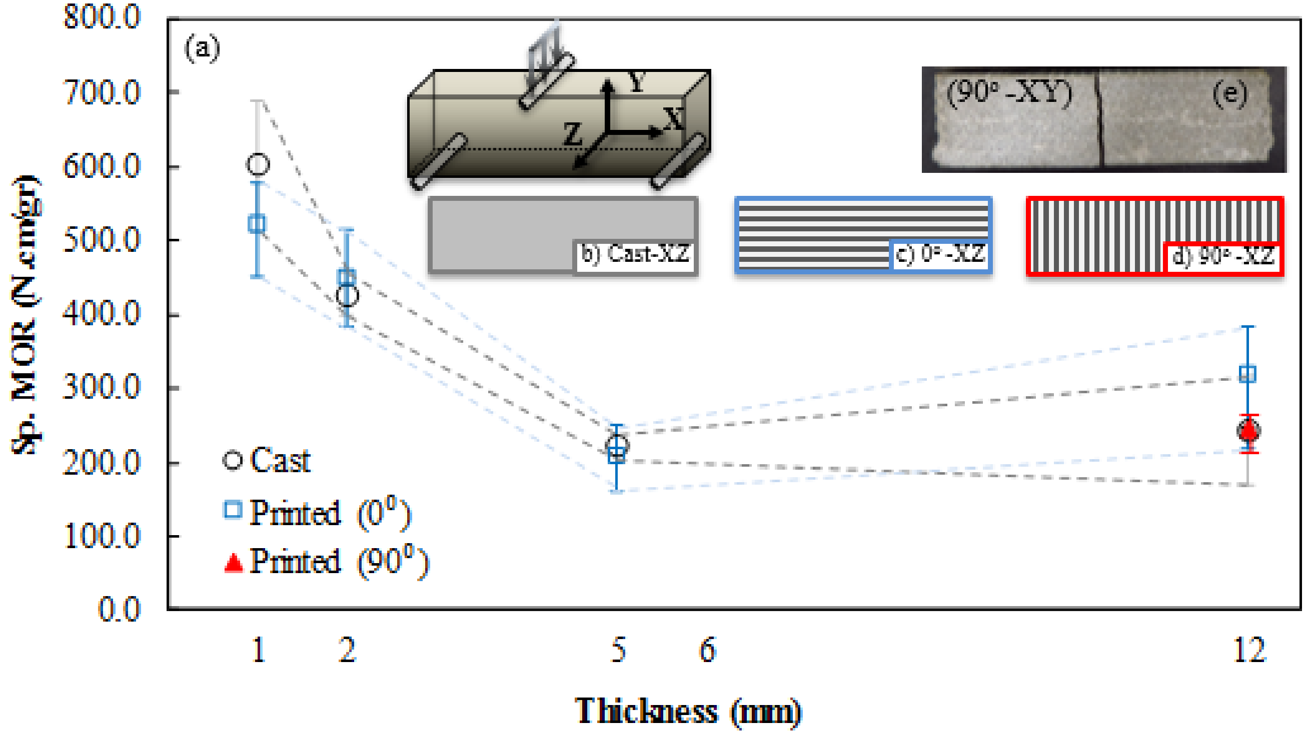






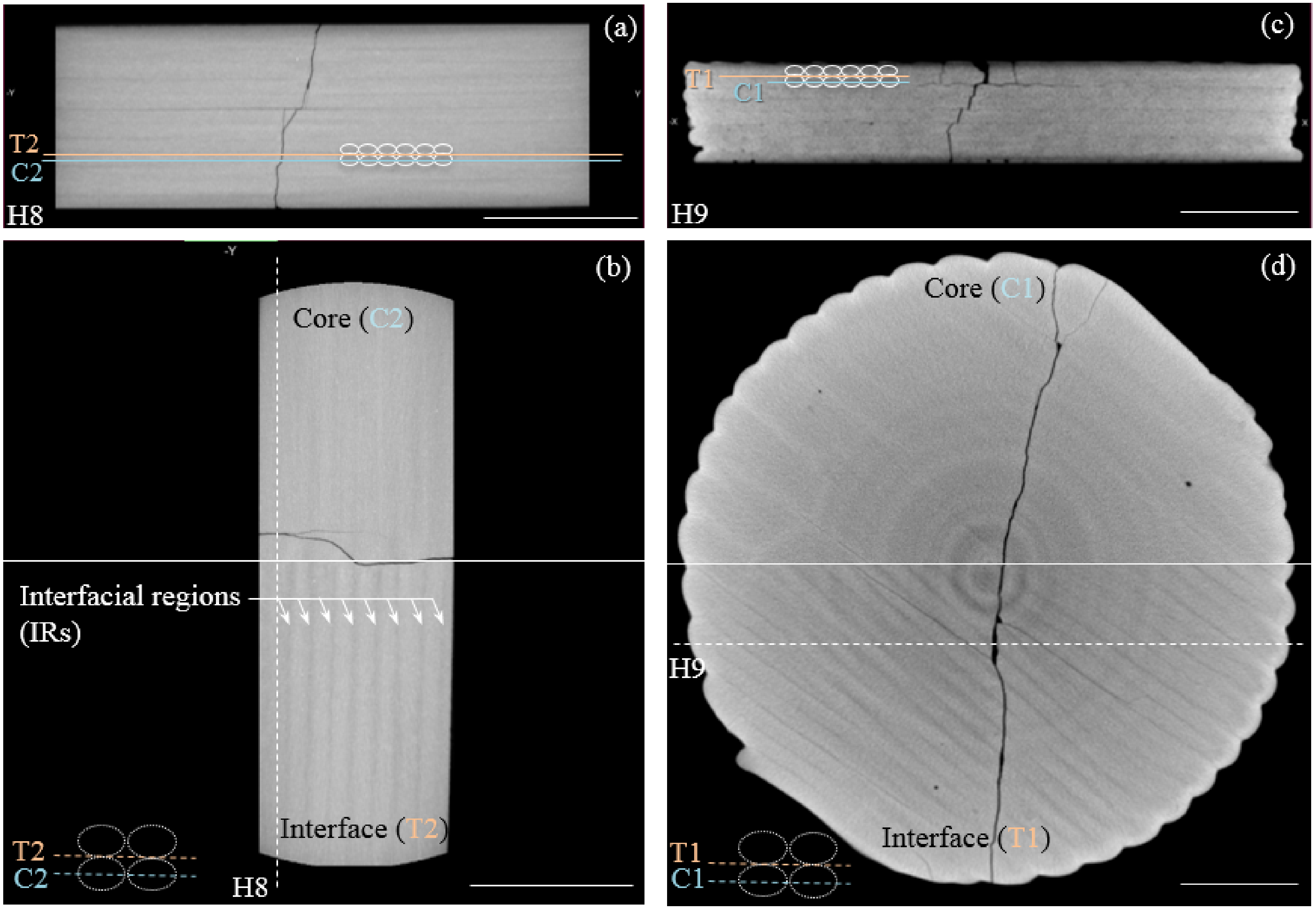
Interface is porous, but how weak is its strength?

Using 3PB:

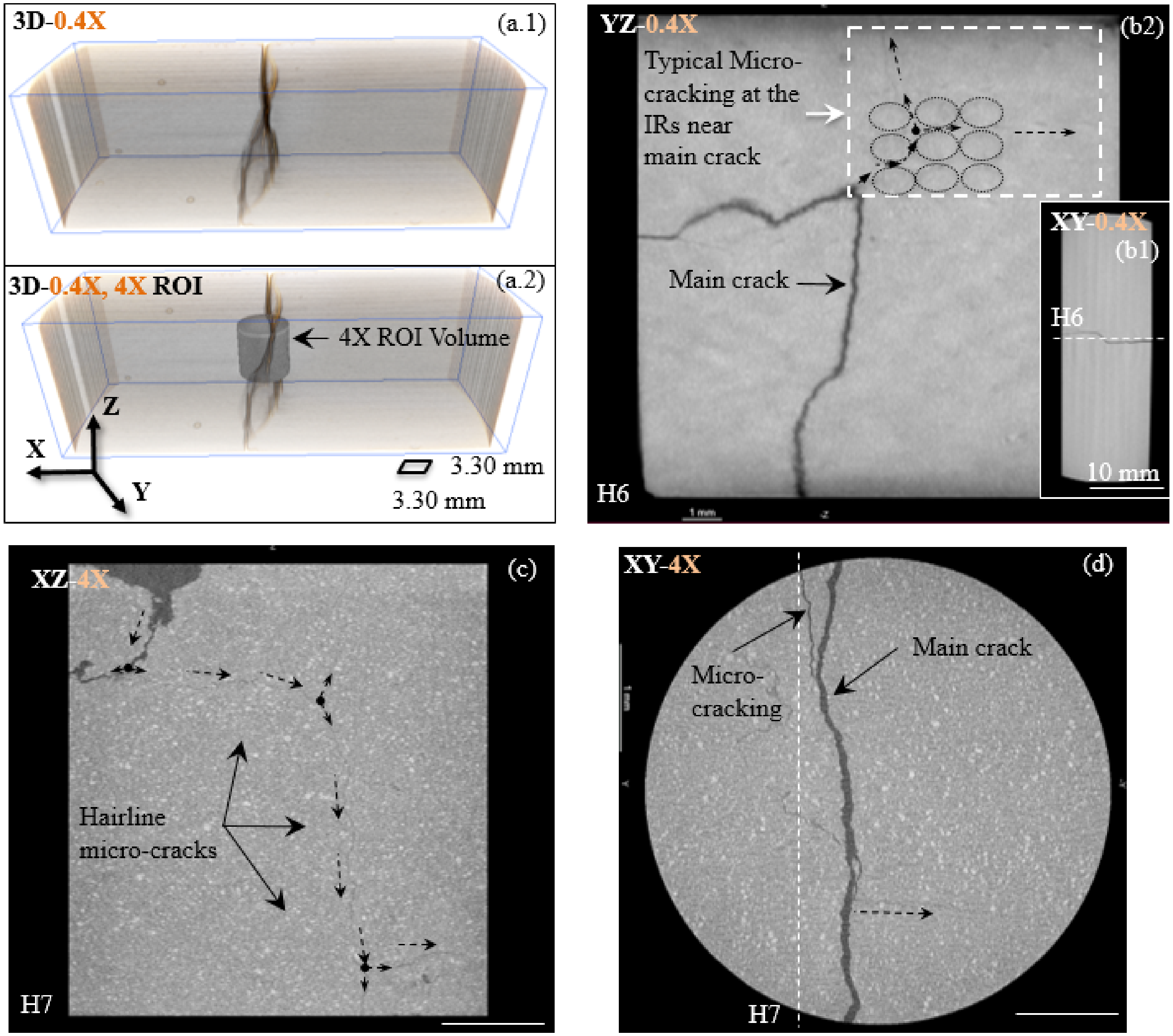


- Although the interfaces was identified weak and porous
- No significant difference was found between interfacial strength and bulk materials strength when structure was collectively tested in 3PB

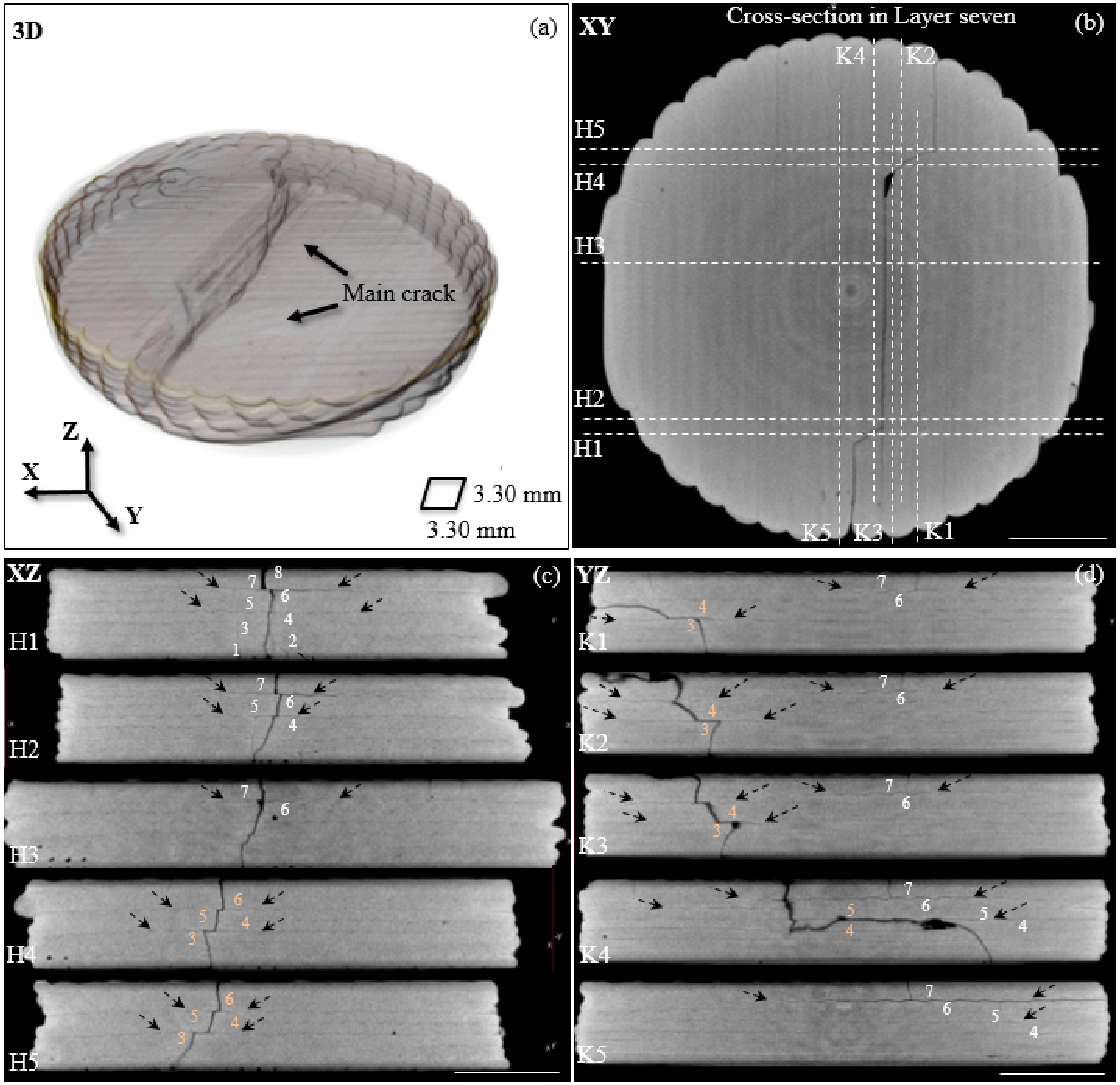
…by controlling crack path during failure

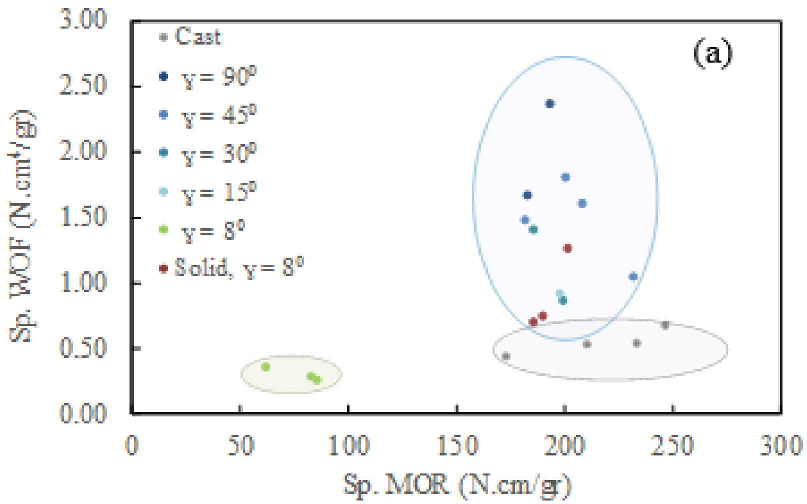


…by controlling crack path during failure

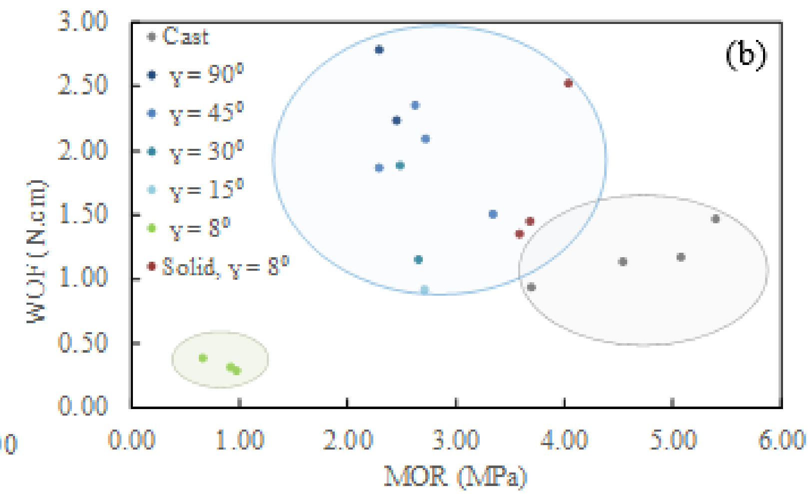


…by controlling crack path during failure



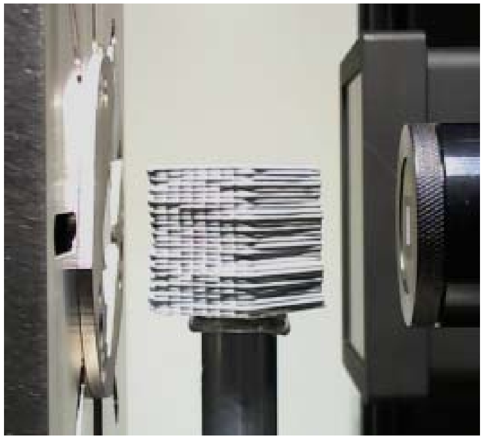


(a)
Cast
γ= 90⁰
γ= 45⁰
γ= 30⁰
γ= 15⁰
γ= 8⁰
Solid, γ= 8⁰
Sp. WOF (N.cm⁴/gr)
Sp. MOR (N.cm/gr)
3.00
2.50
2.00
1.50
1.00
0.50
0.00
0
50
100
150
200
250
300



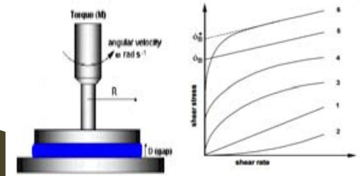
(b)
Cast
γ= 90⁰
γ= 45⁰
γ= 30⁰
γ= 15⁰
γ= 8⁰
Solid, γ= 8⁰
WOF (N.cm)
MOR (MPa)
3.00
2.50
2.00
1.50
1.00
0.50
0.00
0.00
1.00
2.00
3.00
4.00
5.00
6.00


ZEISS
Xradia 510 Versa

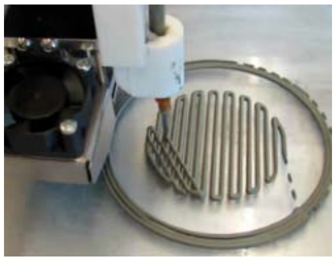


Key Factors in 3D-printing cementitious materials

Viscosity
Yield
…



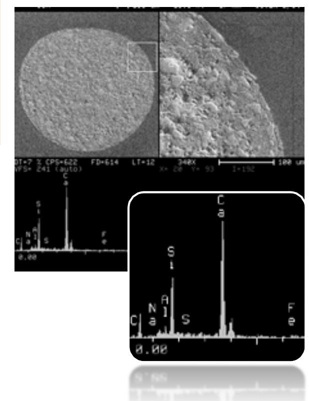
Paste Rheology/ Ink Development



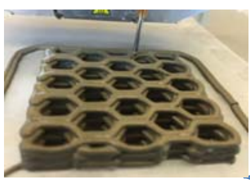
Flow Rate
Speed (XY)
Nozzle Size

Processing Parameters

Chemistry



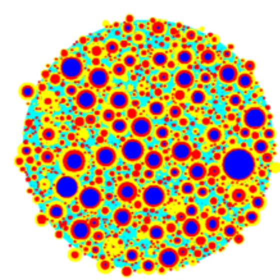
Key Factors in Objective I. & II



Print Quality
Height Stability
Width Consistency

Sample Outcome

Hydration



Hydr. Phases