Microstructural Heterogeneity, Fracture, and Transport in Layered 3D-Printed Cementitious Materials

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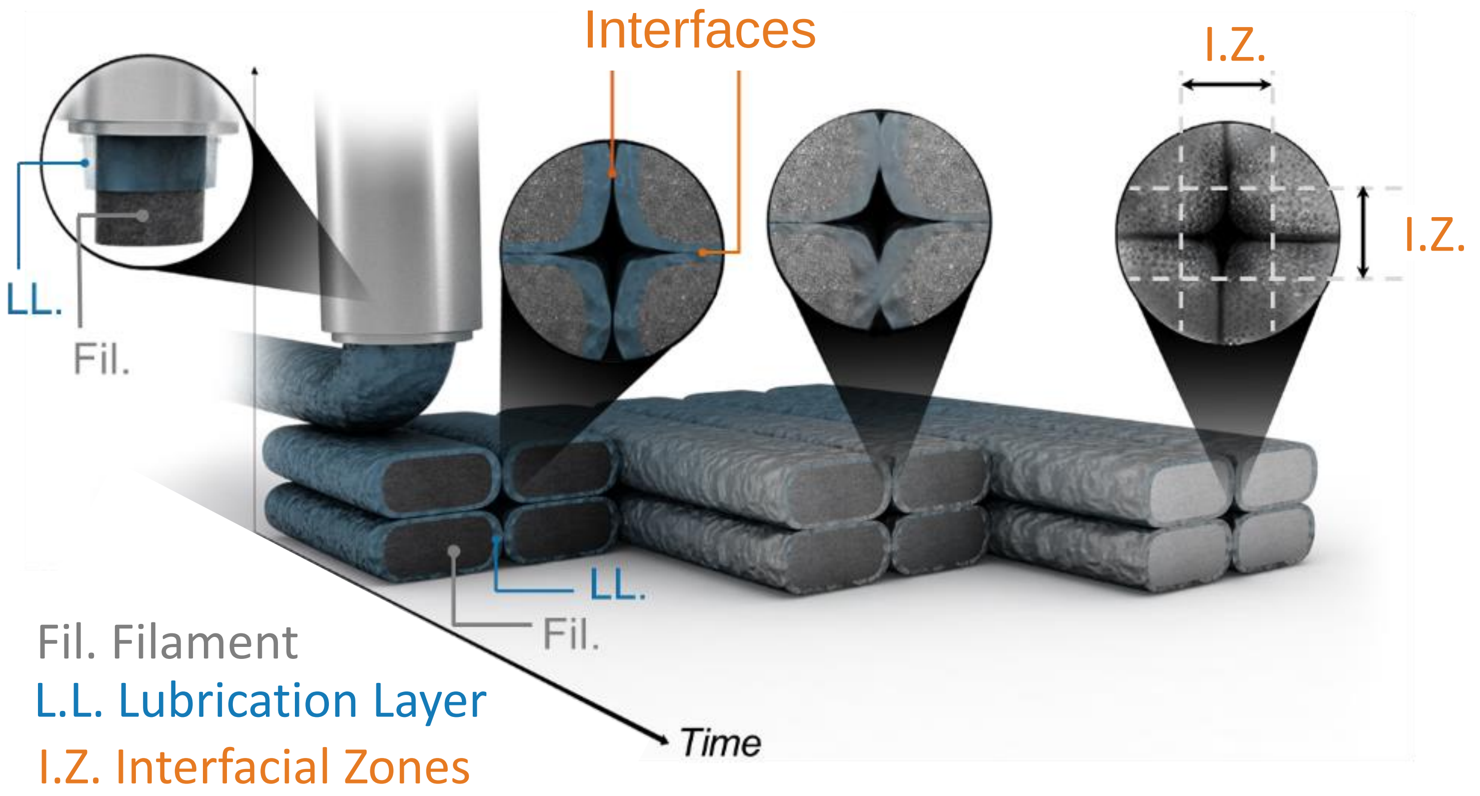
ARCHITECTED MATERIALS AND
ADDITIVE MANUFACTURING LAB

NSF

aci CONCRETE CONVENTION
April 02, 2023

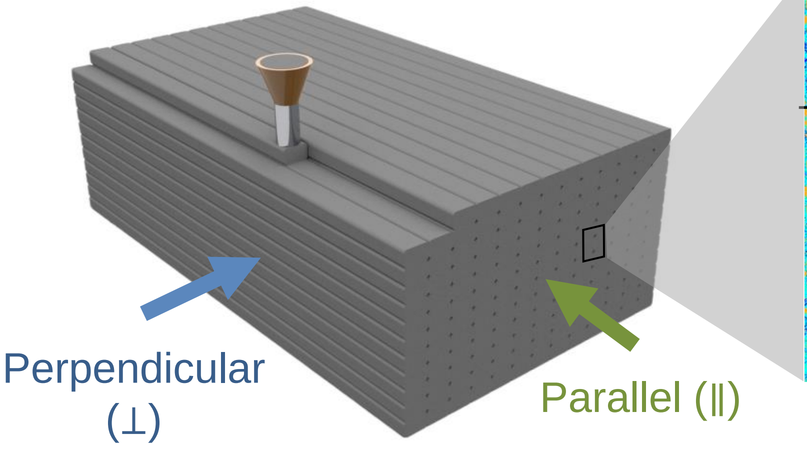
Background: Interfacial Zone in 3D-Printed Cementitious Materials Can be Weak

- Interfacial zones stem from the “Lubrication layer (LL)” during extrusion
- LL is water-rich region which may lead to flaws and heterogeneities in Interfacial zones
- Q. What does the microstructure of the interfacial zone look like?
- Q. What is the role of the interfacial zone on the material properties?

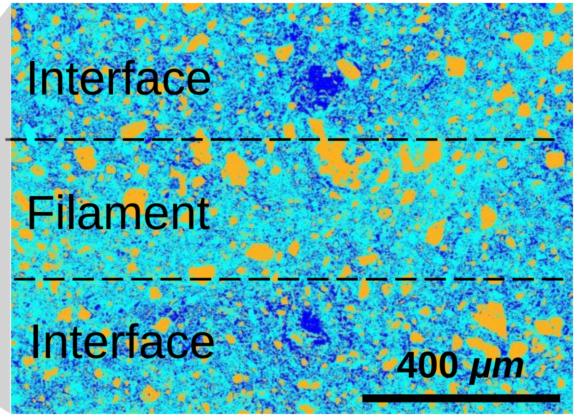


Objectives

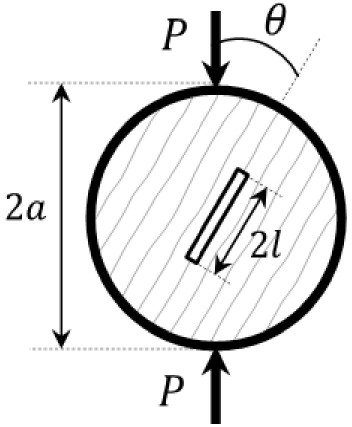
3D-printing of Cement Paste



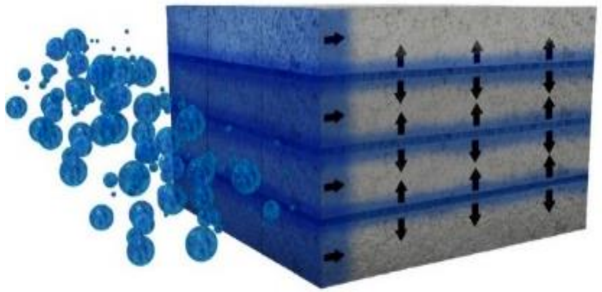
Interfacial Heterogeneities



Fracture Response



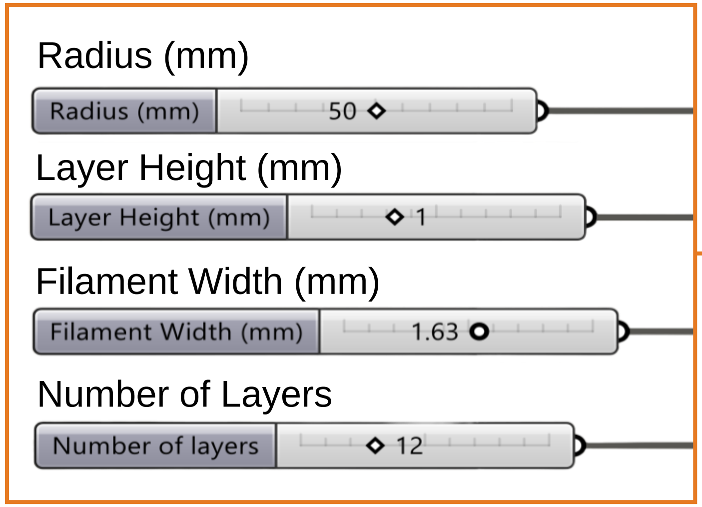
Transport Behavior



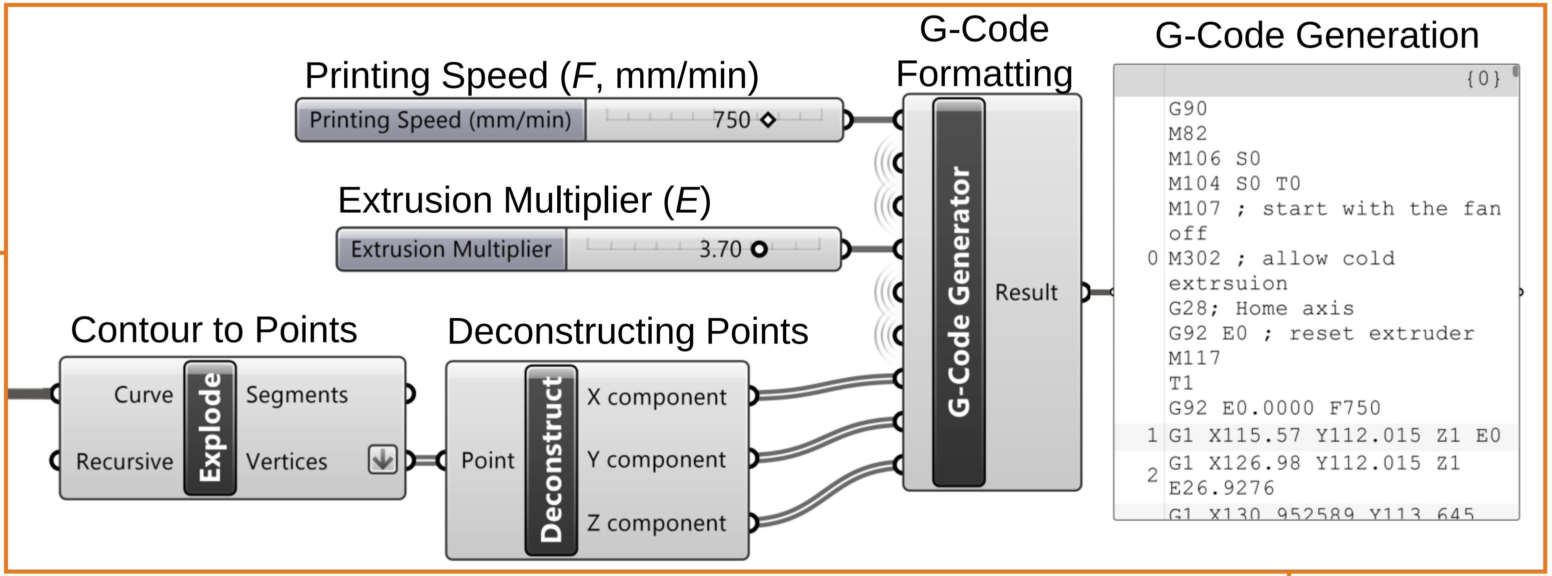
- Understand the role of interfacial heterogeneities in the **fracture** response and cracking mechanism
- Understand the **microstructural phases** at the core filament and interfacial zones using SEM and μ-CT
- Understand water **transport** in the presence of interfaces using neutron radiography (NR)

From Design of (any) Architecture to G-code using Grasshopper

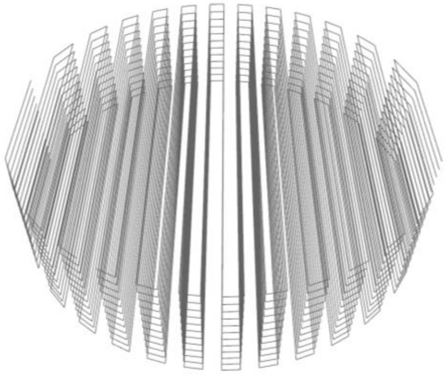
Defining Input Geometrical Parameters



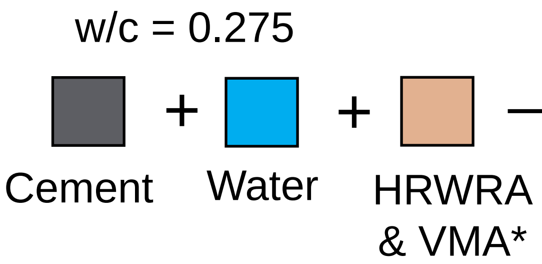
Generating G-Code from the Contour



```
  G90
  M82
  M106 S0
  M104 S0 T0
  M107 ; start with the fan
  off
0 M302 ; allow cold
  extrsuion
  G28; Home axis
  G92 E0 ; reset extruder
  M117
  T1
  G92 E0.0000 F750
1 G1 X115.57 Y112.015 Z1 E0
2 G1 X126.98 Y112.015 Z1
  E26.9276
  G1 X130.952589 Y113.645
```

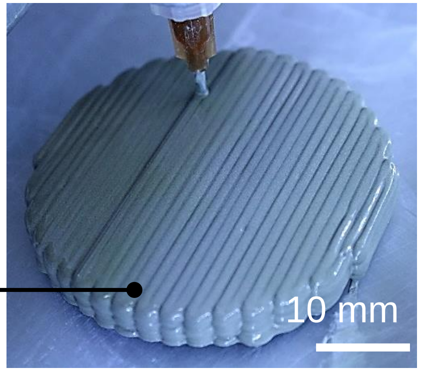


Parametric Contour



w/c = 0.275

Cement + Water + HRWRA & VMA*

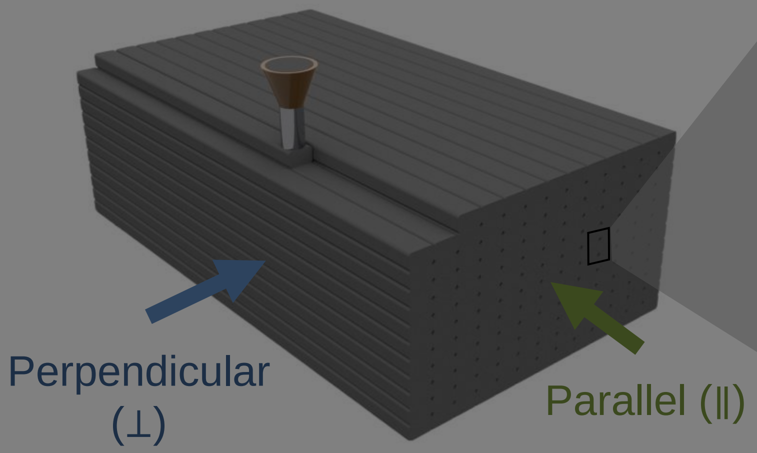


3D-printed sample with lamellar architecture

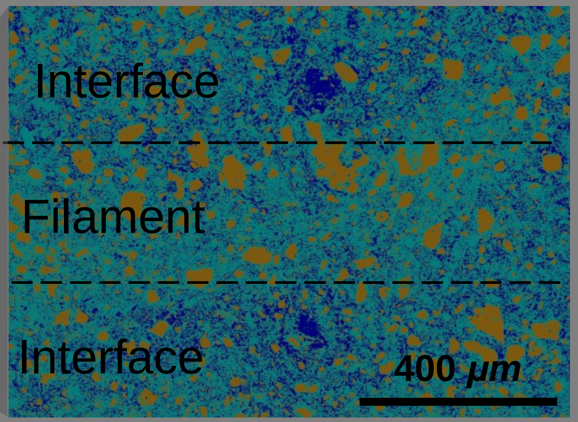
*HRWRA – High range water reducing admixture; VMA – Viscosity Modifying agent

Understand the Role of Interfacial Heterogeneities on Fracture Response

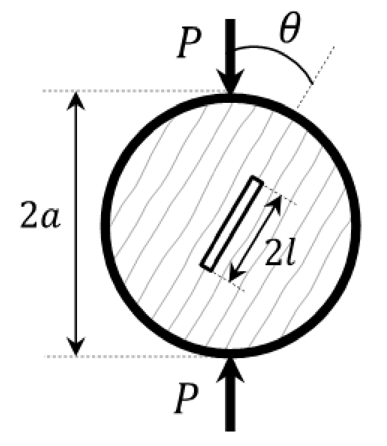
3D-printing of Cement Paste



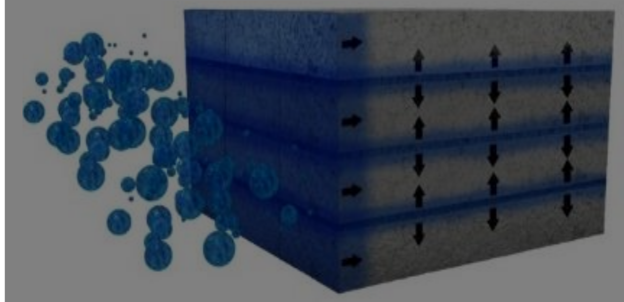
Interfacial Heterogeneities



Fracture Response



Transport Behavior



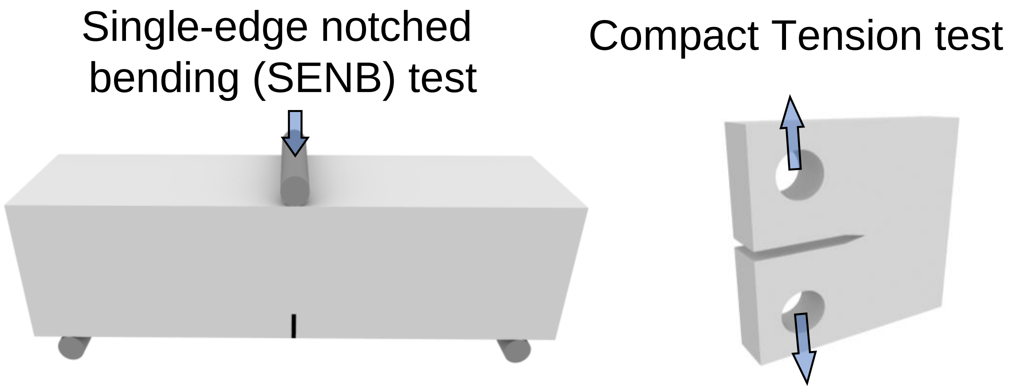
Fracture Characterization in Cast VS. Layered Cementitious Material

- **Cast** materials are assumed to have uniformly distributed microstructural heterogeneities
- **3D-Printed** materials, on the other hand, are microstructurally layered.

➢ In **Cast** fracture toughness tests:
  - Different setups are required
  - Notching is cumbersome and not sharp

➢ In **3D-printed** materials fracture is sensitive to:
  - The notch location due to interfaces
  - The interface orientation

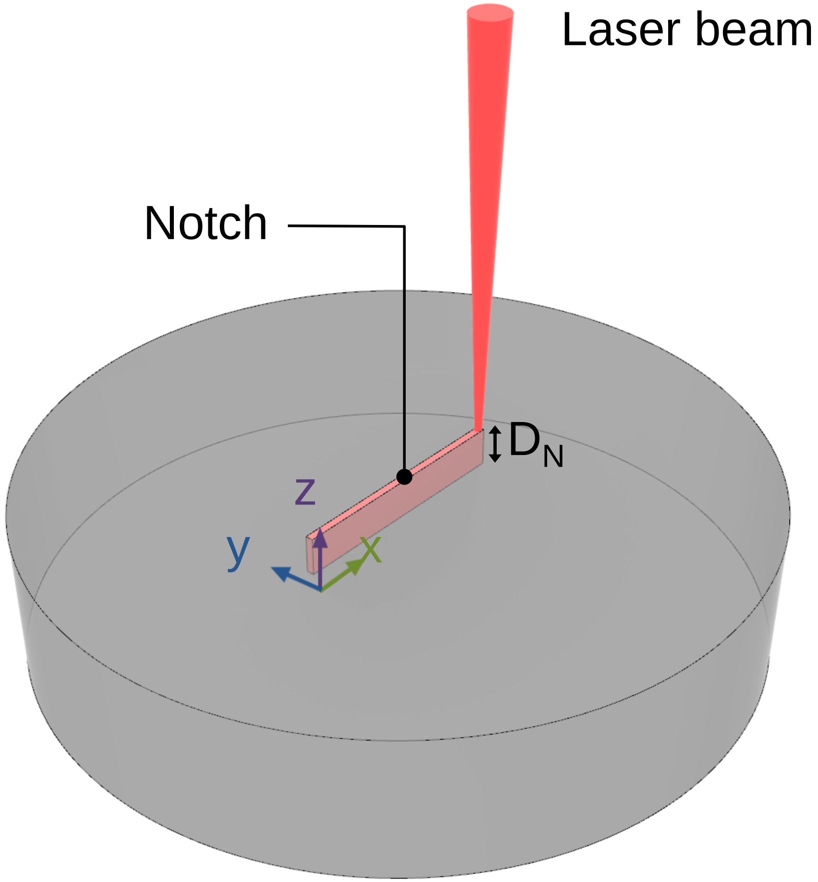
Mode-I (Tension) Fracture Toughness (K_{IC})



Q. How do you locate these notches in precise location?

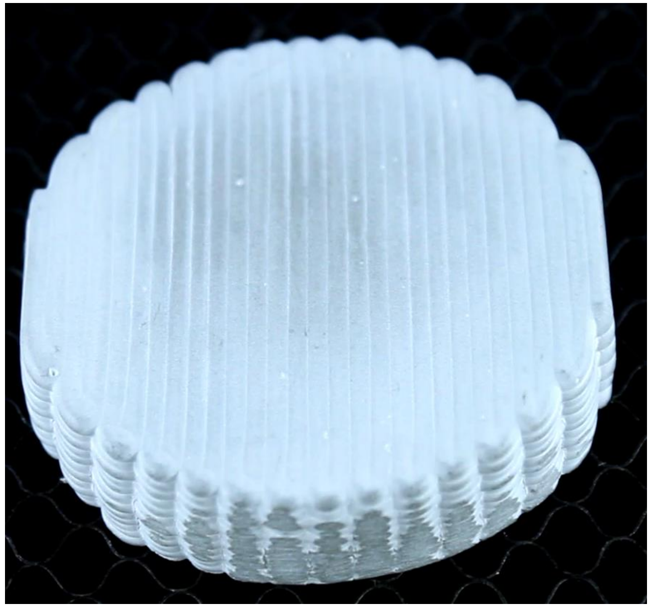
Q. How do you determine fracture sensitivity to interface orientation?

Laser-processing Is Used to Create Notches at Precise Locations in 3D-P Materials



D_N = Depth of notch

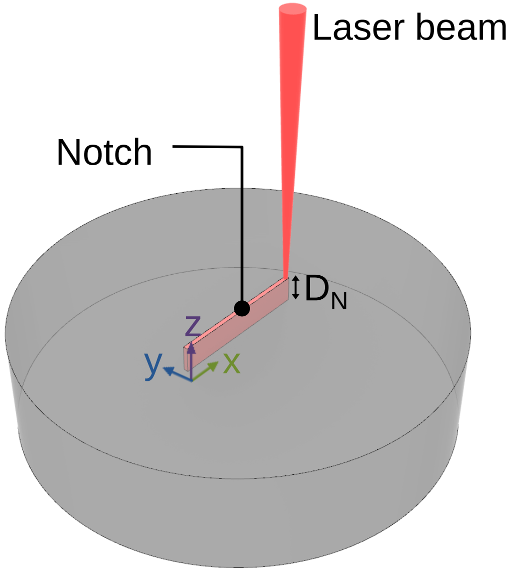
- **Precise** notch location relative to interfaces vs. filaments
- **Accurate** notch position compared to to saw-cutting
- **Sharp** notch tip shape compared to blade-saw tip
- **Tunable** laser processing parameters to engineer depth and sharpness



Laser notching of 3D-printed sample

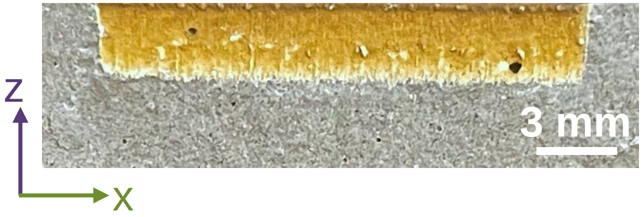
Effect of Laser Processing Parameters on the Notch Depth and Sharpness

Laser notching of Brazil-Nut sample

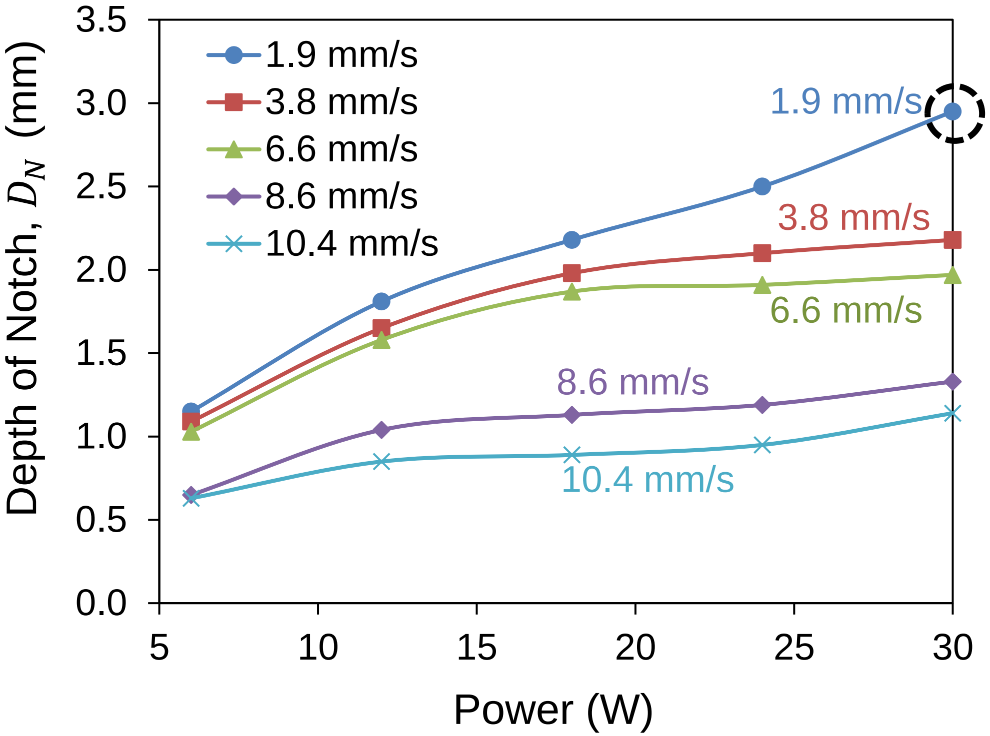


$D_N = Depth\ of\ Notch$

X-Z view of notch depth

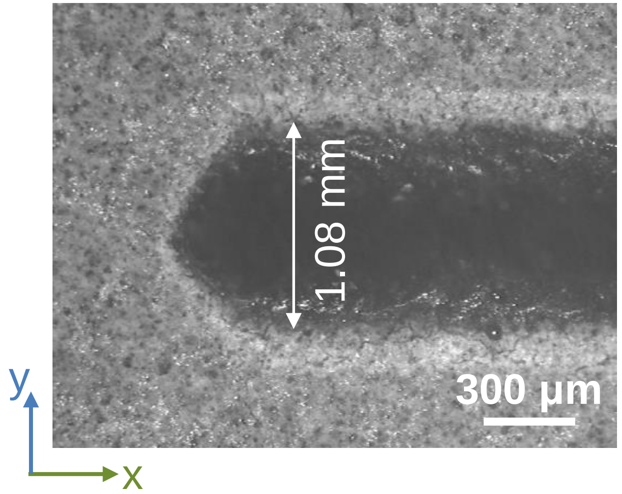


Depth of notch vs speed vs power of laser

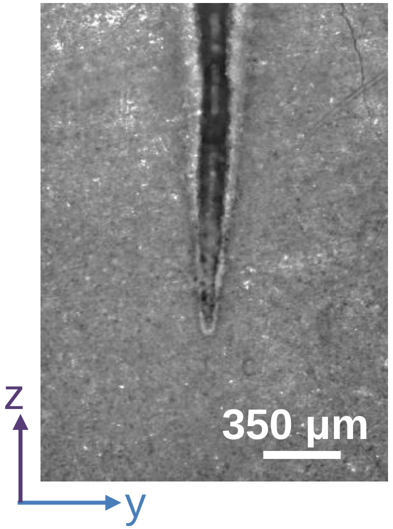


- Controlled the depth of notch as f(speed, Power)
- Sharp V-shaped notch was achieved

X-Y view of notch tip

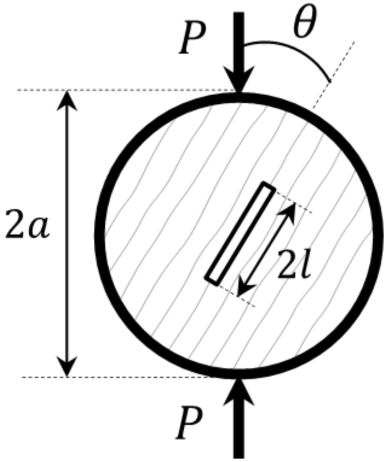


Y-Z view of notch depth

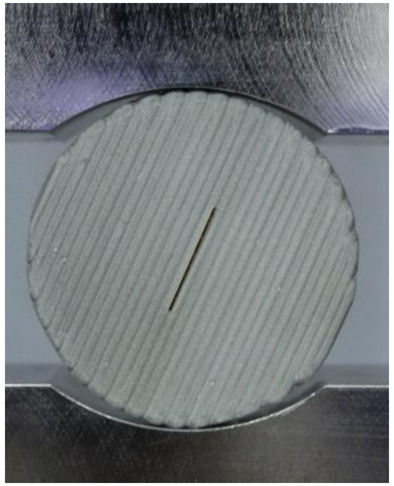


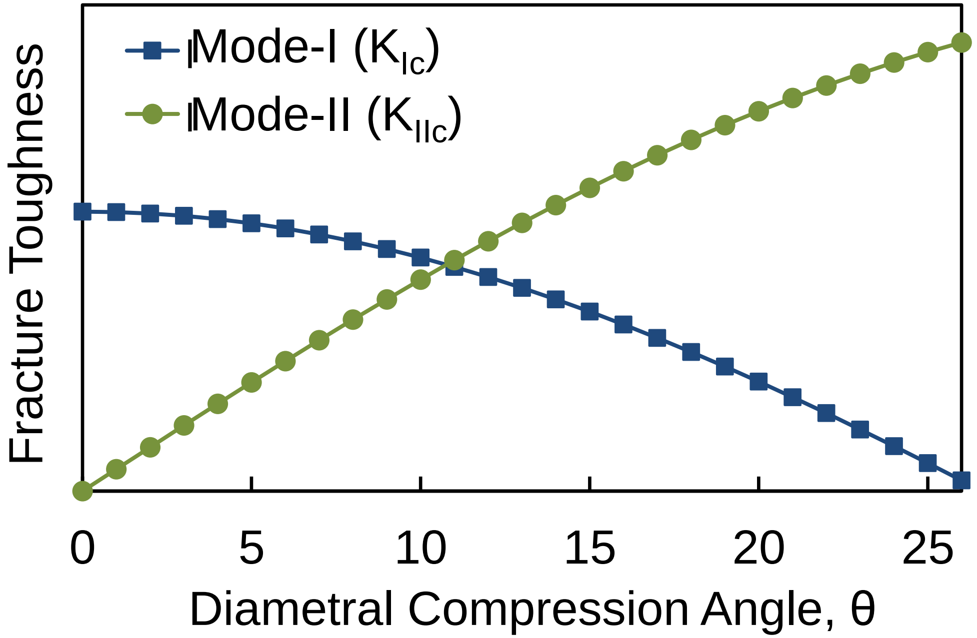
Brazil-Nut Test Is Used to Study Fracture Sensitivity to Interface

Schematic of Brazil-Nut test



Brazil-Nut Setup





Mode-I Fracture Toughness, $K_{I,c} = f_I\,(\theta, a, l)\,\frac{P}{\pi a D}\sqrt{\pi l}$

Mode-II Fracture Toughness, $K_{II,c} = f_{II}\,(\theta, a, l)\,\frac{P}{\pi a D}\sqrt{\pi l}$

where, f_I and f_{II} are normalized stress intensity factor

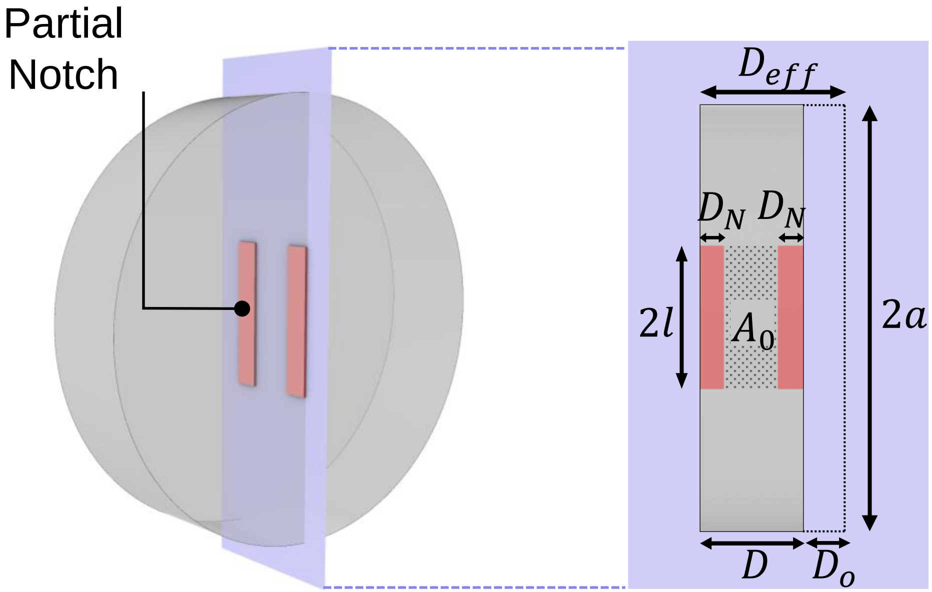
+ No need for different setups or sample geometry
+ Mode-I/II and mixed-Mode $[\theta]$
- Only partial notch at 30W laser power
- Modification of analytical equations for non-full-depth notch

Modification of the Analytical Calculations Is Proposed Due to Partial Notch

Consideration:

- Unnotched region required additional fracture energy
- Effective depth, D_{eff}, was introduced to account for additional fracture energy

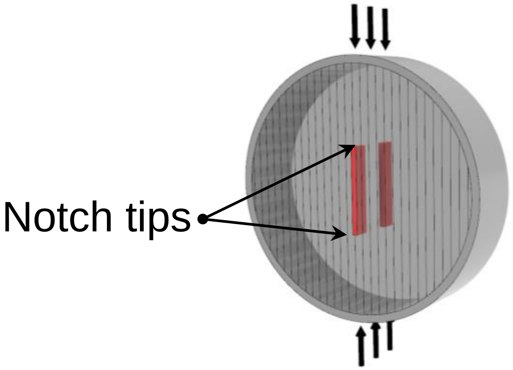
$$D_{eff} = D + A_0/2a$$



Verification:

- Fracture toughness of the **cast** agreed with both
  - Single edge notched bending result
  - Literature data of cast of same composition

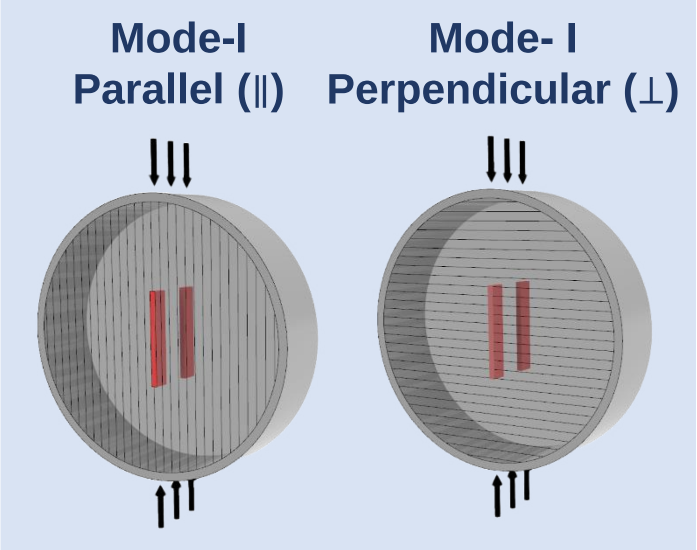
Consideration In 3D-Printed Lamellar Samples:

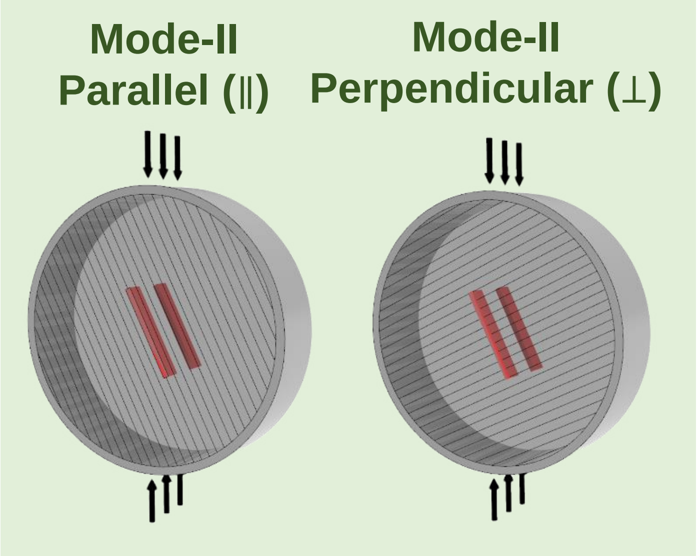


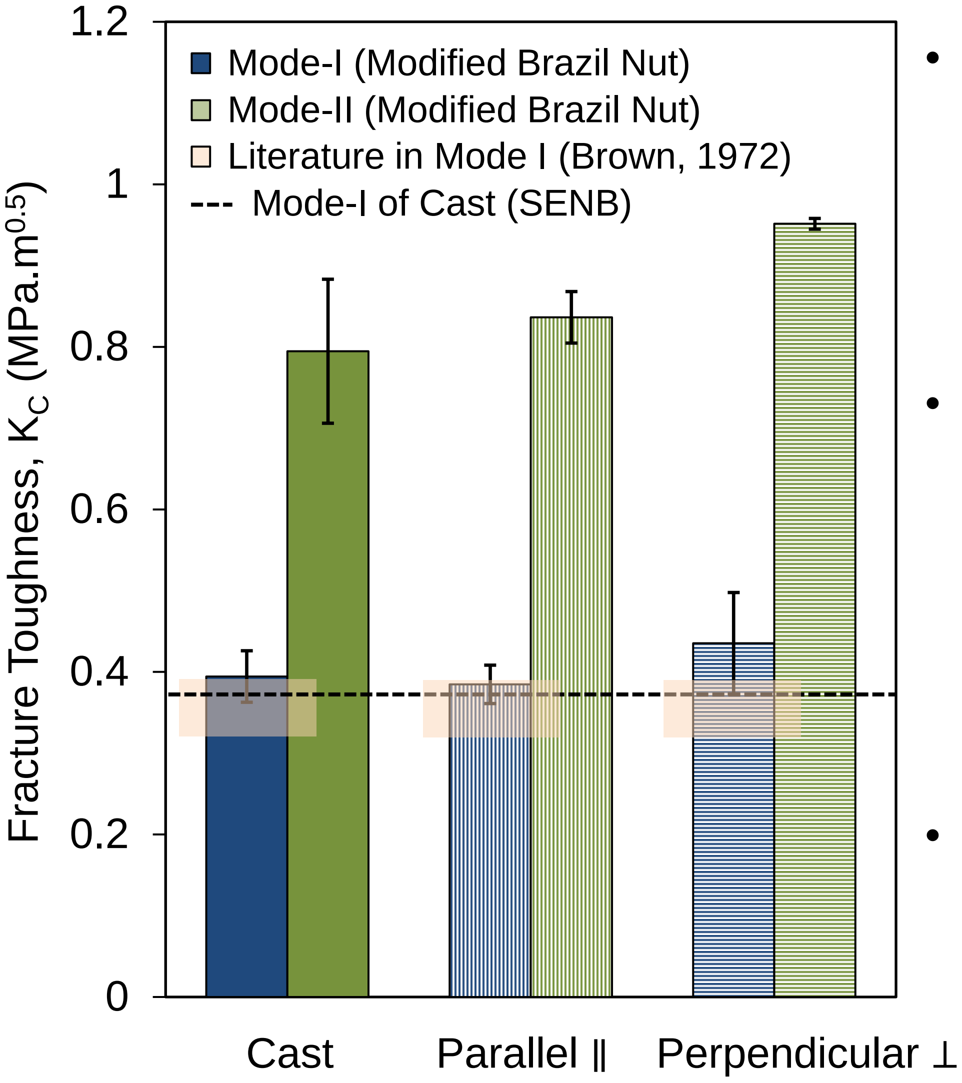
Notch tips were positioned at

- Interface in parallel direction
- Filament in perpendicular direction

Fracture Toughness of 3DP is Higher in Mode-II (Shear) in Perpendicular Direction







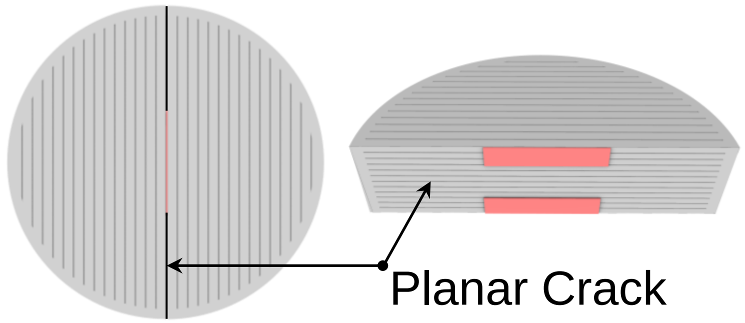
- Mode-I toughness was statistically similar in cast, parallel, & perpendicular

- Mode-II toughness in **perpendicular direction** was statistically higher than cast & parallel.

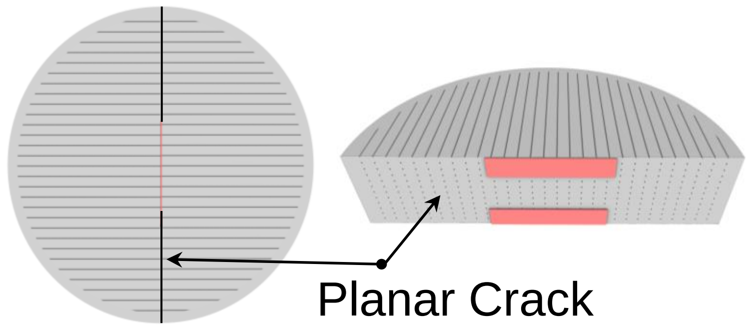
- **Q. Why?**

Crack Deflection acted as Toughening Mechanism in Shear in 3DP Materials

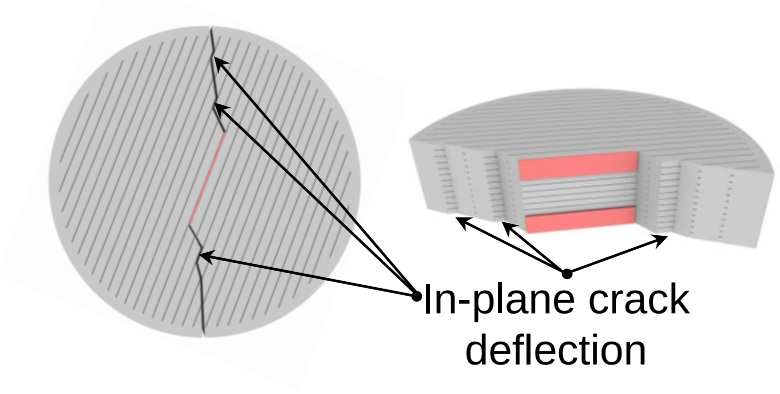
Mode-I. Parallel (∥)



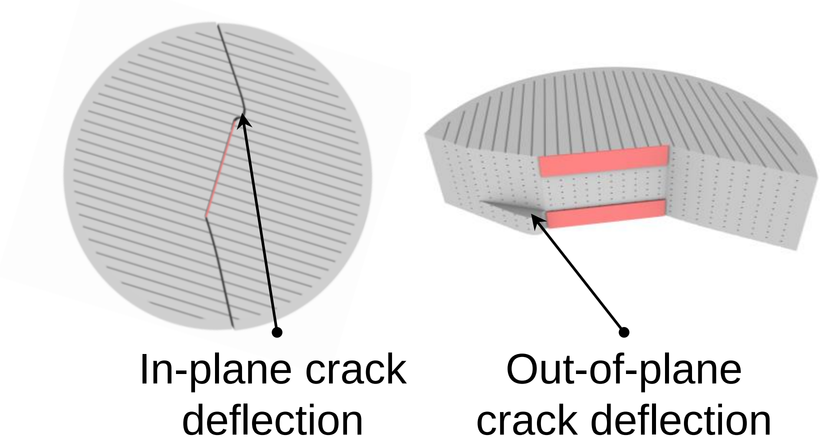
Mode-I. Perpendicular (⊥)



Mode-II. Parallel (∥)



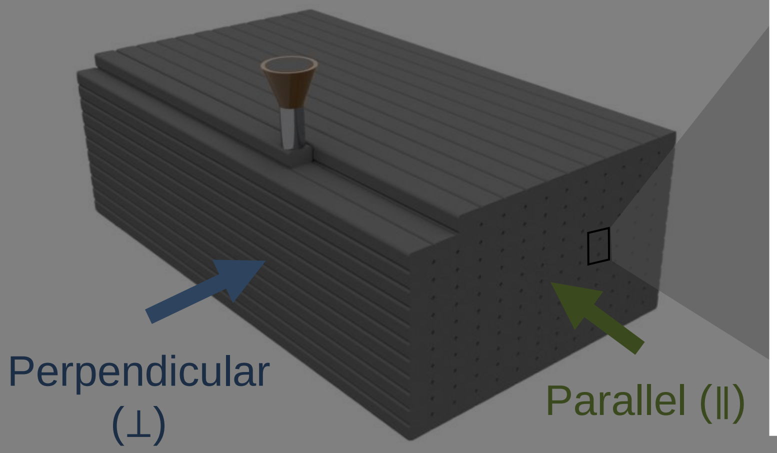
Mode-II. Perpendicular (⊥)



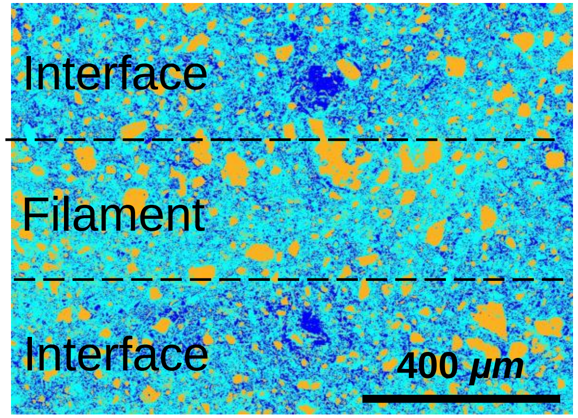
- In Mode-I, ***planar crack propagation*** was observed in both directions
- In Mode-II, ***in-plane and out-of-plane crack deflection*** was observed
- Hypothesis.
  - ➢ Presence of the ***weak/porous interfacial zone*** leads to mixed-mode (I-II) cracking

Understand the Microstructural Phases at the Core Filament and Interfacial Zone

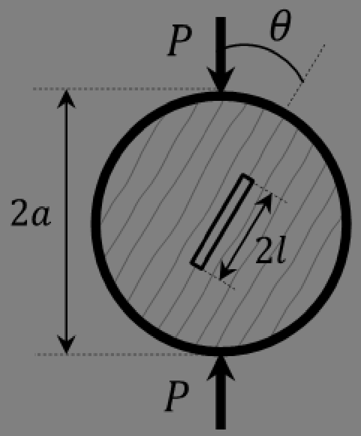
3D-printing of Cement Paste



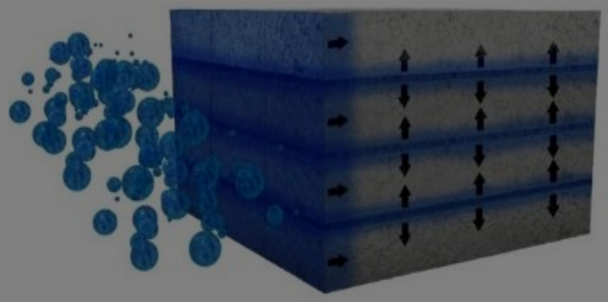
Interfacial Heterogeneities



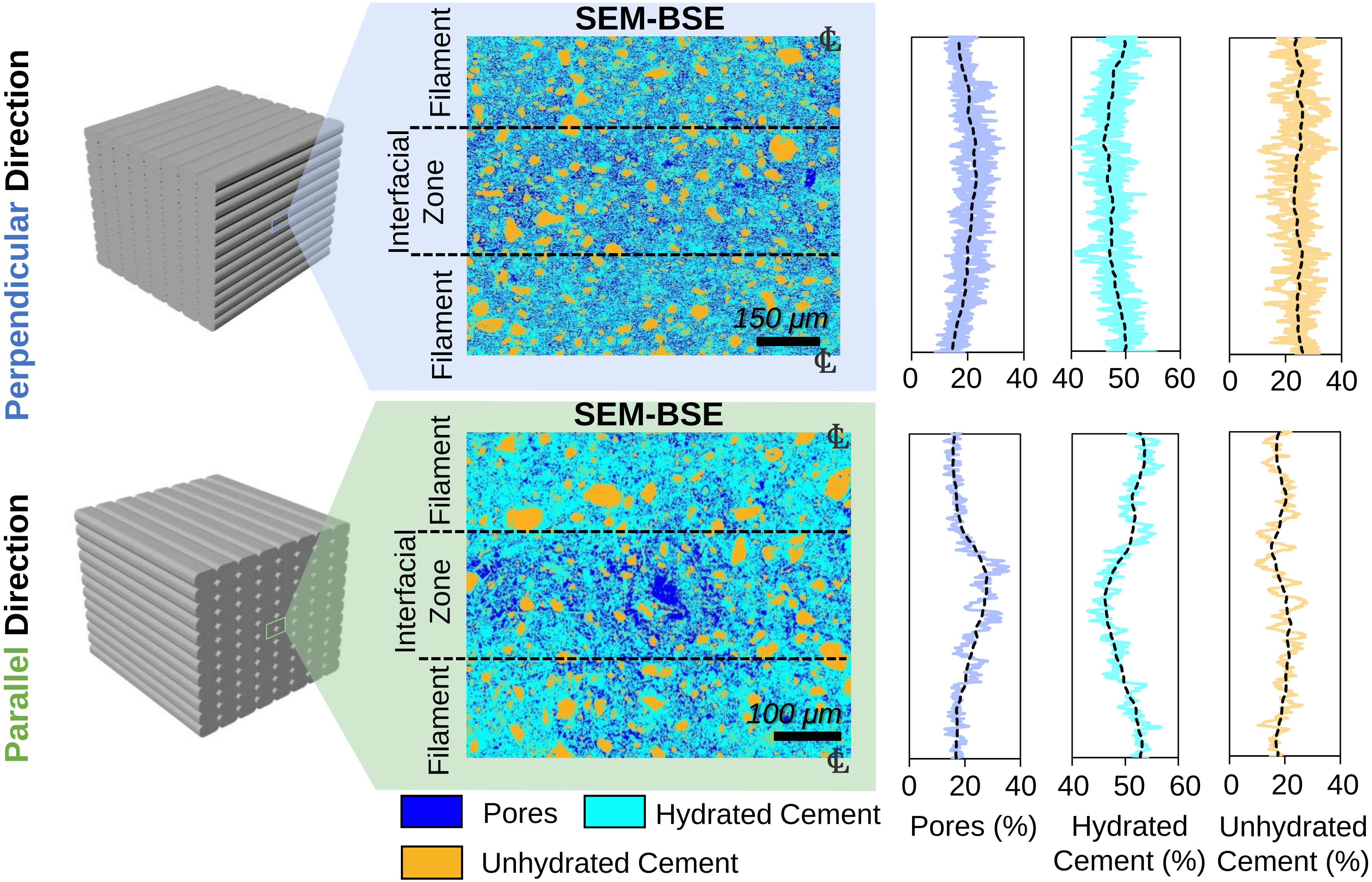
Fracture Response



Transport Behavior

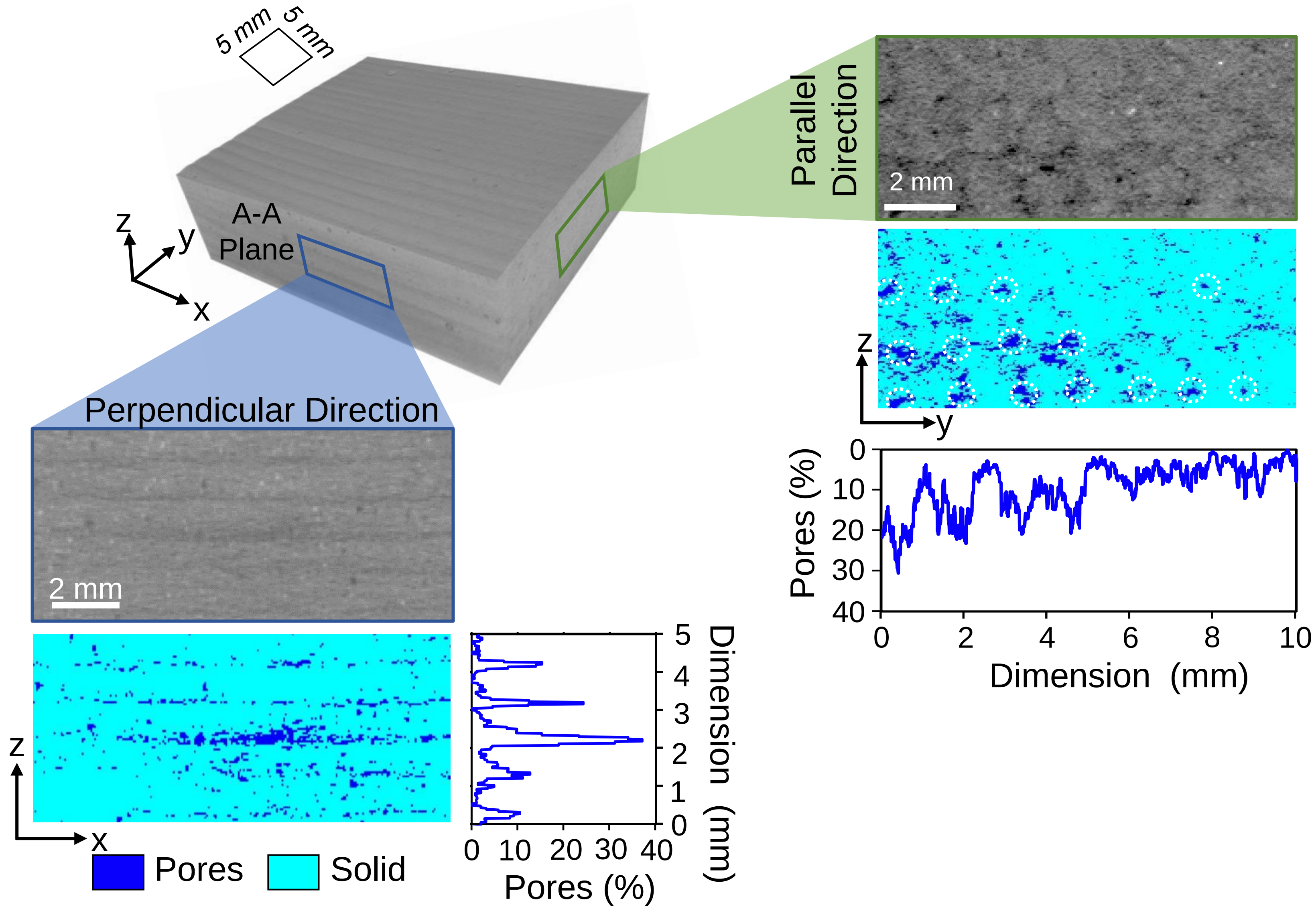


Interfacial Zone Is Heterogeneous (By Porosity & Hydrated Products Measure)

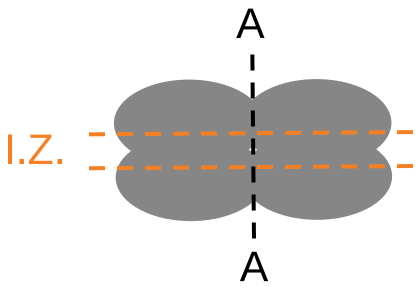


- Porosity was 7.2% higher in interfacial zones than in the filament
- Hydrated cement was 8.5% lower in interfacial zone than in filament
- Unhydrated Cement remained constant
- Microchannels constituted 2.5% of total porosity

Interfacial Zone Contains Non-uniform Heterogeneities



- Porosity was ~370% times higher in interfacial zone than in filament
- Porosity was not uniformly distributed!

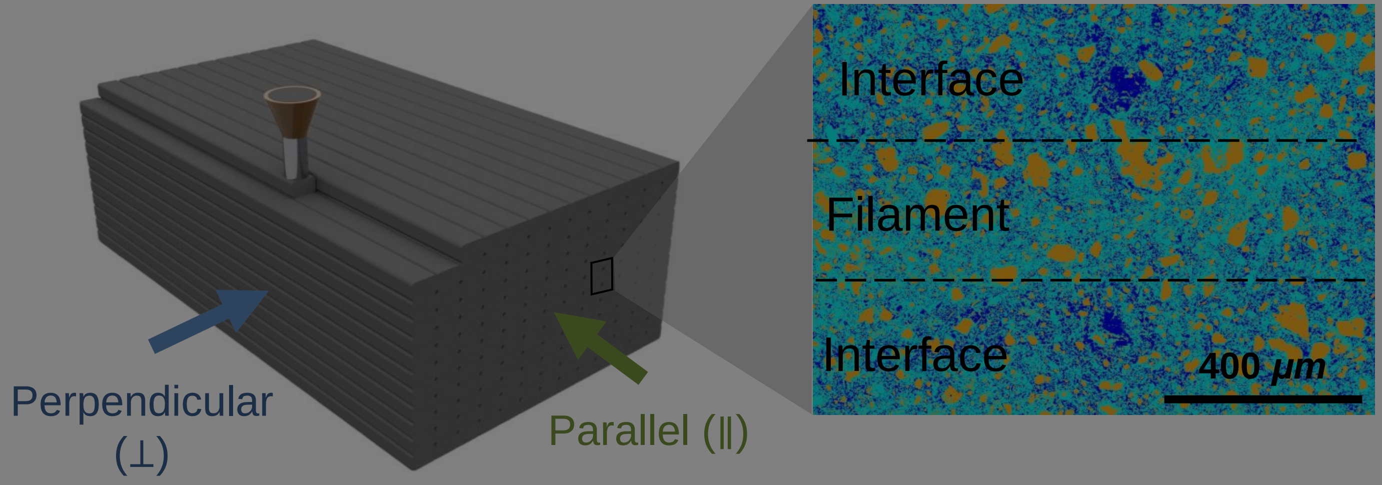


I.Z. = Interfacial Zone

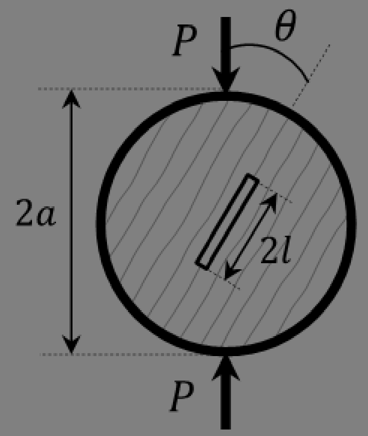
Understand Water Transport at the Presence of Interfaces

3D-printing of Cement Paste

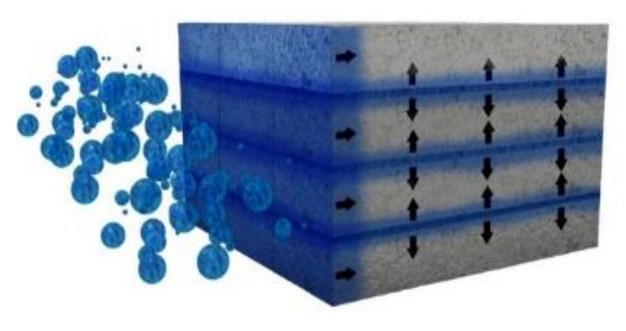
Interfacial Heterogeneities



Fracture Response

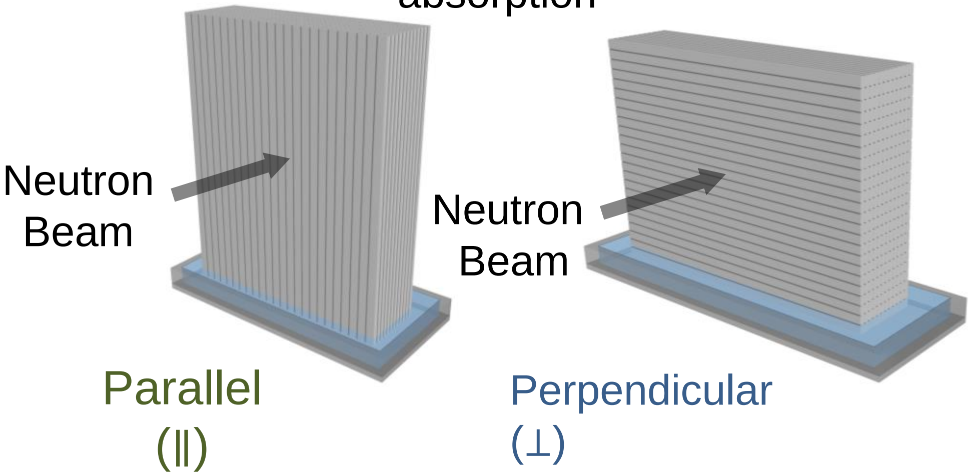


Transport Behavior

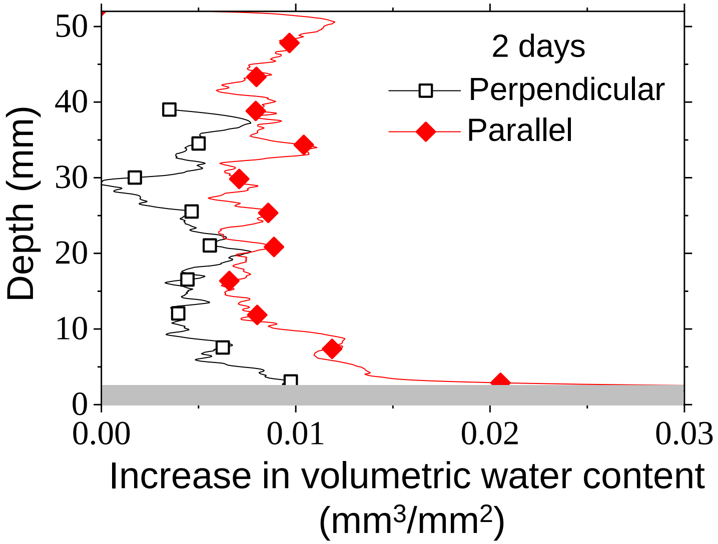


Transport Is Anisotropic Due to Presence of Heterogeneities

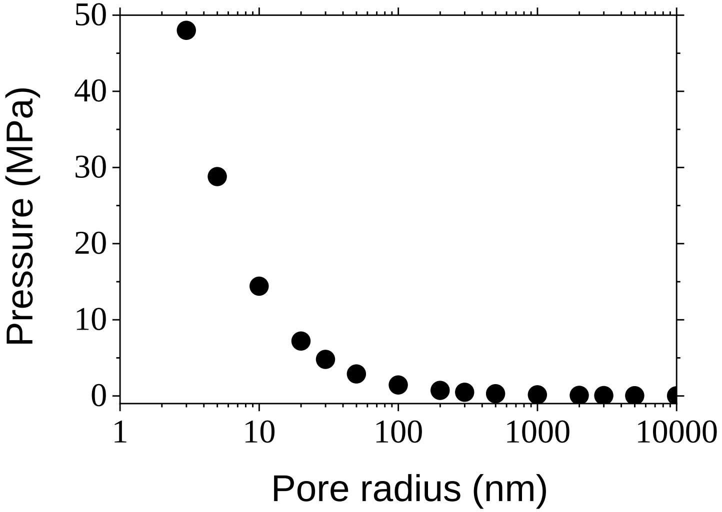
Schematic of Neutron Radiography for water absorption



Water Absorption at t = 2 days



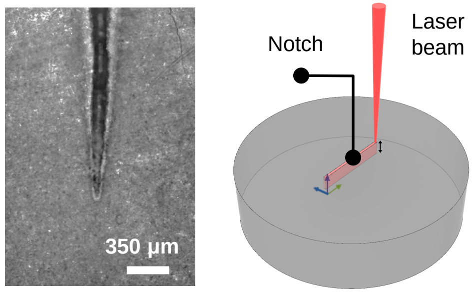
Capillary Pressure vs. Pore Radius



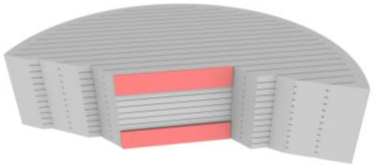
- Water absorption was higher in parallel direction
- Hypothesis.
  - In perpendicular direction, interfacial zone contains larger pores which serve as a large capillary break
  - In parallel direction, higher capillary uptake takes place in the filament compared to interfacial zone

Conclusions

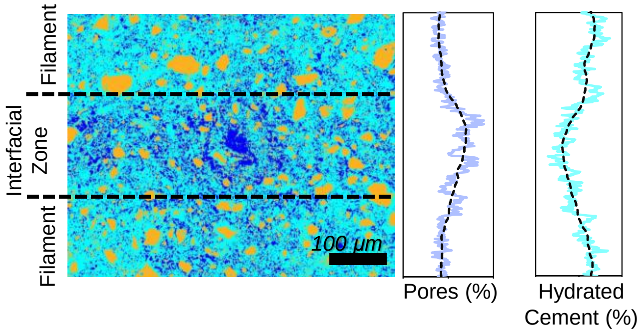
- A novel approach to use **laser processing** for notching cementitious materials



- Interface can be harnessed to **shear resistance to fracture** in 3D-printed Materials by giving rise to crack deflection toughening mechanism



- Pore size and **heterogeneities** in the interfaces and filaments can control the directionally of water transport



Acknowledgements

- NSF-ECI program for the generous support
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- Princeton School of Engineering and Applied Science (SEAS) Machine-shop for use of Laser Cutter
- Oregon State TRIGA® Reactor (OSTR)

NSF

CMMI 2129566, 2129606

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School of Engineering and Applied Science

Oregon State
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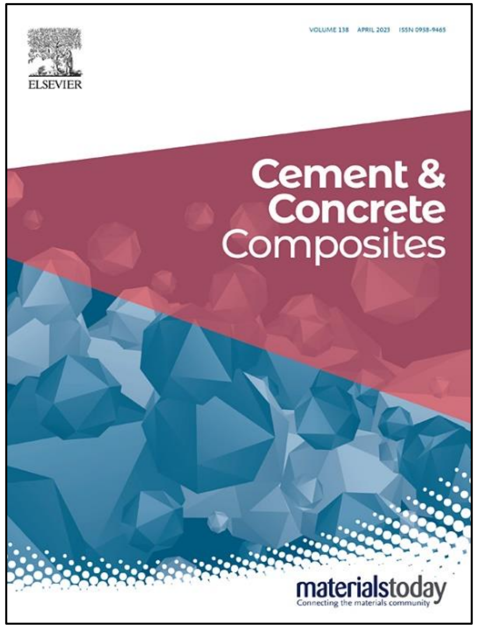
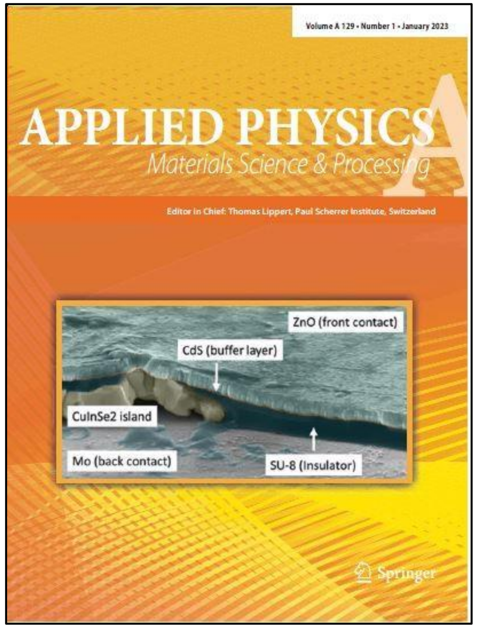
Thank you

Questions?

For more detail:





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Marco Rupp, *et al.* 2023 *Appl. Phys. A.*

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