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Understanding print stability in material extrusion additive manufacturing of thermoset composites

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Understanding Print Stability in Material Extrusion Additive Manufacturing of Thermoset Composites

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Introduction – Direct ink writing

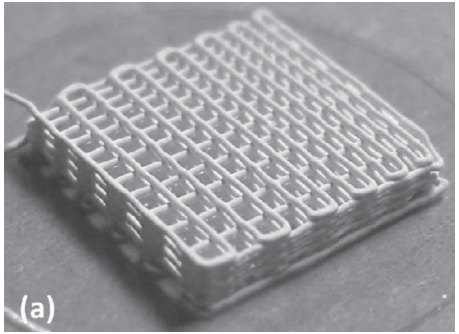
Direct ink writing (DIW)

- Subset of *material extrusion* additive manufacturing (AM)

Small-scale developments

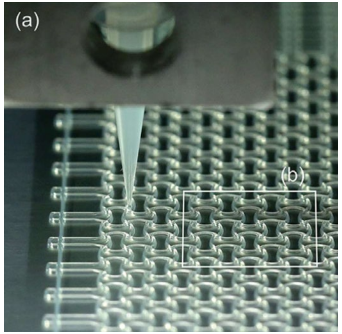
- Bio-materials
- Elastomers
- Thermosets

Bio-materials



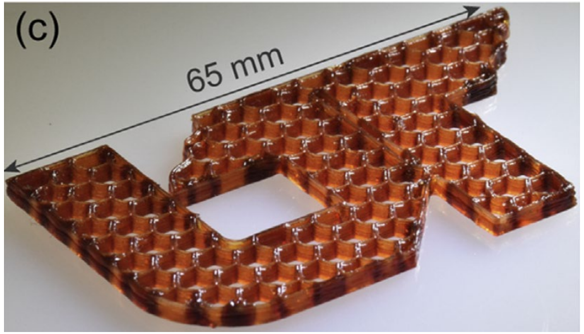
Sun et al., *Advanced Materials*, 2012

Elastomers



Clausen et al., *Advanced Materials*, 2015

Thermoset composites



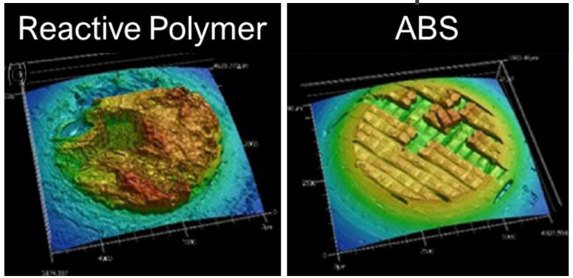
Hmeidat et al., *Compos Sci Technol*, 2018

Introduction – Large-scale thermoset DIW

Large-scale thermoset DIW

- Desirable characteristics
  - Extrusion does not require heat
  - Attractive mechanical properties
  - High thermal resistance
- Machine at Oak Ridge National Laboratory (ORNL)
- Limited by structural **stability** during printing

Fracture surface comparison

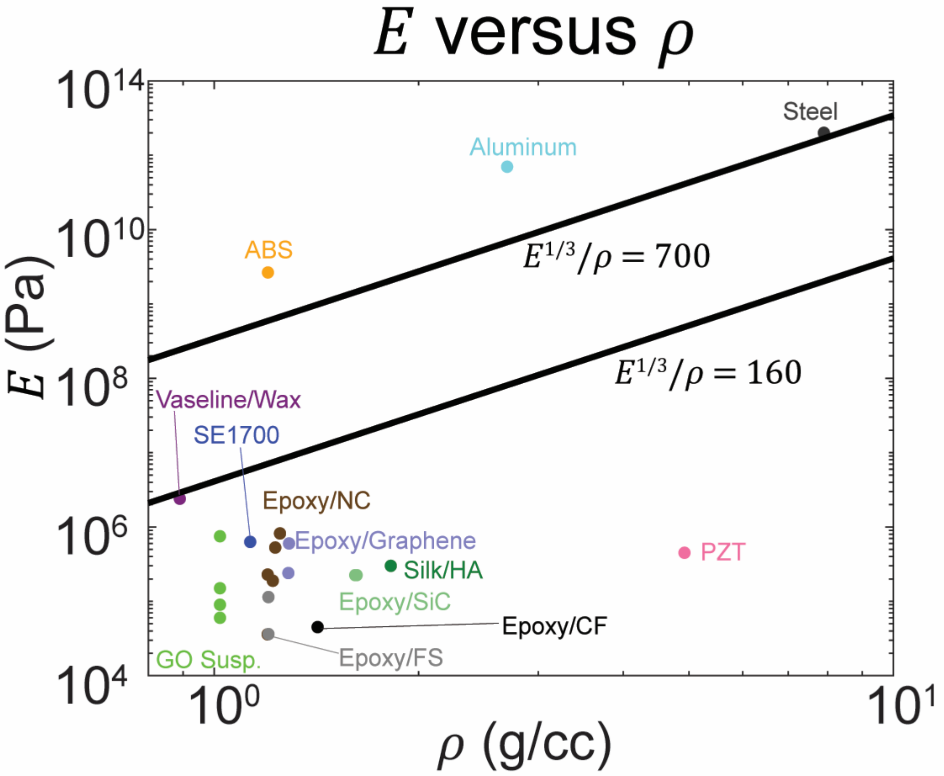


Rios et al., *Mater. Today Commun.*, 2018

Background – Viscoelastic properties

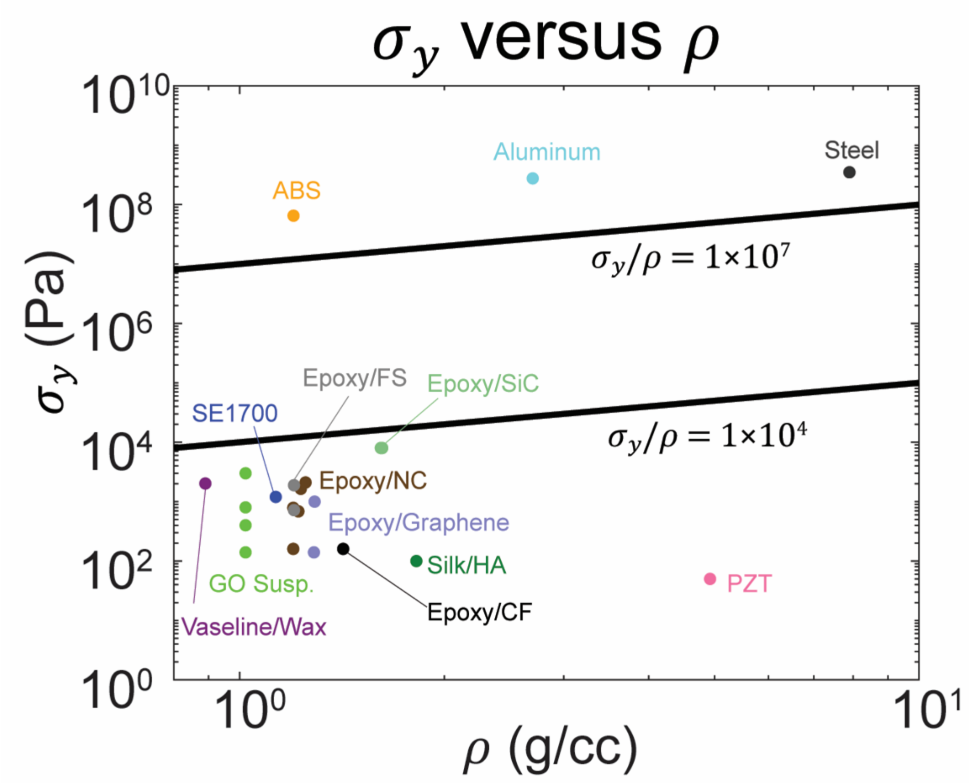
Before cure, thermoset DIW inks

- Low specific stiffness



Background – Viscoelastic properties

Before cure, thermoset DIW inks
- Low specific stiffness
- Low specific strength



Background – Challenges

Collapse on small scale

Falling of thin walls

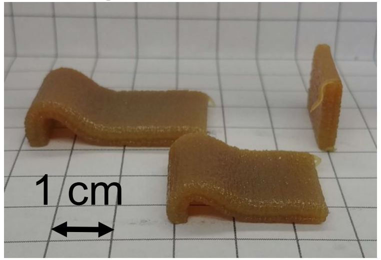
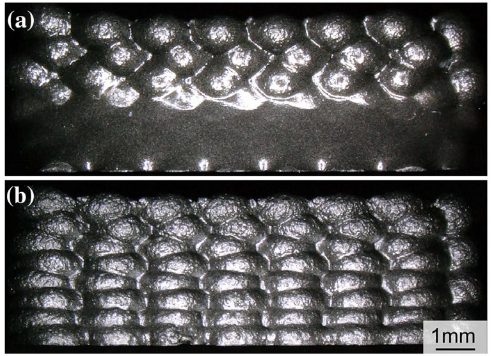


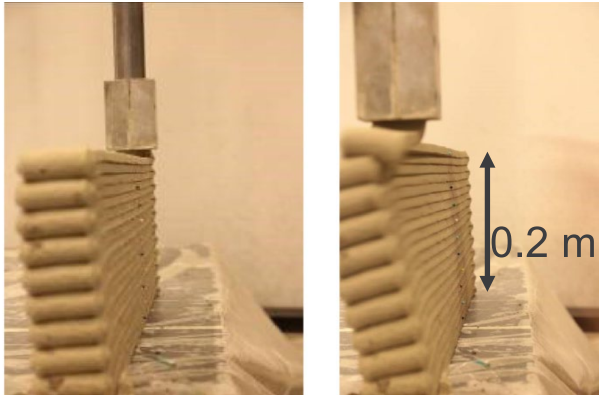
Image courtesy of Madeline Wimmer

Slumping of lower layers

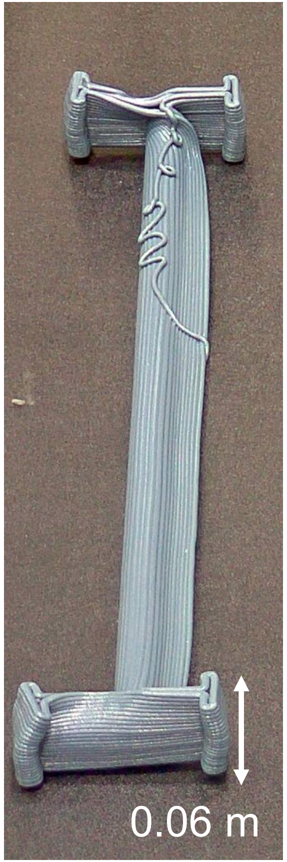


Compton et al., *JOM,* 2017

Collapse on large scale



Suiker, *Int. J. Mech. Sci.*, 2018

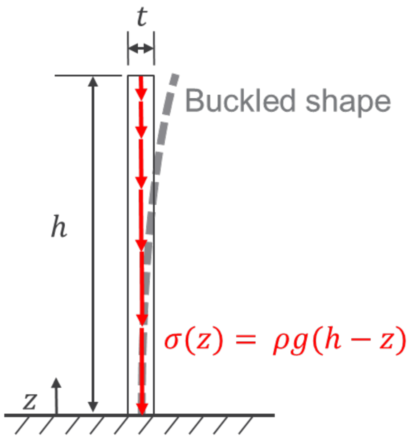


Romberg, *SAMPE J*, 2019

Background – Stability models

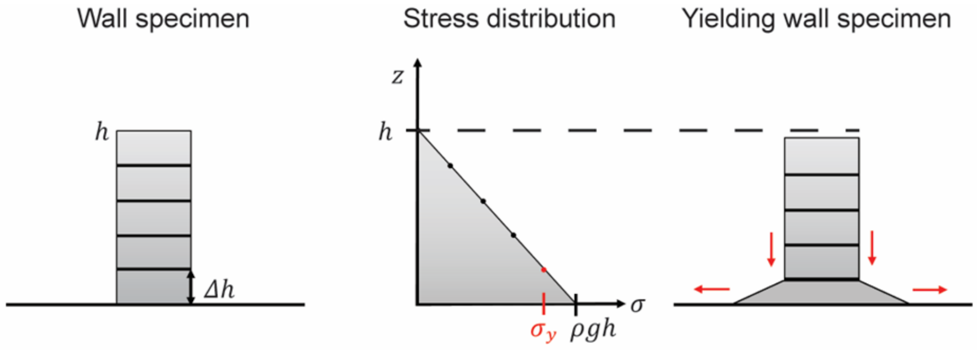
Buckling under self-weight

- Greenhill (1881)*
  - Inspired by the height capacity of trees
  - $h_{column,b} = \left(7.8373 \frac{EI}{\rho g A}\right)^{1/3}$



Yielding under self-weight

- $h_y = \frac{\sigma_y}{\rho g}$
- Suiker (2018) – concrete print stability
  - Stiffening
  - Strengthening

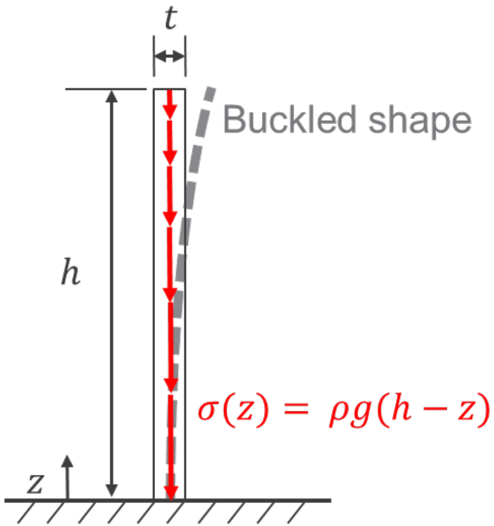


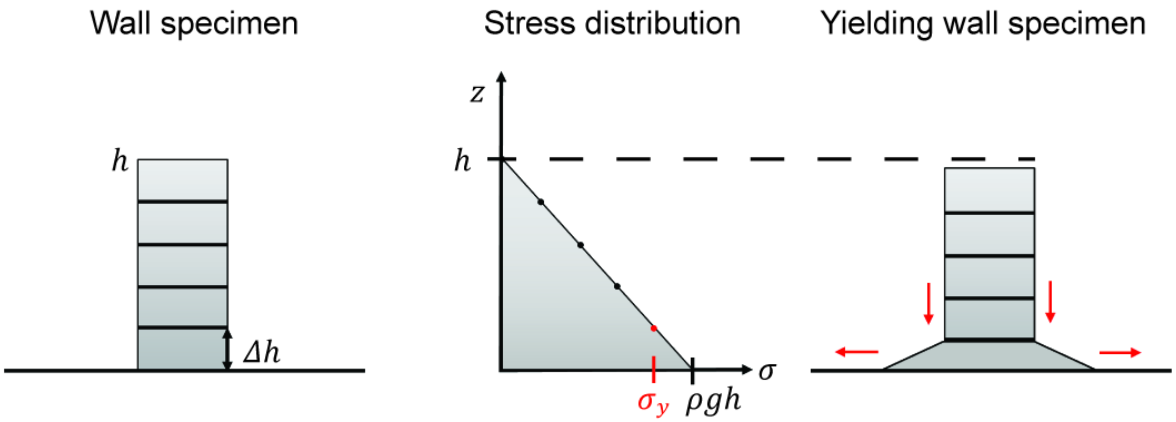
*Greenhill predicted a maximum height of 300 feet for a pine tree with a 20-inch diameter

Hypothesis – Stability of DIW inks

Collapse of thermoset DIW walls is caused by self-weight and depends on 3 ink properties

- Density (ρ)
- Shear plateau storage modulus (E)
- Shear yield stress (σ_y)



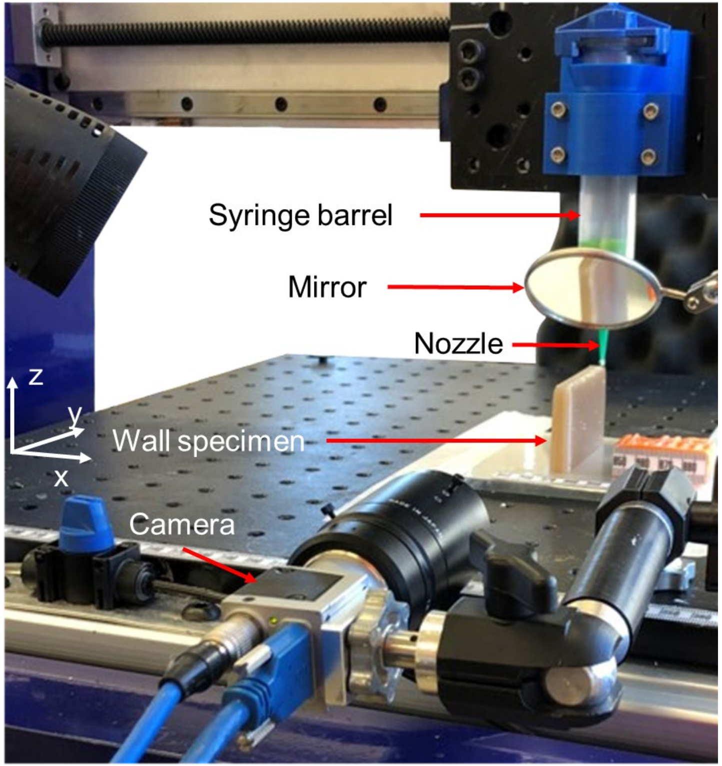


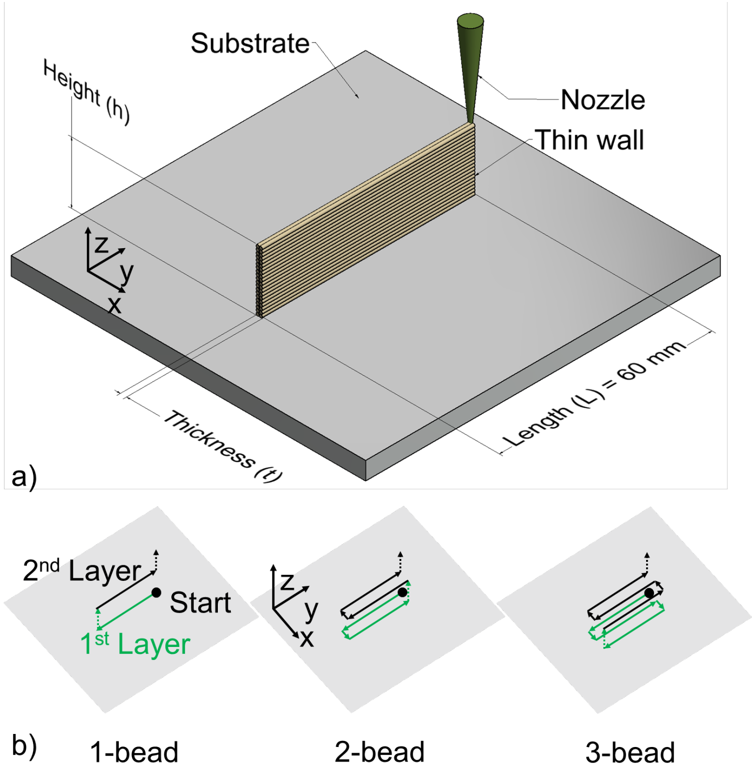
Stability tests – Setup

Nozzle diameters
- 0.404 mm
- 0.872 mm

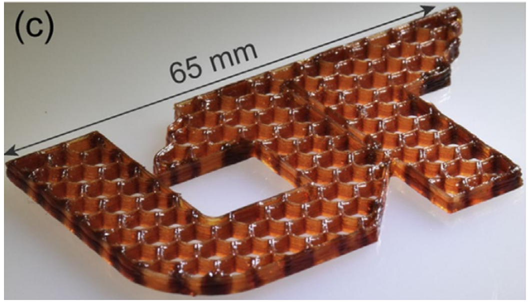
Number of beads
- 1
- 2
- 3
- 6
- Enough to yield

- **1 image/layer**



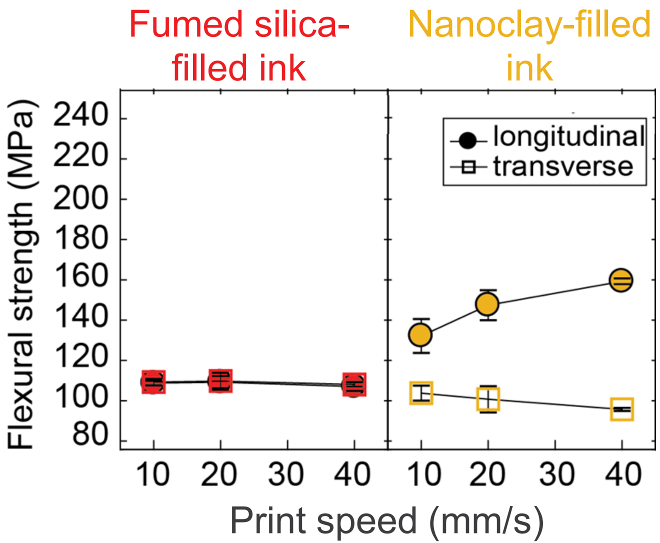


Approach – Material

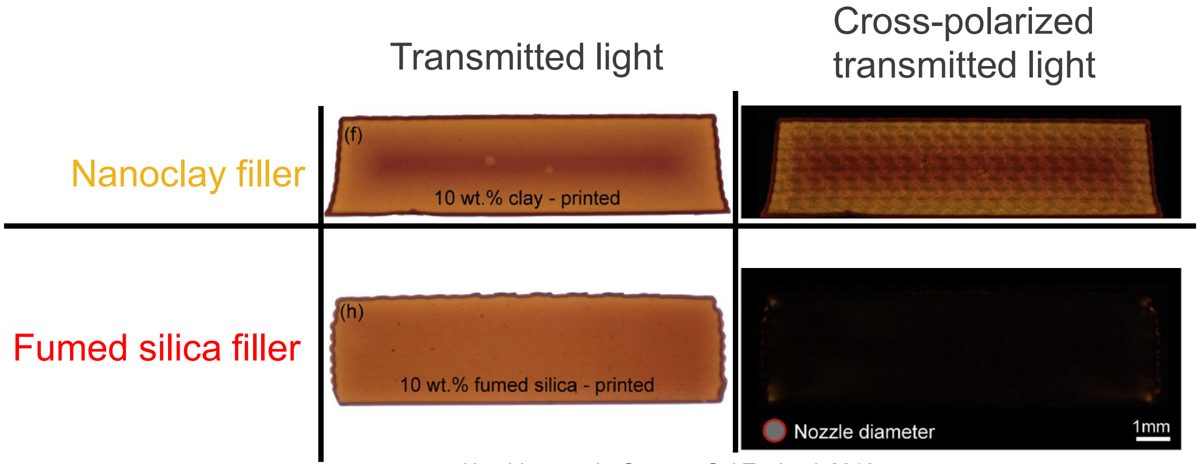


Two epoxy-based systems

- Printed and cured properties can be filler dependent
- Nanoclay is more anisotropic than fumed silica
- Cross-polarized images highlight differences

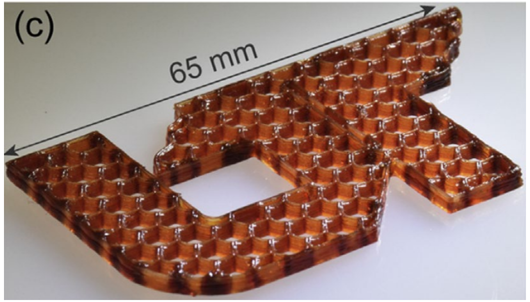


Hmeidat, et. al., *Addit. Manuf.*, in review 2020



Hmeidat, et. al., *Compos Sci Technol*, 2018

Approach – Material



Two epoxy-based systems

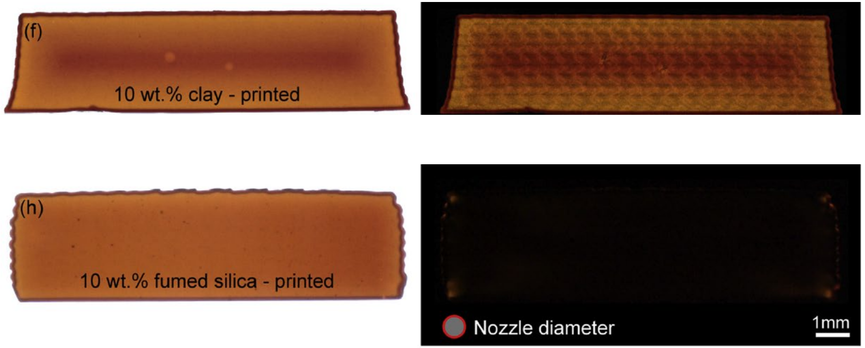
- Printed and cured properties can be filler dependent
- Nanoclay is more anisotropic than fumed silica
- Cross-polarized images highlight differences

- 10 wt% nanoclay (NC)
  - Garamite 7305
- 10 wt% fumed silica (FS)
  - Cab-o-sil TS-720

Without curing agent

- Minimize time-dependence

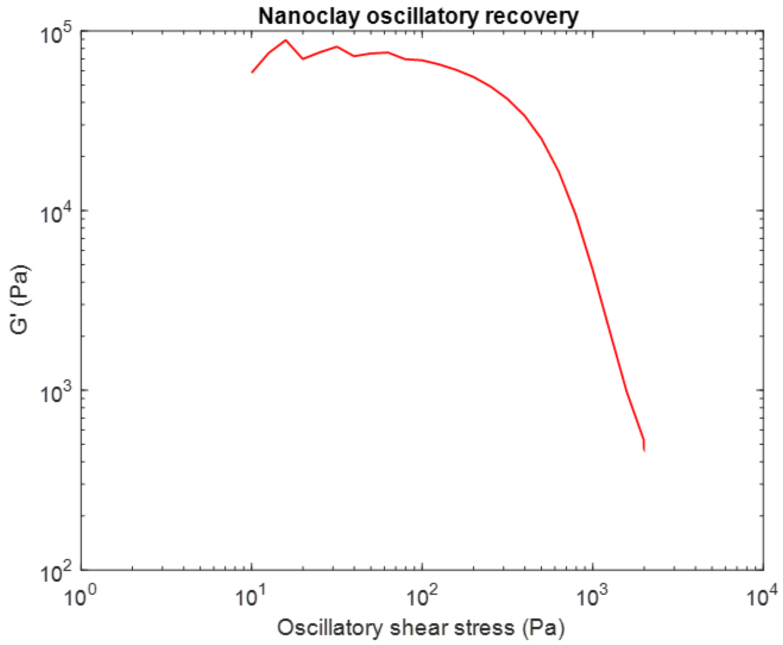
Transmitted light | Cross-polarized transmitted light



Hmeidat, et. al., *Compos Sci Technol*, 2018

Approach – Research plan

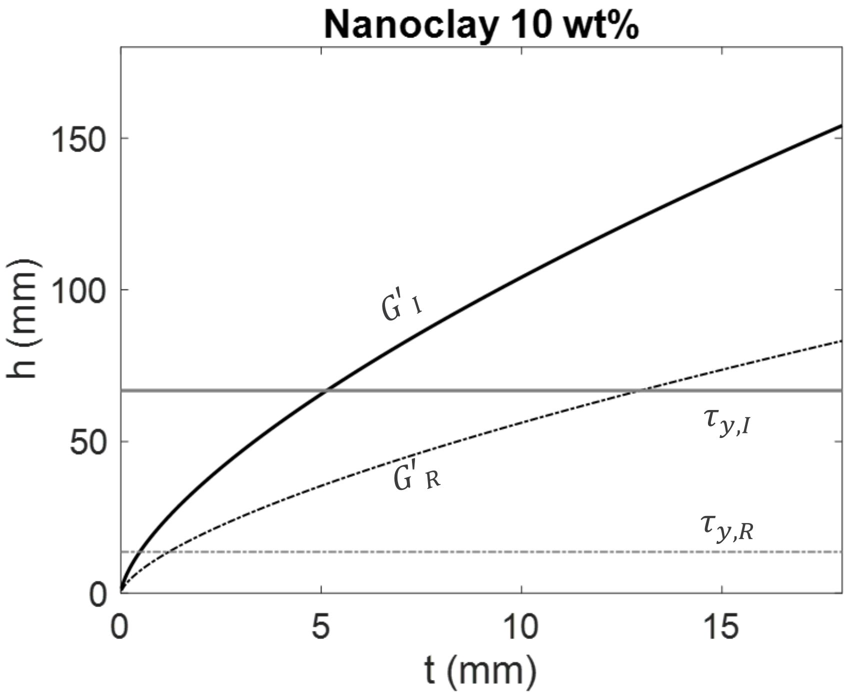
1. **Rheology**
   - G'_P and τ_y
   - Initial and recovered properties



Approach – Research plan

1. **Rheology**
   - G'_P and τ_y
   - Initial and recovered properties

2. **Model wall height predictions**
   - G'_P → Self-buckling
   - τ_y → Self-yielding



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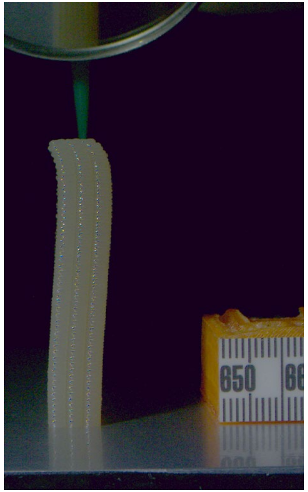
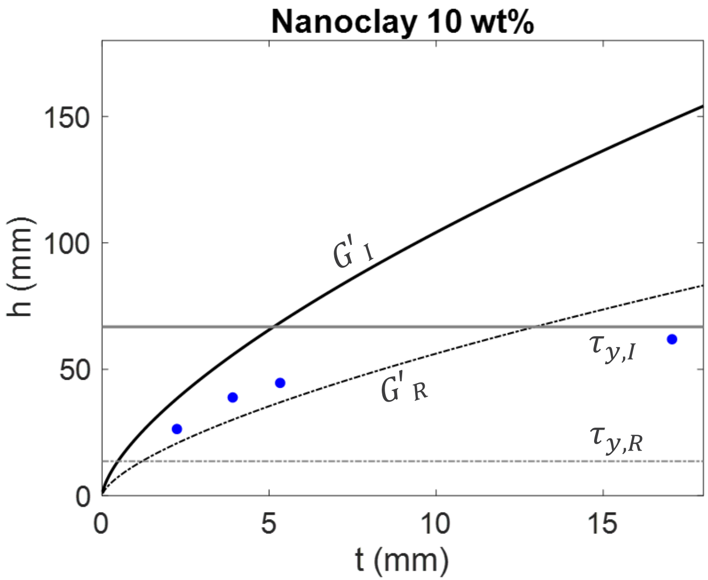
Approach – Research plan

1. **Rheology**
   - G'_P and τ_y
   - Initial and recovered properties

2. **Model wall height predictions**
   - G'_P → Self-buckling
   - τ_y → Self-yielding

3. **Print walls to assess model**





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Rheological tests

Extrusion may affect G'_P and τ_y

Oscillatory recovery studies

1. Began in linear viscoelastic region (LVR)
2. Ramped to a maximum stress
3. Held at maximum stress
4. Retraced stress back to LVR

Maximum stress

- Just past yield (lowest)
- Intermediate
- Before material loss (highest)

Time hold at each stress

- No hold
- 5-min hold
- 60-min hold

40-mm upper platen

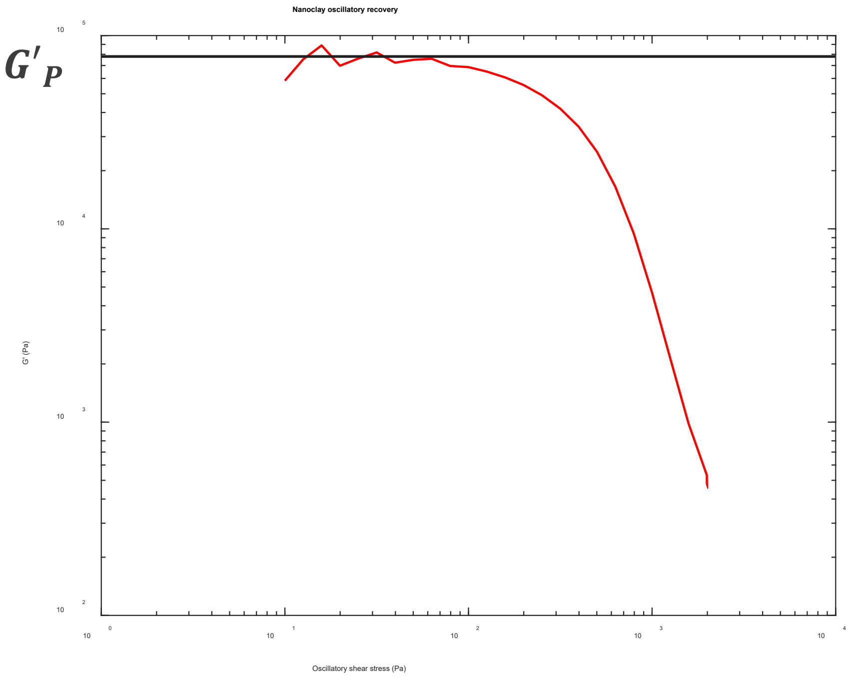
Peltier plate (22°C)

	Nanoclay ink	Fumed silica ink
Lowest max stress (Pa)	700	1200
Intermediate max stress (Pa)	1200	2000
Highest max stress (Pa)	2000	3000

Rheological definitions

G'_P

- Averaged G' in the LVR



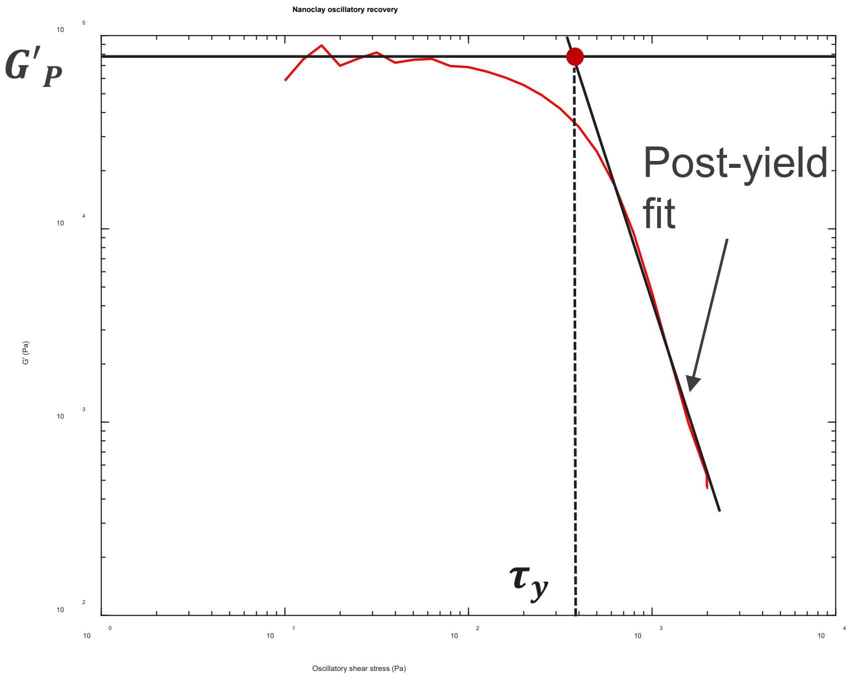
Rheological definitions

G'_P

- Averaged G' in the LVR

τ_y

- G'_P and post-yield fit intersection



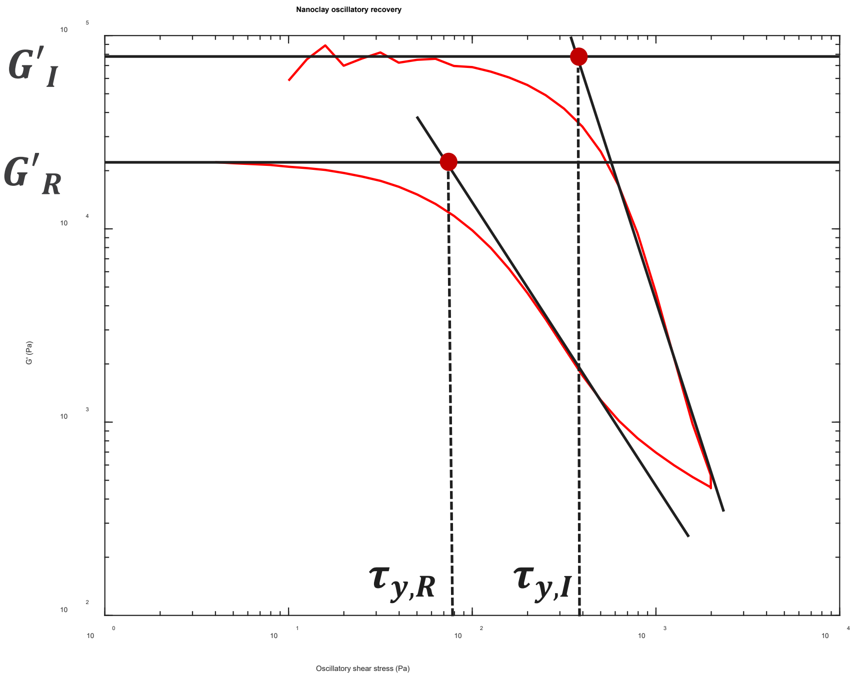
Rheological definitions

G'_P
- Averaged G' in the LVR

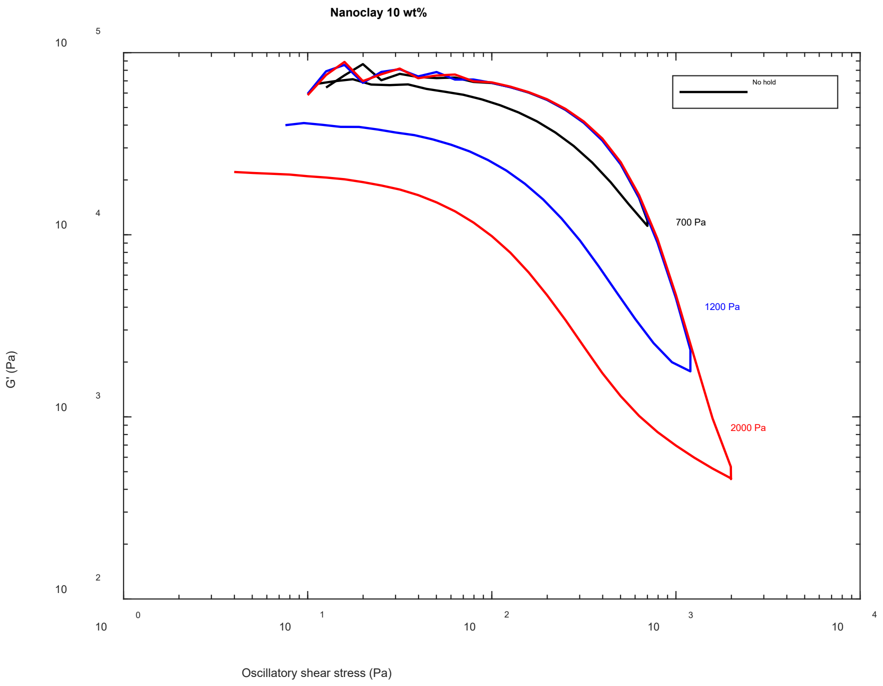
τ_y
- G'_P and post-yield fit intersection

G'_I **and** $\tau_{y,I}$
- Determined on the "initial curve"

G'_R **and** $\tau_{y,R}$
- Determined on the "recovered curve"



Rheological results – Nanoclay

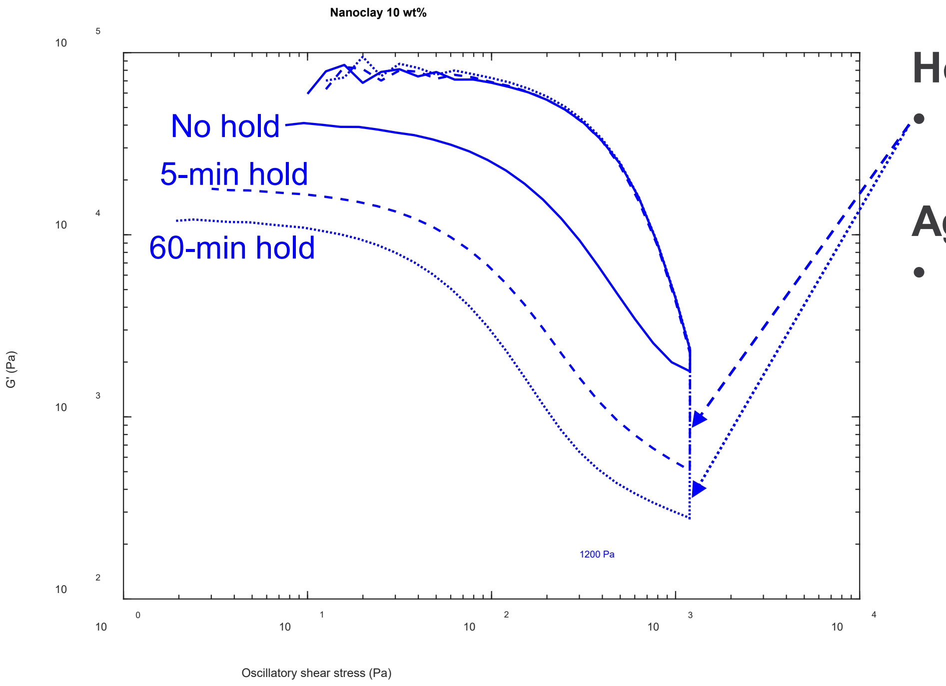


Increased maximum stress decreases recovery

Recovery is dependent on shear history

- Extrusion defines the shear history

Rheological results – Nanoclay



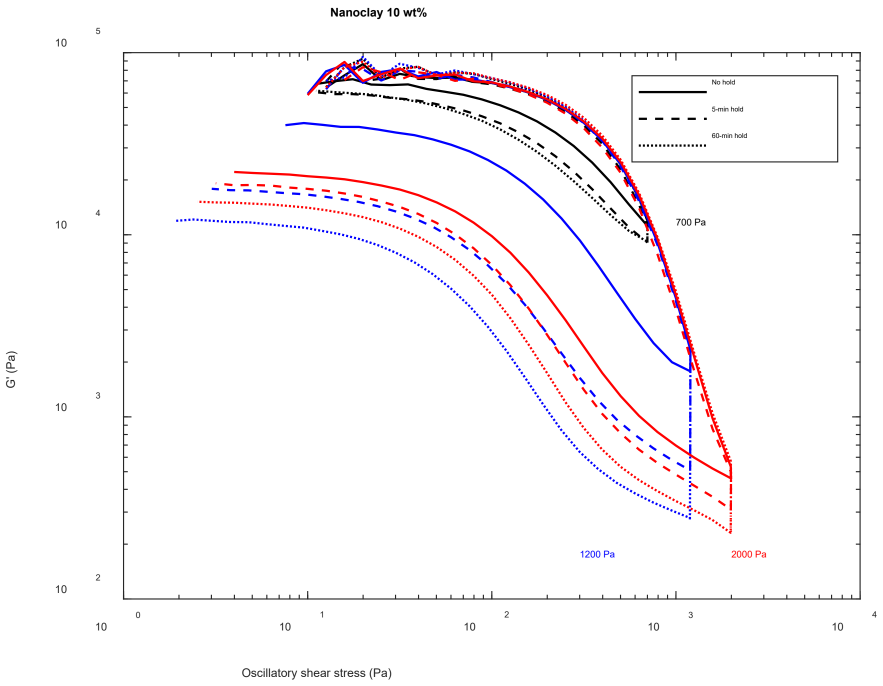
Holding at maximum stress decreases recovery

- Vertical drop at maximum stress

Again, recovery is dependent on shear history

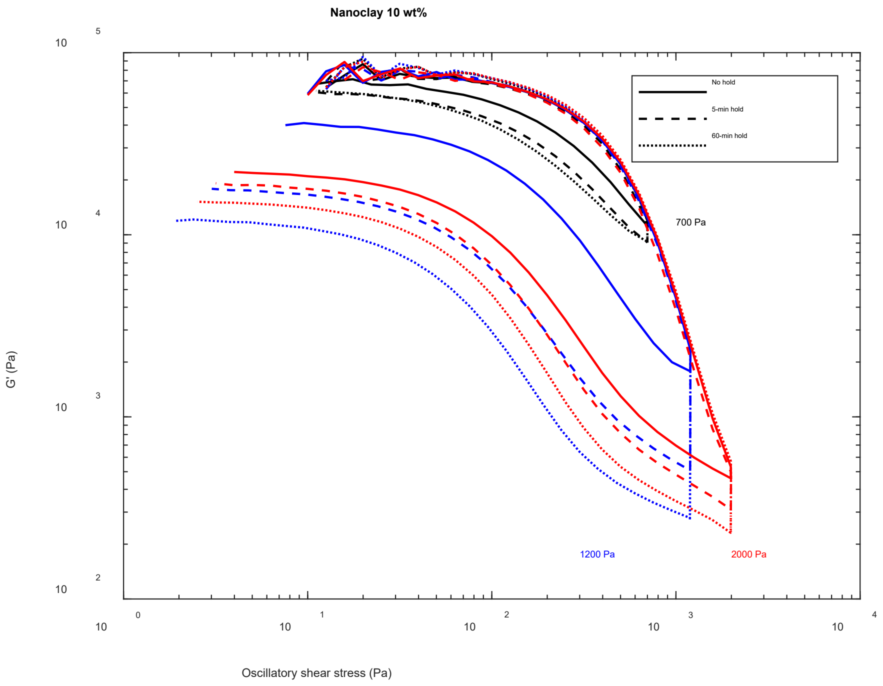
- Extrusion defines the shear history

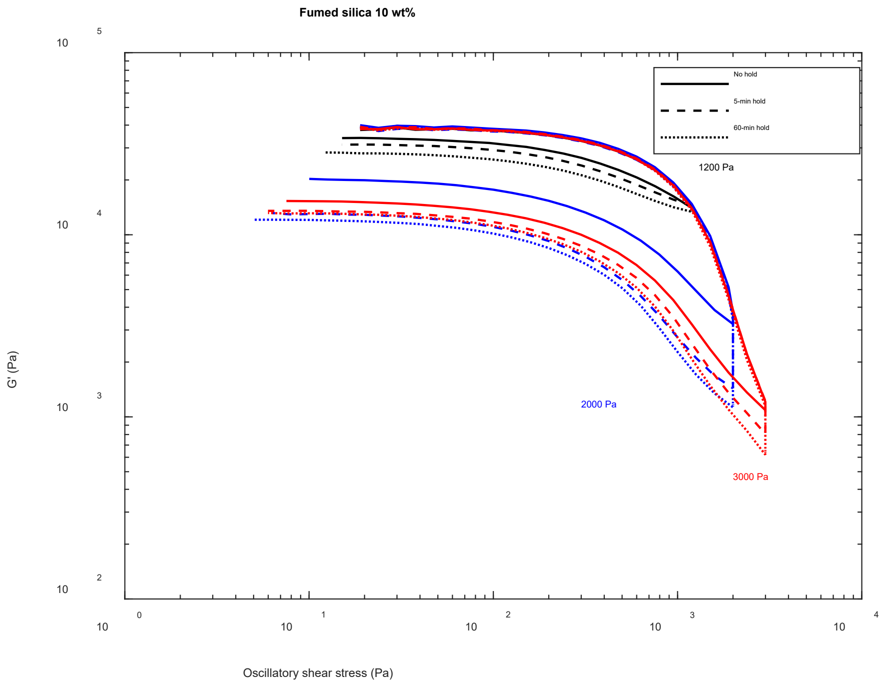
Rheological results – Nanoclay



Result: Array of potential material properties

Rheological results – Fumed silica





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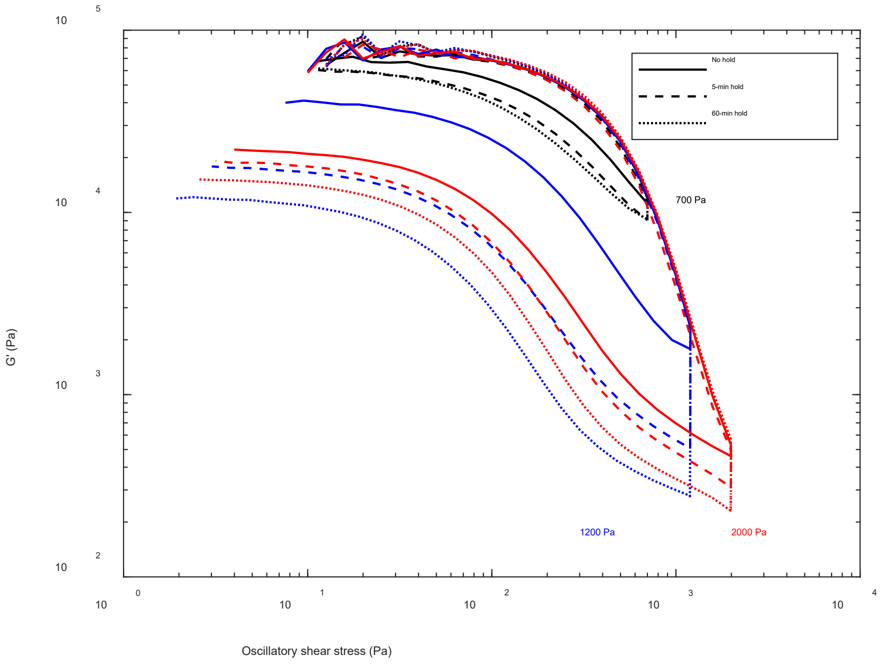
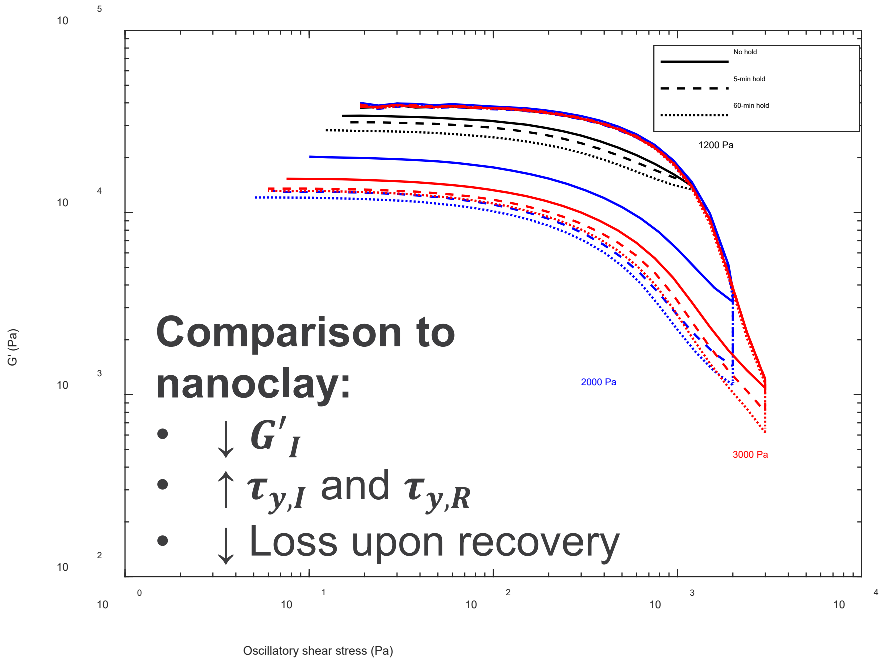
Rheological results – Fumed silica

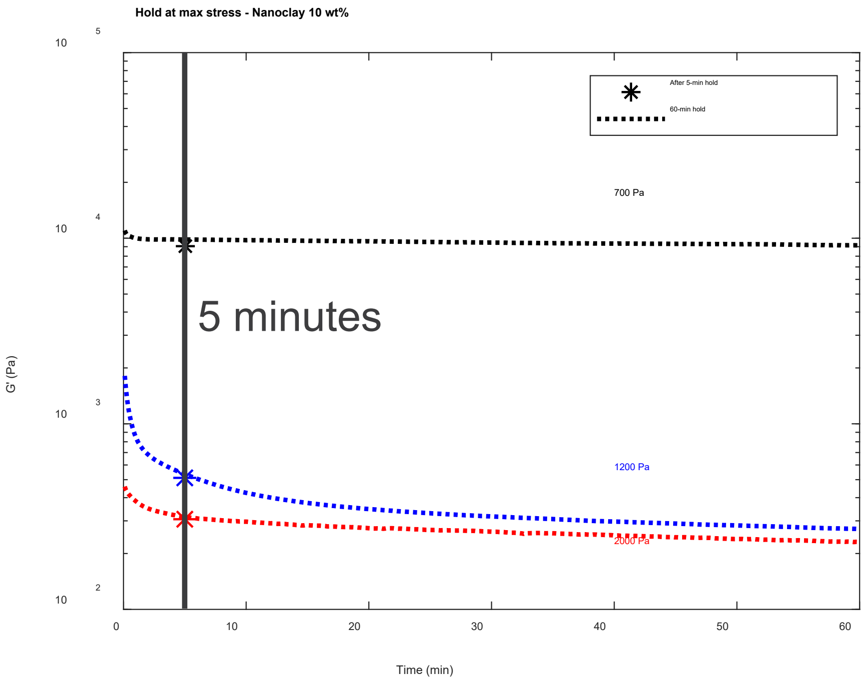




Comparison to nanoclay:

- ↓ G'_I
- ↑ $\tau_{y,I}$ and $\tau_{y,R}$
- ↓ Loss upon recovery

Rheological results – Time hold behavior

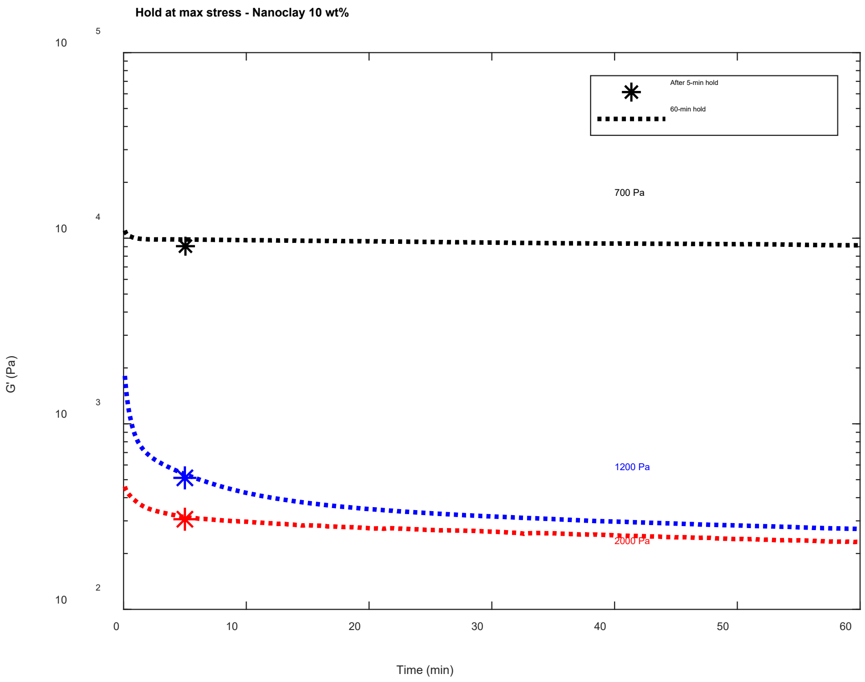


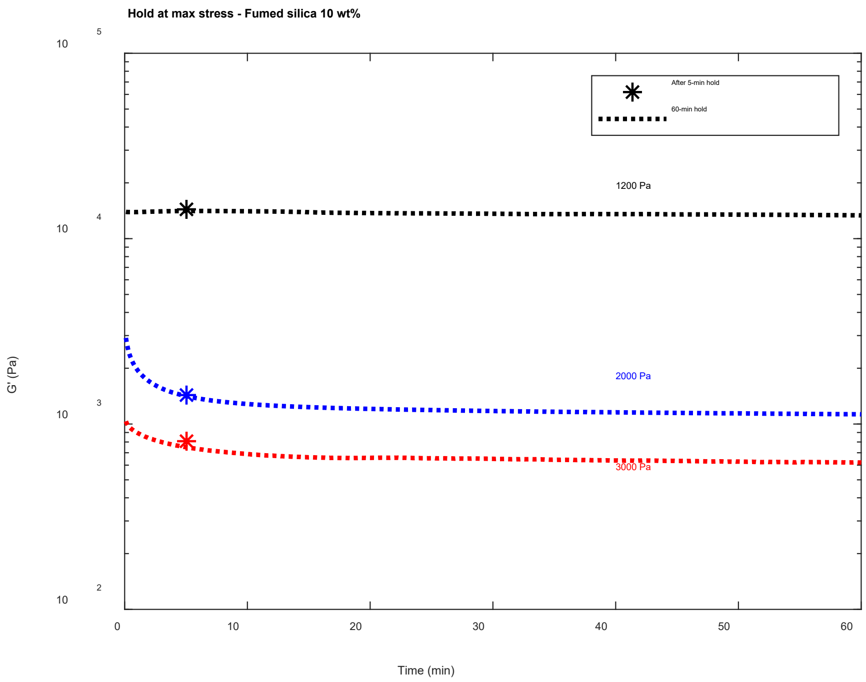
5 minutes – G' is still decreasing

60 minutes – Near asymptotic behavior

- Most aggressive shear history

Rheological results – Time hold behavior





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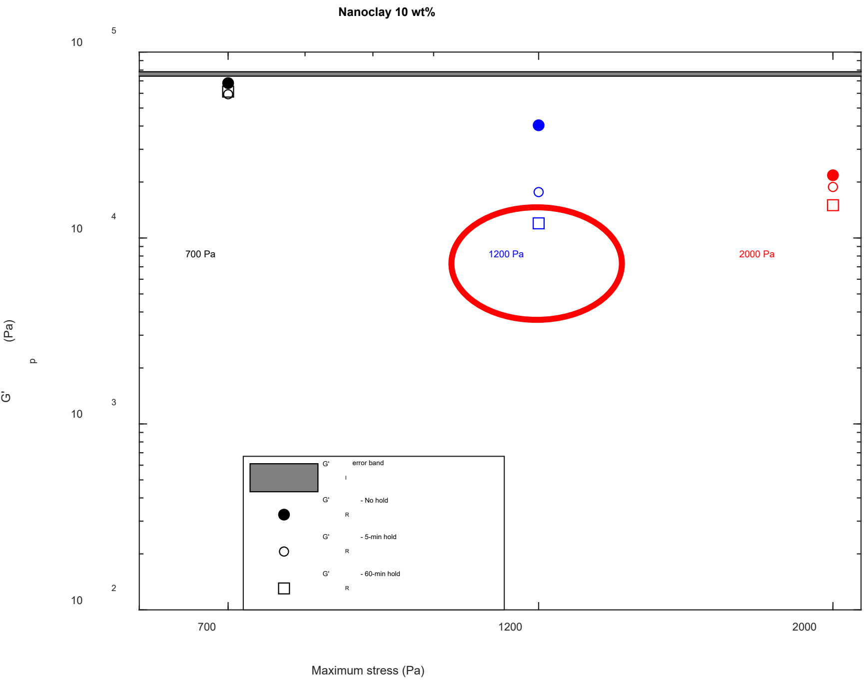
Rheological results – G'_P



Summary of oscillatory shear curves

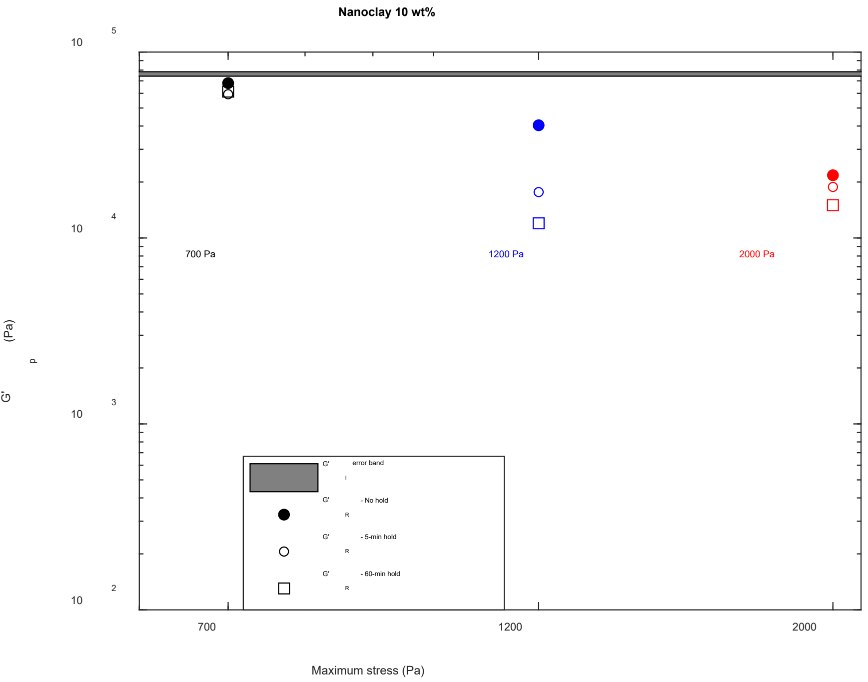
G'_P is inversely proportional to:

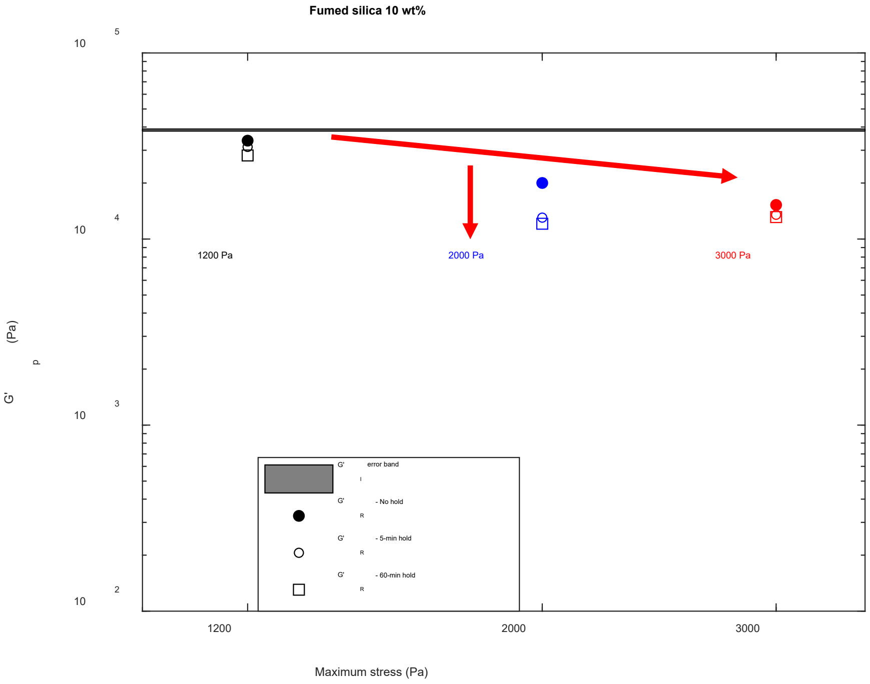
- Maximum stress
- Time held at maximum stress

1200 Pa returns the lowest recovered modulus

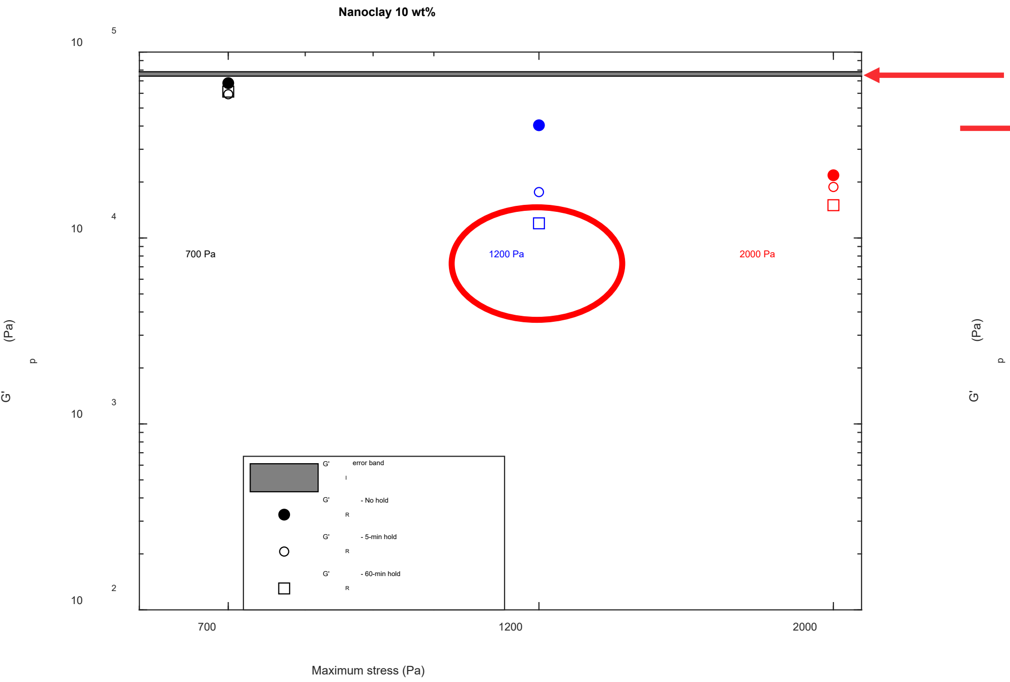
- Warrants further investigation

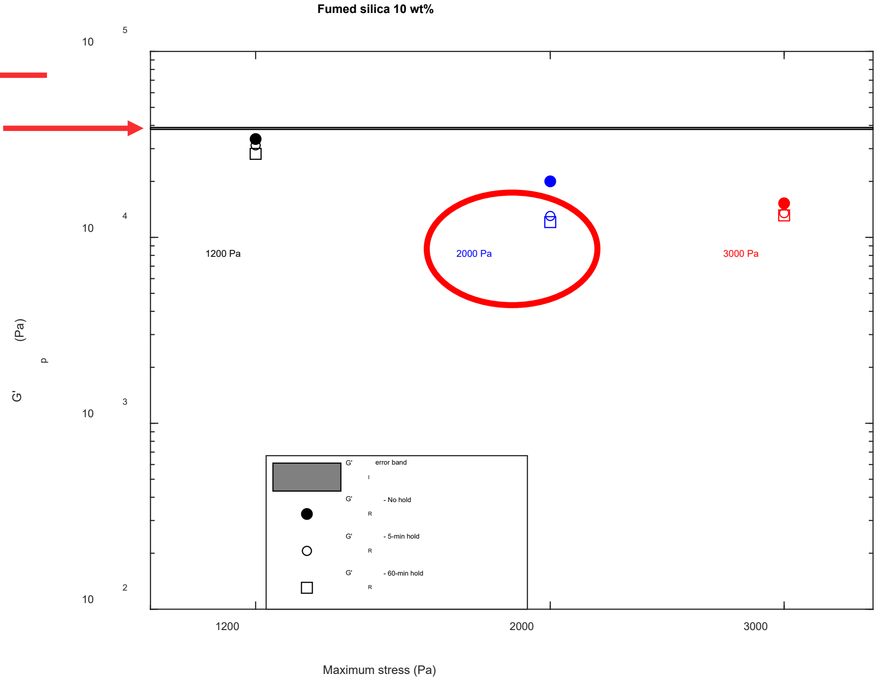
Rheological results – G'_P





Rheological results – G'_P





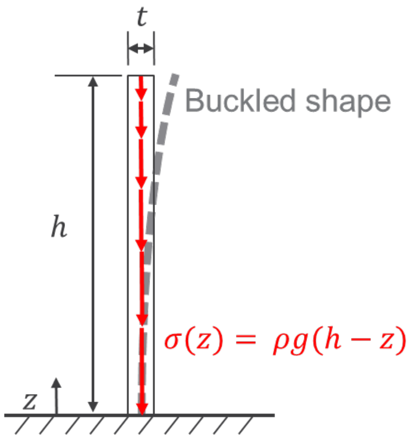
Rheological results – Model inputs

Rheological properties are used as wall height model inputs

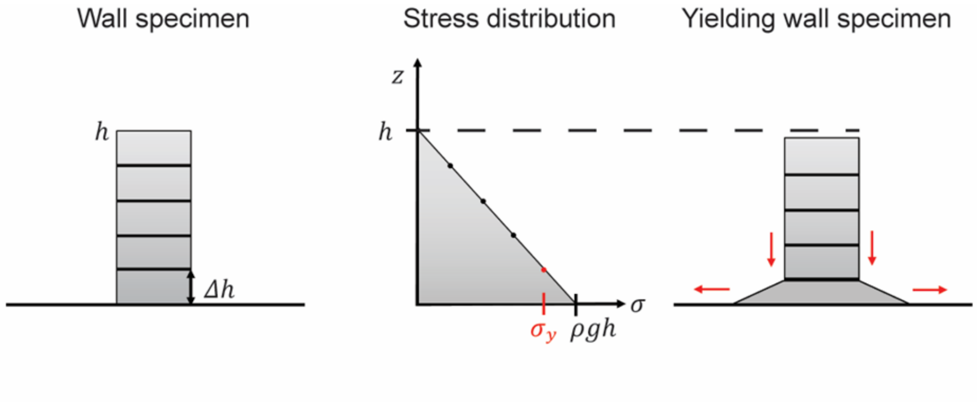
	G'_I (Pa)	G'_R (Pa)	$\tau_{y,I}$ (Pa)	$\tau_{y,R}$ (Pa)	ρ (kg/m^3)
Nanoclay	76,282	11,990	393	80	1199
Fumed silica	38,040	12,080	944	360	1202

Model predictions

Buckling under self-weight



Yielding under self-weight



*Greenhill predicted a maximum height of 300 feet for a pine tree with a 20-inch diameter

Model predictions – Self-weight buckling

Self-weight column buckling height

- Greenhill 1881: $h_{c,b} = \left(7.8373 \frac{EI}{\rho g A}\right)^{1/3}$

Generalize column expression to plates

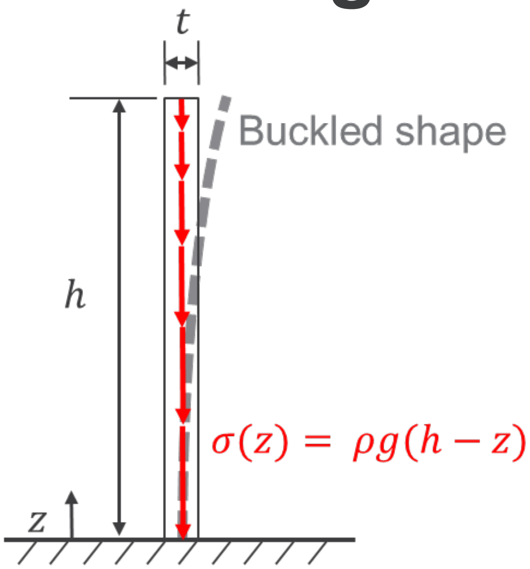
- Plane-strain modulus: $\tilde{E} = \frac{E}{1-\nu^2}$

Below yield, elastic behavior is assumed

- $G = G'$

Isotropy is assumed

- $E = 2G(1+\nu) = 2G'(1+\nu)$



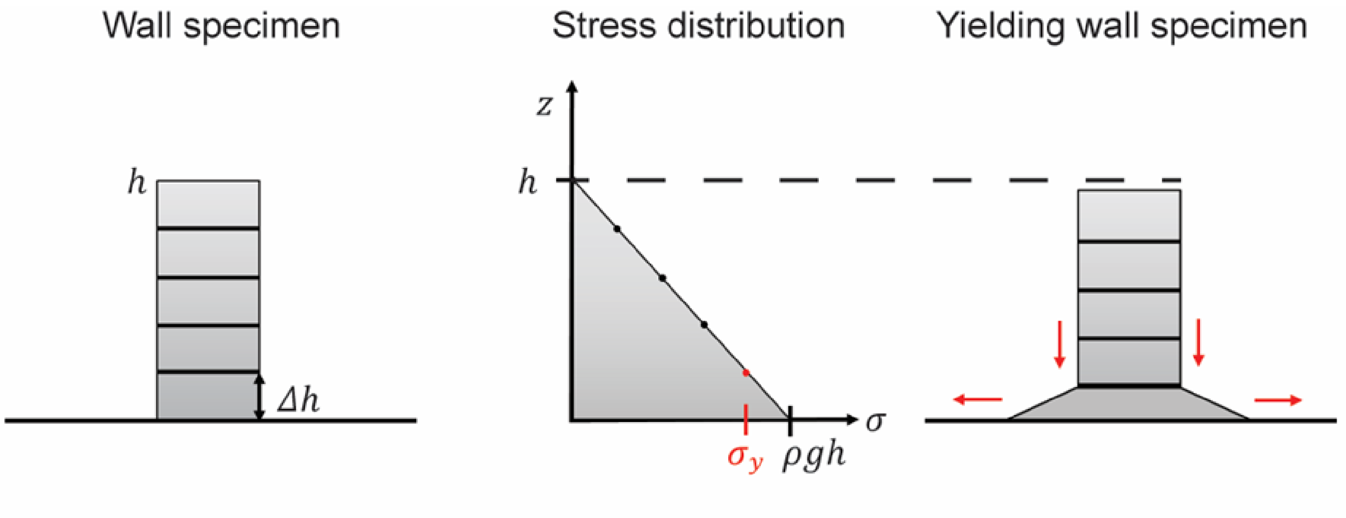
Printed wall buckling height:

$$h_b = \left(7.8373 \left(\frac{G'_P t^2}{6\rho g}\right)\left(\frac{1+\nu}{1-\nu^2}\right)\right)^{1/3}$$

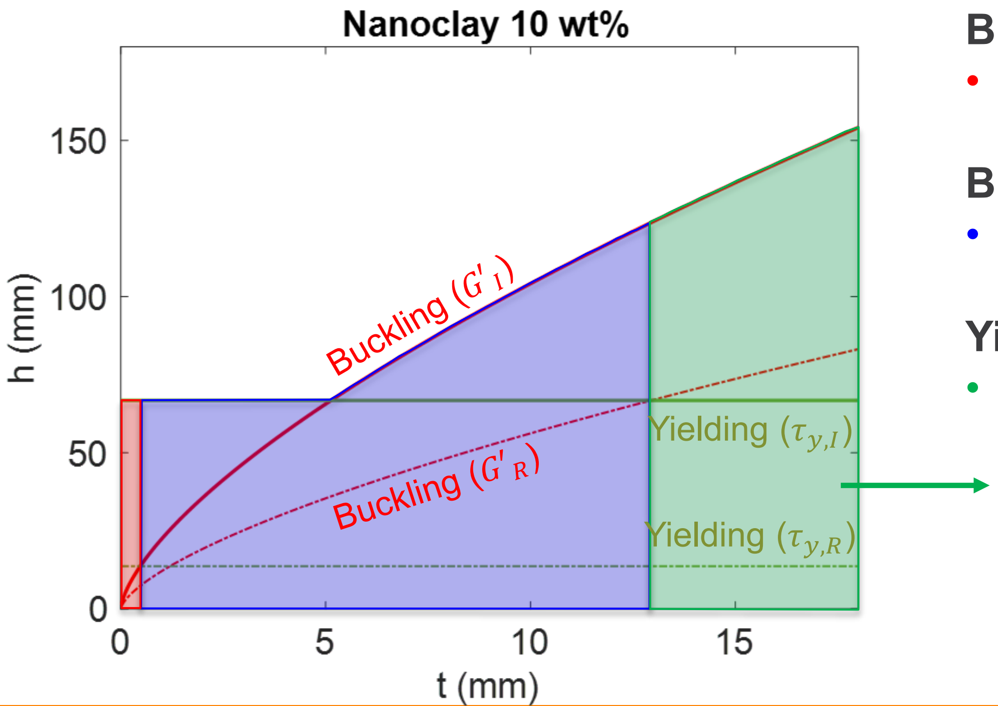
Model predictions – Self-weight yielding

- Yield height: $h_y = \frac{\sigma_y}{\rho g}$
- Rheological tests provide τ_y
- Max shear (Tresca) yield criterion for uniaxial loading: $\sigma_y = 2\tau_y$

$$h_y = \frac{2\tau_y}{\rho g}$$



Model predictions – Results



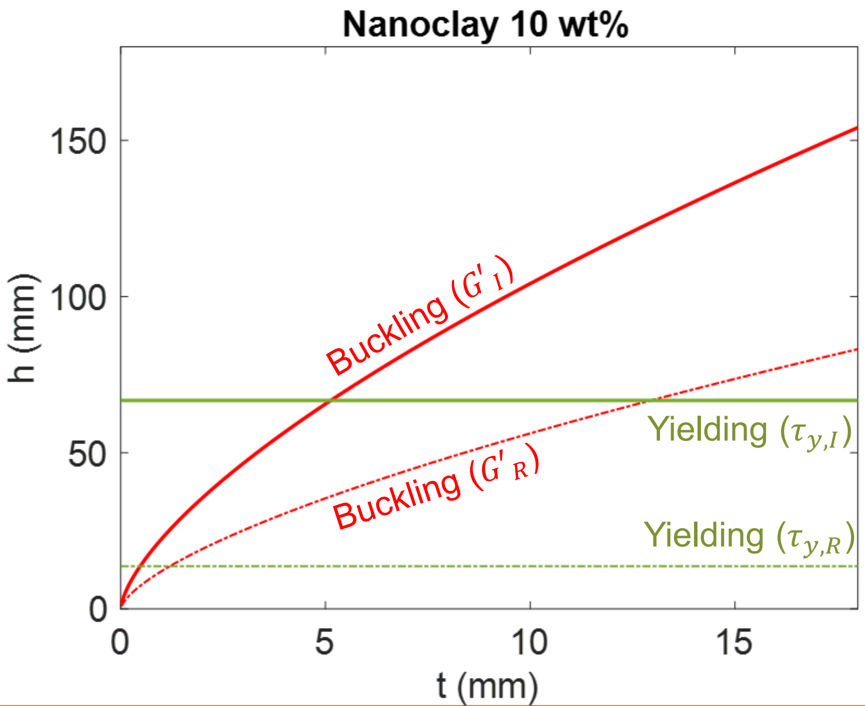
Buckling at small thickness
- **Red**

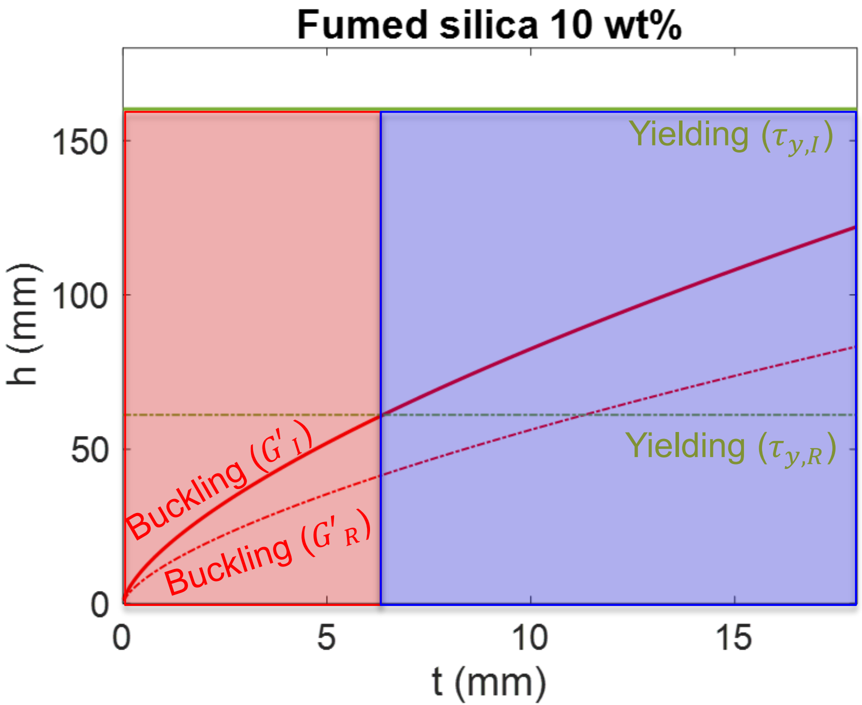
Buckling or yielding at intermediate thickness
- **Blue**

Yielding at large thickness
- **Green**

Model predictions – Results

Yielding at large thickness





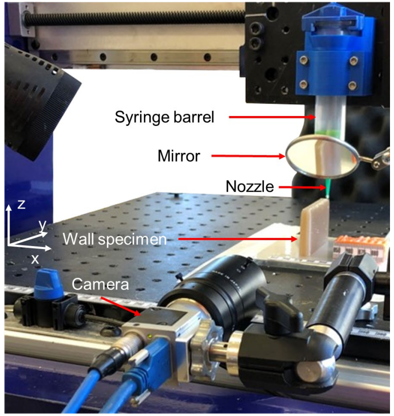
Stability tests – Data analysis

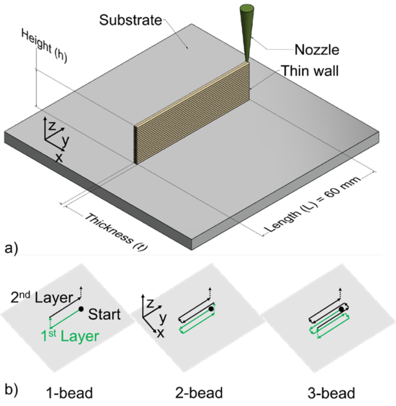
Printing video

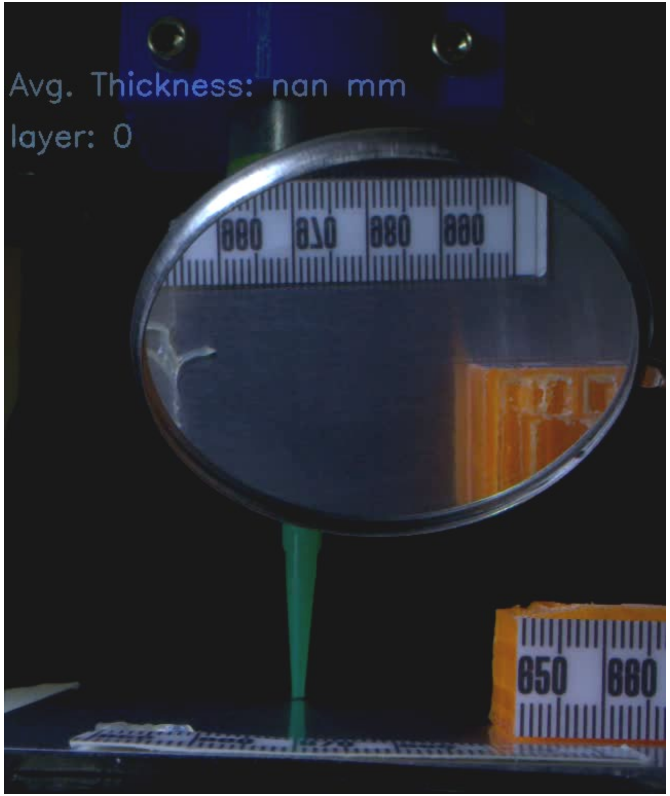
Image processing – onset of buckling

1. Identifies wall edges
2. Calculates wall thickness
3. Identifies the onset of buckling ($\boldsymbol{h_b}$)

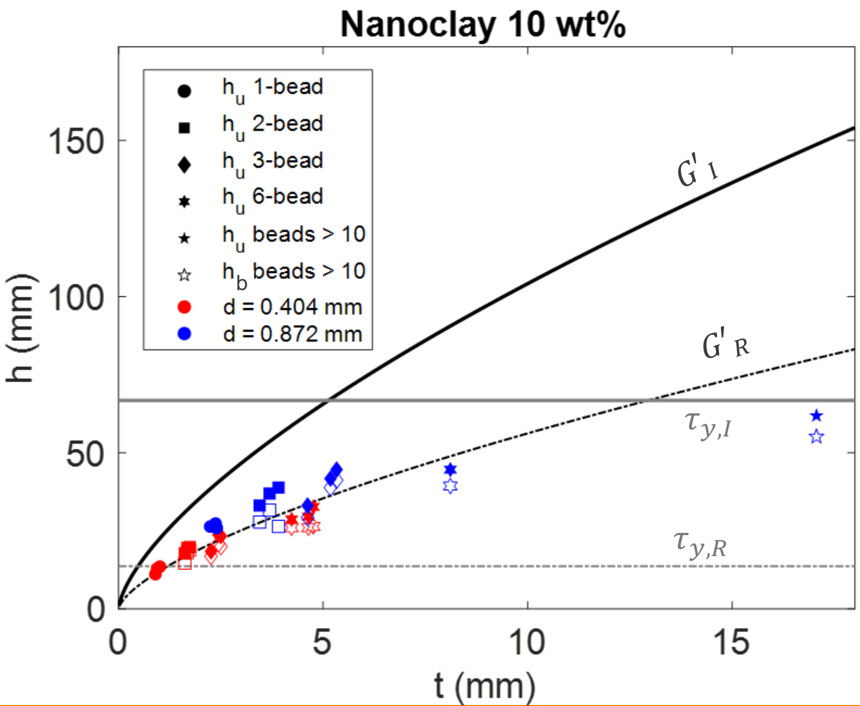
Full collapse ($\boldsymbol{h_u}$) is defined when material is no longer deposited on the top of the wall







Stability tests – Results



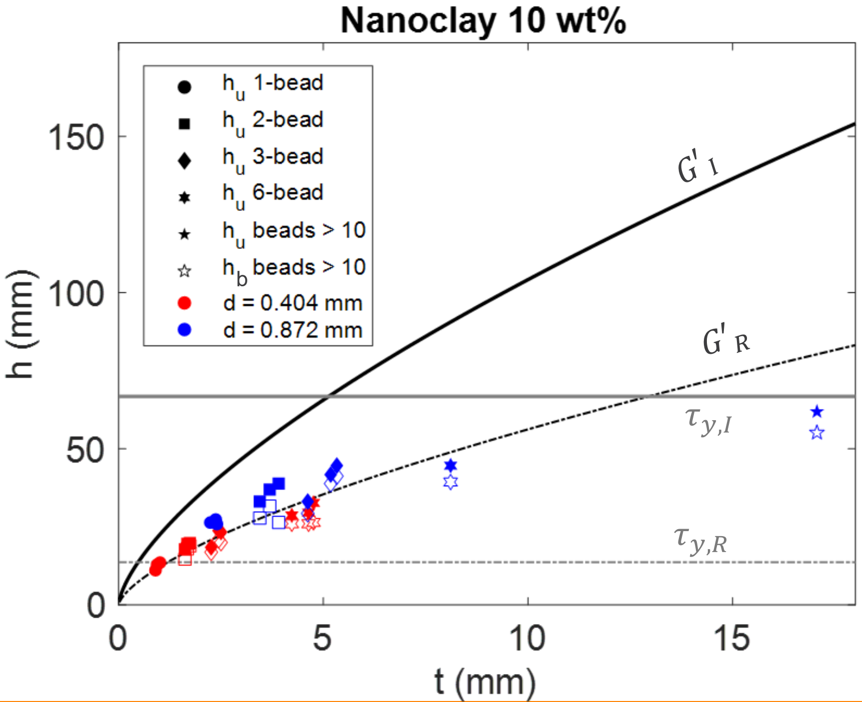
Nanoclay ink

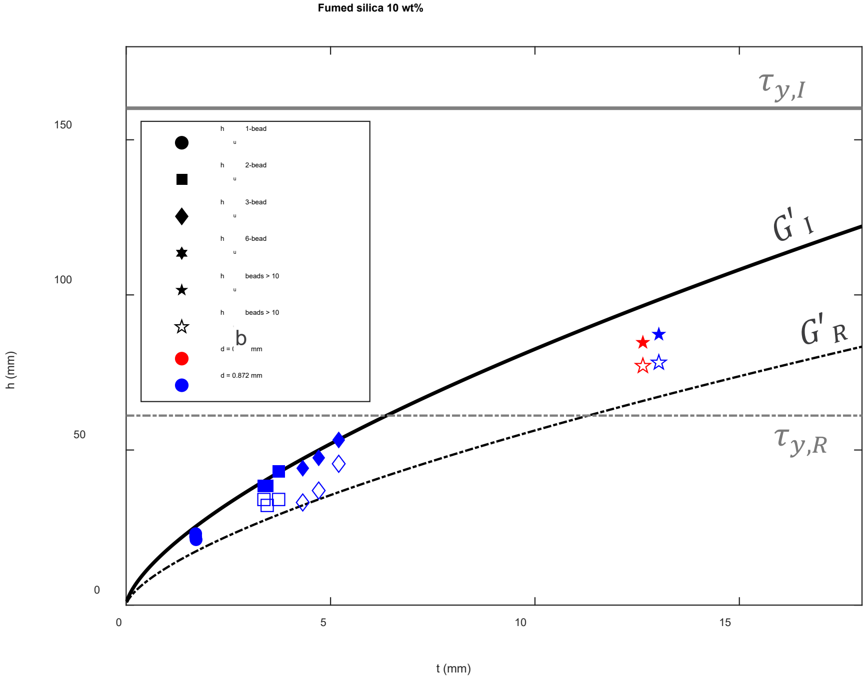
- Data follows "Buckling (G'_R)" at low thickness
- Transitions to "Yielding ($\tau_{y,I}$)" around 13 mm

Stability tests – Results

Fumed silica ink

- Between Buckling (G'_R) and (G'_I)

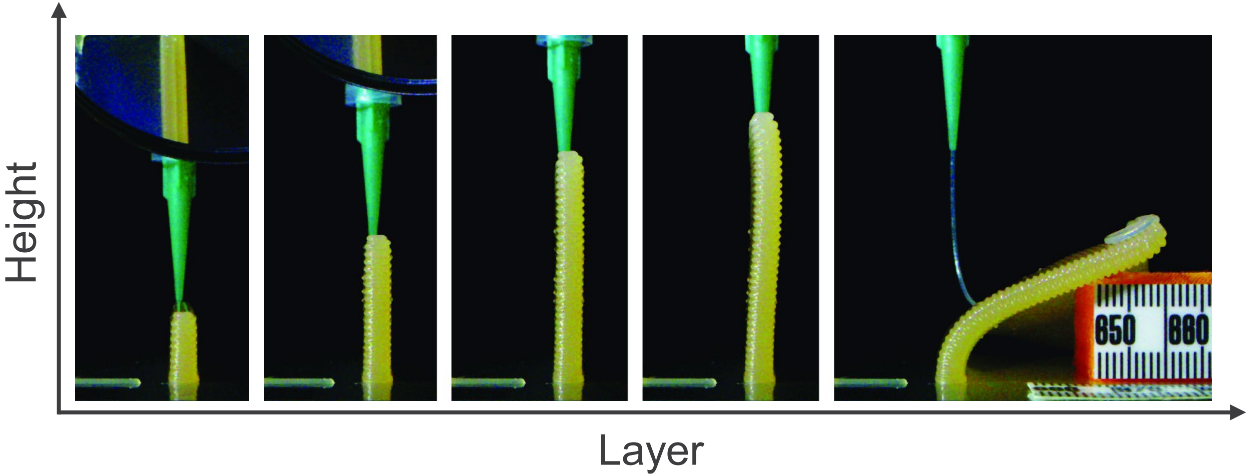




Stability tests – Wall profile

Nanoclay ink

- Slender walls buckle



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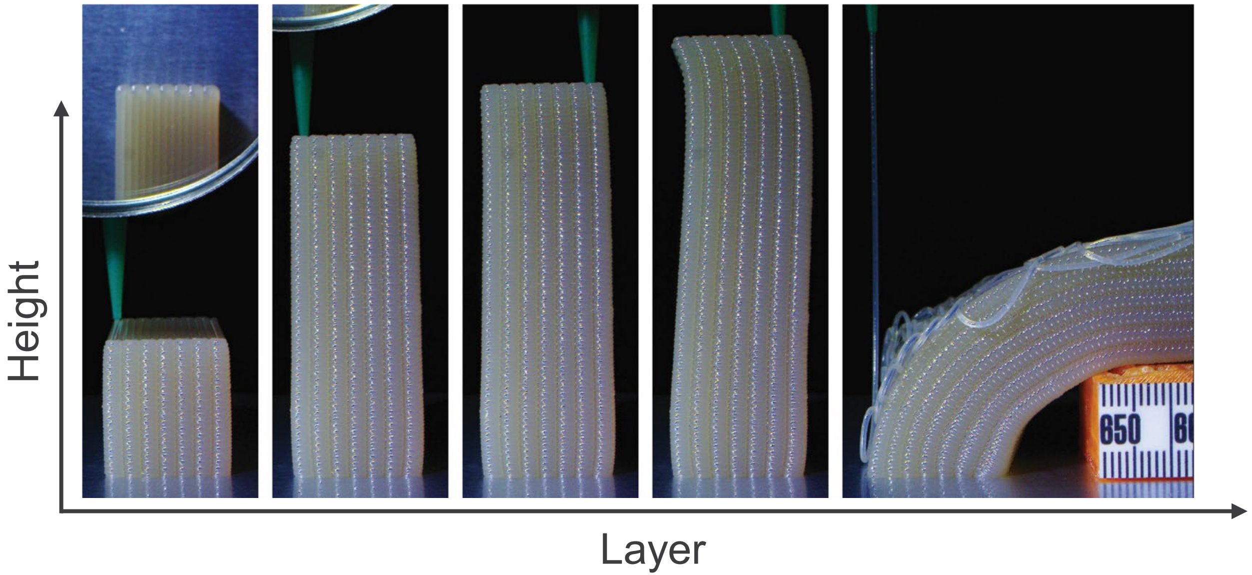
Stability tests – Wall profile

Nanoclay ink

- Thick walls yield then buckle



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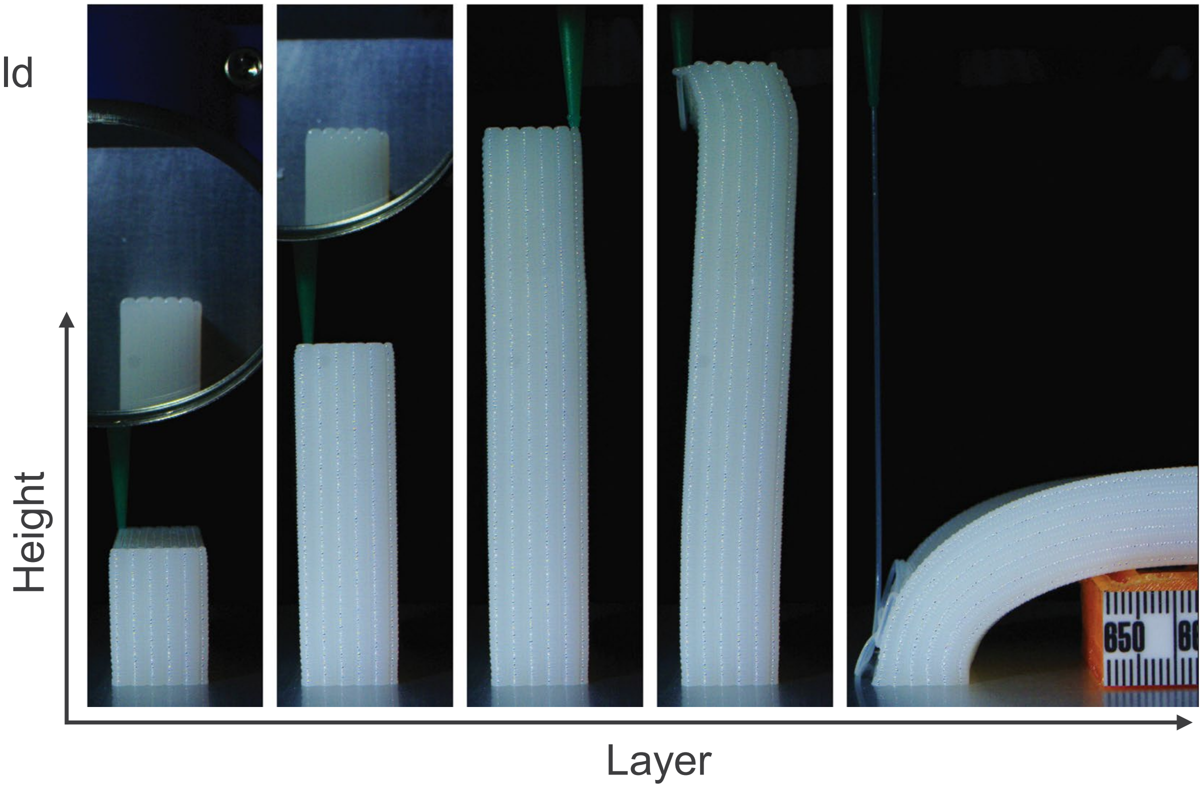
Stability tests – Wall profile

Fumed silica ink

- Thick walls buckled without yield



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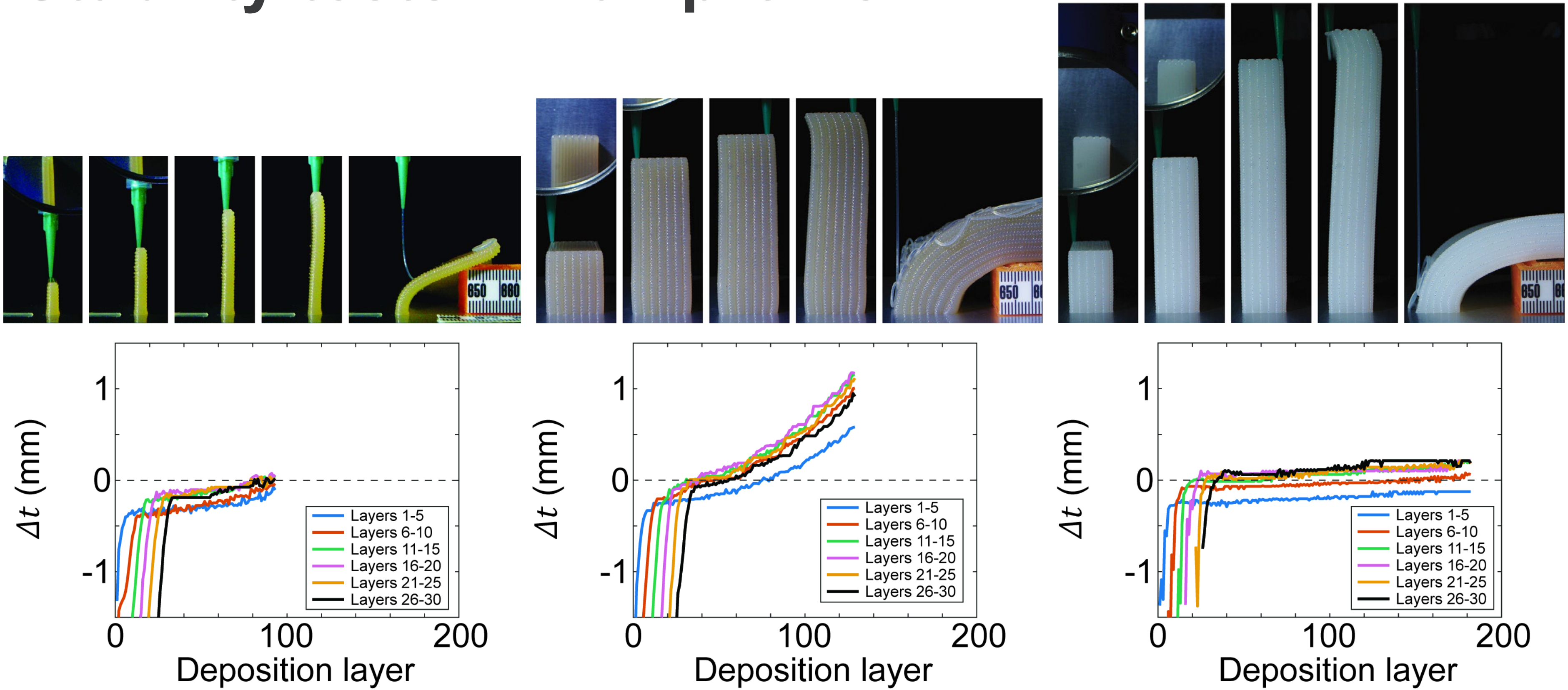
Stability tests – Wall profile



Summary

Established a link between measurable rheology and stability

Geometry also plays a role in collapse

- Thinner walls exhibited buckling behavior
- Thicker walls demonstrated yielding behavior

Recovery is key to appropriately bounding predictions

Filler type affects recovery and therefore stability

Unclear whether initial or recovered properties dominate behavior

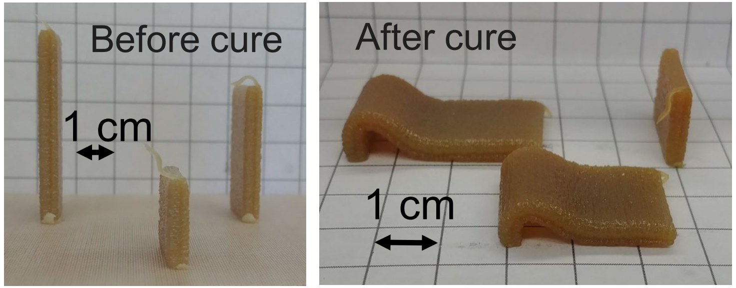
Future work

Informed material design

Characterize the time-dependence of recovery

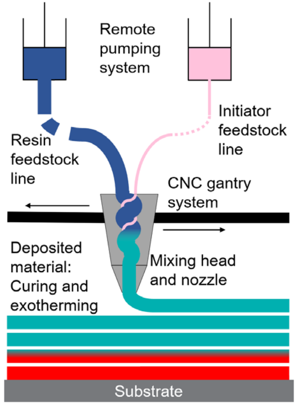
Understand collapse during heated cure

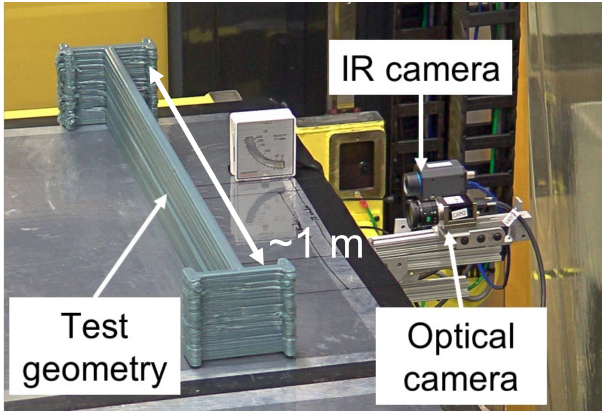
Evaluate materials that cure during printing



Images courtesy of Madeline Wimmer

Video showing exothermic behavior of in-situ curing





Acknowledgements

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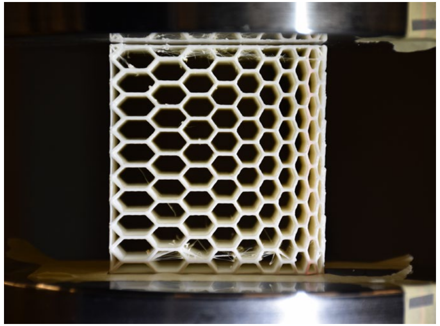
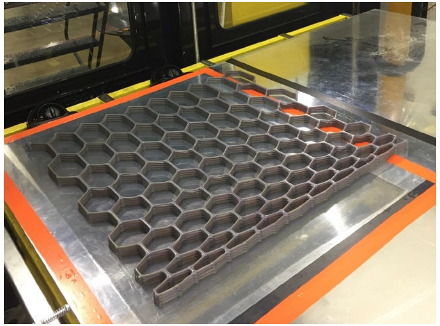
MVP
Magnum Venus Products

Polynt Composites





Thank you for your attention!

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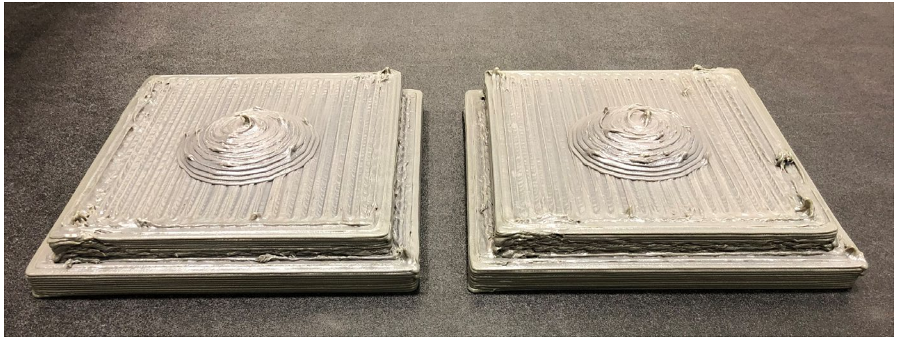

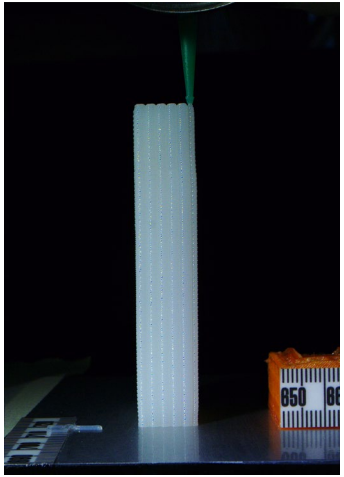

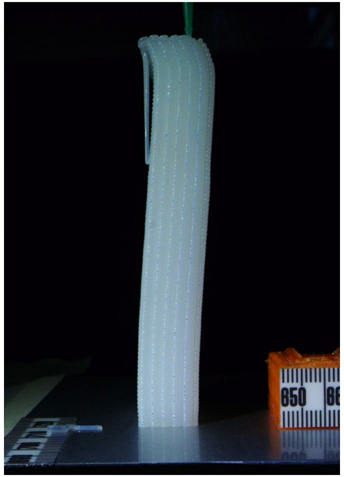

Questions?

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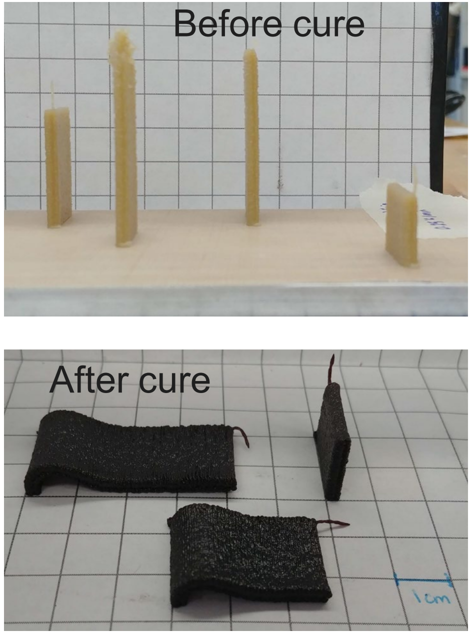
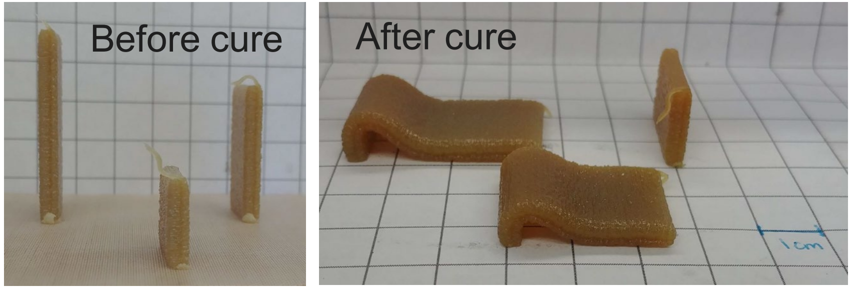
Stability challenges



Images courtesy of Madeline Wimmer



Images courtesy of Madeline Wimmer

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Future work – Recovery vs time