Additively Manufactured Ceramics for High-Temperature Heat Rejection Systems

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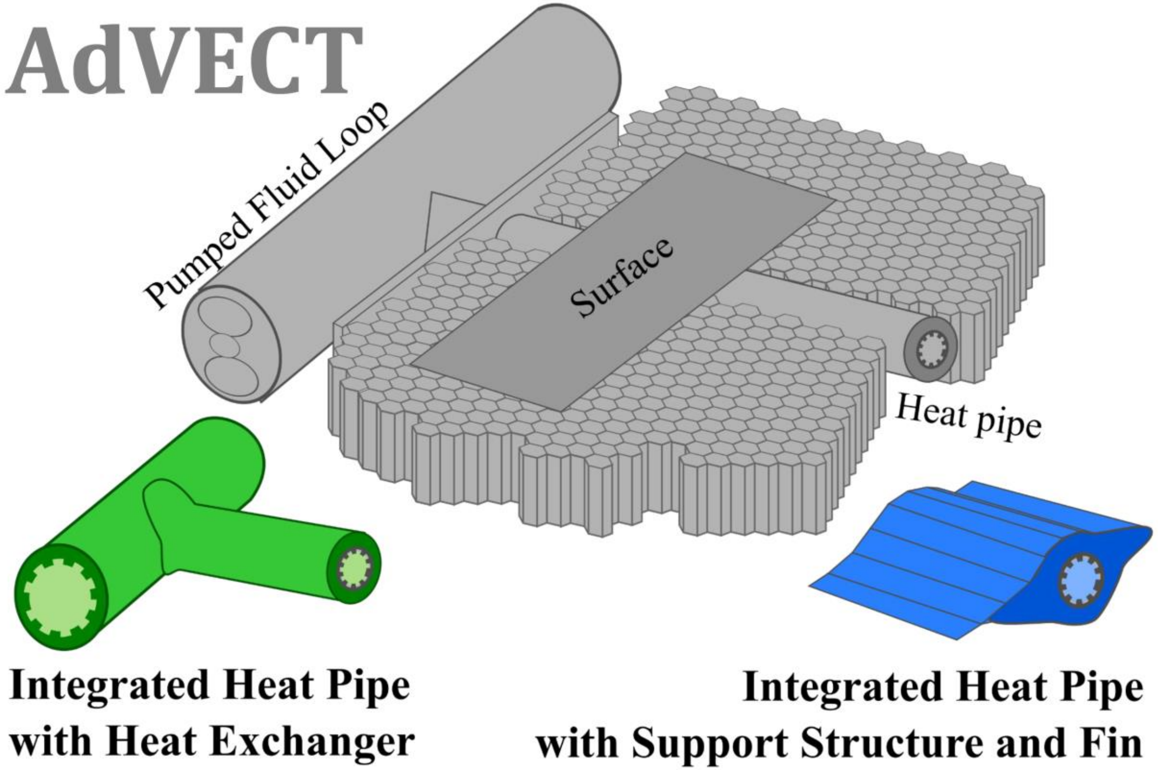
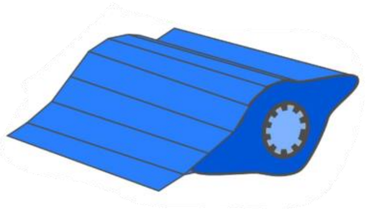
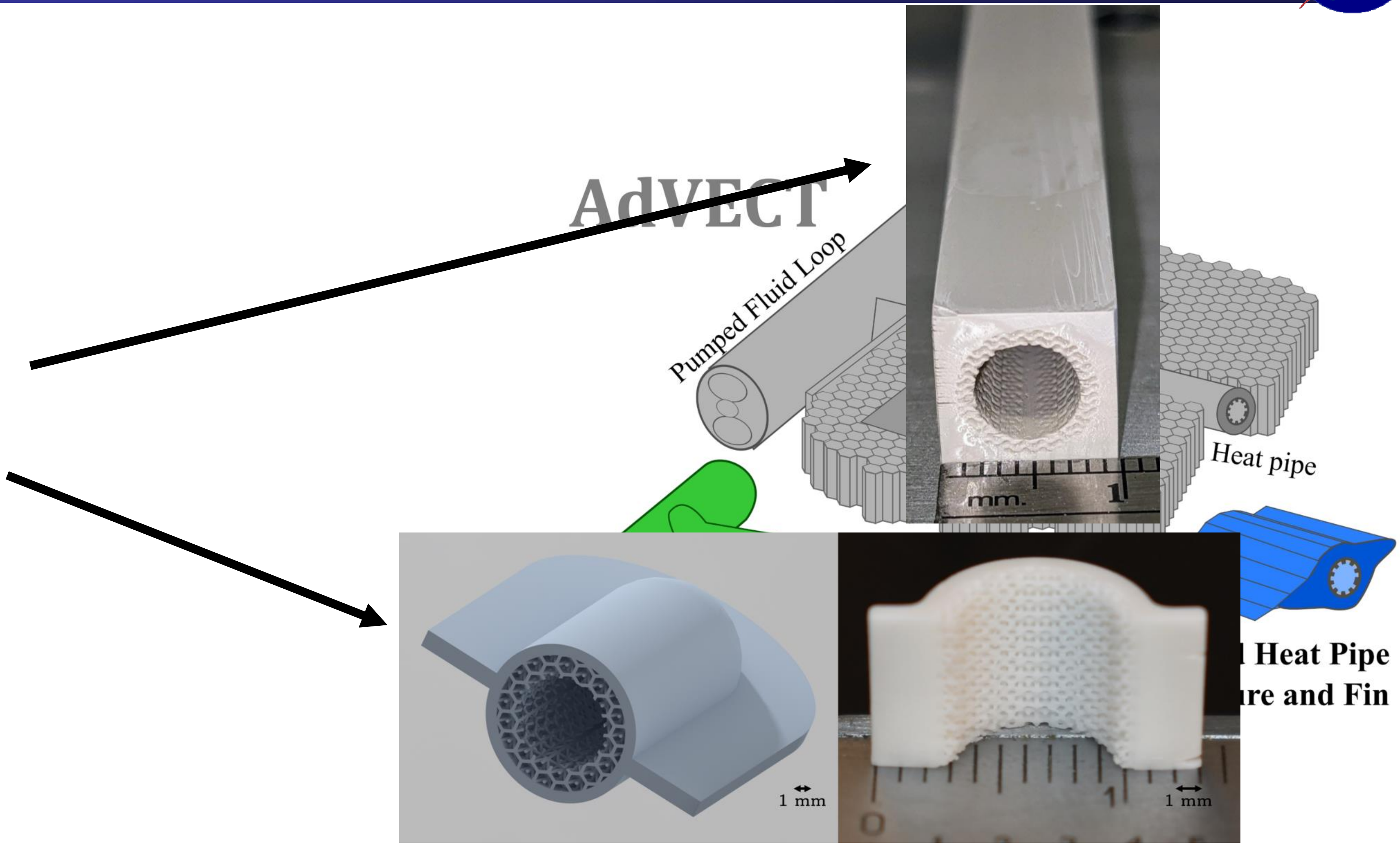
Additive Vehicle-Embedded Cooling Technologies

- AdVECT
  - Novel, high temperature heat pipes
  - Leverage AM
    - Consolidate parts
    - Complex internal structures
  - Decrease system mass
  - Increase working fluid compatibility



Additive Vehicle-Embedded Cooling Technologies

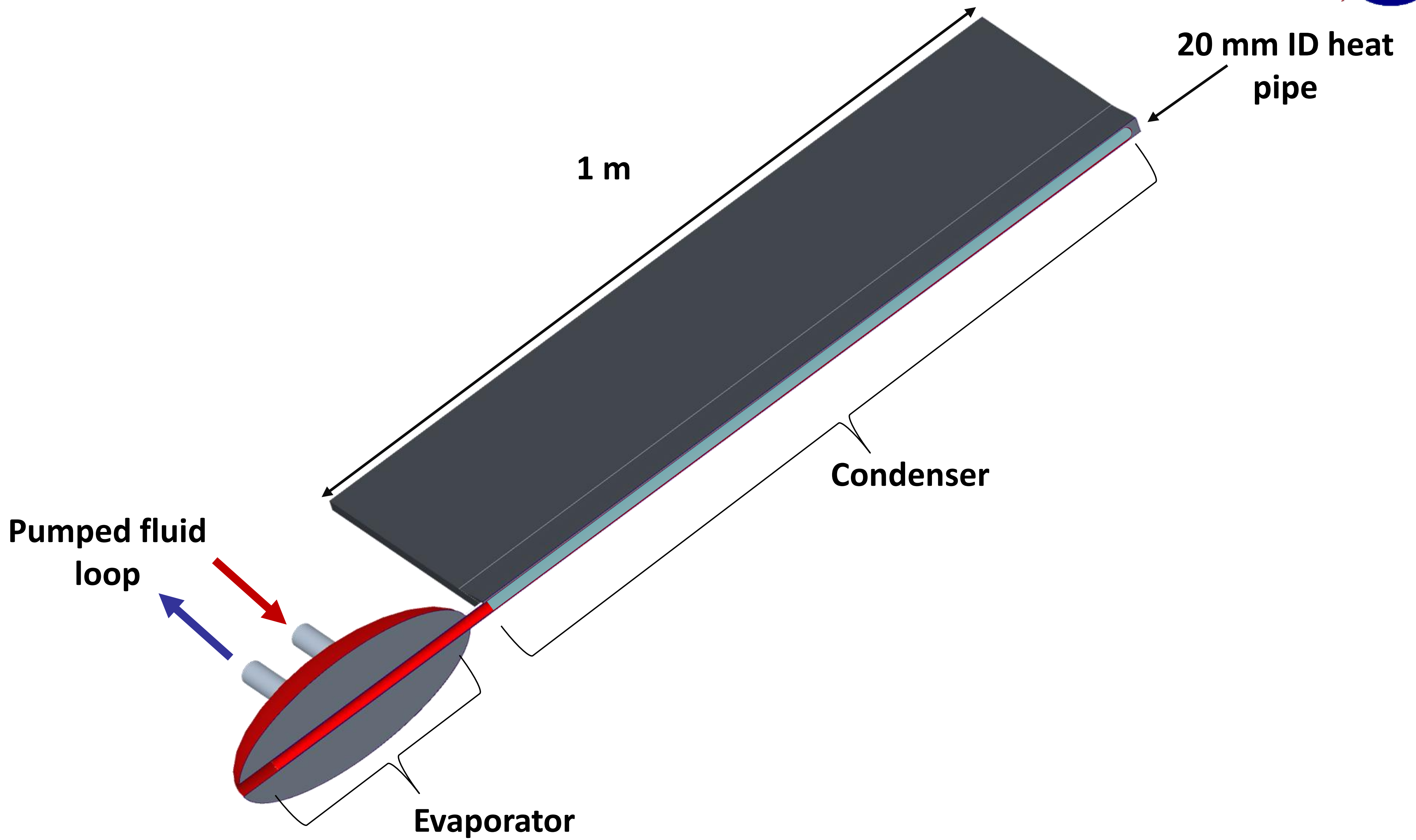




Additive Vehicle-Embedded Cooling Technologies

- 500-600 K operating regime
  - Operating temp above conventional Ti/Al & water/ammonia systems
- Low mass (< 2 kg/m^2)
  - Additively manufactured (AM) ceramics reduce part density
- Potential Working Fluids
  - Halides
    - Iodine
    - Aluminum chloride
    - Aluminum bromide
    - Iron (III) chloride
  - Eutectics
    - Dowtherm A
- TRL 3 development goal

Additive Vehicle-Embedded Cooling Technologies



Additively Manufactured Ceramics

- Three primary fabrication methods
  - Binder Jetting
    - Low resolution (50-200 μm layers,
    - Requires large ceramic particles (generally ≥ 50 μm), porous
  - Direct Ink Writing
    - Low resolution (minimum feature size tip dependent, generally > 100 μm, > 50 μm layer height)
    - Can use small or large ceramic particles
  - Digital Light Processing (DLP)/Stereolithography
    - High resolution (≤ 50 μm minimum feature size, ≤ 50 μm layer height)
    - Small ceramic particles (< 1 μm)

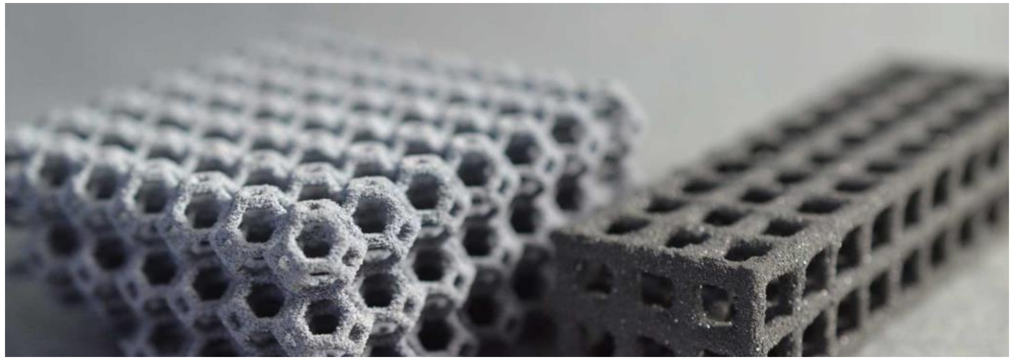
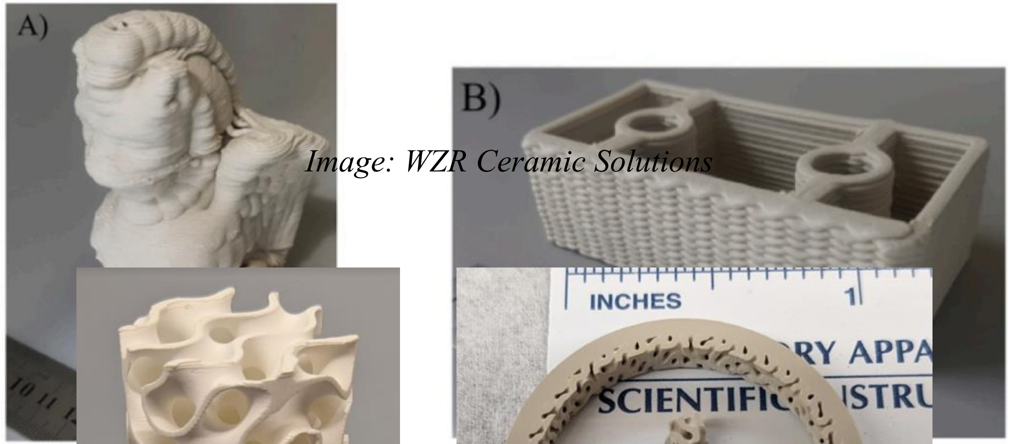
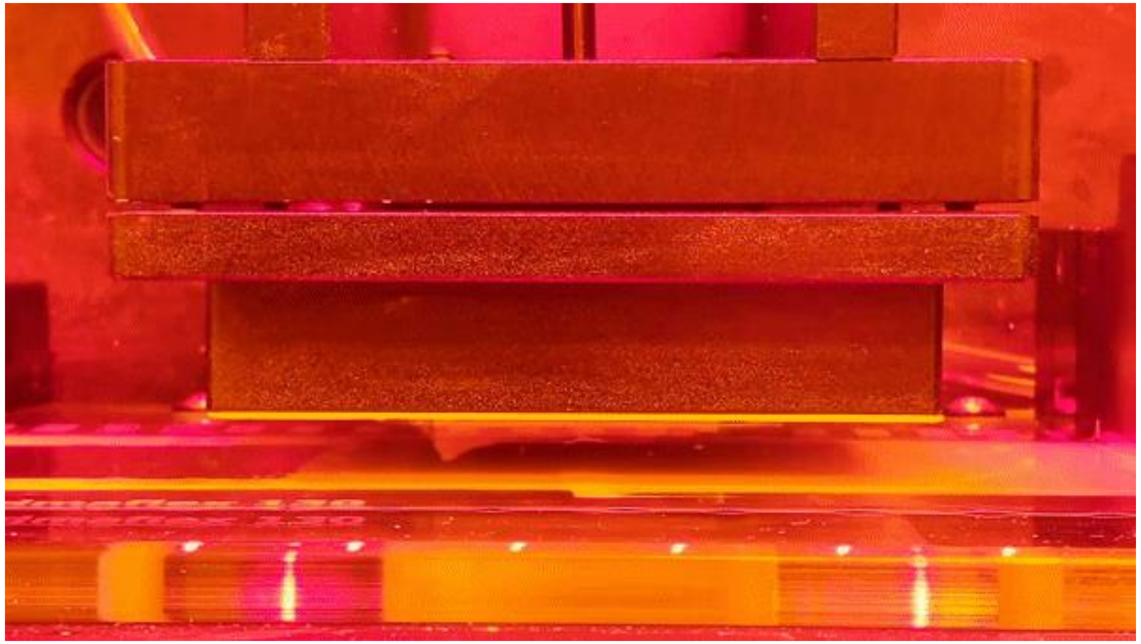


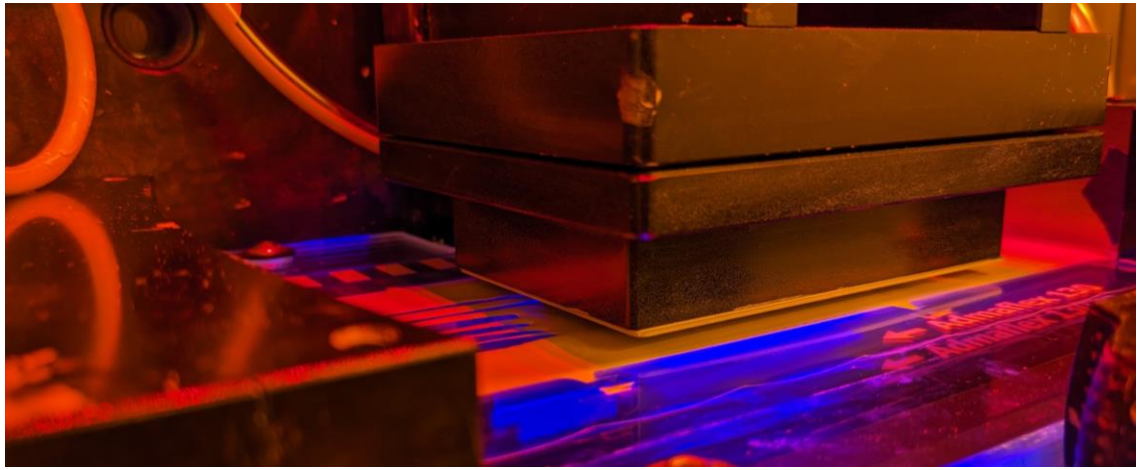
Image: WZR Ceramic Solutions



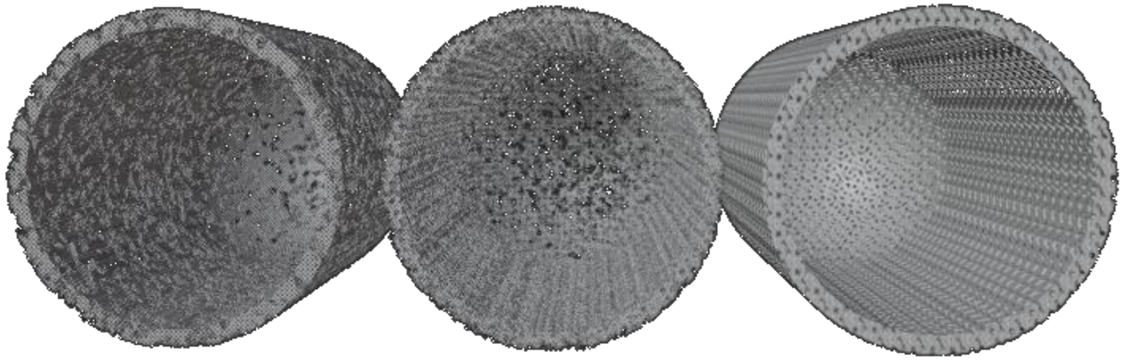
Digital Light Processing

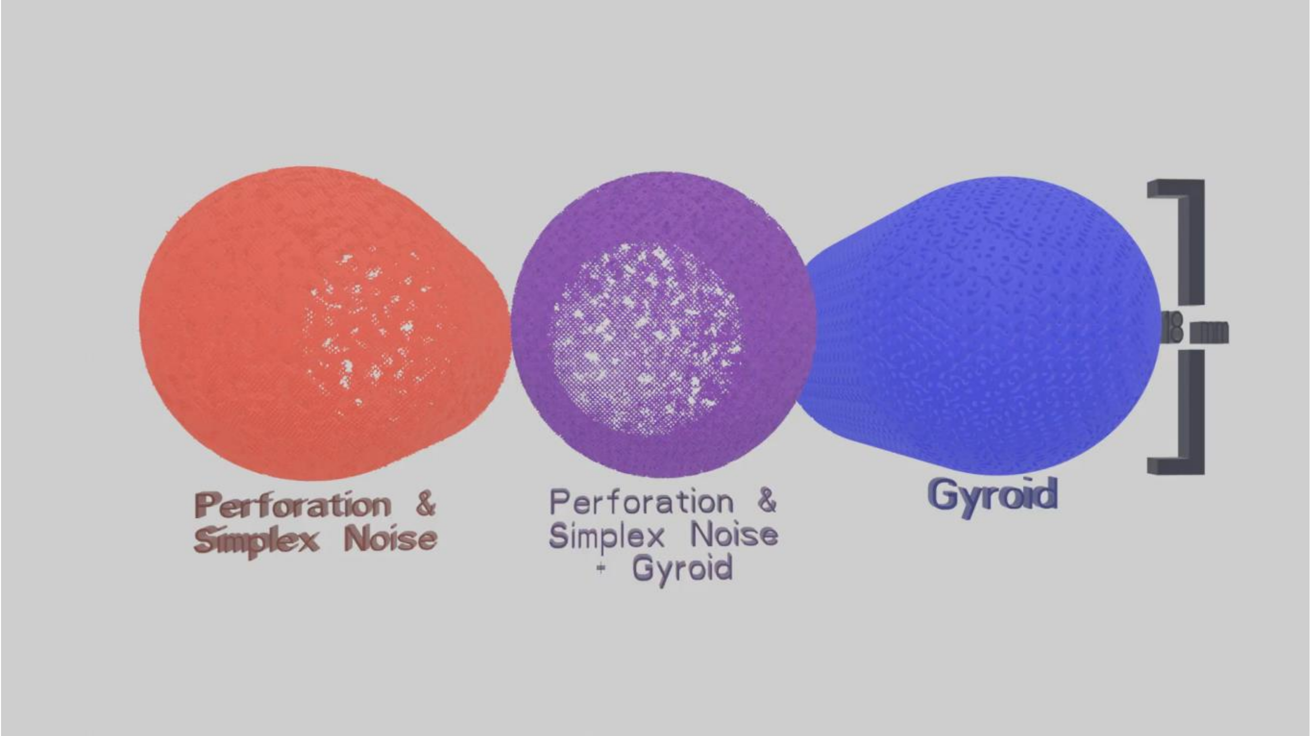
- Admatec Admaflex 130 DLP Printer
  - Exposes photosensitive resin with ceramic particles in suspension
    - 405 nm wavelength
  - 50 µm resolution, 20 µm layer height
  - 54 x 96 x 100 mm build volume
  - > 99% final part density





- AM enables arbitrary complexity
  - Stochastic
  - Functional lattice (triply periodic minimal surfaces)
  - Strut-based
  - Combination of the above





Aluminum Nitride (AlN) Ceramics

- High Thermal Conductivity
  - > 120 W/m·K
- Moderate to High Flexural Strength
  - > 500 MPa
- Low Bulk Density
  - 3.26 g/cm^3
- Outstanding Thermal Shock Resistance
  - >900° C
- High Melting Point
  - ~2200° C
- Excellent Chemical Resistance
  - Wide range of potential working fluids

AM AlN Development

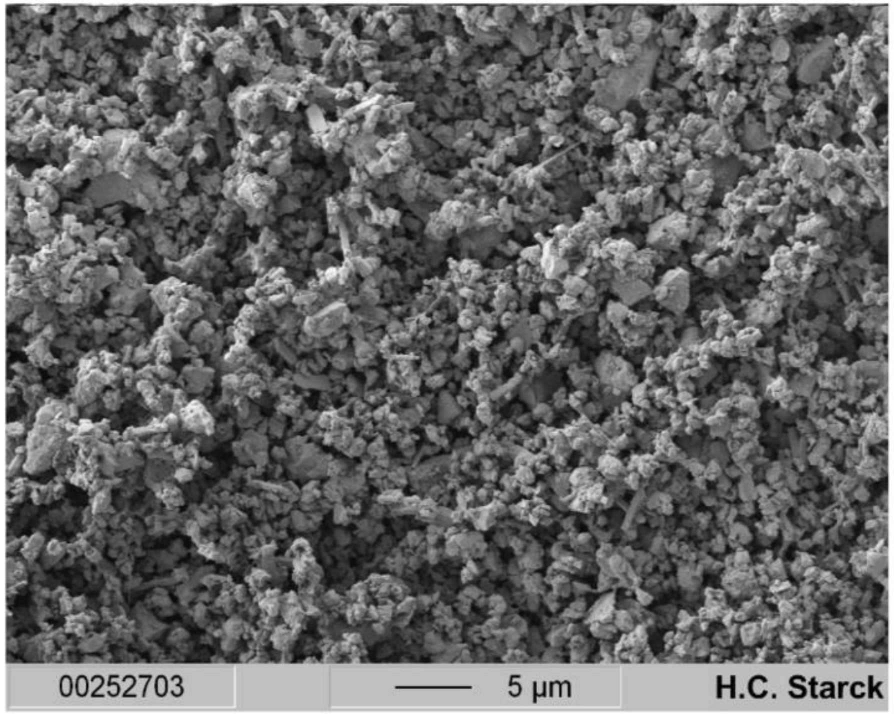
	Composition (wt %)	Sintering Temperature (°C)	Flexural Strength (MPa)	Thermal Conductivity (W/m·K)
Duan et al. 2020	95:5 $AlN:Y_2O_3$	1845	265 ± 20	155
Ożóg et al. 2020	90:6:4 $AlN:Y_2O_3:Al_2O_3$	1800	Not tested	4.34
Lin et al. 2022	100:5 $AlN:Y_2O_3$	1850	365-400	135-150 (appr)
Rauchenecker et al. 2022	96:3:1 $AlN:Y_2O_3:CaO$	1700	320-498	162.1-166.2
Lee and Kim 2014* (Basis for this work)	98:1:1 $AlN:Y_2O_3:CaZrO_3$	1600	579	120

** Conventional ceramics*

AlN Slurry Development

- 60%/vol Admatec Development Resin C
  - Water-soluble acrylate resin with BAPO photoinitiator

- 40%/vol ceramic solids
  - **98%/wt Höganäs Grade C AlN powder**
    - Specific Surface Area : 1.8 – 3.8 m^2/g
    - Particle Size: 0.8 - 2.0 micron
  - **1%/wt Y_2O_3 nanopowder**
    - Particle Size: 10 nm
  - **1%/wt $CaZrO_3$ nanopowder**
    - Particle Size: 40 nm
  - Ceramics mixed with 1%/wt with Hypermer KD1 Dispersant



AlN Grade C
Image: Höganäs GmbH

Silicon Nitride Slurry Development

- High refractive index materials challenging to print

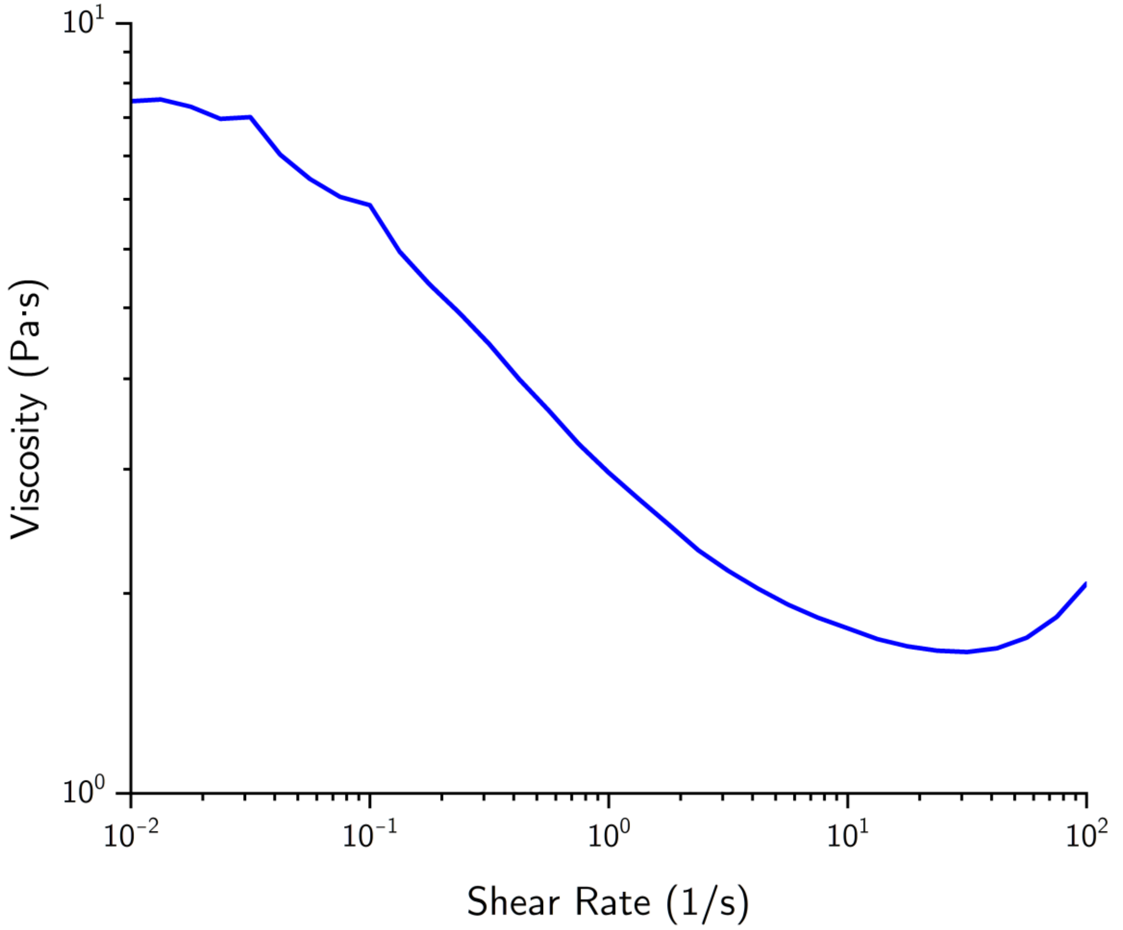
$$D_{cure} \approx \frac{C}{\Phi} \cdot d_{part} \left(\frac{n_0}{\Delta_n}\right)^2 \cdot ln\left(\frac{I}{I_0}\right)$$

- d_{part} encompasses MFP of light
- Resin refractive index (n_0) = 1.46
  - n_{Al2O3} = 1.786 – n_0 = Δ_n = 0.326
  - n_{AlN} = 2.110 – n_0 = Δ_n = 0.650
  - ~ 4 times higher intensity for AlN at same D_{cure}

C = constant (e.g. light wavelength)
D_{cure} = depth of cure
d_{part} = particle diameter
I = light intensity
I_0 = light intensity to cure resin
n_0 = resin refractive index
Δ_n = difference between refractive index of n_0 and ceramic
ϕ = volumetric particle loading

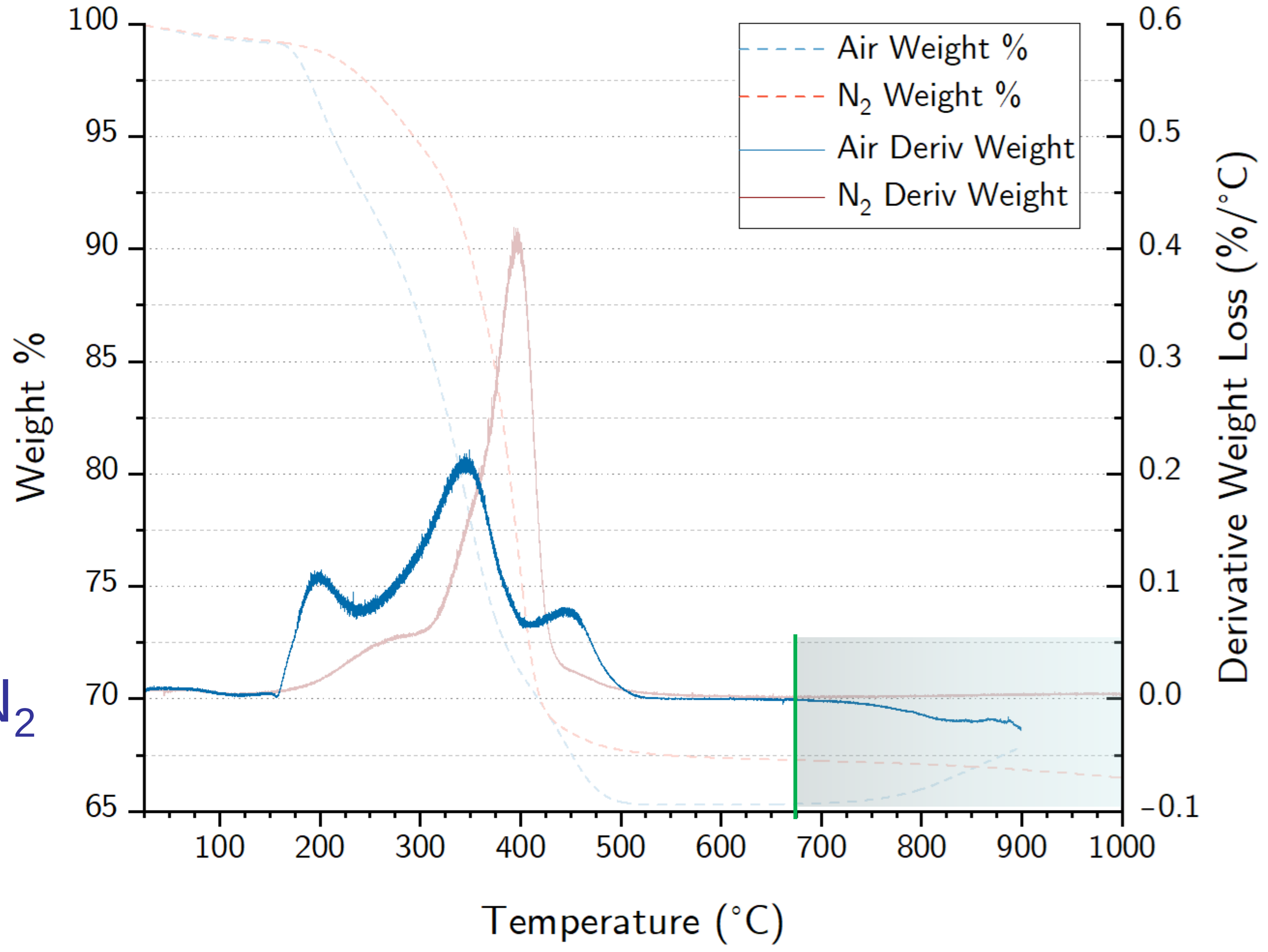
Slurry Characterization

- Rheometry testing confirms adherence to printer specifications
  - Shear thinning
    - Doctor blade, peristaltic pump
- Increasing volumetric solid loading (Φ) → shear thickening, decreased D_{cure}
- Decreasing Φ → low final part density, increased D_{cure}



Thermogravimetric Analysis

- TGA run in N_2 and air
- Informs debinding curve
- Notable mass gain in air nearing 700C
  - Oxynitride formation from 550-900C
  - Oxidation > 900C
- Initial processing in air, switch to N_2 atmosphere for future work



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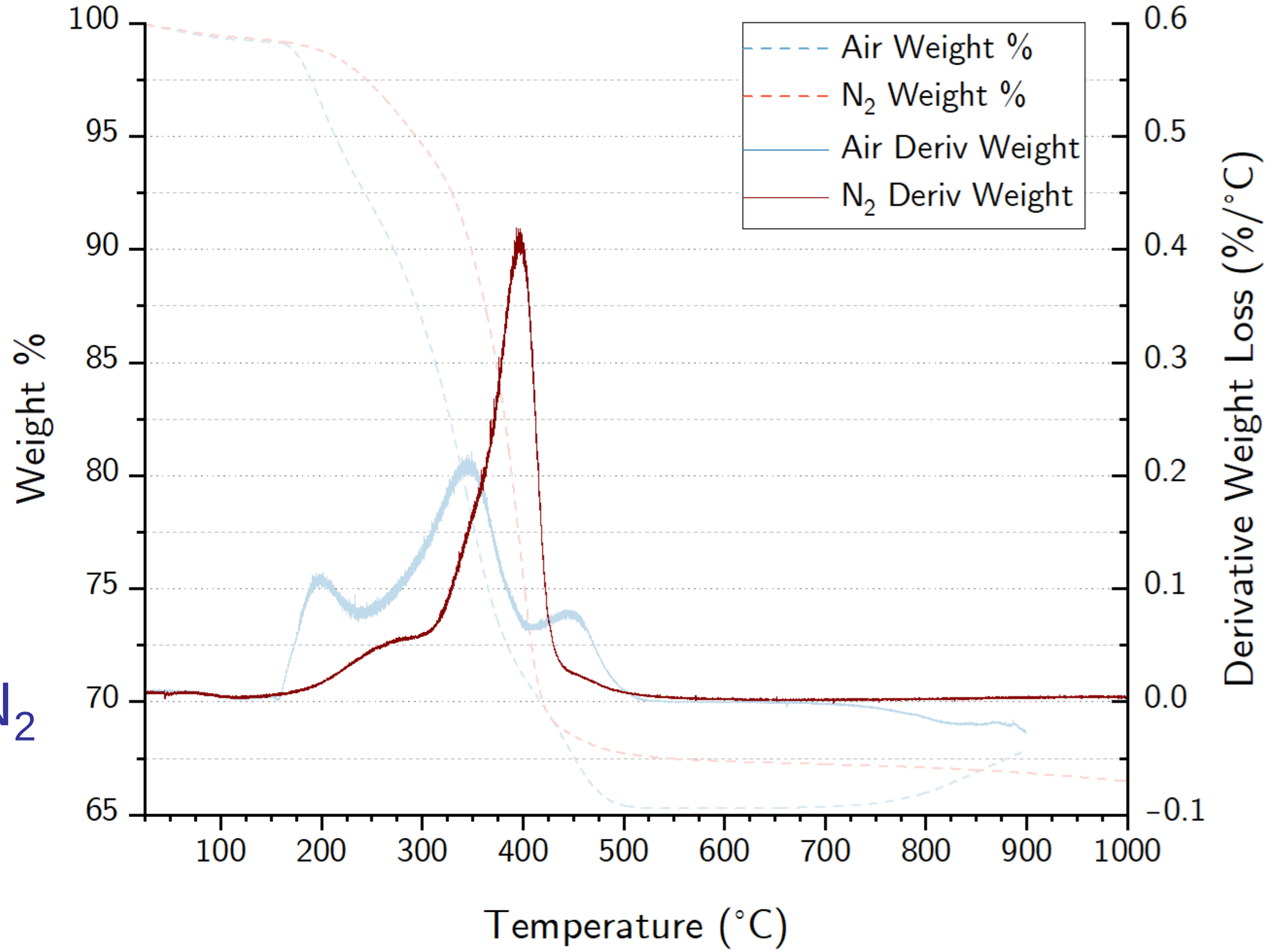
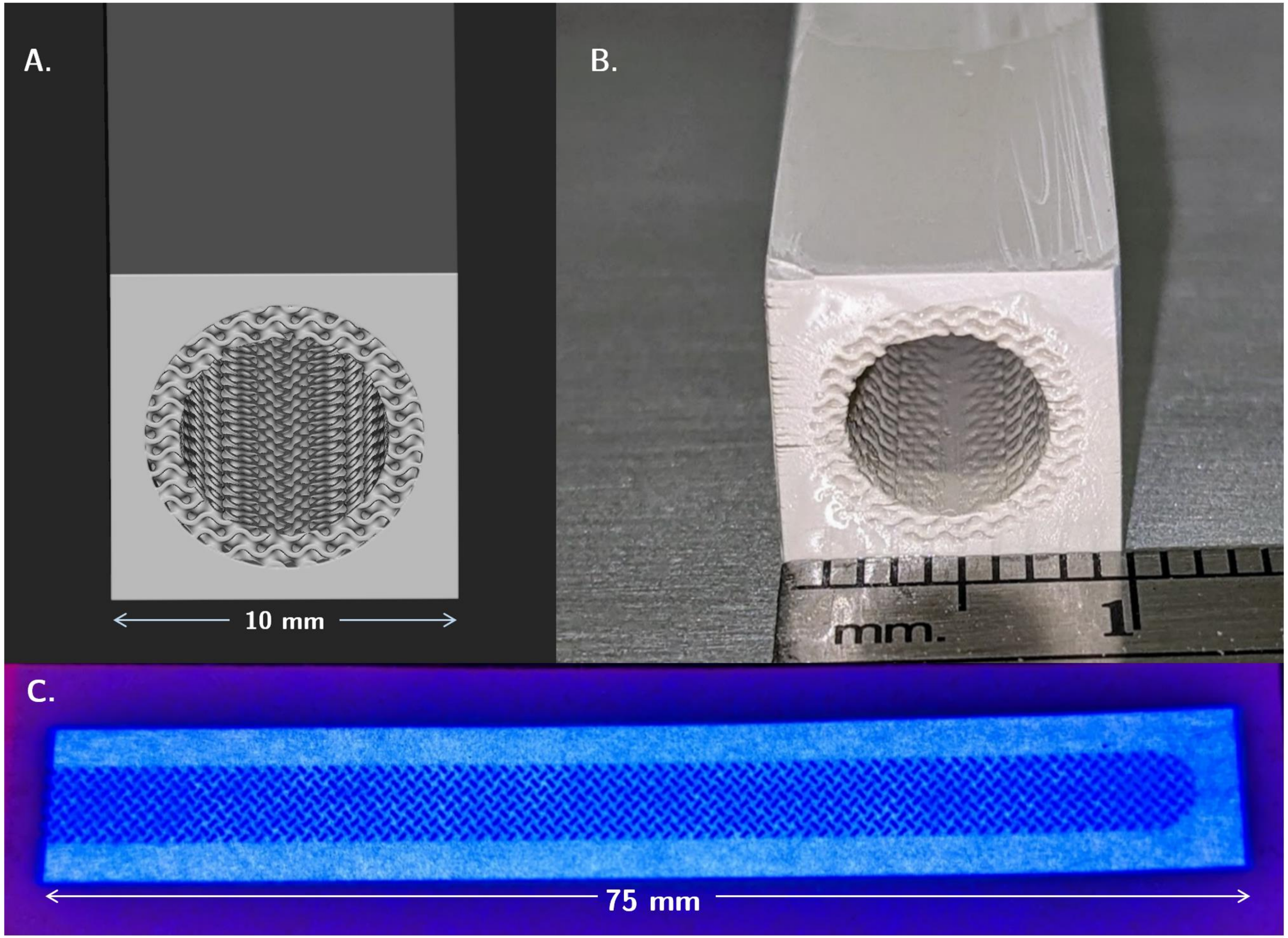


Printing Results

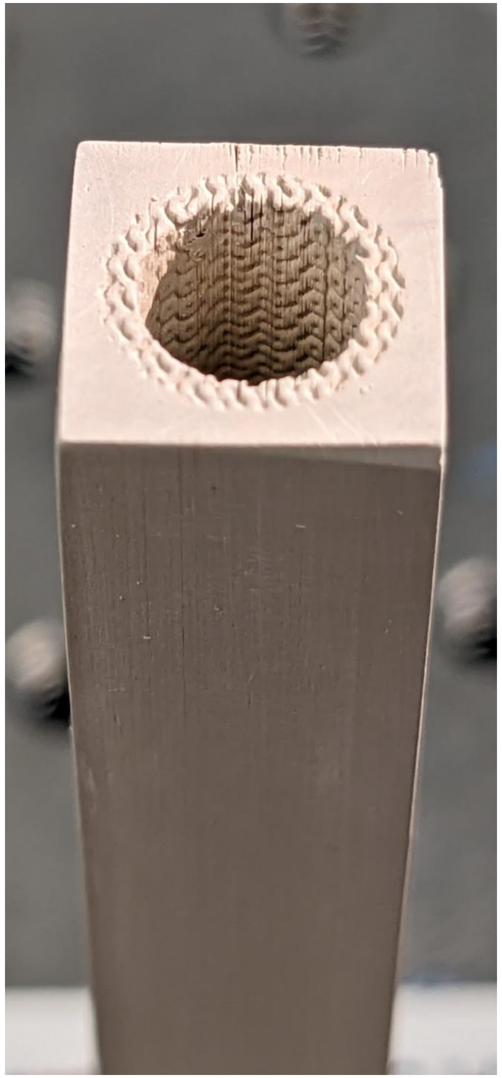
- Initial prints show good interlayer adhesion
  - Some overdevelopment, loss of fine features compared to CAD model in Fig. A
  - Fig. C shows as-exposed layer

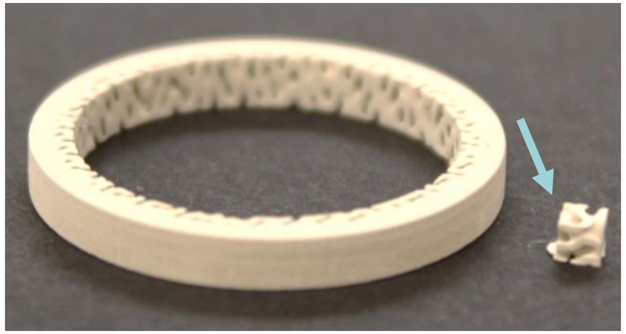


Printing Results

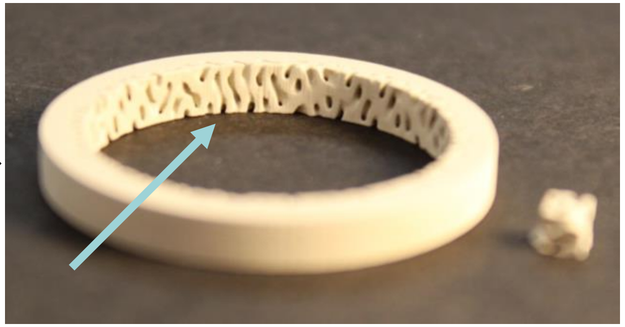
- Initial prints show good interlayer adhesion
  - Some overdevelopment, loss of fine features

- Tailored print settings decrease overall light dose
  - ~586 mJ/cm^2 → 414 mJ/cm^2
    - 36 µm D_{cure} → 34 µm D_{cure}
    - Ideal: 40 µm D_{cure}
  - Fine features preserved





Small features well defined



Wick structure not overexposed

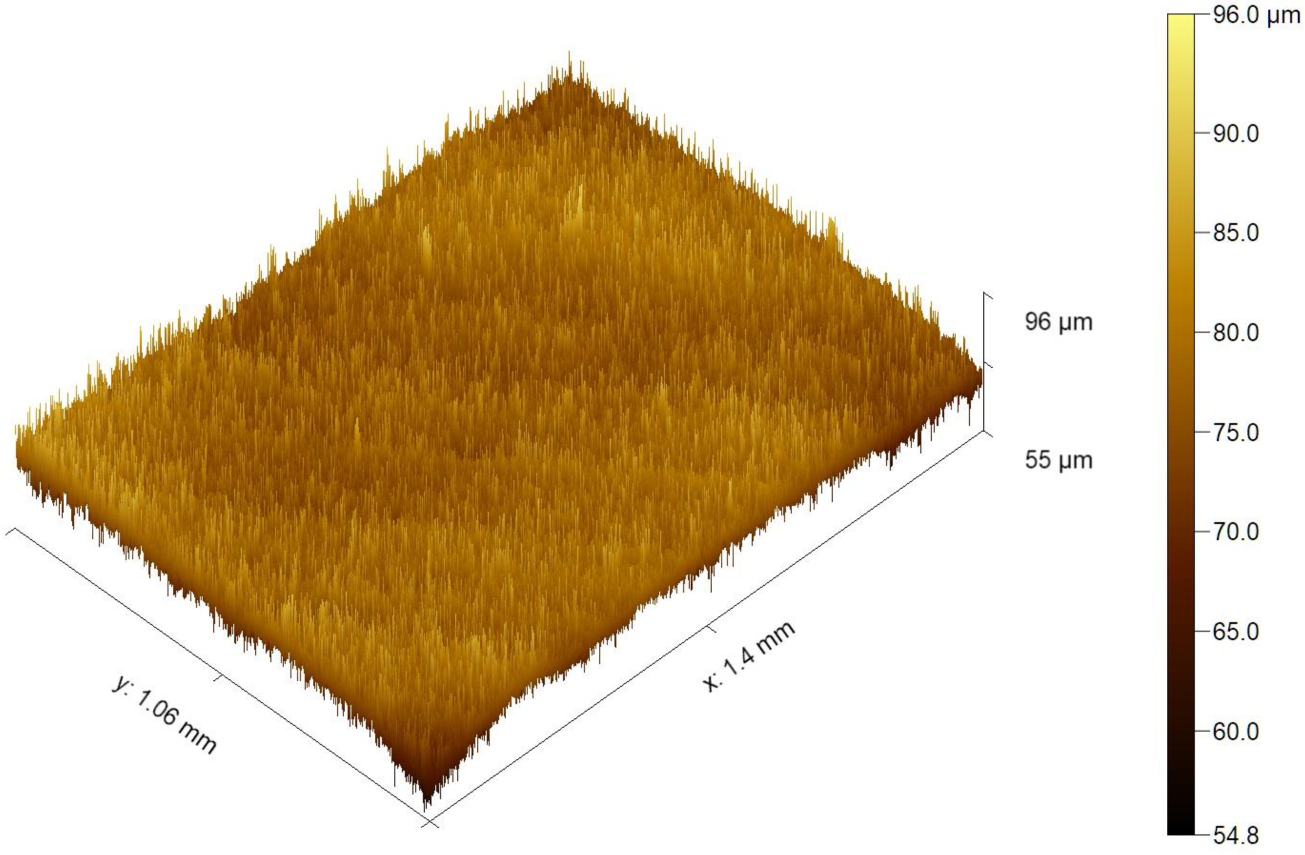
Clean layer lines

Sintering Results

- ASTM C1161 flexural bending beams sintered after air debind
  - 45 x 4 x 3 mm
- Two part sintering at 1600° C (3 hrs) and 1400° C (2 hrs) in N2 atmosphere
- Good part density but lower shrinkage than conventional
  - ~12% observed, 30% typical
- Oxide formation
  - YAG, YAM, and YAP phases, ambient oxidation from processing

Surface Characterization

- Surface roughness of as-printed alumina ceramics measured via optical profilometry
  - Not AlN, but considered representative
- AlN emissivity ~0.87 in literature
  - Testing forthcoming on properly sintered samples
- High roughness along layer lines (from radiation perspective)
  - $S_q \approx 2.07\ \mu m > 0.780\text{-}1.400\ \mu m\ (\lambda_{IR})$
- Emissivity characterization planned for as-printed



Surface Characterization

- Surface roughness of as-printed ceramics measured via optical profilometry (alumina test print)
- High roughness along layer lines (from radiation perspective)
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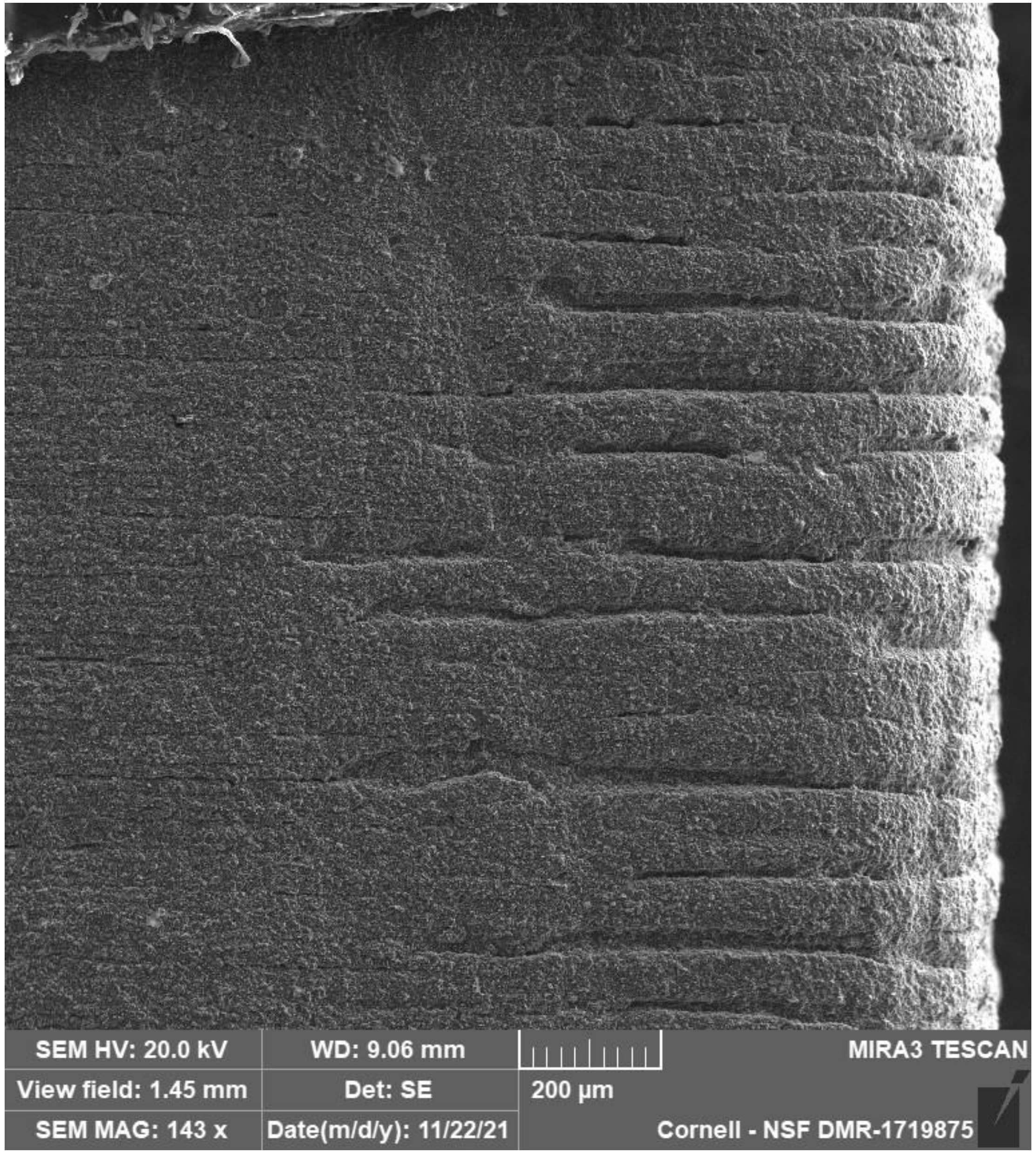

Future Work

- Working fluid compatibility testing
  - $AlBr_3$, $AlCl_3$, $FeCl_3$, I_2, Dowtherm A

- Wick rate of rise testing

- Improved debind & sintering programs

- Scale heat pipe testing in atmosphere & vacuum

Conclusions

- Demonstration of AlN printing
- TGA performed, curves in air and N_2 developed
- Initial sintering conducted
  - Requires increased N_2 flow & pressure
- High resolution parts demonstrate fine feature
- Material compatibility testing currently ongoing

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