Literature Review: Additive Manufacturing of Technical Ceramics

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Mechanical and Aerospace Engineering

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Why Technical Ceramics?

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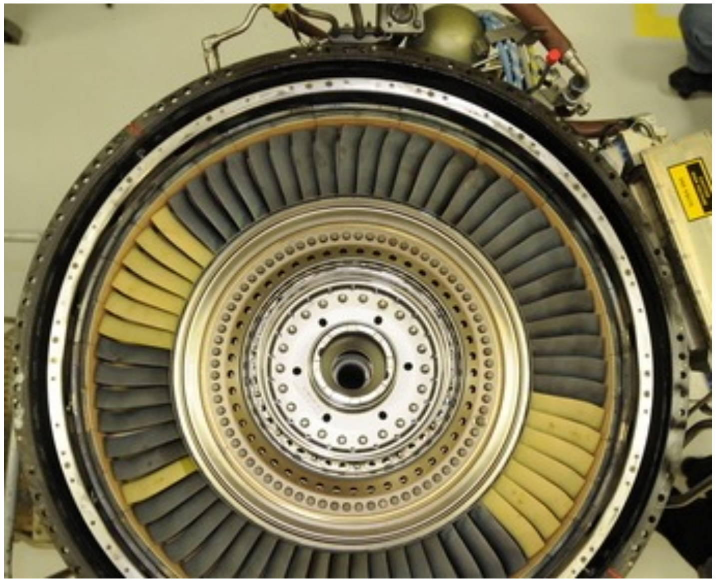
- Exceptional wear, corrosion, and temperature resistance
- Biocompatibility, electrical, optical
- Applications for aerospace, automotive, defense, and biomedical



geaviation.com

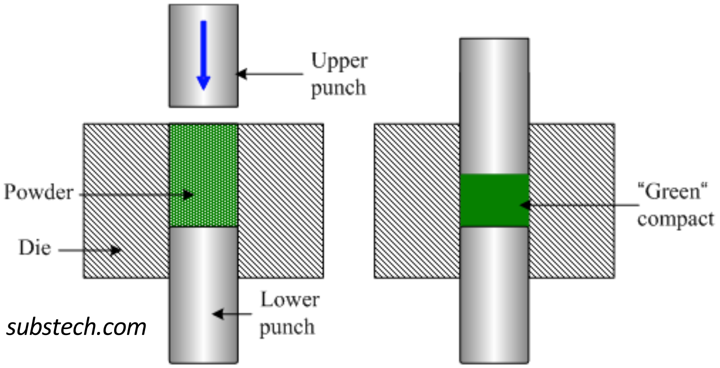
coorstek.com

coorstek.com

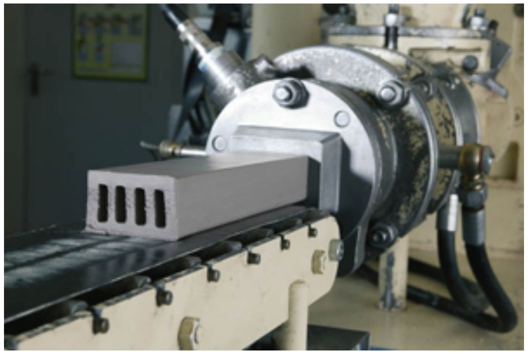
[1] C. B. Carter et at., *Springer*, 2007

Ceramic Forming Techniques

Pressing - Extrusion - Slip Casting - Tape Casting - Injection Molding



substech.com



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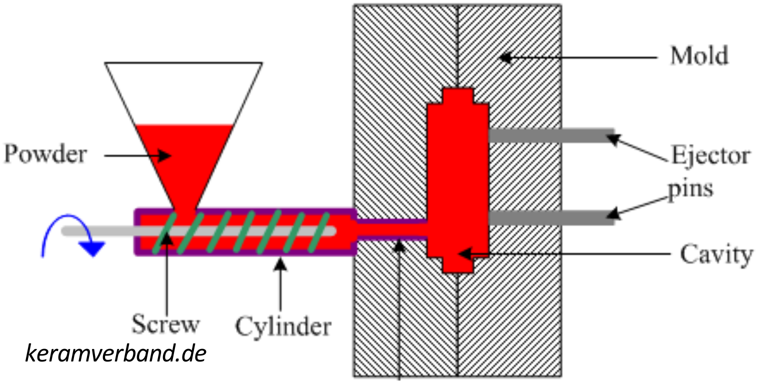


keramverband.de

Forming technology limits geometry

- Often constrained to 2D
- Limited internal features
- No controlled compositional variation

Post-Forming Techniques difficult

- 80% part cost [2]
- Time consuming
- Low tool life

[1] C. B. Carter et al., *Springer*, 2007
[2] N. Travitzky et al., *Adv. Eng. Mater.*, 2014

Additive Manufacturing (AM)

Rapid prototyping

- Quick design iteration, testing

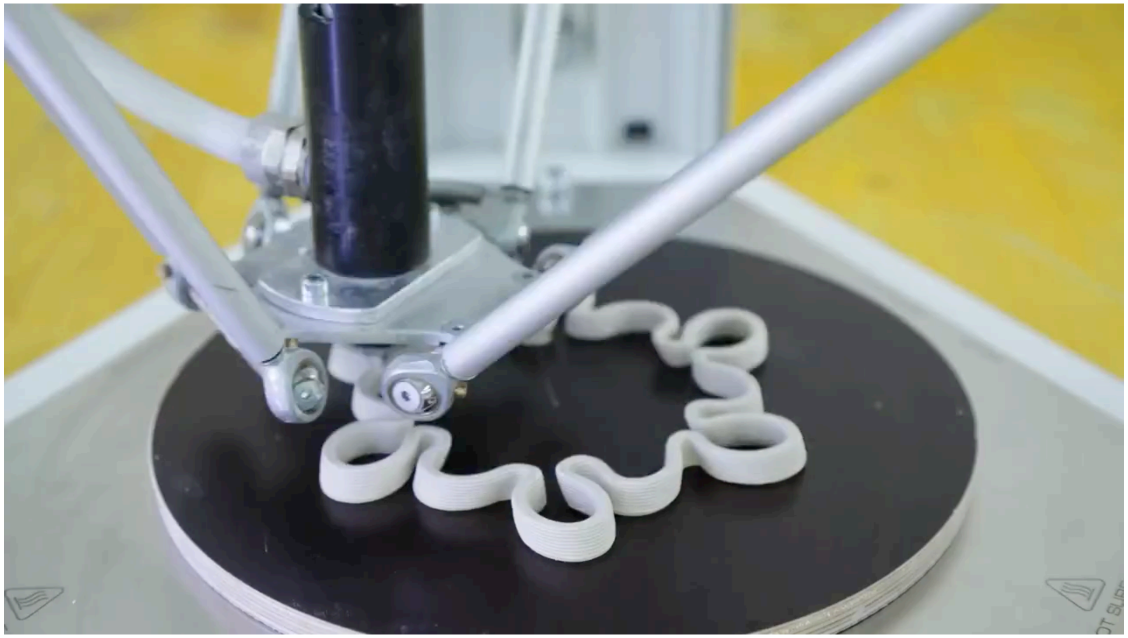
Material Selection

- Polymers, metals, and ceramics

Complexity is free

- Design at multiple length scales in 3D

3dsystems.com

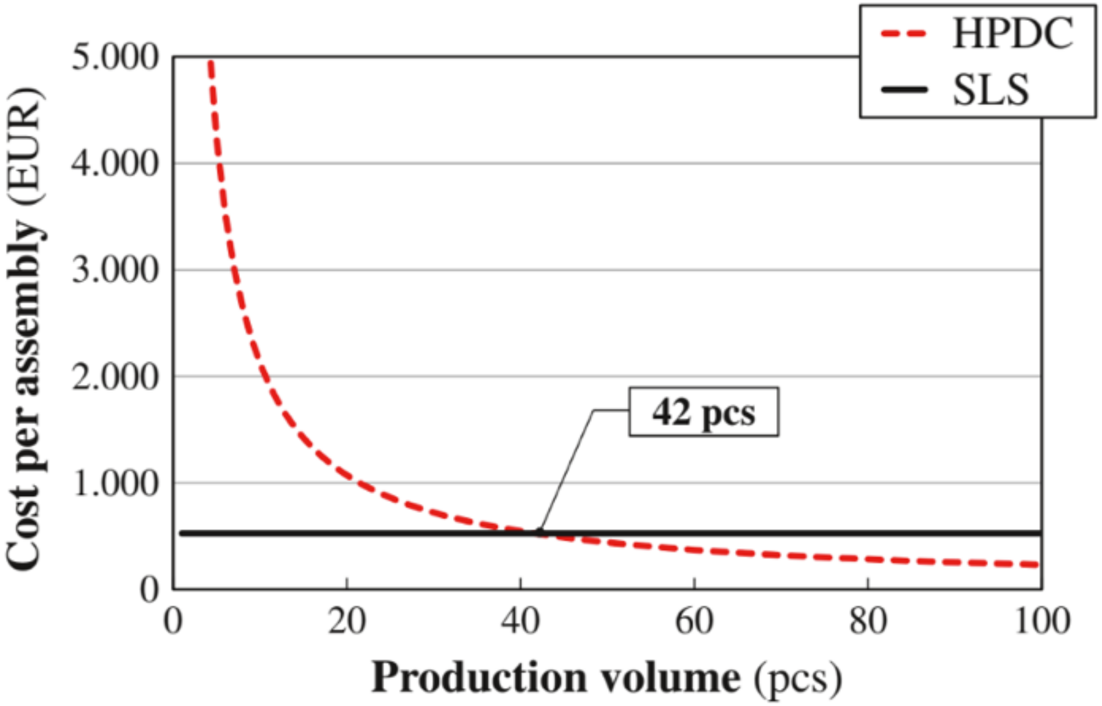


3dwasp.com

[3] B. Gibson et al., *Springer*, 2010

Is AM Viable?

- AM suitable for customized features, low-volume production, and increased geometric complexity.
- Trade-off between resolution and scalability, due to the large increases in build time at lower layer heights
- Material selection is still quite limited
- Part properties are anisotropic due to directionality of layers and weaker interlayer bonding.
- Currently, very little standardization exists which leads to poor quality control

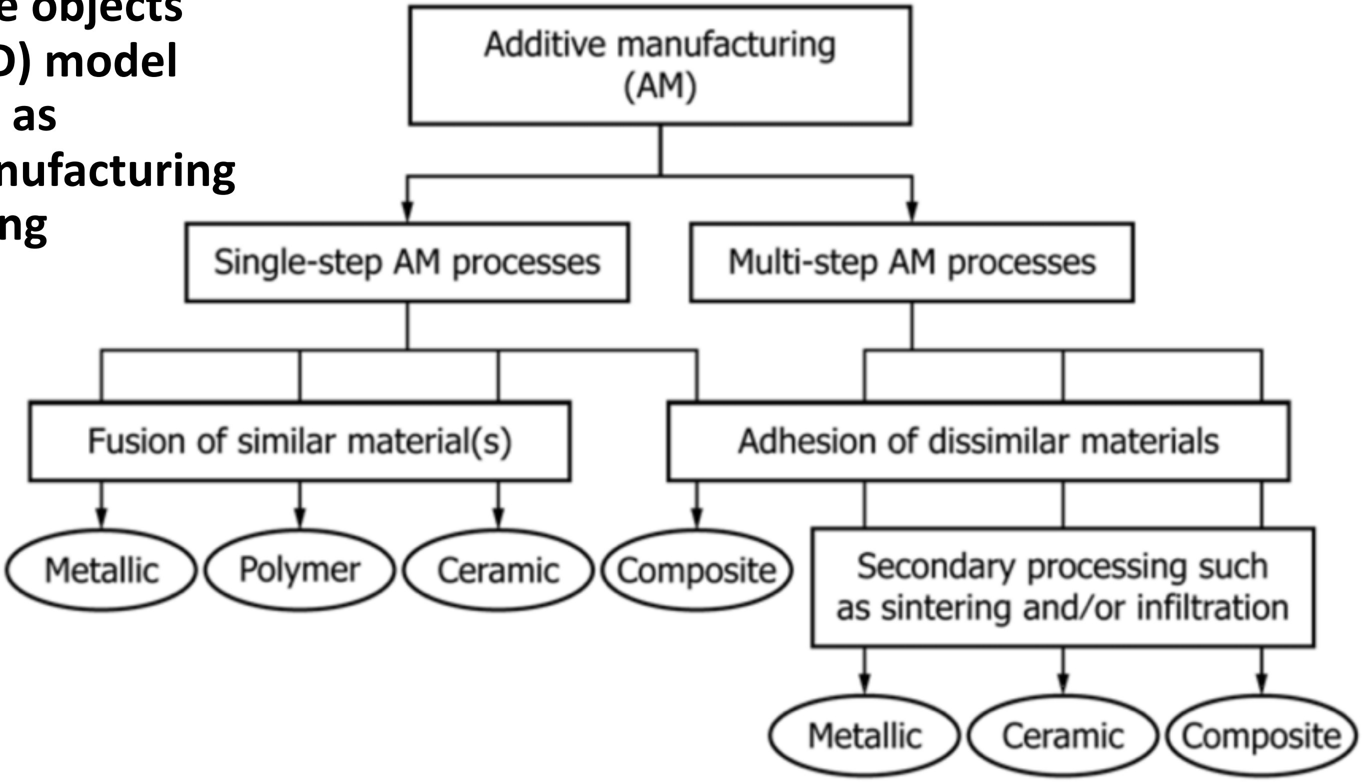


Cost analysis versus part number for selective laser sintering (SLS) vs high-pressure die casting (HPDC) for aluminum parts [5]

[4] W. Gao et al., *CAD Comput. Aided Des.*, 2015
[5] E. Atzeni et al., *Int. J. Adv. Manuf. Technol.*, 2012

ISO/ASTM Standard

- Additive manufacturing is the “**process of joining materials to make objects from three-dimensional (3D) model data, usually layer by layer, as opposed to subtractive manufacturing and formative manufacturing methodologies**”



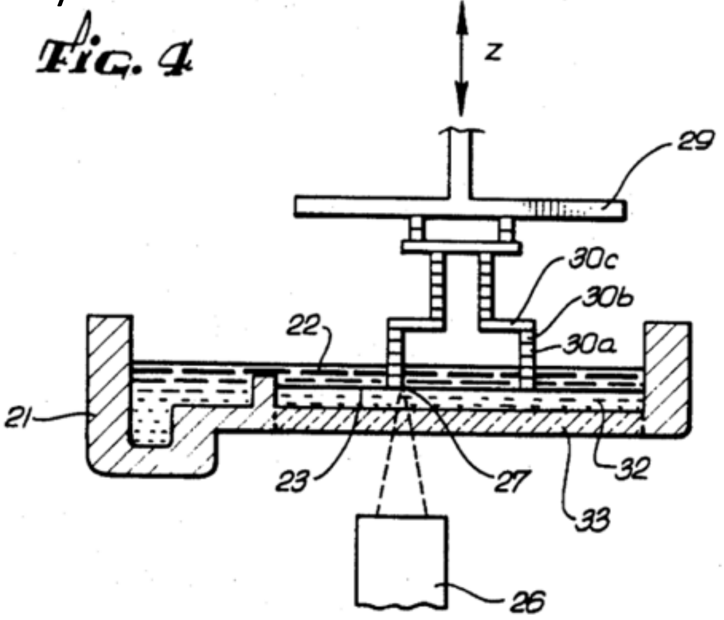
AM processes are single- or multi-step, meaning there may be post-processing required following some techniques [6]

[6] ISO/ASTM 52900, *ASTM B. Stand.*, 2015

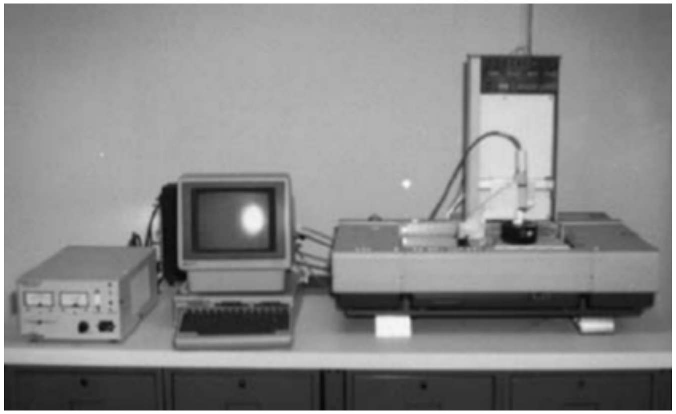
AM Inception

- Charles Hull patented first commercial AM technology in 1984
  - Concerned the use of an “apparatus for production of three-dimensional parts by stereolithography” [8]
- (SLA) means StereoLithography Apparatus
- The STL file format, an abbreviation of stereolithography, was developed by Dr. Hull’s company 3D Systems for use by their SLA machines

Fig. 4



C. W. Hull, *U.S. Patent Office*, 1984



B. Gibson et al., *Springer*, 2010

[3] B. Gibson et al., *Springer*, 2010
[7] T. Gornet et al., *Wohlers Report*, 2014
[8] C. W. Hull, *U.S. Patent Office*, 1984

Major Patents

1984

Stereolithography (SLA)

- Charles Hull
- 3D Systems

1986

Selective Laser Sintering (SLS)

- C. Deckard, J. Beaman, J. Darrah
- DMT (acquired by 3D Systems)

1989

Fused Deposition Modeling (FDM)

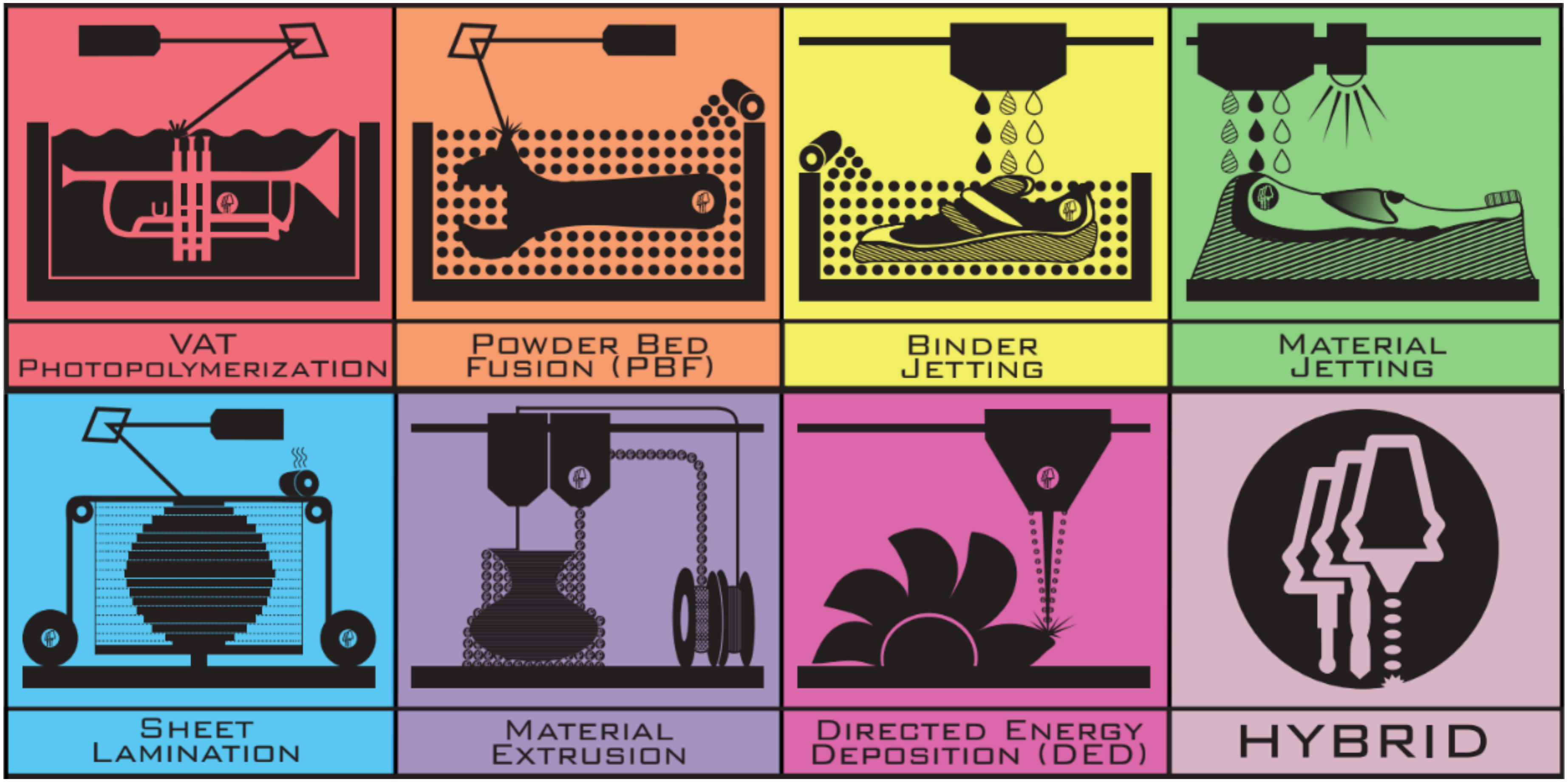
- Scott Crump
- Stratasys

3D Printing (3DP)

- E. Sachs, J. Haggerty, M. Cima, P. Williams
- MIT licensed to several companies

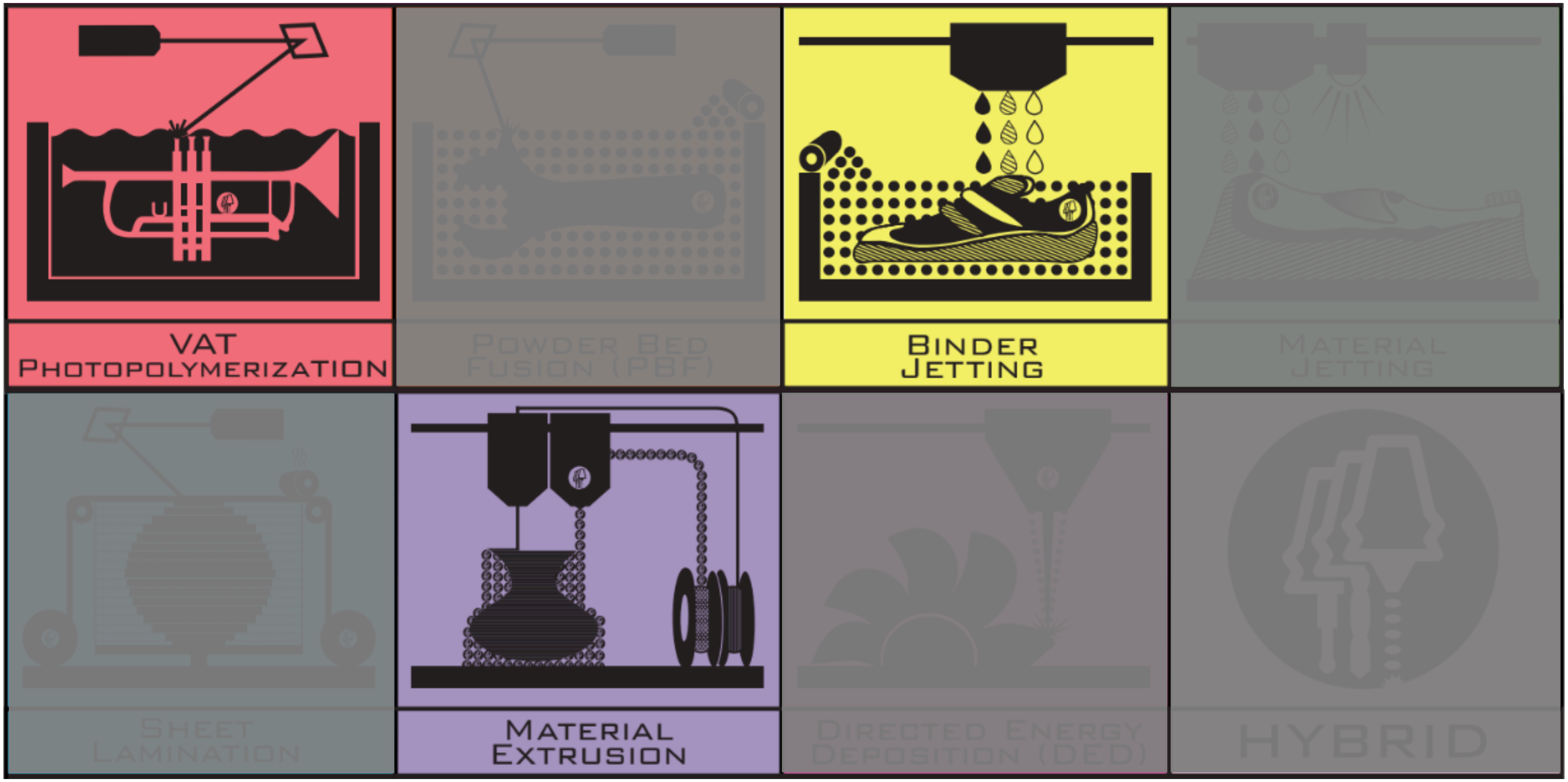
[8] C. W. Hull, *U.S. Patent Office*, 1984.
[9] C. R. Deckard et al., *U.S. Patent Office*, 1986.
[10] S. S. Crump, *U.S. Patent Office*, 1989.
[11] E. Sachs et al., *U.S. Patent Office*, 1989.

General AM Processes



[6] ISO/ASTM 52900, *ASTM B. Stand.*, 2015

General AM Processes



[6] ISO/ASTM 52900, *ASTM B. Stand.*, 2015

Vat Polymerization

Commonly called:

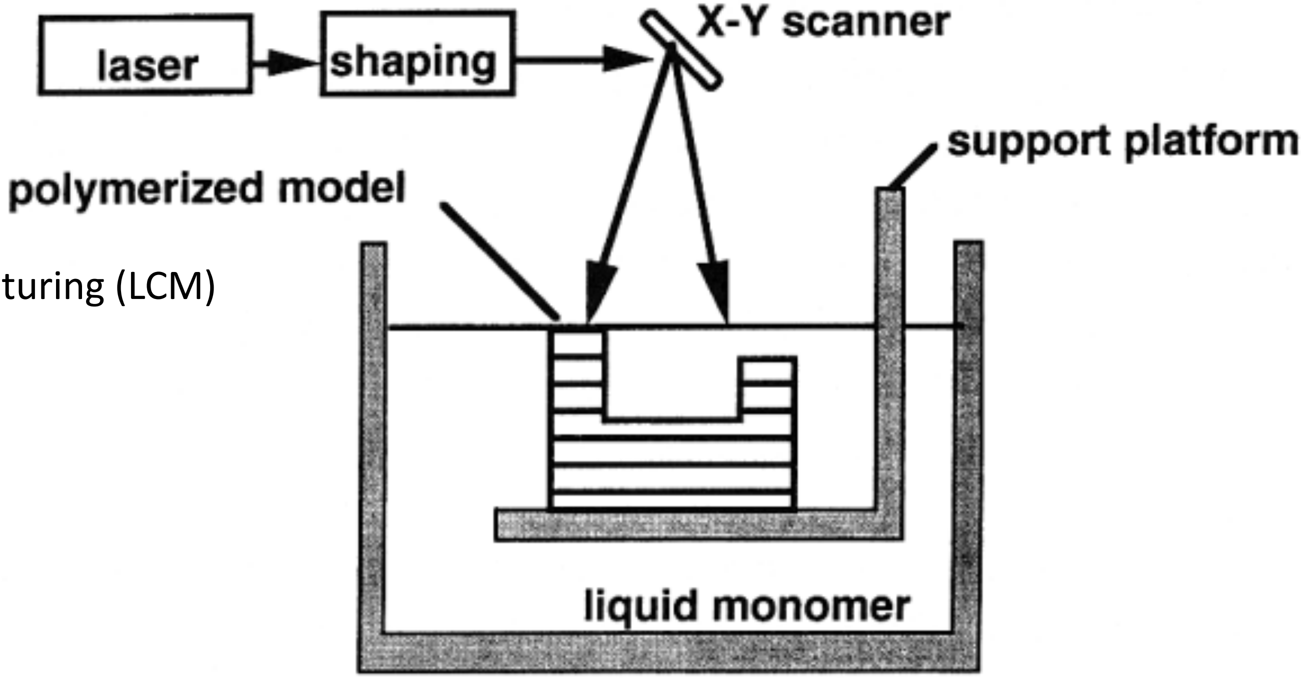
- Stereolithography (SLA)
- Digital Light Processing (DLP)
- Lithography-based Ceramic Manufacturing (LCM)

Strengths:

- High accuracy and complexity
- Good surface finish
- Large build volume

Material:

- UV-curable photopolymer resin
  - Load with ceramic powder



Resin is selectively cured by a laser or light projector [12]

[12] J. W. Halloran, *Annu. Rev. Mater. Res.*, 2016

Binder Jetting

Commonly called:

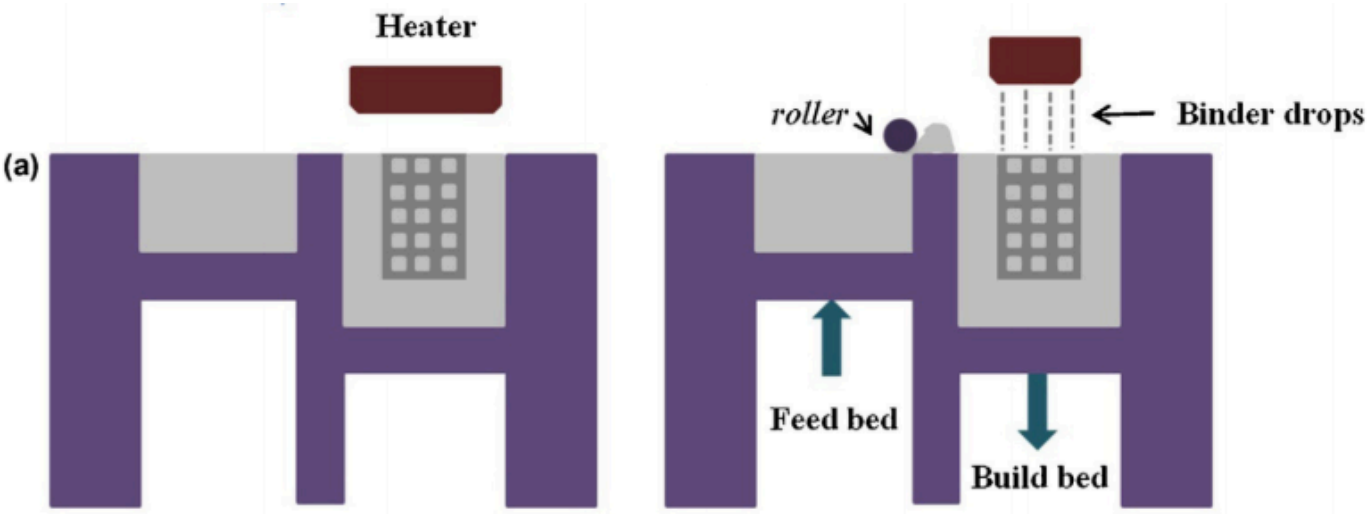
- 3D Printing (3DP)
- Voxeljet^{TM}

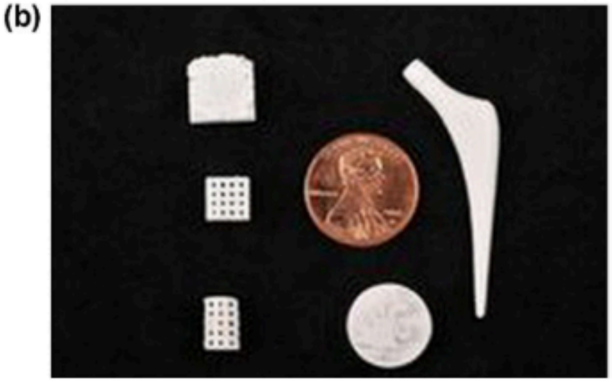
Strengths:

- High resolution
- Powder acts as support material
  - Scaffold structures
- Wide range of materials

Material:

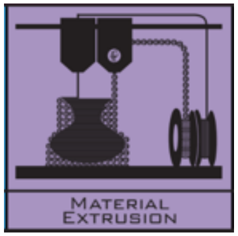
- Powdered
  - Plastic
  - Metal
  - Ceramic





Liquid binder is applied to thin layers of powder. Loose powder must be removed [13].

[13] S. Bose et al., *Mater. Today*, 2013



Material Extrusion

Commonly called:

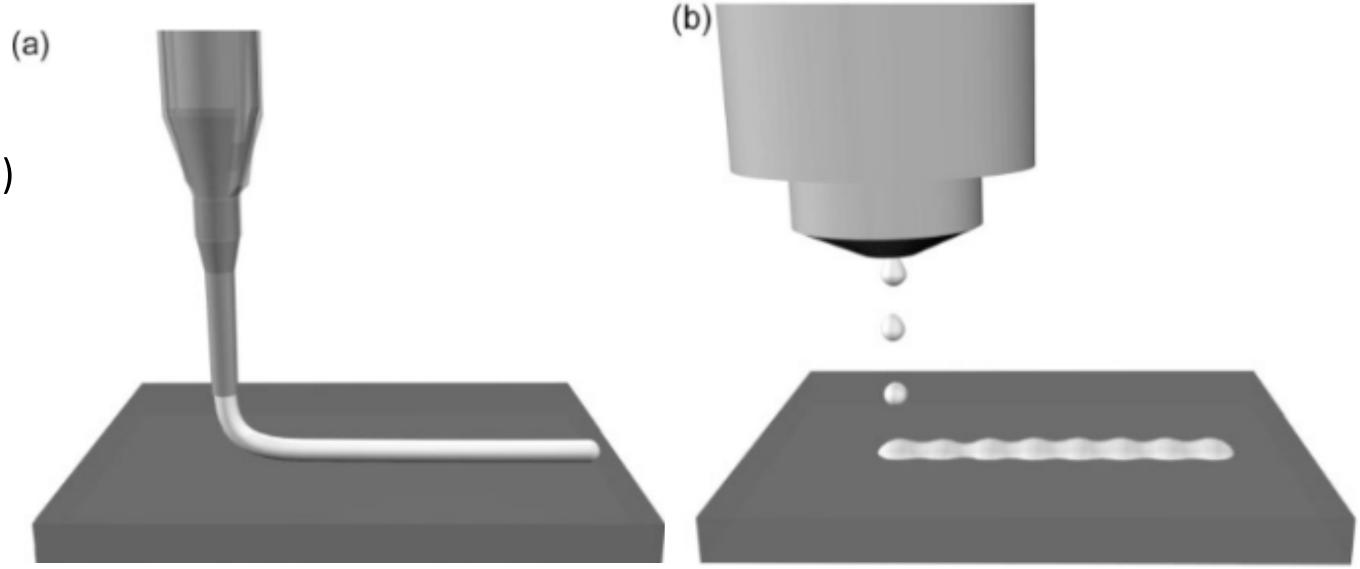
- Direct Ink Writing (DIW)
- Fused Deposition Modeling (FDM)
- Fused Filament Fabrication (FFF)

Strengths:

- Inexpensive, low-complexity
- Multi-material capability

Material:

- Thermoplastic filaments
  - Load with ceramic/metallic powder
- Pastes or slurries
  - Load with ceramic/metallic powder



Material is extruded through a nozzle in traces, layer-by-layer. Material self-supports via cooling, gelation, or other rheological effects [14].

[14] J. A. Lewis, *Adv. Funct. Mater.*, 2006

Ceramic AM Challenges

Multi-step AM processes

- Pyrolysis and Sintering

Printing Defects

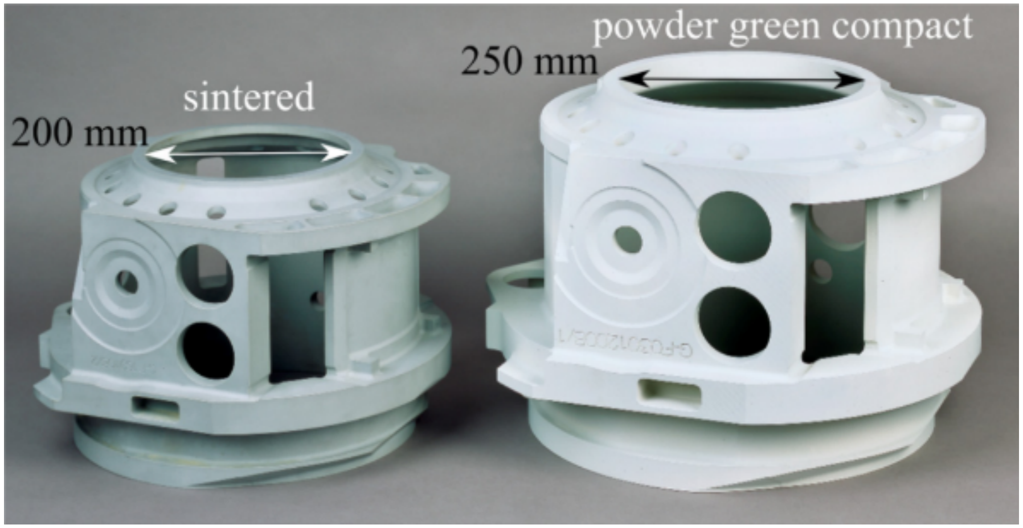
- Layer deadhesion, slumping, pores

Near-net Shape Difficult

- Optimal powder packing between 65-72%
- Post-processing causes shrinkage (>30%)

Poor Quality Control

- Large variation in properties for an identical process



A. Zocca et al., *J. Am. Ceram. Soc.*, 2015

[15] J. A. Lewis, *J. Am. Ceram. Soc.*, 2004
[16] A. Zocca et al., *J. Am. Ceram. Soc.*, 2015
[17] C. Y. Yap *et al.*, *Appl. Phys. Rev.*, 2015

Ceramic AM Case Studies

3DP for Rapid Prototyping

Emanuel Sachs at MIT (1990)

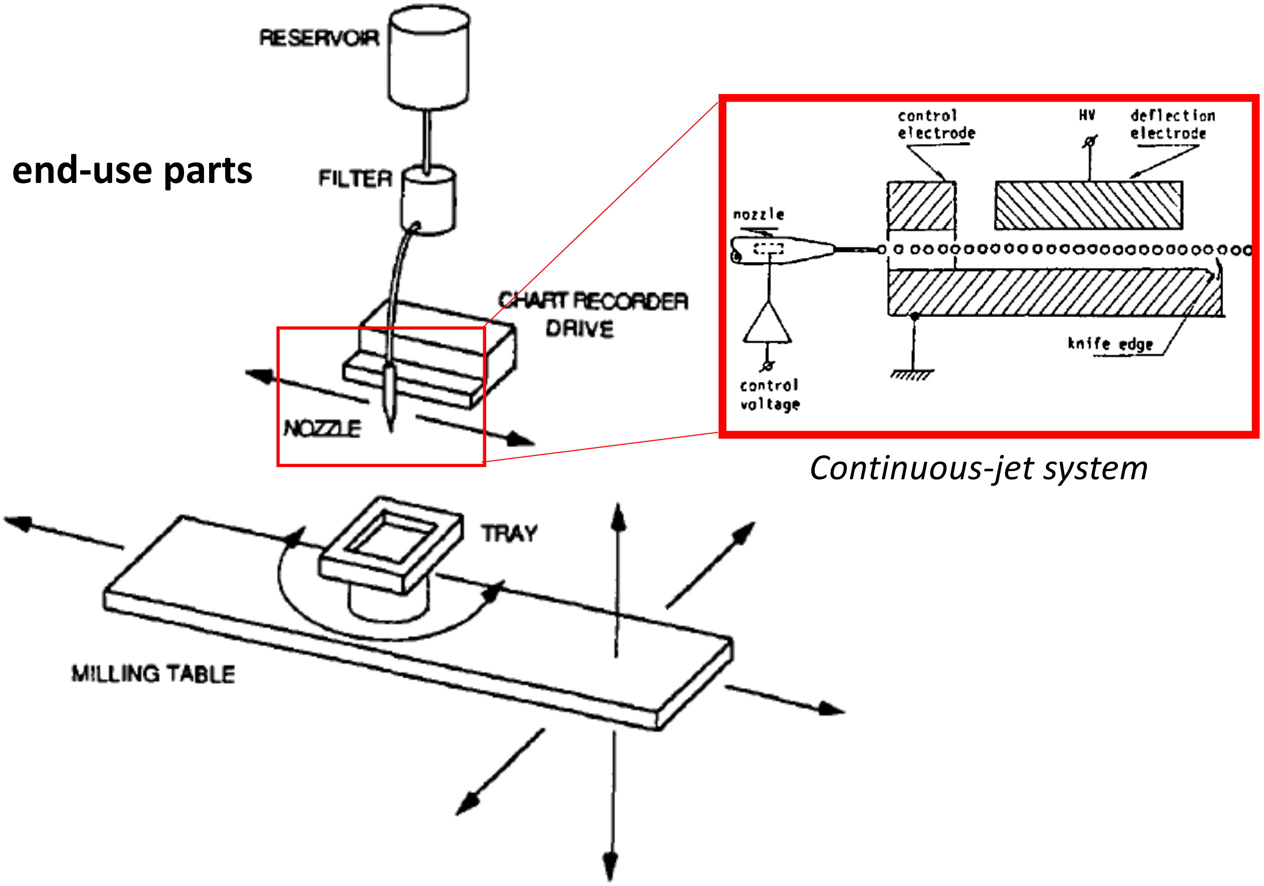
- First AM study for non-polymeric materials

Motivation - ceramic molds for cast metal end-use parts

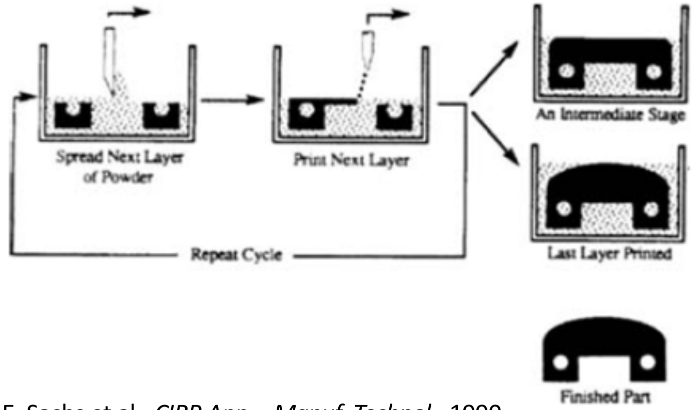
- Quicker part development
- Lower cost for unique, one-off molds
- Replace lost-wax casting
  - $5k-50k and 2-20 weeks per die

Tested systems

- Drop-on-demand
- Continuous-jet



Continuous-jet system



[18] E. Sachs et al., *CIRP Ann. - Manuf. Technol.*, 1990

3DP for Rapid Prototyping

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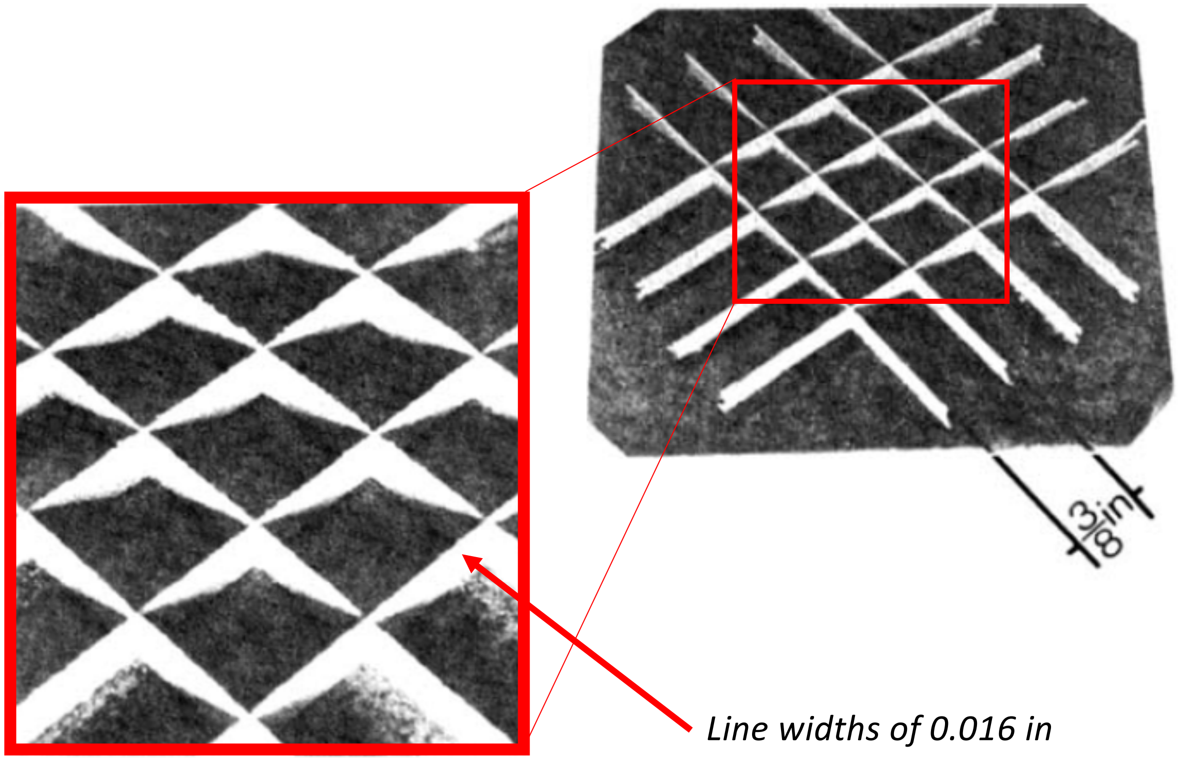
Technical challenges

- Powder flowability (50 micron)
- Binder properties
- Post-processing

Results

- Excellent dimensional accuracy
  - 10-100 micron powder
  - Nozzle size 0.002 in. (apparent resolution)
  - No detectable shrinkage from curing
- Printing Rate
  - Powder spreading: dry (0.1-1 s) and wet (0.1-10 s)
  - Continuous jet (20 m/s) and DoD (0.1 m/s)
  - Binder setting (<1 s)
  - Build-up rate of 0.01-0.27 m/hr for 25 micron layers



Alumina powder bound with silica

Line widths of 0.016 in
Tolerance of +/- 0.0005 in
40 layers

[18] E. Sachs et al., *CIRP Ann. - Manuf. Technol.*, 1990

3DP without Sintering

Uwe Gbureck et al. (2007)

- Modern 3DP study for bone scaffolds

Motivation – bioresorbable bone scaffolds

- Room temperature processing
- Thermally instable materials usable
- Sufficient mechanical properties

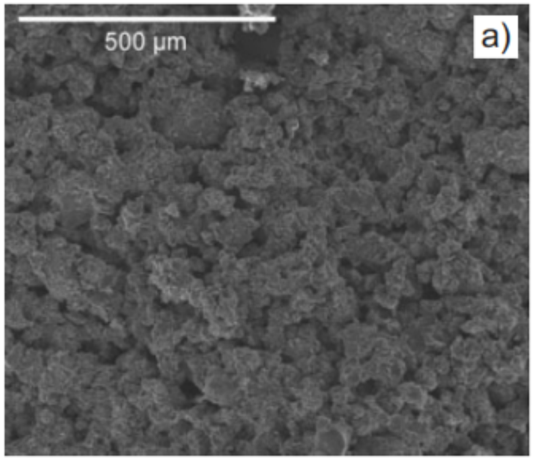
Technical challenges

- Low temperature densification workflow

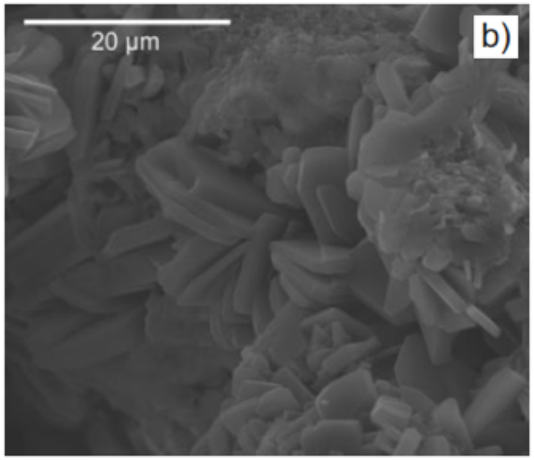
Tricalcium Phosphate (TCP) powder bound with phosphoric acid

Workflow

1. Tricalcium Phosphate (TCP) powder + Phosphoric acid solution -> forms a brushite matrix with unreacted TCP.
2. Strengthen parts by washing three times in phosphoric acid solution for one minute
3. Brushite converted to monetite by hydrothermal reaction



Brushite/TCP matrix from 20% phosphoric acid solution



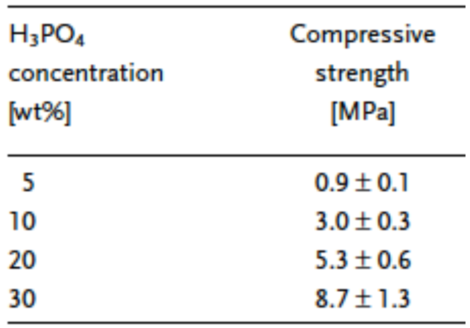
Brushite/TCP matrix (a) with three subsequent phosphoric acid washes

[19] Gbureck U et al., *Advanced Functional Materials*, 2007

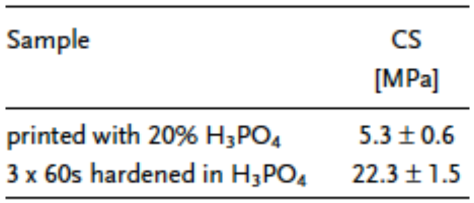
3DP without Sintering

Results

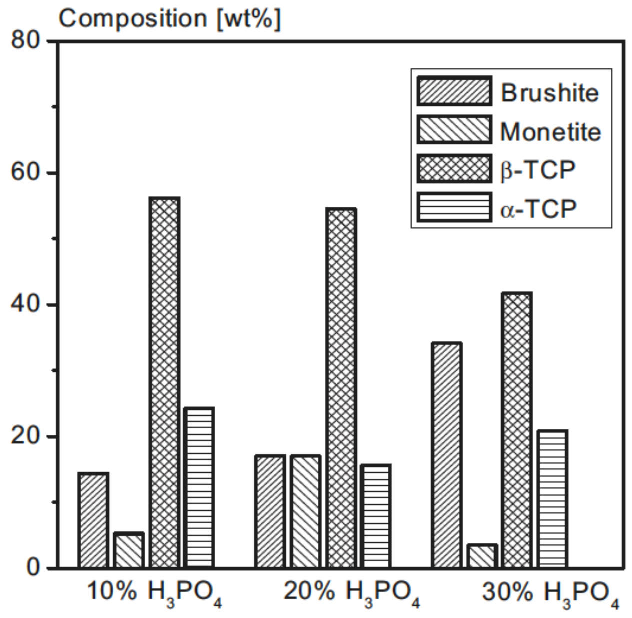
- Compressive strength increases with phosphoric acid concentration
- Compressive strength increases after washing
- Compressive strength decreases after hydrothermal reaction, but biosorption increases

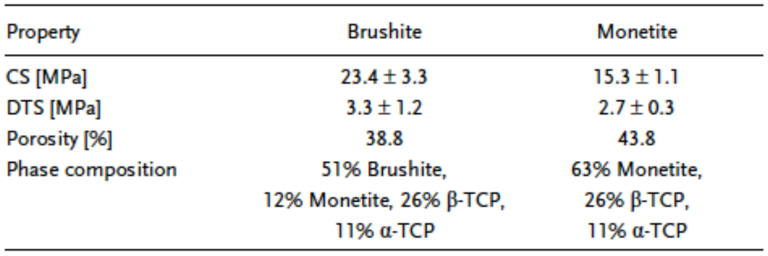


H_3PO_4 concentration [wt%]	Compressive strength [MPa]
5	0.9 ± 0.1
10	3.0 ± 0.3
20	5.3 ± 0.6
30	8.7 ± 1.3

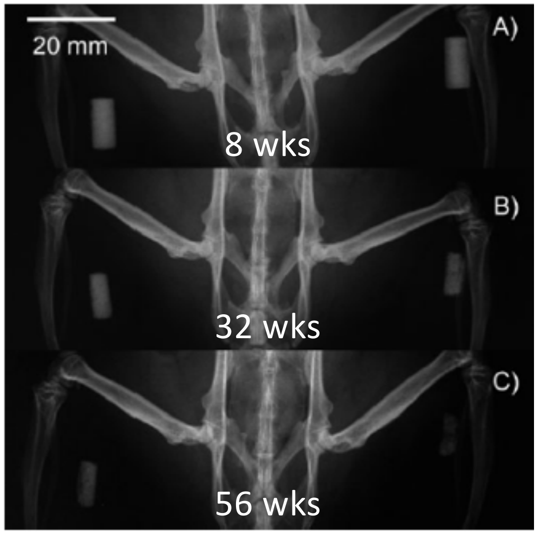


Sample	CS [MPa]
printed with 20% H_3PO_4	5.3 ± 0.6
3 x 60s hardened in H_3PO_4	22.3 ± 1.5

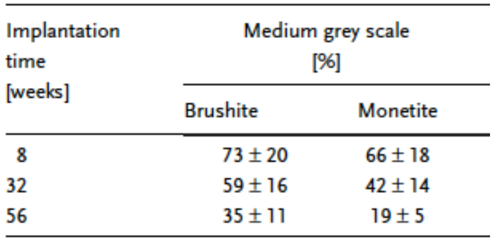




Property	Brushite	Monetite
CS [MPa]	23.4 ± 3.3	15.3 ± 1.1
DTS [MPa]	3.3 ± 1.2	2.7 ± 0.3
Porosity [%]	38.8	43.8
Phase composition	51% Brushite, 12% Monetite, 26% β-TCP, 11% α-TCP	63% Monetite, 26% β-TCP, 11% α-TCP



Printed Brushite (left) and Monetite (right) scaffolds as intramuscular implants



Implantation time [weeks]	Medium grey scale [%]	
	Brushite	Monetite
8	73 ± 20	66 ± 18
32	59 ± 16	42 ± 14
56	35 ± 11	19 ± 5

[19] Gbureck U et al., *Advanced Functional Materials*, 2007

Vat Polymerization First Steps

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Michelle Griffith and John Halloran at U. Mich. (1996)

- First Vat Polymerization study for ceramic materials

Motivation - casting molds and end-use parts

- Quicker development and lower cost for unique parts
- Study efficacy for end-use parts
- Explore process limitations to better understand part design

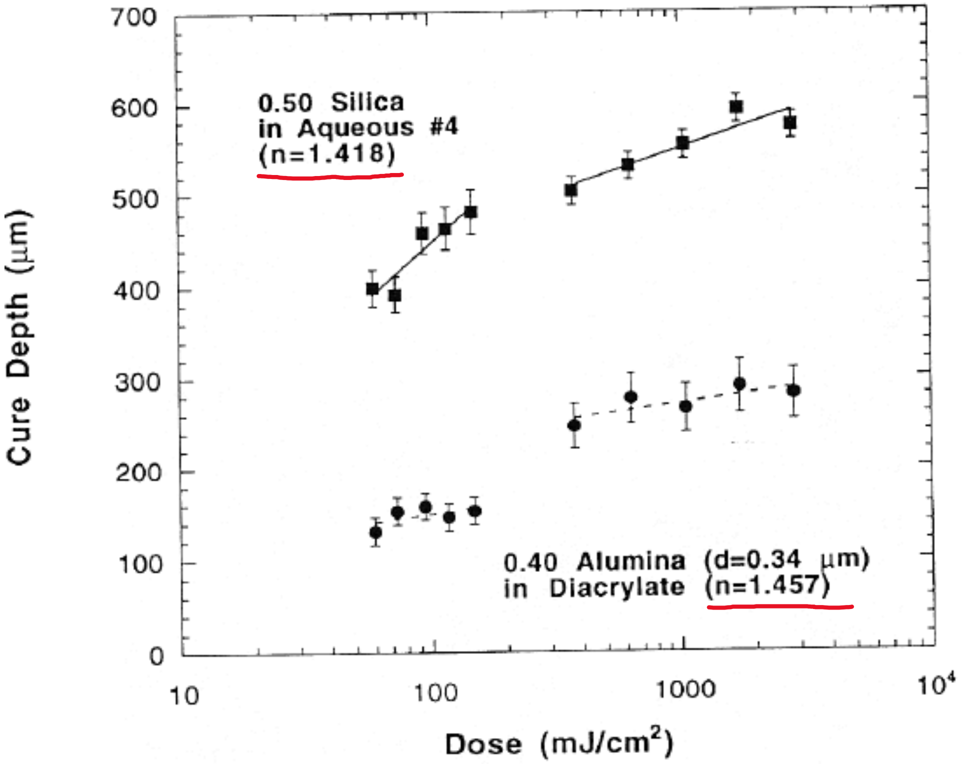
Technical challenges

- Resin solids loading, rheology
- Powder refractive index, cure depth

Results

- Silica, Alumina: 100^{s} um (40 vol.%)
- Silicon Nitride: 21 um (10 vol.%) and 10 um (20 vol.%)
- Cure depth is hypothesized to be scattering limited

Varying powder type

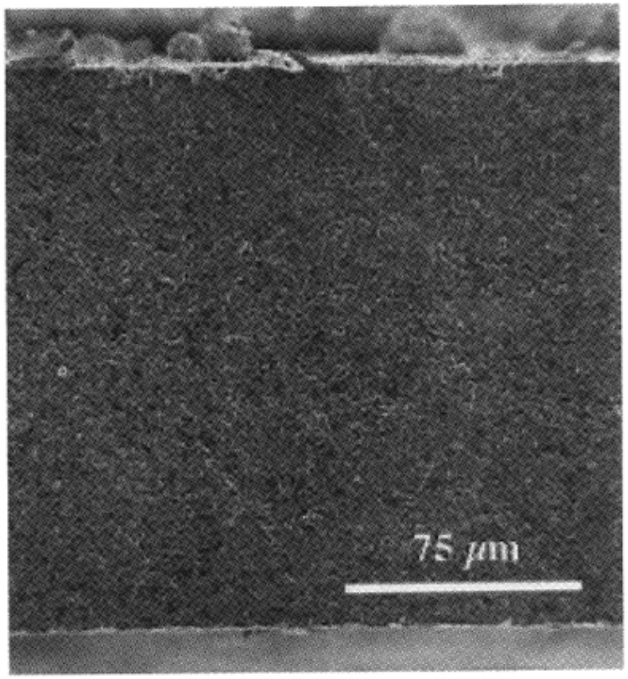


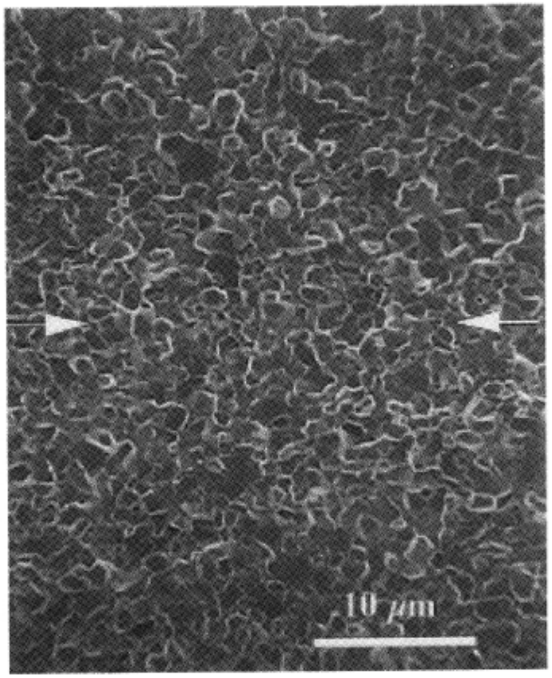
[20] M. L. Griffith and J. W. Halloran, *Journal of the American Ceramic Society*, 1996

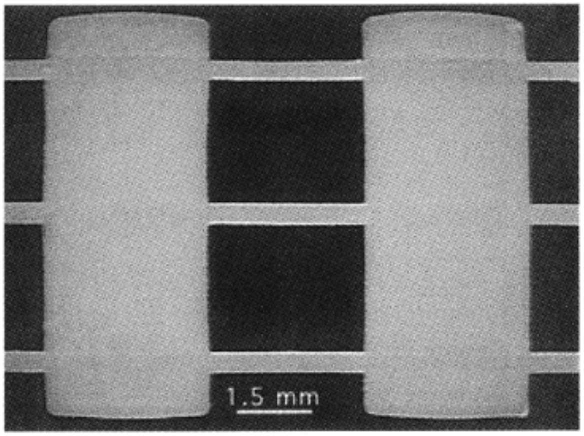
Vat Polymerization First Steps

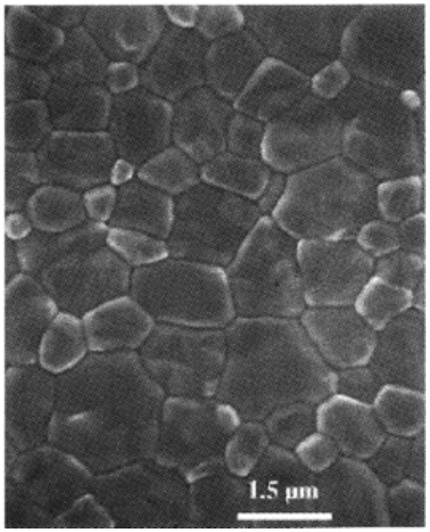
Results

- Fracture surfaces free from printing artifacts
- Layers are not distinguishable
- Geometric stability in both thick and thin sections
- Dense alumina parts (1550 °C)









SLA-250: 40 vol.% alumina powder in diacrylate resin

[20] M. L. Griffith and J. W. Halloran, *Journal of the American Ceramic Society*, 1996

Predicting Cure Depth

Michelle Griffith and John Halloran at U. Mich. (1997)

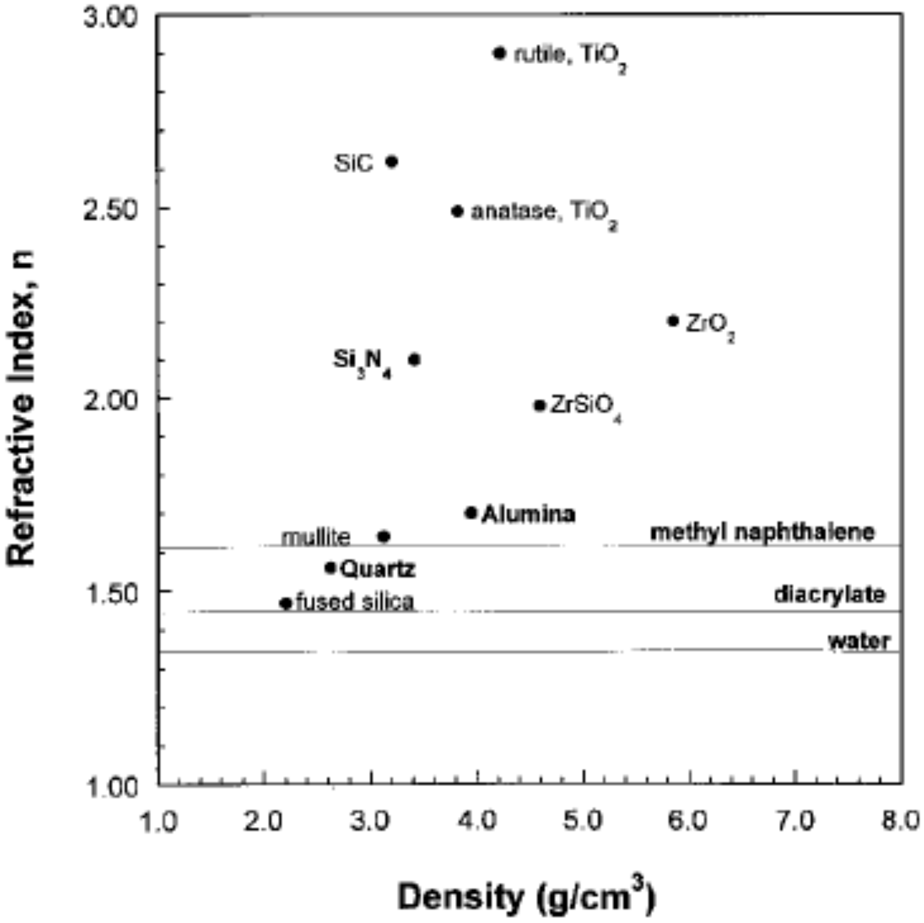
- Follow up study for ceramic vat polymerization

Motivation – predict cure depth

- More effective laser parameters

Results

- Modeled depth of cure for 40-50 vol.% slurries
- Refractive index difference main factor
- Interparticle spacing as a secondary factor
- Particle size less related than others predicted



[21] M. L. Griffith and J. W. Halloran, *Journal of Applied Physics*, 1997

Biomedical Application

Probst et al. (2010)

- Bone scaffold study using vat polymerization

Motivation – biomedical implant workflow

- Quick production of patient-specific bone scaffolds
- Determine printed cell-free-scaffold efficacy

Technical challenges

- Multi-step workflow
  - Imaging, model reconstruction, printing, implantation
- Powder refractive index, cure depth

Results

- Calvarial defect reconstruction using CT imaging
- Polycaprolactone-calcium phosphate
- Long-range growth into scaffold at 6 months

[22] Probst, F. A., et. al., *Handchir Mikrochir Plast Chir*, 2010

Dense Ceramics by Robocasting

Joseph Cesarano and Thomas Baer at Sandia (1997)

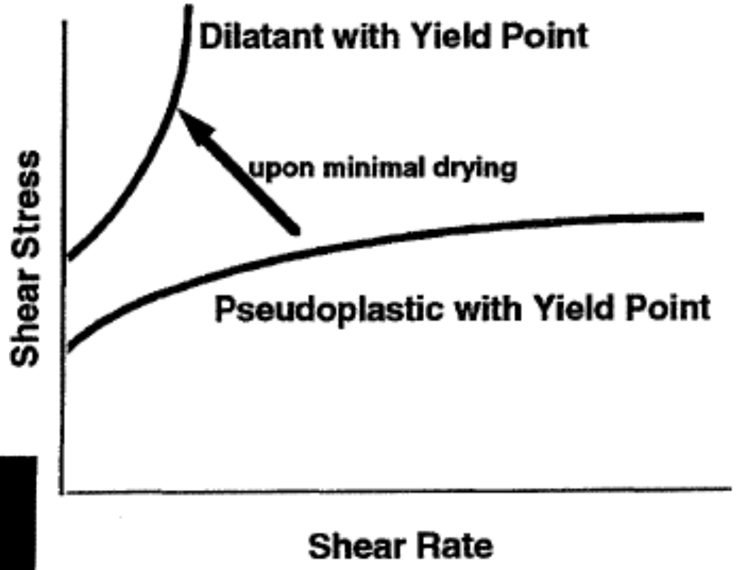
- First Material Extrusion study for ceramic materials

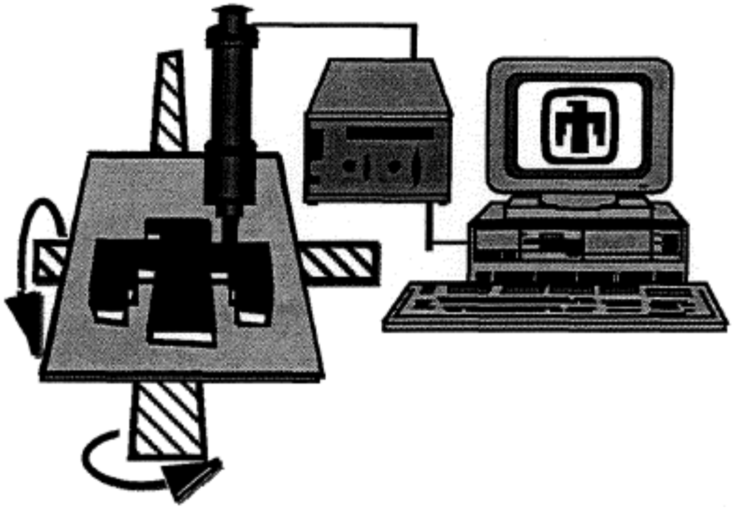
Motivation - High-solids loading

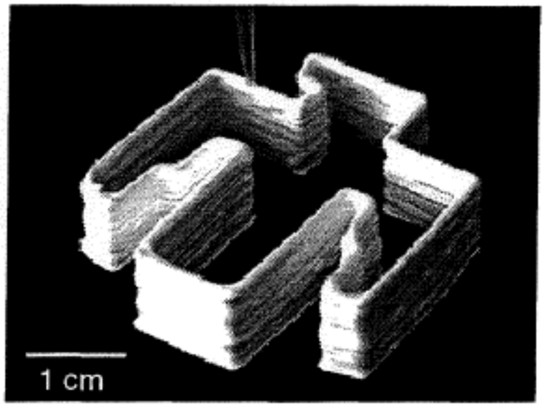
- Print slurries of 50-65 vol.% ceramic and <1 vol.% organic
- Much quicker drying and pyrolyzation process (<24 hours vs. days at a heating rate of ~0.2 °C /min)
- Create denser green bodies, and therefore denser final parts

Technical challenges

- Slurry rheology and Drying Kinetics







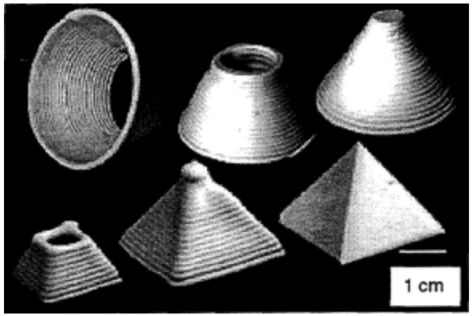
Thunderbird printed from alumina with 20 layers and sintered to 96 %TD

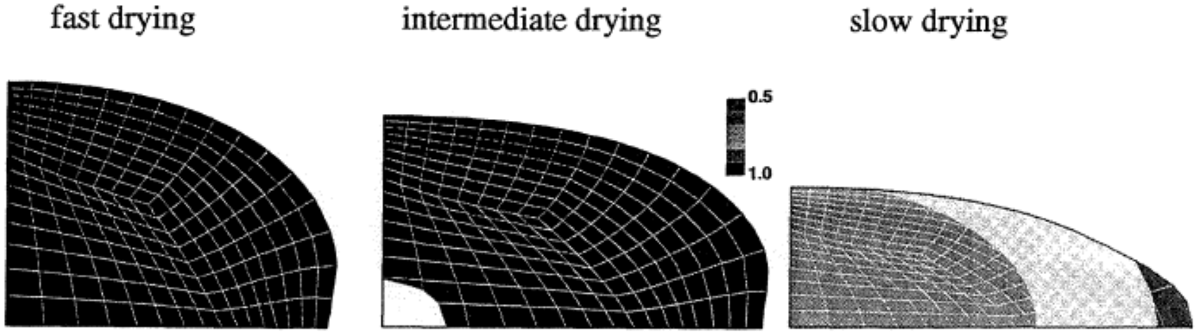
[23] Joseph Cesarano III and Thomas A. Baer, *Sandia National Laboratories*, 1997

Dense Ceramics by Robocasting

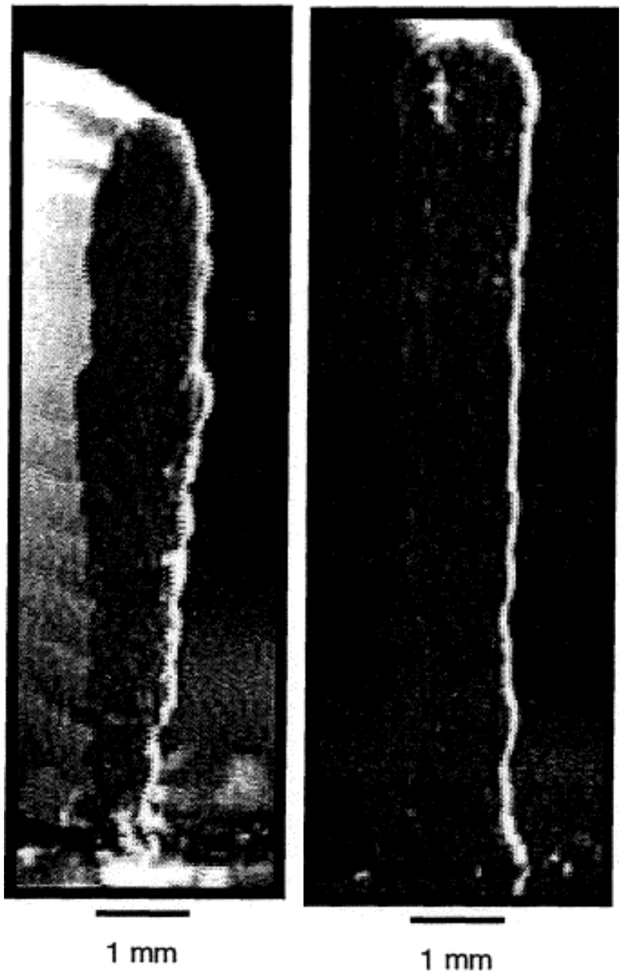
Results

- Robocasting technique for ceramics
- Yield-pseudoplastic slurries consist of:
    - Alumina
    - Darvan-821A
    - DIH_2O
- Viscosity reduces from 1e6 to 4e4 cP with shear rates increasing from 0.07 to 1.7 s^{-1}
- FEA simulation of drying kinetics to better understand how to develop slurries and how to control bead geometry





FEA results for three drying rates after 5 minutes



[23] Joseph Cesarano III and Thomas A. Baer, *Sandia National Laboratories*, 1997

Rheology in Al_2O_3 Printing

Jennifer Lewis and Joseph Cesarano (2000)

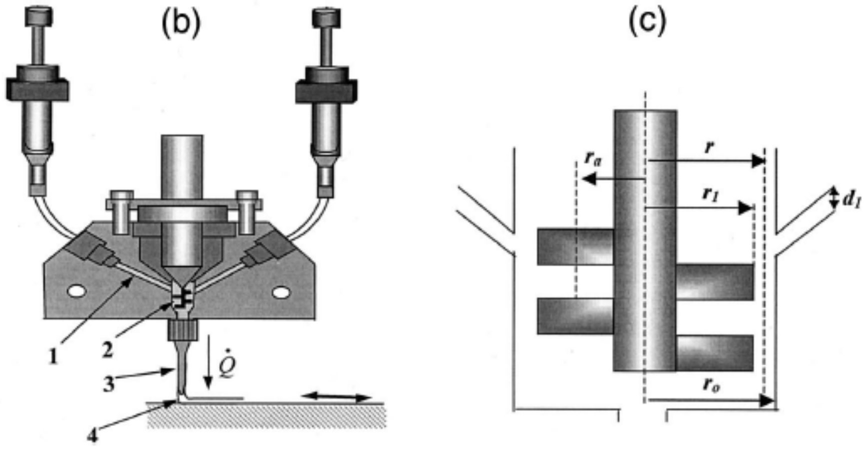
- High impact study concerning rheological effects on printability

Motivation – Rheology and Printability

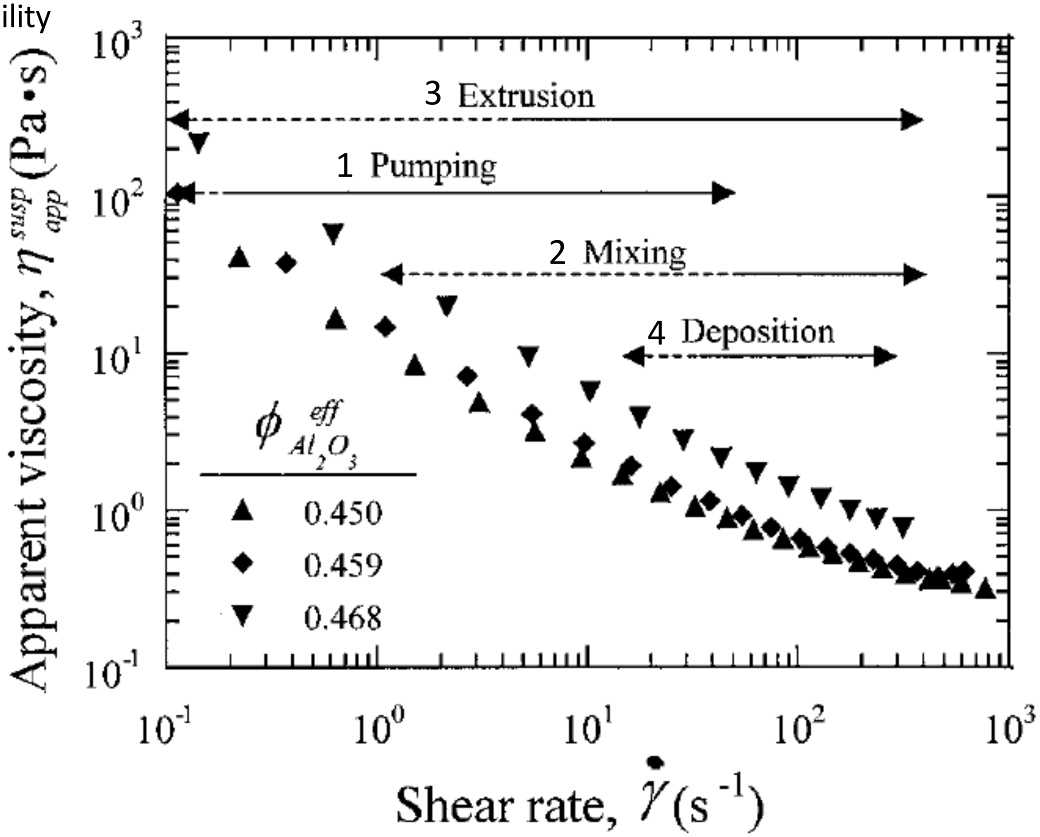
- Reduce nozzle size
- Increase shape retention
- Create defect free parts

Technical challenges

- Adjust slurry viscosity using mixing chamber
- Isolate print parameters



[24] J. Lewis et al., *J. Am. Ceram. Soc.*, 2000

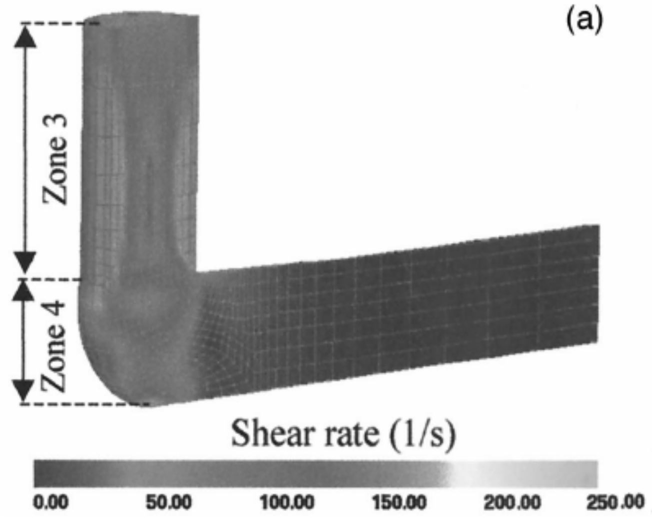


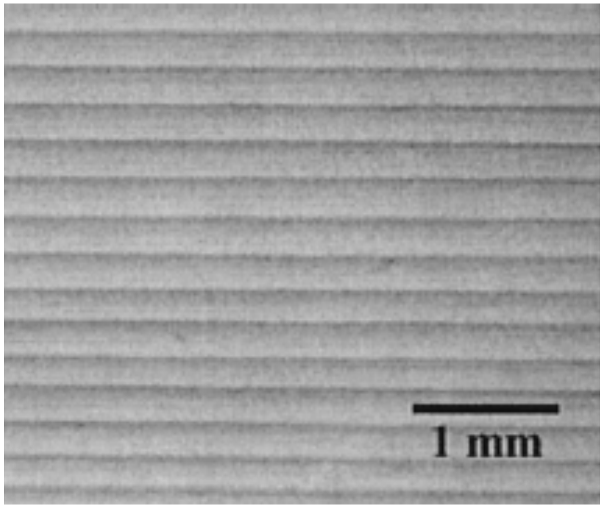
Viscosity versus shear rate for varying solids loading. Shear rate ranges are given for each segment of the printing process.

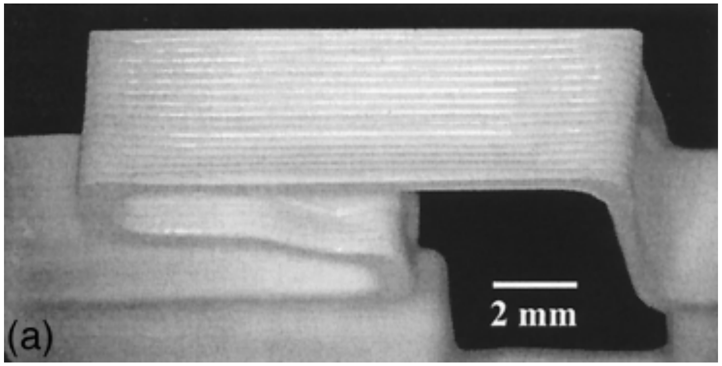
Rheology in Al_2O_3 Printing

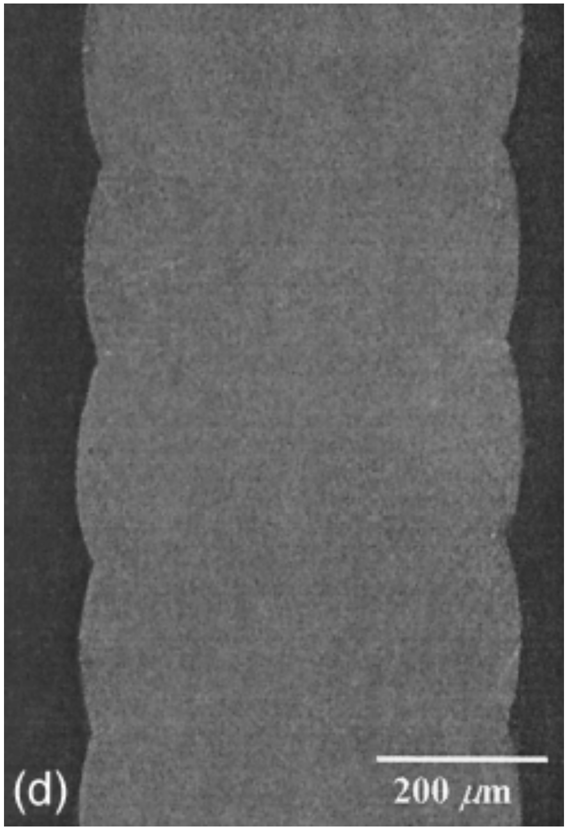
Results

- Printable nozzles 0.254–1.370 mm
- Good line, edge, and shape retention
- Defect-free parts
- FEA simulation of shear rate









[24] J. Lewis et al., *J. Am. Ceram. Soc.*, 2000

Dense Al_2O_3

Lisa Rueschhoff group at Purdue (2016)

- Inexpensive, commercially available material extrusion printer

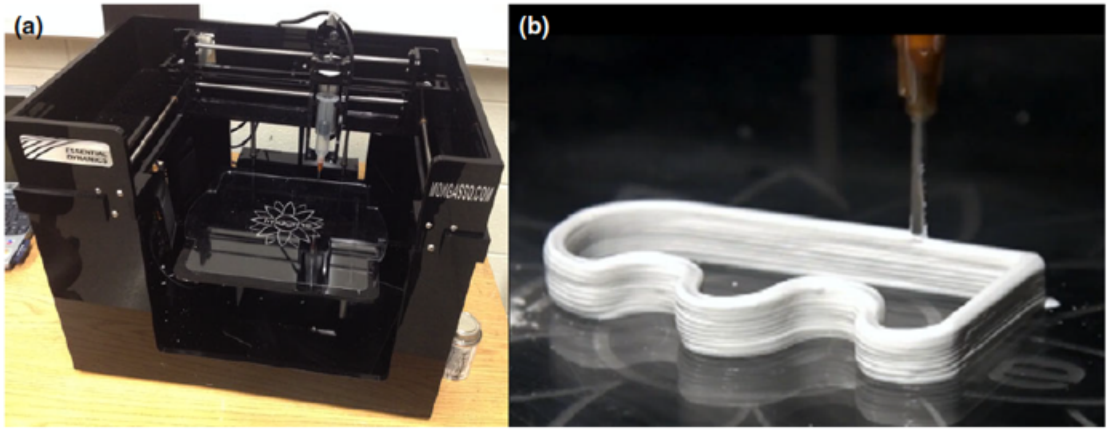
Motivation – Strong, Dense Al_2O_3

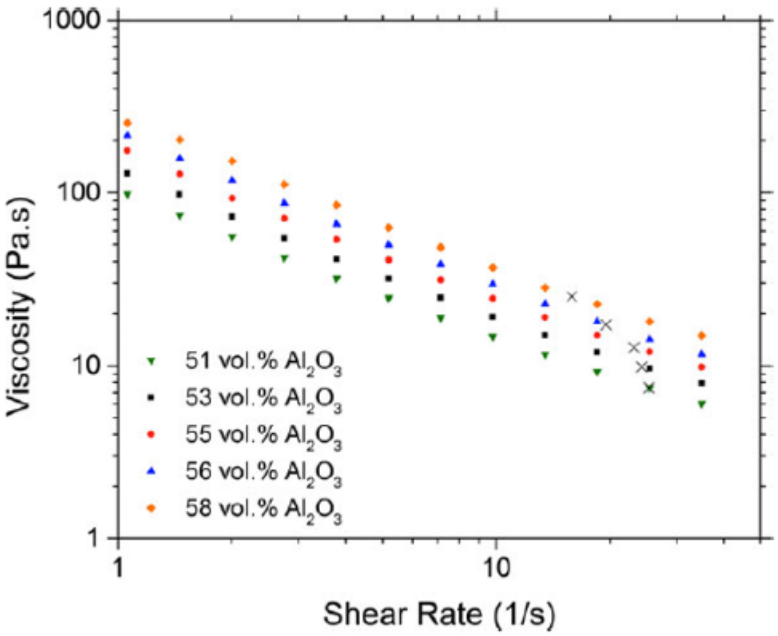
- Cheap printer
- Full densification without pressure
- Design yield-pseudoplastic Al_2O_3 ink
    - 55 vol.% = 122 Pa*s yield stress

Technical challenges

- High solids loading
- Slumping

Al_2O_3 (vol.%)	Shear Rate during forming 1/s	Nominal viscosity during forming (Pa·s)
51	25.3	7.5
53	24.2	9.9
55	23.2	12.8
56	19.5	17.2
58	15.9	25.0





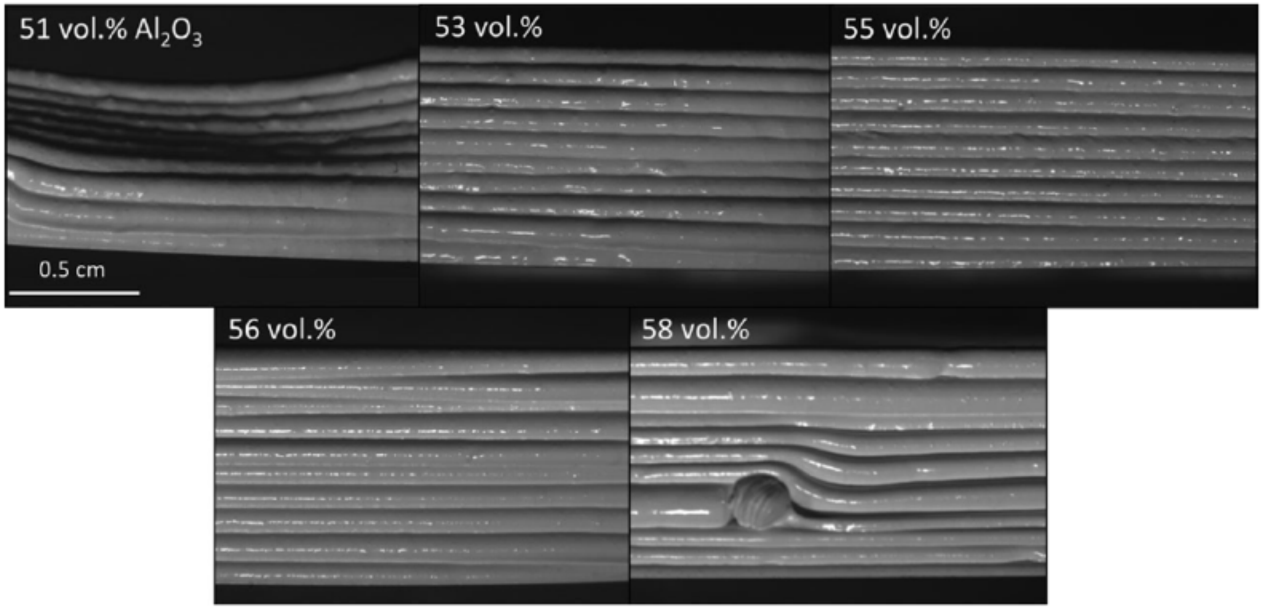
Decreasing viscosity with solids loading

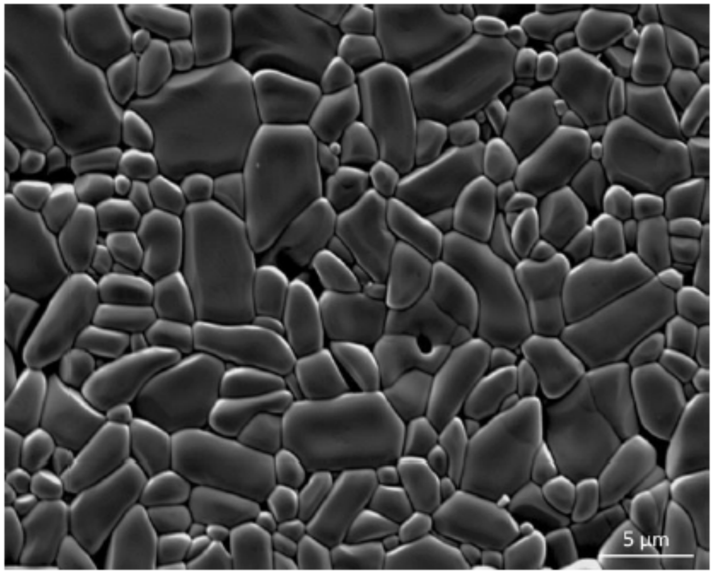
[25] L. Rueschhoff et al., *Int. J. Appl. Ceram. Technol.*, 2016

Dense Al_2O_3

Results

- Inexpensive, commercially available material extrusion printer
- 55 vol.% optimal - even, defect-free layers
  - 4.2 vol.% Darvan 821A
  - 4.9 vol.% PVP
- Pyrolysis at 700 °C
- Sintering at 1600 °C
  - No pressure or sintering additives





Densification > 98 %TD
Grain size = 3.17 um
Flexural Strength = 156.6 MPa

[25] L. Rueschhoff et al., *Int. J. Appl. Ceram. Technol.*, 2016

Dense B_4C

Lisa Rueschhoff group at Purdue (2016)

- Follow-up study for Boron Carbide

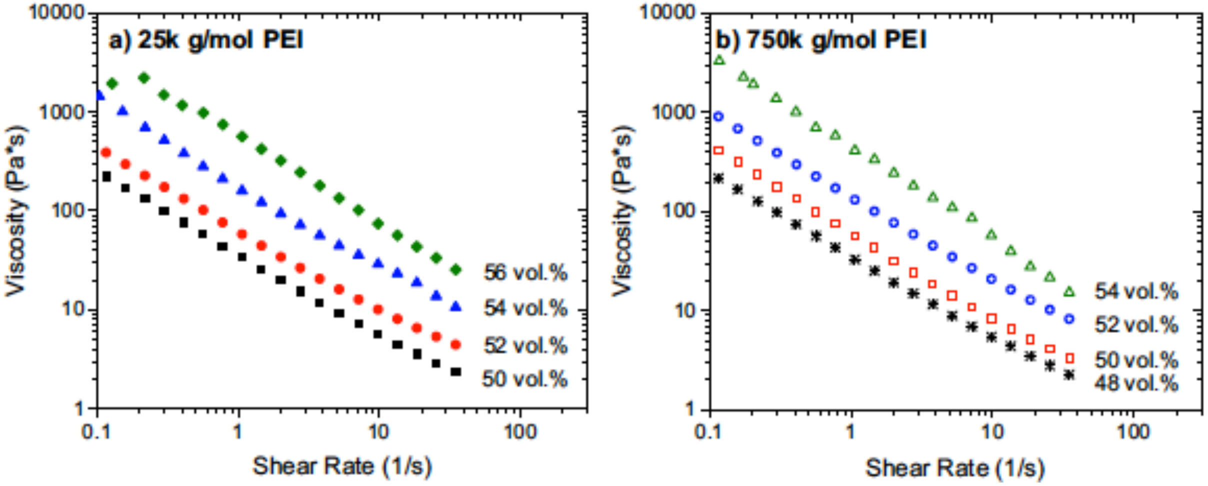
Motivation – Dense B_4C without field-assisted sintering

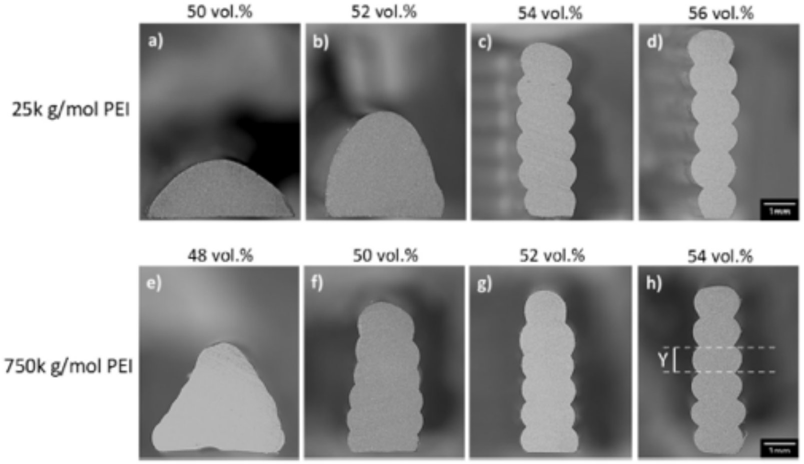
- Dense B_4C can not be printed via binder jetting or vat polymerization
- Design yield-pseudoplastic B_4C ink

Technical challenges

- High solids loading
- Slumping
- High density without field-assisted sintering techniques

Parameter	Value
Slice Height	0.85 mm
Path Speed	4 mm/s
Path Width	1 mm



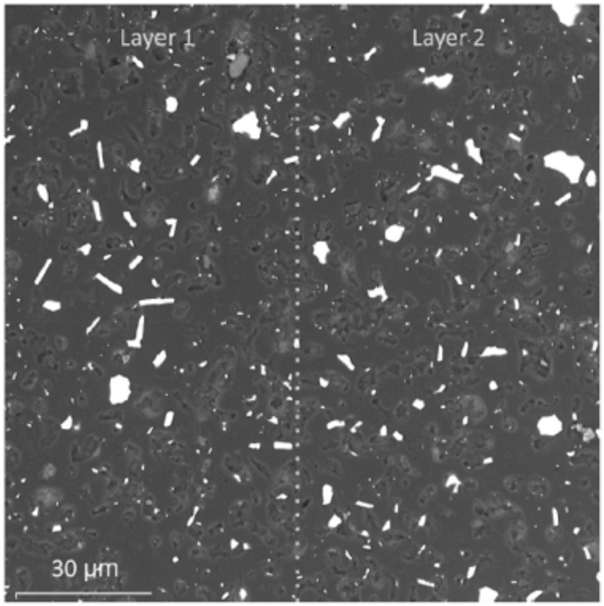


[26] W. J. Costakis et al., *J. Eur. Ceram. Soc.*, 2016

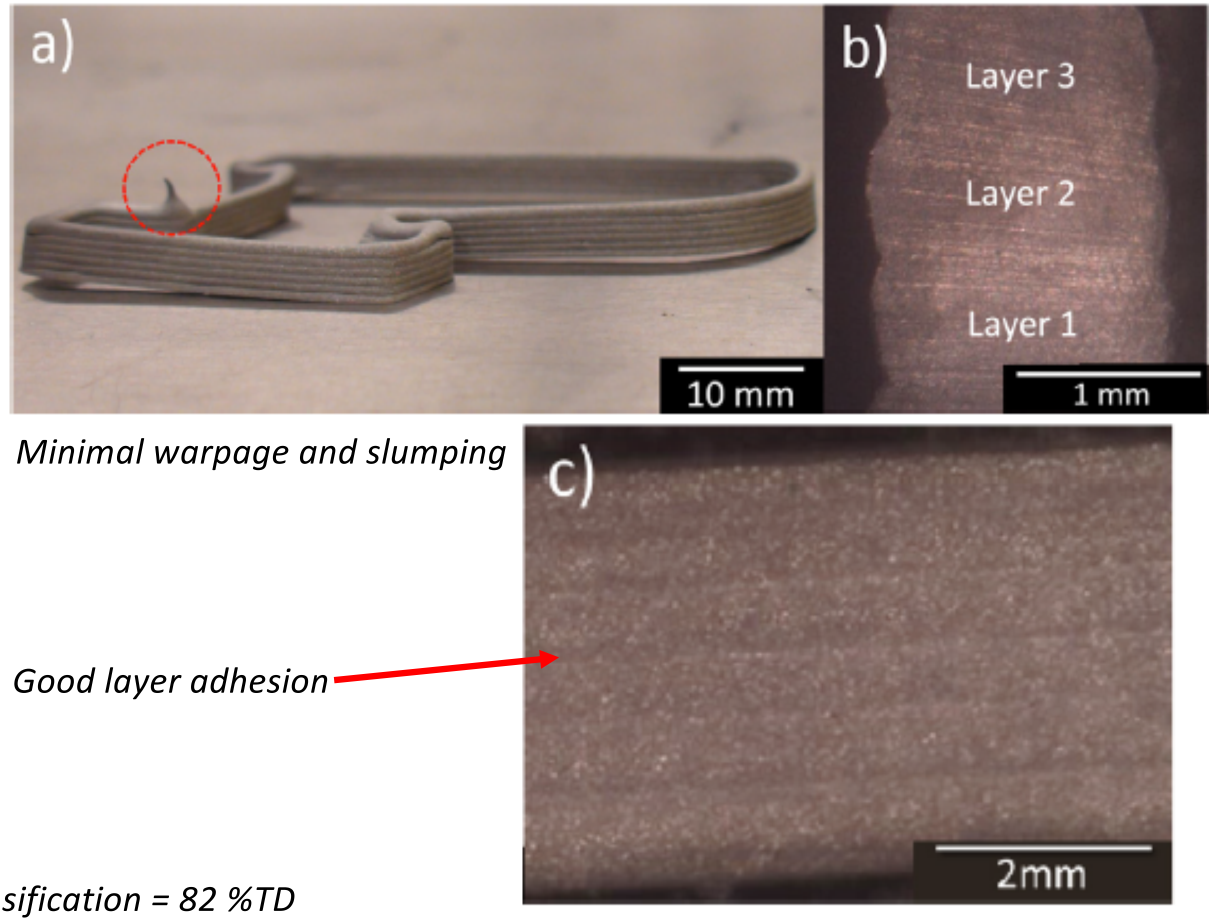
Dense B_4C

Results

- 54 vol.% optimal - even, defect-free layers
    - 5 vol.% PEI (25k g/mol)
    - 5 vol.% HCL
- Pyrolysis at 500 °C
- Sintering at 2000 °C
    - No pressure or sintering additives



Densification = 82 %TD



Minimal warpage and slumping

Good layer adhesion

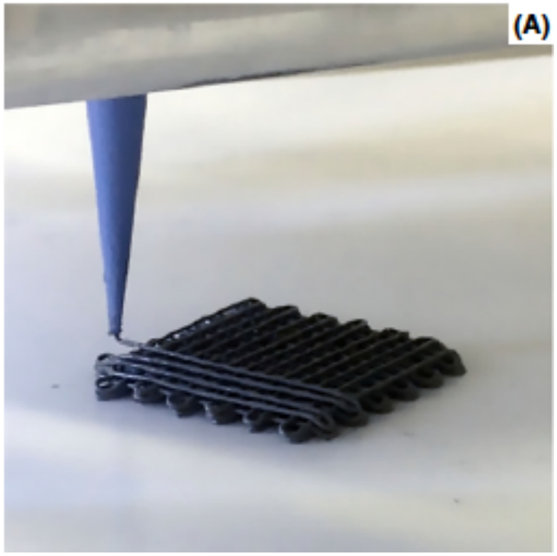
[26] W. J. Costakis et al., *J. Eur. Ceram. Soc.*, 2016

Fiber Alignment

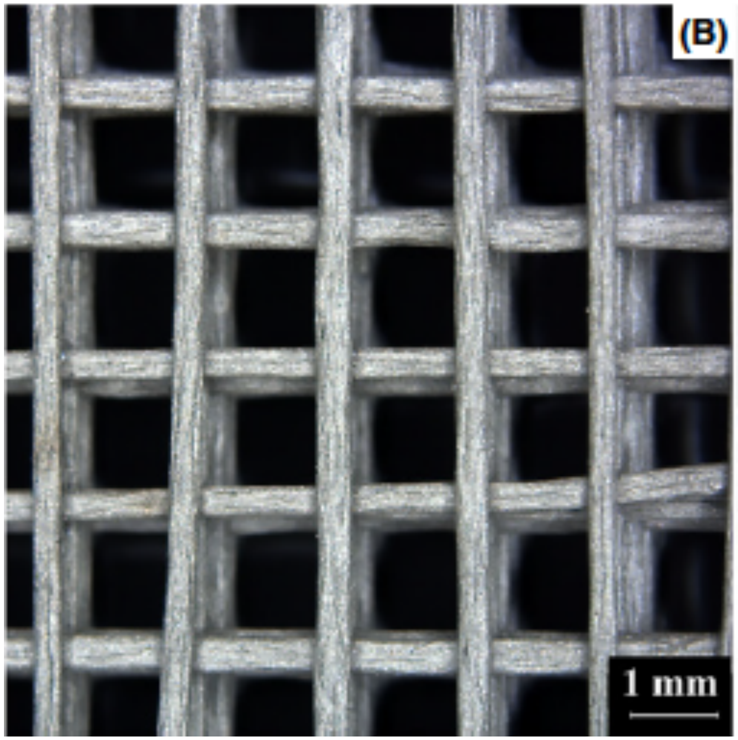
G. Franchin et al. (2017)
- Ceramic Matrix Composite (CMC) design

Motivation – Complex Part with Aligned Carbon-fiber
- Fiber alignment by nozzle pressure gradient
- Alignment not possible for binder jetting or vat polymerization

Technical challenges
- Ink printability with high aspect-ratio fibers
- CAD file design with optimized fiber direction



Chopped-fiber > 30 vol.%
Nozzle = 840 um



SiC Matrix with carbon fiber
Porosity > 75%
Compressive Strength = 4 MPa

[27] G. Franchin et al., *J. Am. Ceram. Soc.*, 2017

Fiber Alignment

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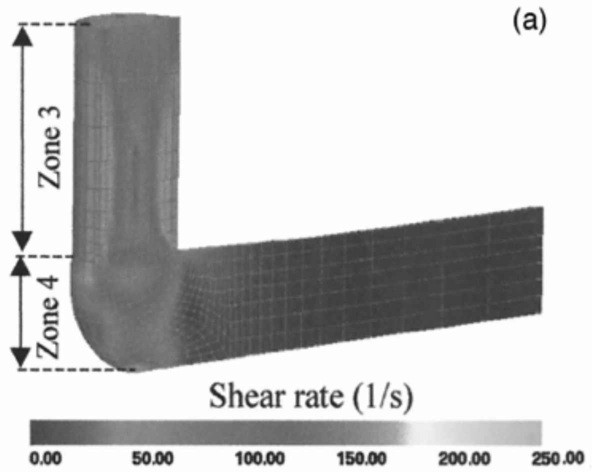
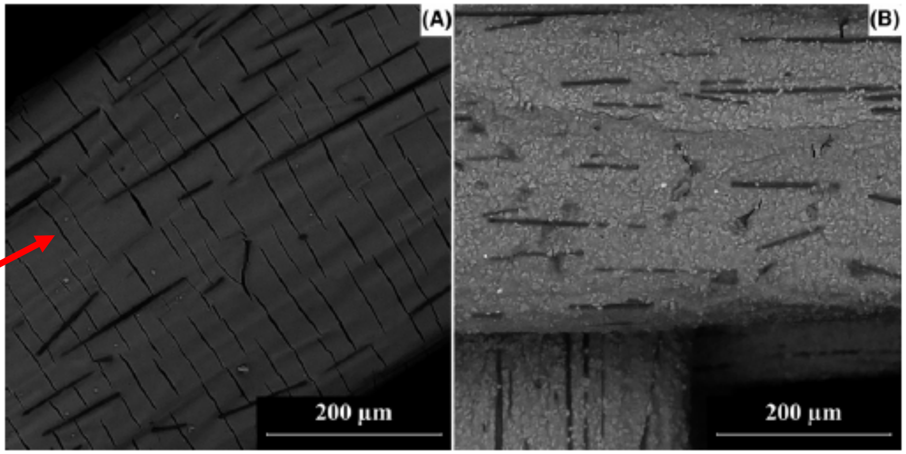
Results

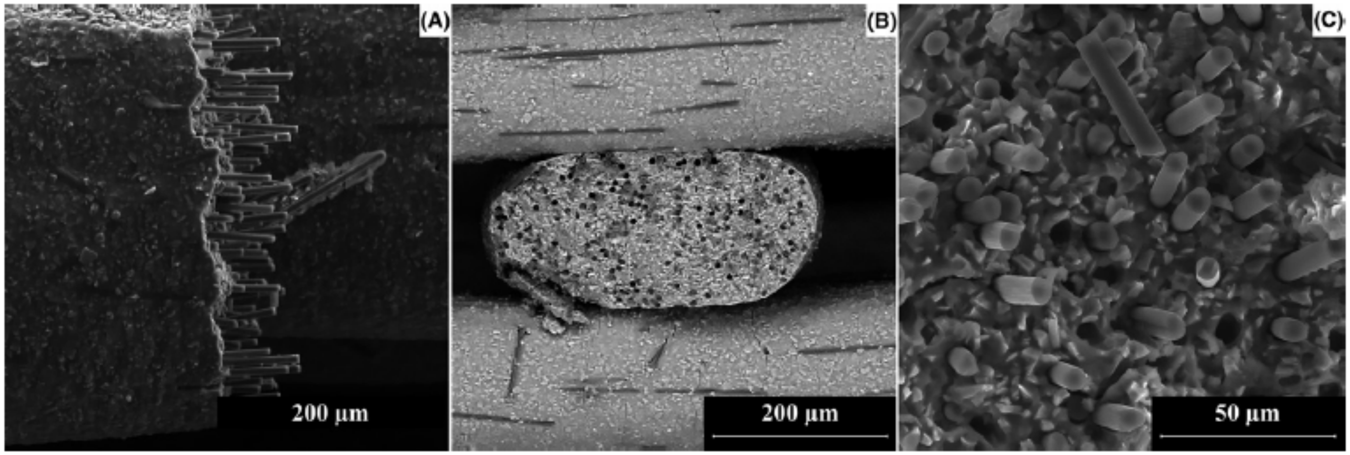
- Ceramic Matrix Composite with aligned fibers
  - SiC matrix
  - Chopped carbon fibers (L = 100 um, t =7.5 um)
- Shear stresses at nozzle orifice cause alignment
- Optimized ink reduces stress cracking



J. Lewis et al., J. Am. Ceram. Soc., 2000



Stress-cracking



Fiber alignment with printing direction

[27] G. Franchin et al., *J. Am. Ceram. Soc.*, 2017

Multi-material Printing

James Smay et al. (2007)

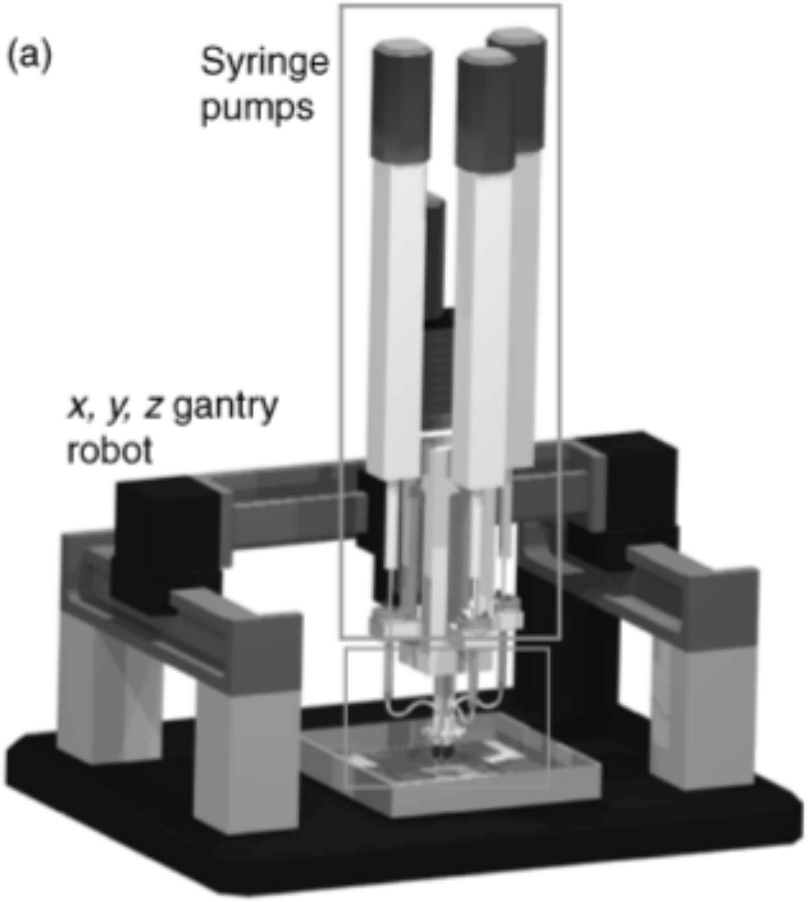
- Multi-material material extrusion study

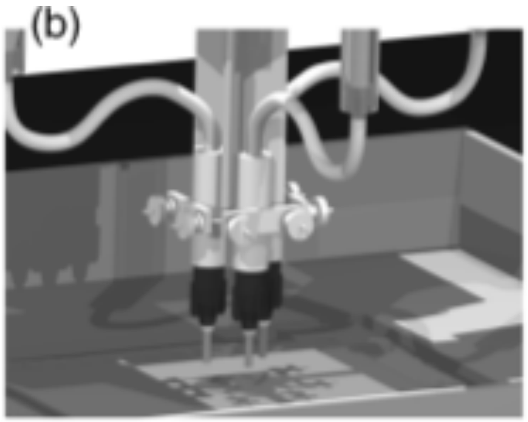
Motivation – Cermet Composites and Gradient Compositions

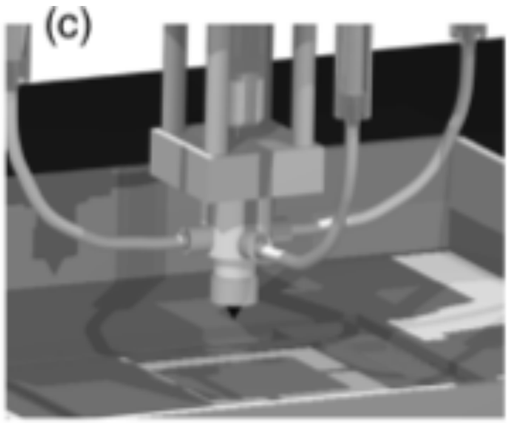
- Ternary mixtures for rapid screenings of technical ceramics *(c)*
- Ceramic-Metal (Cermet) composites *(b)*
- Explore active mixing nozzle strategies

Technical challenges

- Variation in rheology between multiple slurries
    - Viscosity
    - Compressibility
- Variation in surface chemistry between metallic and ceramic powder





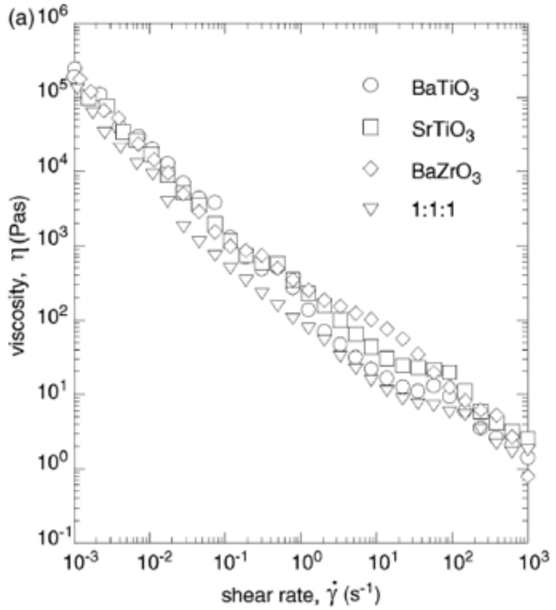


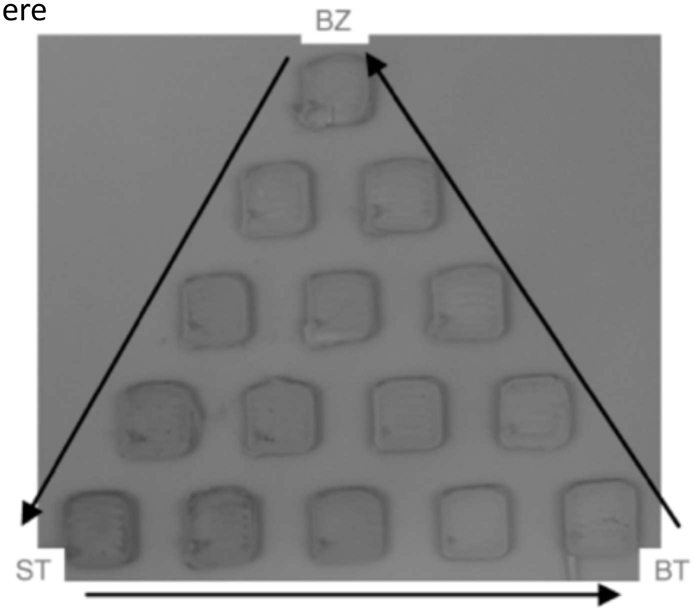
[28] J. E. Smay et al., *Int. J. Appl. Ceram. Technol.*, 2007

Multi-material Printing

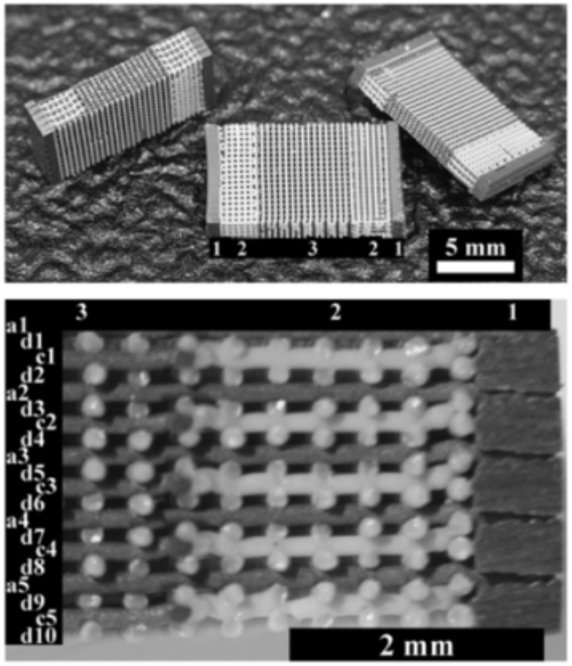
Results

- Developed inks of similar rheology
- Discrete and gradient multi-material printing capabilty shown
- Created ternary gradients of parts for rapid dielectric screening
    - Better bulk property representation than thin film screening techniques
- Cermet parts demonstrate good shape retention during densification
    - Sintering done in a reducing atmosphere





Ternary made by gradient compositions for rapid screening of advanced oxide ceramics



BaTiO$_3$ – Ni composite structures for dielectric applications
Sintering done at 1350 ℃ with flowing gettered-Ar.

[28] J. E. Smay et al., *Int. J. Appl. Ceram. Technol.*, 2007

Vat Polymerization

Common Names
- Stereolithography (SLA)
- Digital Light Processing (DLP)
- Lithography-based Ceramic Manufacturing (LCM)

Advantages
- High accuracy and complexity
- Good surface finish
- Best resolution = 10 um
  - Related to powder size

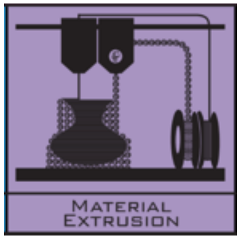
Applications
- Biomedical implants where surface texture is important
- Low refractive index materials

Binder Jetting

Common Names
- 3D Printing (3DP)
- VoxelJet™

Advantages
- High resolution
- Unbound-powder supports overhangs
- Wide range of materials
  - Any powdered material
- Best resolution = 20 um
  - Based on powder size

Applications
- High resolution scaffold structures
- Low density bioresorbable structures



Material Extrusion

Common Names
- Direct ink writing (DIW)
- Fused Deposition Modeling (FDM)
- Fused Filament Fabrication (FFF)

Advantages
- Inexpensive
- Low complexity equipment
- Dense green bodies
- Small powder size
  - Improved sintering
- Best resolution = 150 um

Applications
- Dense technical ceramics
- Near-net shape parts
- Fiber alignment
- Multiple materials

Acknowledgments

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Members of the Meyers Group

Meyers Group

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Questions?