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Ceramic feedstock for "Additive" Manufacturing

Chris DiAntonio, Adam Cook, Tom Chavez, Lindsey Evans, William Reinholtz, Kristin Meyer

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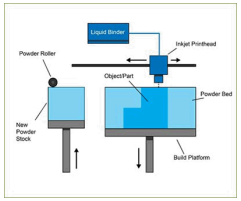
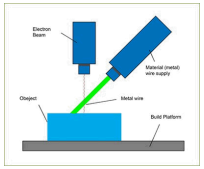
“Additive Manufacturing” Landscape

- Presently – AM (additive manufacturing)
  - Polymers – State-of-the-art

  - Metals – many materials can be processed
    - Metal binder jetting



    - Powder Bed Fusion
      - Selective Laser Sintering (SLS)
      - Selective Laser Melting (SLM)
      - Direct metal laser sintering (DMLS)



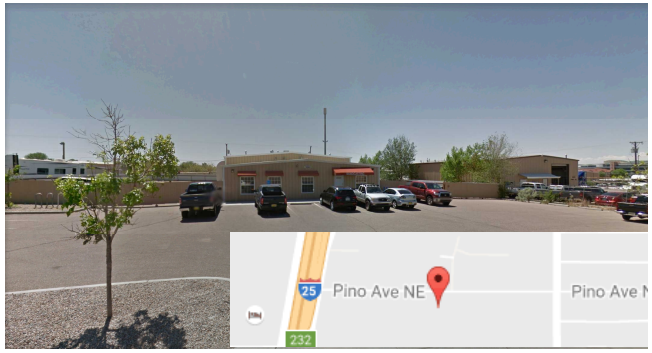
    - Directed Energy Deposition
  - Ceramics – the technical application of AM technologies is yet limited
    - Ceramic materials space is enormous and extremely wide
    - Concepts for “additive manufacturing” is enormous and extremely wide
    - “Ceramic feedstock” variability – ceramic processing not easy and not flashy
    - Printing platforms tend to be over and under developed
    - Post-printing dilemma
    - Desire for flexibility

Additive Manufacturing in Ceramics Land

- Ceramic materials
  - Have been studied in AM processes ab initio for about 25 years
  - The popular AM technologies, formerly referred to as
    - Rapid Prototyping (RP)
    - Solid Free Form Fabrication (SFF)
  - Conventional stereolithography (STL) process – photopolymerizable ceramic suspension is cured by a UV laser
  - Selective laser sintering (SLS) and 3D powder bed printing
  - Robocasting – computer controlled deposition of colloidal paste or slurries
    - Carrier fluid is a volatile solvent (water or organic liquid)
    - Highly dispersed ceramic suspensions – specific rheology
  - Conventional fused deposition modeling (FDM) – uses a thermoplastic ceramic feedstock that is liquefied by heating and pressed through a fine nozzle (physical sense a suspension is used)
    - Efforts for the preparation of the thermoplastic ceramic feedstock in the form of spooled filaments constrain the FDM application for ceramics

Exploration of Fabrication options: Direct Write Method

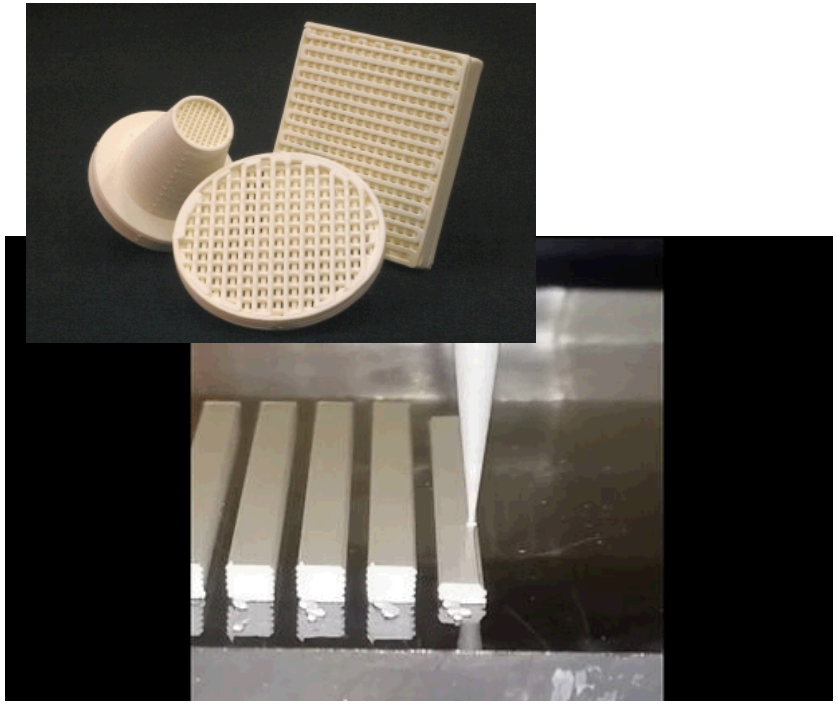
Robocast



- Developed at SNL by Joe Cesarano and eventually became private spin-off company in Albuquerque

- Utilizes non-Newtonian fluids that undergo shear thinning (e.g. ketchup)

- Rheology control very complicated
  - Stability of slurry an issue … same day use
  - Cost/time associated with managing this one aspect of the technology exceeds allotted budget



Exploration of Fabrication options: Direct Laser Melting

- 3D promised Al_2O_3 processing on ProX 300
- Many concerns about whether this platform could deliver on promise
  - Coupling of laser into materials that may absorb poorly
  - High melting point of ceramic materials lead to high thermal stress
  - Inability to choose low CTE material (design constraints) like SiO_2 further exacerbates high stress problems
- Very limited set of publications on direct laser melting of ceramics
  - Often more advertisement than technical
  - Applied Surface Science ref in 2007 showed picture of ZrO_2 part printed on **PM 100 (phenix powder bed system)** with reported density of 56%
  - Authors gave little description of process
    - Mentioned importance of "furnace" to minimize cracks
  - Max temp of ProX 300 is ~100 C

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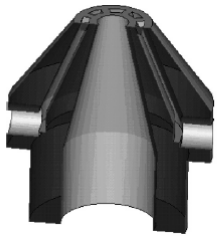
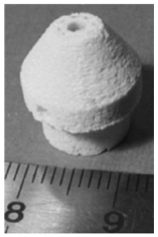
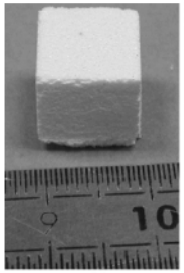
Ceramic components manufacturing by selective laser sintering

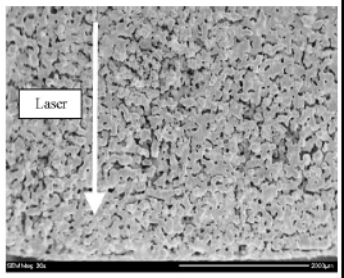
Ph. Bertrand [a], F. Bayle [a], C. Combe [a], P. Goeuriot [b], I. Smurov [a,*]
[a] École Nationale d'Ingénieurs de Saint-Etienne (ENISE), DIPI Laboratory, 58 rue Jean Parot, 42023 Saint-Etienne Cedex 2, France
[b] École Nationale Supérieure des Mines de Saint-Etienne (ENSMSE), Centre SMS 158, Cours Fauriel, 42023 Saint-Etienne Cedex 2, France

Available online 1 September 2007

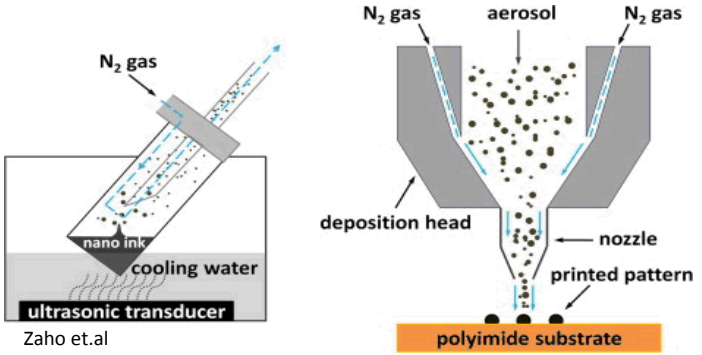








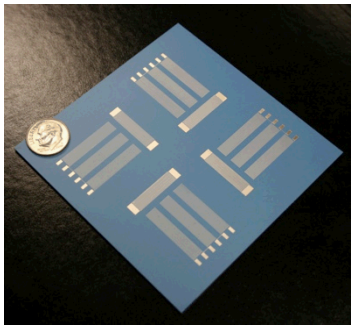
Ceramic AM at SNL

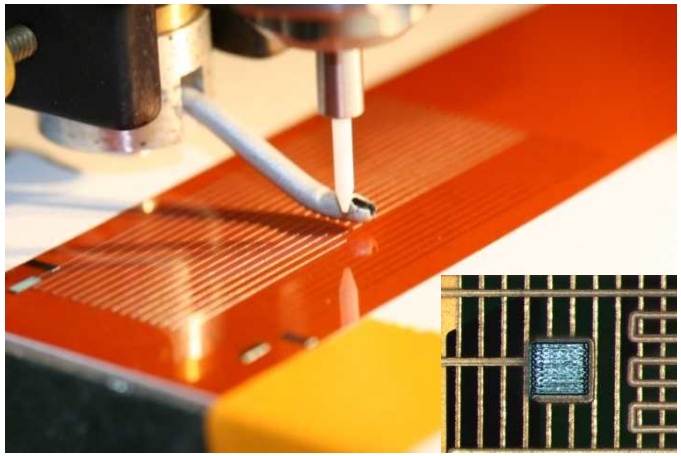


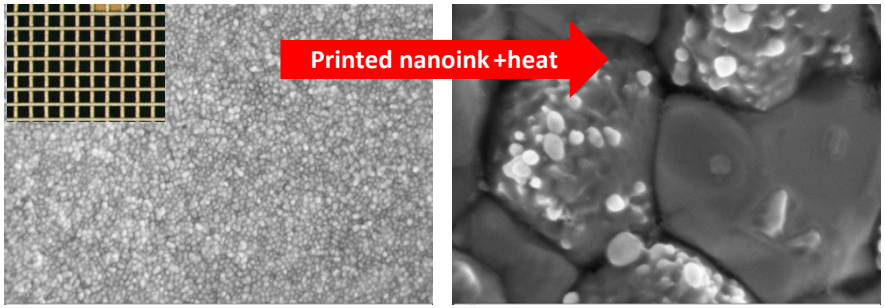
Zaho et.al

Aerosol Jet Printing Method (Optomec)

- Aerosol can be focused using inert gas streams and a small nozzle
- Atomization of liquid ink to produce a dense aerosol mist
- Line widths as narrow as 10 µm with 0.5-3 µm heights (silver nanoink)
- Broad materials compatibility
- Expanded post processing capabilities
- Rapid design iteration

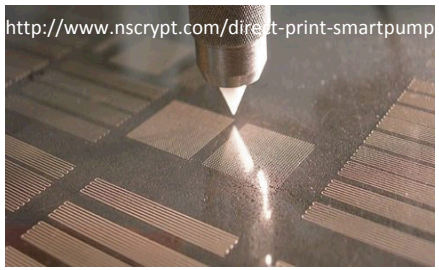


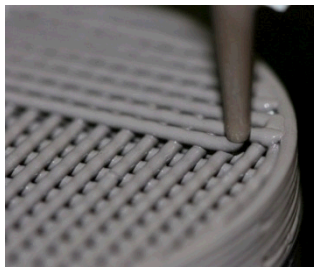




- DC and RF pathways for interconnect and antenna applications on planar or arbitrary surfaces
- Strain and crack sensors for structural health monitoring, resistance temperature devices (RTD)
- Integration of packaged components with external sensing networks for value added functionality

Ceramic AM at SNL



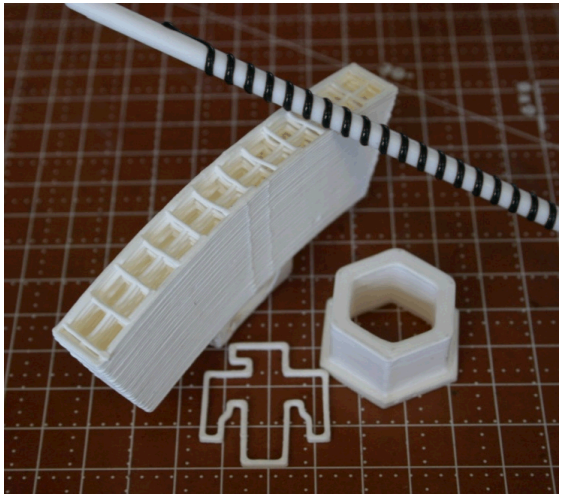


Extrusion Casting (nScrypt, Robocasting)

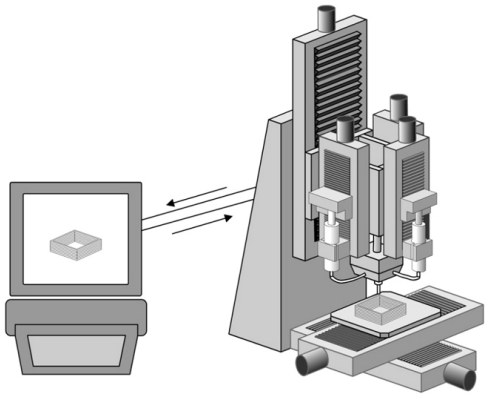
- Low volume print head
- 20 Pico-liter discrete volume depositions (minimum)
- Compatible with materials ranging from 1-1,000,000 cPs

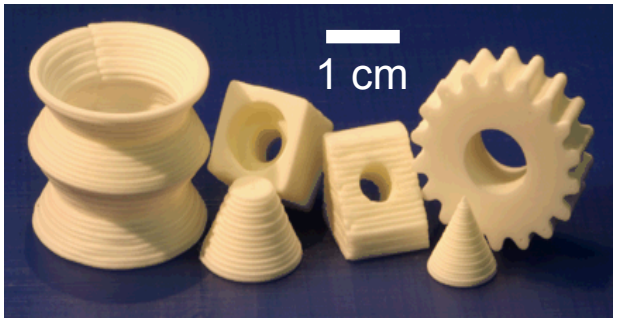
Compatible Materials:

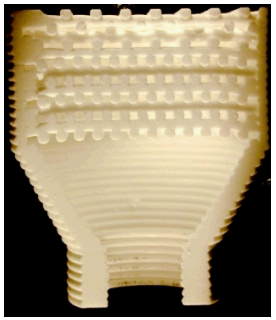
Alumina	**UV Curable Epoxies**	**Conductive inks**
Zirconia	**Silicones**	**Novel Formulation**

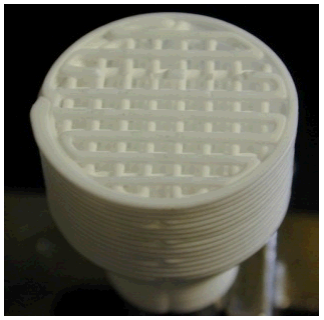


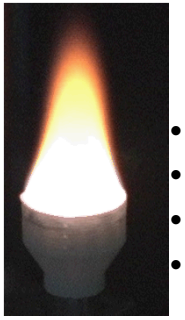
"Robocast" Ceramic Parts







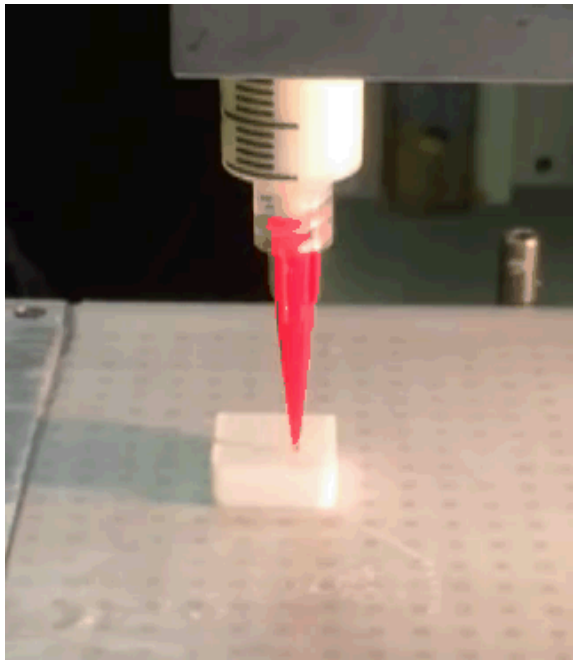


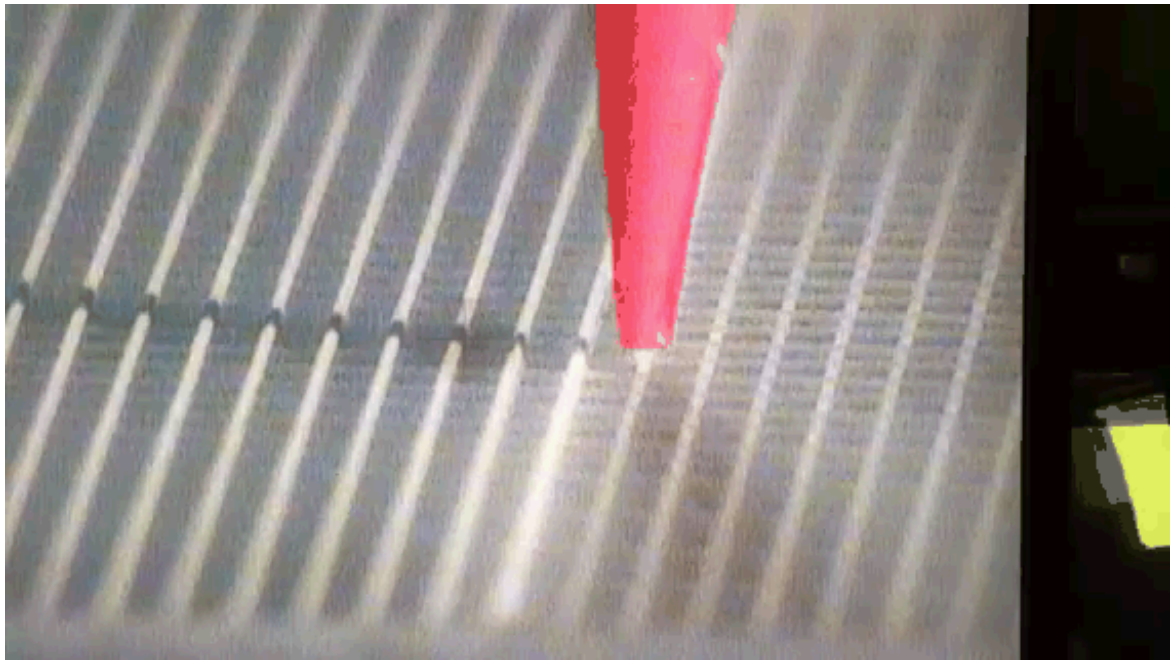


Challenges:

- Will shrink slightly when fired
- May require surface finishing
- Rheology dictates performance
- Surface chemistry is critical

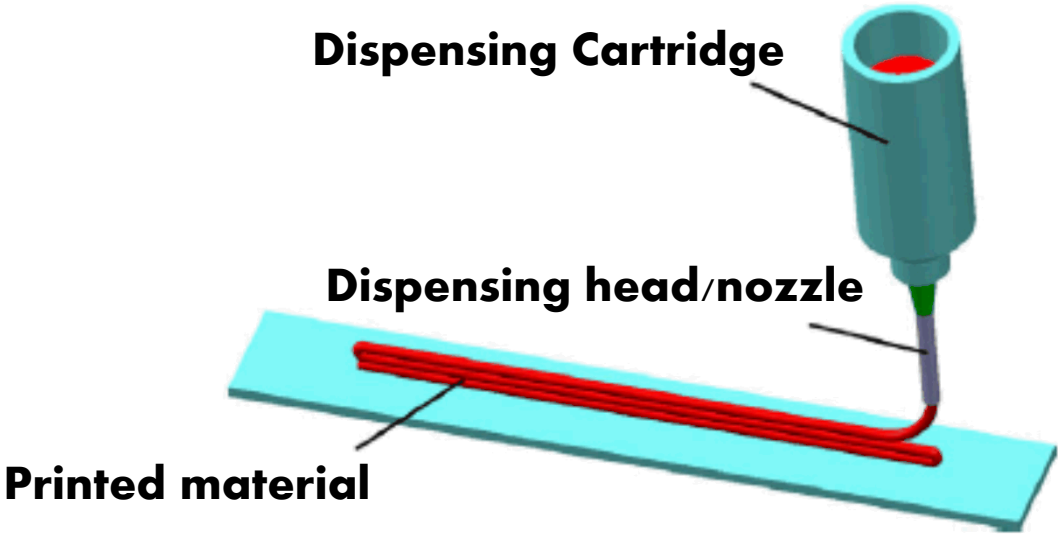
Ceramic AM at SNL





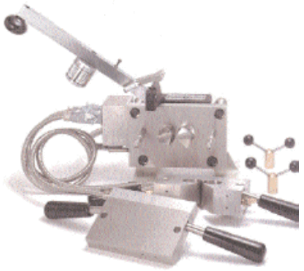
Ceramic-Thermoplastic 3D (CT3D) Printing

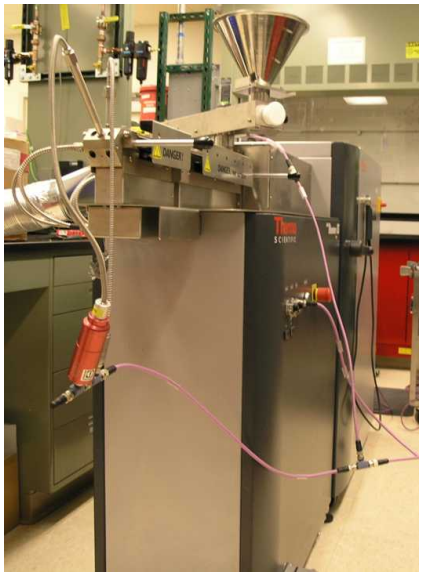
- Proposed Approach – Combine FDM and Robocasting
- Ceramic-Thermoplastic 3D Printing (CT3D) – An "additive manufacturing" method for producing ceramic components
  - Highly-filled ceramic suspension based on thermoplastic binder systems is used to produce ceramic components by "additive manufacturing"
  - Utilize a thermoplastic binder system to prepare loaded feedstocks that are processed in a temperature controlled dispensing unit with xyz positioning



Ceramic (CT3D) Feedstock Processing

- Brabender Intelli-Torque, Plasti-Corder – used for compounding/mixing the feedstock
- ThermoScientific – HAAKE PolyLab OS Rheodrive extruder
- Dynisco LCR 7000 – Capillary Rheometer





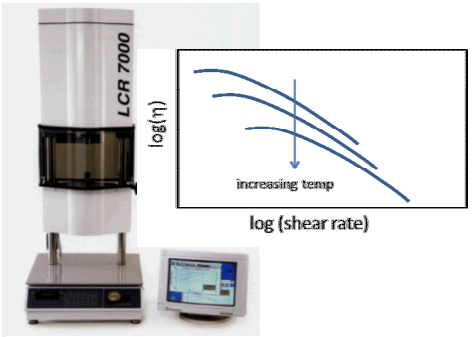
Exploration of Fabrication options: Direct Write Method

Extrusion Method

Feedstock Fabrication

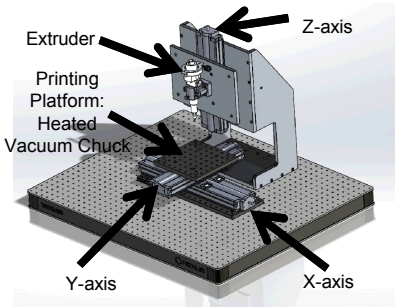
Brabender Intelli-Torque, Plasti-Corder

Feedstock Characterization

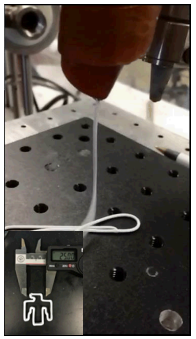


Dynisco Capillary Rheometer

Printing on Direct Write Platform



Modular direct writing printing system



Al_2O_3

Characterize, feedback & optimize

Technique is modified version of FDM

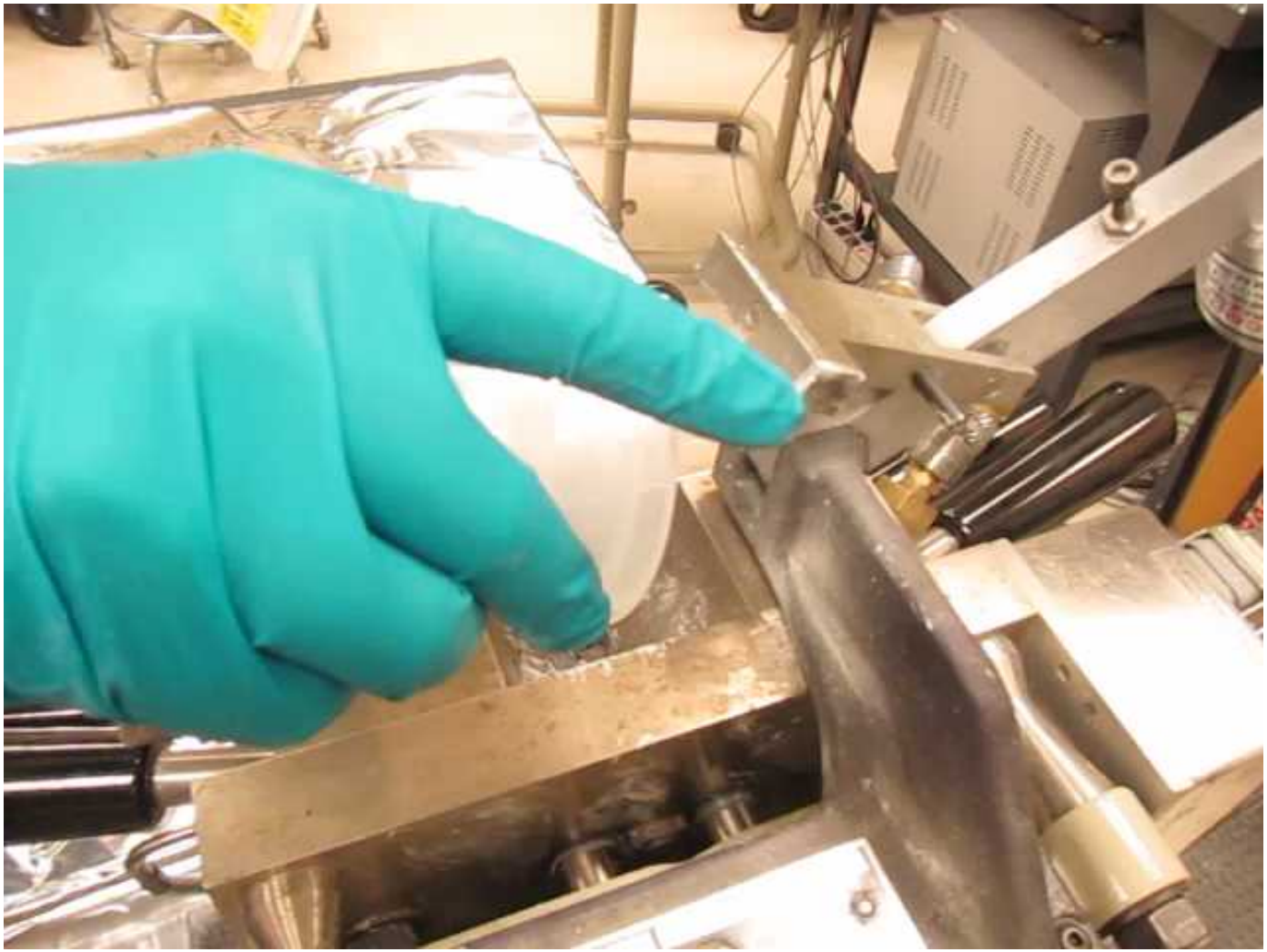
Feedstock Development

- Thermoplastic systems chosen as binder
- Initial attempt with Al_2O_3,

Direct write platform

- 3-axis automated motion control, Heated platen, Syringe dispense module with heater
- Pressure & temperature data can be monitored from nozzle – will go into build file for analysis

Ceramic (CT3D) Feedstock Processing



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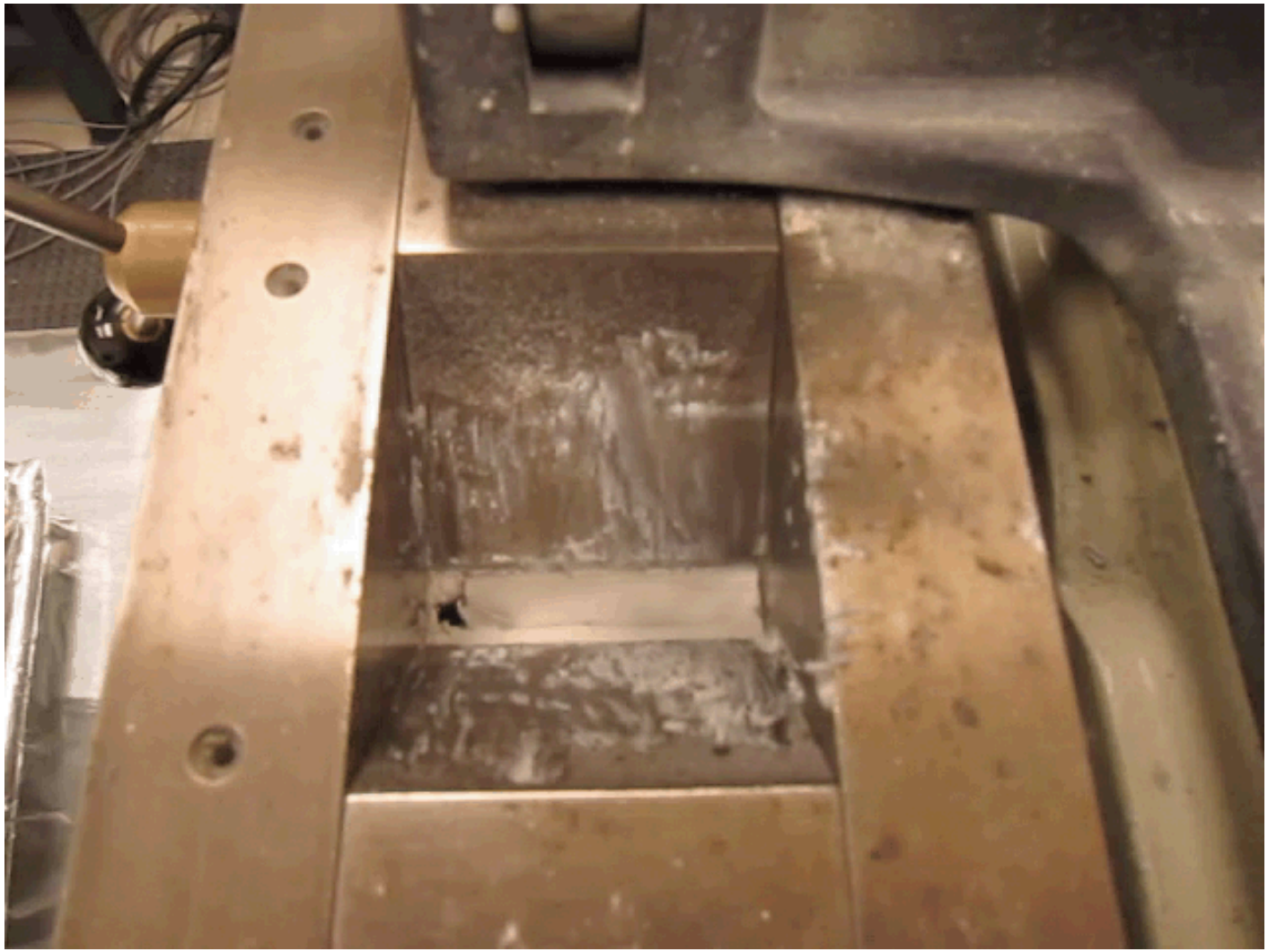

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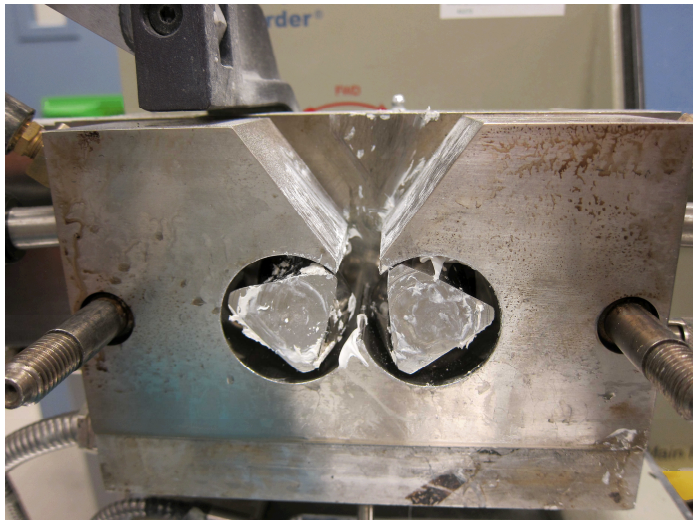

Ceramic (CT3D) Feedstock Processing

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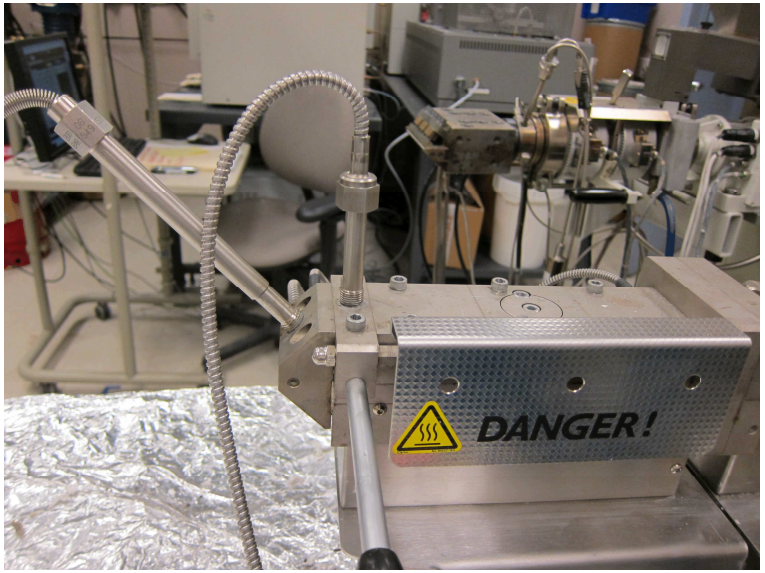
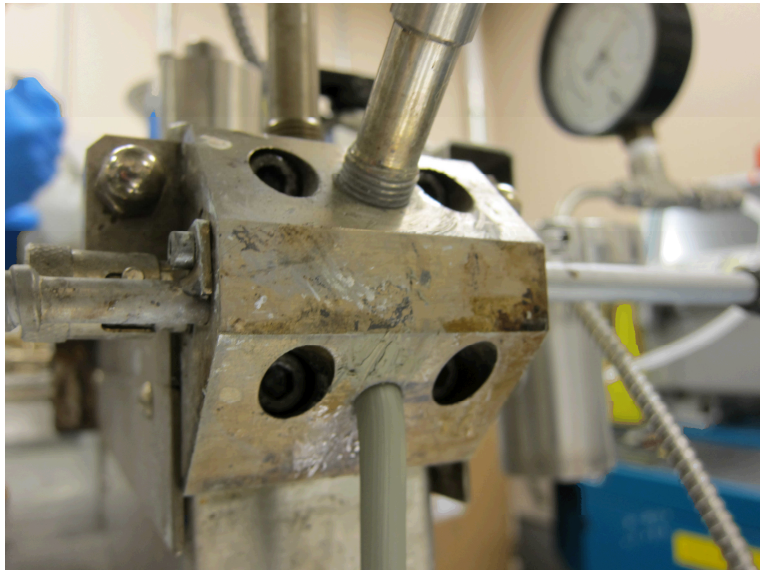
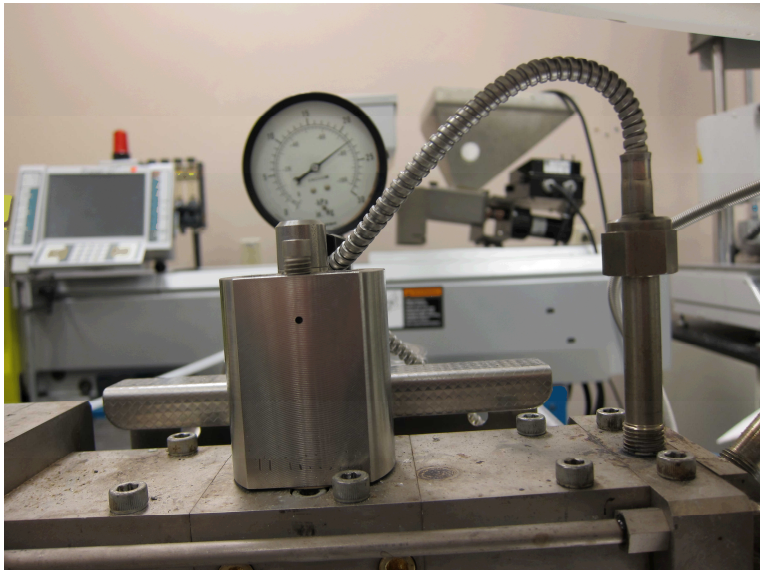
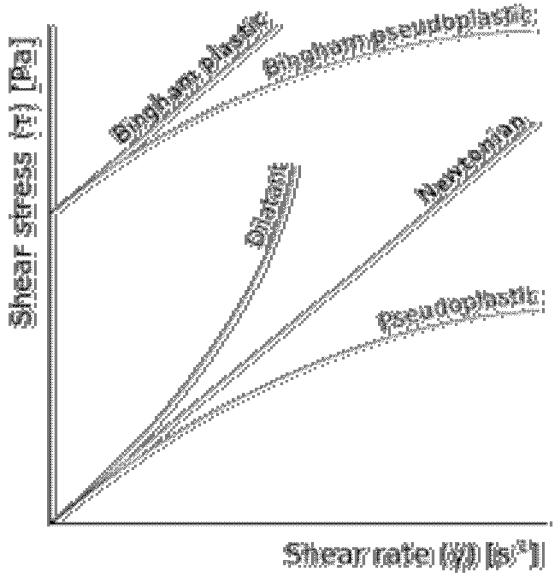


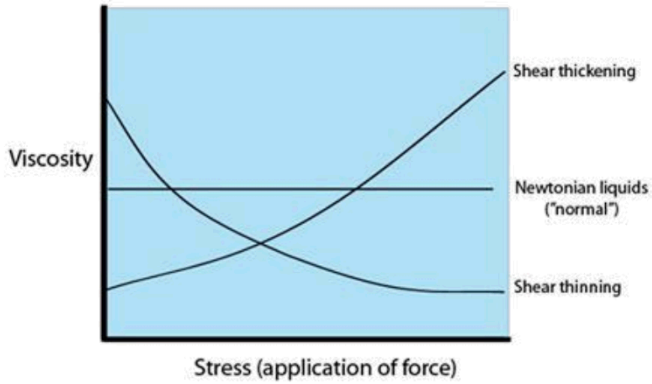




Rheology

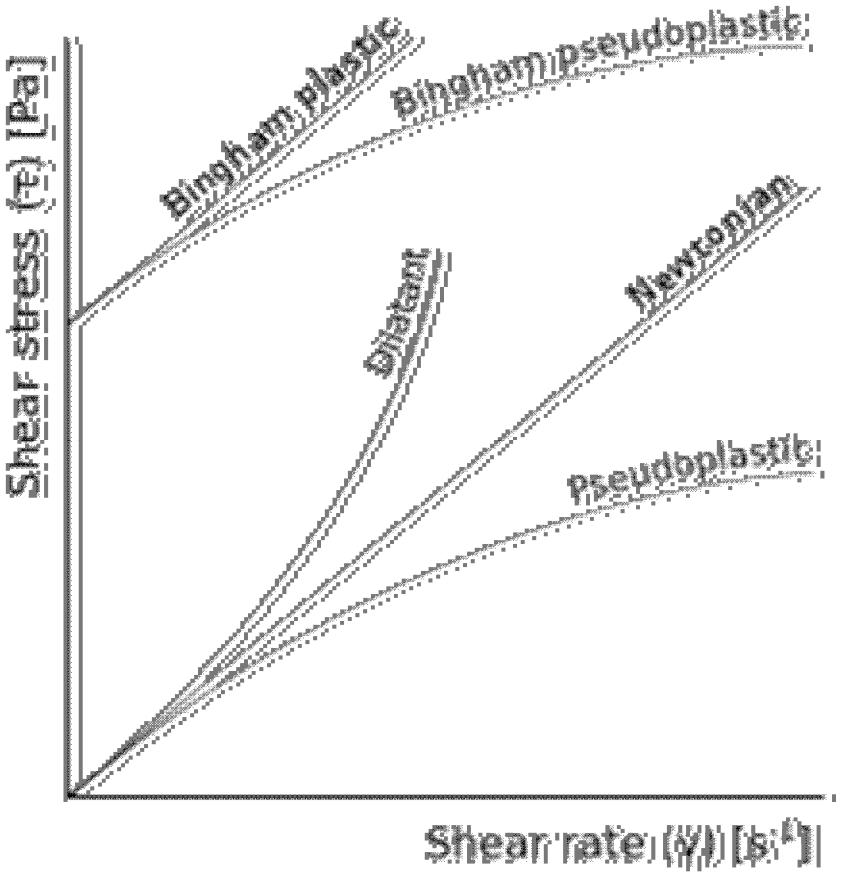
- Rheology
  - Characterization is very important to evaluate the processability of the suspension
    - Estimation of the ability to meter small volumes
    - Dynamic fluid flow characteristics
    - Differences in materials
      - Powder properties – particle size distribution, surface area, density, morphology
  - Ideal suspension should have a pseudoplastic (i.e. shear thinning) behavior in a low viscosity range
    - non-Newtonian fluid which has a decreased viscosity when subjected to shear strain, excluding time-dependent effects, such as thixotropy





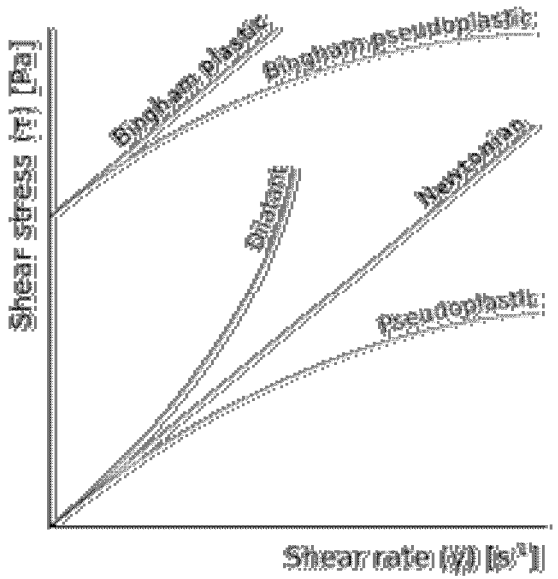
Rheology

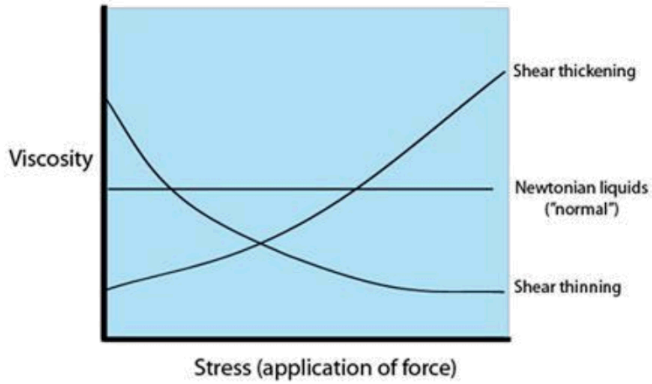
- Rheology
  - Bingham Plastic and Bingham Pseudoplastic – “Yield Materials”
  - Bingham Pseudoplastic with Yield Stress – Shear Thinning
    - Thermoplastics, Clay, Tar, Sludge
  - Pseudoplastic – Shear Thinning
    - Paper pulp, grease, soap, paint
  - Dilatant – Shear Thickening
    - Beach sand, Starch in water
  - Newtonian
    - Water, gasoline, motor oils



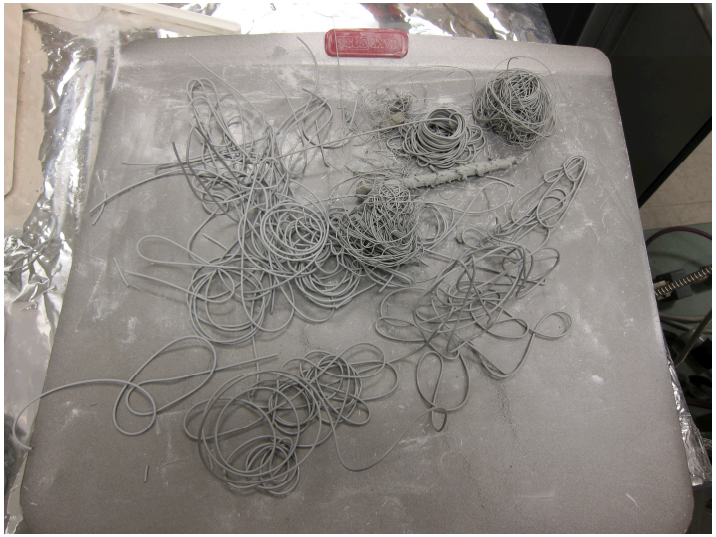
Rheology

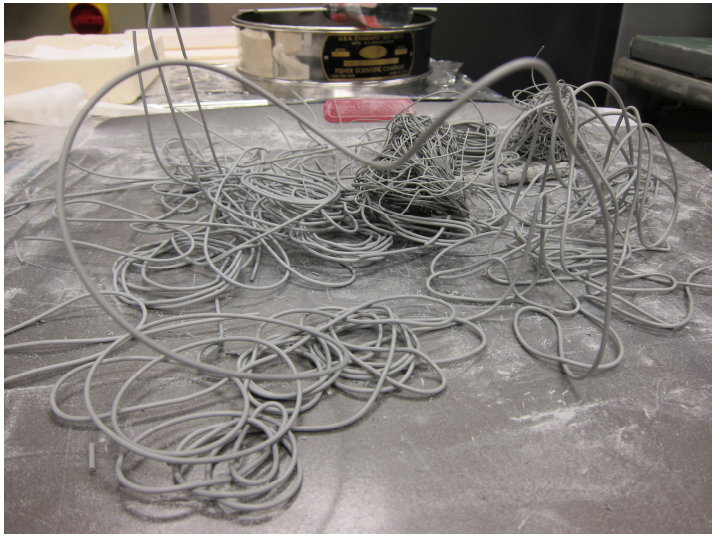
- Rheology
  - Viscoelastic behavior means that the suspension has a very low viscosity at high shear rates
  - Important for metering small volumes through small geometries at low pressures
  - High viscosity at low shear rates to be fixed at the point of application
  - Flow behavior dependent on several factors – temperature, pressure, material structure
  - Essential to optimize this parameter because the realizable resolution is strongly dependent on the metering (i.e. pressure), which in turn depends on the viscosity

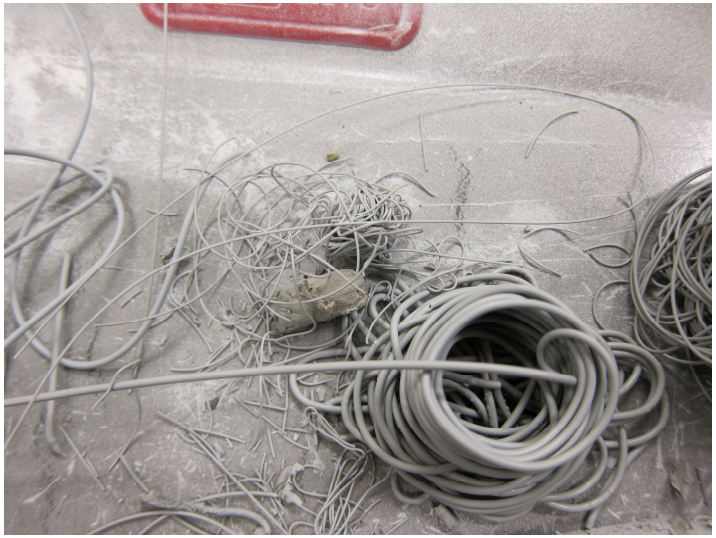




Rheology

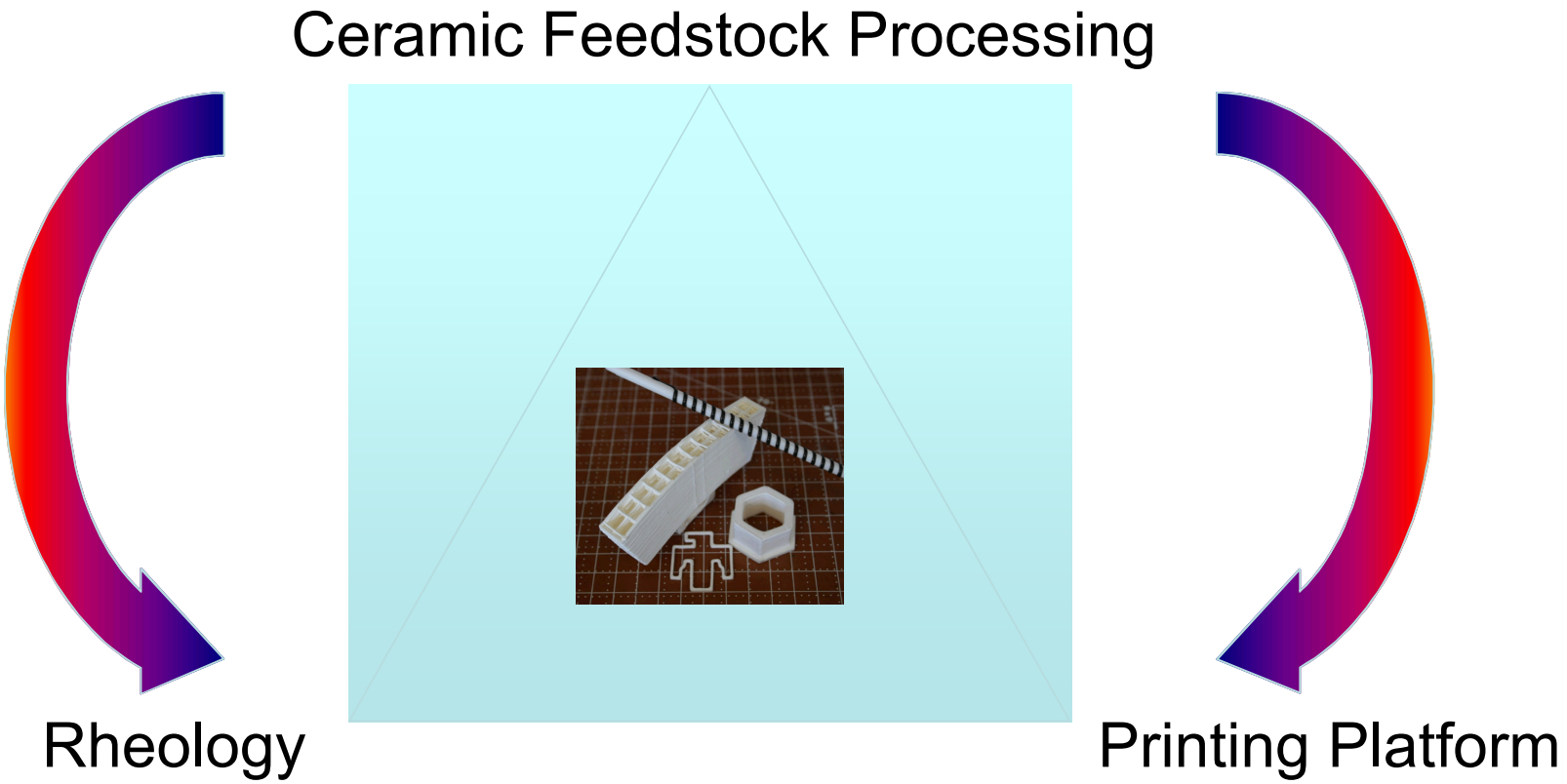




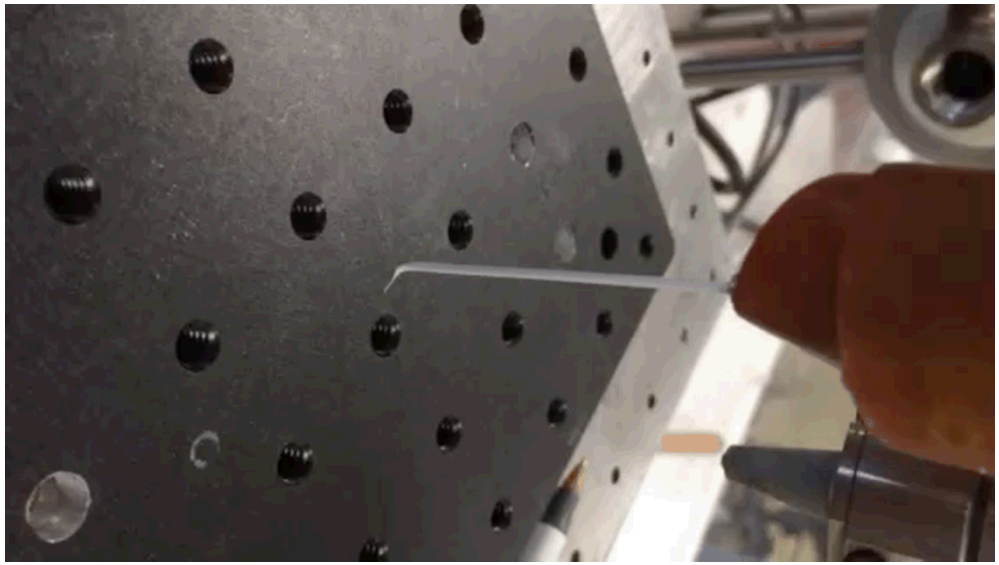


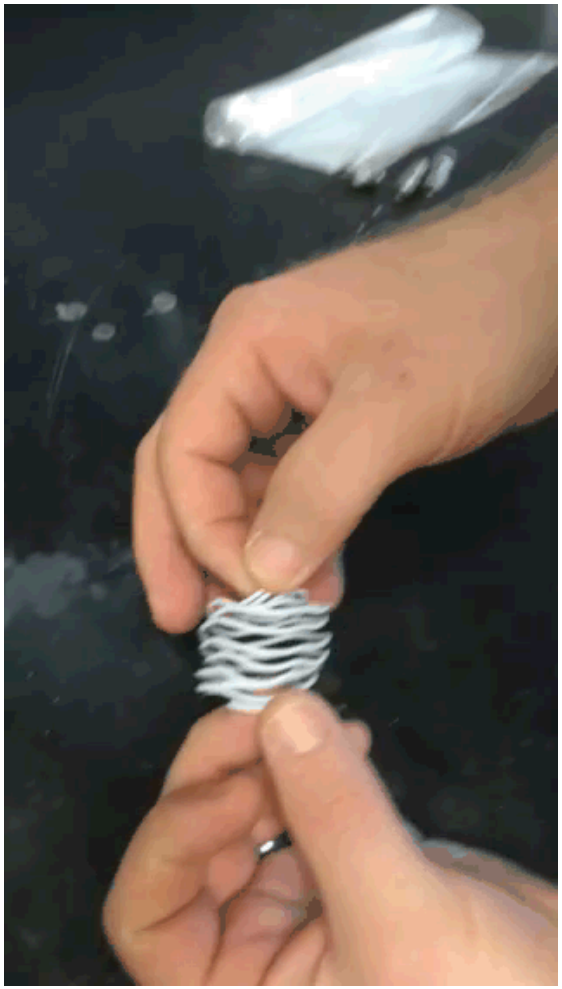
Development of the CT3D Printing Platform

- Co-dependent

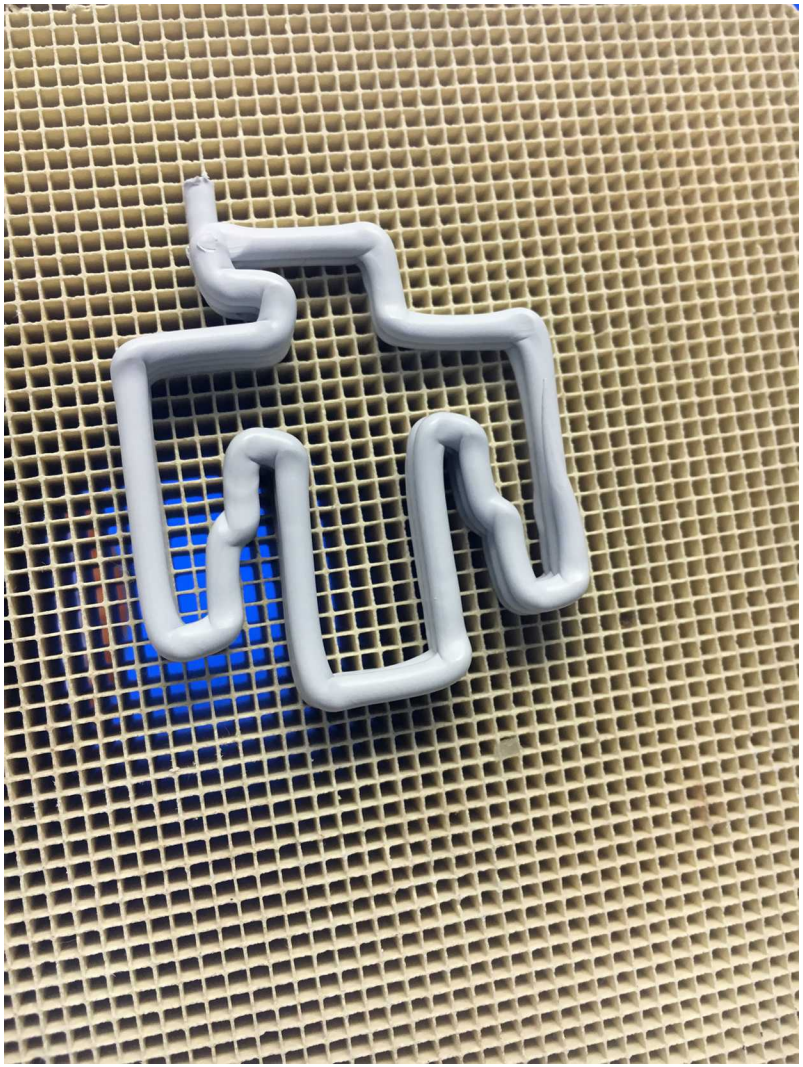


Development of the CT3D Printing Platform





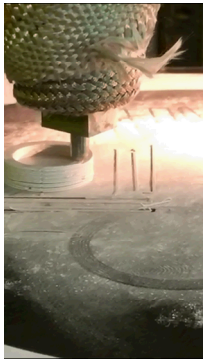
Development of the CT3D Printing Platform



CT3D Direct Write Progress to date: Initial test parts

- Numerous initial test parts have been printed and sintered. Characterization has started as well.

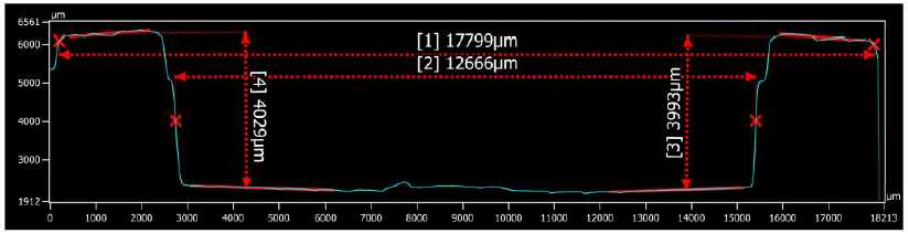
Early test part



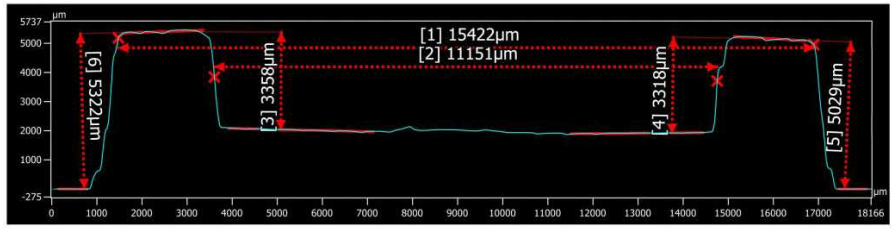
20 mm diameter crucibles, 3 mm wall thickness, 6 mm height

- Solids loading effects rheology and microstructure
- Archimedes density of sintered alumina samples is greater than 93%TD
- Linear shrinkage after sintering between 12-17%

As-printed



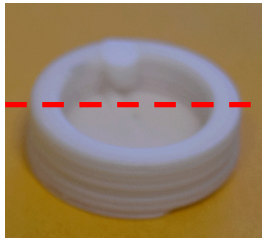
Sintered



Measurement equipment : KEYENCE VR-3000 Series

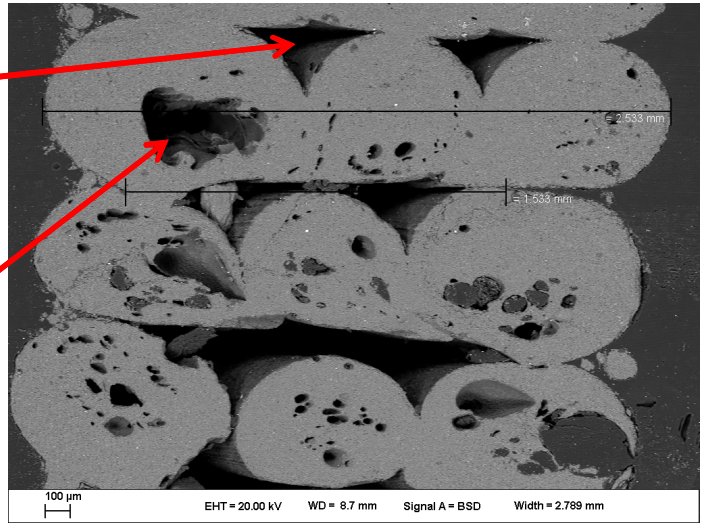
CT3D Direct Write Progress: Characterization

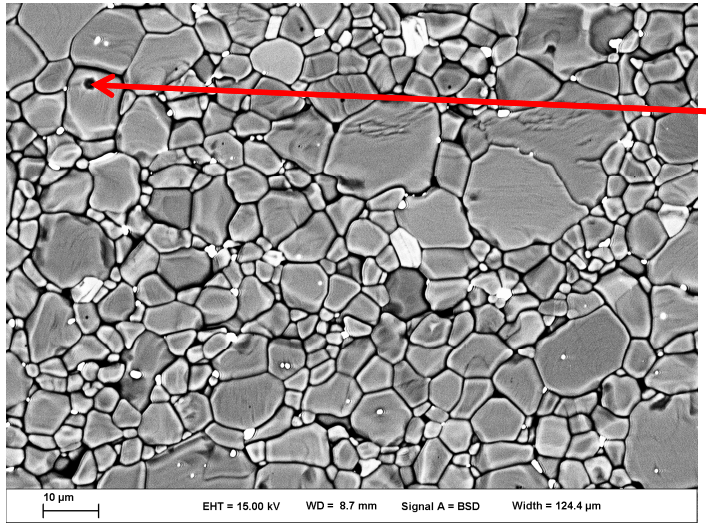
- Porosity one three different size scales
  - Between adjacent strands (results from tool path choice)
  - Within a strand (from air bubble introduced during compounding)
  - Between and within grains (incomplete densification during sintering)



Between strands

Within strand

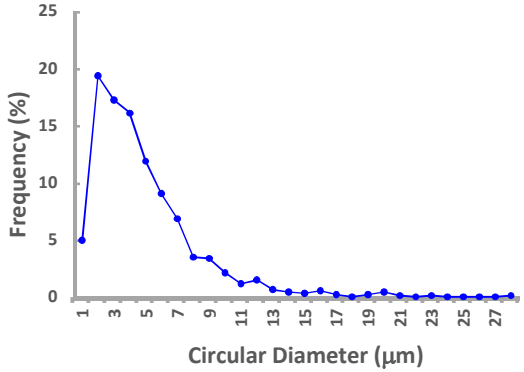




intragranular

- Sintered microstructure within strands is typical of many alumina ceramics

- Grain size distribution compares to initial particle size distribution in source material



Lisa Deibler
Amy Allen
(1819)

Full Disclosure – The Good, The Bad, and the Ugly

- The Good
  - Thermoplastics are a mature field – can be based on compositions from the compounding, extrusion and injection molding (LPIM)
  - Relatively low melting points can be realized
  - Ceramic feedstock (dynamic fluid) can be dispensed via a nozzle as a nearly endless filament (similar to FDM and robocasting) but also discontinuous as droplets by micro-dispensing technology
    - Potential for the realization of very fine structures with smaller tolerances
  - Minimal aging
  - "Green" Machine
  - Characterized and behaved rheological properties – rheological adaptation as a function of temperature
  - Piggyback MakerBot
  - Suspension solidification can be controlled – heat transfer from the printed suspension to the underlying layer or to the surrounding atmosphere (environment)

Full Disclosure – The Good, The Bad, and the Ugly

- The Bad, The Ugly – The Challenges
  - Specialized equipment and subject matter experts
    - Compounders, extruders, capillary rheometers
    - Print platform with temperature uniformity and monitoring
  - Thermal degradation of the organic constituents
  - Non-flexible filaments
  - Post-processing
    - Thermal pyrolysis of the organics – timely, costly, sensitive – heating rates for thermal debinding may be very low and may need to be carried out in the powder bed – spanning structures and “sagging”
    - Sintering - shrinkage
  - Achievable final densities based on competition between solids loading and rheology
  - Degradation (abrasion) of the equipment components can be very high due to the high solids loaded suspensions

Pie in the Sky

- Multi Tool Platform
  - Multiple print heads
  - Laser Machining
  - Micromachining
- Composite structures / Graded Materials
- Potential for multi material classes – ceramics, metals and glasses living together on the same platform