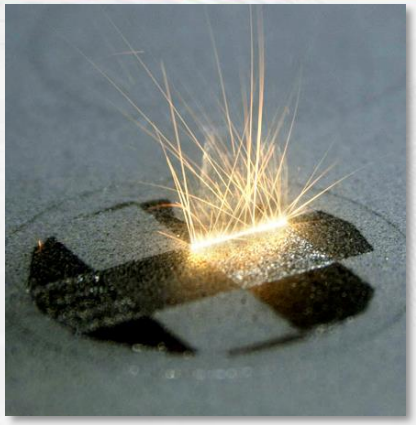
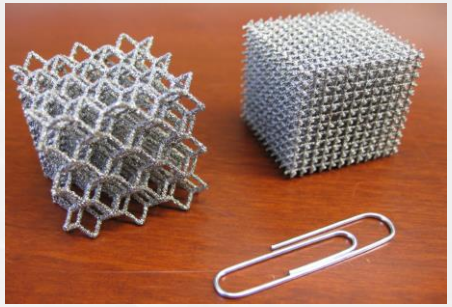


# Key Parameters and Issues:

## Effects of Particle Size, Particle Size Distribution, and Particle Morphology



*Justin Whiting*[1], Ed Garboczi[1], Michelle Bernhardt[2], Stephen Geer[2]
[1]NIST; [2]University of Arkansas

UNIVERSITY OF ARKANSAS

NIST
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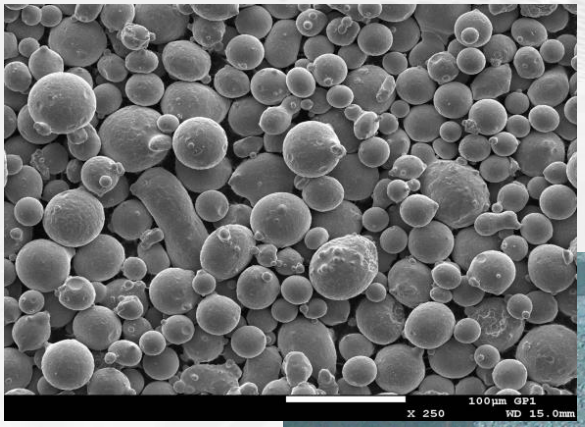
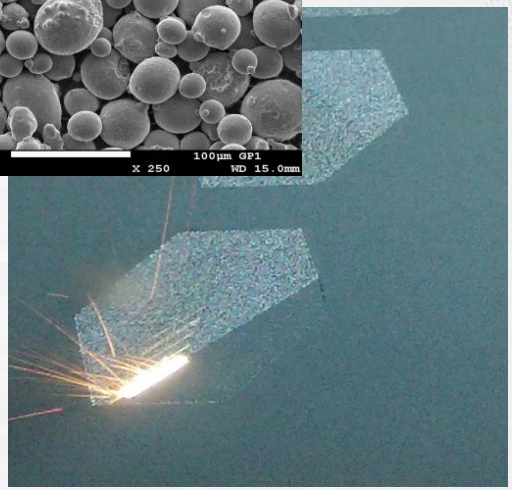
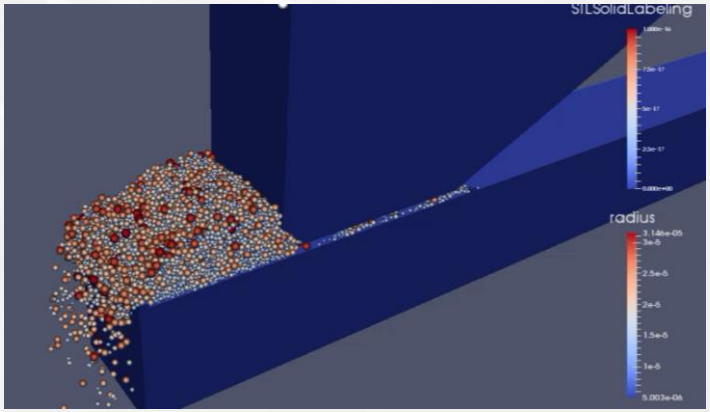
# Overview

- Powder Spreading in Laser Powder Bed Fusion
  - **<u>Variability in PS(D) of spread powder</u>**
  - Density
  - Height
- Discrete Element Method (DEM) Modeling
  - Predict how powder will behave when spread
  - Correlation of Model/Empirical
- PS(D)/Morphology Measurement
- Rheology (time permitting)

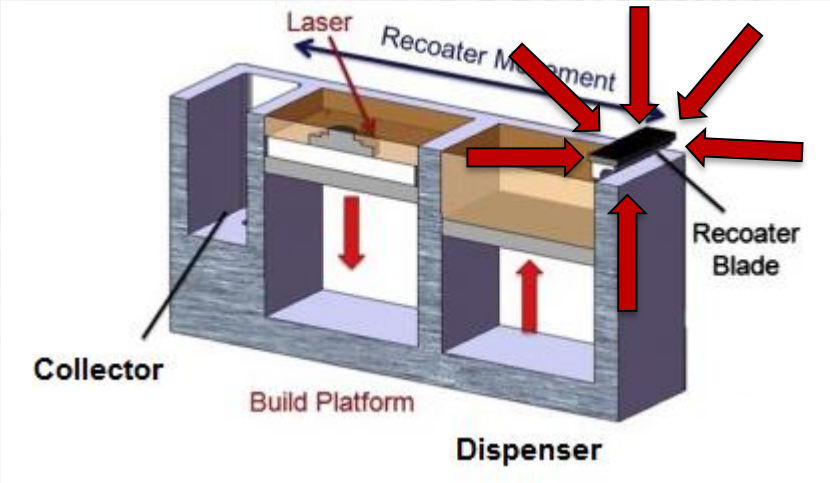


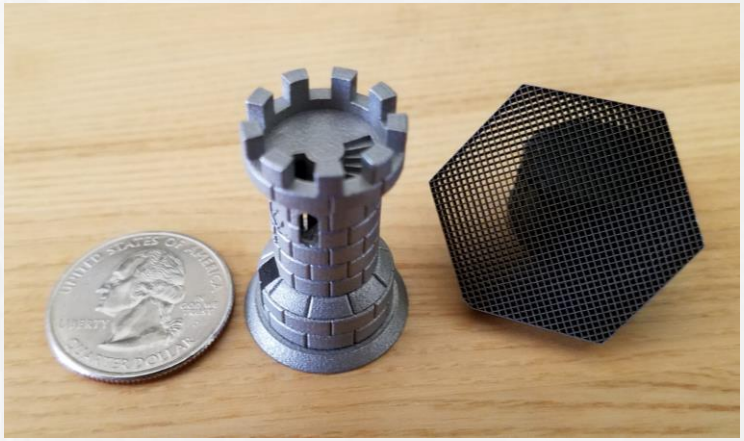


# LPBF Process

- Complex process with final properties dependent on solidification rates
- Rates of solidification altered by:
  - Residual heat from parts in vicinity
  - Time between subsequent scans or layers
  - Performance of ‘Air blanket’ if Present
  - Part geometry & Scan strategies
  - many more….

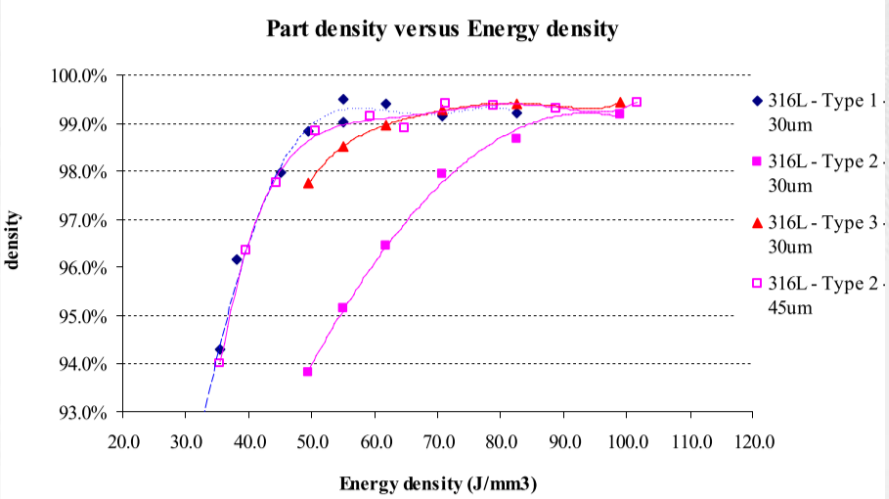



*General powder bed fusion machine layout [1]*

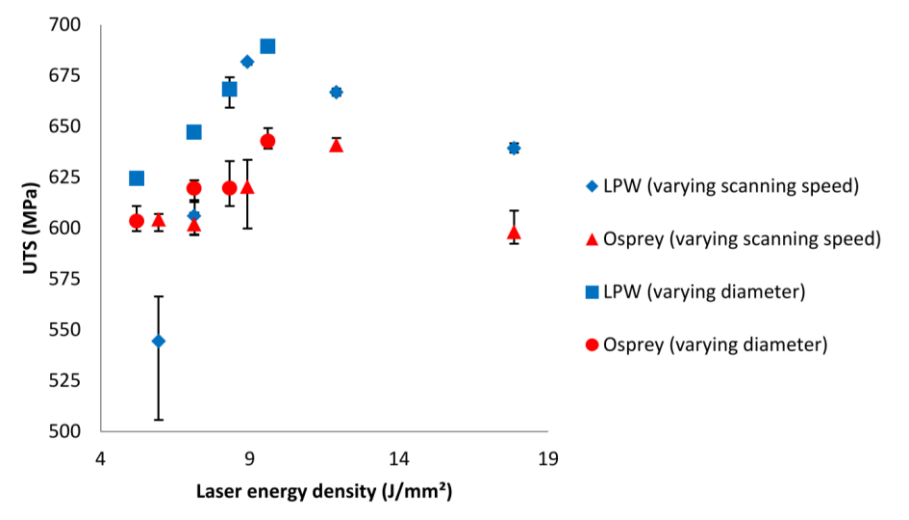


# Feedstock/Material Property Relationship

- Literature Review:
  - PSD and PBF:
    - Yadroitsev *et al*: single track geometry [2]
    - Spierings *et al*: apparent, tapped, and final part density, roughness, UTS [3]
    - Liu *et al*: apparent, tapped, powder bed, and final part density, UTS, hardness, roughness [4]



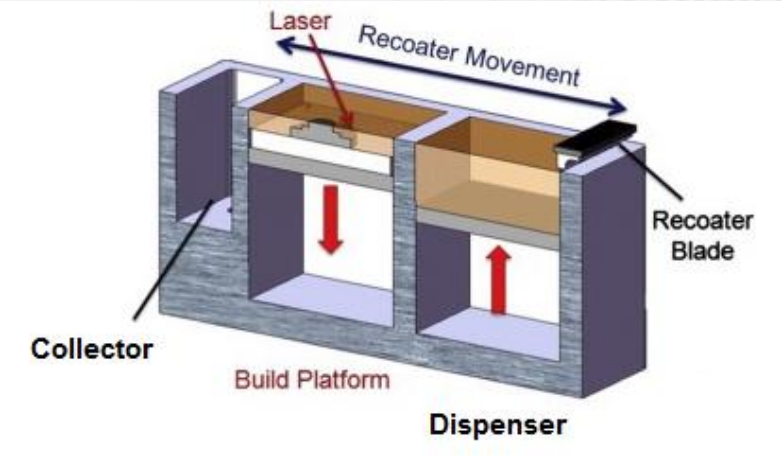

*Density and PSD [3]*



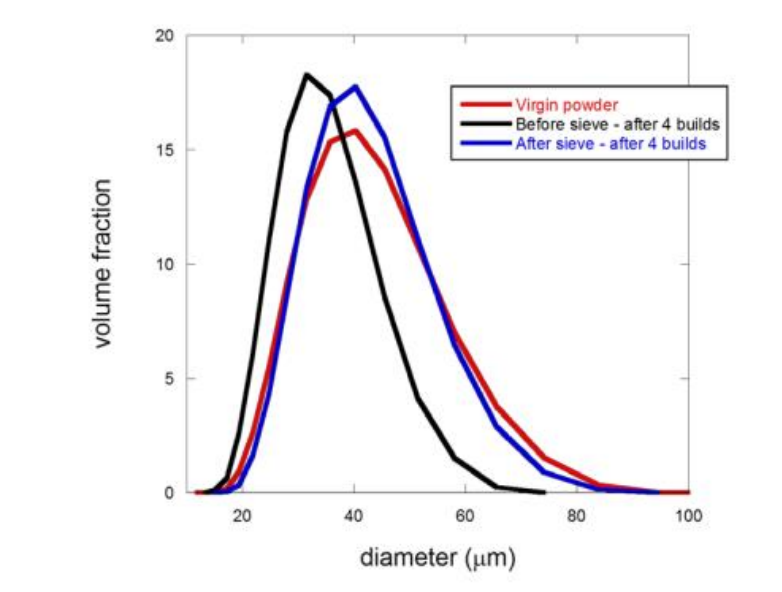

*UTS and PSD [4]*

# Slotwinski: effects of recycling [5]

- Recycling and PSD
- PSD of virgin
- 4 Builds
  - PSD of powder before sieve
  - PSD of powder after sieve
- Sieving makes powder smaller!!
- Preferential spreading of large particles

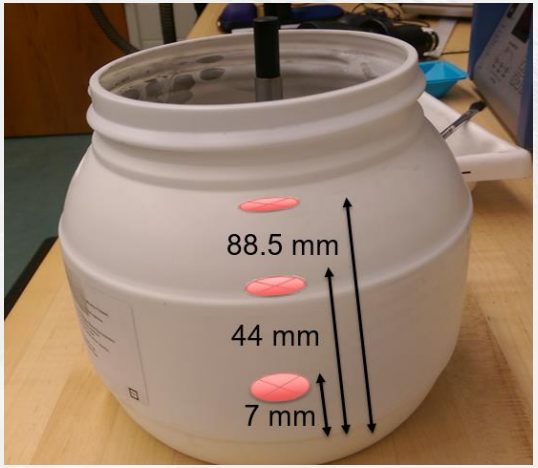



*General powder bed fusion machine layout [1]*

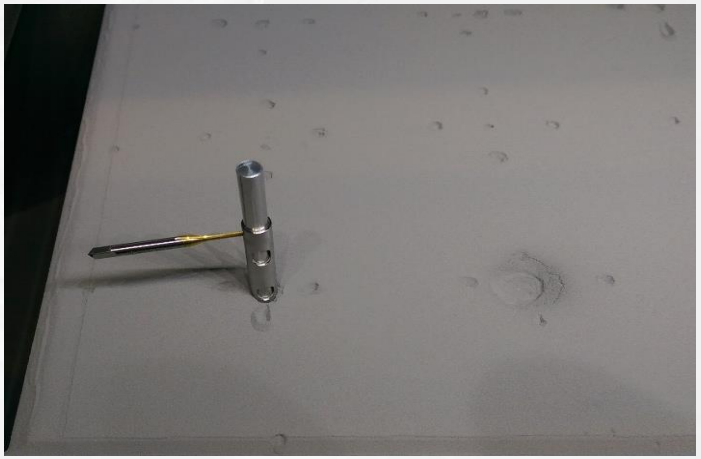



*Effects of recycling [5]*

# Sources of Variability

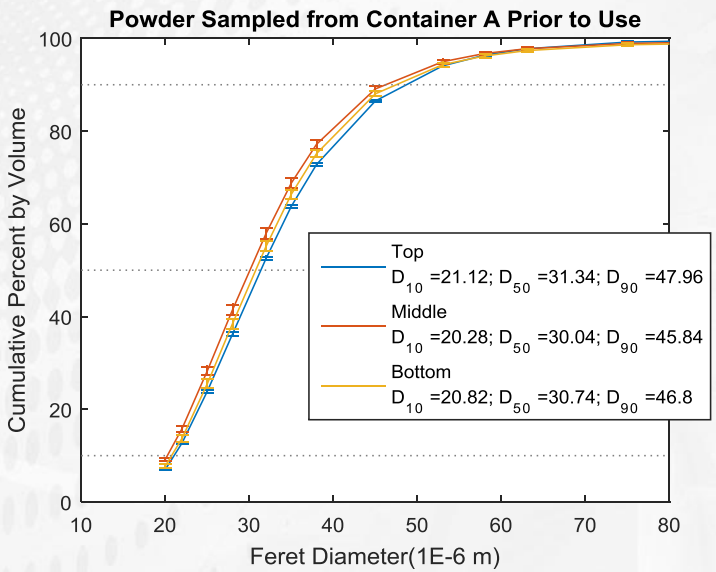


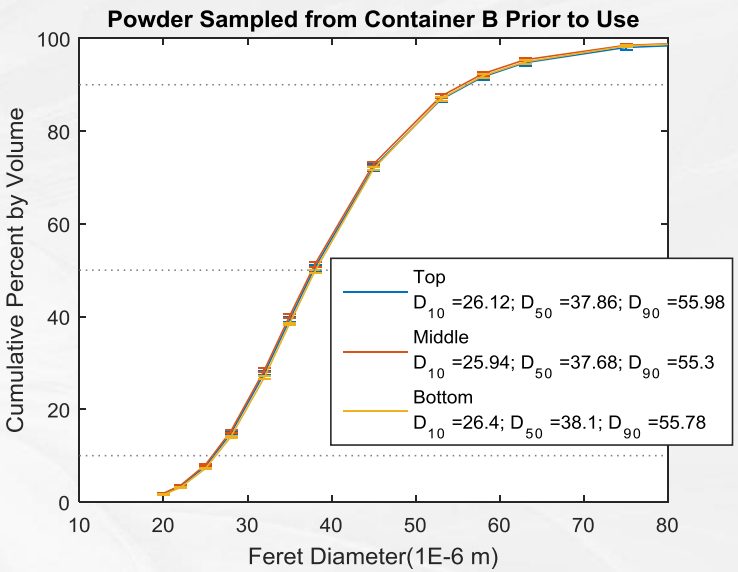

- Potential sources in:
  - Container
  - Loose Powder Bed
  - On/Off Part
- Situation specific sampling
  - Loose bulk powder
  - Powder on part

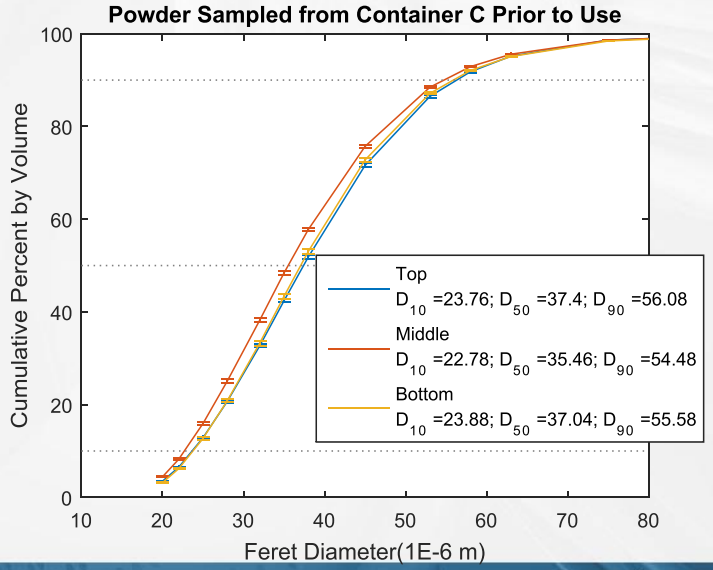


Whiting, J. G., & Fox, J. (2016). “Characterization of Feedstock in the Powder Bed Fusion Process: Sources of Variation in Particle Size Distribution and the Factors that Influence them.” In 25th Annual International Solid Freeform Fabrication Symposium - An Additive Manufacturing Conference, SFF 2016.

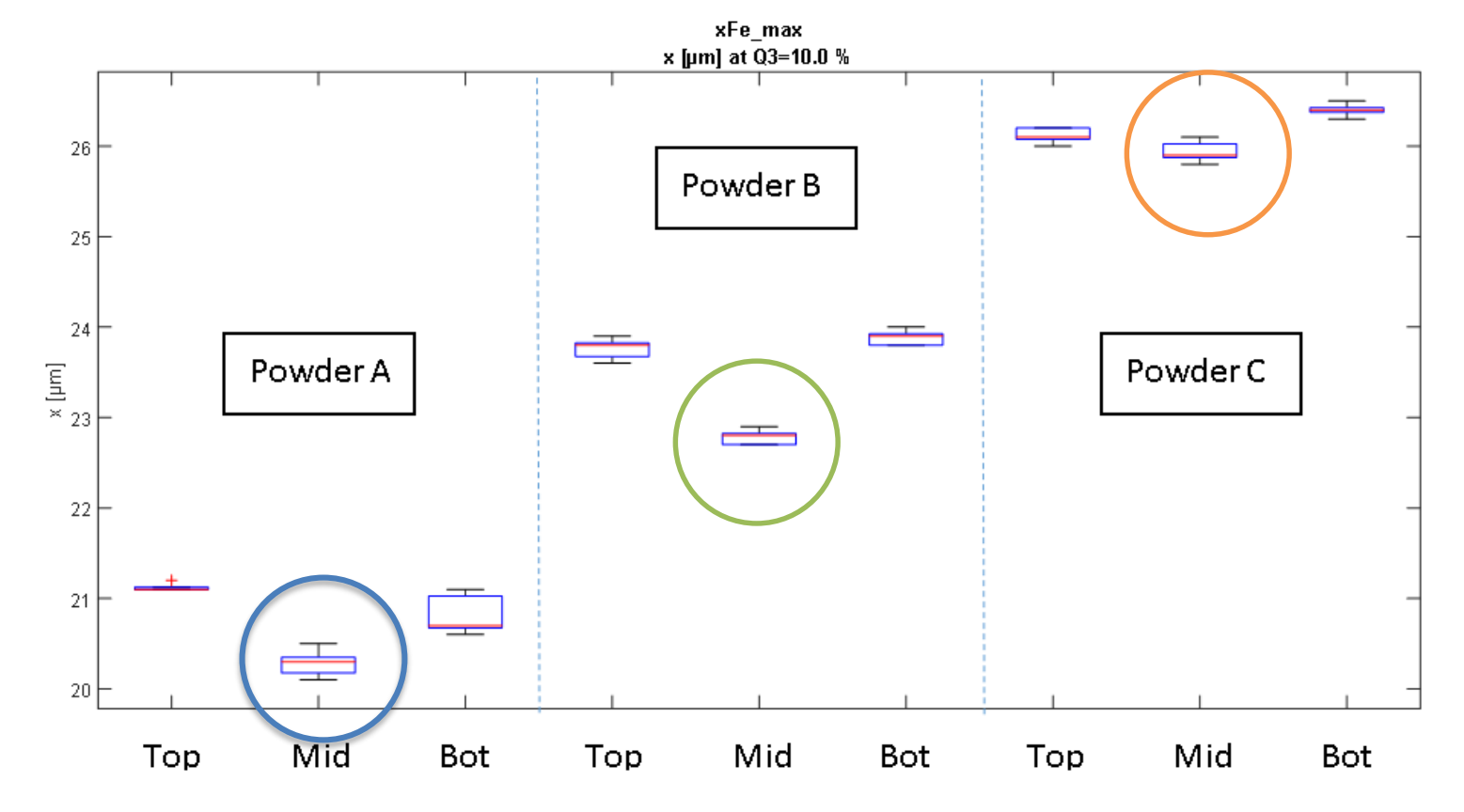
# Powder As-received



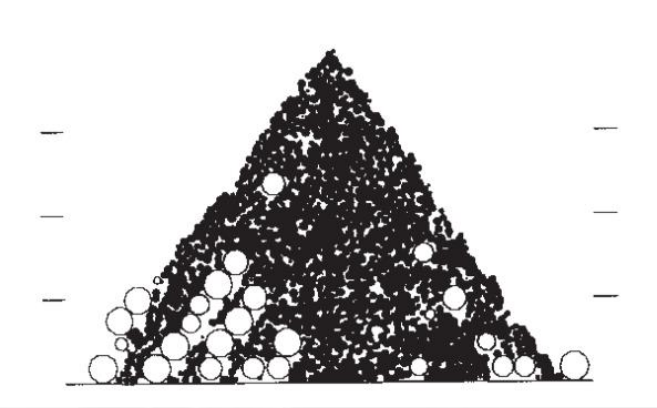



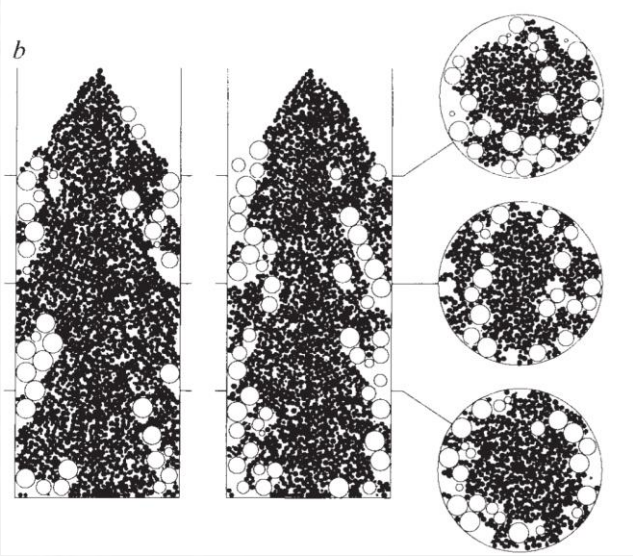
# Powder As-Received



# Particle Segregation

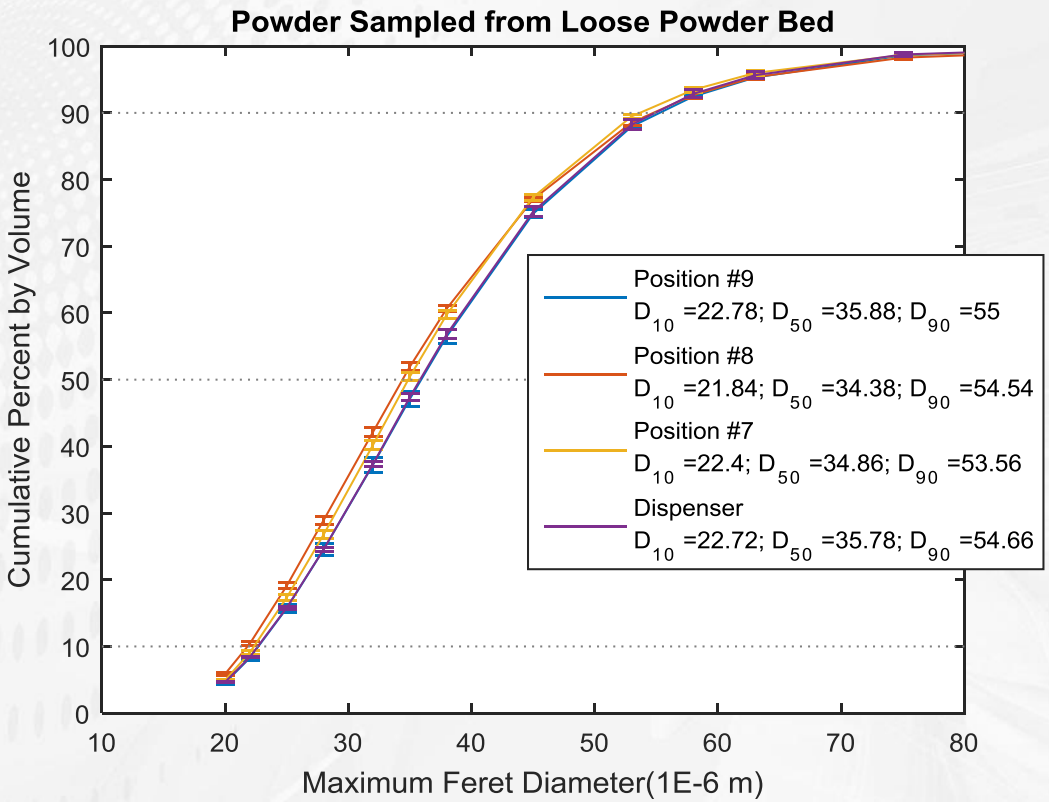


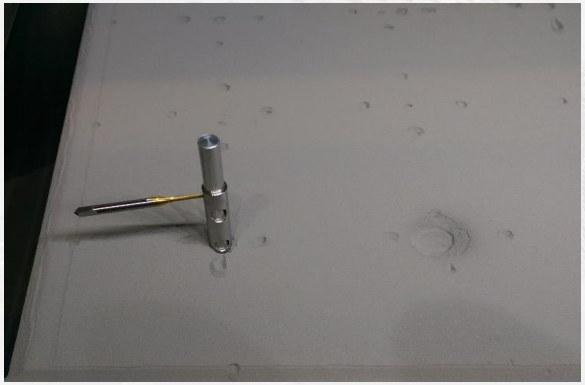
- Published in Nature, 1990
- Simulation of bidisperse pouring of powder
- Fines concentrated centrally
- Has since been shown empirically
- Handling leads to fines concentrated vertically as well?

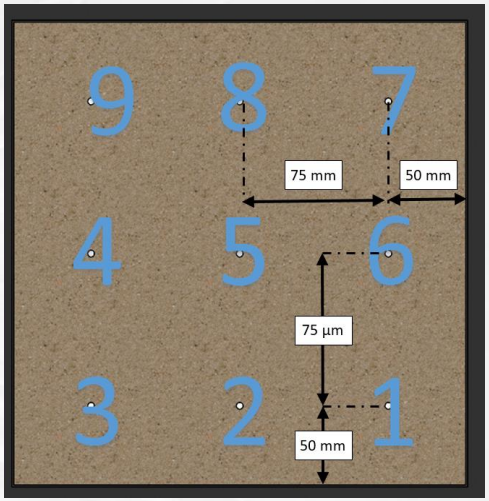



*Simulation of the deposition of bidisperse powder [9]*

# Loose Powder Bed



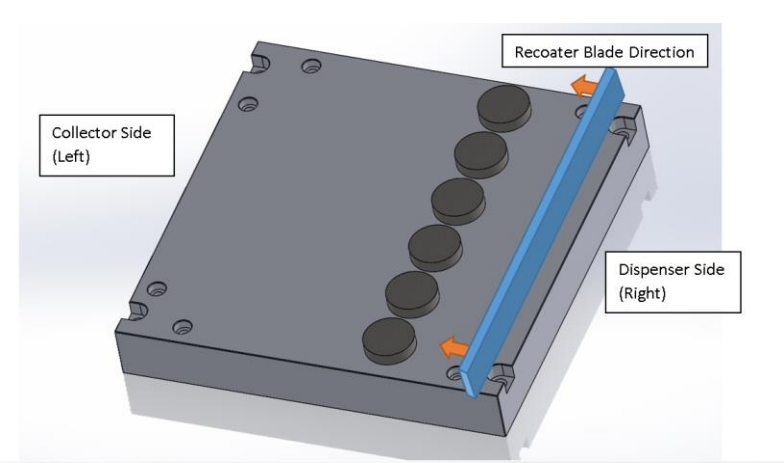



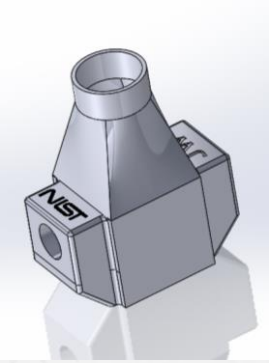



| | $D_{10}$ | | $D_{50}$ | | $D_{90}$ | |
|---|---|---|---|---|---|---|
| Powder Location | Average (µm) | σ (µm) | Average (µm) | σ (µm) | Average (µm) | σ (µm) |
| 7 | 22.4 | 0.12 | 34.86 | 0.17 | 53.56 | 0.29 |
| 8 | 21.84 | 0.09 | 34.38 | 0.22 | 54.54 | 0.51 |
| 9 | 22.78 | 0.19 | 35.88 | 0.36 | 55 | 0.19 |
| Dispenser (center) | 22.72 | 0.08 | 35.78 | 0.16 | 54.66 | 0.74 |

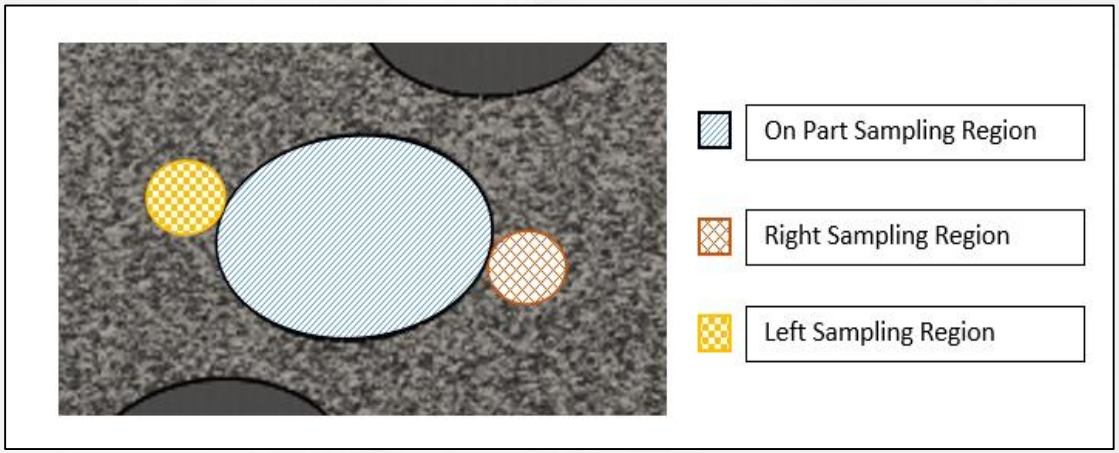
# On/Off Part Sampling

- Collector/Dispenser = Left/Right =
- Vacuum apparatus w/ filter paper

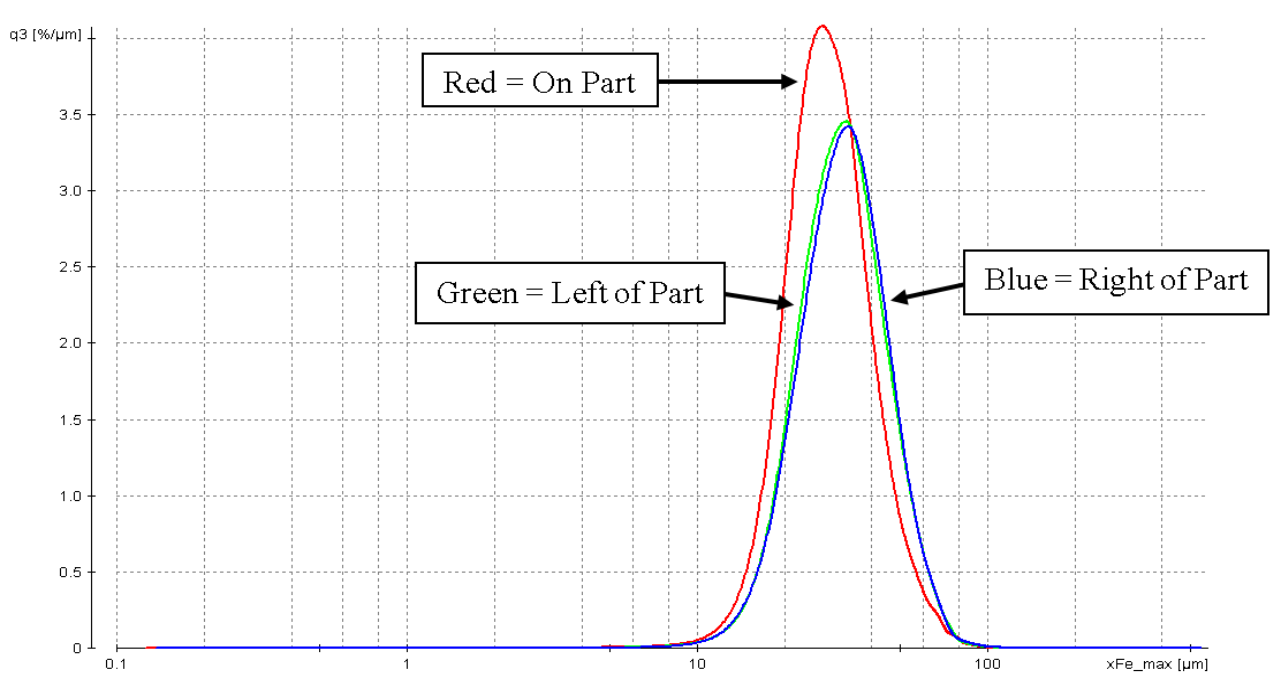


*Vacuum throttle apparatus*



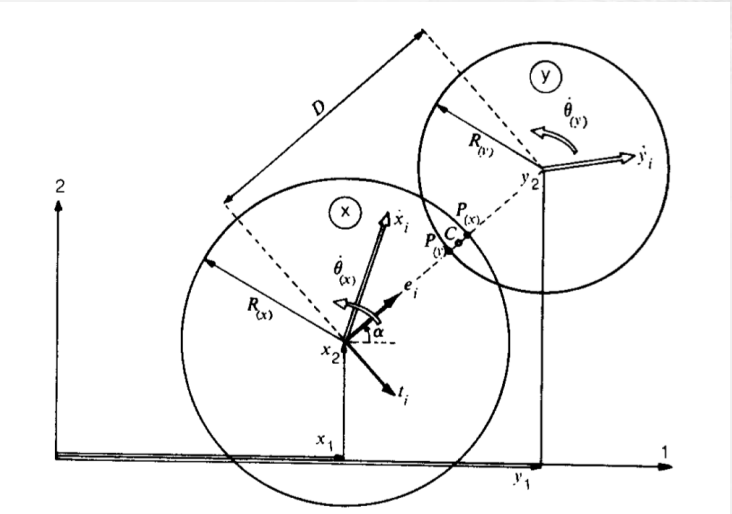
# Distribution of Powder On Part vs. Powder Bed



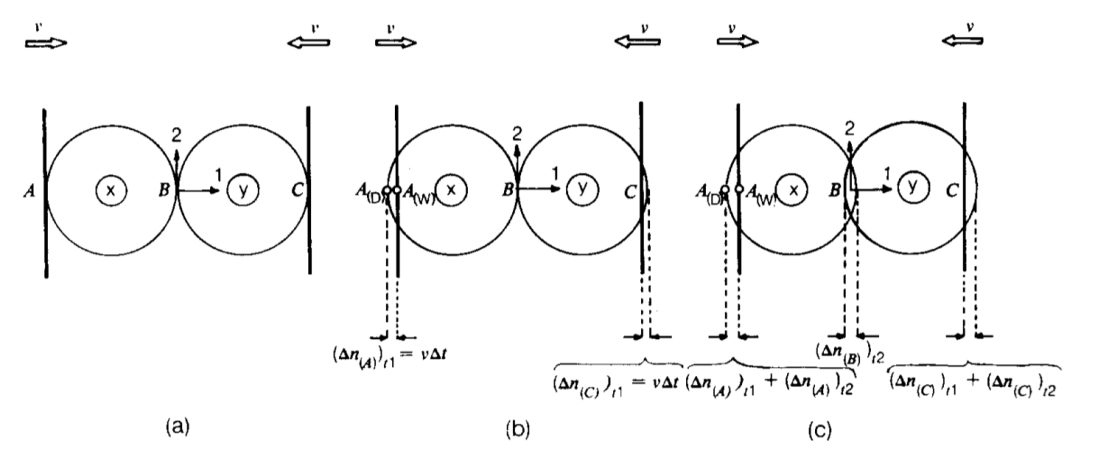

- Shift of magnitude
- Shape of distribution
- Different packing density?
- Obvious variance in PSD of powder on part when comparing to bulk powder

# Discrete Element Method (DEM) Model

- Cundall (1971)
  - Rock Mechanics
- Force-Displacement
- Newton's 2nd Law
- Exciting potential to predict spreading performance




*Discrete Element Method Model: force-displacement model[6]*




*Discrete Element Method Model: two discs compressed between rigid walls[6]*

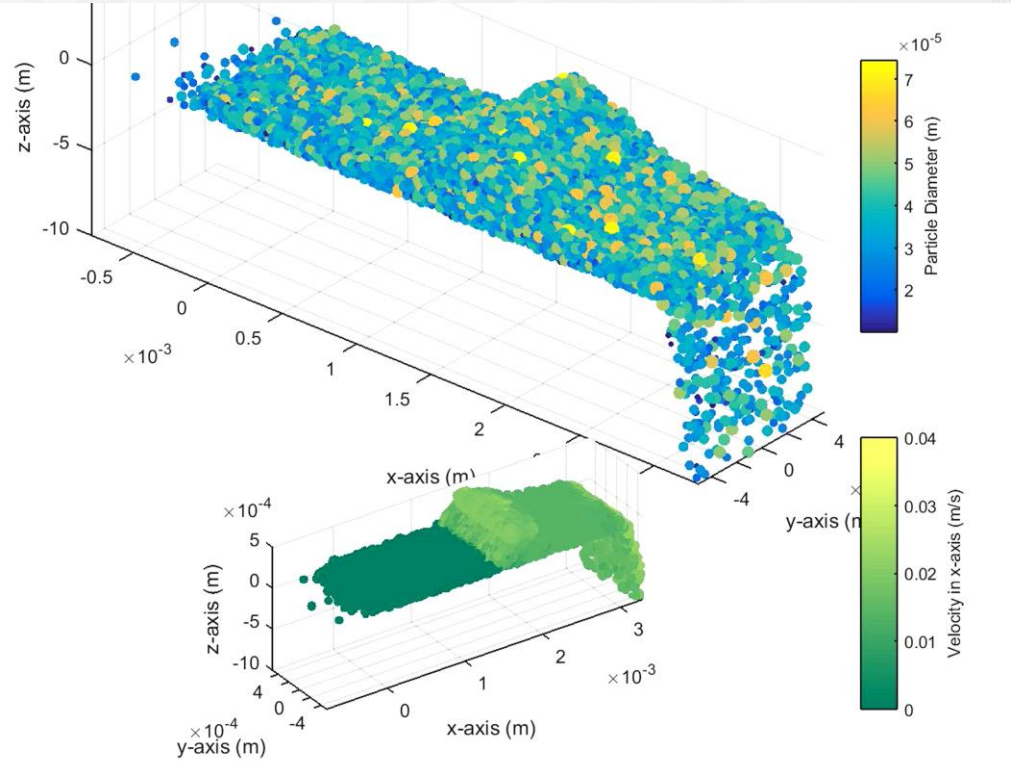
# DEM Model Basics

- Stiffness
- Coulomb friction
- Damping ratio
- Rolling friction

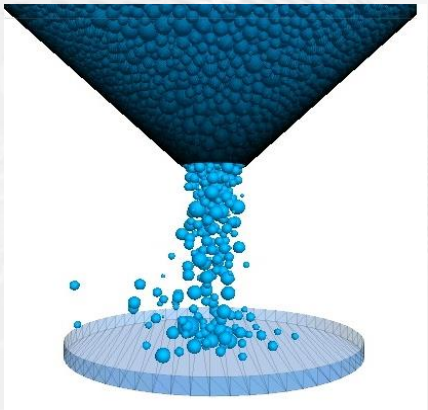
✓ $\Delta Fn = k_n(\Delta n)$

✓ $\sum F(x)_i = m(x)\,\ddot{x}_i$

✓ $\sum M(x) = I(x)\,\ddot{\theta}_i$



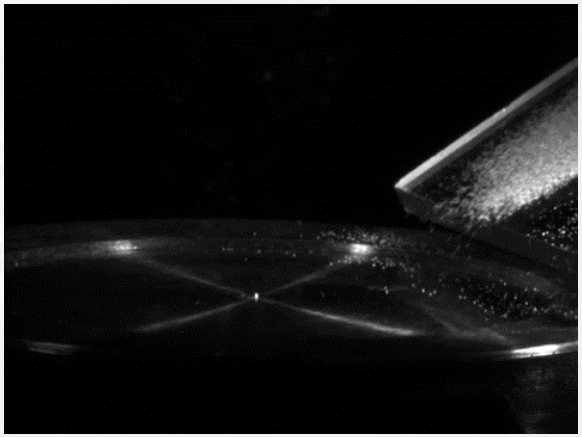
# Calibration of DEM Model



- Coefficient of Restitution (COR)
  - High speed imaging of dropped particles
- Angle of Repose (AOR)
  - Similar to ASTM D6393-14 (…Carr Indices), but with Hall Flowmeter Apparatus

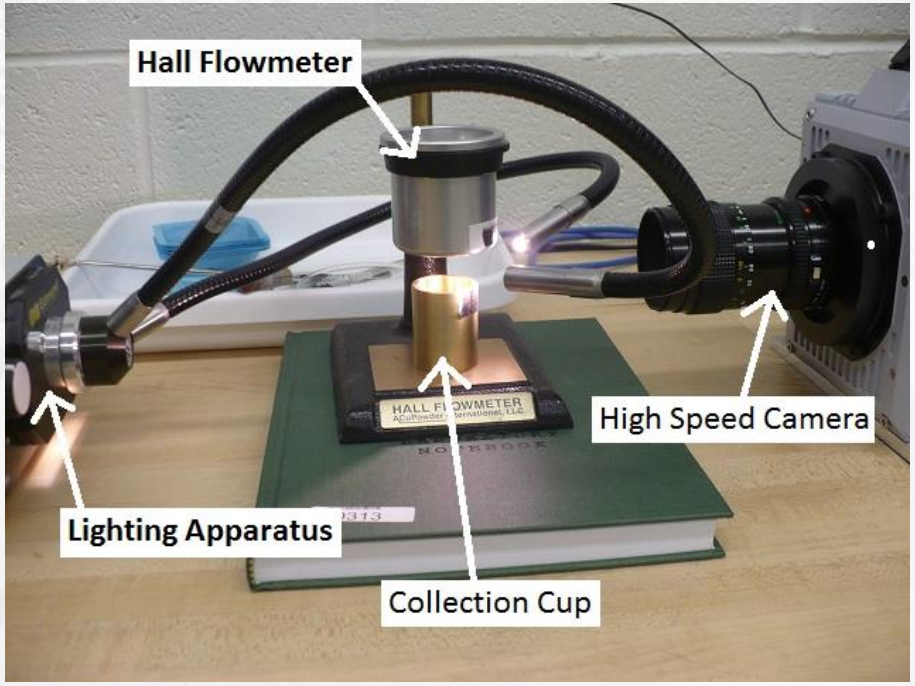
| Parameter | Values/Type Considered |
|---|---|
| Simulation method | Cloud, Funnel |
| Contact model<br>Particle Young's modulus | Linear, Hertz-Mindlin<br>200 MPa, 200 GPa |
| Coulomb friction coefficient, $\mu_C$ | 0.1, 0.3, 0.5 |
| Critical damping ratio, $\beta_n$ | 0.1 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9 |
| Rolling friction coefficient, $\mu_r$ | 0.05, 0.15, 0.20, 0.30, 0.05 |

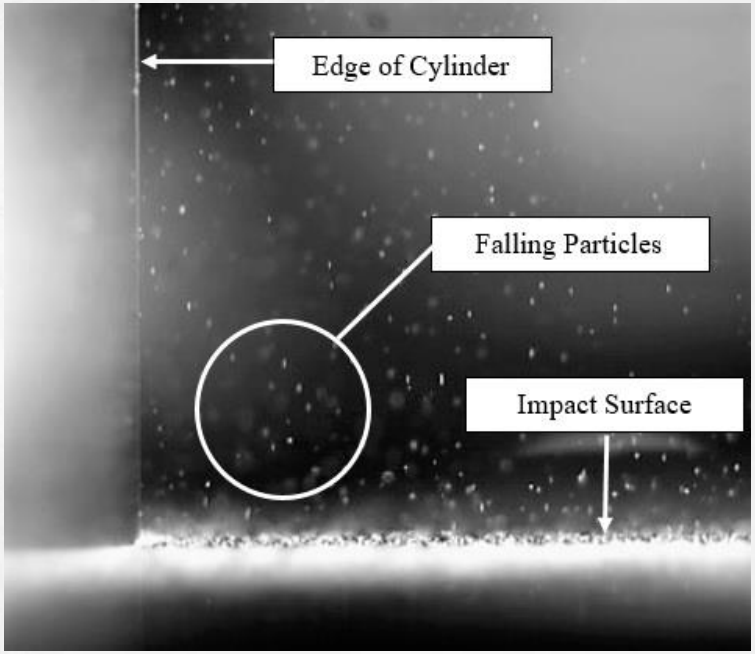
*Parametric studies conducted using PFC3D and LIGGGHTS [7]*



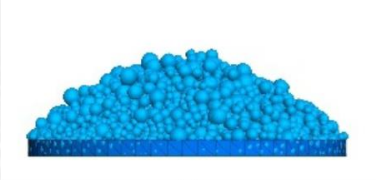
Geer, Stephen, Bernhardt, Michelle L., Garboczi, Edward J., Whiting, Justin G., Donmez, Alkan M. "A More Efficient Method for Calibrating Discrete Element Method Parameters for Simulations of Metallic Powder Used in Additive Manufacturing," to appear in *Granular Matter.*

# DEM Calibration: COR

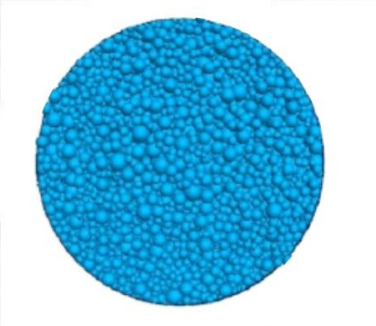


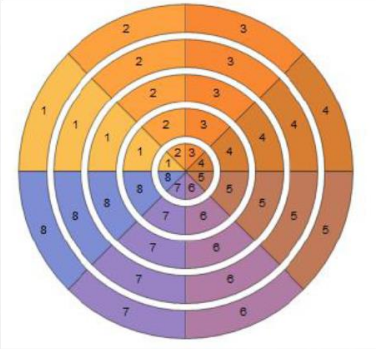
# DEM Calibration: AOR



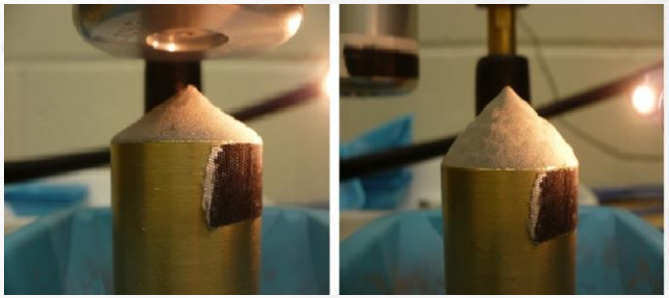
(a)

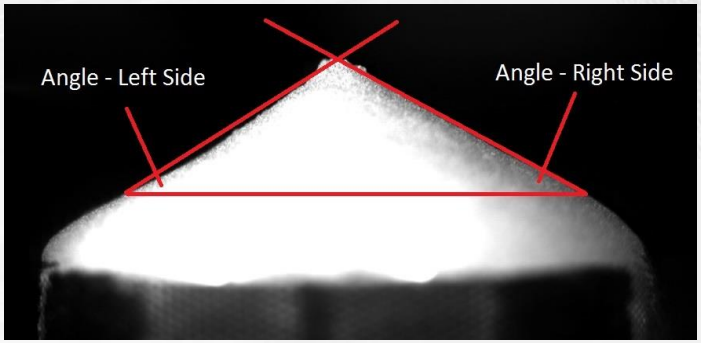


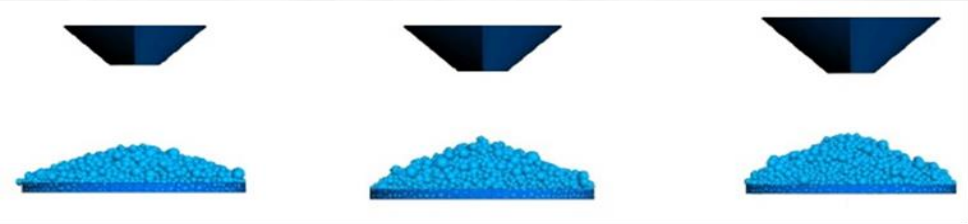
(b)



(c)



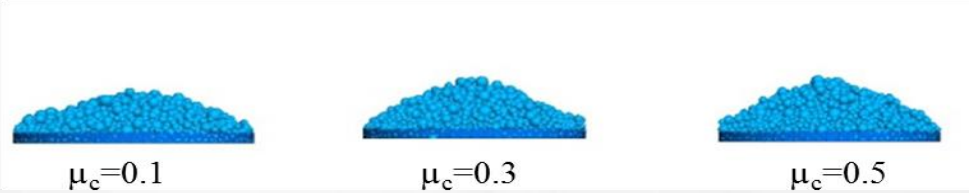


$\mu_c$=0.1 $\mu_c$=0.3 $\mu_c$=0.5

(a)

- Funnel Method



$\mu_c$=0.1 $\mu_c$=0.3 $\mu_c$=0.5

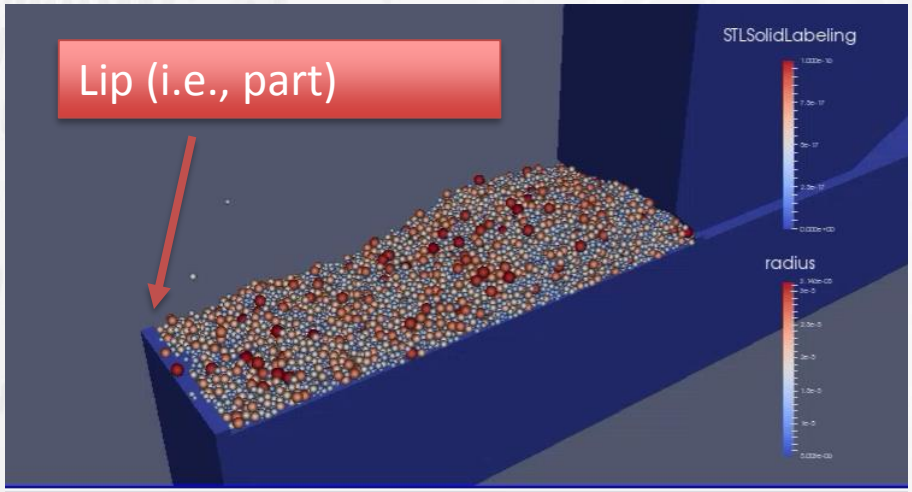
(b)

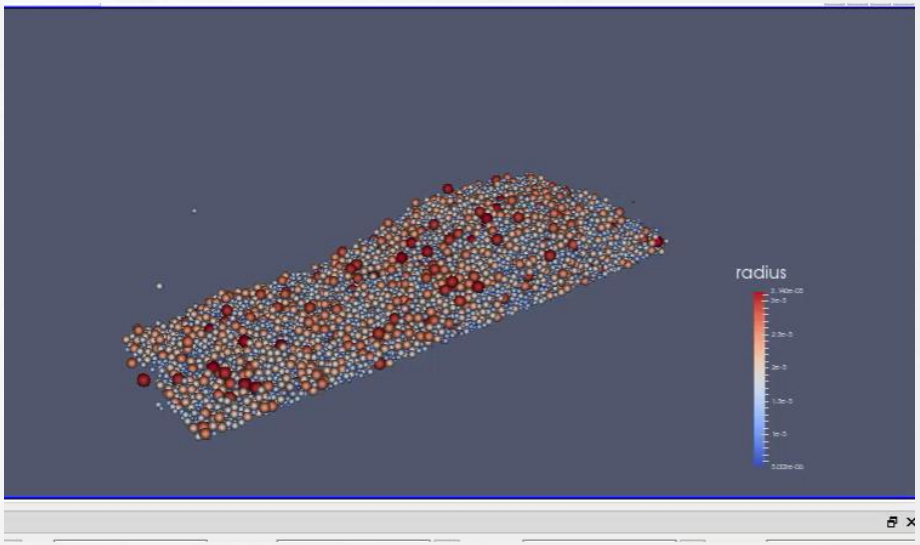
- Cloud Method

# Early Model

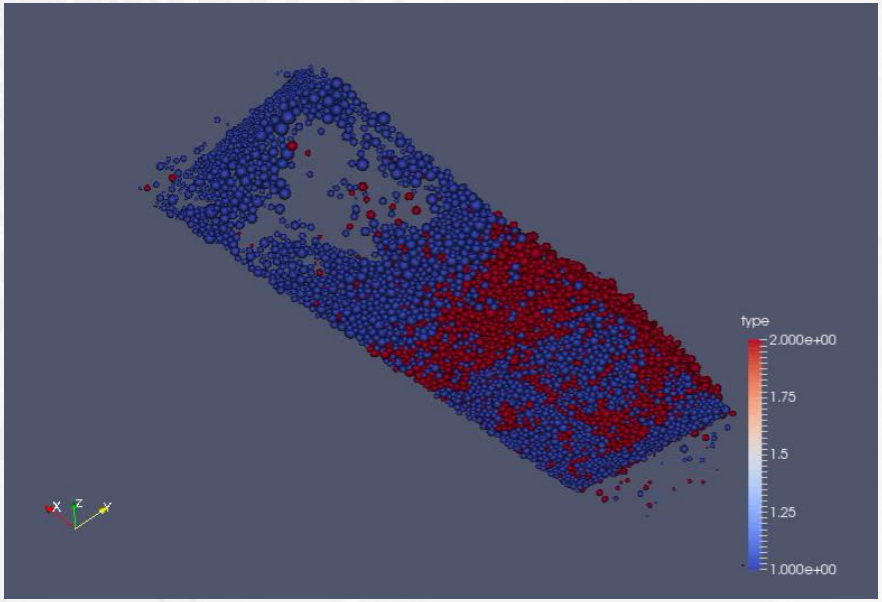
- Blade speed: .02 m/s
- Blade width: 1 mm
- Linear Contact Model
- ~13,000 particles

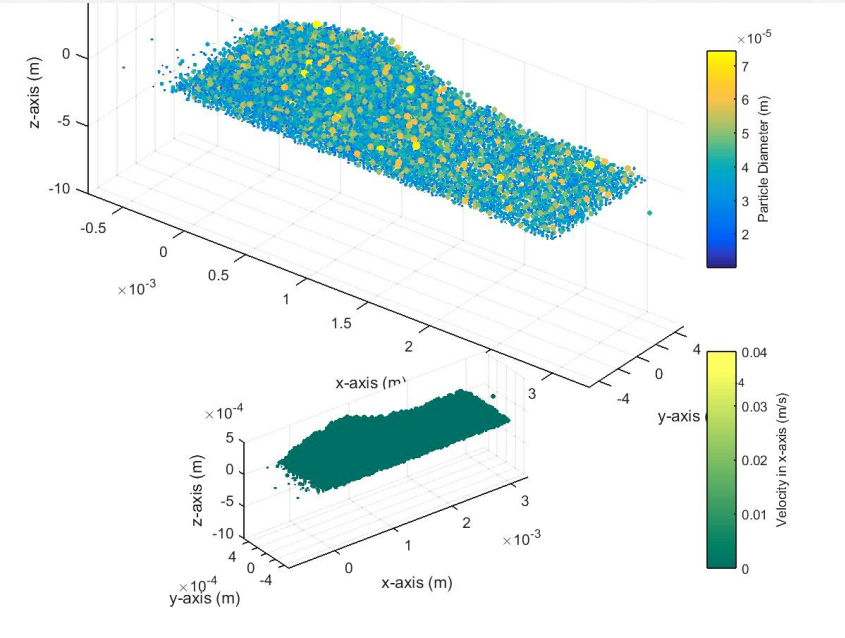
- 35 μm gap between arm and lip
- Lip is 35 μm high
- Coulomb Friction μ=0.3
- Rolling Friction μ=0.3



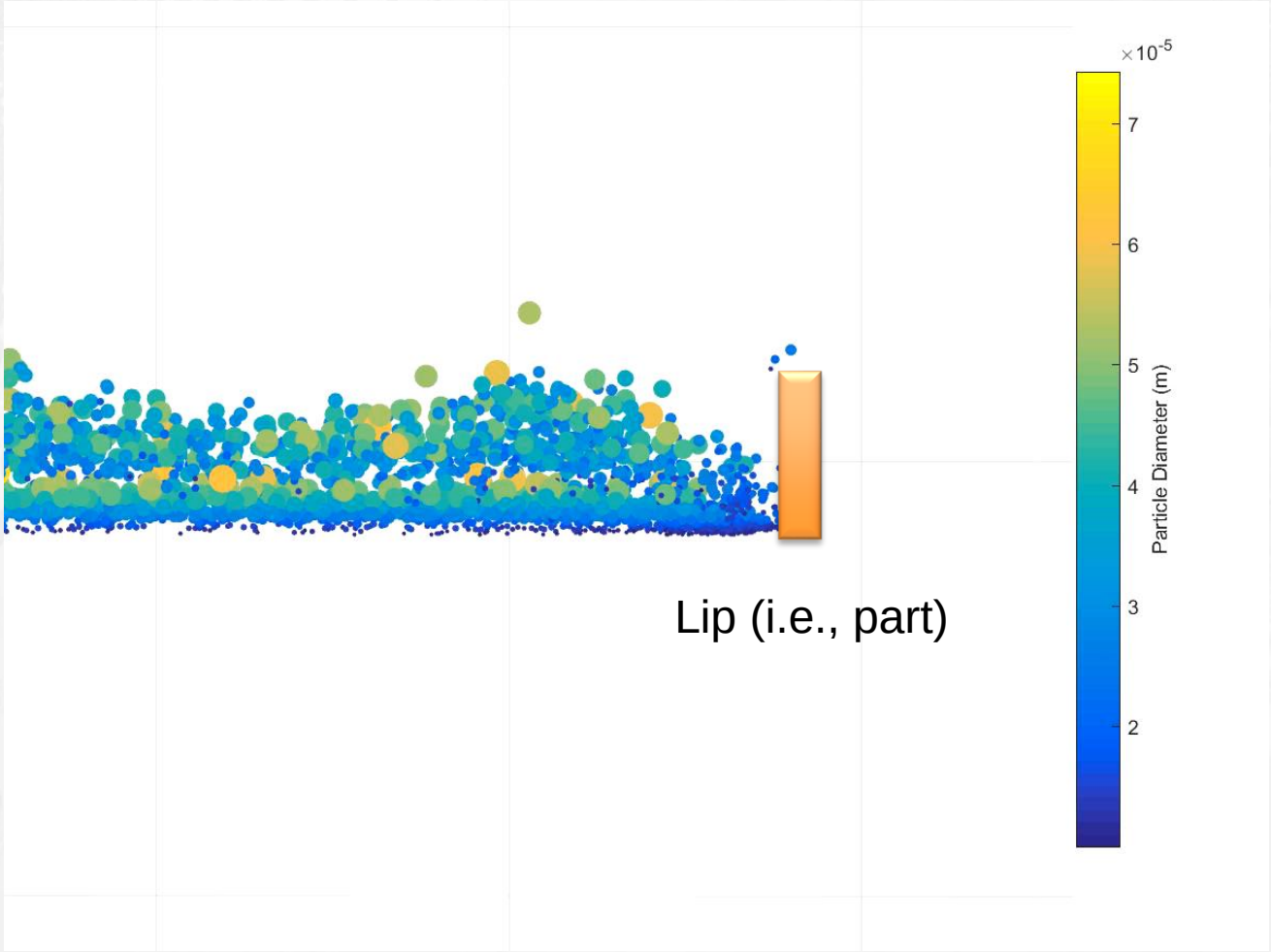
# Multi-layer Spreading




- View from bottom of substrate
  - Red = 2nd layer powder
  - Blue = 1st layer powder
- Uniform PSD along spreading direction

# Loose Powder Bed - Part Interface




- 3rd Spread
- 110 µm gap between recoater and substrate
- 35 µm gap between part and recoater
- Fines concentrated near part/powder interface

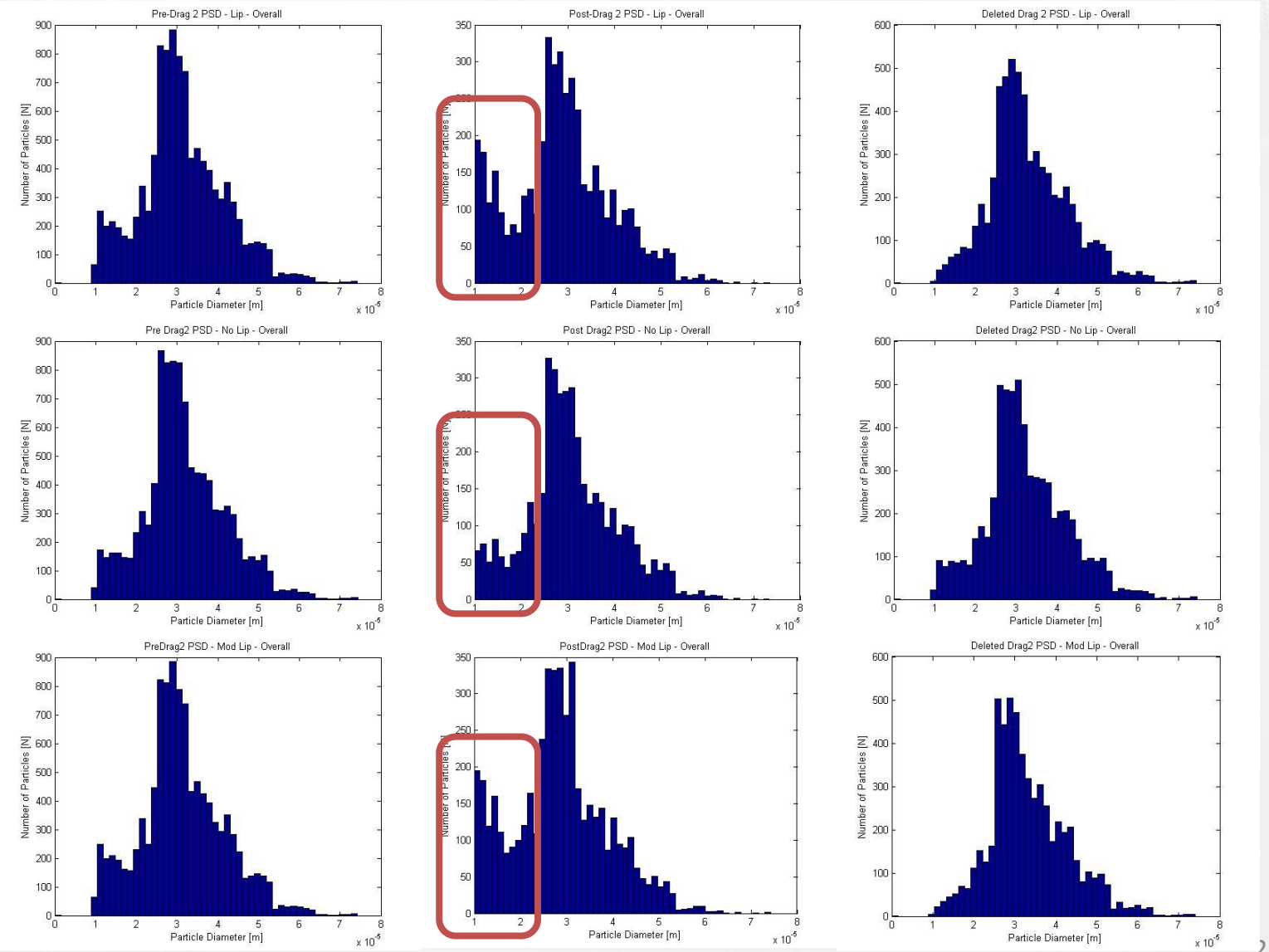
# PSD of Spread Powder

Pre-spread | Post-spread | Over lip

Lip

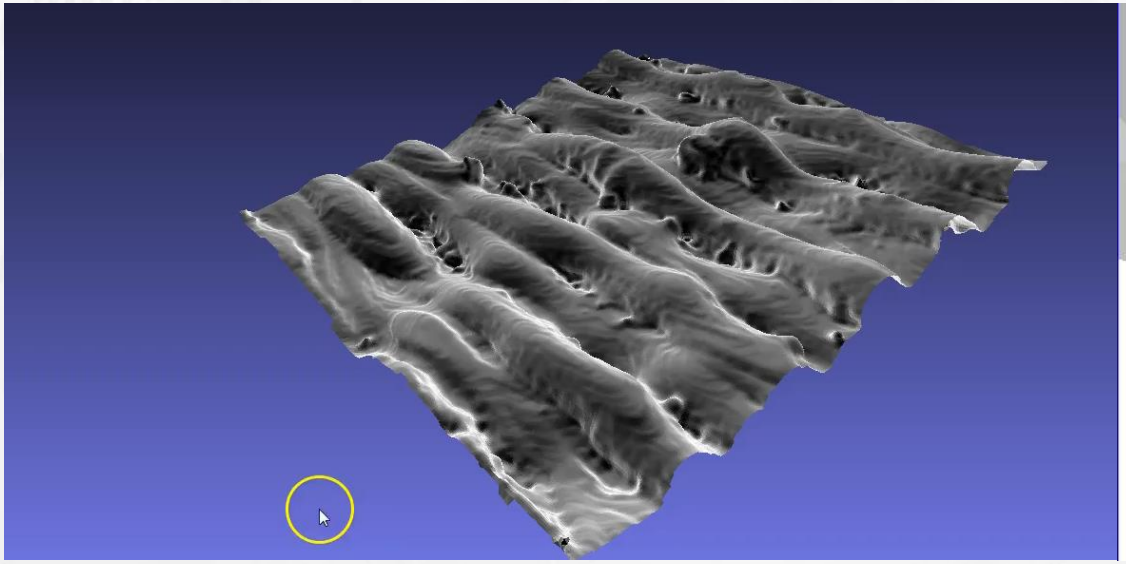
No Lip

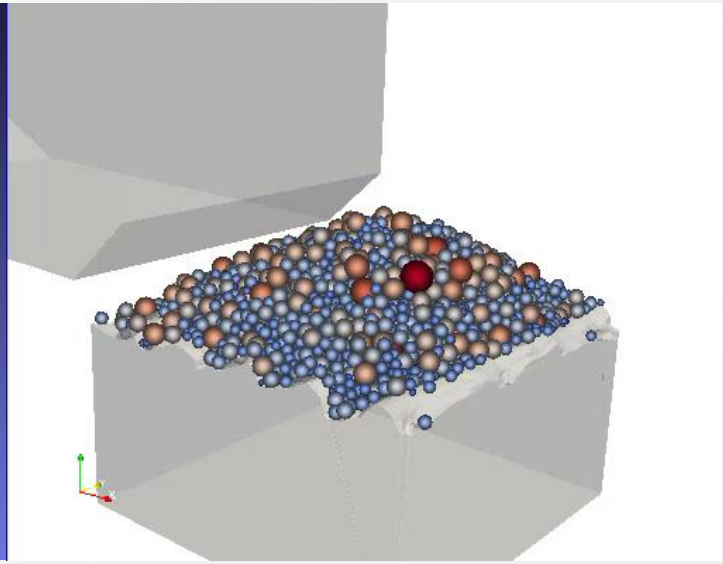
Moving Lip



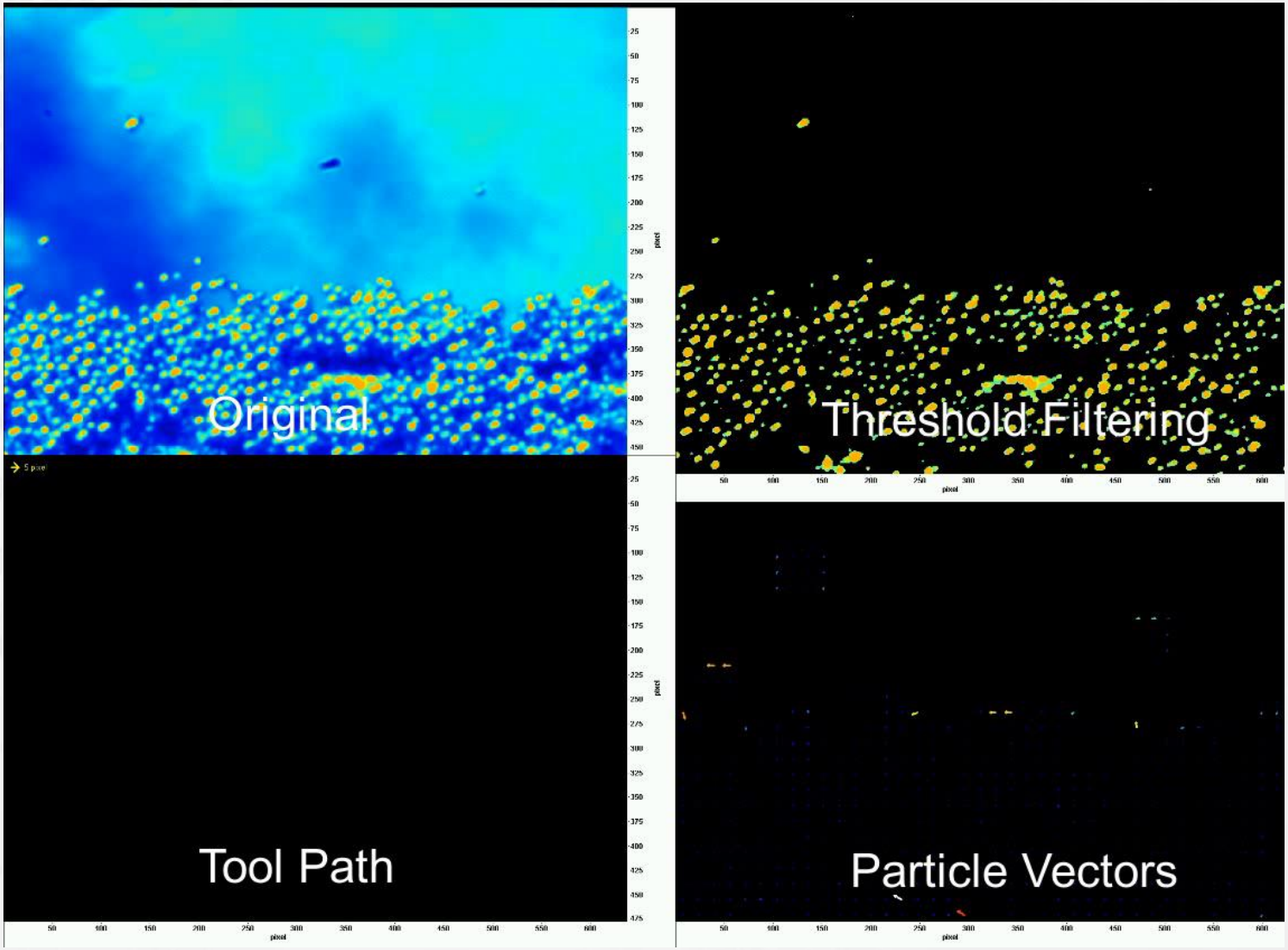
# STL of measured solidified LPBF surface

- Part's surface (measured using scanning white light interferometer) inserted into model
  - Hatch, speed, power
  - Scan orientation
- Charge and spreading method (hopper hybrid vs. stationary dispenser)
- Endless configurations



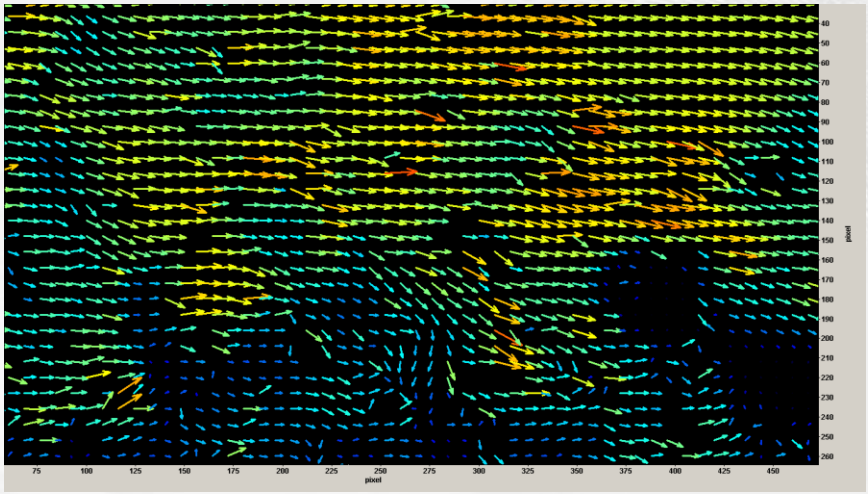


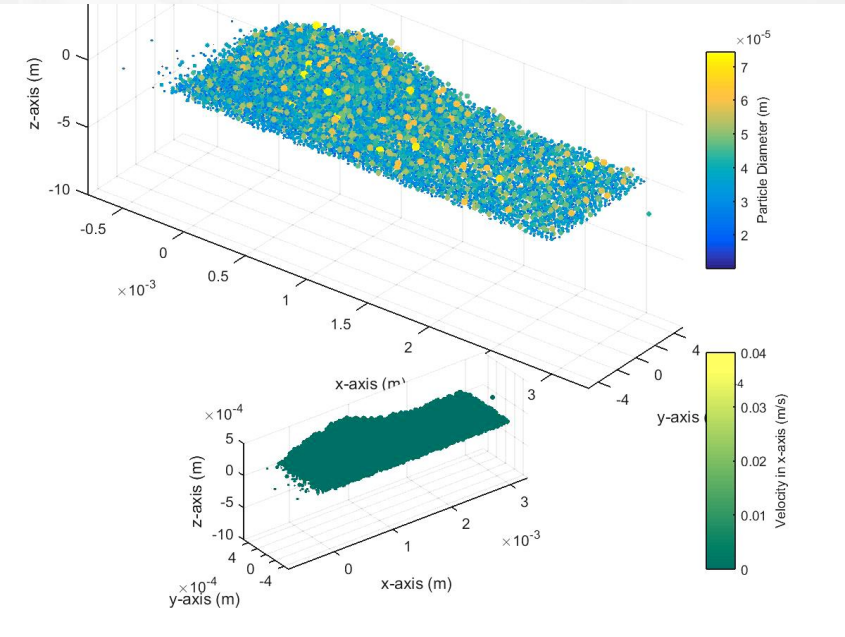
# Preliminary DIC of Powder Spreading



# Capabilities/Potential

- DEM
- High speed imaging
- Layer Density
- Local PSD
- Effect of:
  - Humidity
  - Static Charge
  - Recoater geometry, speed, stiffness
  - PS(D) of powder
  - Charge factor





# Particle Size Measurement, XCT and real world applications

- Comparisons to LD, ASEM, DIA, Sieve provides insight into how each of the technique's mechanisms alter how they measure

**LD ≠ XCT ≠ DIA ≠ Sieve ≠ aSEM**

- Correction factors can be used with little information (e.g., general morphology, size range)
- Standardized reporting!

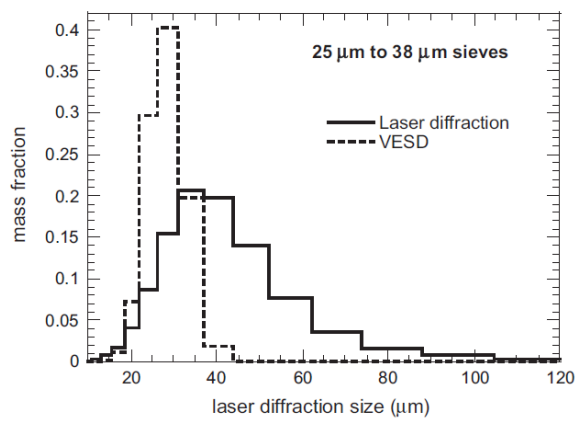



**Fig. 2.** The particle size distribution of the 25–38 µm sieve particles, as determined by laser diffraction and by X-ray CT, which uses the VESD parameter as a measure of particle size.

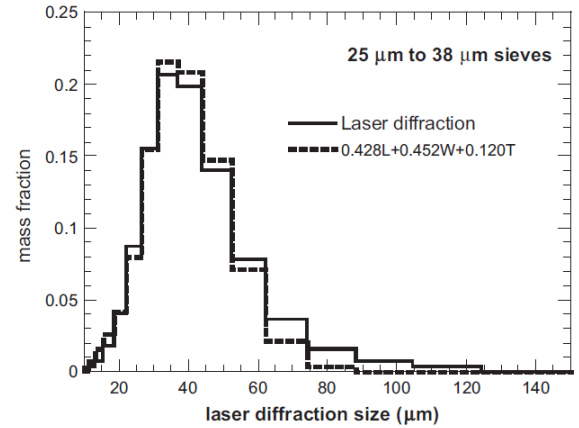



**Fig. 6.** The particle size distribution of the 25–38 µm sieve particles, as determined by laser diffraction and by a weighted sum of the *L*, *W*, and *T* parameters computed from the X-ray CT data.

E.J. Garboczi, K.A. Riding, and M. Mirzahosseini, Particle shape effects on particle size measurement for crushed waste glass, Advanced Powder Technology 28, 648-657 (2017).

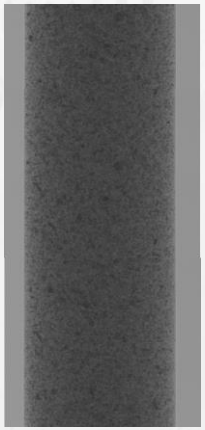
# X-ray Micro CT (Ed Garboczi)

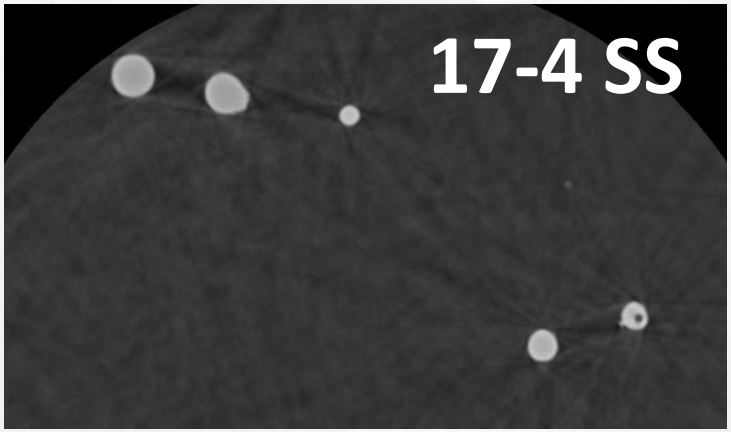
- For additive manufacturing (AM), the shape of the particles affects (at least) spreading characteristics and density of packing
  - Non-spherical single particles; multi-particles, even in virgin powder
- Combination of X-ray microCT and mathematical analysis can give 3D shape characteristics for thousands of particles with only a few scans

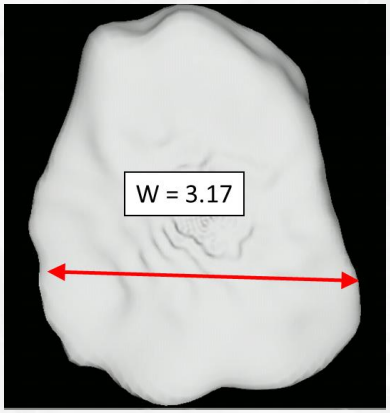

- J. A. Slotwinski, E. J. Garboczi, P. E. Stutzman, C. F. Ferraris, S. S. Watson, and M. A. Peltz, Characterization of Metal Powders Used for Additive Manufacturing, Journal of Research of NIST, 119 (2014) http://dx.doi.org/10.6028/jres.119.018.
- E.J. Garboczi and J.W. Bullard, 3D analytical mathematical models of random star-shape particles via a combination of X-ray computed microtomography and spherical harmonic analysis, Advanced Powder Technology 28, 325–339 (2017).
- R. Cepuritis, E.J. Garboczi, S. Jacobsen, K.A. Snyder, Comparison of 2-D and 3-D shape analysis of concrete aggregate fines from VSI crushing, Powder Technology 309, 110–125 (2017).
- E.J. Garboczi, K.A. Riding, and M. Mirzahosseini, Particle shape effects on particle size measurement for crushed waste glass, Advanced Powder Technology 28, 648-657 (2017).

# X-ray Micro CT (Ed Garboczi)

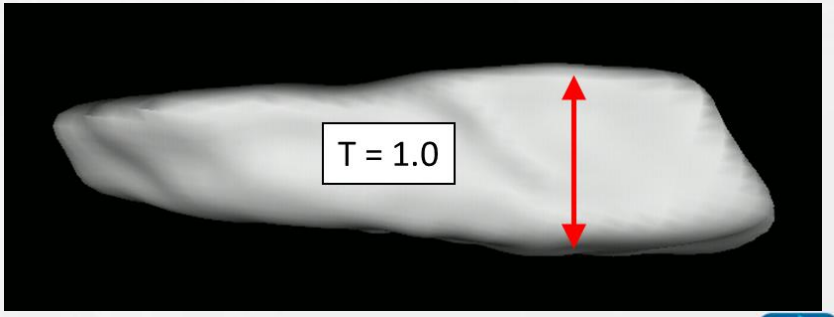
- Embed particles in epoxy cylinder, scan in X-ray CT
- Zeiss Versa XRM500, Skyscan 1172, North Star Imaging X50
- $L \geq W \geq T$ $\quad L \perp W \perp T$





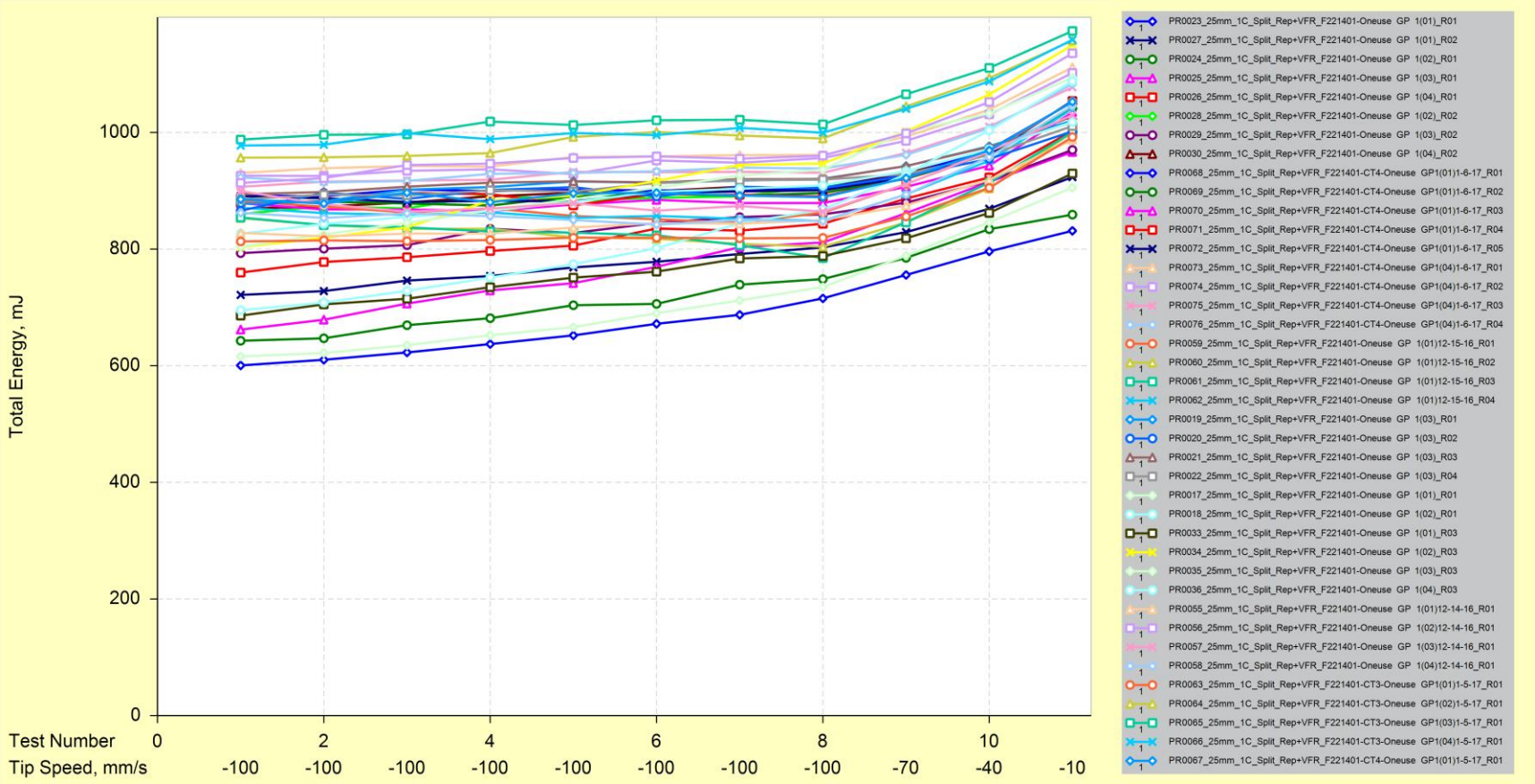

What I have found to be the most useful size/shape parameters: **Length, Width, Thickness**



# Repeatability of Testing with Commercial Rheometer: same material

- ~ 40% change in total energy

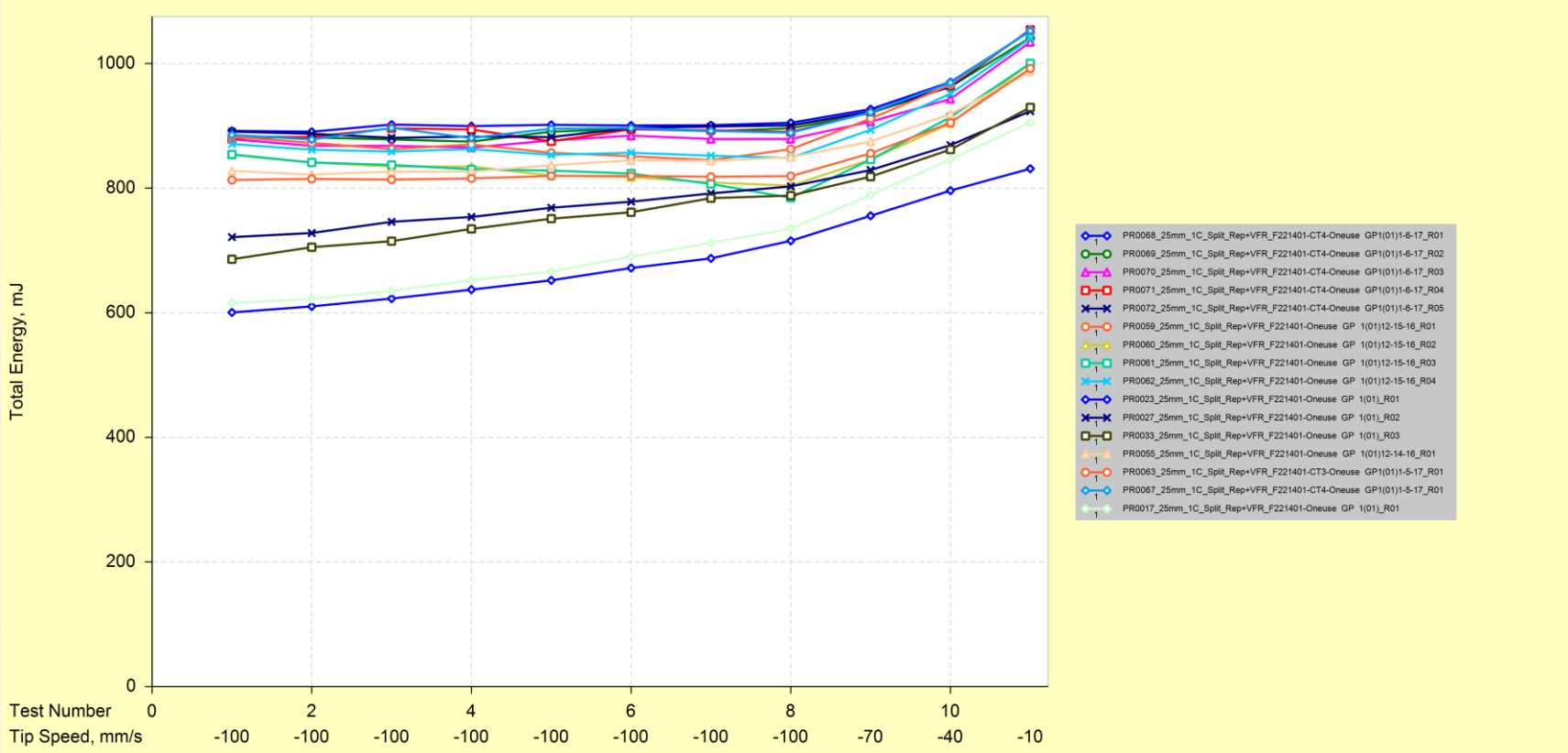
$$E_{flow} = \int_0^H \left( \frac{T}{R \ \tan \ \alpha} + F_{base} \right) dH$$



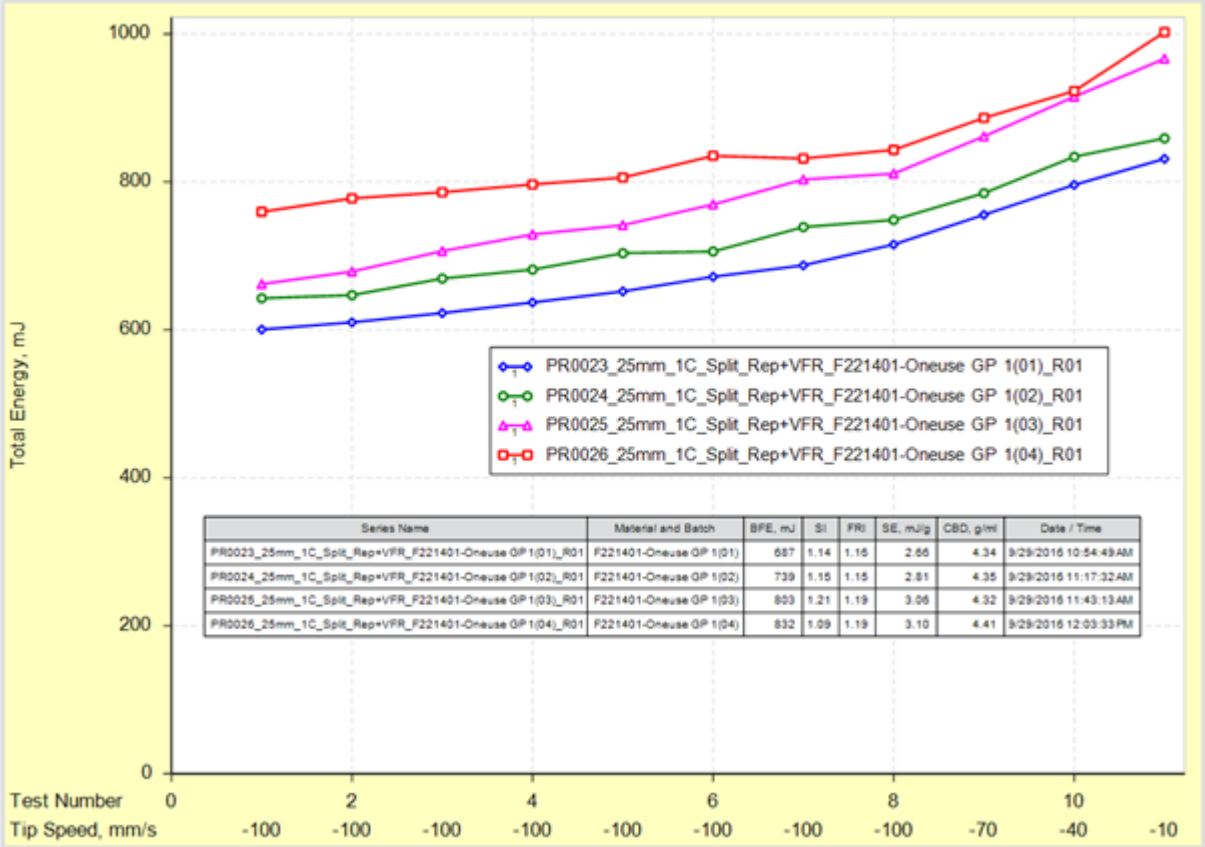
# Repeatability of Testing with Commercial Rheometer: same sample

- ~ 33% change in total energy

$$E_{flow} = \int_0^H \left( \frac{T}{R \ \tan \ \alpha} + F_{base} \right) dH$$



# Rheological Results: subsequent tests, cleaning between

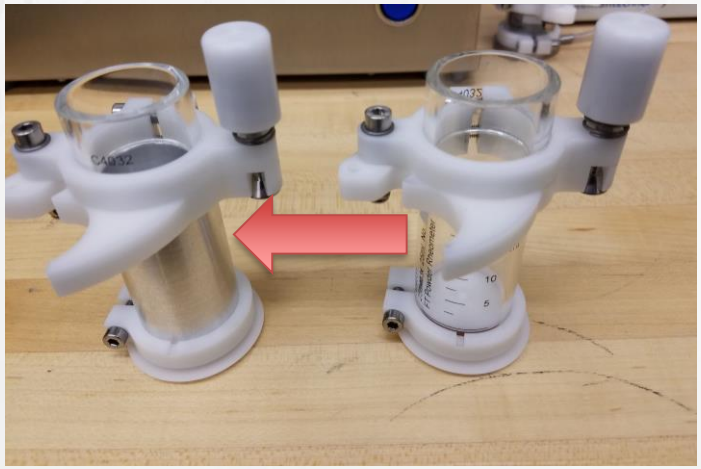



| Series Name | Material and Batch | BFE, mJ | SI | FRI | SE, mJ/g | CBD, g/ml | Date / Time |
|---|---|---|---|---|---|---|---|
| PR0023_25mm_1C_Split_Rep+VFR_F221401-Oneuse GP 1(01)_R01 | F221401-Oneuse GP 1(01) | 687 | 1.14 | 1.16 | 2.66 | 4.34 | 9/29/2016 10:54:49 AM |
| PR0024_25mm_1C_Split_Rep+VFR_F221401-Oneuse GP 1(02)_R01 | F221401-Oneuse GP 1(02) | 739 | 1.15 | 1.15 | 2.81 | 4.35 | 9/29/2016 11:17:32 AM |
| PR0025_25mm_1C_Split_Rep+VFR_F221401-Oneuse GP 1(03)_R01 | F221401-Oneuse GP 1(03) | 803 | 1.21 | 1.19 | 3.06 | 4.32 | 9/29/2016 11:43:13 AM |
| PR0026_25mm_1C_Split_Rep+VFR_F221401-Oneuse GP 1(04)_R01 | F221401-Oneuse GP 1(04) | 832 | 1.09 | 1.19 | 3.10 | 4.41 | 9/29/2016 12:03:33 PM |

# Triboelectric Effect

- History of powder plays an important role
- Moisture? Device not stable?
- **<u>Static charge</u>**
- Vessel change?

| | | | |
|---|---|---|---|
| Glass | +25 nC/J | +N | Slightly conductive. (Depends on humidity). |

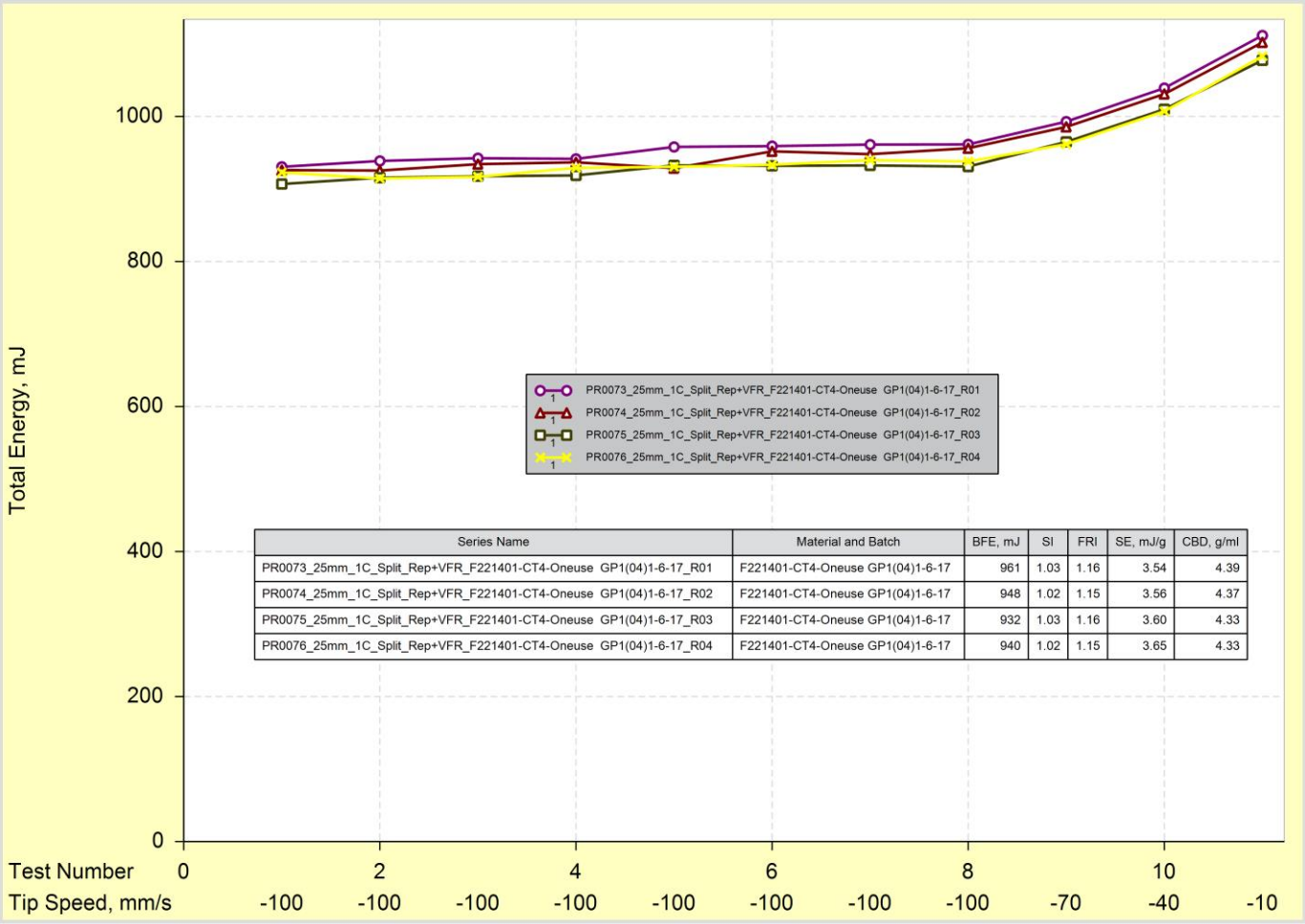


## TriboElectric Table

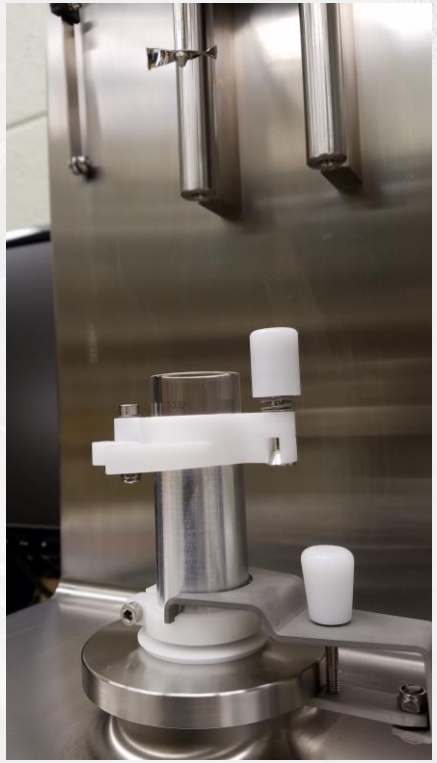
| Column 1 (this col.): Insulator name. Col.2: Charge affinity in nC/J (nano ampsec/wattsec of friction). Col.3: Charge acquired if rubbed with metal (W=weak, N=normal, or consistent with the affinity). Col.4: Notes. | Affinity nC/J | Metal effect | **Triboelectric Table** Tests were performed by Bill Lee (Ph.D., physics). ©2009 by AlphaLab, Inc. (TriField.com), which also manufactured the test equipment used. This table may be reproduced only if reproduced in whole. |
|---|---|---|---|
| Polyurethane foam | +60 | +N | All materials are good insulators (>1000 T ohm cm) unless noted. |
| Sorbothane | +58 | -W | Slightly conductive. (120 G ohm cm). |
| Box sealing tape (BOPP) | +55 | +W | Non-sticky side. Becomes more negative if sanded down to the BOPP film. |
| Hair, oily skin | +45 | +N | Skin is conductive. Cannot be charged by metal rubbing. |
| Solid polyurethane, filled | +40 | +N | Slightly conductive. (8 T ohm cm). |
| Magnesium fluoride (MgF2) | +35 | +N | Anti-reflective optical coating. |
| Nylon, dry skin | +30 | +N | Skin is conductive. Cannot be charged by metal rubbing. |
| Machine oil | +29 | +N | |
| Nylatron (nylon filled with MoS2) | +28 | +N | |
| Glass (soda) | +25 | +N | Slightly conductive. (Depends on humidity). |
| Paper (uncoated copy) | +10 | -W | Most papers & cardboard have similar affinity. Slightly conductive. |
| Wood (pine) | +7 | -W | |
| GE brand Silicone II (hardens in air) | +6 | +N | More positive than the other silicone chemistry (see below). |
| Cotton | +5 | +N | Slightly conductive. (Depends on humidity). |
| Nitrile rubber | +3 | -W | |
| Wool | 0 | -W | |
| Polycarbonate | -5 | -W | |
| ABS | -5 | -N | |
| Acrylic (polymethyl methacrylate) and adhesive side of clear carton-sealing and office tape | -10 | -N | Several clear tape adhesives are have an affinity almost identical to acrylic, even though various compositions are listed. |
| Epoxy (circuit board) | -32 | -N | |
| Styrene-butadiene rubber (SBR, Buna S) | -35 | -N | Sometimes inaccurately called "neoprene" (see below). |
| Solvent-based spray paints | -38 | -N | May vary. |
| PET (mylar) cloth | -40 | -W | |
| PET (mylar) solid | -40 | +W | |
| EVA rubber for gaskets, filled | -55 | -N | Slightly conductive. (10 T ohm cm). Filled rubber will usually conduct. |
| Gum rubber | -60 | -N | Barely conductive. (500 T ohm cm). |
| Hot melt glue | -62 | -N | |
| Polystyrene | -70 | -N | |
| Polyimide | -70 | -N | |
| Silicones (air harden & thermoset, but *not* GE) | -72 | -N | |
| Vinyl: flexible (clear tubing) | -75 | -N | |
| Carton-sealing tape (BOPP), sanded down | -85 | -N | Raw surface is very + (see above), but close to PP when sanded. |
| Olefins (alkenes): LDPE, HDPE, PP | -90 | -N | UHMWPE is below. Against metals, PP is more neg than PE. |
| Cellulose nitrate | -93 | -N | |
| Office tape backing (vinyl copolymer ?) | -95 | -N | |
| UHMWPE | -95 | -N | |
| Neoprene (polychloroprene, *not* SBR) | -98 | -N | Slightly conductive if filled (1.5 T ohm cm). |
| PVC (rigid vinyl) | -100 | -N | |
| Latex (natural) rubber | -105 | -N | |
| Viton, filled | -117 | -N | Slightly conductive. (40 T ohm cm). |
| Epichlorohydrin rubber, filled | -118 | -N | Slightly conductive. (250 G ohm cm). |
| Santoprene rubber | -120 | -N | |
| Hypalon rubber, filled | -130 | -N | Slightly conductive. (30 T ohm cm). |
| Butyl rubber, filled | -135 | -N | Conductive. (900 M ohm cm). Test was done fast. |
| EDPM rubber, filled | -140 | -N | Slightly conductive. (40 T ohm cm). |
| Teflon | -190 | -N | Surface is fluorine atoms-- very electronegative. |

*From <https://www.trifield.com/content/tribo-electric-series/>*

# Rheological Results: subsequent tests, AL vessel




| Series Name | Material and Batch | BFE, mJ | SI | FRI | SE, mJ/g | CBD, g/ml |
|---|---|---|---|---|---|---|
| PR0073_25mm_1C_Split_Rep+VFR_F221401-CT4-Oneuse GP1(04)1-6-17_R01 | F221401-CT4-Oneuse GP1(04)1-6-17 | 961 | 1.03 | 1.16 | 3.54 | 4.39 |
| PR0074_25mm_1C_Split_Rep+VFR_F221401-CT4-Oneuse GP1(04)1-6-17_R02 | F221401-CT4-Oneuse GP1(04)1-6-17 | 948 | 1.02 | 1.15 | 3.56 | 4.37 |
| PR0075_25mm_1C_Split_Rep+VFR_F221401-CT4-Oneuse GP1(04)1-6-17_R03 | F221401-CT4-Oneuse GP1(04)1-6-17 | 932 | 1.03 | 1.16 | 3.60 | 4.33 |
| PR0076_25mm_1C_Split_Rep+VFR_F221401-CT4-Oneuse GP1(04)1-6-17_R04 | F221401-CT4-Oneuse GP1(04)1-6-17 | 940 | 1.02 | 1.15 | 3.65 | 4.33 |



# Static Measurements

- Externally mounted surface volt-meter
- Prior to testing voltages are high (~700-1300 V)
- During testing voltages drop to 200-400 V
- After testing voltages jump higher (1300 – 1500V)
- **<u>Static charge causing increased cohesion</u>**
- Grounded vessel helps, but does not eliminate

# References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[6] Cundall, Peter A., and Otto DL Strack. "A discrete numerical model for granular assemblies." Geotechnique 29.1 (1979): 47-65.

[7] Geer, Stephen, Bernhardt, Michelle L., Garboczi, Edward J., Whiting, Justin G., Donmez, Alkan M., “Calibration of Discrete Element Method Parameters for the Simulation of Stainless Steel Powder,” to appear in Particuology.

# Questions and Riffling