3D PBF – "A Miniaturized Solids Processing Plant in a Box" Key Aspects of Powder Handling

Goals:

- Highlight accomplishments/principles of powder technology field
- Introduce approach to solids processing problems
  - Identify relevant scale of study (particle vs. bulk vs. system)
  - Bulk or mesoscale – material properties
    - Material properties directly used for first principle process design or powder formulation, and scaling
    - Fluid flow design approach; less common for granular solids. Why?
  - Particle scale – controls bulk material properties
    - Best practice: Multivariate correlation of bulk material properties, in turn used for "powder" design (opposed to machine design)
- Impact on Powder Bed Fusion
- Powder Workshops (Week of January 15th or 22nd)
  - *Contact us for details, or to be added to our workshop mailing list. (contact@powdernotes.com)*

E&G Associates, 100 Cherokee Blvd. Ste 332, Chattanooga, TN 37405

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Webinar abstract

Dr. Bryan J. Ennis
E&G Associates, Inc., 100 Cherokee Blvd., Chattanooga, TN 37405
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Abstract

Modern additive manufacturing systems based on powder bed technology integrate several well-established solids processing unit operations. Examples of relevant solids processing steps include mixing, fluidization, powder dosing, powder transport and storage, roller spreading, sintering and powder classification. Powder based systems can be viewed essentially as a miniaturized solids processing plant in a box. Though additive systems present their own unique set of challenges, established design principles are often overlooked. Solids processing plants often have poor efficiencies, long start-up times, and large sensitivities to feedstock material property variations, and powder bed fusion printers have not proven to be an exception.

This E&G Associates' webinar will focus on one of the essential foundations of solids processing, namely solids handling and storage. In particular, the problem of bin design will be presented as an introduction to the language and methodologies of solids processing and powder mechanics. Using well-established design principles, attendees will be shown that it is possible to design a storage system to prevent powder arching, ratholing, and segregation. Traditional characterization methods will be emphasized, including material property measurements of powder cohesion, powder/wall friction, and permeability, as well as related particle scale characterization of particle size, shape, and surface energy and hardness. Extensions as well as limitations of powder mechanics to related areas will be highlighted, namely roller spreading, fluidized and partially aerated flows.

Remote WebEx Webinar Link:

Check our website for an video recording of Oct.17th live webinar.

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Company background

E&G Associates, Inc.

E&G is a recent recipient of two SBIR awards from the Department of Defense to undertake feedstock development for additive manufacturing (3D Printing) and solids mixing design for energetic materials of interest to the U.S. Navy. “The Small Business Innovation Research (SBIR) program is a highly competitive program that encourages domestic small businesses to engage in Federal Research & Development that has the potential for commercialization [https://www.sbir.gov].” In terms of additive manufacturing, these two additional SBIR awards combined with ongoing AM client support and matching state funding brings its funding in 3D printing and solids processing research to 1.4 million dollars.

E&G is a leading engineering consulting firm with specific expertise in bulk solids & powder processing, both formulation and equipment design, supported by a 3000-square foot laboratory facility incorporating well over two million dollars in characterization equipment. E&G is part of the INCubator program of the Chattanooga Area Chamber of Commerce located at the Business Development Center (BDC) of Hamilton County, Tennessee, and is managed by four alumni of the College of Computer Science and Engineering at the University of Tennessee at Chattanooga.

E&G was also a sponsor of a recent conference in 3D printing hosted by the national laboratories in Austin, TX: “Modeling of Powder Dynamics in Additive Metal Manufacturing, August 2017”. As a follow-up to this conference, E&G is presenting this webinar for previous attendees on the impact of bulk solids properties on 3D powder bed fusion hosted by Lawrence Livermore National Laboratory.

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Presenter background

Dr. Bryan J. Ennis

Dr. Ennis is a founder of E&G Associates, Inc. and iPowder Systems, and is an Associate Professor of Chemical Engineering at The University of Tennessee at Chattanooga. He has three decades of experience in powder process manufacturing, and has consulted for over 100 clients, including all major US pharmaceutical and consumer products companies, the US FDA, and the State of MN (Steel Processing Consortia). He led granulation research, solids handling, & powder characterization programs of DuPont Engineering, and served as an Adjunct Professor of Vanderbilt Univ. He received his B.S. (Chemical Engineering) from Rensselaer Polytechnic Institute & Ph.D. from The City College of New York. Dr. Ennis is a founder and previous Technical Vice-Chair of the Particle Technology Forum (PTF) and previous Chair of the Powder Technology Programming Group of the American Institute of Chemical Engineering (AIChE). Honors include two national AIChE awards for service to the profession, Deutscher Akademischer Austausch Dienst Award (Germany), Stanley Katz Memorial Award (City College of NY), and a Visiting Research Fellow of Delft Technische Hogeschool (Netherlands). He is the author of several other invited contributions on particle processing, including presentations to National Science Foundation, and Section Editor of Section 21: Solid-Solids Operations & Equipment (Powder Processing) of the Perry's Chemical Engineer's Handbook (8th Ed.) and Section 20: Size Enlargement (7th Ed.); Theory of Granulation: An Engineering Perspective, in Handbook of Pharmaceutical Granulation, 2e., The Science & Engineering of Granulation Processes, Kluwer Academic, Powder & Bulk Engineering, and Chemical Engineering. Website: www.powdernotes.com

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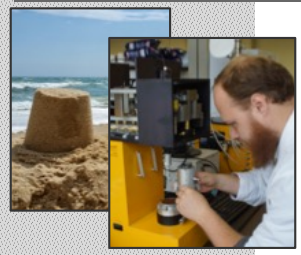
Related Materials Characterization for Powder Bed Fusion

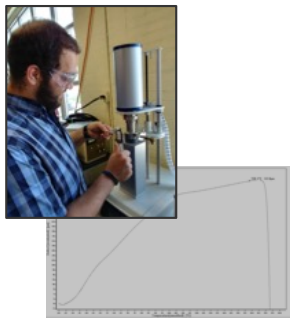
E&G Associates Laboratory and Design Capabilities

Rotary & translational shear cell



- Hopper & feeder design
- Transfer points, platen design
- Mix formulation and flow aids
- Powder flowability (powder/wall friction, roll friction, cohesion)
- iShear™ & Jenike™ split cells

Dilatometry

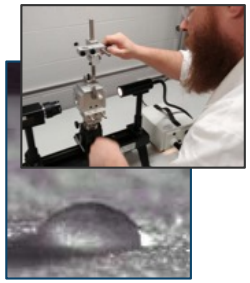


- Thermal behavior of bulk powders
- Cyclic thermal loading
- Softening temperature, powder sintering
- Linear thermal expansion
- Linseis & Netzch dilatometer

Permeability/fluidization cell

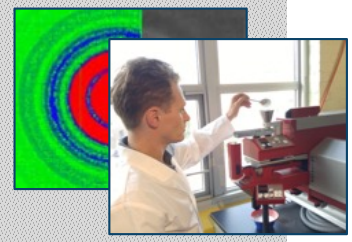
- Fluidization behavior, rate limiting flow, part extraction
- Permeability, minimum fluidization & bubbling velocity
- Triboelectric charging
- iFluid™ permeability cell

Contact angle goniometer



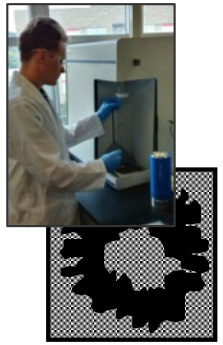
- Dynamic contact angle & surface tension
- Surfactant studies
- Binder wetting & penetration
- Surface chemistry & IGC
- Dynamic contact angle goniometry
- Rame-hart goniometer

Imaging & laser diffraction



- Segregation & blend uniformity
- Impact of recycling
- Powder flowability & qualification
- Conveying operations, safety
- Cyclone efficiency
- Particle size & shape distributions
- Sympatec HELOS™ & QICPIC™
- Zeiss-Clemex microscope

BET surface area & porosity



- Surface area and porosity metrics
- Both feedstocks and finished parts
- Binder and feedstock interaction
- BET Surface Area and Porosity
- Micromeritics Tristar 3000
- Micromeritics Accupyc II 1340

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3D PBF – "A Miniaturized Solids Processing Plant in a Box" Key Aspects of Powder Handling

Structure:

- Introduction to PBF as a solids process
- E&G background & capabilities
- Some manifestations of granular flow
- Bin design illustration: What is powder flowability?
- Extension to other flows
  - Tableting, extrusion
  - Roll pressing
- Related powder bed fusion challenges?
  - Blade & roller spreading
  - Print bed motion
  - Powder-part interactions
- Conclusions/Questions

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3D Powder Bed Fusion – "A Manufacturing Plant in a Box"

Common Solids Processing Unit Operations of AM

Fresh

Fresh

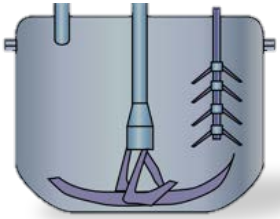
Recycle

Storage, transfer, feeding:

- Powder flow function & density
- Wall & powder friction
- Aeration & permeability
- Conveying

Mixing:

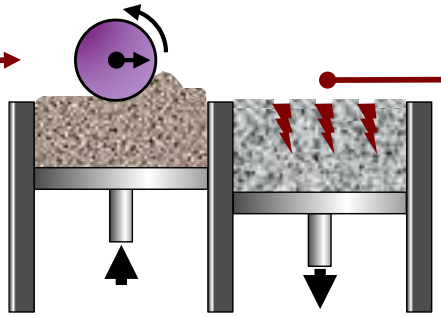
- Vertical or horizontal
- Batch or continuous
- Mechanical or fluidized
- PSSD & powder cohesion
- Scale of scrutiny

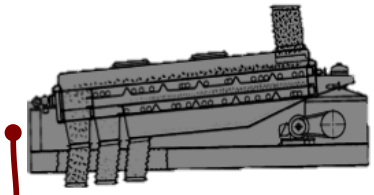


Blended feedstock

Print Bed:

- Spreading, wetting & fusion
- Powder flowability & density
- Roll, wall & powder friction
- Thermal softening
- PSSD & permeability
- Binder viscosity, contact angle





Extraction & classification:

- Particle size and shape (PSSD)
- Cake strength
- Triboelectric charging
- Reconditioning of recycle

Overview:

- PBF integrates traditional solids processing steps; essentially a miniature plant in a box.
- Solids plants often require significant start-up times, and poor efficiencies.
- Solids plant design principles (e.g. solids mixing, feeding, storage) can be applied.
- Solids handling steps are critical for the success of an AM architecture, and are as key as the fusion step.

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Examples in Powder Bed Fusion

- **Solids Handling**
  - Powder cohesion/friction, wall friction
  - Permeability and deareation
  - Hopper design
  - Fluid-bed design
  - Mechanical & pneumatic transport
  - Mixing
  - Extraction/classification
  - Related print bed issues

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Examples in Powder Bed Fusion

- **Solids Handling**
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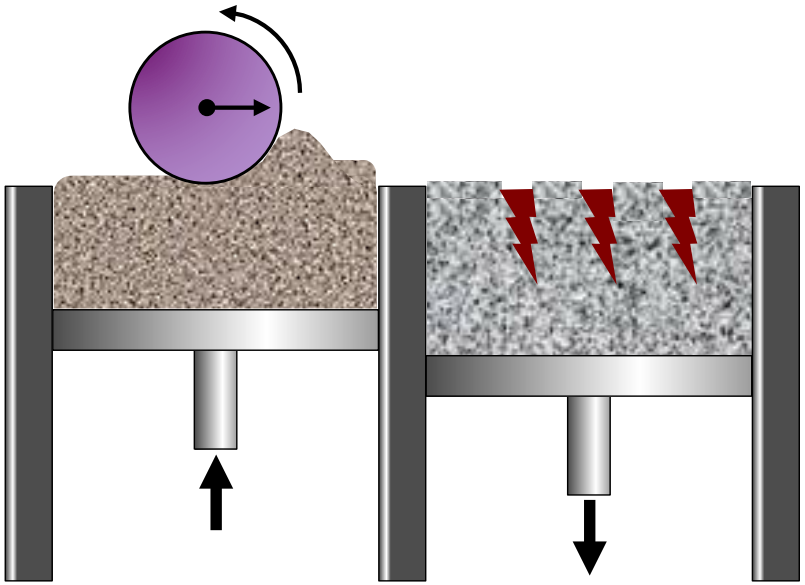
  - **Fluid-bed design**
  - **Mechanical & pneumatic transport**
  - **Mixing**
  - **Extraction/classification**
  - **Related print bed issues**

Print Bed Properties:
- Spreading, wetting & fusion
- Powder flowability & density
- Roll, wall & powder friction
- Thermal softening
- PSSD & permeability
- Binder viscosity, contact angle
- Bed motion
- Part-powder interactions



Feed bed Build bed

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Granular solids background: The ideal Coulomb material

- **Material properties:**
  - Angles of powder friction (internal, effective)
  - Wall friction and adhesion
  - Cohesive yield strength
  - Measured by shear cell
  - Impact on powder spreading?

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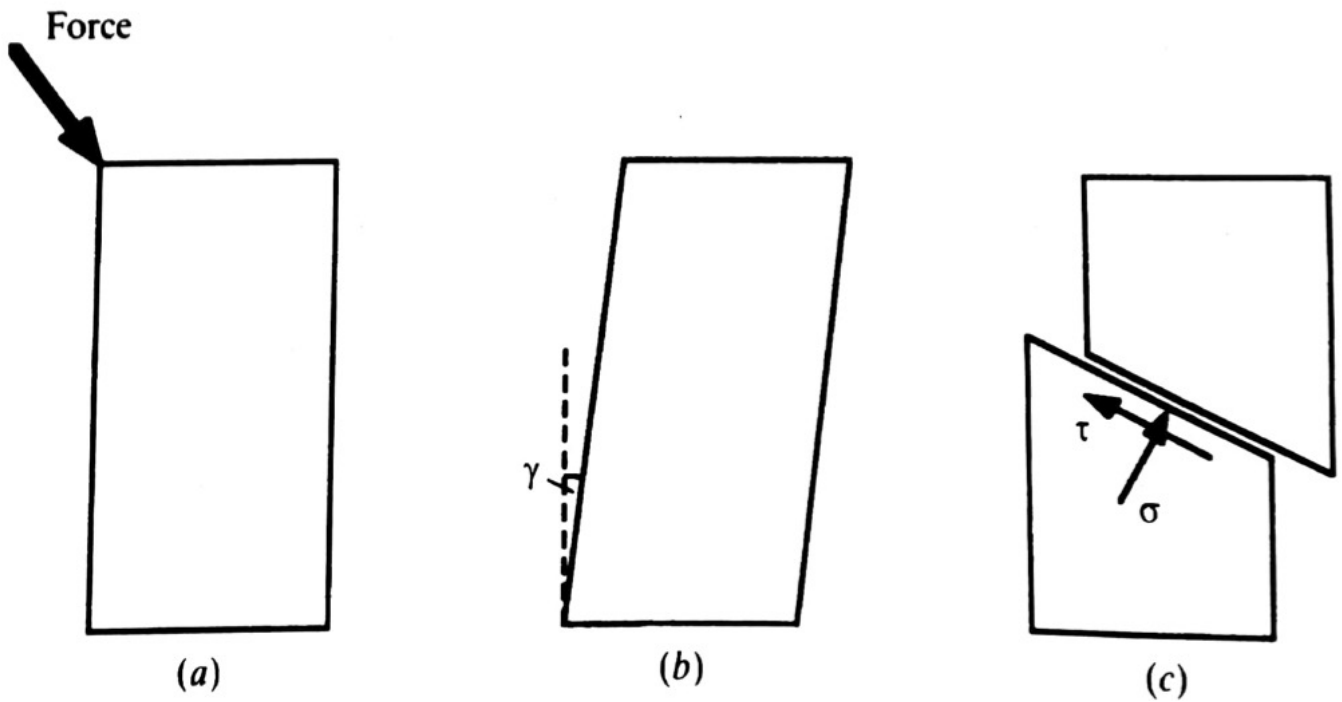
The ideal Coulomb material & implications for strength

(Ref: Nedderman, Statics & Kinematics of Granular Materials, 1992, 2005)



Hookean solid:

$$\tau = f(\gamma) = G\gamma$$

Ideal Coulomb material:

$$\boxed{\begin{aligned}\tau &= f(\sigma) = \mu\sigma + c \\ &= \sigma \tan\phi + c\end{aligned}}$$

Newtonian fluid:

$$\tau = f(\dot{\gamma}) = \mu_f \dot{\gamma}$$

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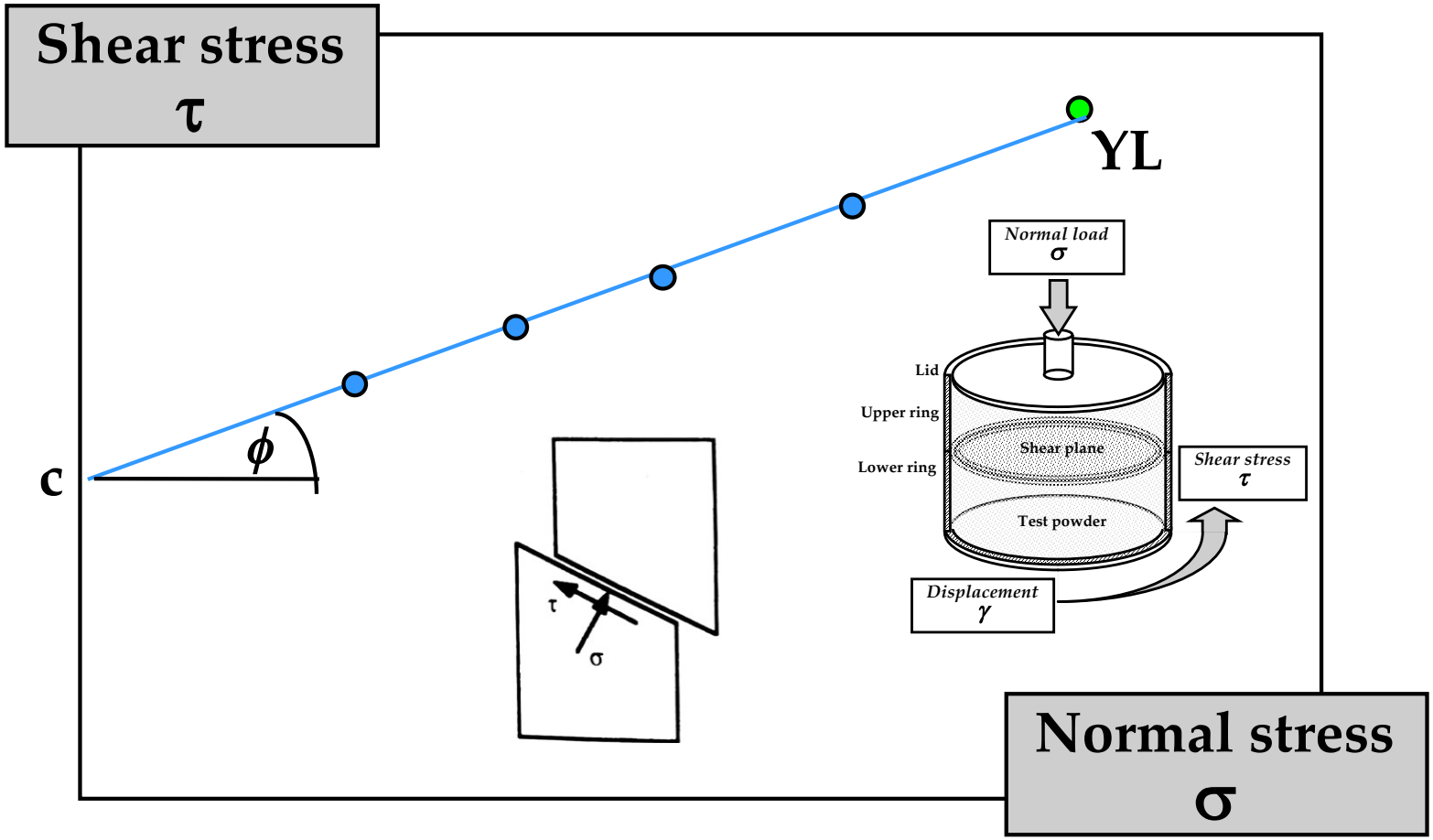
The Mohr-Coulomb Solid: Yield loci - Effect of stress history



- Force required to shear a powder is a function of current stress

Ideal Coulomb material:

$$\tau = f(\sigma) = \sigma \tan\phi + c$$

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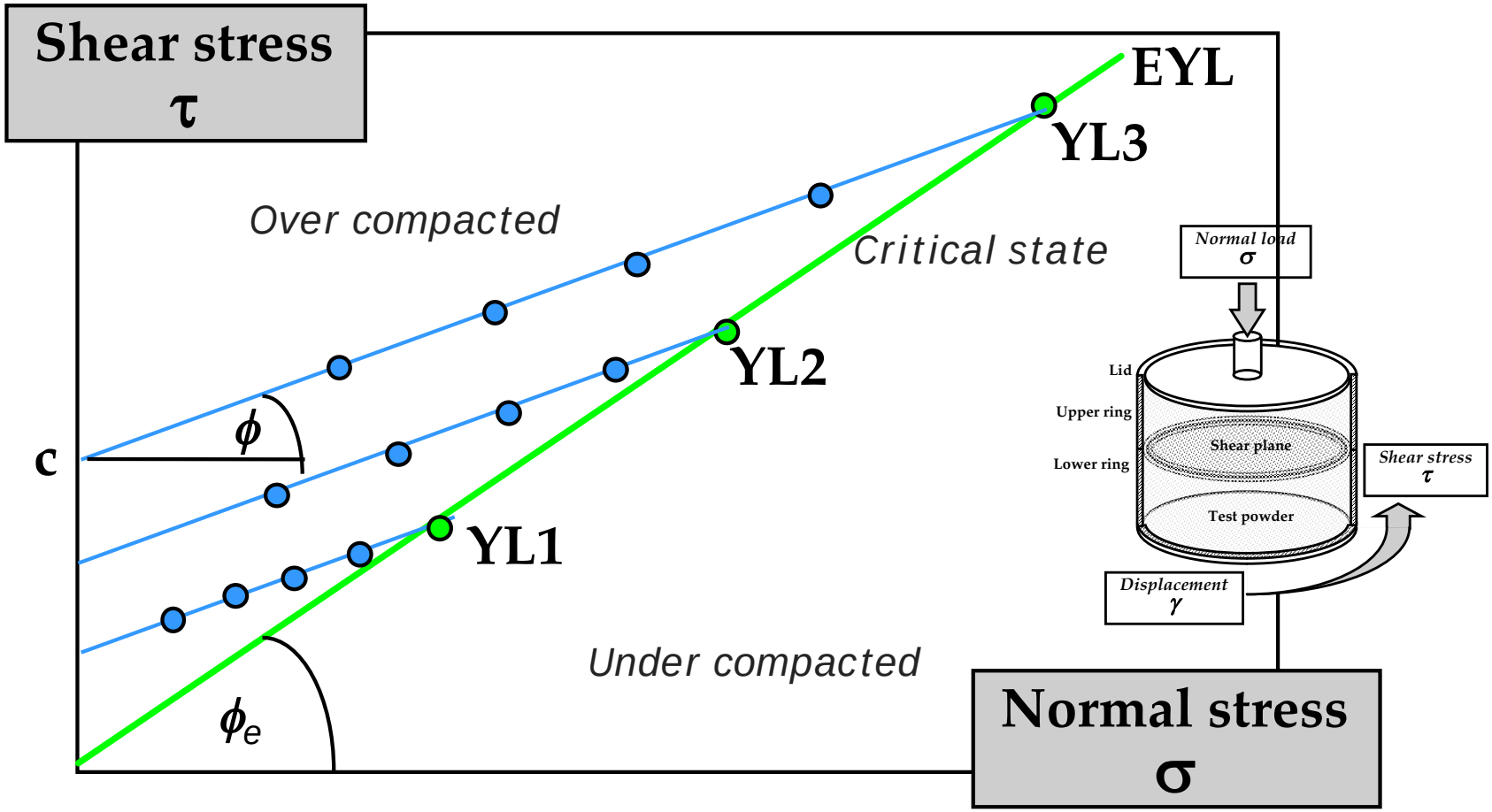
The Mohr-Coulomb Solid: Yield loci - Effect of stress history



- What if I repeat previous experiment at several values of the initial consolidation stress (on the EYL)?
- There are multiple yield loci (YL1, YL2, …)
- Force required to shear a powder is a function of current stress, and stress history (previous compaction.)

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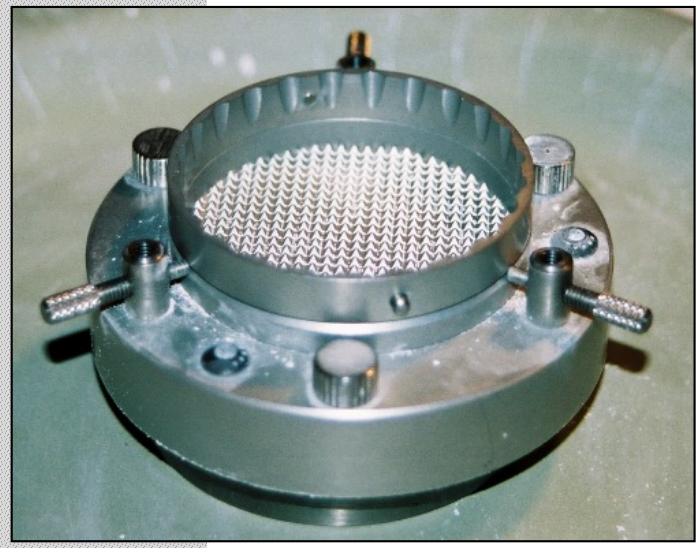
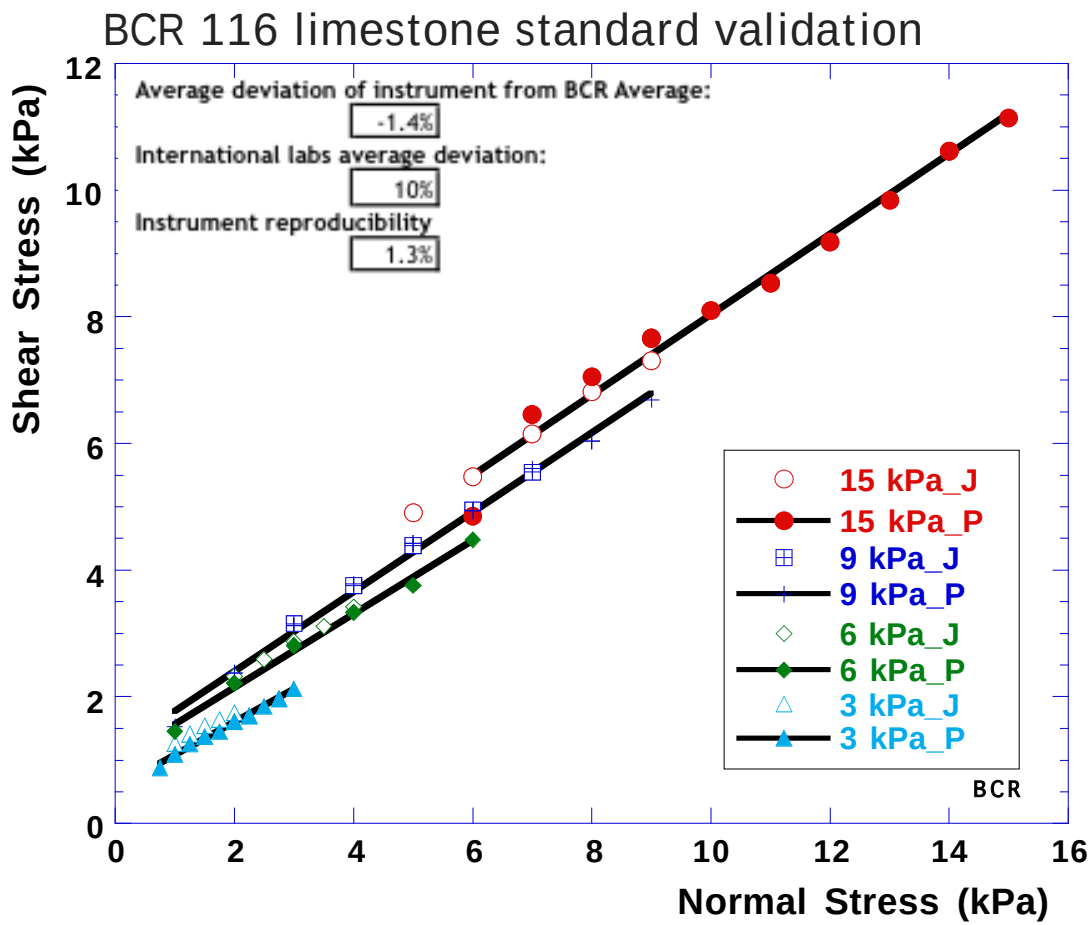
Shear cell measurement & validation





- Full automated w/ unlimited displacement
- Complete flow function w/one sample
- Critical state conditioning through protocol
- Split cell ensures powder friction/failure (not at wall), with free dilation of shear plane

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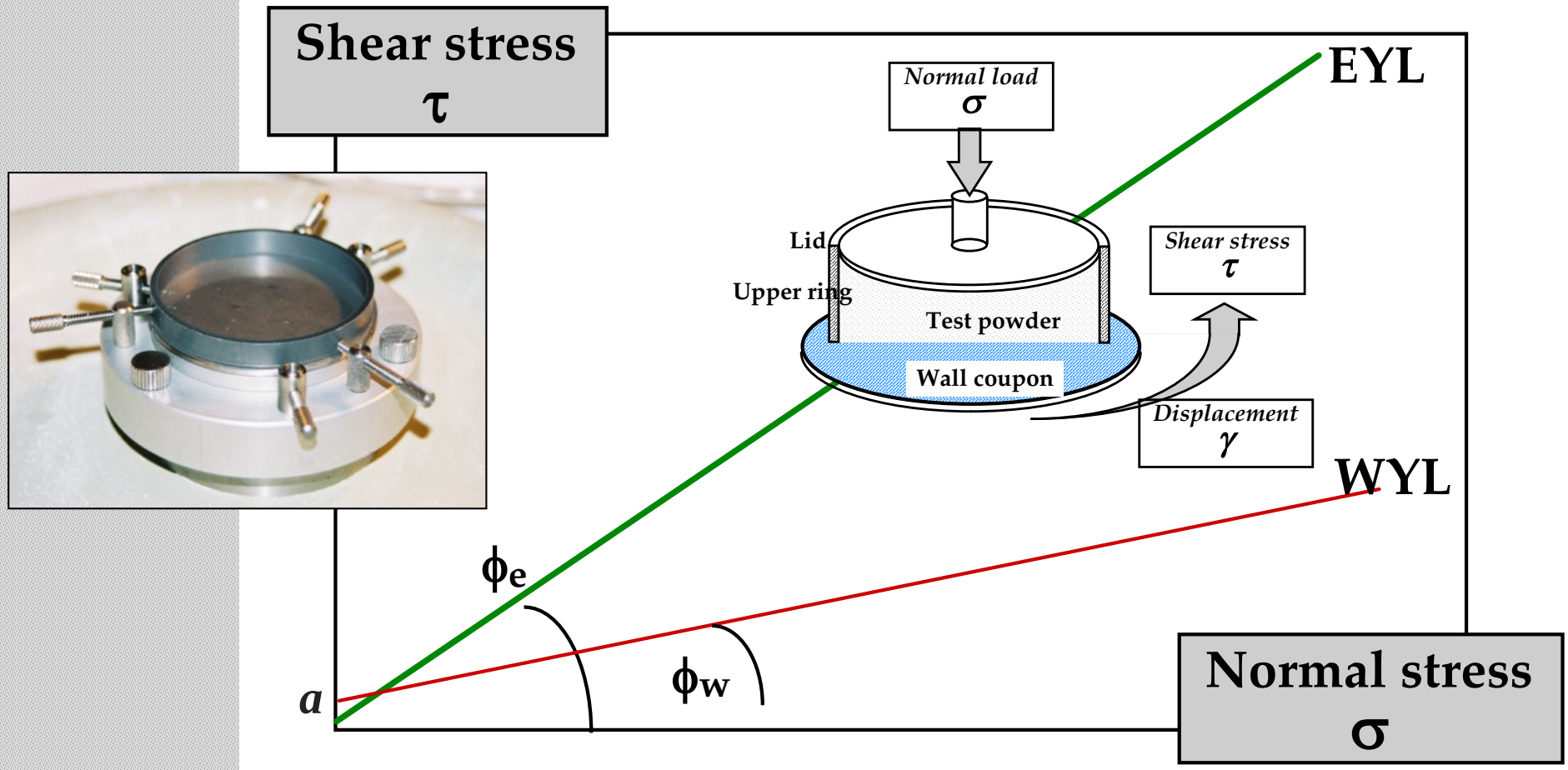
Powder friction vs. wall friction



- ϕ_w, angle of wall friction, or external friction
- ϕ_e, angle of effective friction, or powder friction
- Both control hopper pressures and also compaction processes

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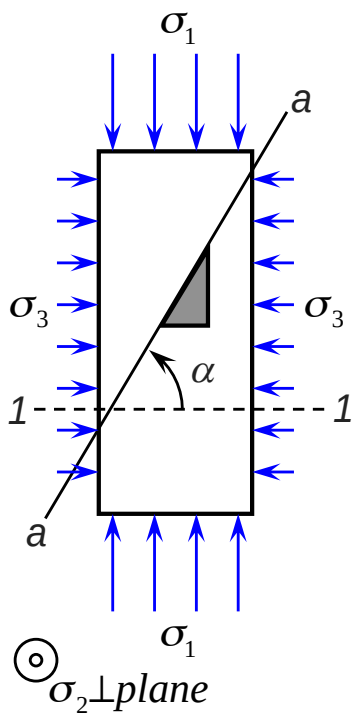
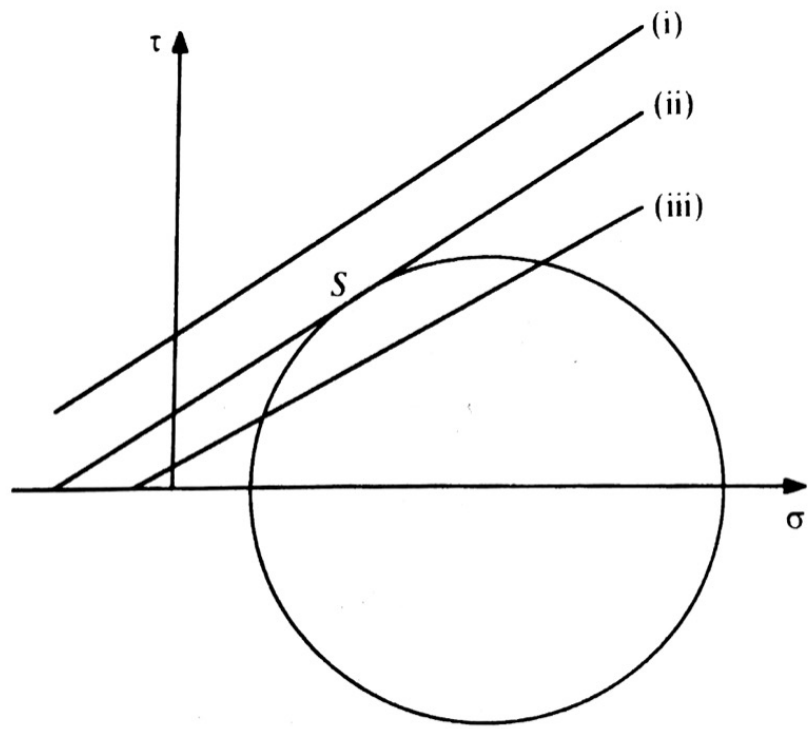
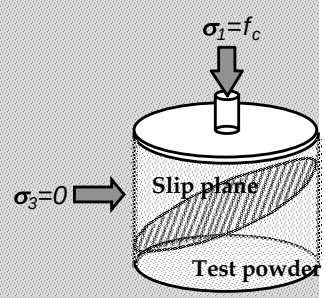
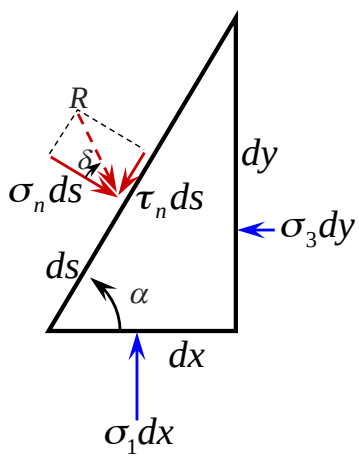
The Mohr stress circle









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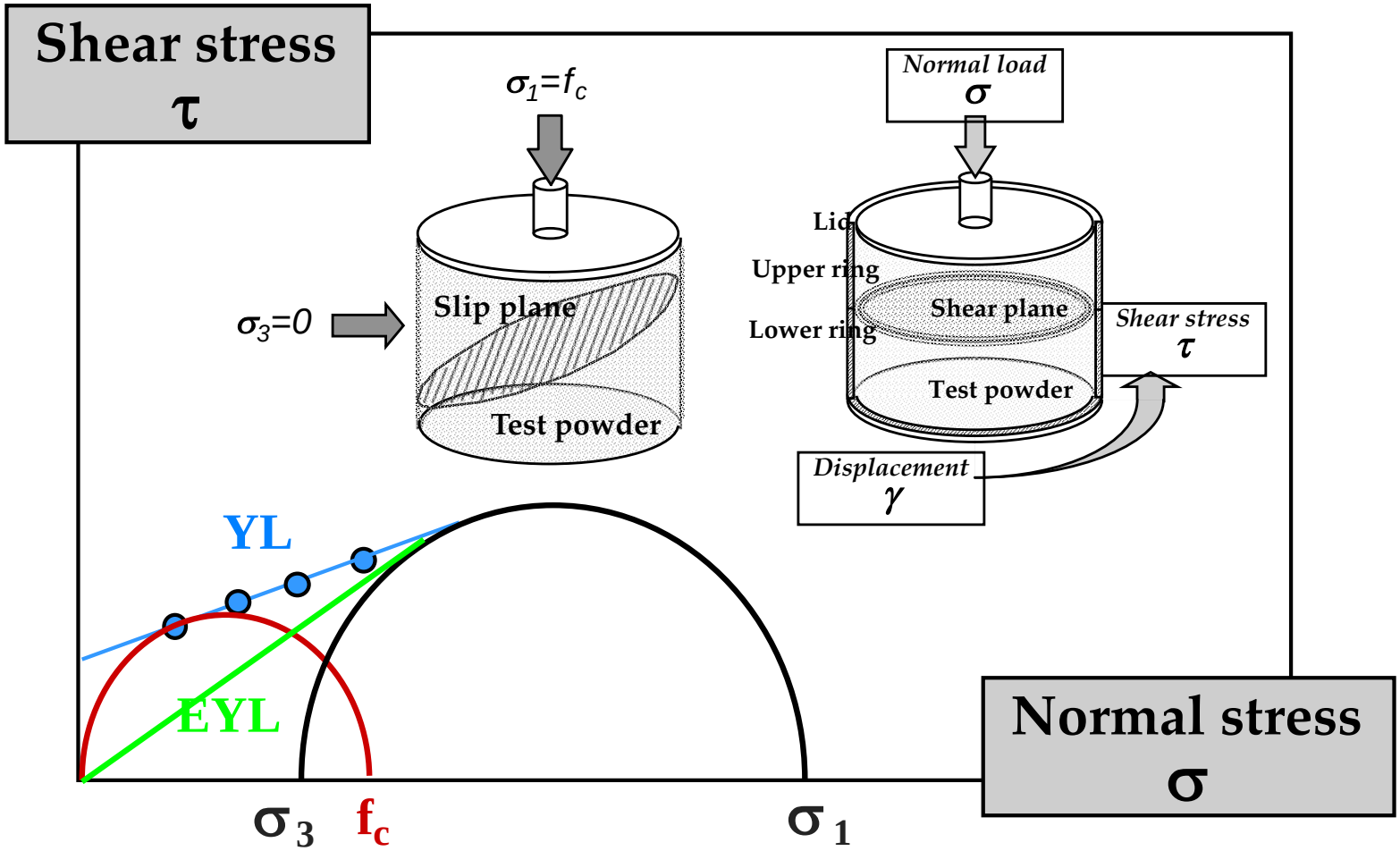
Unconfined yield strength – a special case



- **Tangent Mohr circles determine fc and σ1, the major compaction stress**

$$f_c = \left\{ \frac{2}{\cos\phi} \left(\frac{1+\sin\phi}{1+\sin\phi_e} \right) \left[\sin\phi_e - \sin\phi \right] \right\} \sigma_1$$

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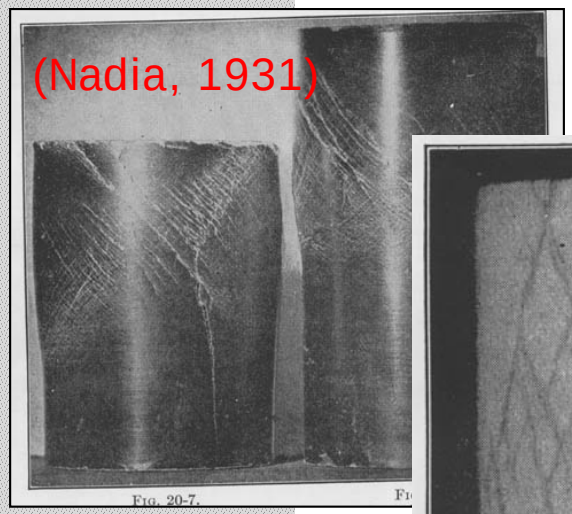
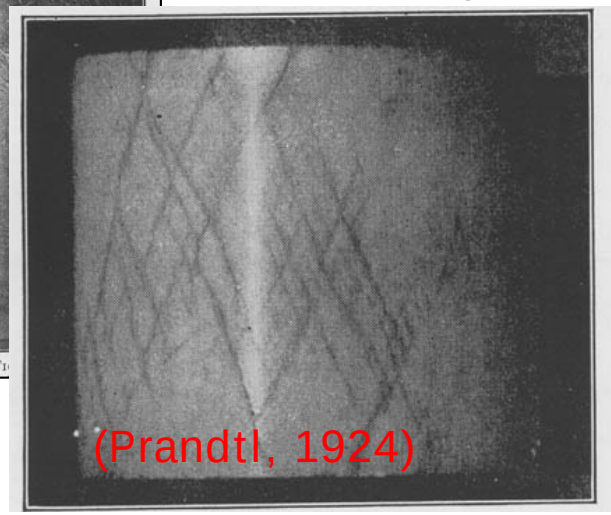
Meaning of uniaxial strength & internal friction?

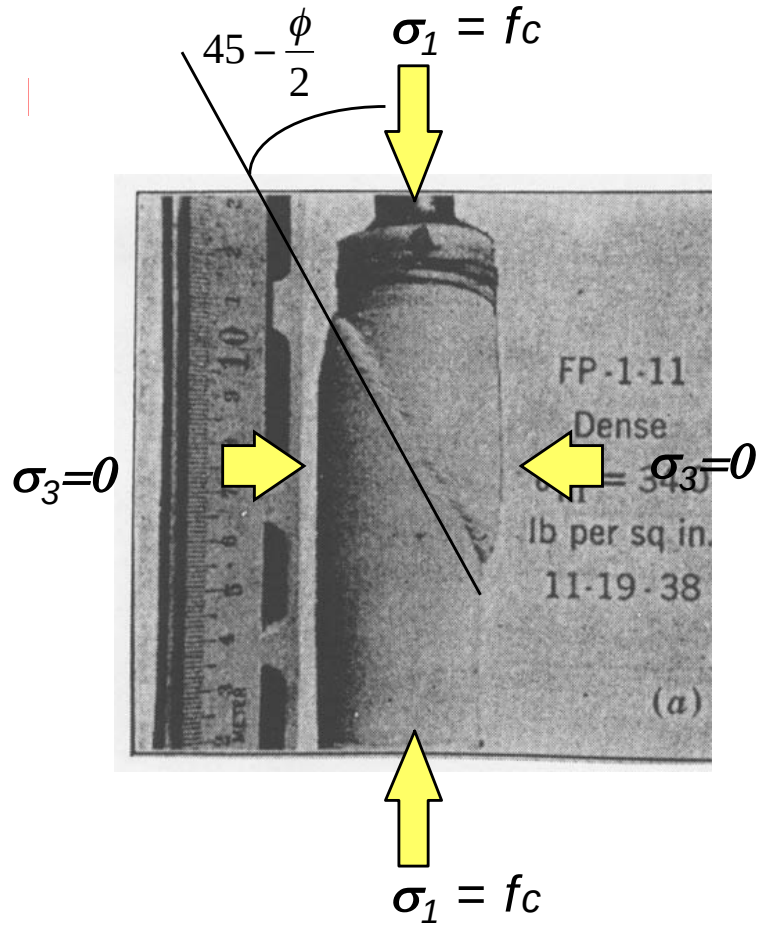
(Nadia, Theory of Flow & Fracture of Solids, Vol.1&2, 1931,1963)



Paraffin slip=45°

Marble slip<45°





- *Powders fail on slip planes at a weak point, rather than transmit stress like a fluid under shear.*
- To a starting approx., shear stress due to powder is strain rate independent. Frictional process, neglecting interstitial pore pressure.
- Powder flow: Osborne Reynolds (1850s), Coulomb, Mohr & Prandlt

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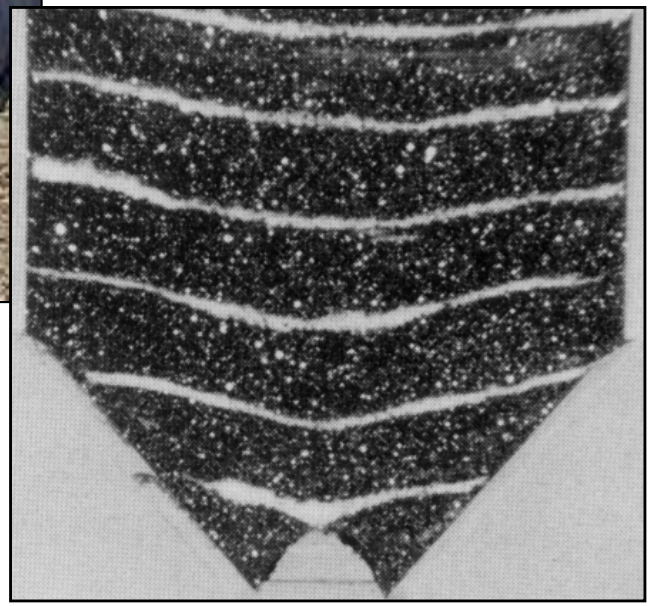
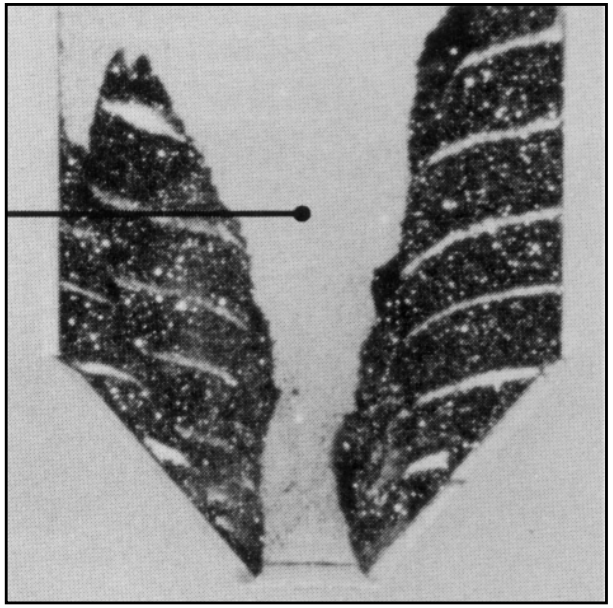
Sand castles & flow loss

- Powders have a cohesive yield strength.
- Think “sand castle” strength.
- Leads to solids flow loss in a bin by a variety of mechanisms.
- This plastic yield strength is a complex function of stress/bulk density path or history, as well as interstitial pore pressure.

Mass flow: A stable arch



Funnel flow: A stable rathole



Johanson et al., ASME, 1970s, Courtesy US Steel.

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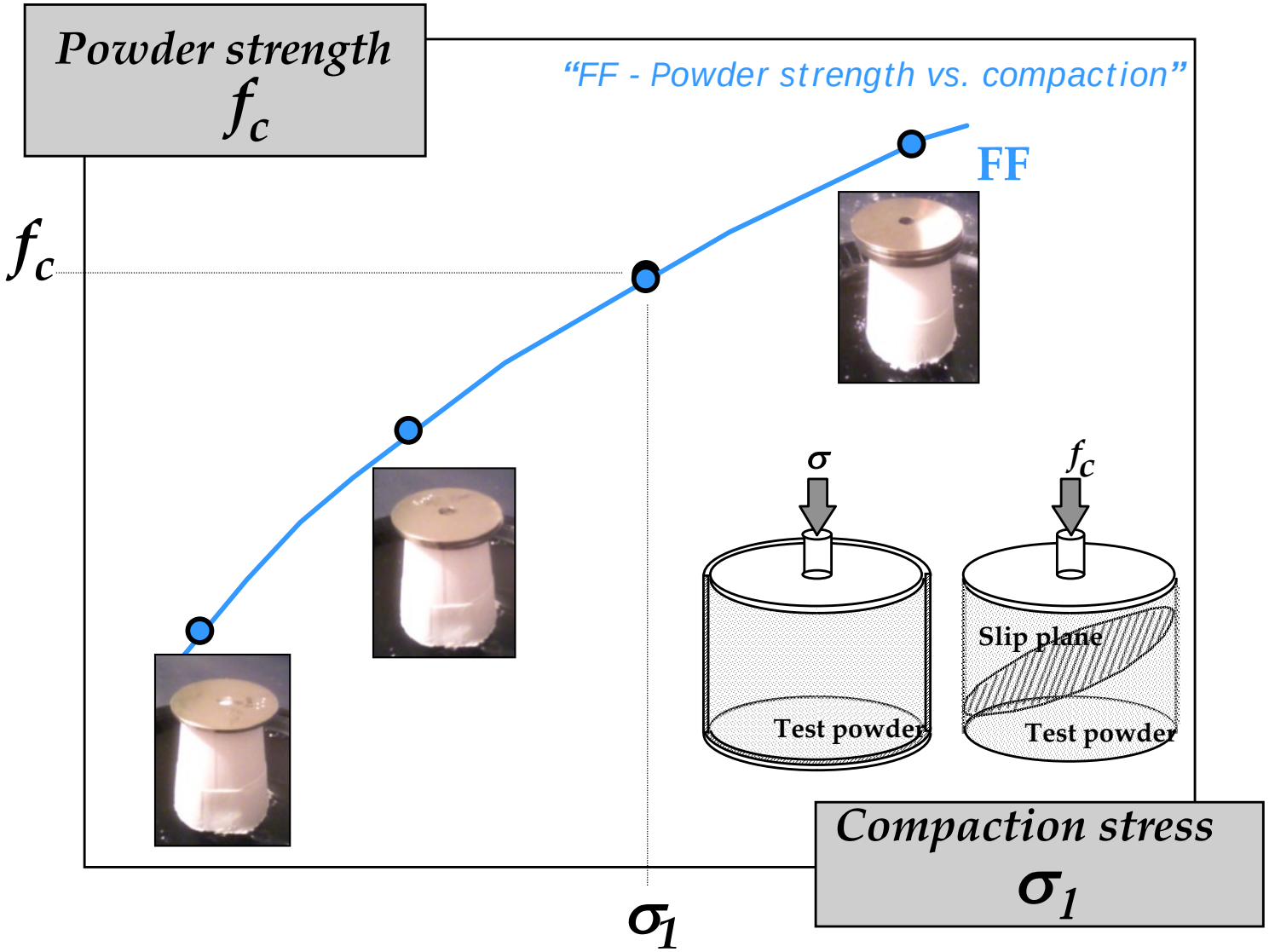
Powder strength vs. compaction pressure (The powder flow function - FF)



Increasing diameter, B --->

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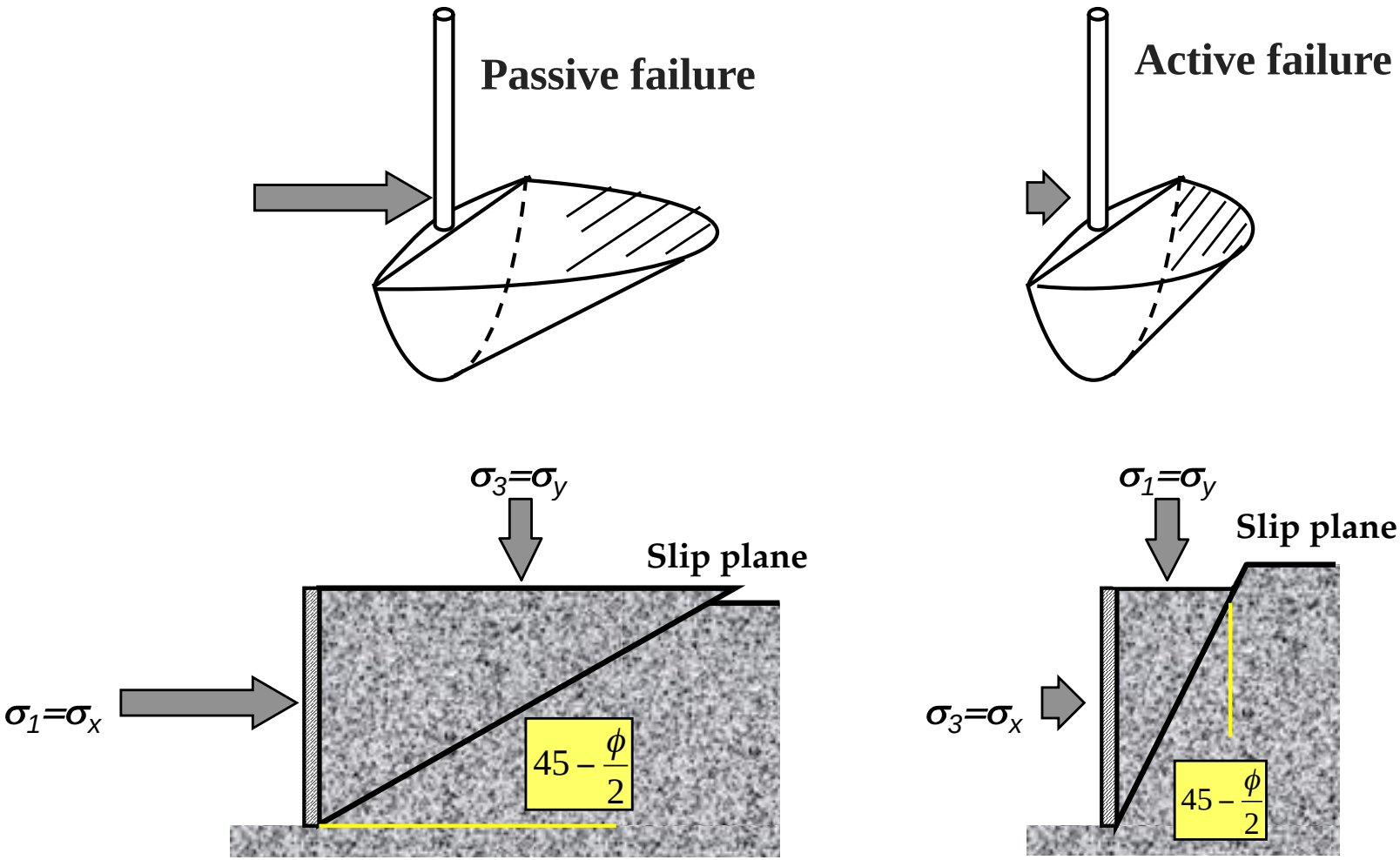
There are two possible conditions of failure!

More experiments on the beach - Rankine (1856), after Nadia



- Passive, push a shovel into a pile
- Active, let the shovel hold the pile up, and relieve this pressure
- Impact on doctor blade spreading?

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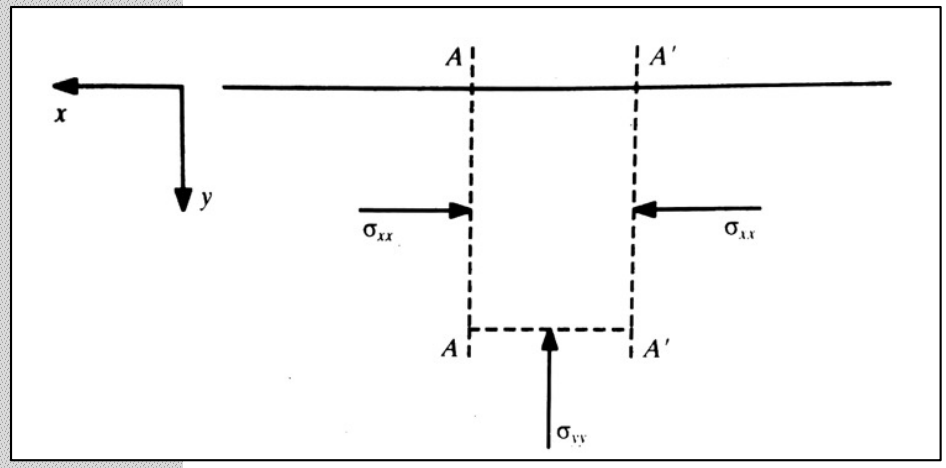
Implications for stress distribution and failure



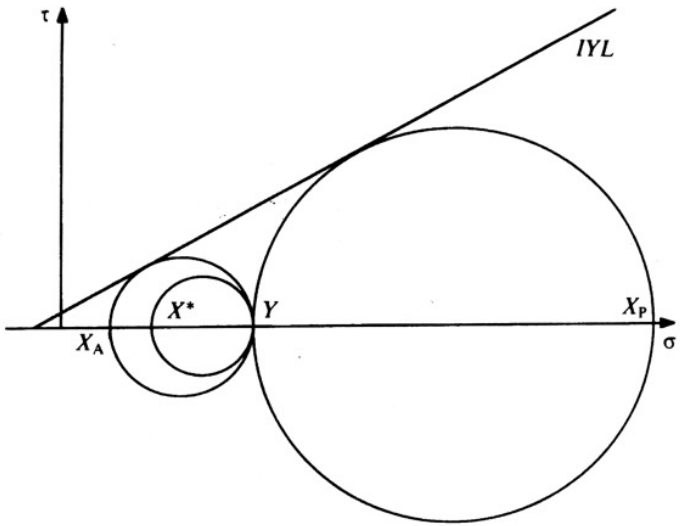


Figure 3.6 Mohr's circles for the Rankine states.

Stress in fluid:

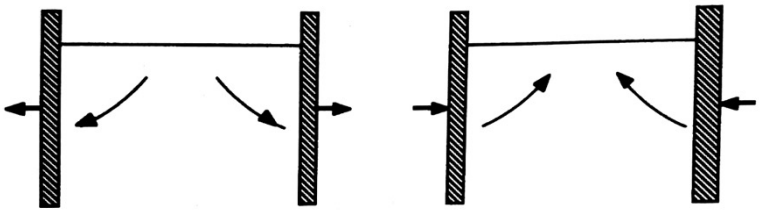
$$\sigma = \rho g h$$

Stress in MC (smooth wall):

$$\sigma = \rho g h \left(\frac{1 - \sin\phi}{1 + \sin\phi}\right)$$

Stress in rough wall:

$$\sigma = \rho g h \left(\frac{\cos^2\phi}{\left[1 + \sqrt{1 + k}\,\sin\phi\right]^2}\right)$$



(a) Active (b) Passive

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Retaining wall forces for failure: Impact on blade spreading?

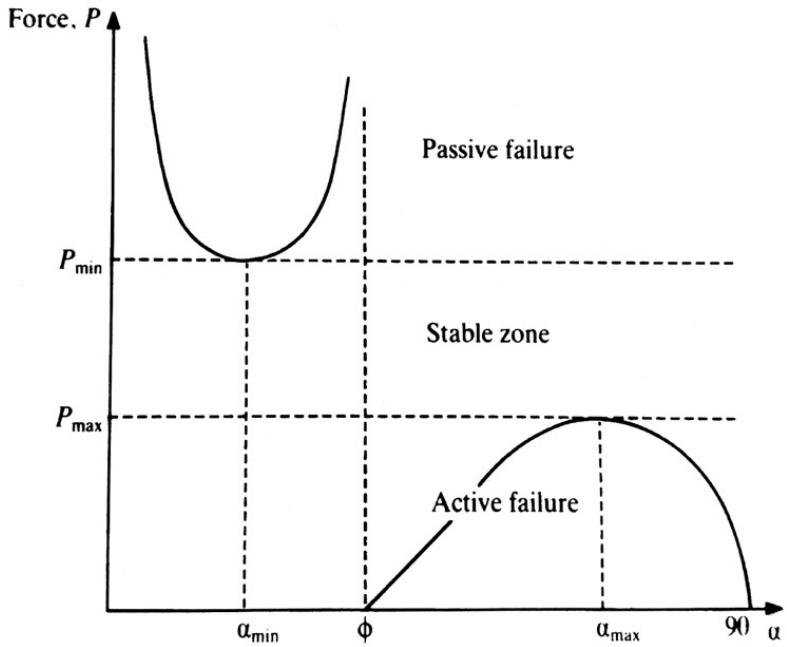
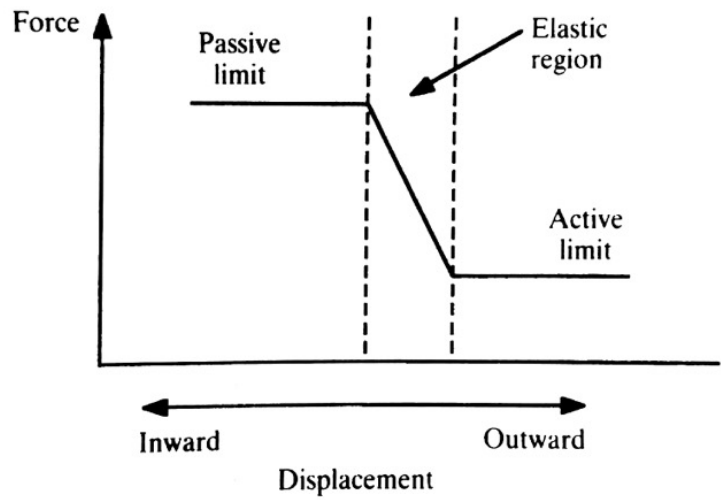
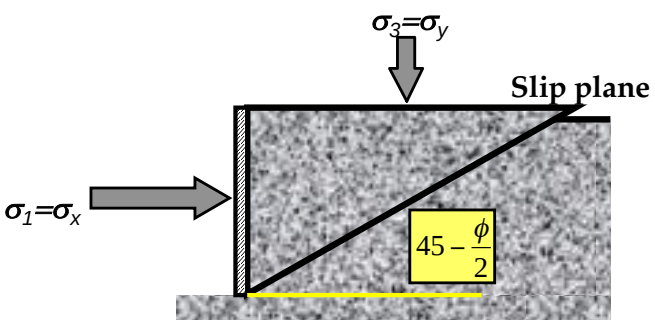
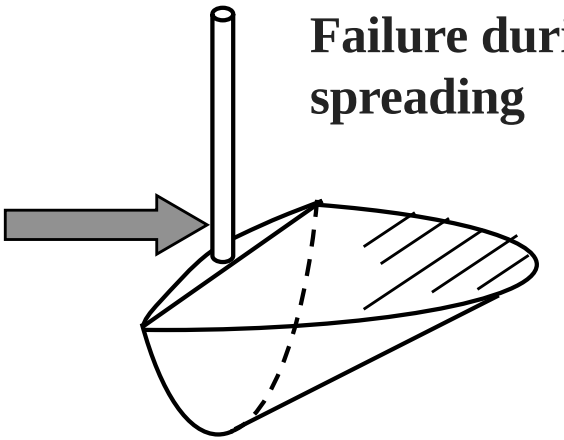


Figure 4.4 Active and passive wall forces.



Dependence of the wall force on the displacement of the wall.





Failure during spreading

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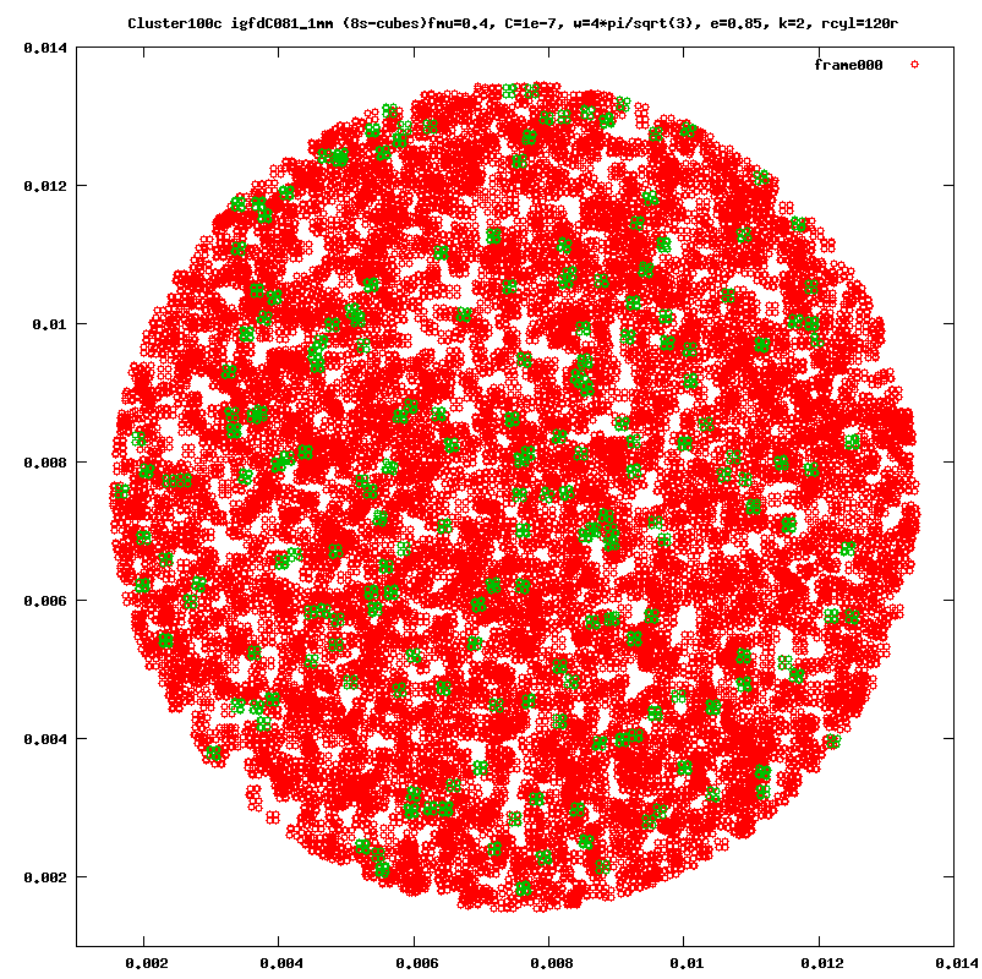
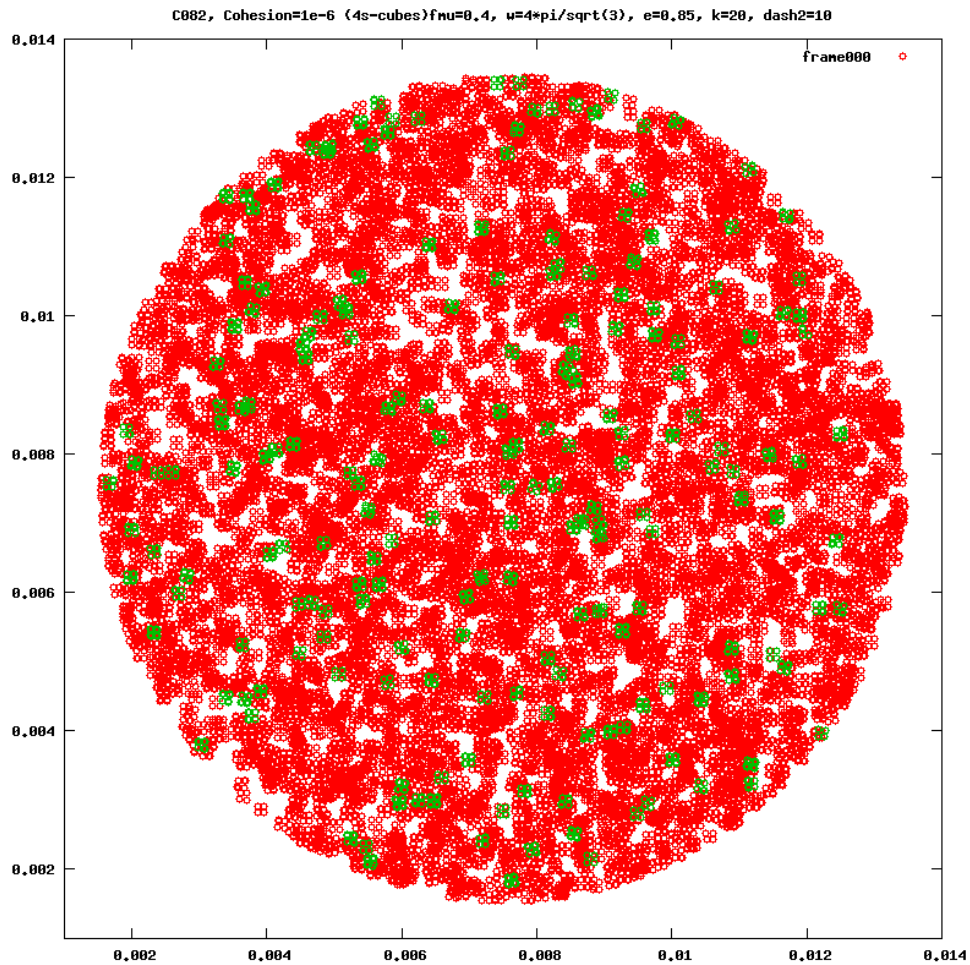
FEM stress distributions based on measured yield surface: DEM Lunar Regolith Example (non-cohesive vs. cohesive)





From: Otis Walton, Grainflow Dynamics

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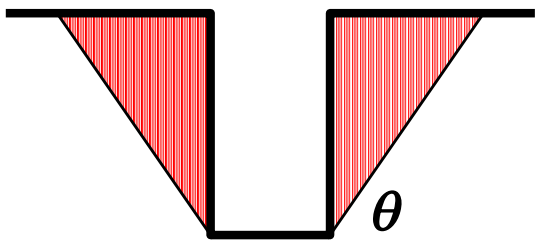
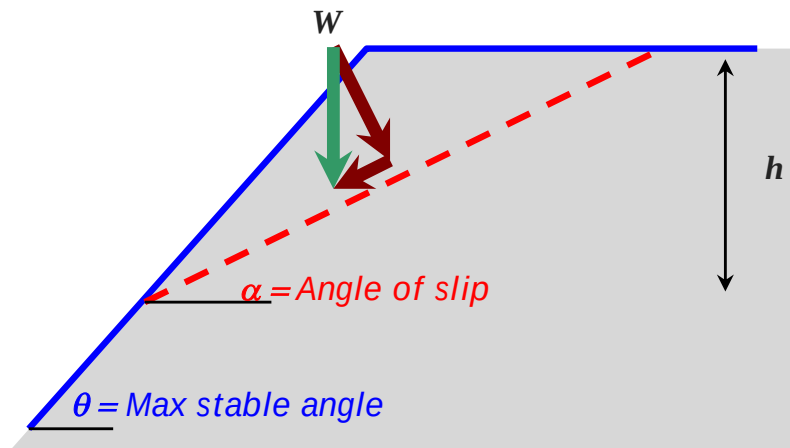
Force balance on a wedge (undergrad civil engineering!)

(after Nedderman)

- **Maximum depth of a trough, dependent on soil cohesion, *c*. (Balance of wt. and shear force on plane)**
- **Solving for *θ*, "angle of repose" is a function of size of wedge, *h*! (or scale of avalanche)**





$$Cohesionless: \quad \theta = \phi = \alpha$$

$$Cohesion: \quad \frac{\rho g h}{2c} = \frac{2\sin\theta\cos\phi}{1-\cos(\theta-\phi)} \quad and \quad \alpha = \frac{1}{2}(\theta+\phi)$$

$$so \quad \theta = fn(\phi, c) \quad AND \quad \theta = fn(h) \;!!$$

$$\boxed{f_c = \left\{ \frac{2}{\cos\phi}\left(\frac{1+\sin\phi}{1+\sin\phi_e}\right)\left[\sin\phi_e - \sin\phi\right] \right\}\sigma_1}$$

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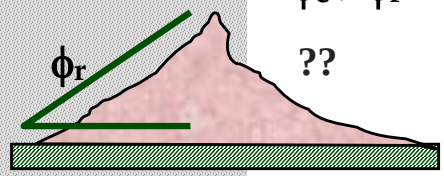
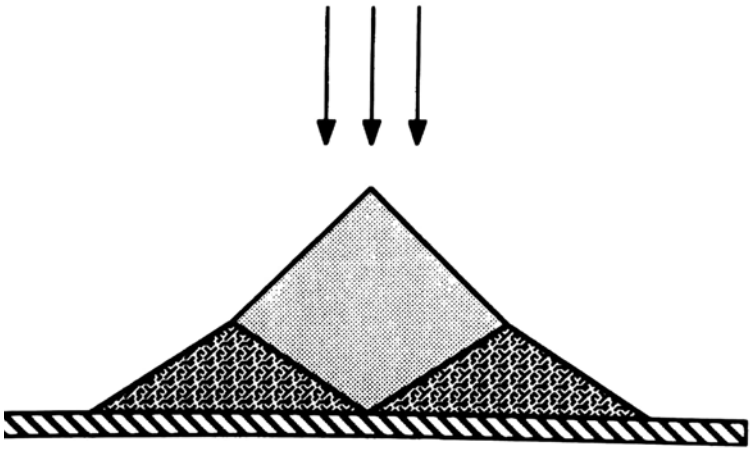
Possible shapes of a pile and variation of angle of repose

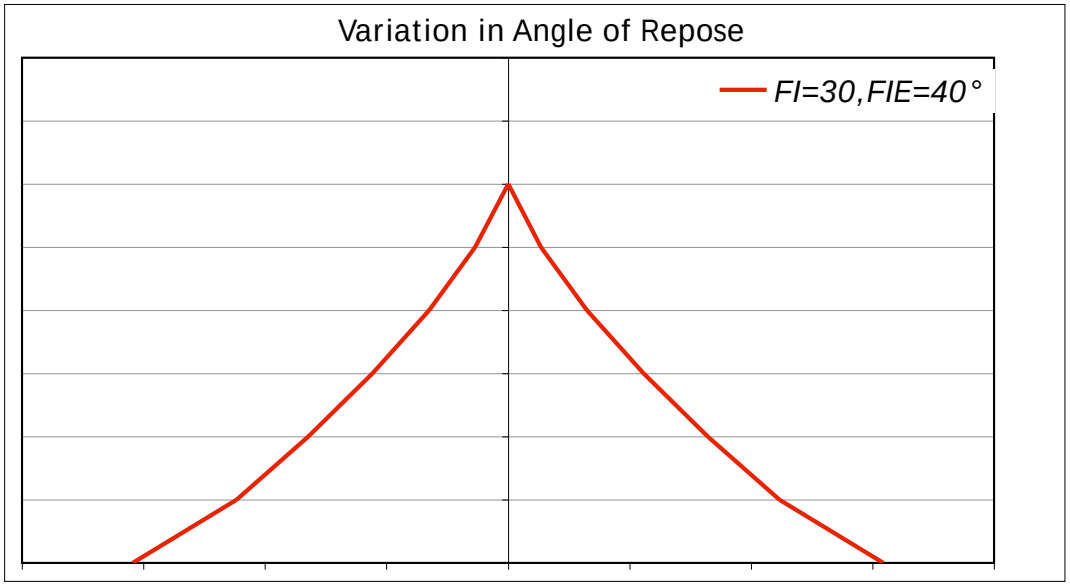


$\phi_e > \phi_r > \phi$

??



Segregation of material during pouring onto a conical pile.



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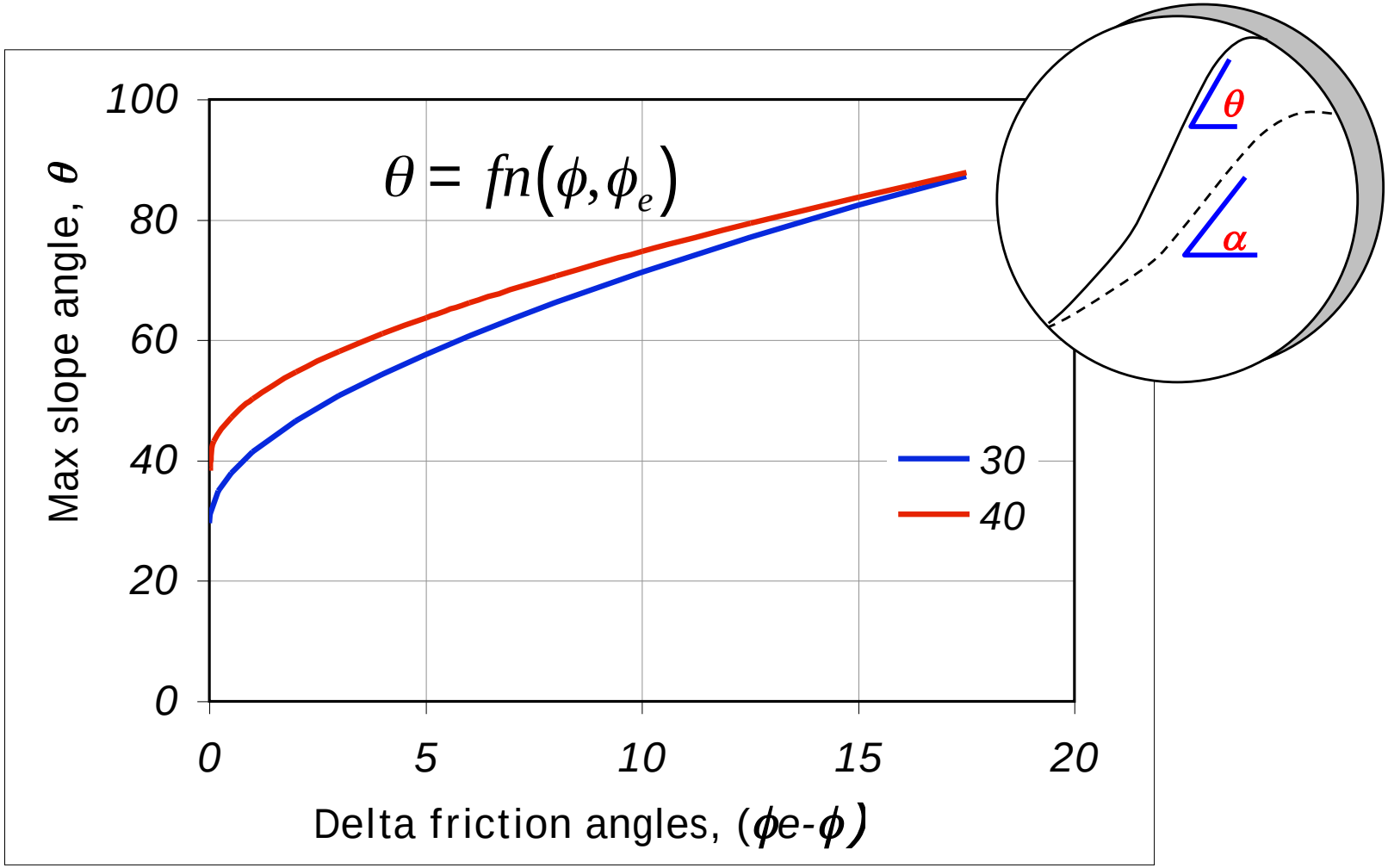
Maximum stable angle depends on friction angle difference, or unconfined strength

$$or \quad \frac{\rho g h}{\sigma_1} = \left(\frac{4\sin\theta}{1-\cos(\theta-\phi)}\right)\left(\frac{\cos\phi}{1+\sin\phi_e}\right)\left[\sin\phi_e - \sin\phi\right]$$



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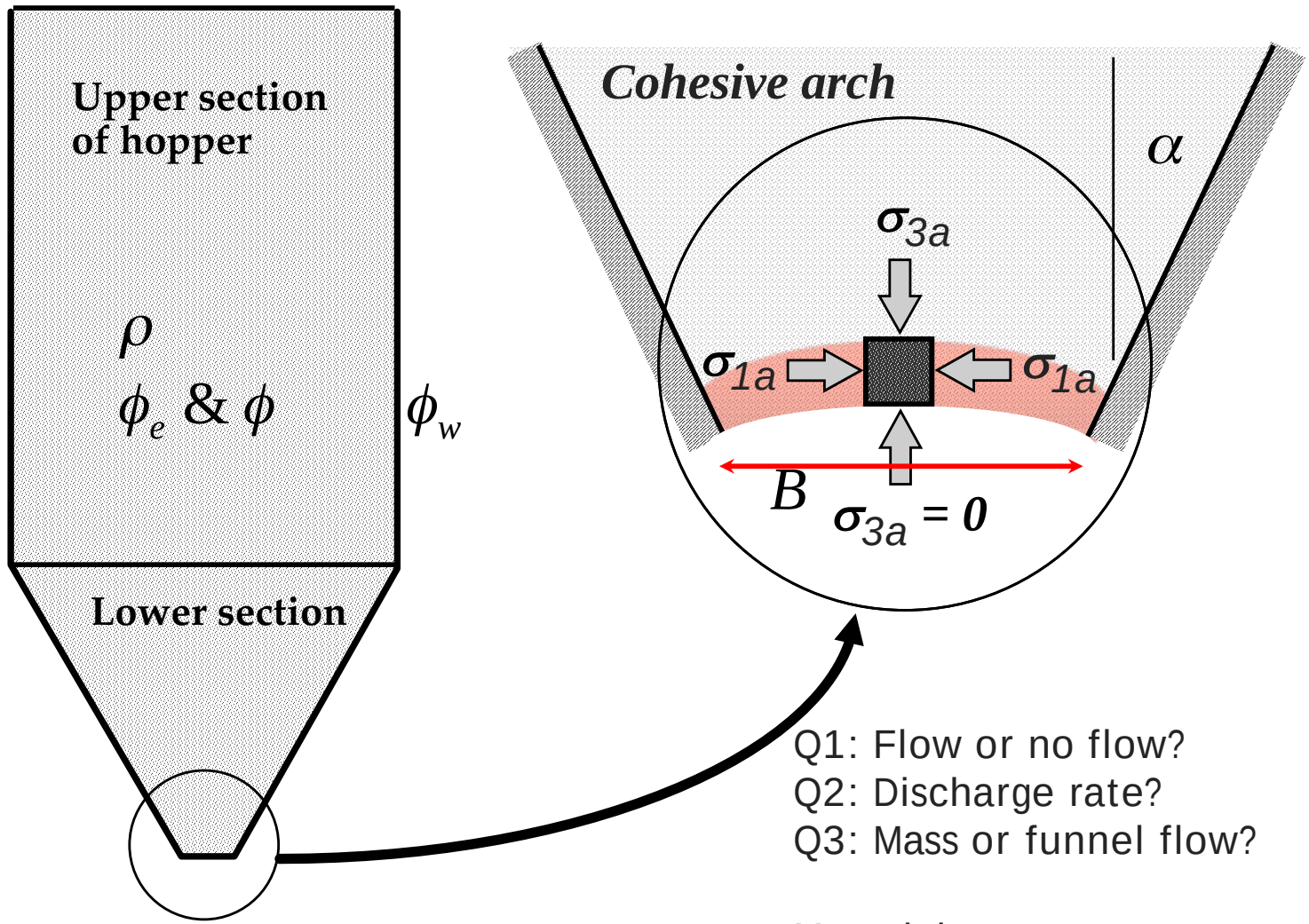
Bin Design Illustration



$$\rho, \phi, \phi_e, \phi_w, (k) = fns(\sigma_1)$$

Q1: Flow or no flow?
Q2: Discharge rate?
Q3: Mass or funnel flow?

Material property:

- Cohesive yield strength
- Minimum opening size

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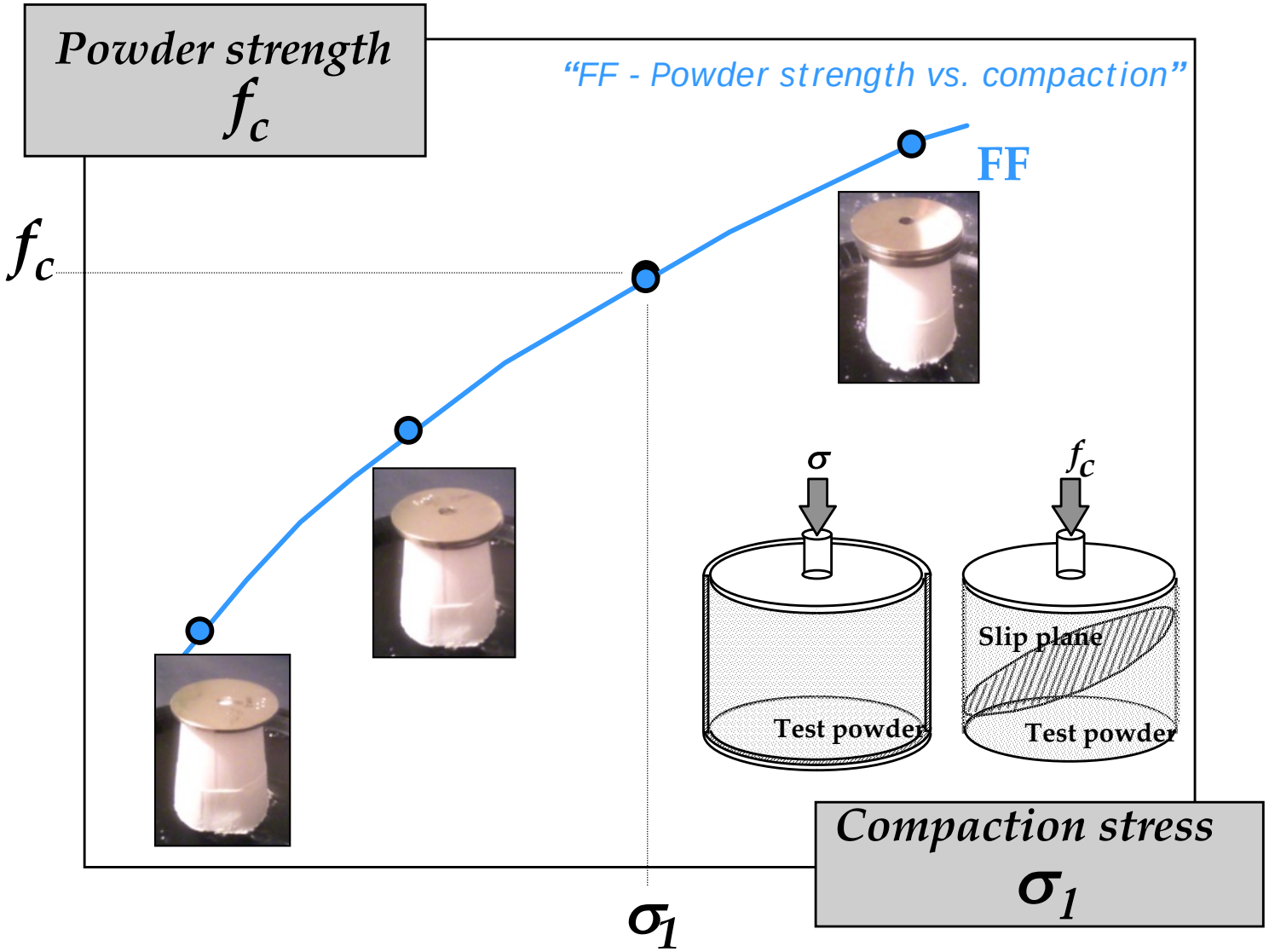
Powder strength vs. compaction pressure (The powder flow function - FF)



Increasing diameter, B --->

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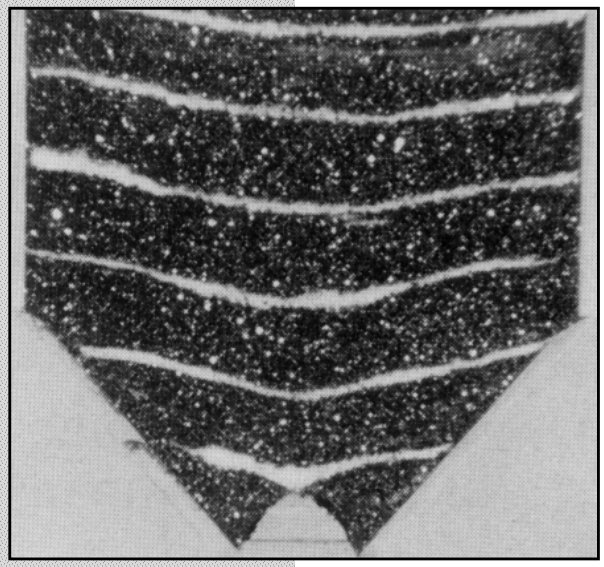
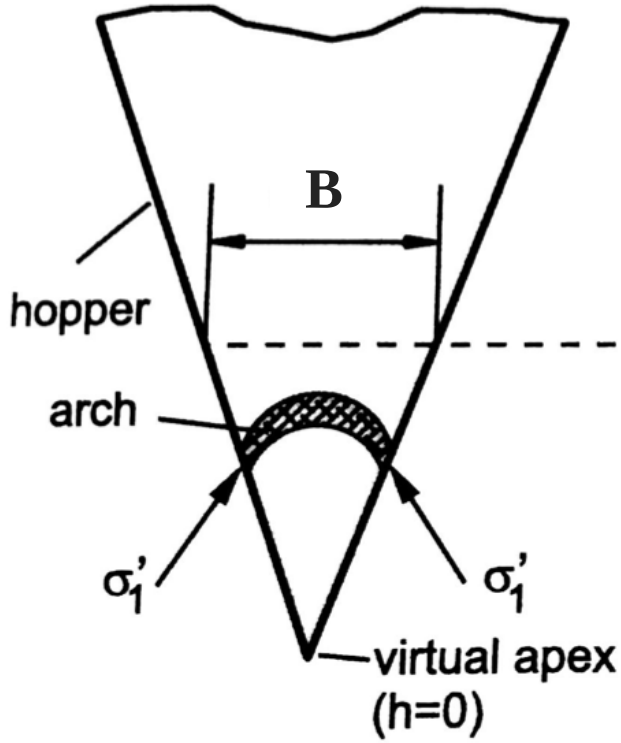
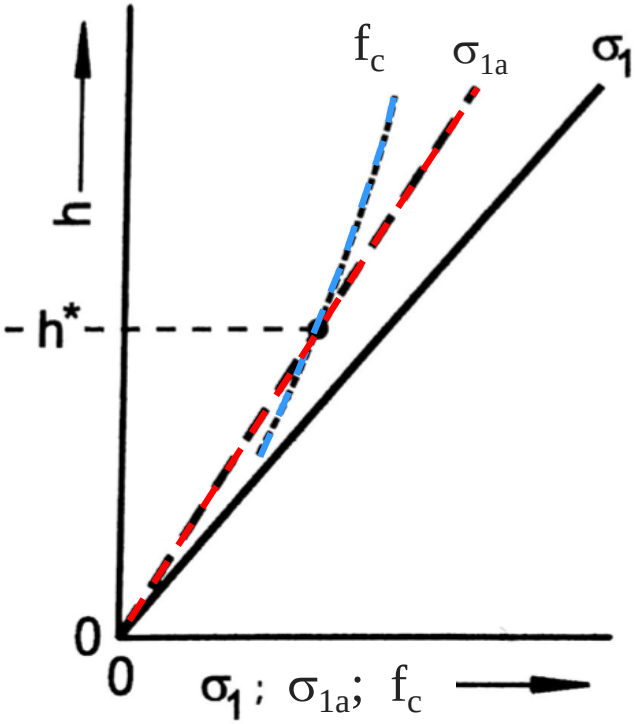
Arching calculation in mass flow – the arching flow factor

Mass flow: A stable arch







Stable arch (dome) broken by abutment stress (breaking stress) σ_{1a}

$$\sigma_{1a} = \frac{2r\rho g \sin\theta}{1+m} = \frac{\rho g B}{1+m} \qquad \sigma_1 = r\rho g\left(1+\sin\phi_e\right)s\left(\theta,\theta',\phi_w,\phi_e\right)$$

$$so \qquad ff = \frac{\sigma_1}{\sigma_{1a}} = \frac{\left(1+m\right)\left(1+\sin\phi_e\right)}{2\sin\theta}s\left(\theta,\theta',\phi_w,\phi_e\right)$$

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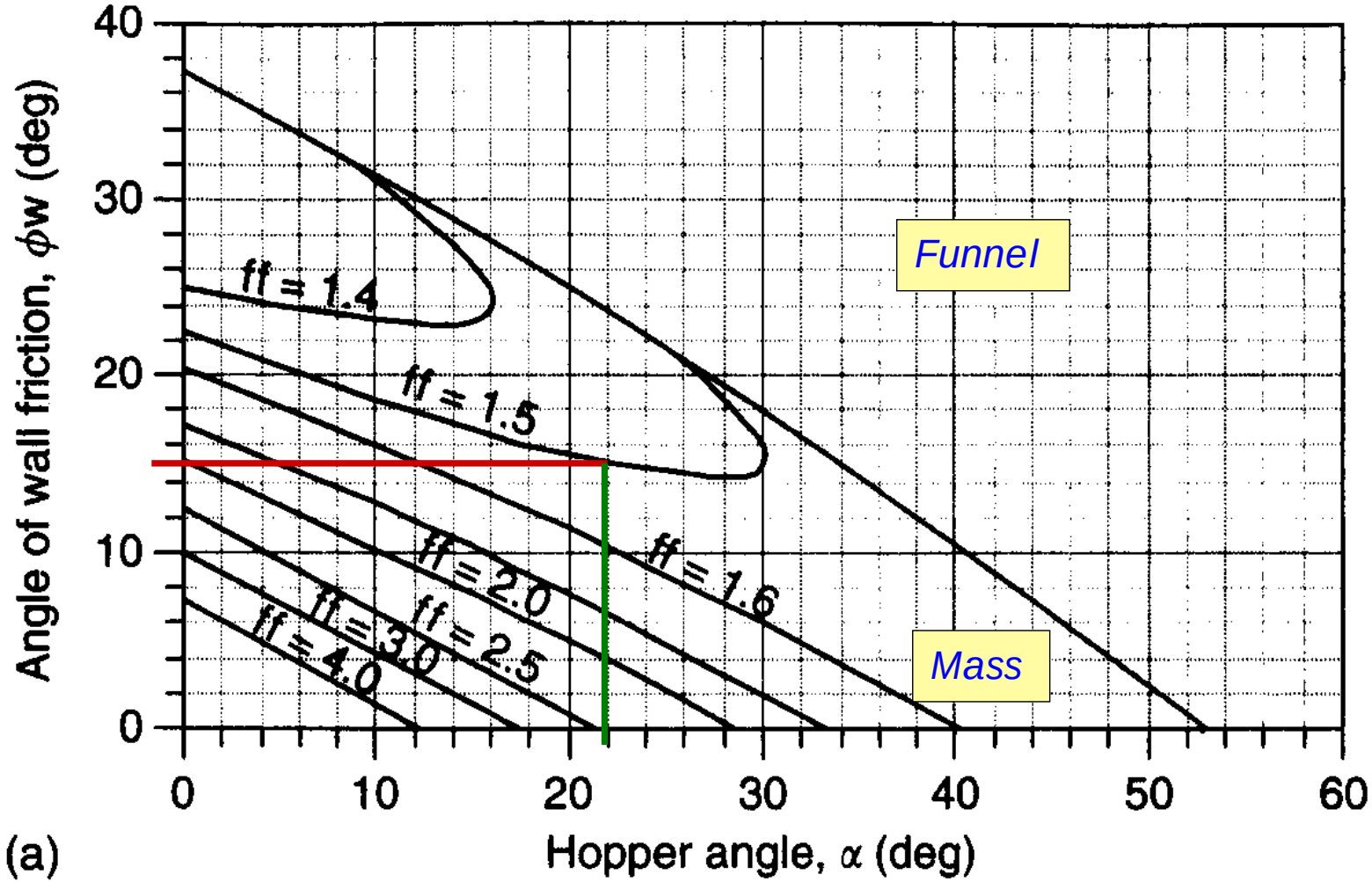
Mass or funnel flow?

(A. Jenike, Bulletins 108 & 123, Univ. of Utah)

Flow factor contours (conical, ϕ_e=40°)

- **Mass vs. funnel flow regimes & flow factor.**
- **The flow factor controls the breaking radial stress, $\sigma_{1a}=\sigma_1/ff$**



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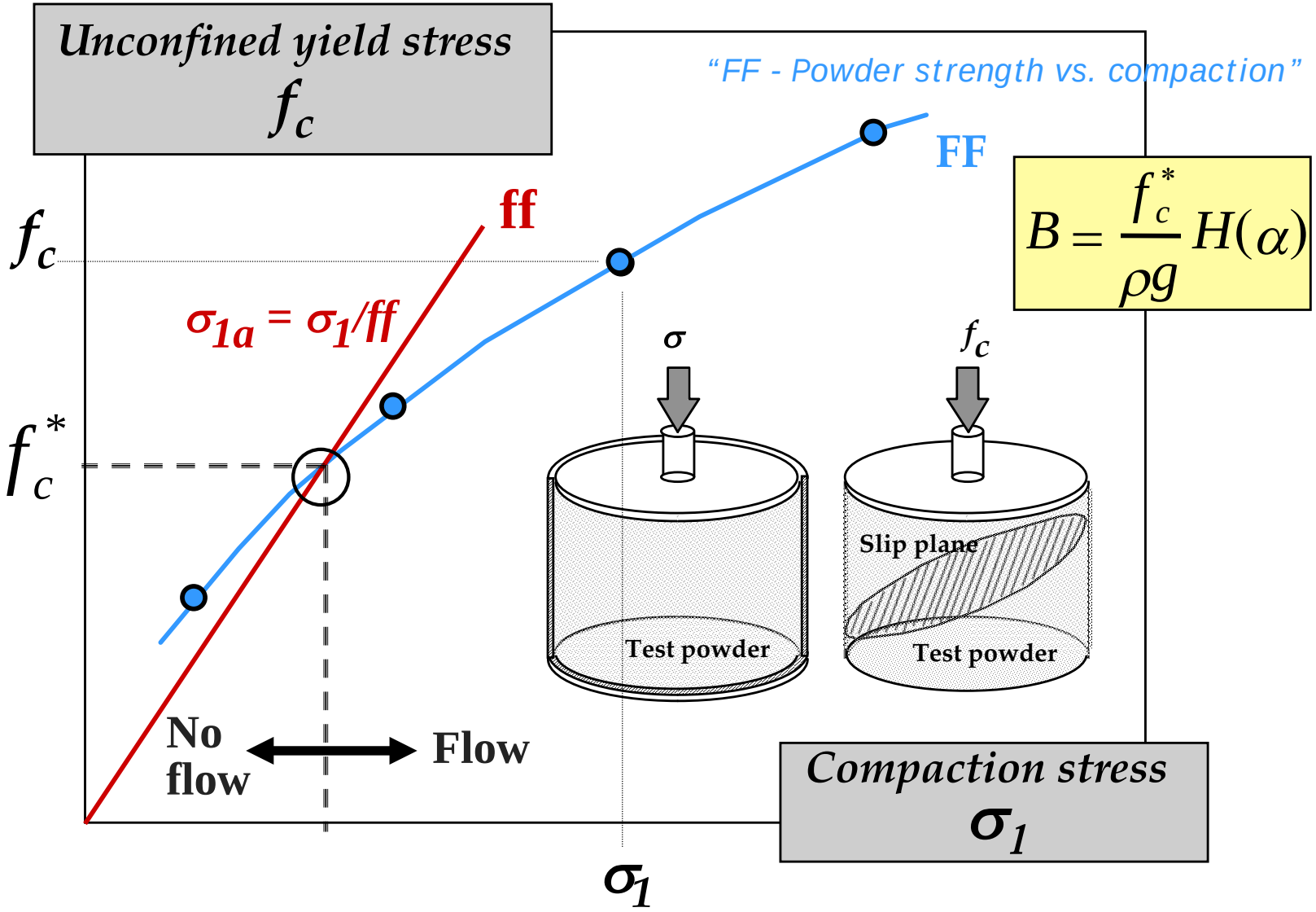
The unconfined yield strength (fc) and flow function FF (FF)



$$B = \frac{f_c^*}{\rho g} H(\alpha)$$

Deeper into hopper from apex
Increasing diameter B

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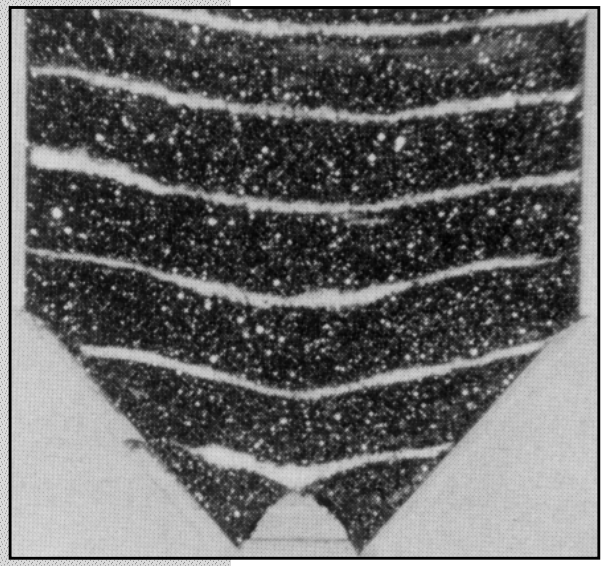
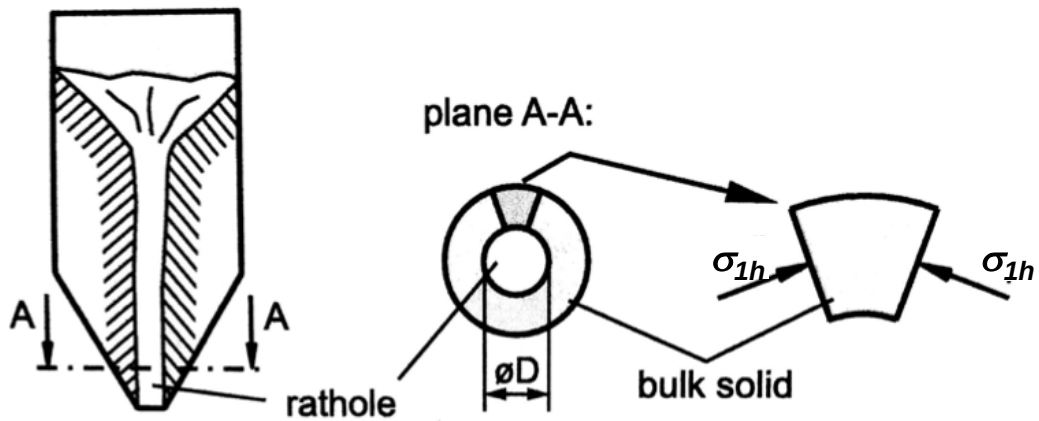
Minimum opening sizes to prevent arching & ratholing

Mass flow: A stable arch



Funnel flow: A stable pipe



$$B = H(\theta)\frac{f_c^*}{\rho g} \approx \begin{cases} \text{conical} & B_c \approx 2.2\dfrac{f_c^*}{\rho g} \\ \text{square} & B_s \approx 2\dfrac{f_c^*}{\rho g} \\ \text{slot} & B_p \approx 1.1\dfrac{f_c^*}{\rho g} \\ \text{funnel slot} & B_p \approx 1.15\dfrac{f_c^*\,(@\,ff = 1.7)}{\rho g} \end{cases}$$

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Bin Design Illustration: Liquid storage vs. powder storage?

Short & wide in aspect ratio

Liquid: Max pressure scales w/height

Powder: Max pressure scales w/diameter

Material property:

- Friction angles
- Bin pressures
- Bin angles for mass flow

Tall & narrow

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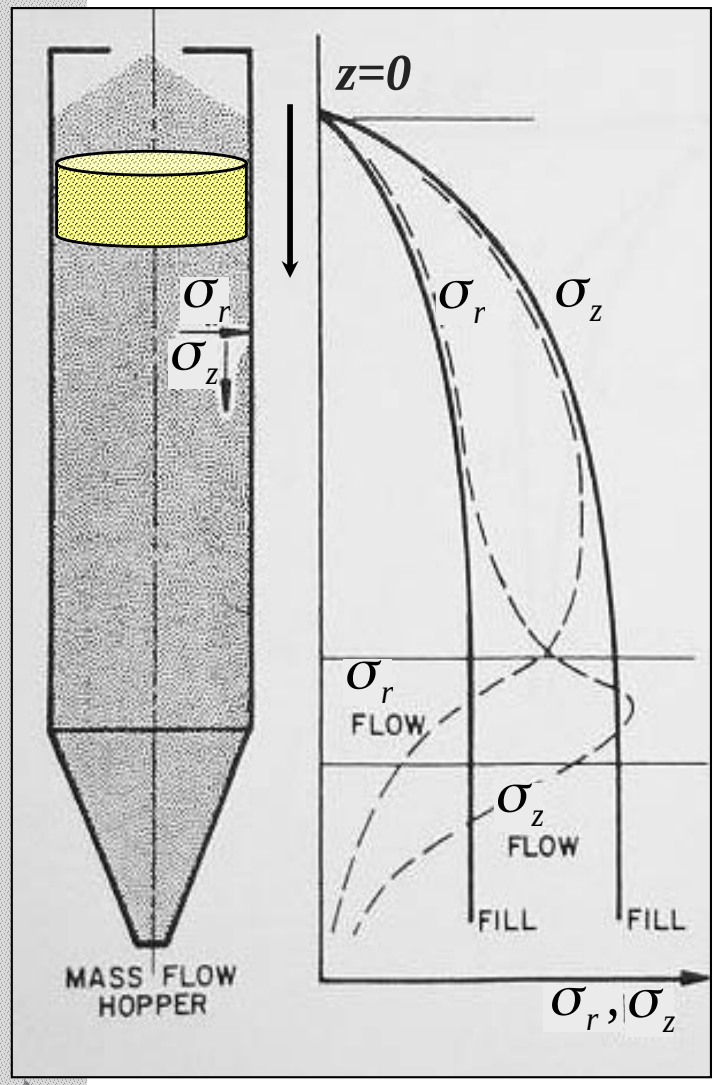
Janseen relation (1895) – Effective head in silo filling



$$\mu_w = \tan\phi_w \qquad K_a = \frac{1-\sin\phi_e}{1+\sin\phi_e} \approx 0.4$$

$$\sigma_r = K_a \sigma_z \qquad \tau_w = \mu_w \sigma_r = \mu_w K_a \sigma_z$$

$$\sigma_1 = \sigma_z = \frac{\rho g D}{4\mu_w K_a}\left(1 - e^{-(4\mu_w K_a/D)z}\right)$$

$$\sigma_3 = \sigma_r = \frac{\rho g D}{4\mu_w}\left(1 - e^{-(4\mu_w K_a/D)z}\right)$$

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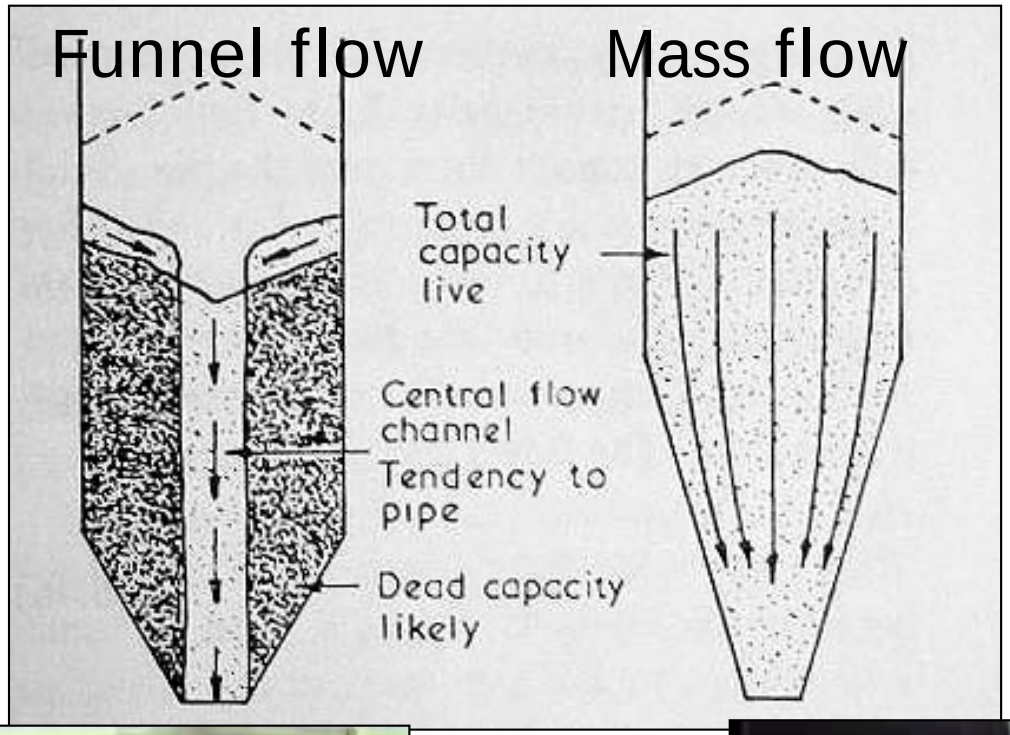
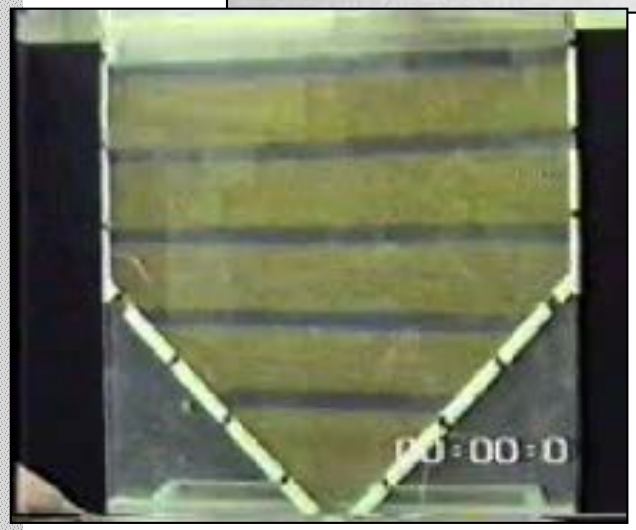
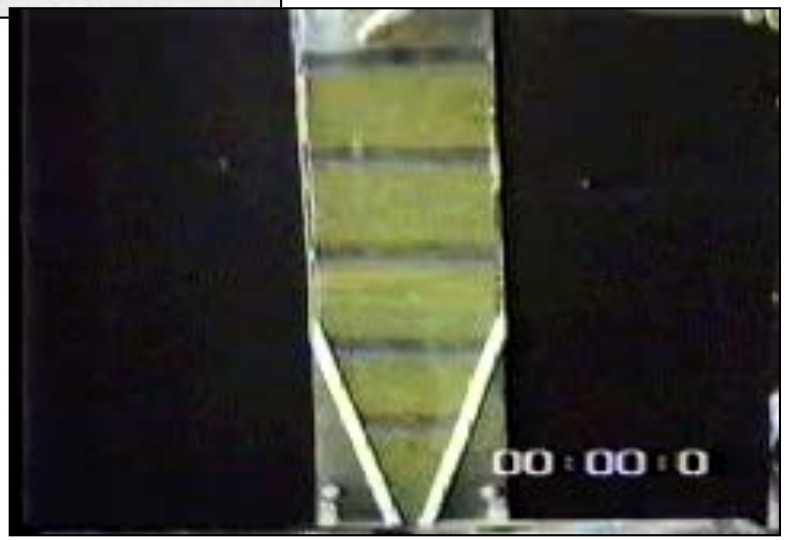
Hopper discharge patterns?

Laboratory Demonstrations in Particle Technology By Martin Rhodes and Alfi Zakhari:
http://users.monash.edu.au/~rhodes/CD.htm







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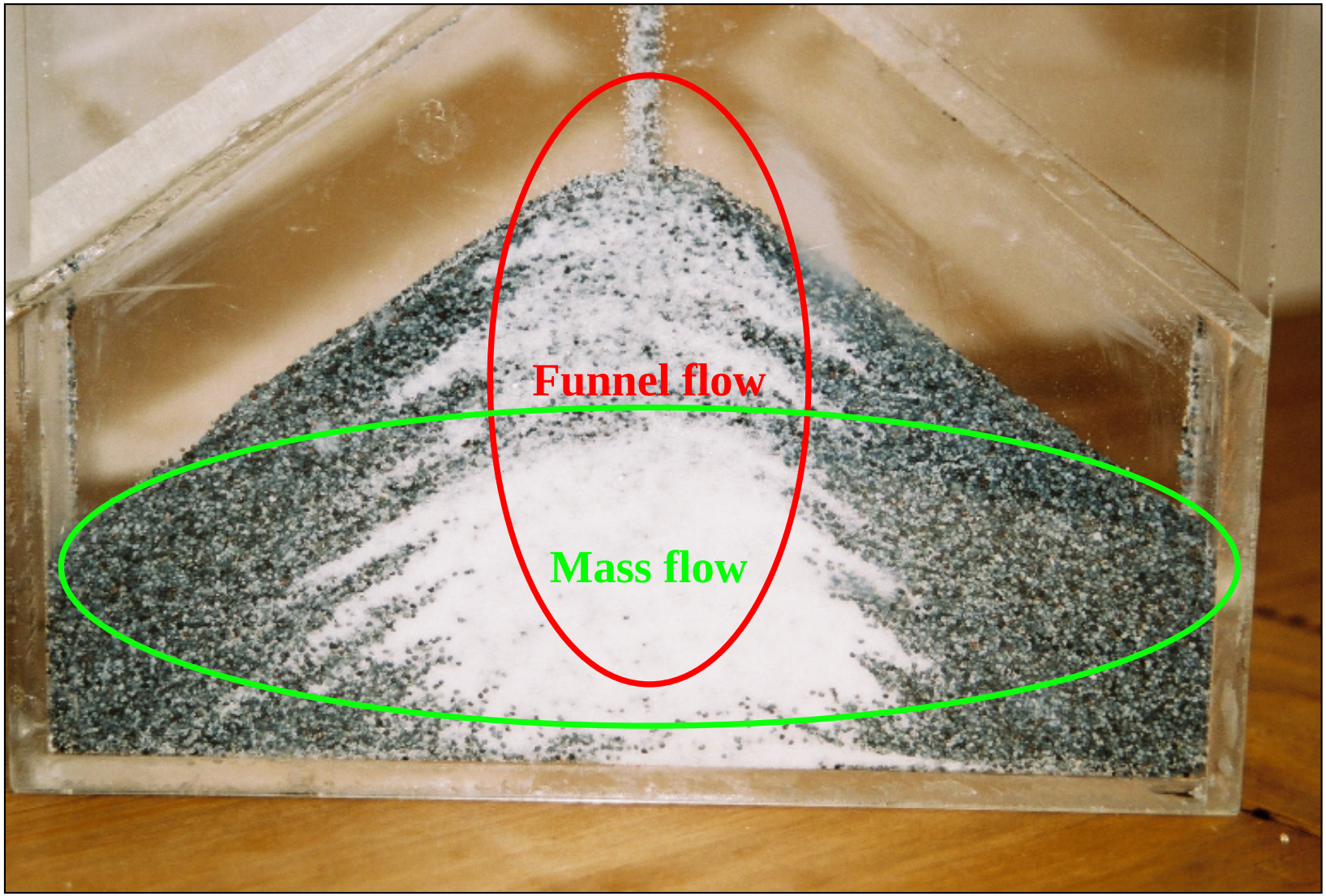
Why mass flow hoppers?

All powders are mixtures (even a single powder), which can segregate.



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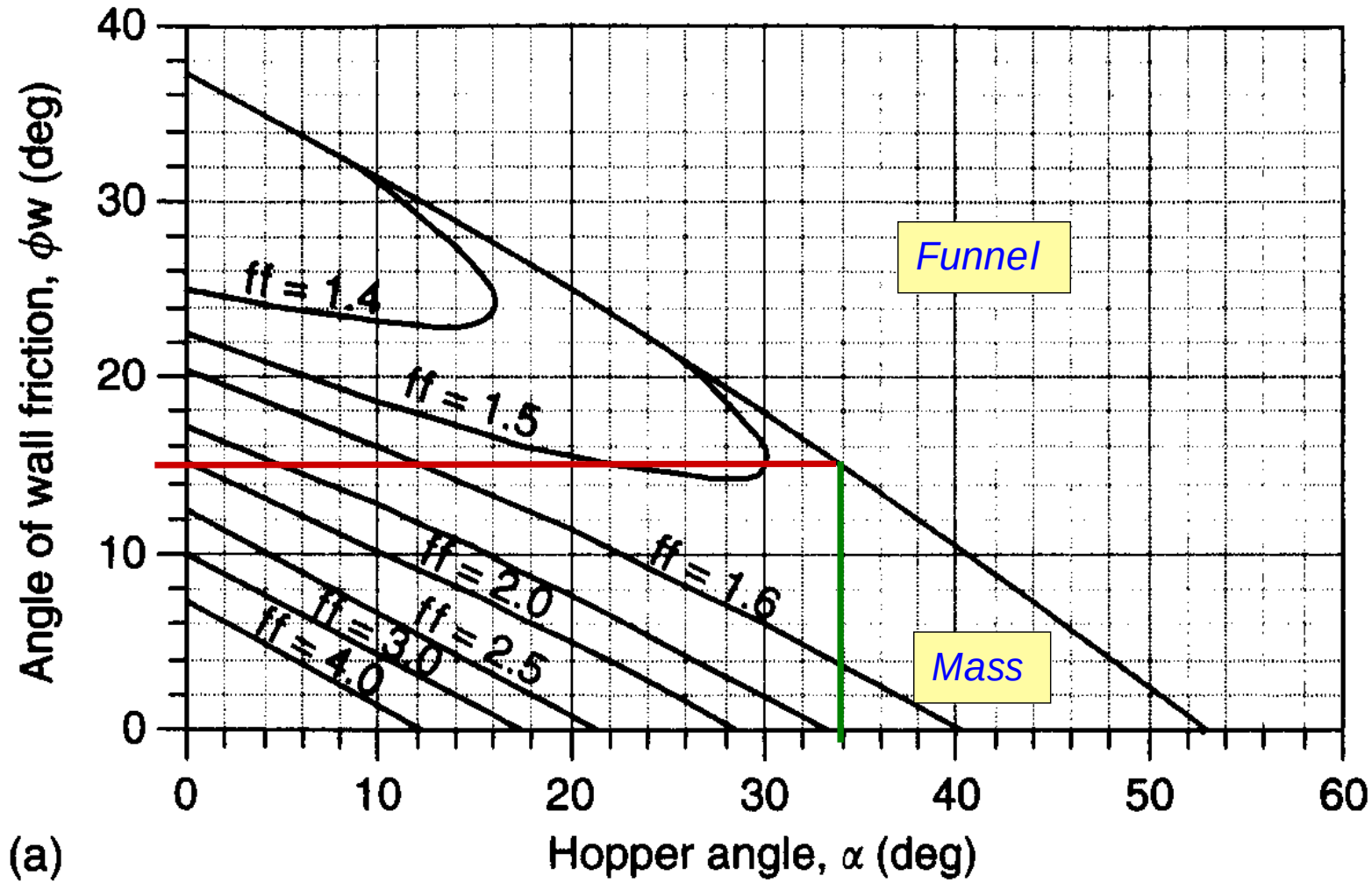
Mass or funnel flow?

(A. Jenike, Bulletins 108 & 123, Univ. of Utah)

Flow factor contours (conical, ϕ_e=40°)

- **Mass vs. funnel flow regimes & flow factor.**
- **The flow factor controls the breaking radial stress, $\sigma_{1a}=\sigma_1/ff$**



(a)

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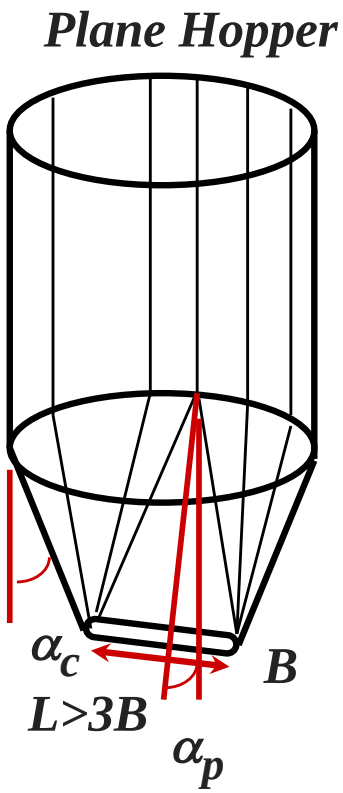
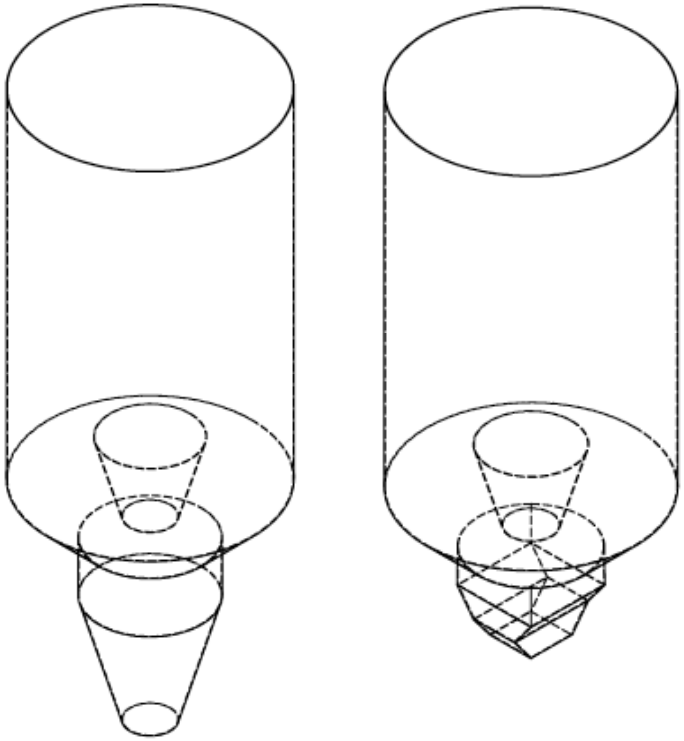
Variations: Slot, Cone-in-Cone, Stacked Wedge





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How "fast" does powder flow? Why an hour glass works!

Beverloo eq. for cohesionless solid:
(Beverloo et al., Chem.Eng.Sci., (1961) 15, 260)

$$W_o \approx 0.58\rho\sqrt{g}\,B^{2.5} \approx 0.52\rho A\sqrt{2gB}$$

Contrasting with inviscid fluid:

$$W = 0.64\rho A\sqrt{2gH}$$

m=1 (slot) or 2 (conical), α=vertical half-angle, B=opening size, A=opening area, ff=flow factor, Rel=flow index=σ_1/f_c

Discharge of cohesion solid:
(Johanson, Trans.Soc.Min.Eng., March (1965))

$$W_s = 1.354\,W_o\sqrt{\frac{1}{2m\tan\alpha}\left(1-\frac{ff}{Rel}\right)}$$

Discharge rate:

- Increases with $B^{2.5}$
- A cohesionless solid does NOT depend on height
- Decreases with increasing cohesive strength
- Decreases with decreasing permeability

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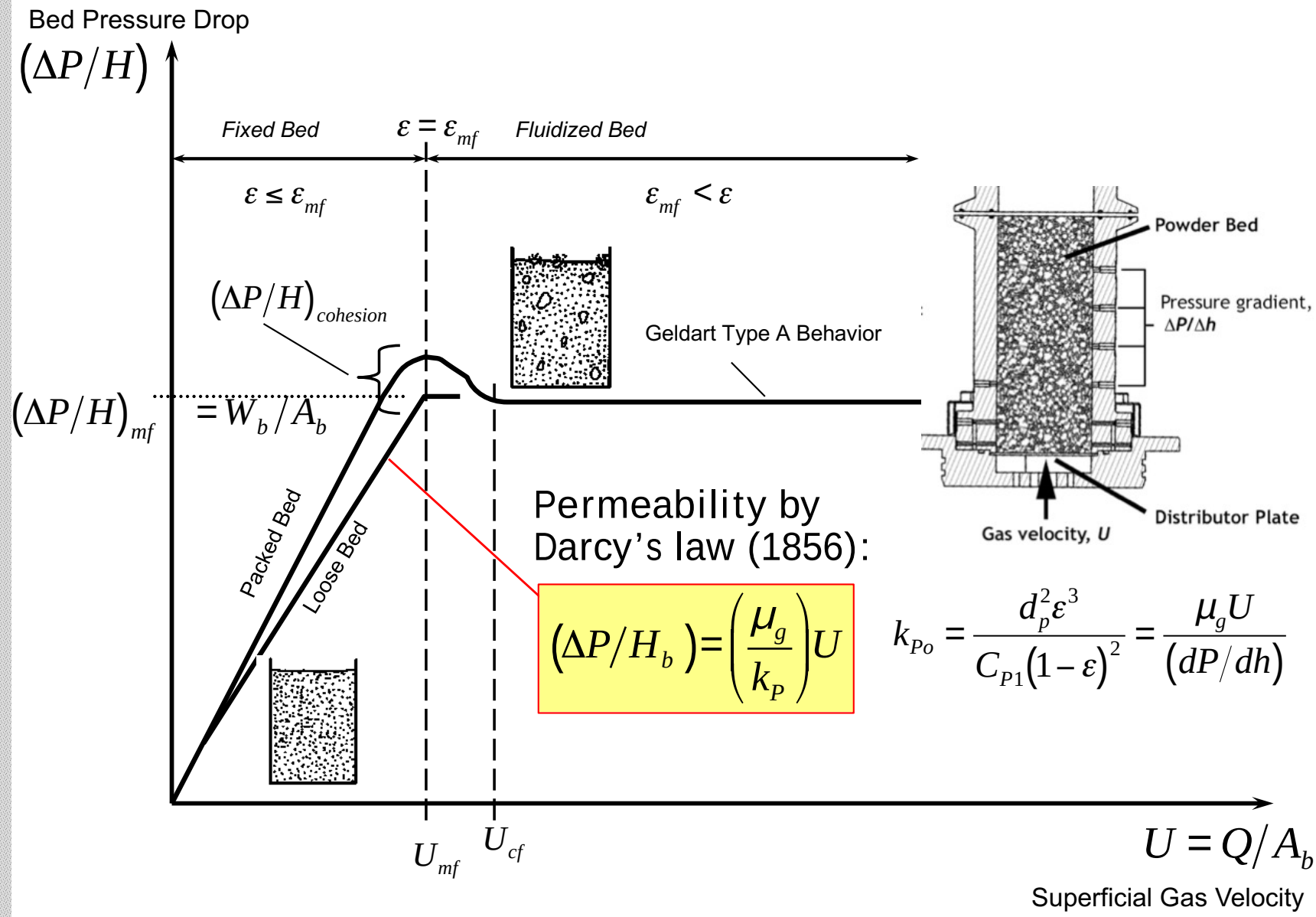
Fixed-Bed Permeability & Fluidization

(Ennis (ed.), Section 21, Perry's Handbook 8th ed.)



Permeability by Darcy's law (1856):

$$(\Delta P/H_b) = \left(\frac{\mu_g}{k_P}\right)U$$

$$k_{Po} = \frac{d_p^2 \varepsilon^3}{C_{P1}(1-\varepsilon)^2} = \frac{\mu_g U}{(dP/dh)}$$

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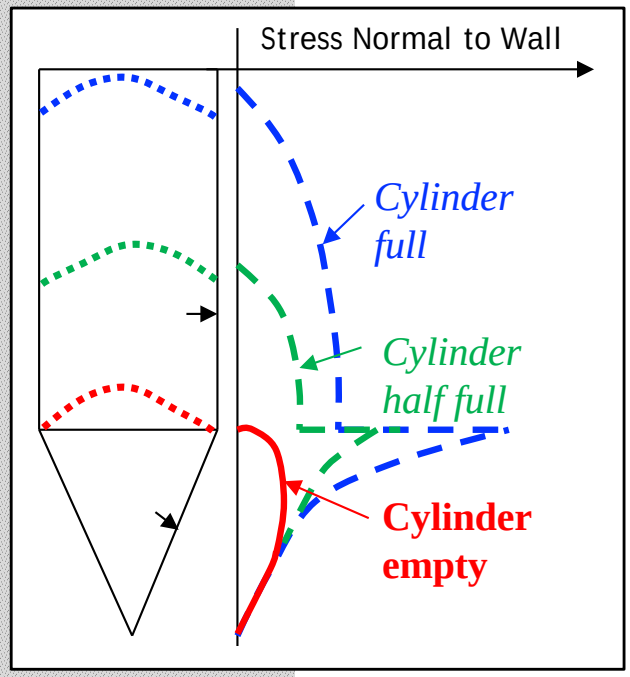
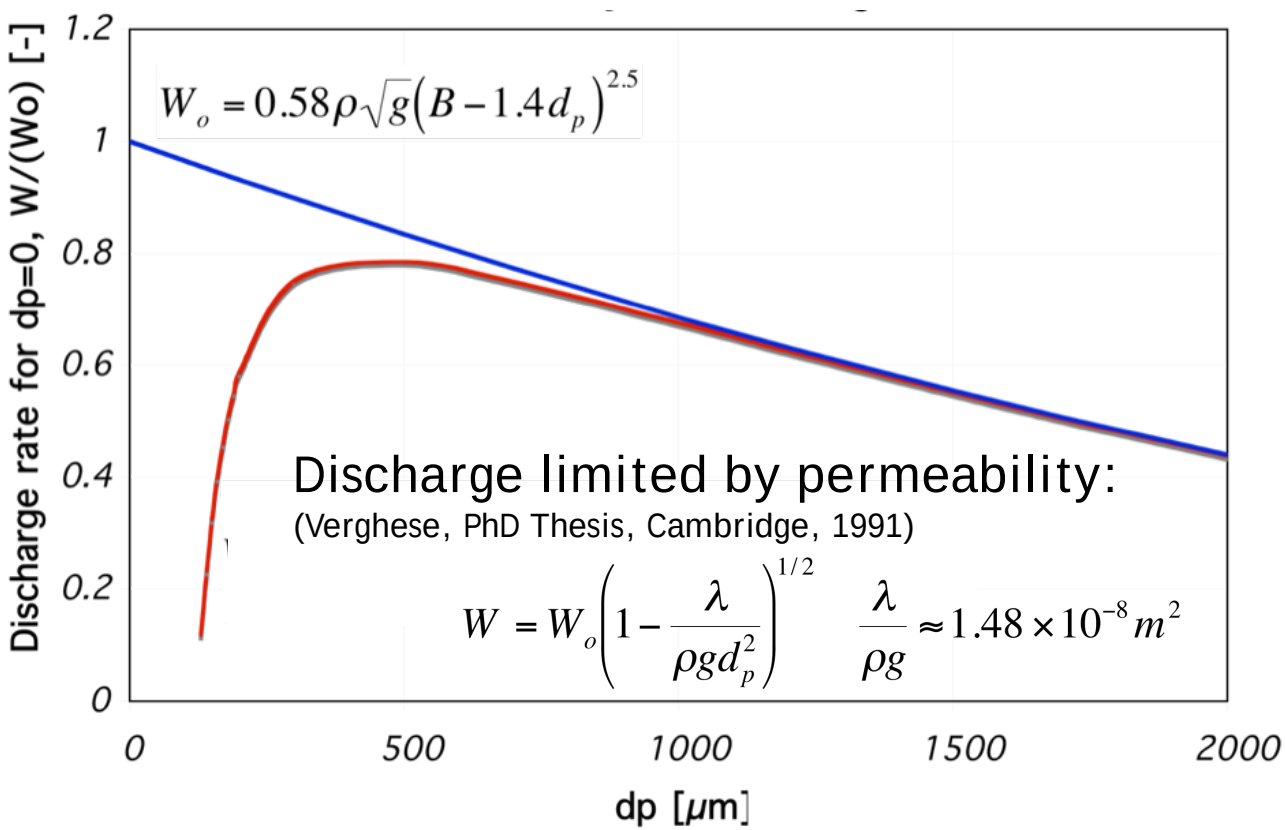
Impact of permeability on mass discharge rate



- Solids pressure decreases towards outlet, giving powder expansion & increasing permeability
- Leads to decreases in pore pressure which can arrest powder flow if sufficient vacuum develops



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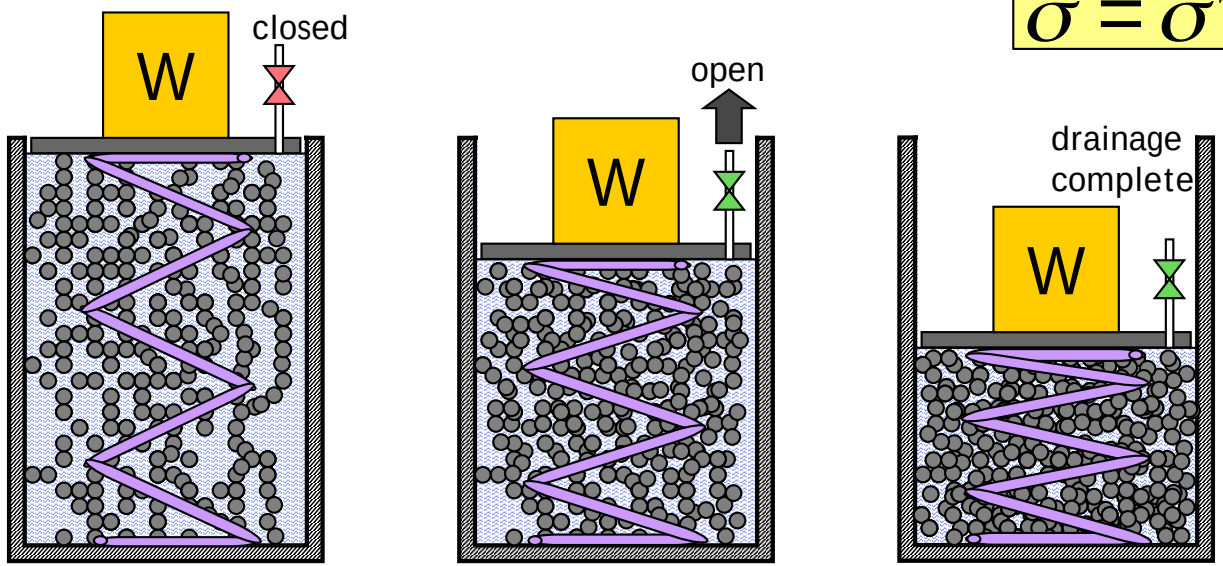
Interaction of interstitial air, solids pressure, deareation

$$\sigma = \sigma' + u$$



- Air pressure initial holds applied stress
- Air pressure decays at a rate given by permeability & void volume decreases
- Stress is transmitted to solids, with solids stress increasing
- Air pressure decay given by wave equation:

$$\frac{\partial u}{\partial t} = C_v \frac{\partial^2 u}{\partial z^2} \qquad and \qquad C_v = \frac{k_p}{m_v \mu_g}$$

$$\bar{U}(t) = \begin{cases} \sqrt{\frac{4}{\pi} T} \approx \sqrt{\frac{k_p t}{m_v \mu_g}} \frac{1}{H} & for \quad T < 0.2827 \\ 1 - \frac{8}{\pi^2} e^{-\pi^2 T/4} & for \quad T \geq 0.2827 \end{cases}$$

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Extension to other process flows & PBF

- Stress transmission
  - Powder & die friction
  - Tabletting & extrusion examples
- Roll pressing
  - Powder & roll friction
  - Compressibility & permeability
  - Nip pressure & limiting roll speed
- Powder bed fusion
  - Mechanical conveying
  - Print bed motion
  - Part/powder friction

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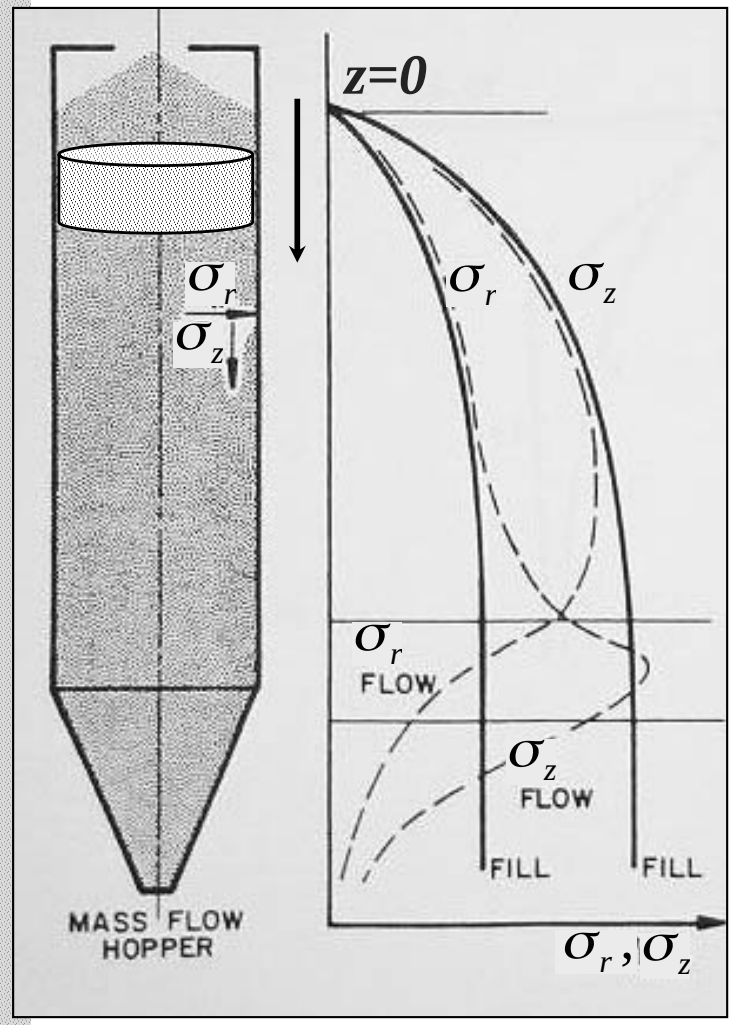
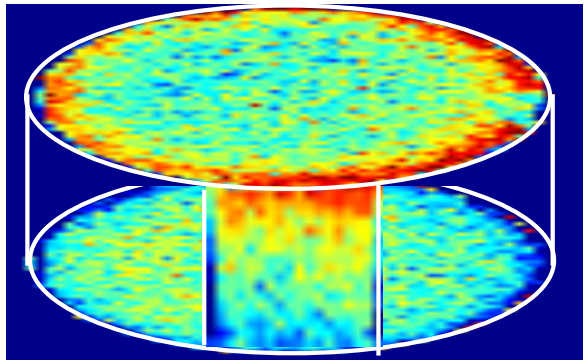
Tabletting, stress transmission & density gradients



NIR chemical imaging density



L2, L316,1% MgS,2000 lb

(Collaborative work with DPQR,FDA)

$$\sigma_z = \frac{\rho g D}{4\mu_w K_a}\left[1 - e^{-(4\mu_w K_a/D)z}\right]$$

$$\sigma_z = \sigma_o e^{-\left(4\mu_w K_\phi/D\right)z}$$

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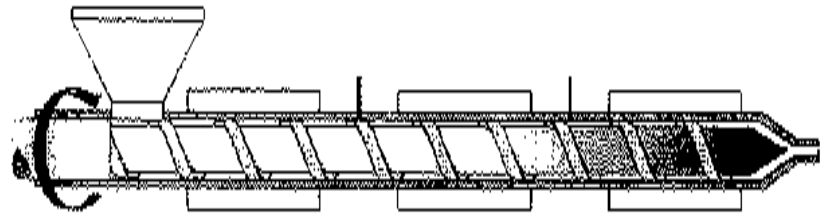
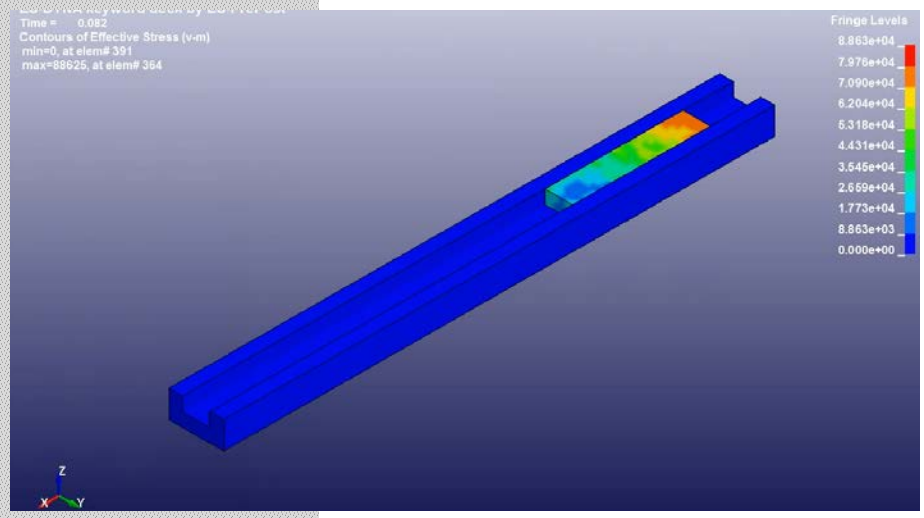
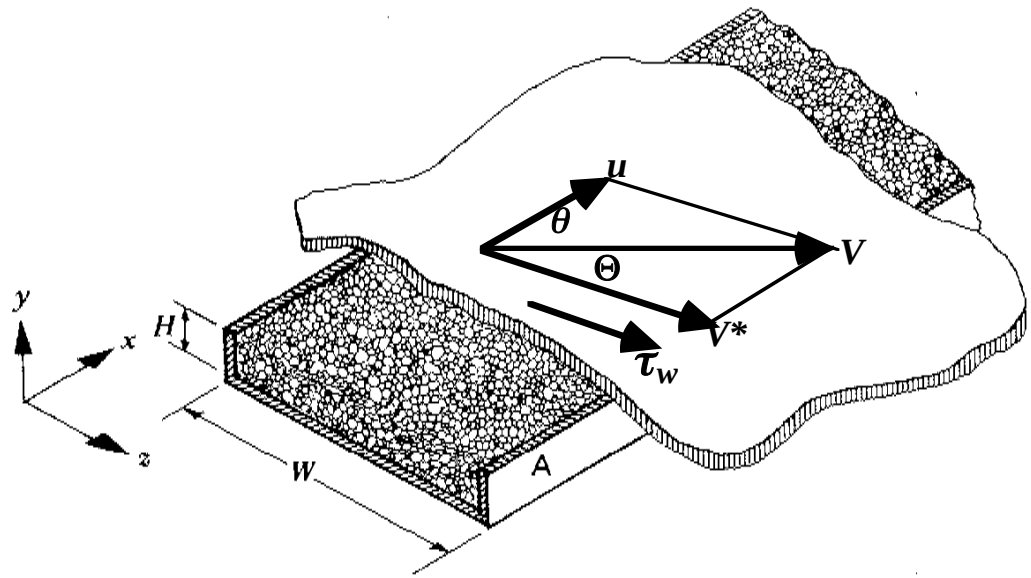
Paste Extrusion



Metering **Conveying** **Die plate extrusion**





$$\sigma_l = \sigma_o e^{+(\mu KC/A)L} = \sigma_o e^{+\mu K(4L/d)} \quad (\textit{for cylinders})$$

$$= \sigma_o e^{+\mu KL(2W+2H)/WH} \, (\textit{for rectangular channels})$$

$$\boxed{P = P_0 \exp\left[\left(w\mu_{barrel}\cos(\theta+\phi) - (w+2h)\mu_{screw}\right)\frac{LK}{wh}\right]}$$

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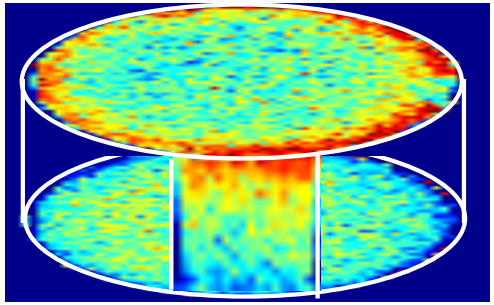
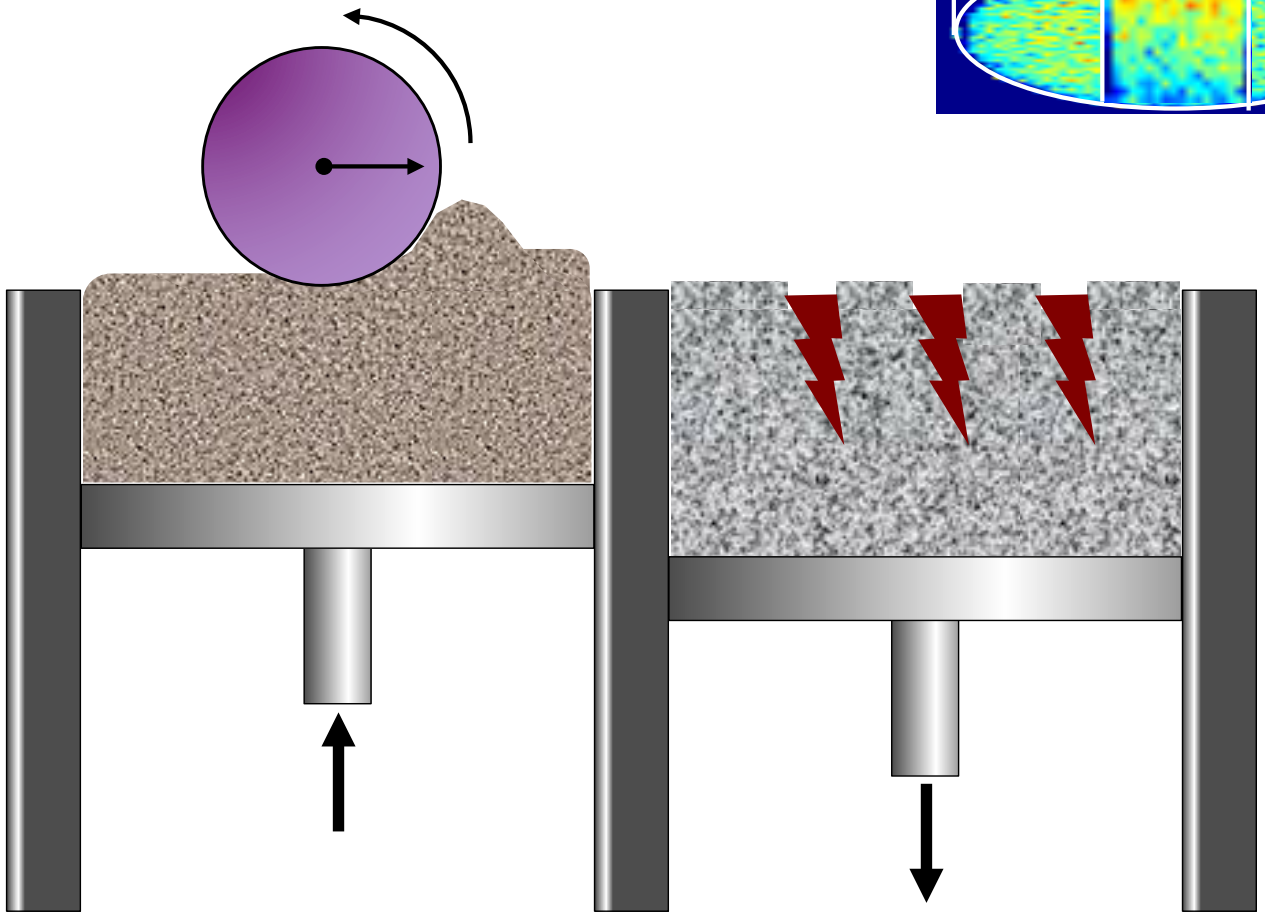
Extension to PBF (mechanical conveying and print bed)





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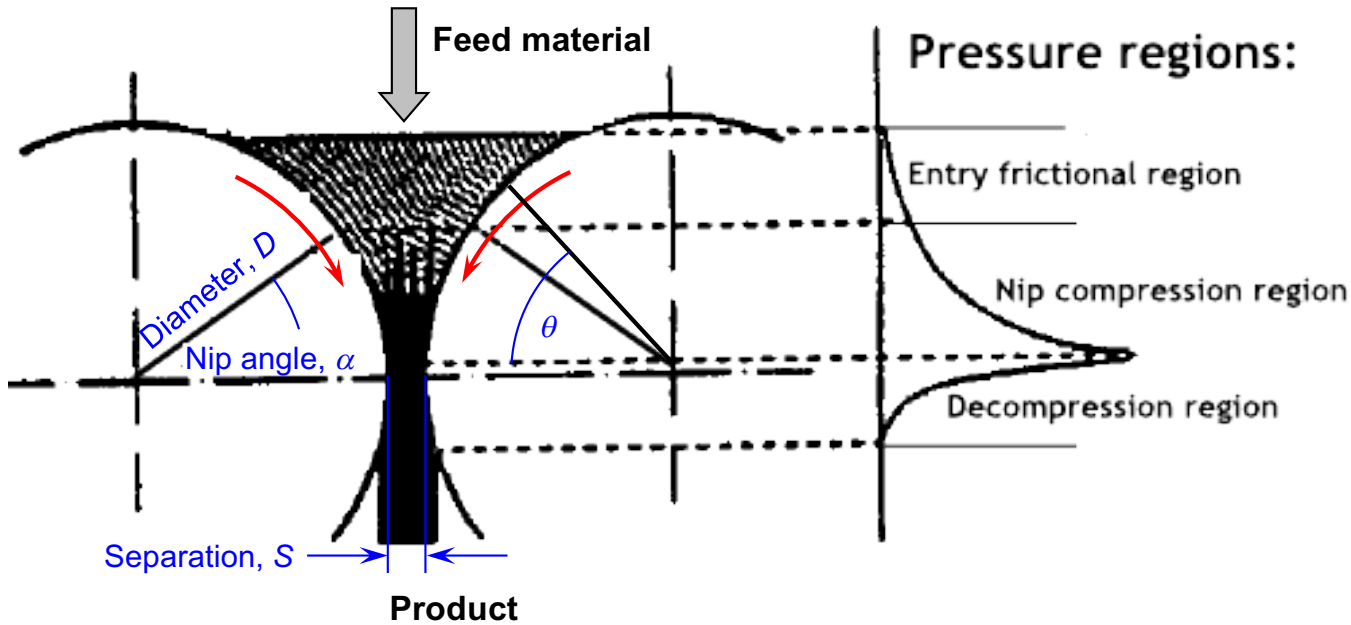
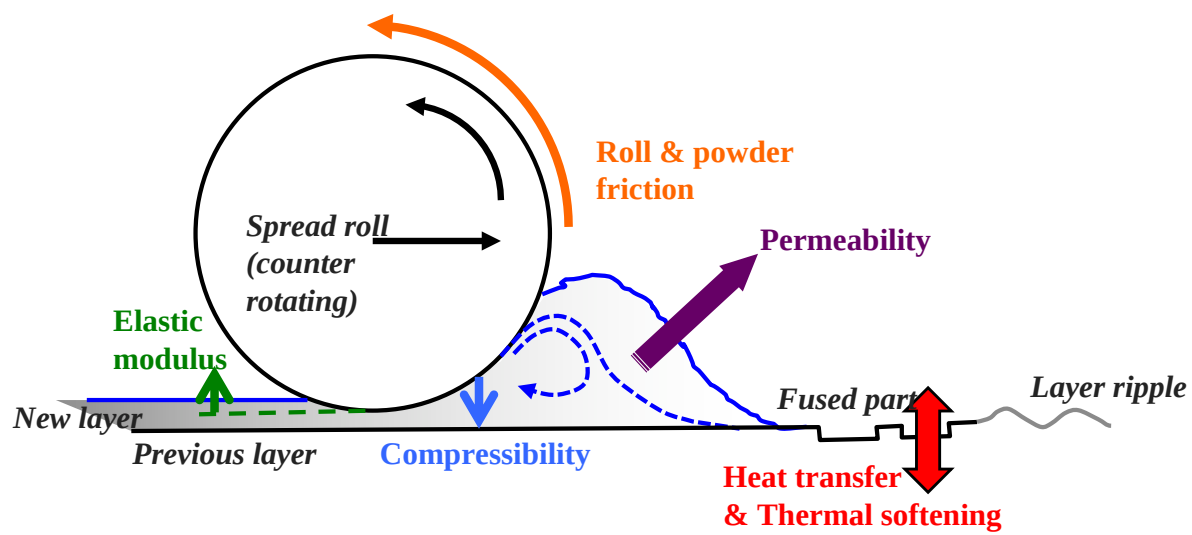
Rolling processes: The roll press & roller spreading





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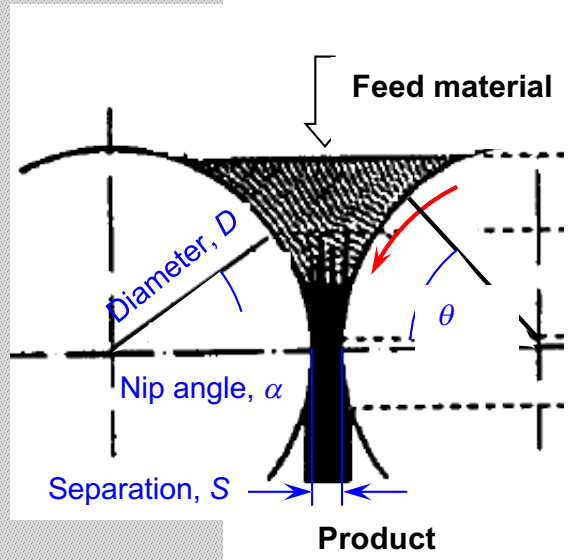
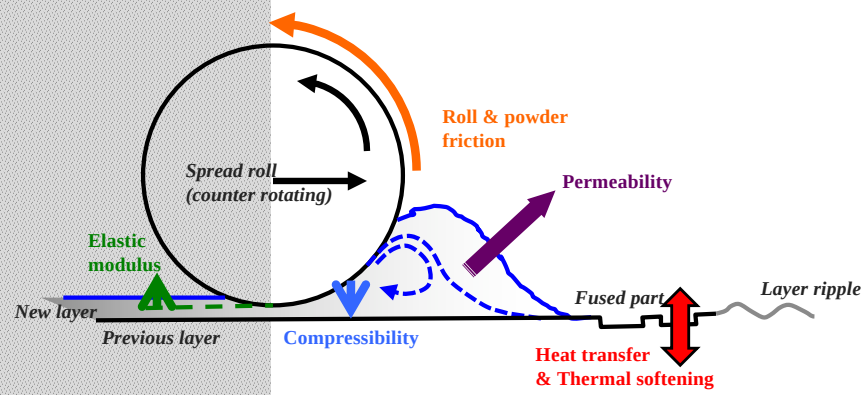
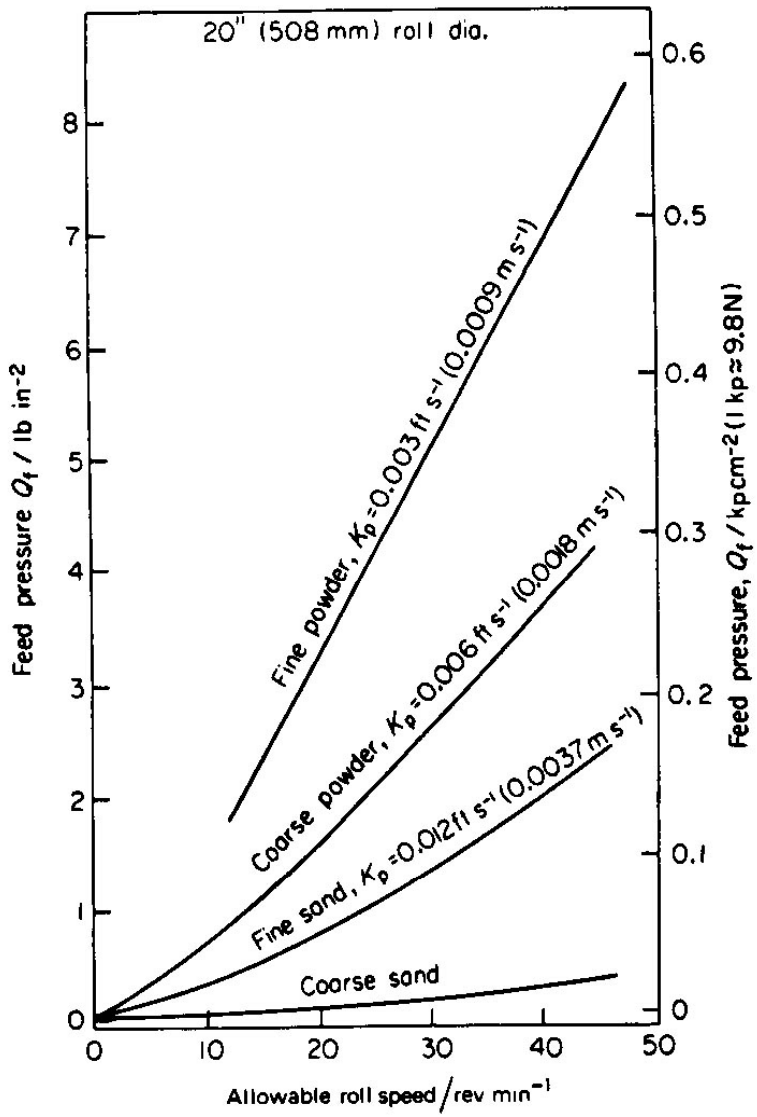
An example of air impact: Limiting roll speed







The effect of roll speed and permeability on the required feed pressure.

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3D PBF – "A Miniaturized Solids Processing Plant in a Box" Problem approach to solids issues:

- **Identify relevant scale of study (particle vs. bulk vs. system)**
- **Bulk, or mesoscale – material properties**
  - **Engineering approach used effectively for unit operation and system design; correlation often not needed**
  - **Well established principles, still unanswered questions**
  - **Multi-variate material properties directly used for first principle process design or powder formulation, and scaling**
  - **Concepts of scale-up (or down), with scale variation of material properties often lost**
  - **Over emphasis of particle level modeling, at the expense of material property level modeling more effective for design and problem resolution**
- **Particle – controls bulk material properties**
  - **Best practice: Multivariate correlation of bulk material properties, in turn used for "powder" design (as opposed to machine/process design)**

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Support slides

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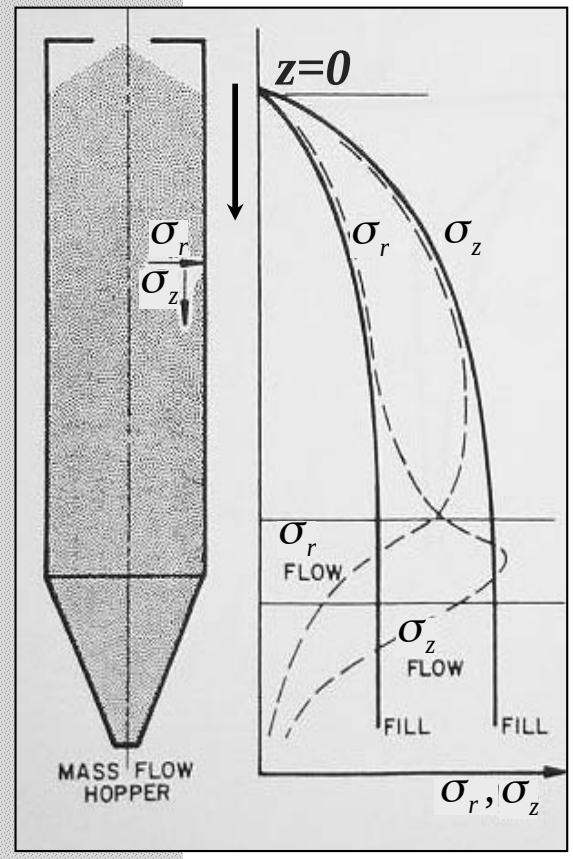
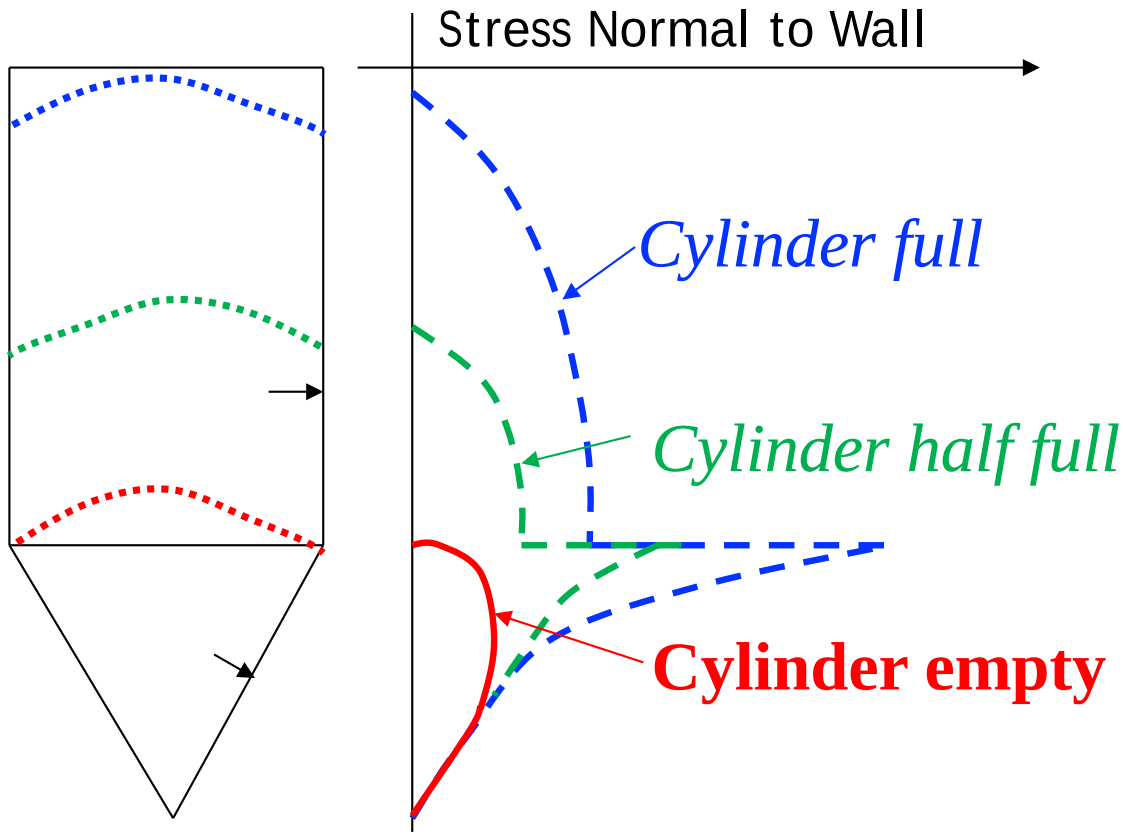
Stresses in hopper (Jenike assumption) - converging section





- In all three cases pressure curve in lower part of hopper is <u>predictable</u> and <u>decreases</u> towards outlet
- Pressure profile in lower part of mass flow hopper is independent of cylinder height (material head).

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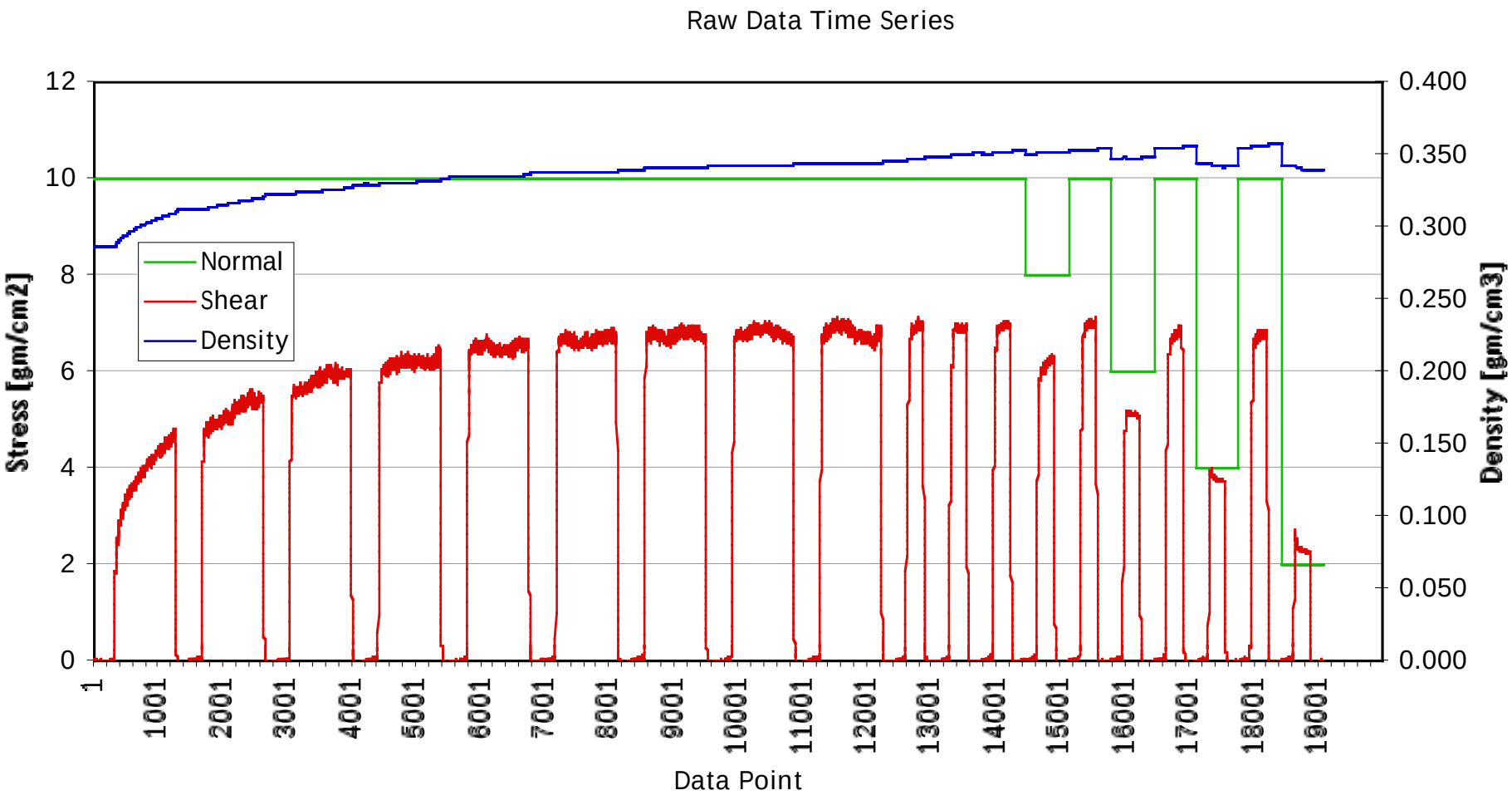
Time series of shear



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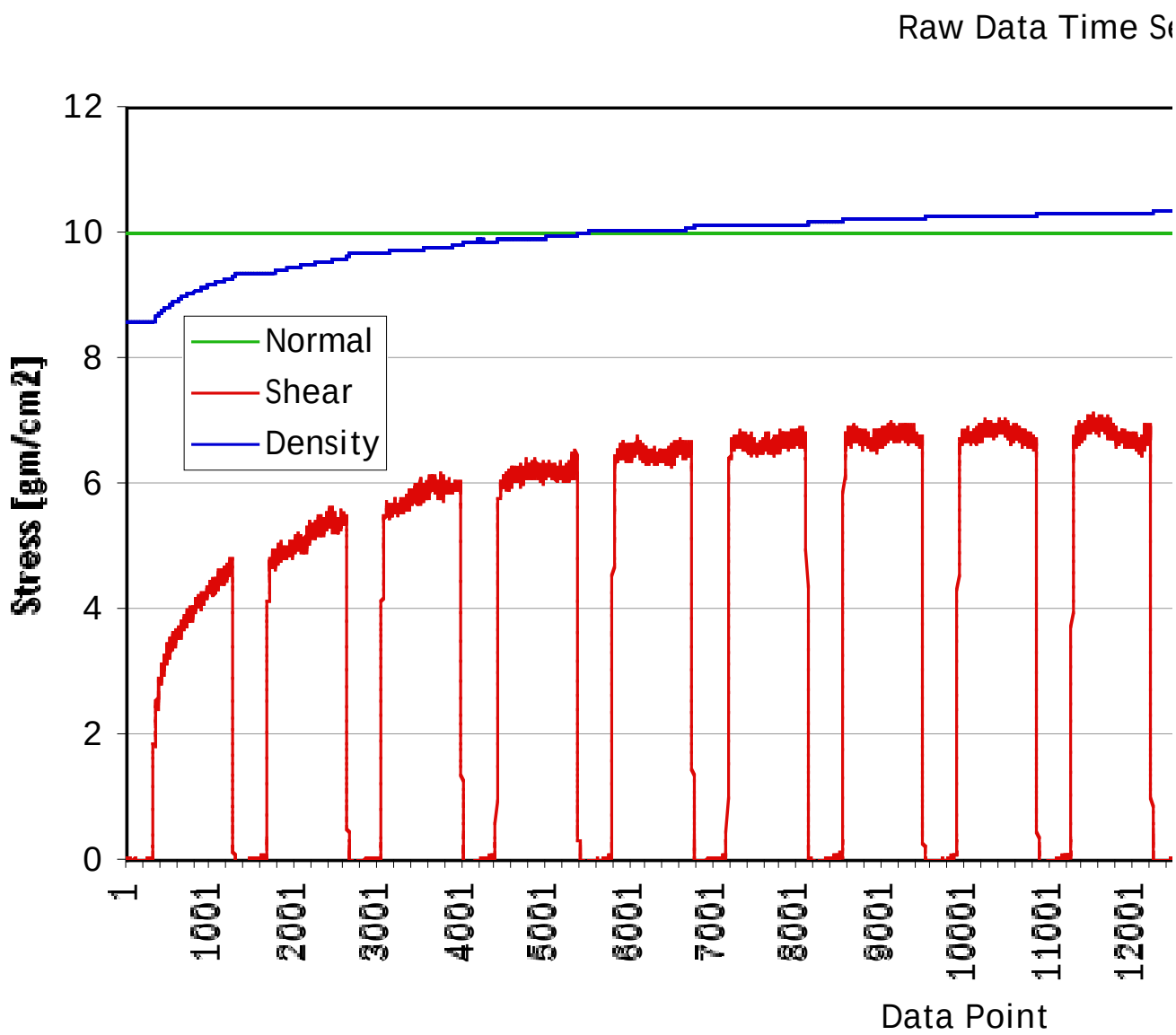
Time series of shear (Preconsolidation)



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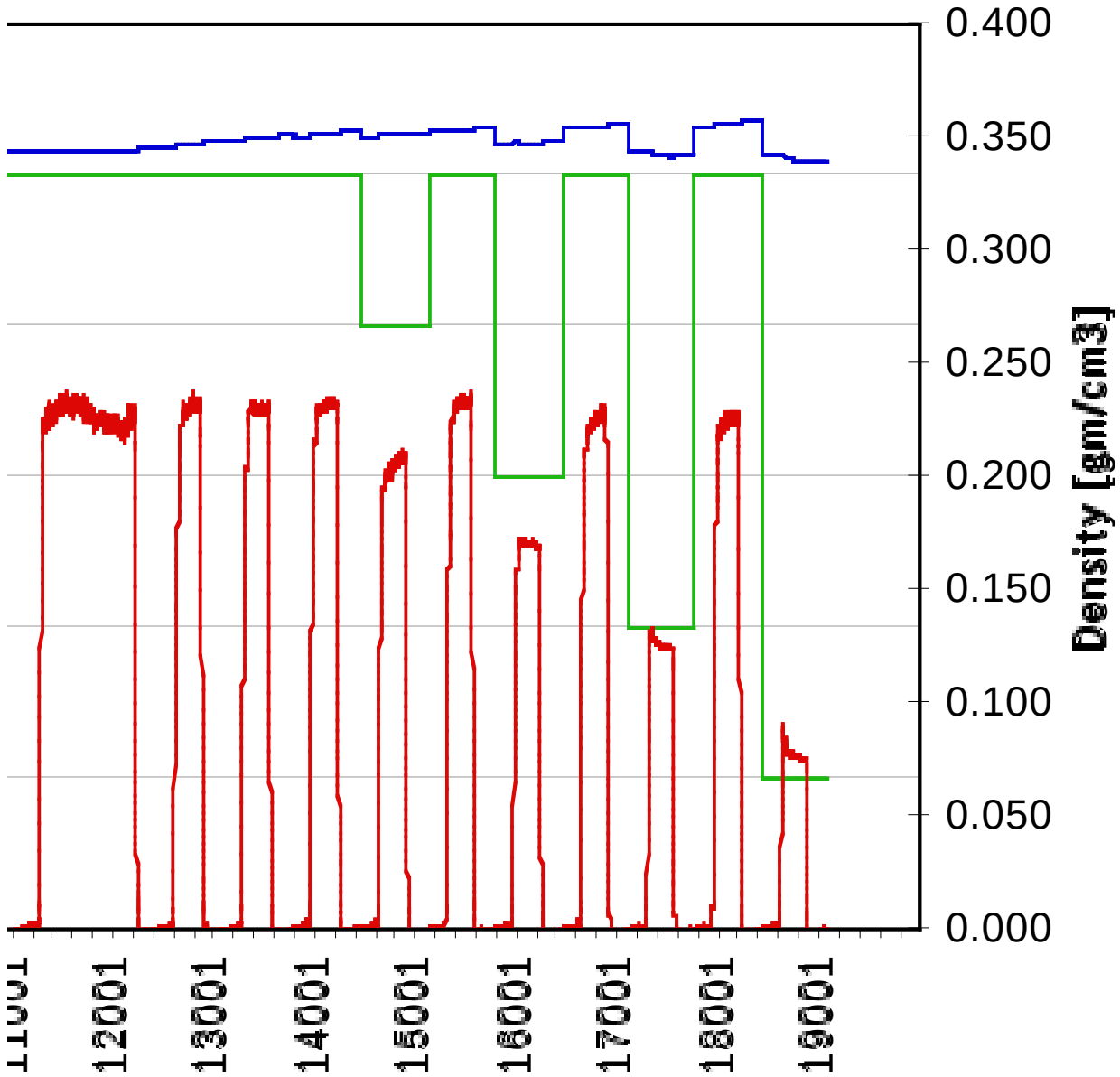
Time series of shear
(Shear steps for Yield Locus)



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Cohesive strength: A family of yield loci

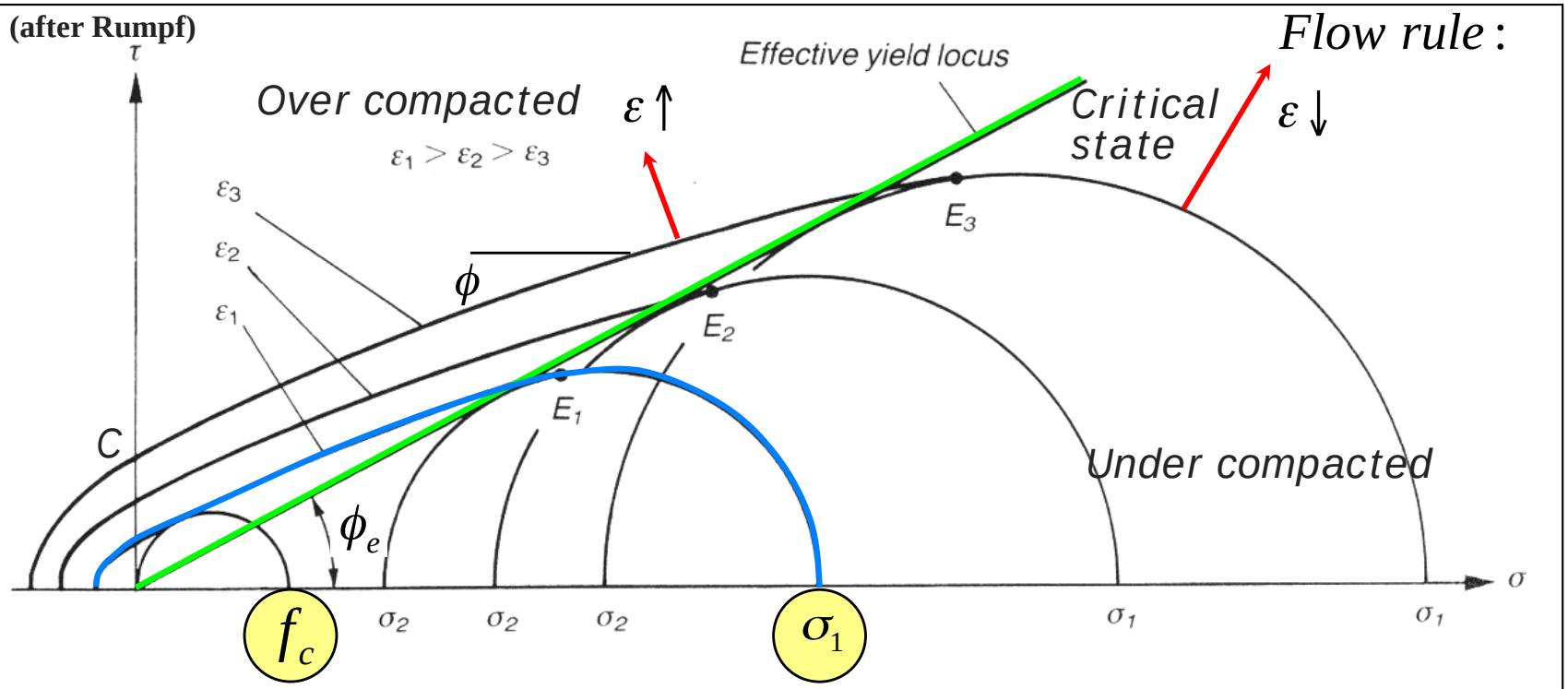

Figure 3.23 Family of yield loci with effective yield locus.

- **Multiple values of strength, as a function of major consolidation stress (history) and current stress state.**
- **Mat prop: Uniaxial cohesive strength, effective angle of friction, internal angle of friction/powder cohesion, wall friction and powder adhesion (not shown), static vs. dynamic yield loci, (and flow rules).**
- **"Drained loci" that neglect interstitial pore pressure.**

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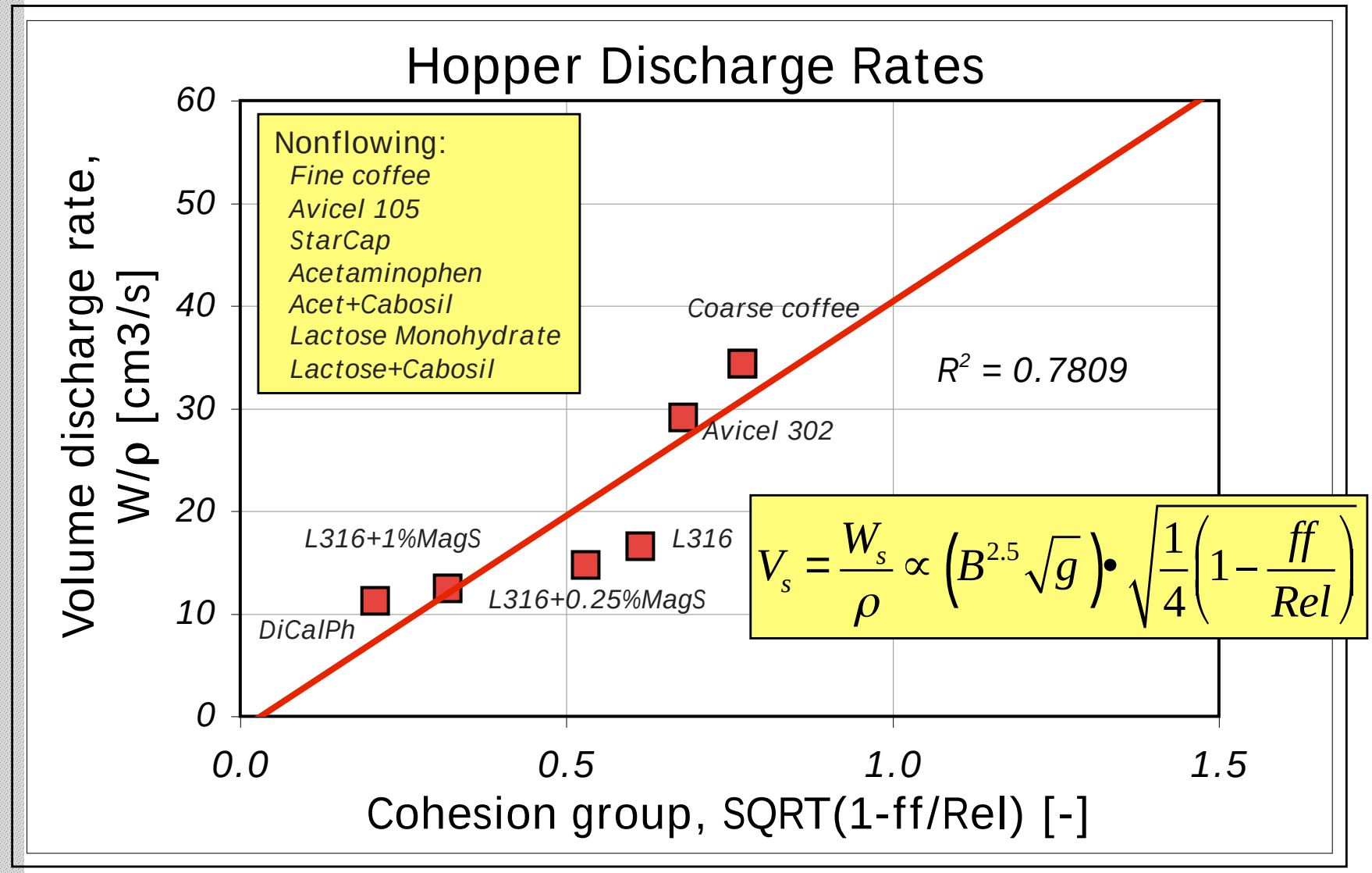
Volume discharge rates from hoppers compared to flow index



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