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CHARACTERISATION OF FLOWABILITY OF COHESIVE POWDERS BY INDENTATION

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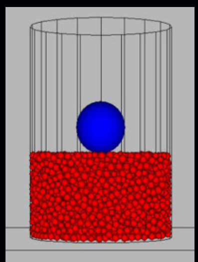


16th December 2009

Powders exhibit extreme flow behaviour

(Cleaver, 2007)

Bulk Powder Flow

Interparticle attraction forces, such as van der Waals, electrostatics and liquid bridges are responsible.

A number of techniques are available for quantifying bulk cohesion of powders:

- Shear cells
- Uniaxial compression test
- Sevilla powder tester
- Raining beds

Also a number of devices operating under less well-defined conditions:

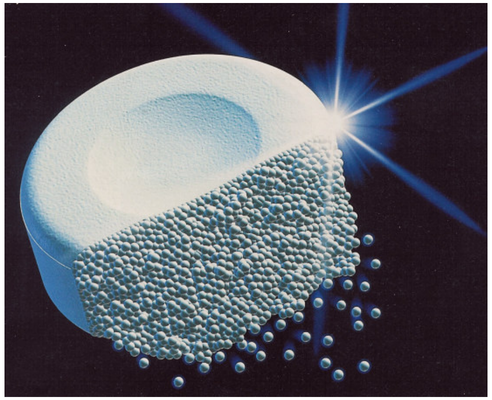
- Funnel tester
- Repose angle measurement
- Freeman powder rheometer
- Stable Micro System powder flow analyser

Technological Needs:

1. In a number of applications, there is insufficient material for testing or material is not easily accessible.

2. There is a need to test powder flowability at very low levels of stresses.

Pharmaceutical powders



Filling of small quantities of powders in capsules or dispersion in dry powder inhalers

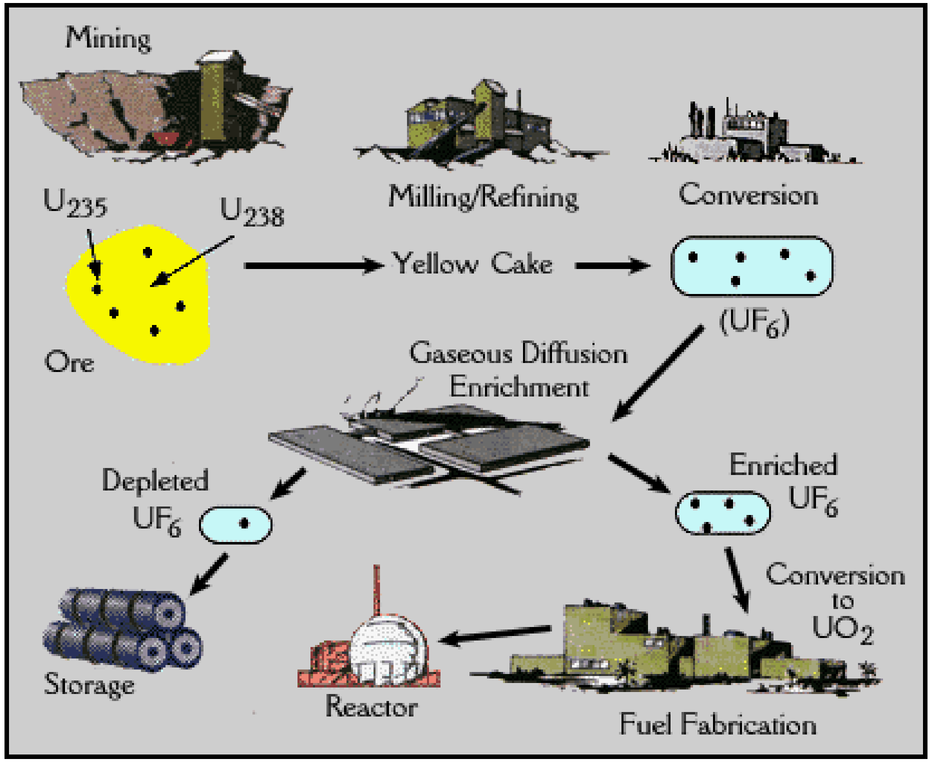
↓

Analysis of the flowability of loosely compacted cohesive powders at low stress levels

↓

Testing on small quantities of powders

Powders in nuclear industry



Ability to successfully predict the flowability of such powders remains a primary research interest

Requirements:

Testing on small quantities of powders at low loads

Why not

Indenting on a powder bed?

Objectives

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- Characterise bulk powders, compacted at low levels of pressure, by indentation and compare with unconfined compression and shear cell techniques

- Analyse the deformation process around the indent based on single particle properties using DEM.

Samples:

α-Lactose monohydrate, Avicel, starch powders, magnesium carbonate, glass beads made cohesive by silanisation

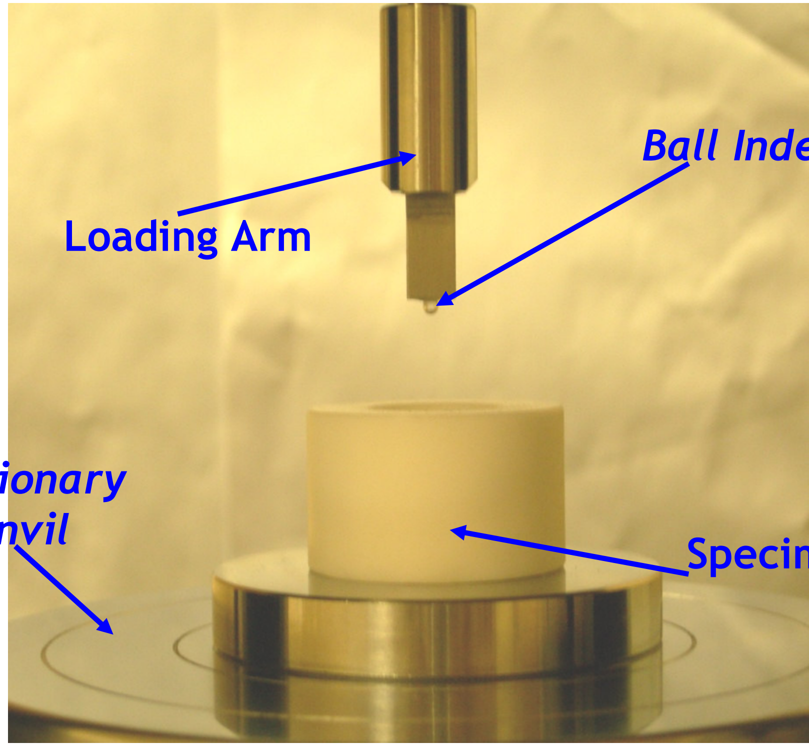
Indentation Method

This is being developed at Leeds to infer the plastic yield stress of powder beds and hence bulk cohesion.

- **Very small scale, a few mm^3.**
- **Very low levels of stresses.**

What does the indentation test give?

Penetration vs load from which the hardness is calculated



Indentation process

Hardness Calculation

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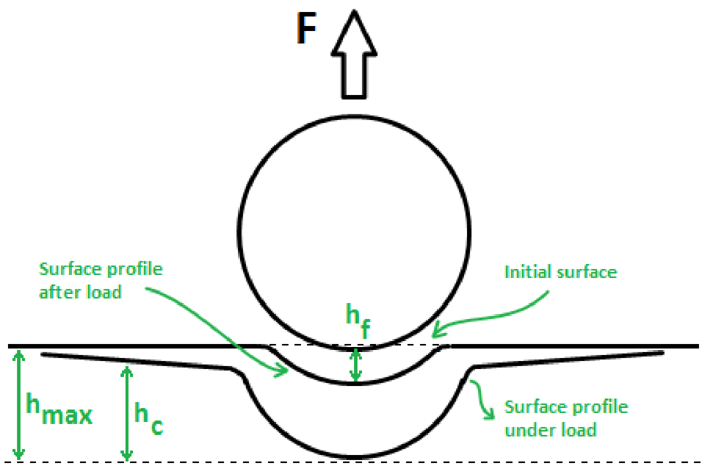

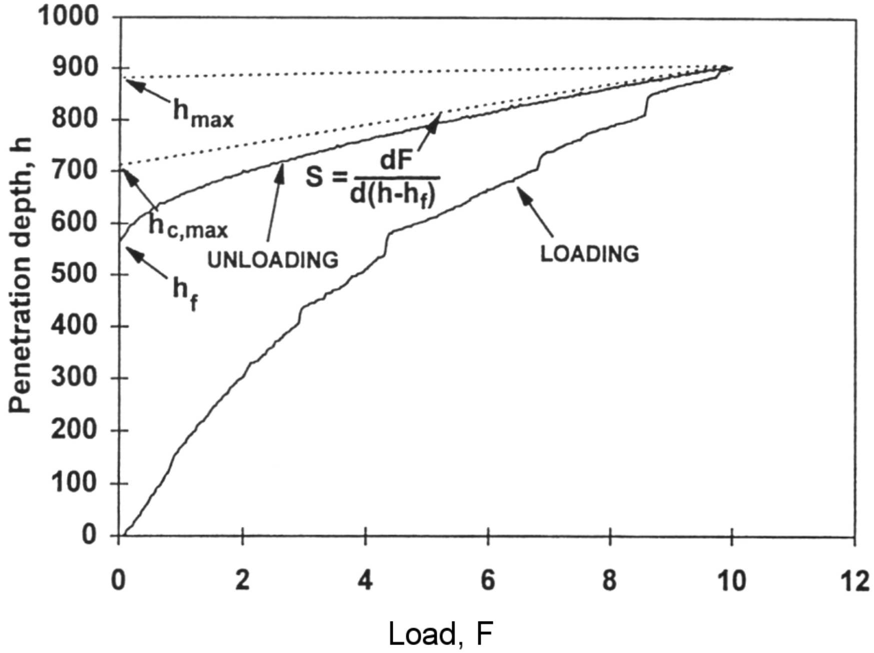

$$H = \frac{F_{max}}{A}$$

For ball indenters: $A = \pi(d_b h_{c,max} - h^2_{c,max})$

If $h_{c,max} << d_b$ $\longrightarrow$ $A = \pi d_b h_{c,max}$

Incipient yield loci and consolidation surfaces (Nedderman's book)

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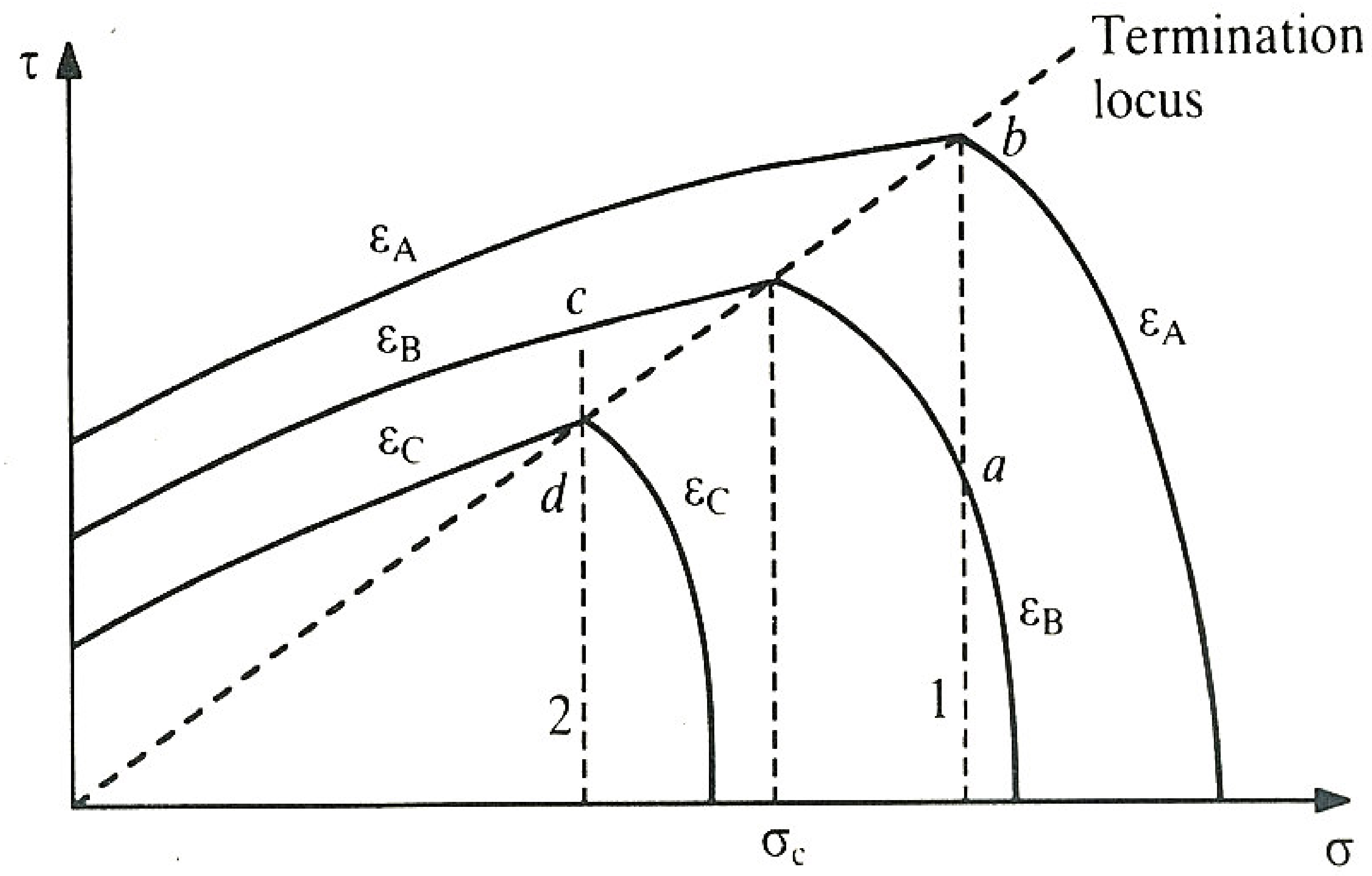

Indentation on a-lactose powder bed

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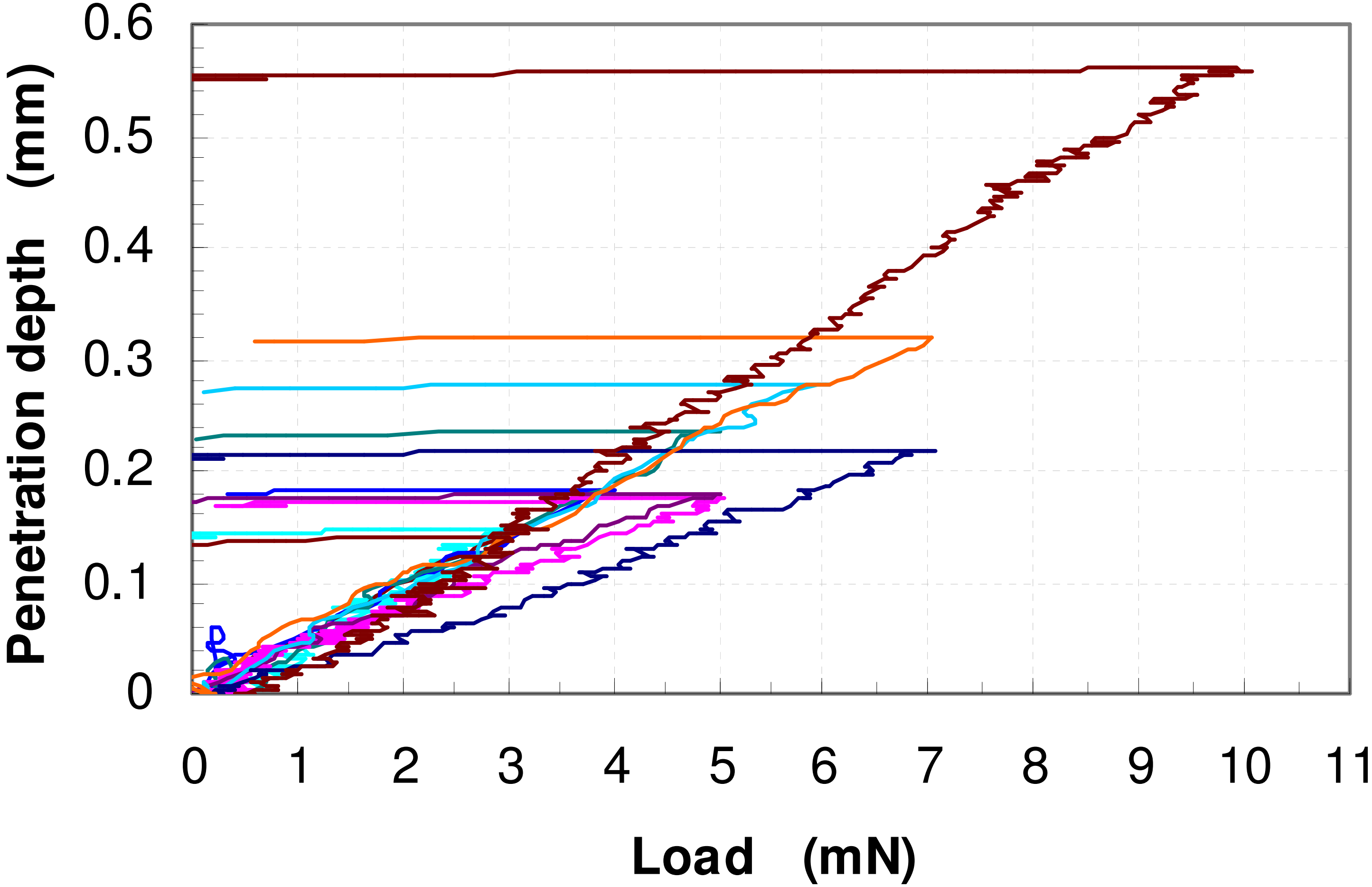
Typical indentation cycles (tablets pre-consolidated to 3 kPa)
Each tablet has been indented with a high precision spherical glass indenter, 1.588 mm in diameter.



Effect of indentation load

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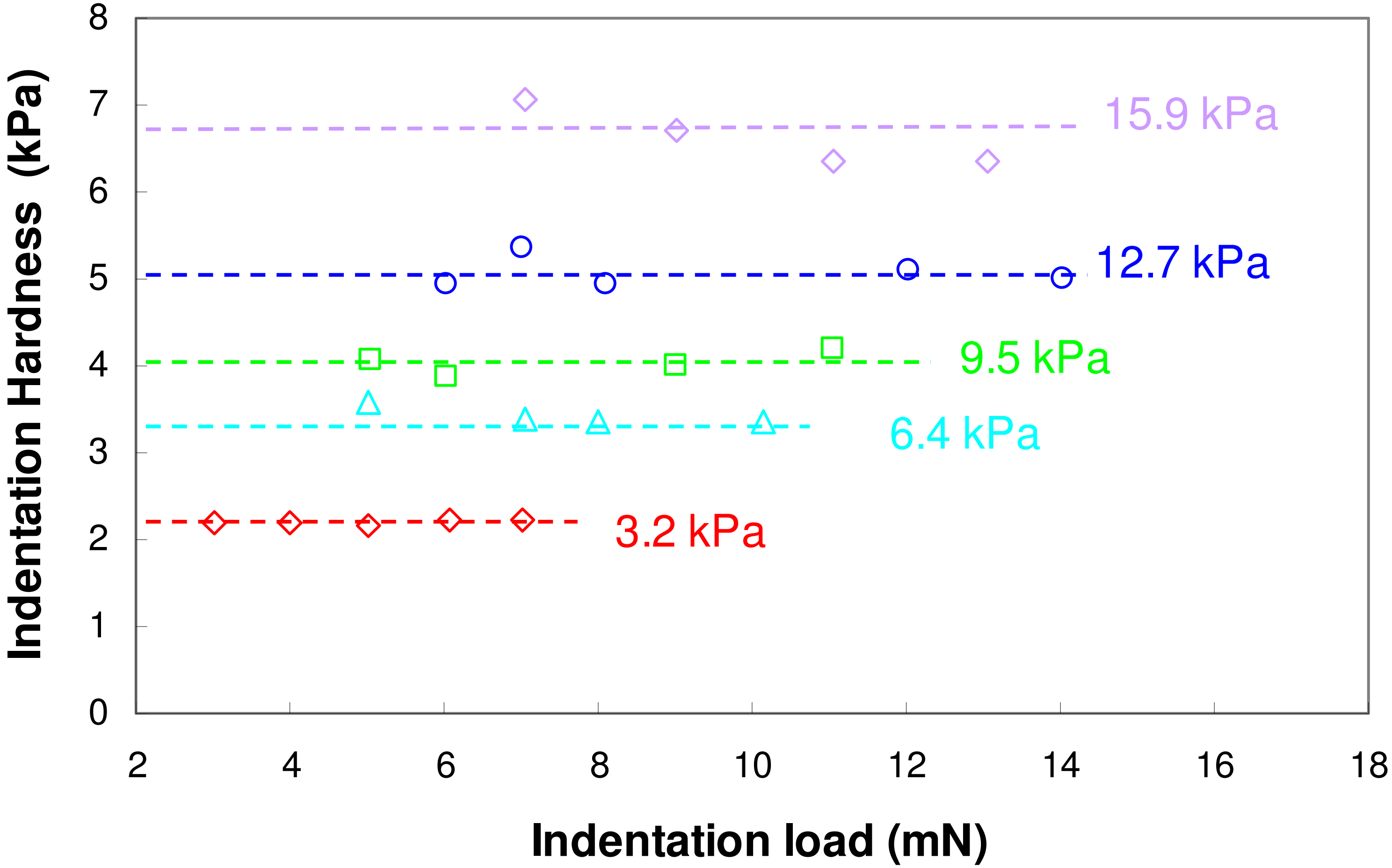
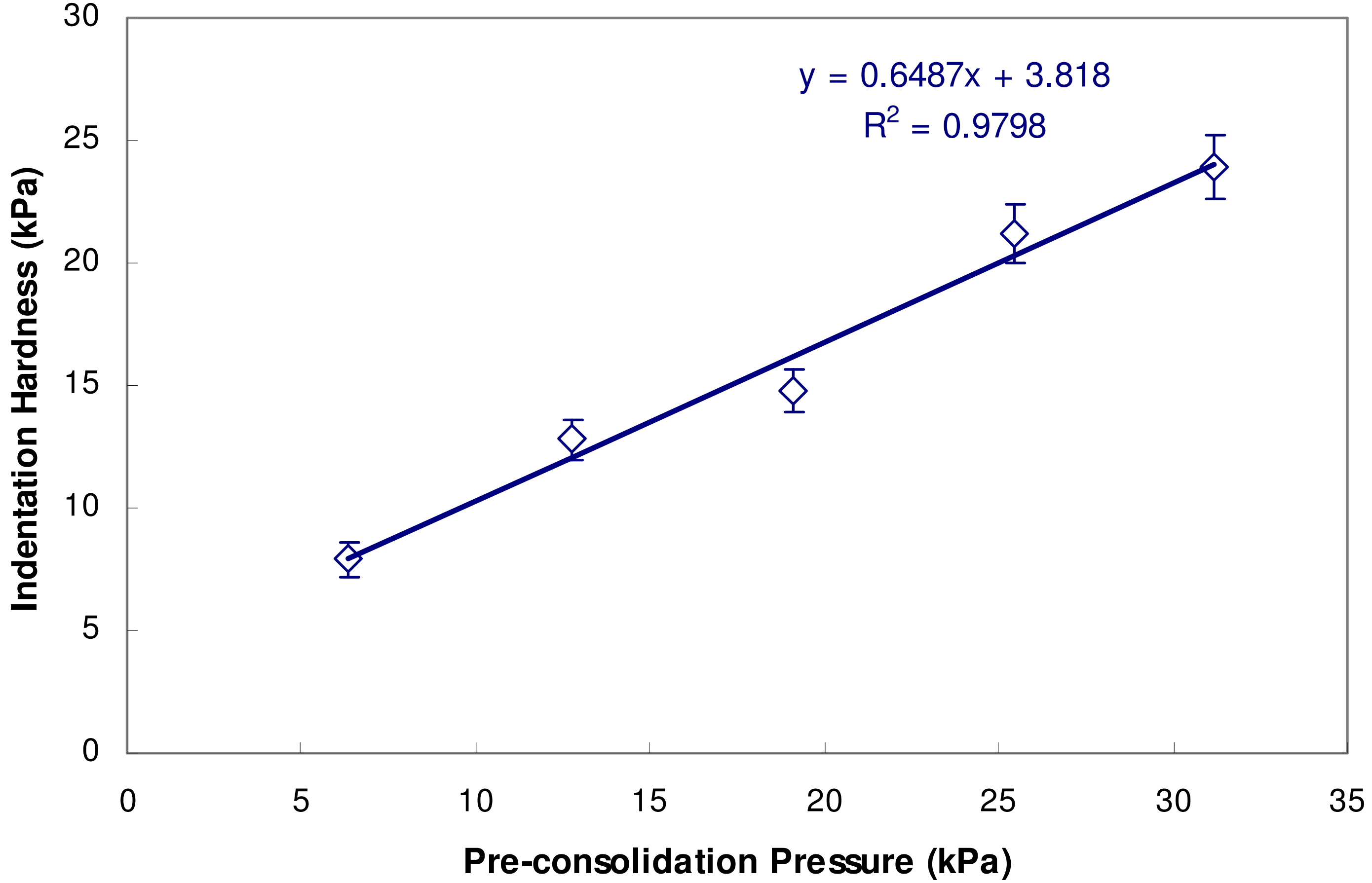
Hardness values of α-lactose pre-consolidated to five different levels

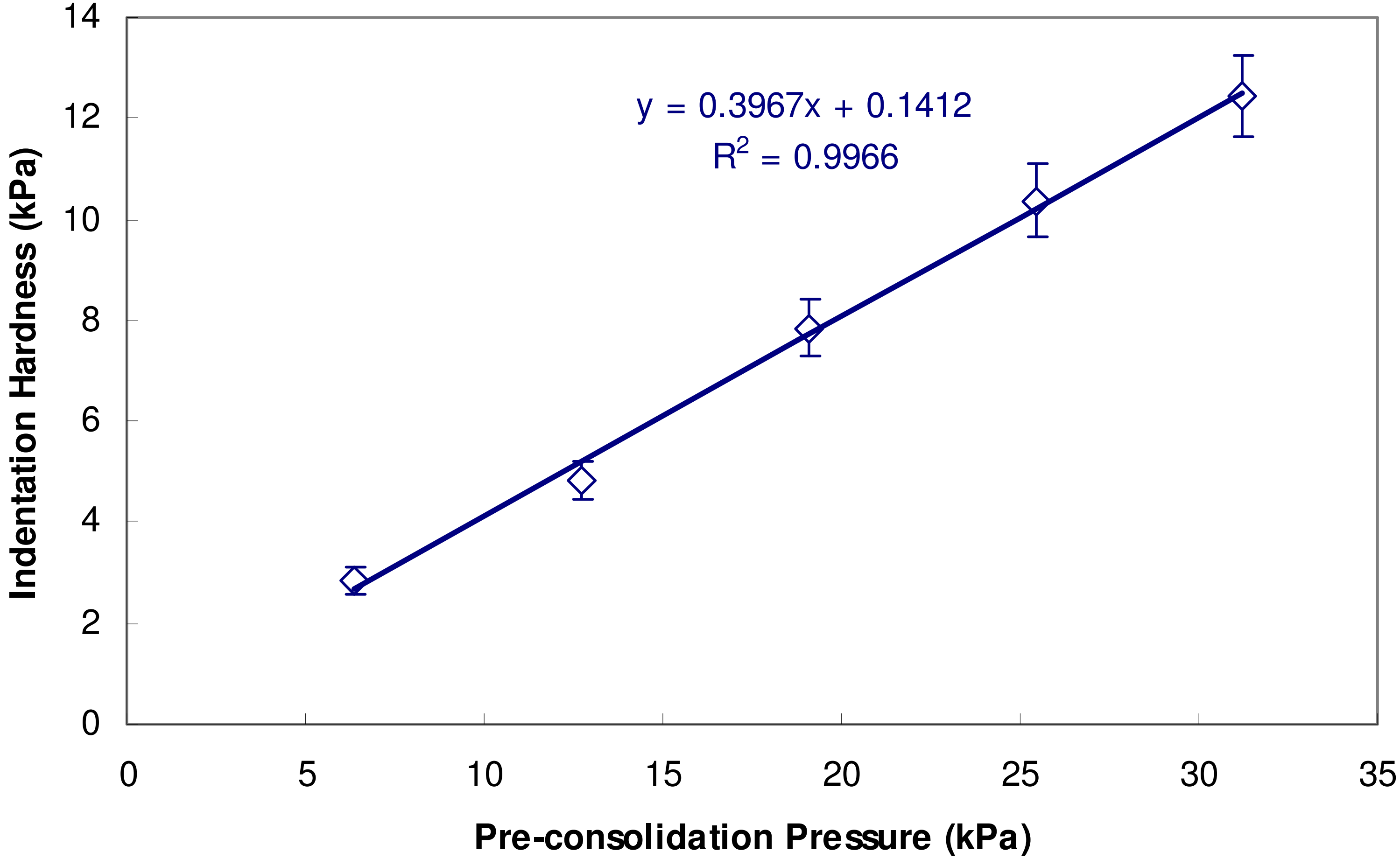


Hardness value is independent of indentation load within a certain range

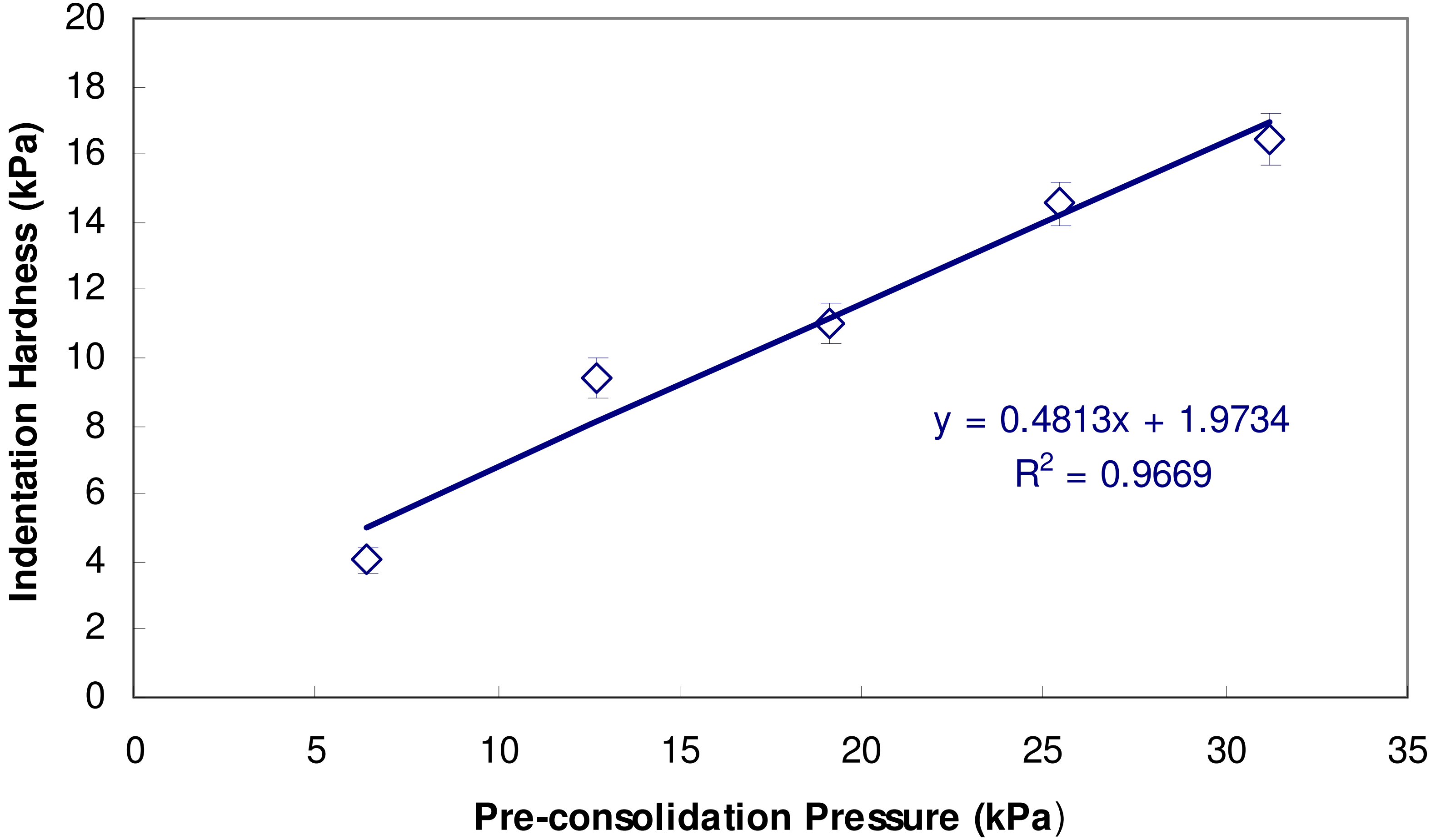
Relationship between indentation hardness and consolidation pressure for α-lactose



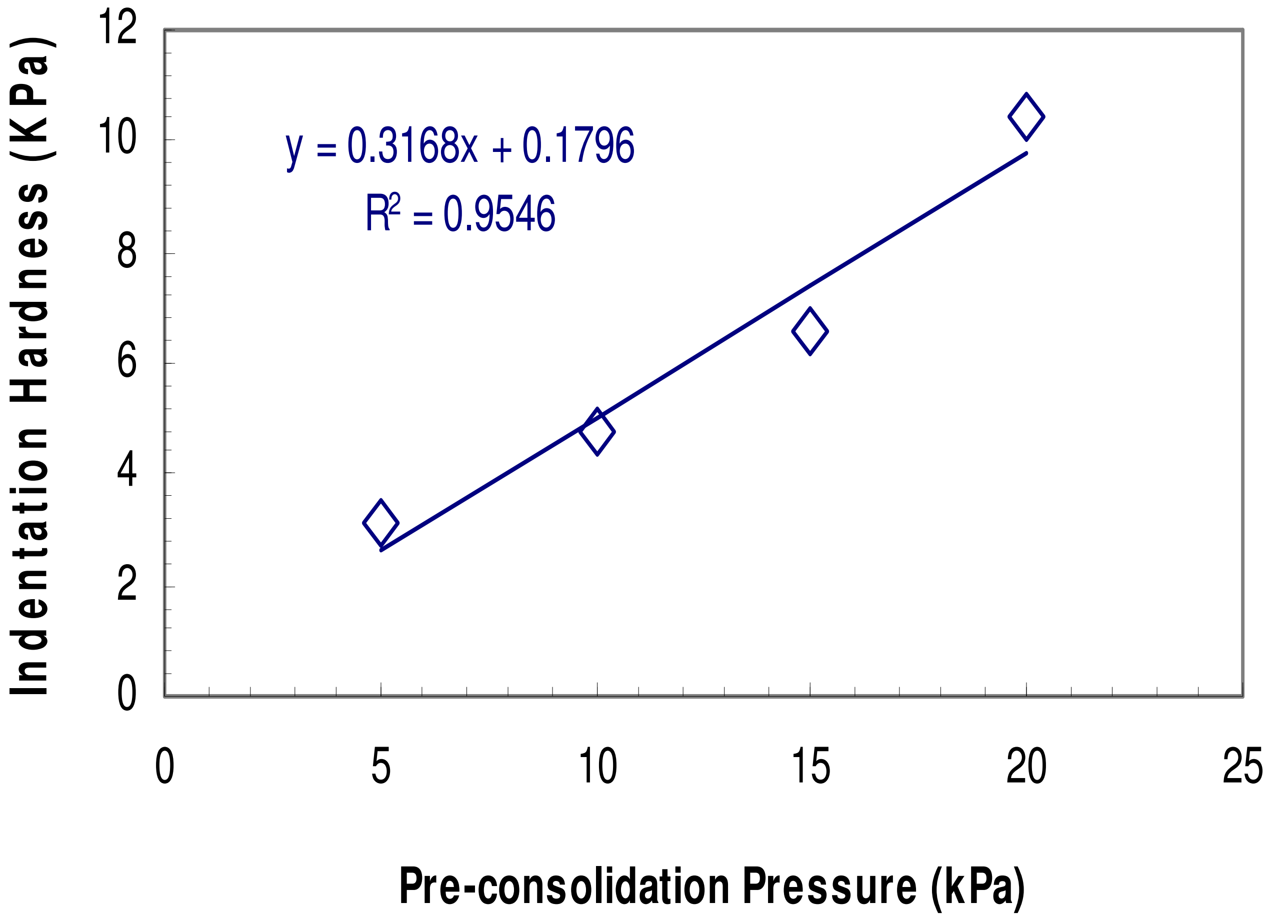
Relationship between indentation hardness and consolidation pressure for Avicel



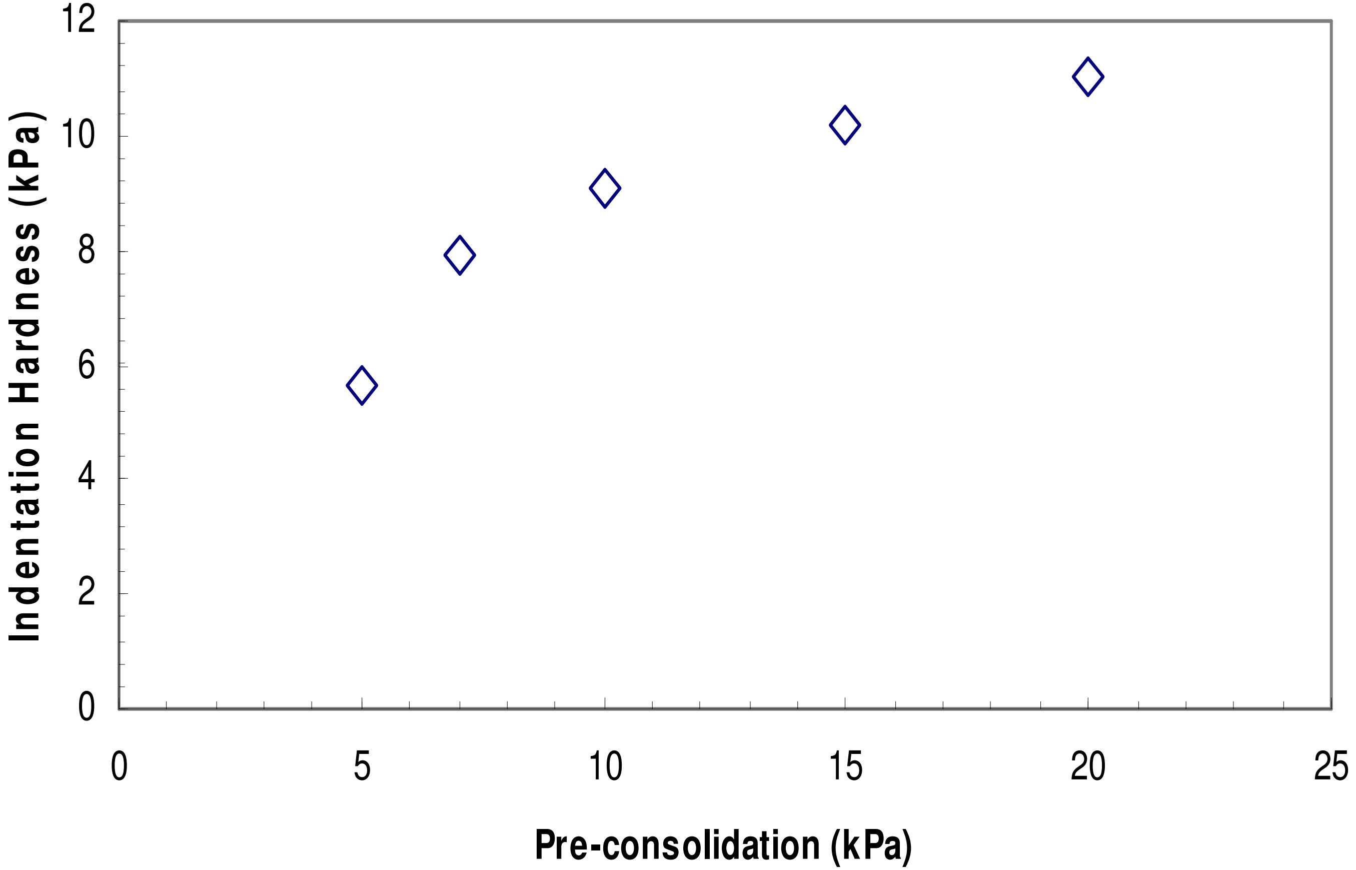
Relationship between indentation hardness and consolidation pressure for starch



Relationship between indentation hardness and consolidation pressure for magnesium carbonate



Relationship between indentation hardness and consolidation pressure for cohesive glass beads

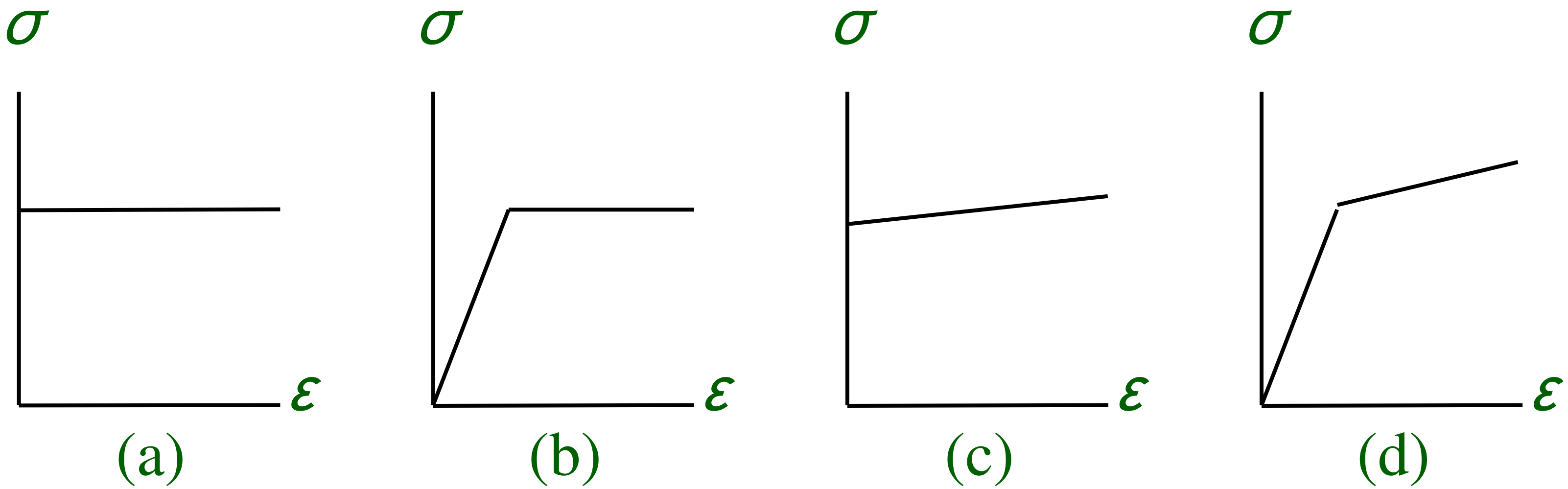


Determination of Yield Stress

- When an indentation is made the plastic deformation is confined, and therefore hardness is much larger than yield stress.
- The relationship: $H=CY$

  C: constraint factor. Affected by material properties (deformation mode), indenter geometry and friction. Relevant properties: elastic modulus, work-hardening, anisotropy.

Effect of Failure Mode



(a) rigid-perfectly plastic,

(b) elastic-perfectly plastic,

(c) rigid-plastic with work-hardening, and

(d) elastic-plastic with work-hardening.

Rigid-Perfectly Plastic Indentation

- Wedge-cutting mechanism for sharp indenters, <30°:

$$H/Y = 3$$

- Radial compression, > 30°: next slide

Elastic-Perfectly Plastic Indentation

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- **Deformation is treated as expansion of a spherical cavity (Hill, 1950):**

$$\frac{p}{Y} = \frac{2}{3}\left\{1 + Ln\left(\frac{E}{3(1-\nu)Y}\right)\right\}$$

Elastic-Perfectly Plastic Indentation

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- **For conical or pyramidal indenters with half-angle θ Johnson (1970) gives:**

$$\frac{H}{Y} = \frac{2}{3}\left(1 + Ln\frac{E\,cot\theta}{3Y} \right)$$

(not valid for sharp indenters)

Work-Hardening

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- $H/Y >> 3$
- Flow stress is strain dependent: $\sigma = Y + \Pi$
- Considering work-hardening rate: Π'

$$\Pi = \Pi' \times (\varepsilon - \varepsilon_o)$$

- Taking the expansion of spherical cavity approach, Bishop *et al*. (1945) give:

$$H = p = \frac{2}{3} Y \left\{ 1 + Ln\left(\frac{2E}{3Y}\right) \right\} + \frac{2\pi^2}{27} \Pi'$$

$$\frac{H}{Y} =$$

Constraint factor	Material	Properties
$f\left(\frac{E}{Y}\right), \quad \phi < 3$	glasses and polymers	low $\frac{E}{Y}$, $\Pi' \cong 0$
$\phi \cong 3$	a wide range of metals	high $\frac{E}{Y}$, low $\frac{\Pi'}{Y}$
$f\left(\frac{E}{Y}, \frac{\Pi'}{Y}\right), \quad \phi > 3$	ionic single crystals	high $\frac{E}{Y}$, high $\frac{\Pi'}{Y}$

Relationship between yield stress and pre-consolidation pressure for α-lactose

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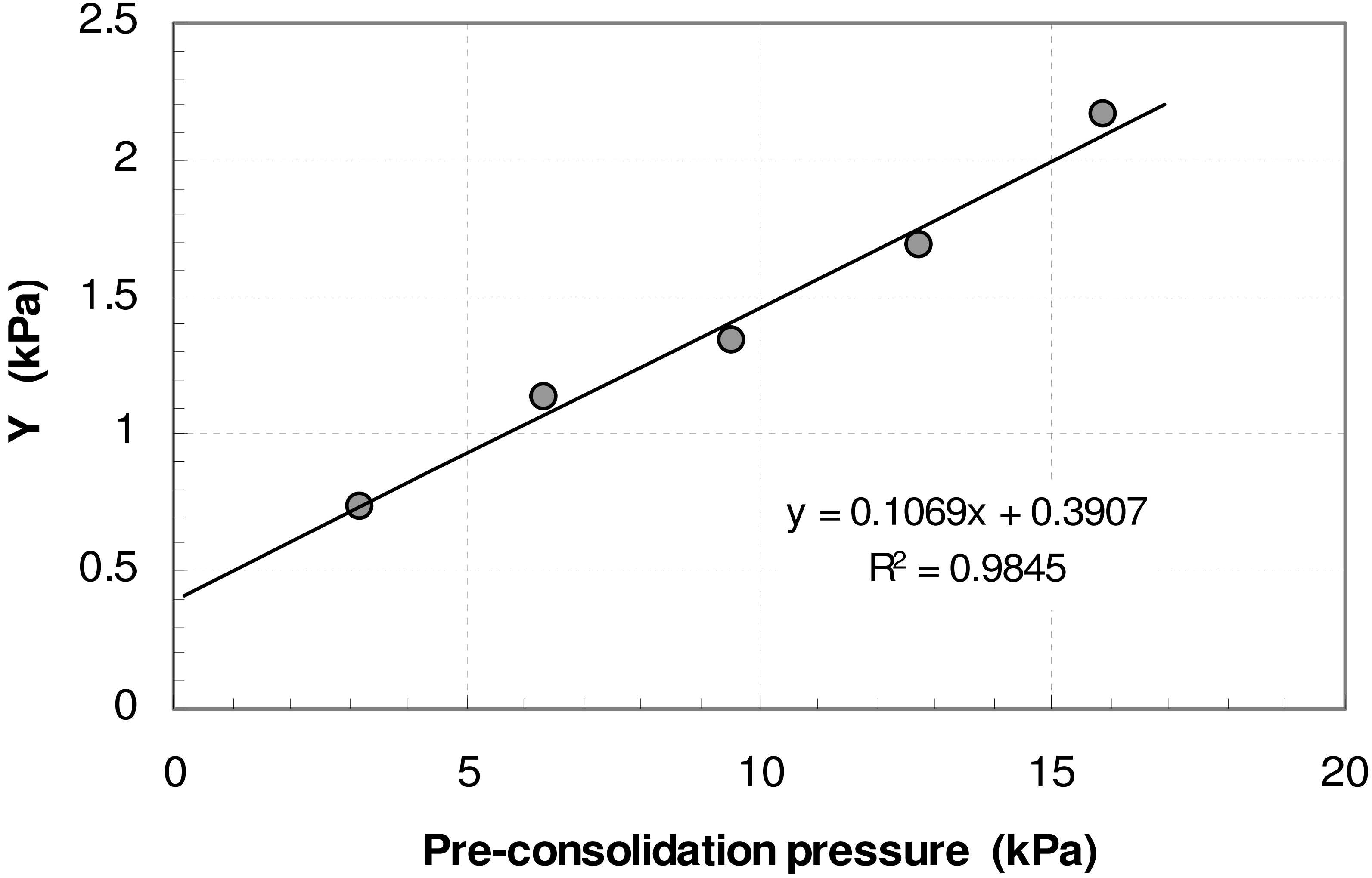

Comparison of Trend

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Experimental Approach

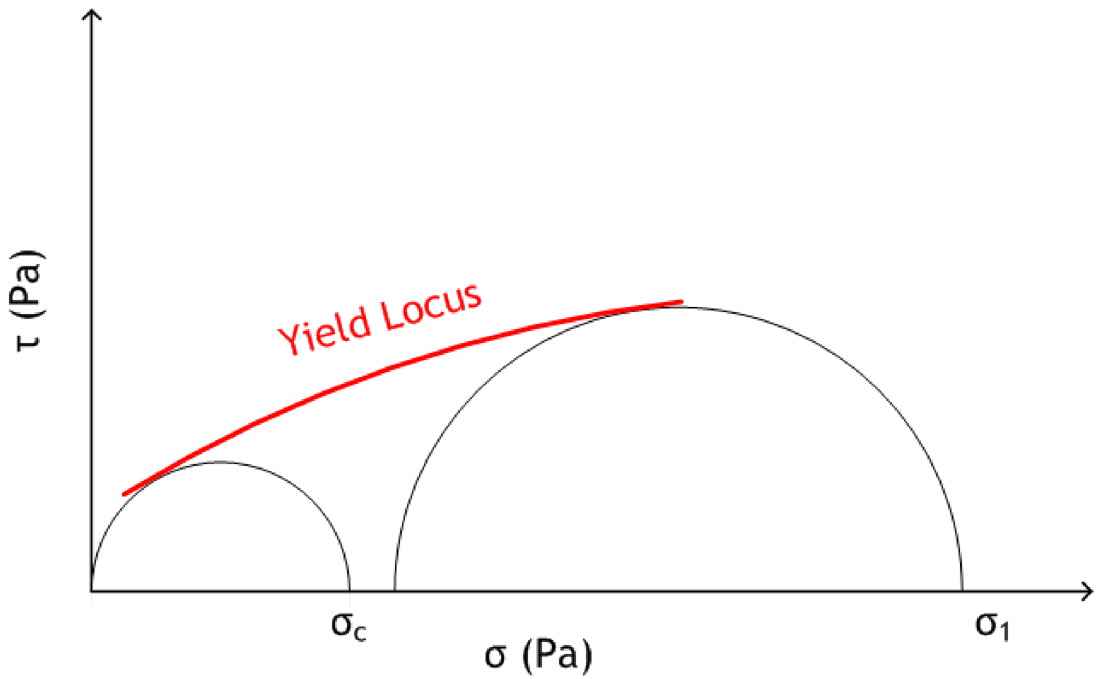
Schulze ring shear tester - used to evaluate unconfined yield strength and relate to bulk powder flowability.

Flow Function → $$ff_c = \frac{\sigma_1}{\sigma_c}$$

σ_1 ← Major Principal Stress

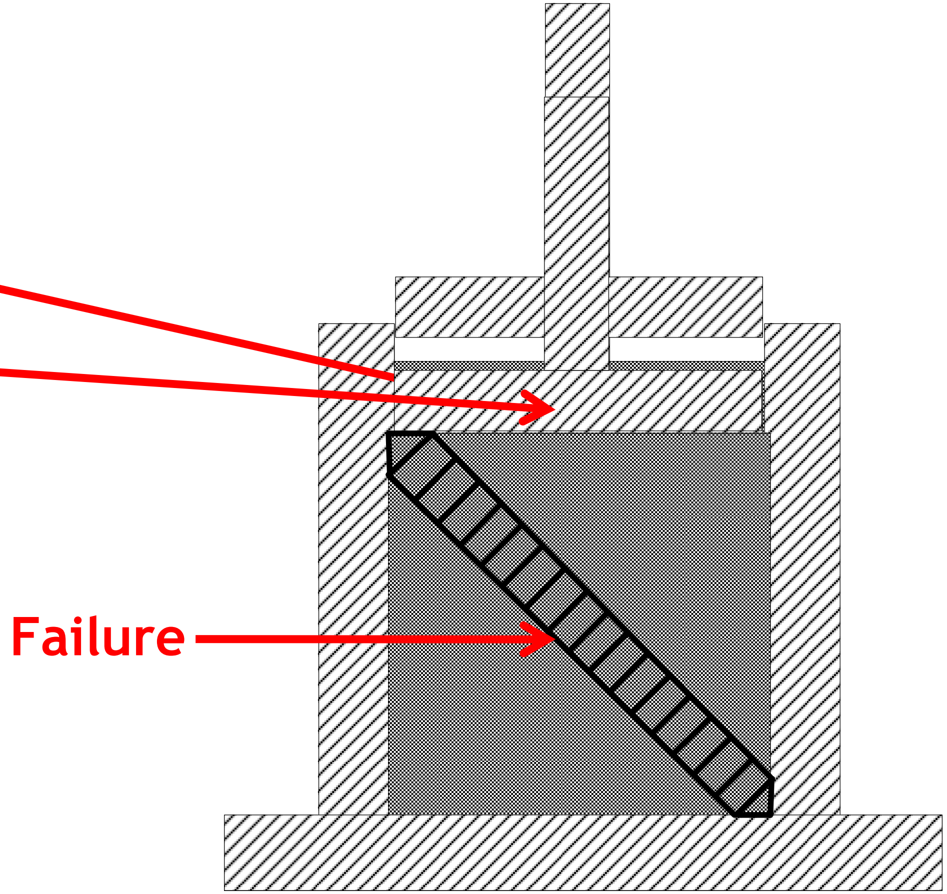
σ_c ← Unconfined Yield Strength

Schulze Ring Shear Tester



Uniaxial Compression

$$ff_c = \frac{\sigma_1}{\sigma_c}$$



Comparison of yield stress of α-lactose determined from shear cell, unconfined compression and indentation

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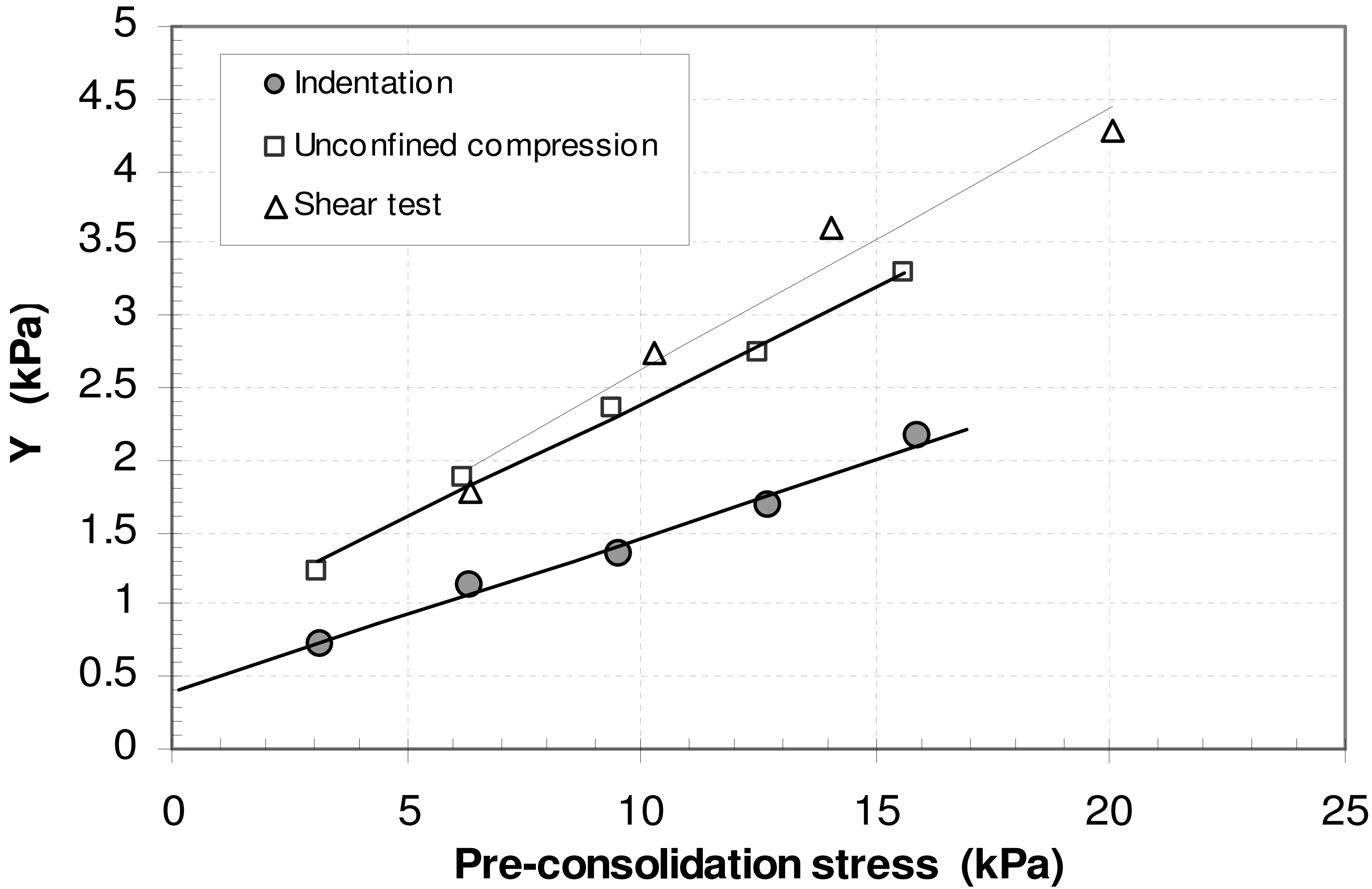
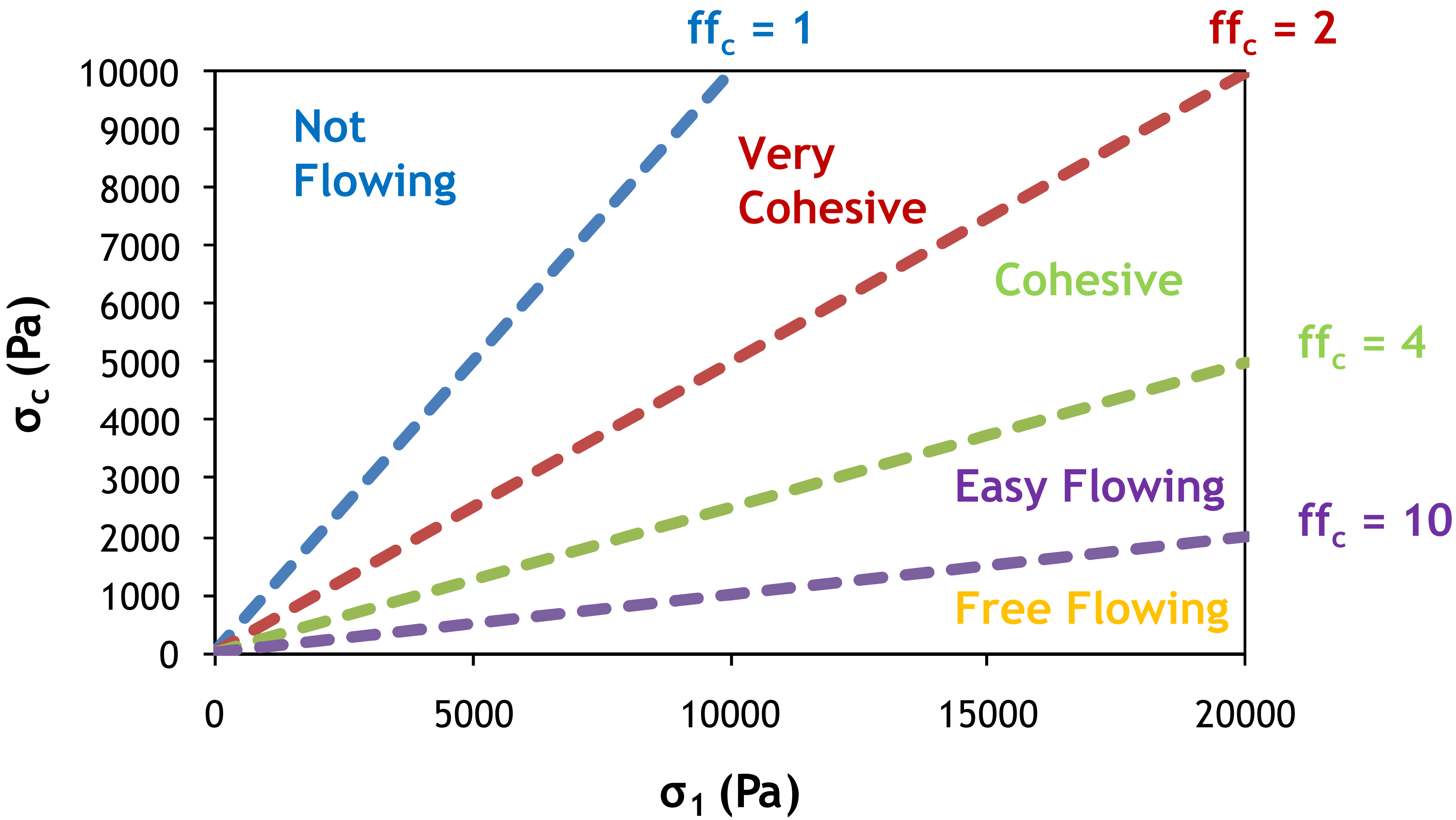


Flowability Guidelines



What do we expect for the value of the constraint factor?

Approaches:

1. Continuum Mechanics
2. Distinct Element Method

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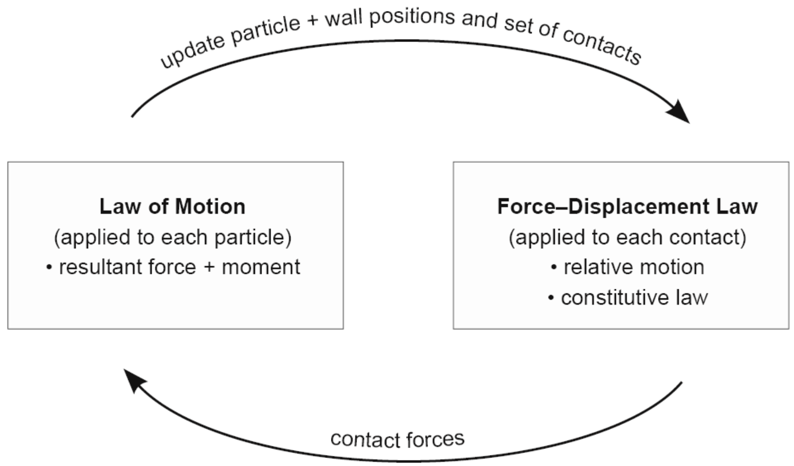
Distinct Element Method

DEM solves Newton's second law of motion and a relative force-displacement law for a set of discrete particles. This tool can provide insightful data otherwise unattainable.

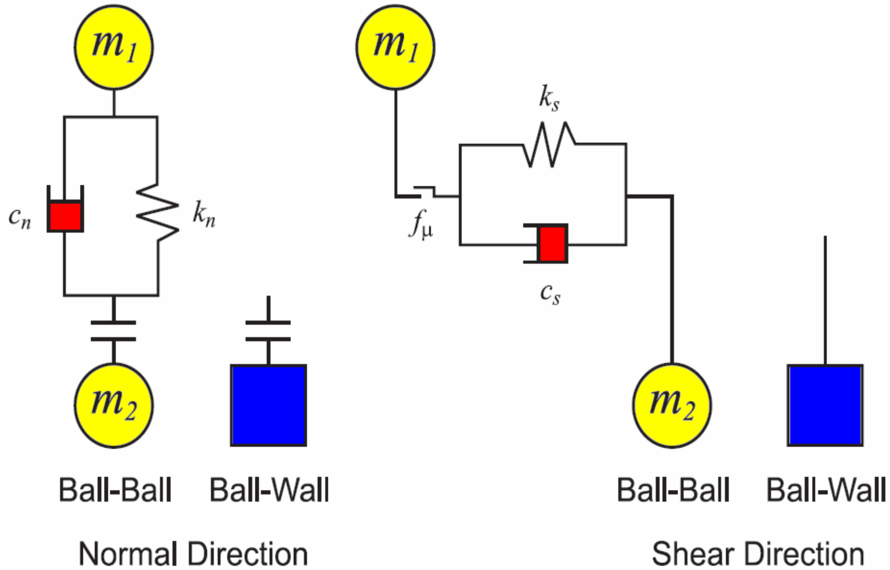


Laws of Motion:

$$ma = \sum F$$

$$I\alpha = \sum M$$

m: Mass of Particle
a: Linear Acceleration
F: Force
I: Moment of Inertia
α: Angular Acceleration
M: Momentum of Tangential force



DEM Principles

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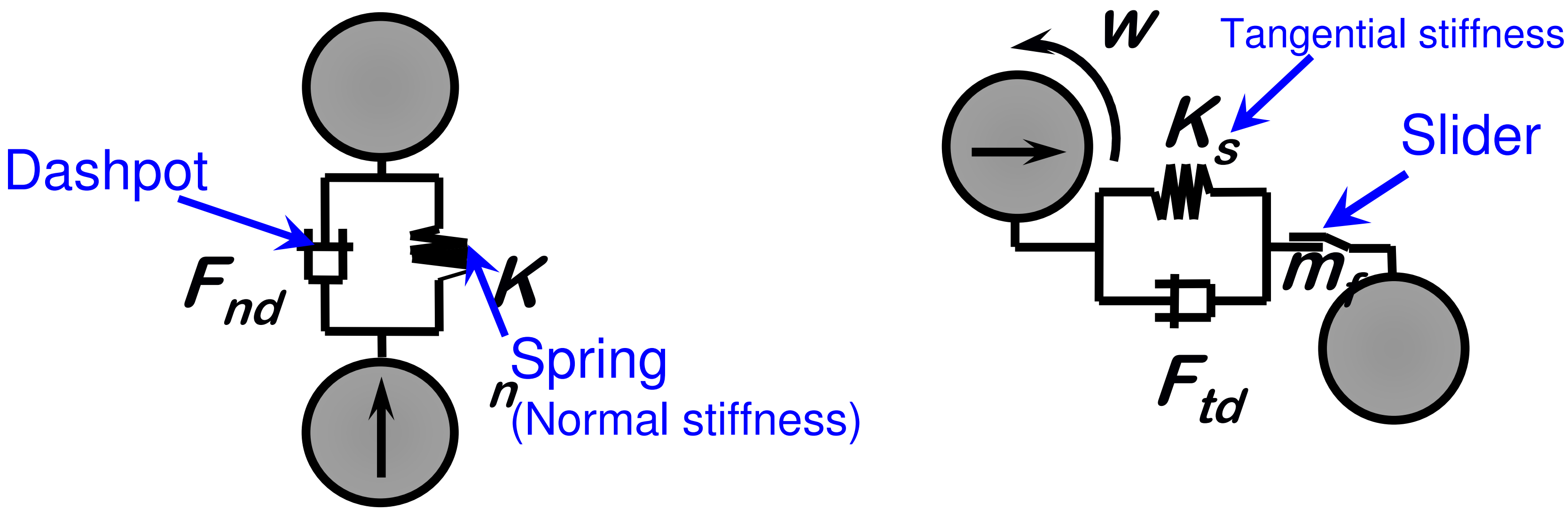
- In a DEM simulation, each particle possesses translational and rotational motion. The motion of each particle can be described by solving Newton's equation of motion according to the external forces applying to the spheres.



Spring-Dashpot Model

Particle Properties

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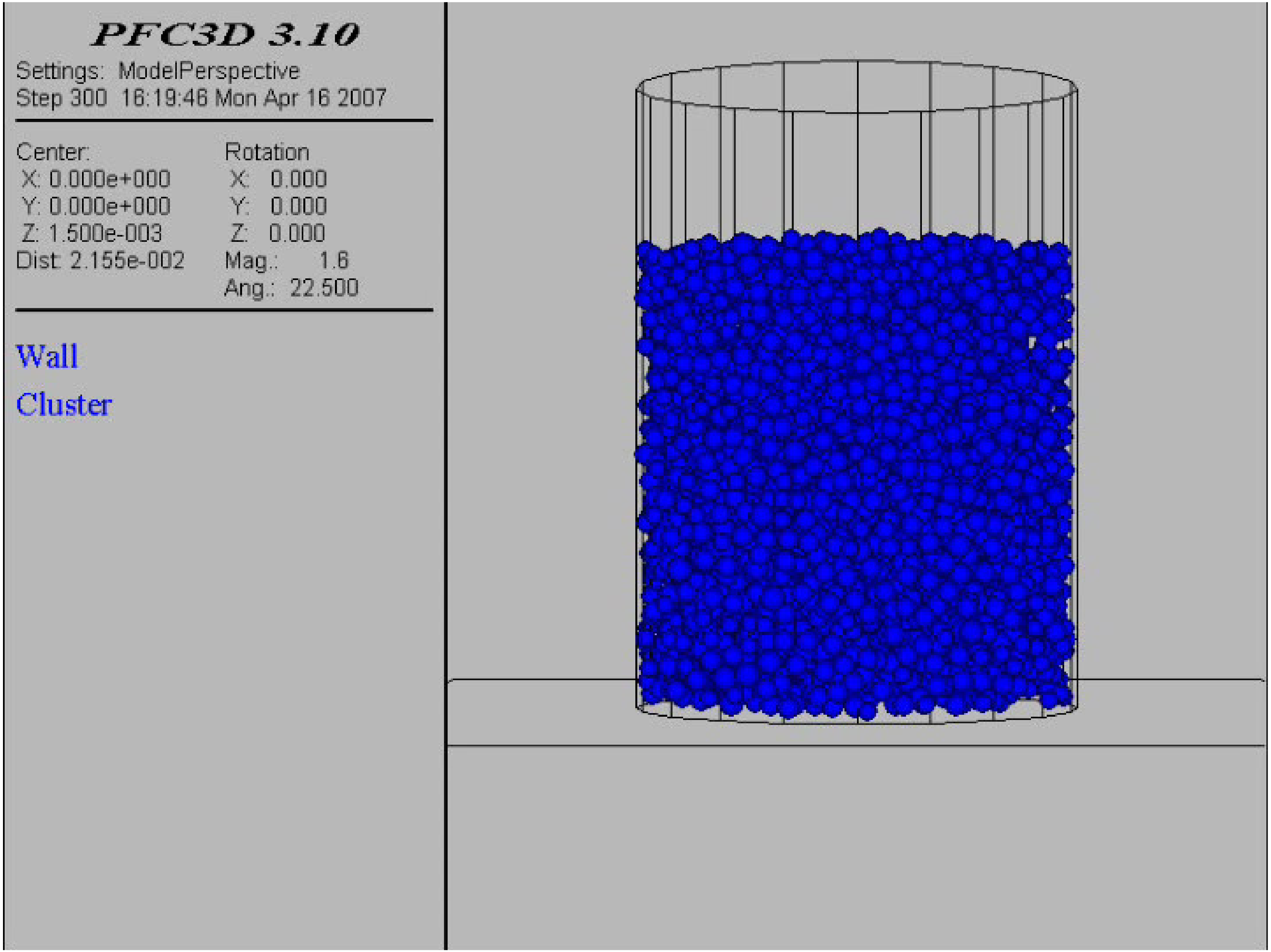
Number of particles: 4800
Average radius of the particles: 70 μm
Diameter of the die: 2.88 mm
Surface energy: 0.2 J m^{-2}

Single Particle Properties	Particle (Cohesive Glass bead)	Cylinder (PTFE wall)	Loading Piston (Steel)
Normal Stiffness (N/m)	69600	2640	44000
Shear Stiffness (N/m)	69600	2640	44000
Friction Coefficient (-)	0.16	0.15	0.3

JKR Theory: $F_{pull-off} = 3\pi\gamma R = 4.1e^{-5}$ N



PFC3D 3.10
Settings: ModelPerspective
Step 300 16:19:46 Mon Apr 16 2007
Center:
X: 0.000e+000
Y: 0.000e+000
Z: 1.500e-003
Dist: 2.155e-002
Rotation
X: 0.000
Y: 0.000
Z: 0.000
Mag.: 1.6
Ang.: 22.500
Wall
Cluster

Pre-consolidation Process

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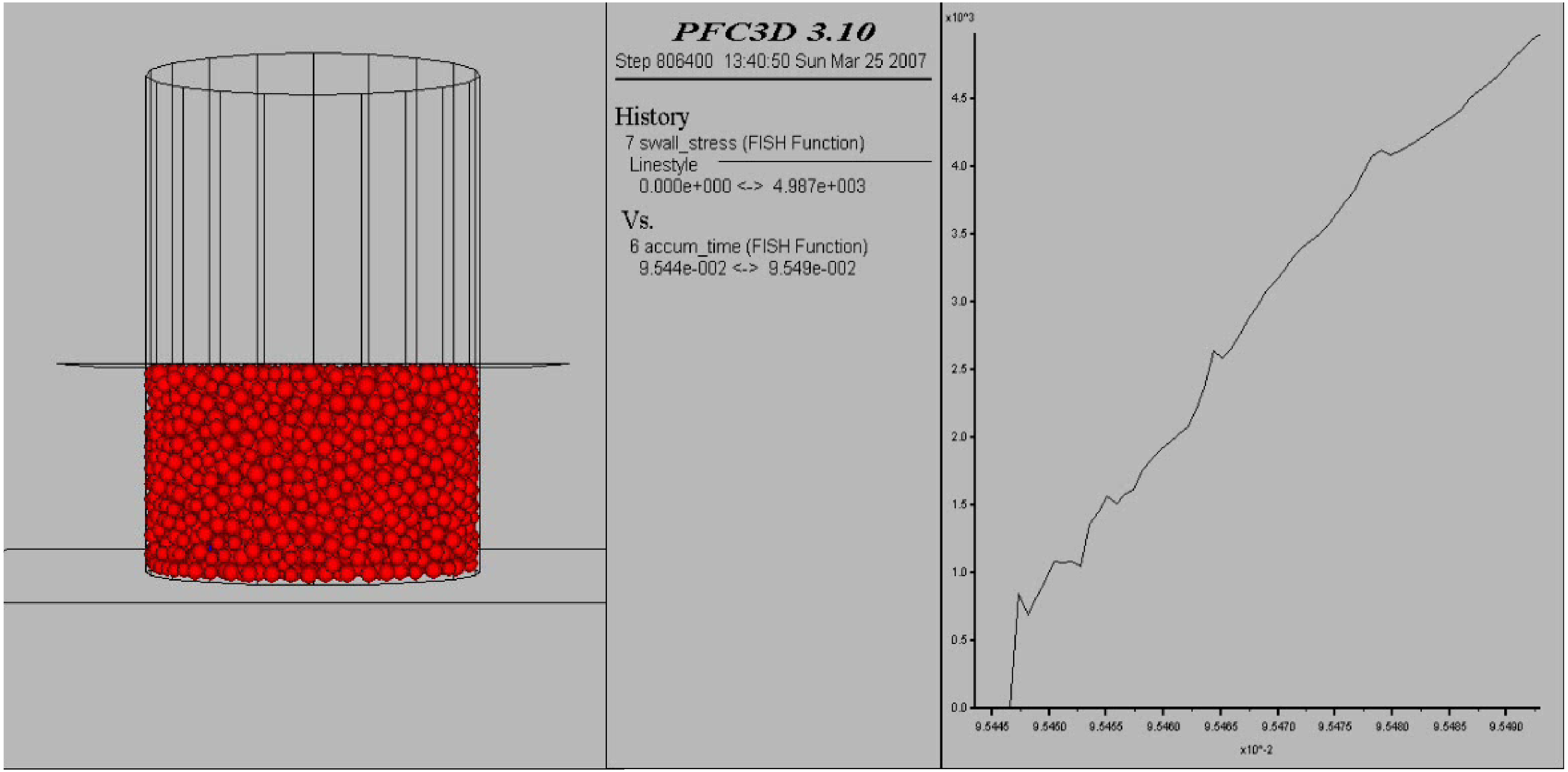

Indentation Process

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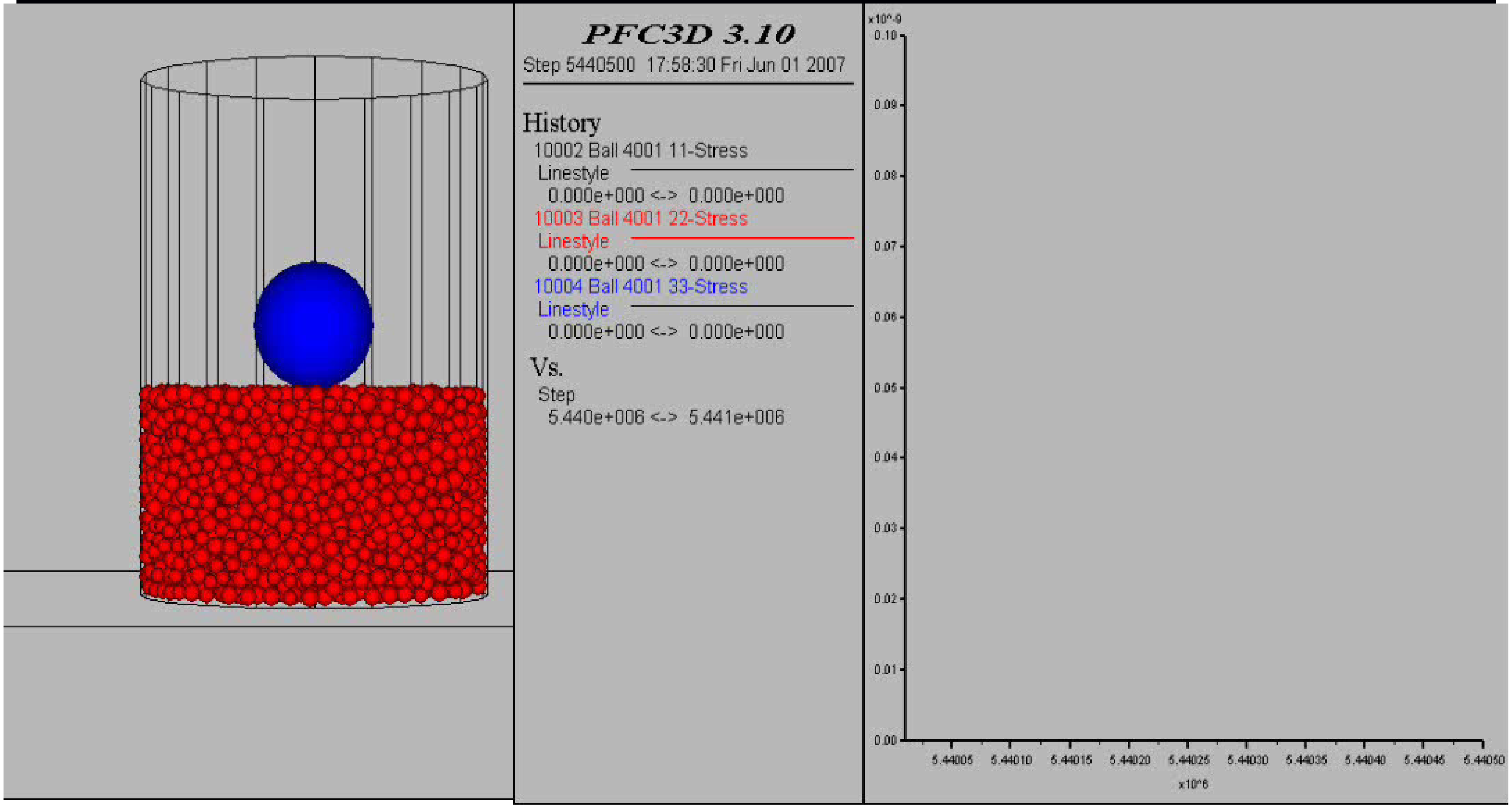

Measurement Area

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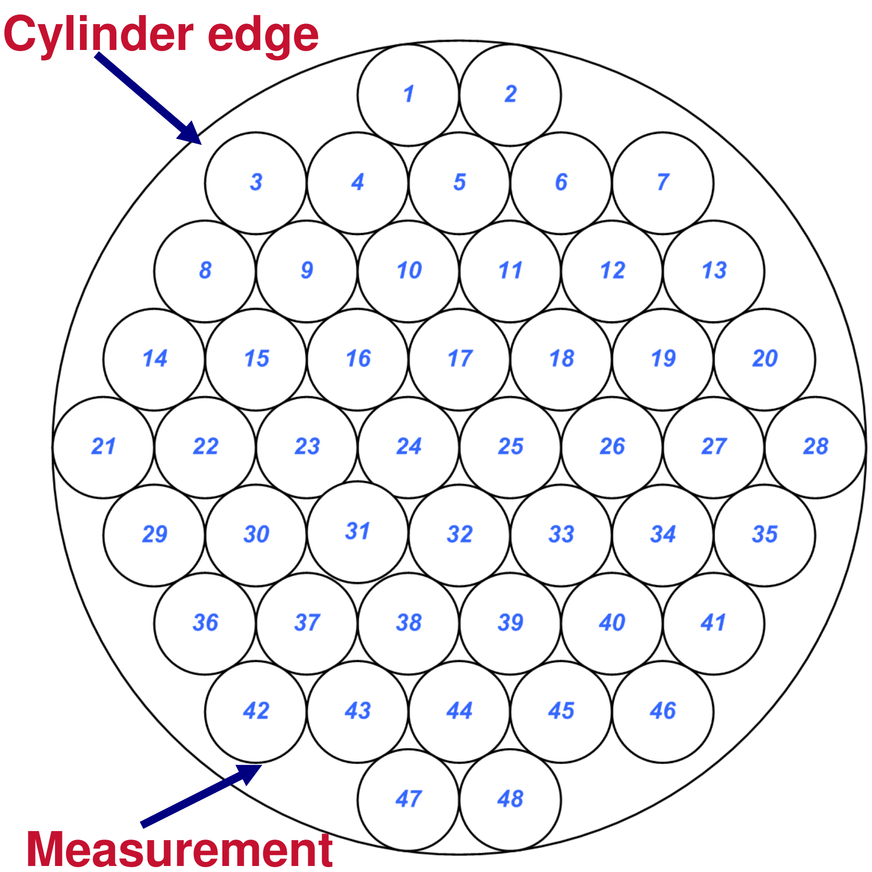
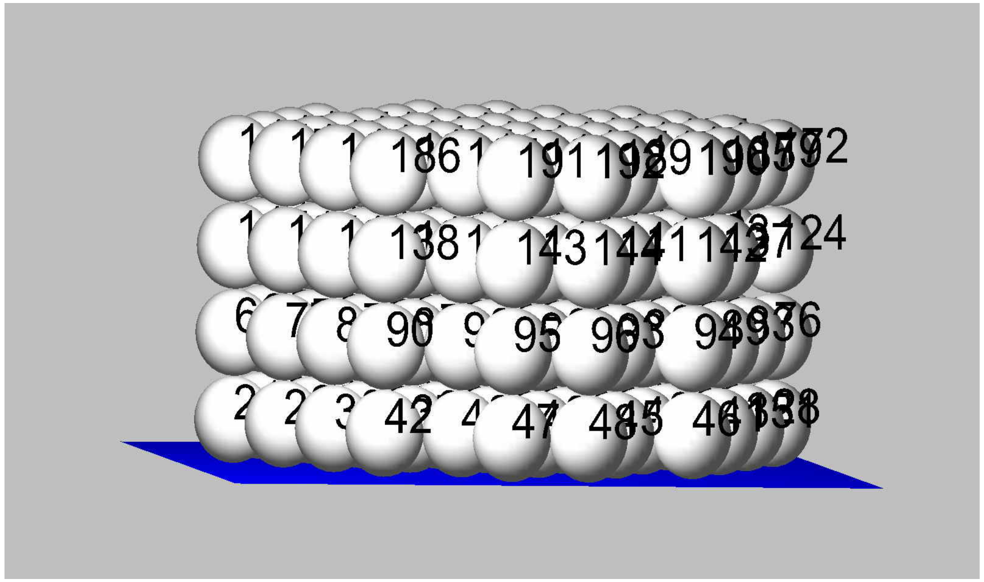
- ***Measurement position***





Cylinder Diameter: 2.88 mm

Measurement Number: 192

Measurement Diameter: 0.36 mm

Around each 9 particles are within a measure sphere.

Stress analysis

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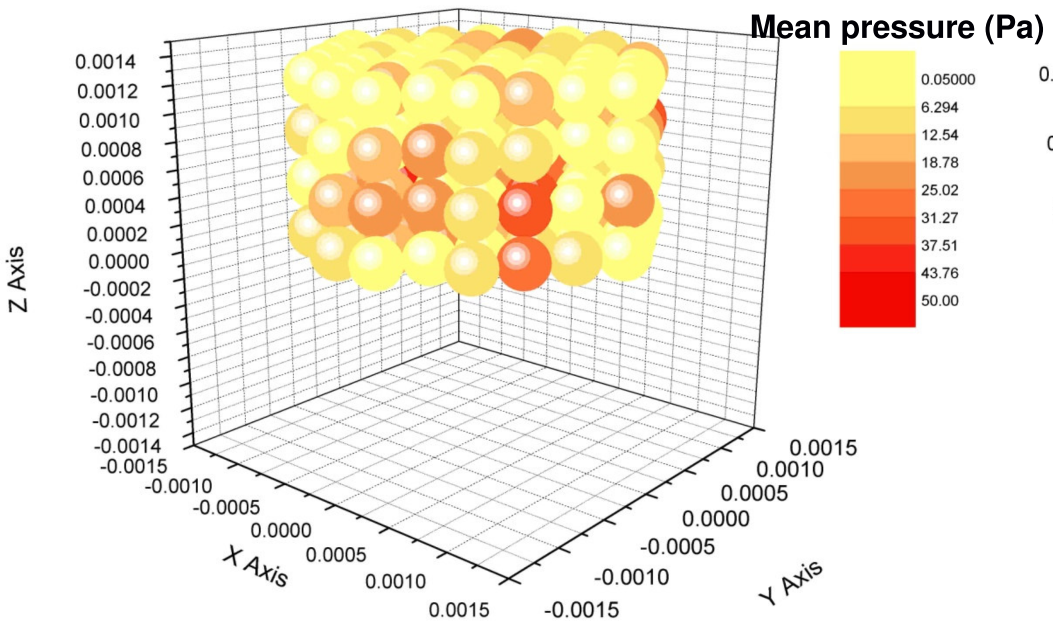

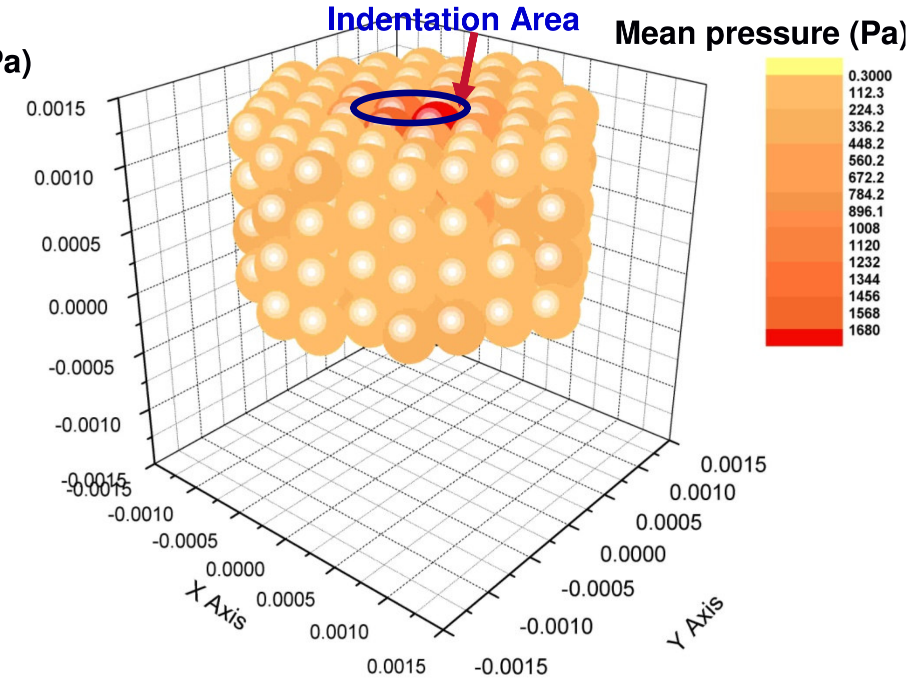

Mean pressure 3-D profile before indentation

Mean pressure 3-D profile after indentation

$$Mean\ \mathrm{Pressure} = \frac{\sigma_{xx} + \sigma_{yy} + \sigma_{zz}}{3}$$

Porosity analysis

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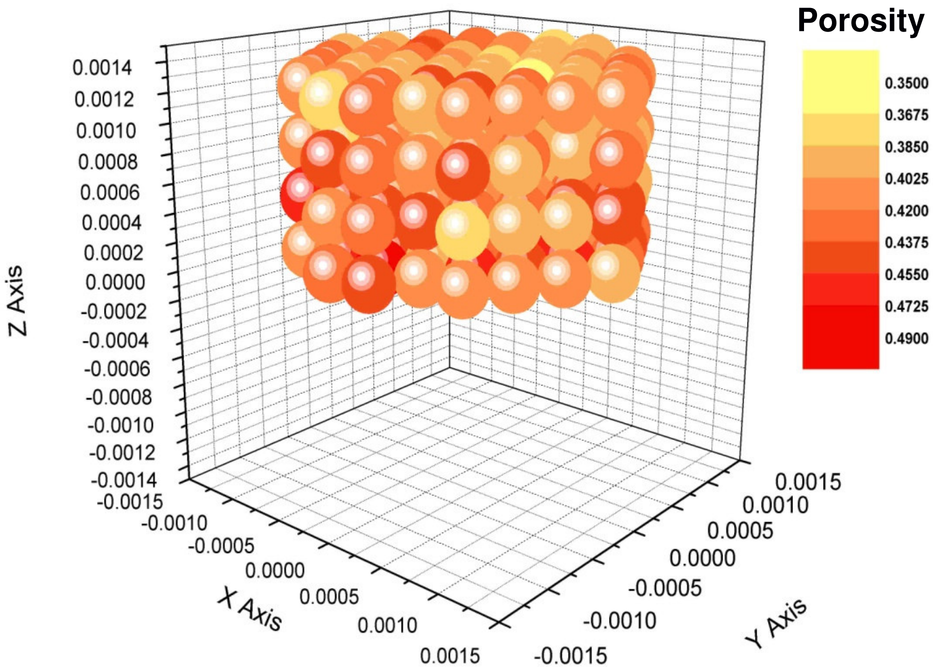
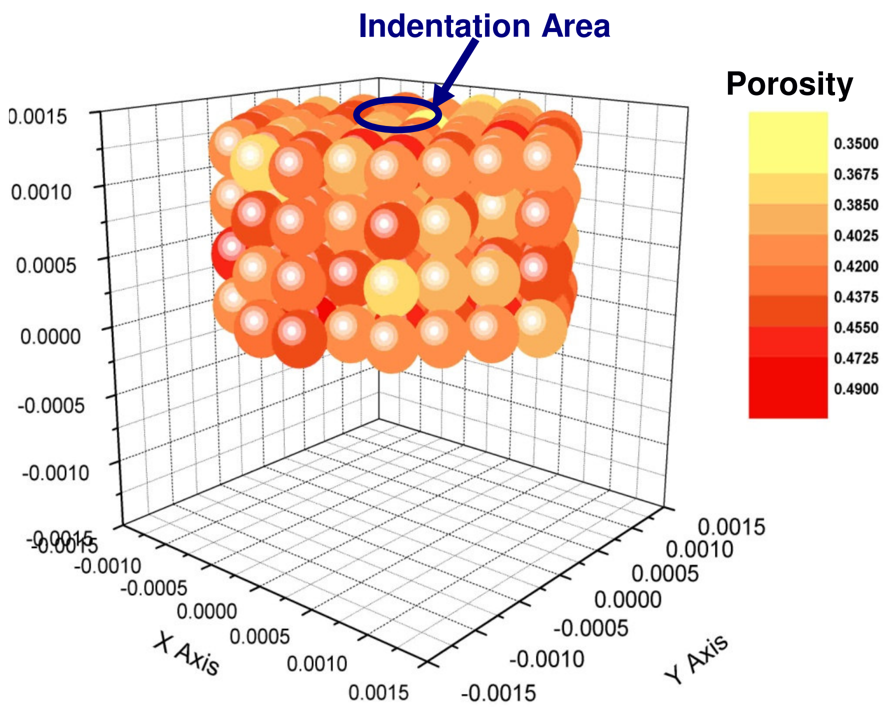


Porosity 3-D profile before indentation



Porosity 3-D profile after indentation

Porosity profile rarely changes during the indentation, which indicates the indentation process does not consolidated the particle assembly.

DEM on Indentation

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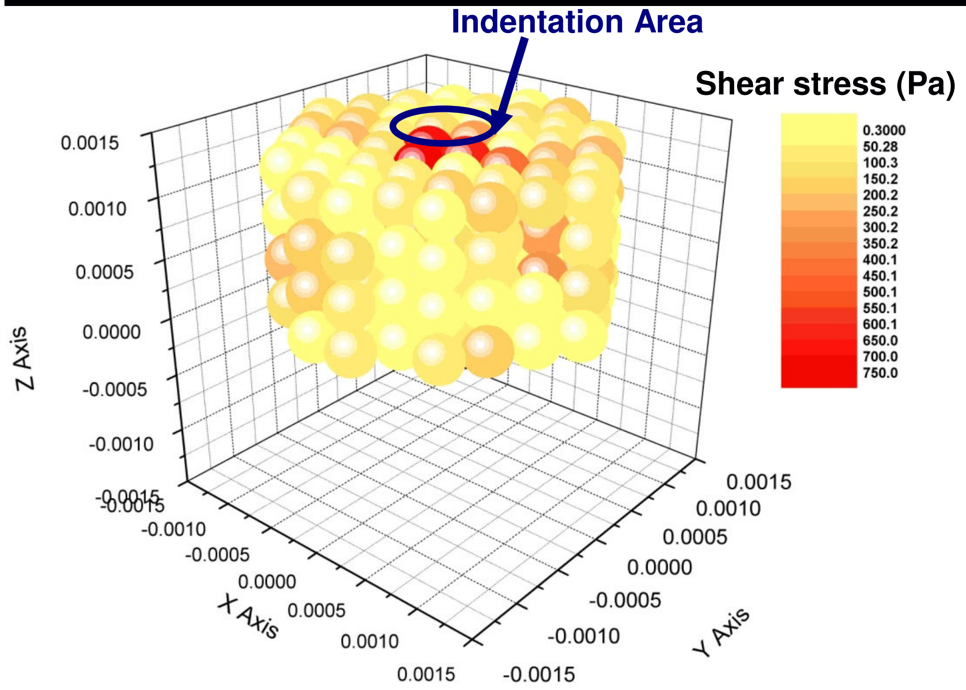

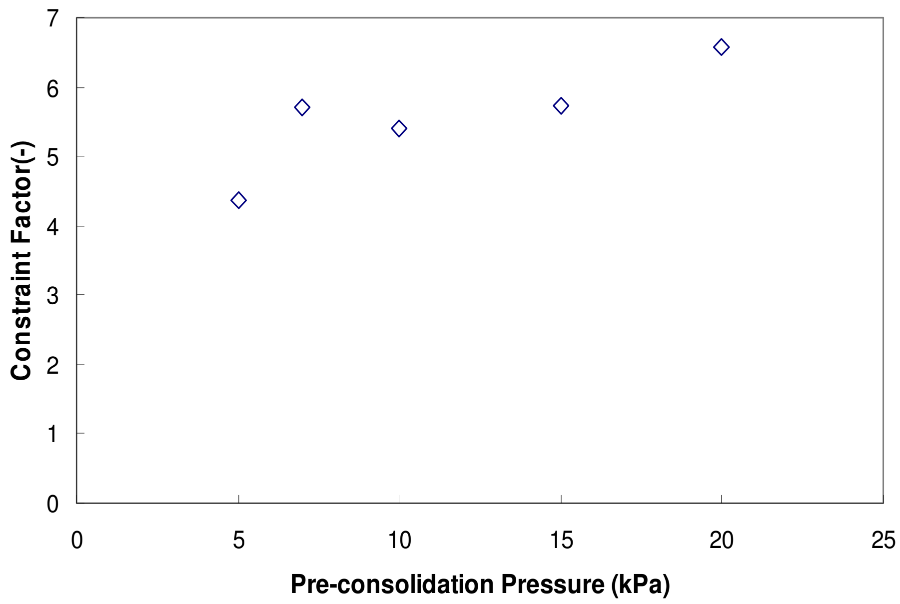

Experimental

Shear stress 3-D profile after indentation

$$Shear\ Stress = \frac{\delta_{zz} - \delta_{xx}}{2}$$

Constraint Factor (*C*)= Hardness/Shear Stress = 2.94 (cohesive glass beads)

Summary

- The indentation hardness and yield stress of weak compacts of α-lactose, Avicel, starch, cohesive glass beads (made cohesive by silanisation) and magnesium carbonate have been assessed using indentation and the trend compared with other techniques.
- The indentation hardness obtained from ball indentation can be related to the unconfined yield stress.
- The constraint factor (C) is being evaluated for a number of different materials.
- The relationship between single particle properties and bulk powder flow behaviour has been analysed by DEM. Further work is needed to provide good predictability.

Thank you for your attention !