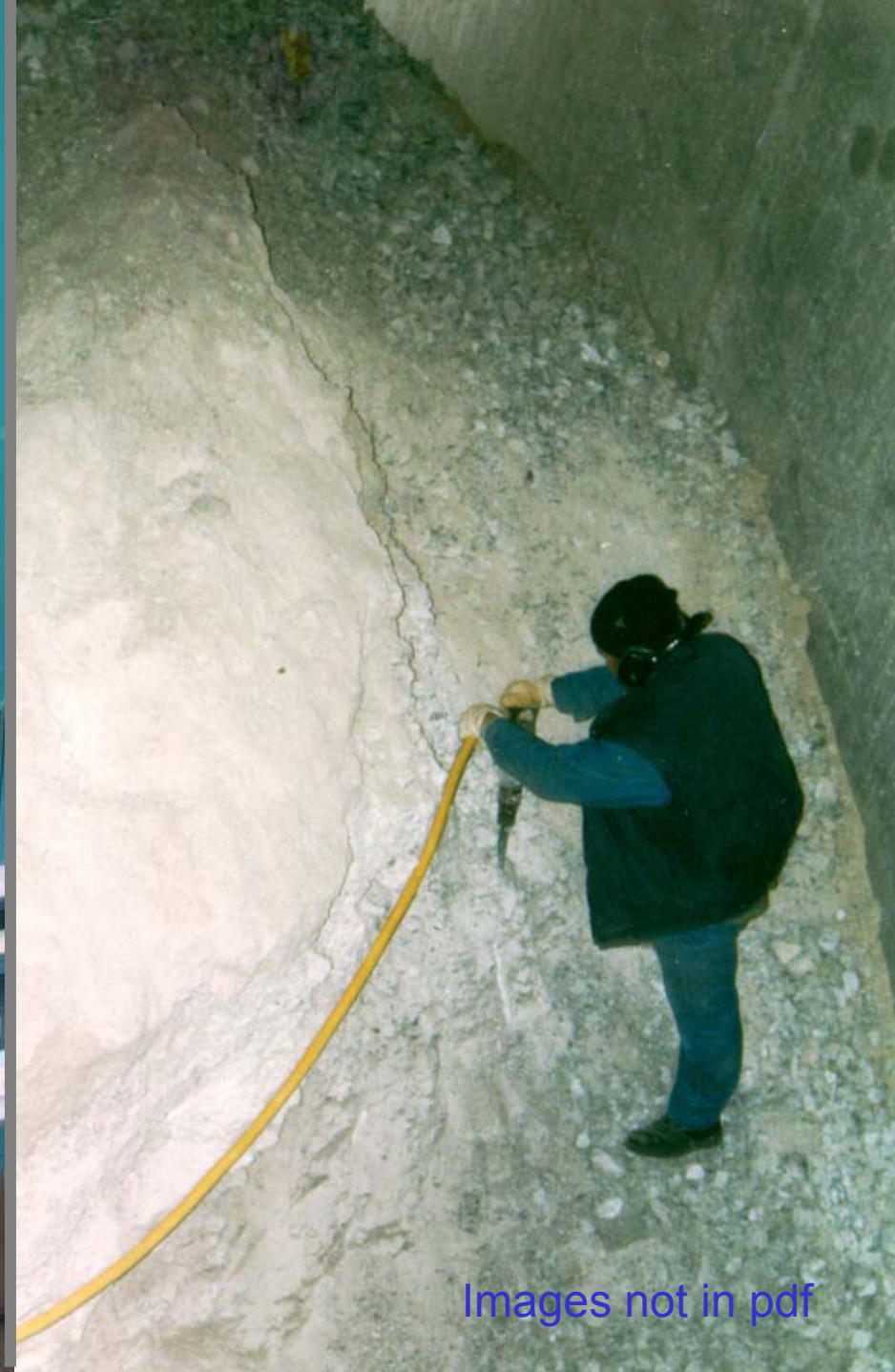# Setting Powder in Motion: Parameters and Descriptors

Dietmar Schulze

1. Why measuring flow properties?
2. The yield locus concept
3. Measurement of flow properties with shear testers
4. Examples

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1. Why measuring flow properties?

Flow problems

Images not in pdf

Why measuring flow properties?

## 1. Qualitative application: Flowability testing

- Will the bulk solid or powder flow in existing equipment (dosage systems, silo trucks, ...)?

- Will it flow uniformly (e.g., when filling dies of tablet presses)?

- Will it tend to caking during long-term storage?

- Can flowability be improved (flow agents, particle size distribution, moisture, surface characteristics, temperature ...)?

- Is a product within specs regarding flowability (quality control)?

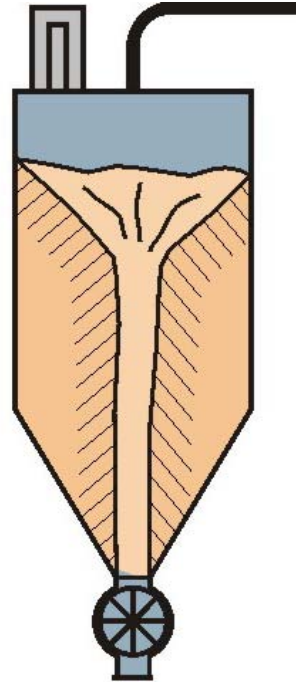## 2. Quantitative application: Equipment design

- Goal: To avoid flow problems with equipment designed according to flow properties →

Sources of flow problems

Problems related to the **strength** of the bulk solid (flow obstructions):
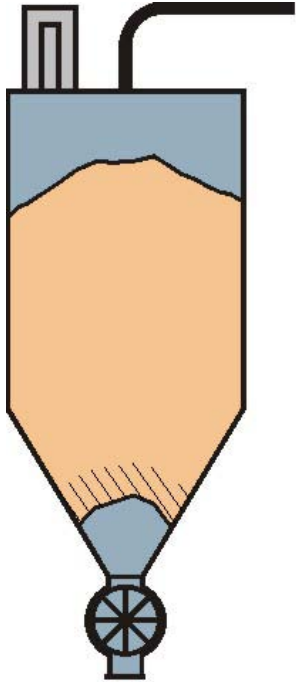
Stable arch (arching)

Stable rathole or pipe
(ratholing, piping)

Strength may increase with time → caking, time consolidation

Ratholing (small scale)

Ratholing (large scale), strong time consolidation

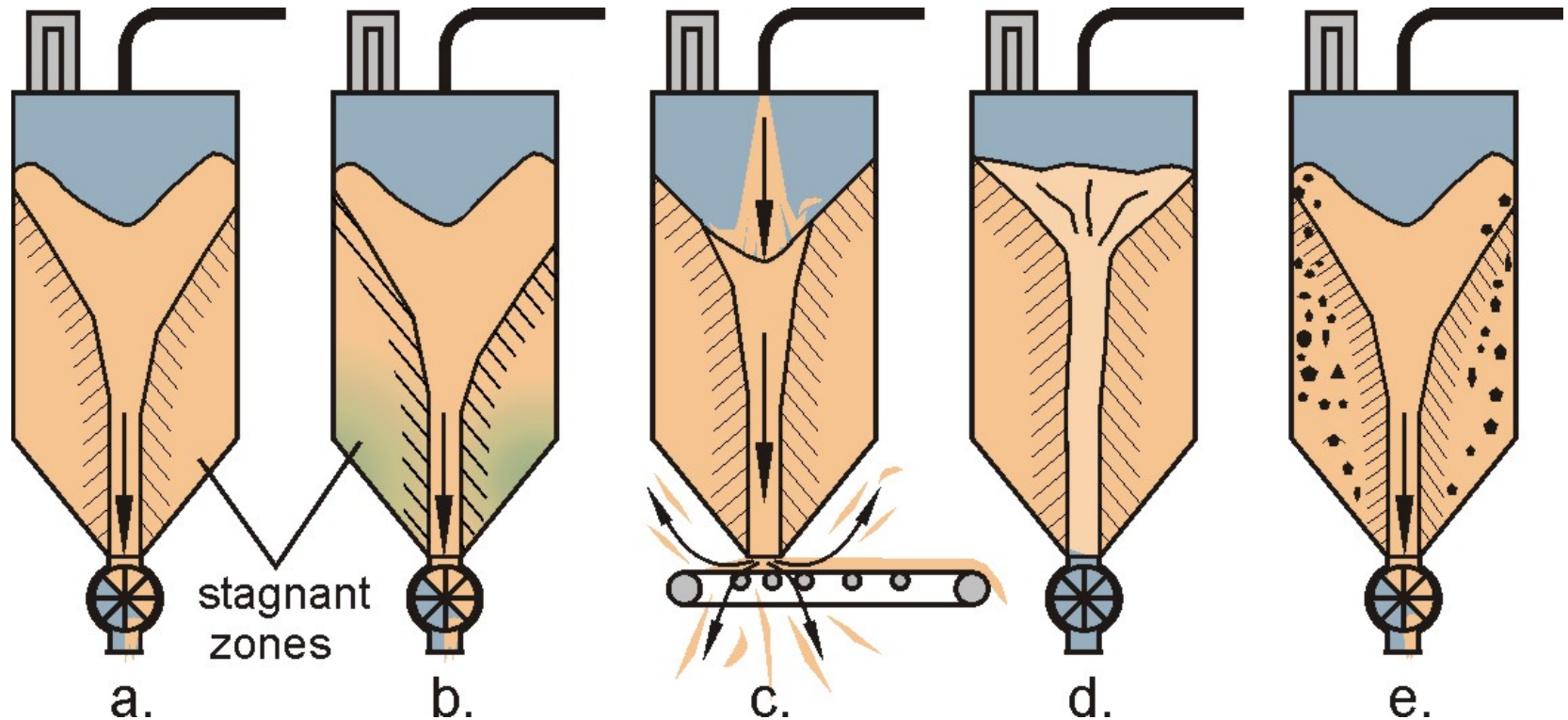Images not in pdf

Adhesive forces

Image not in pdf

Sources of flow problems

Problems related to **funnel flow**
(important parameter: **friction at hopper wall**):

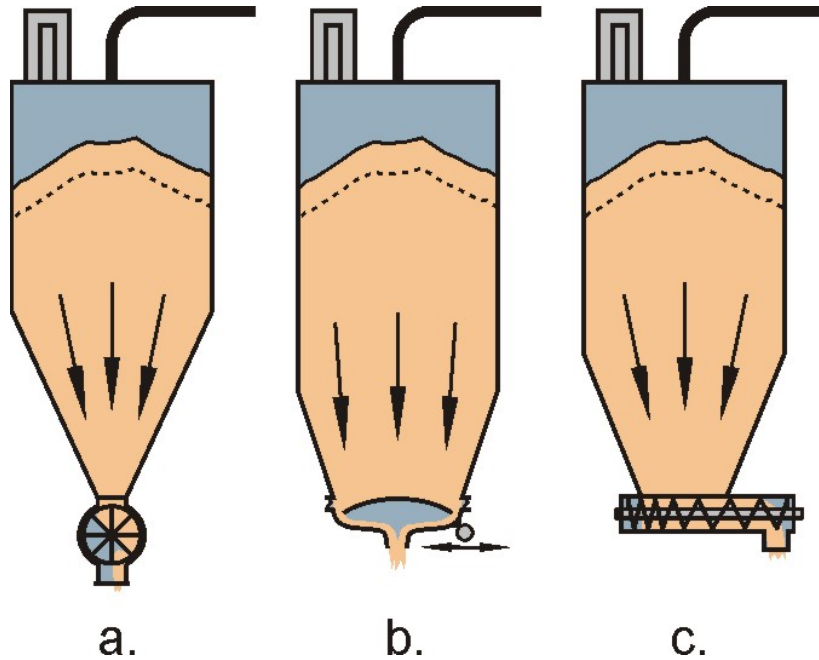a. Funnel flow   b. Wide residence time distribution

c. Irregular flow   d. Ratholing   e. Segregation

Equipment design

Solution: Avoid funnel flow, avoid flow obstructions.

a.   b.   c.

a.   **Mass flow** achieved by sufficiently steep/smooth hopper walls
       (most important parameter: **friction at hopper wall**)

b./c. **Sufficiently large outlet opening** for cohesive products
       (most important parameter: **strength**)

→ **Flow properties have to be known.**

2. The yield locus concept

Jenike's approach

A.W. Jenike

- About 50 years ago Andrew W. Jenike defined the **yield locus** as a means to describe bulk solid properties.
- Furthermore, Jenike introduced an appropriate test method (shear test).
- The yield locus is still state of the art. It is preferred if well-defined physical quantities are needed, e.g.,
  - for quantitative application (e.g., hopper design),
  - for high reproducibility,
  - for test results not being influenced by other parameters (e.g., state of aeration).
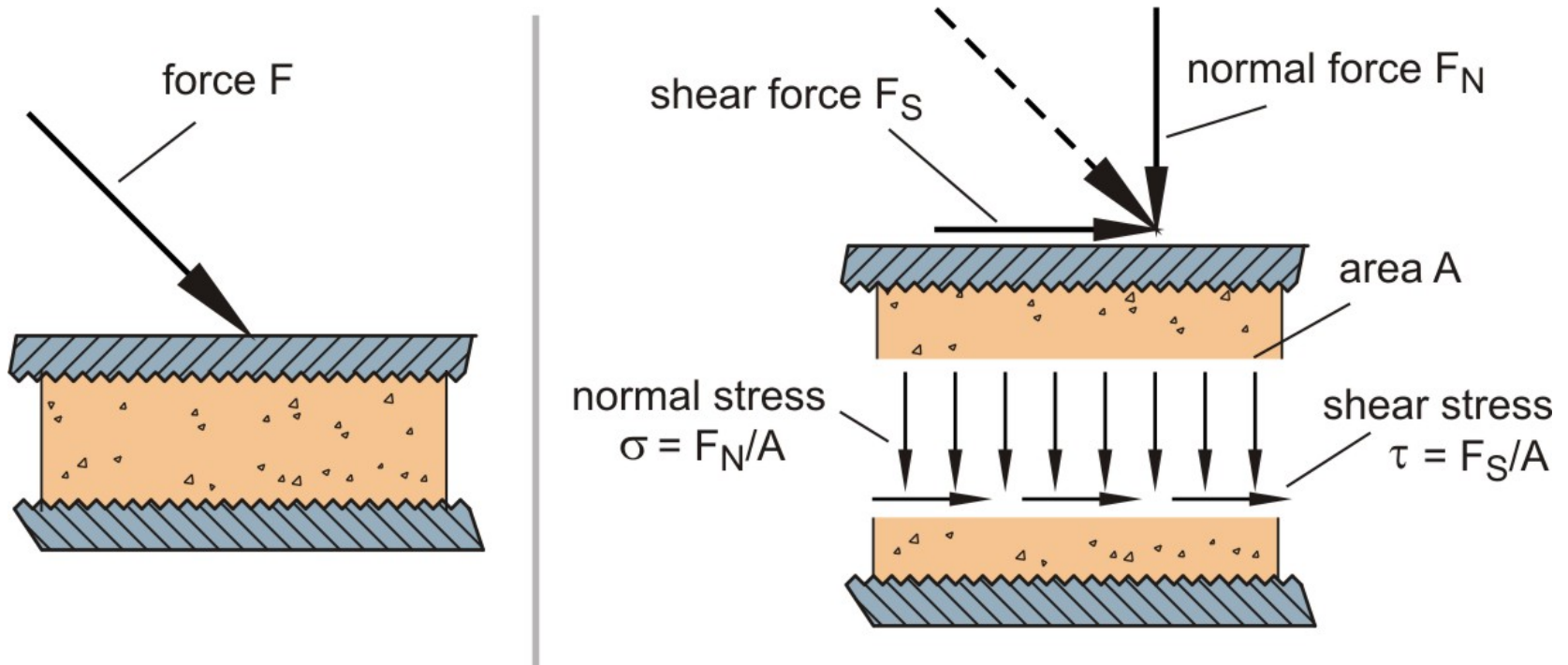
To understand the yield locus concept, it is helpful to know the relationship between stresses and strength.
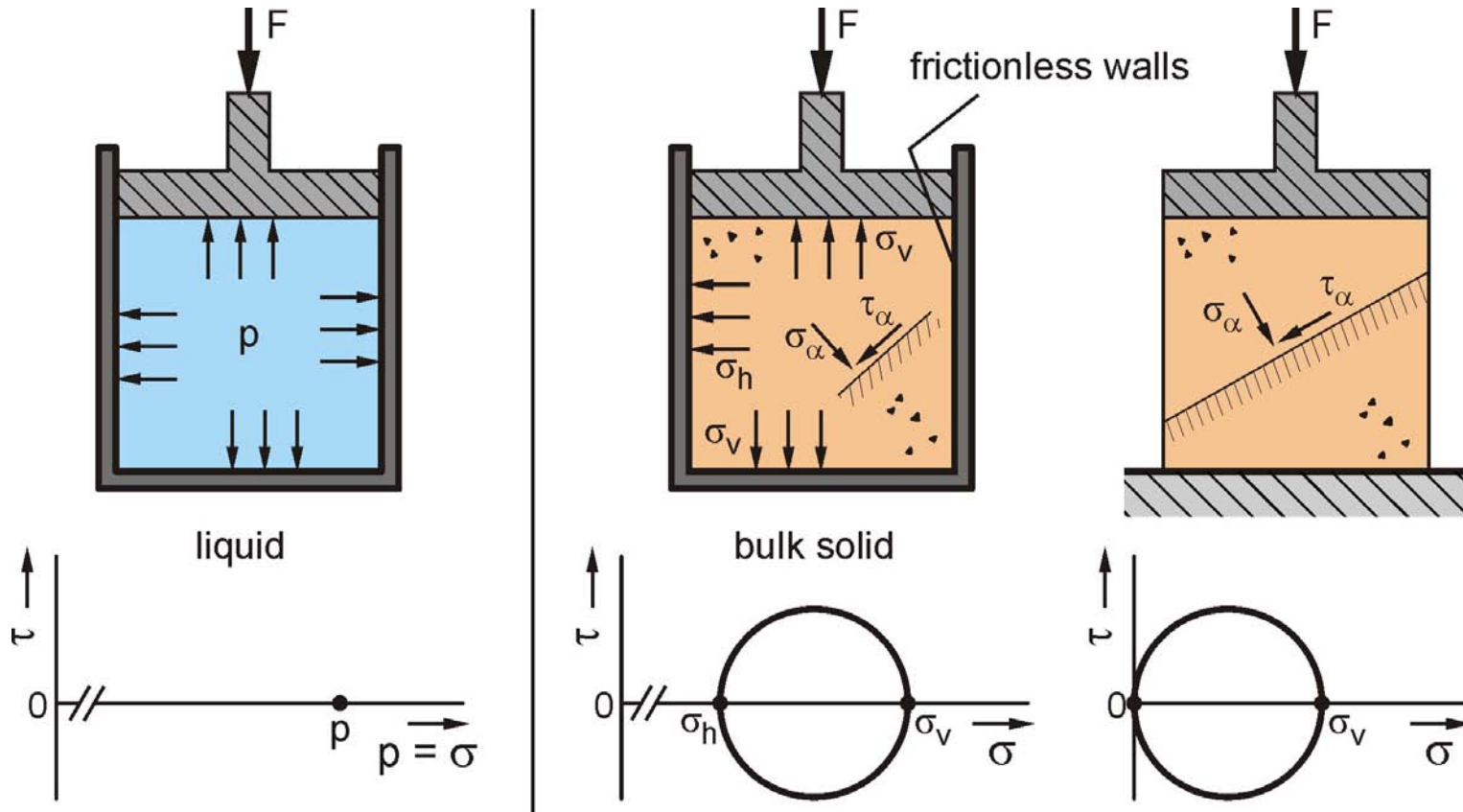
2.1 Stresses

Introduction: Stresses

Bulk solid is regarded as a continuum.

Normal stress $\sigma = F_N/A$:
stress acting normal to the horizontal cutting plane (area A).

Shear stress $\tau = F_S/A$:
stress acting parallel to the horizontal cutting plane (area A).

Stress in powders compared to pressure in liquids

- In a powder different stresses can be found in different cutting planes.
- Even if the material is at rest, shear stresses τ may occur.
- Pairs of values of normal and shear stresses acting in different cutting planes form a Mohr stress circle in a normal stress – shear stress – diagram.
- Liquid: pressure p in all directions → Radius of "stress" circle = 0
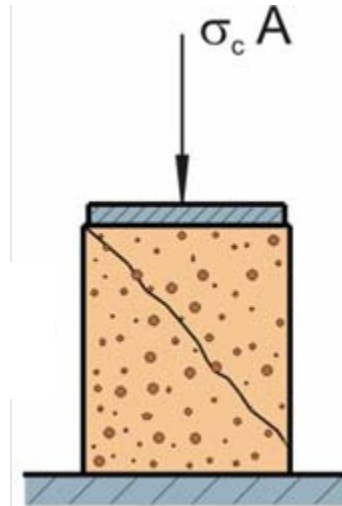
2.2 Strength
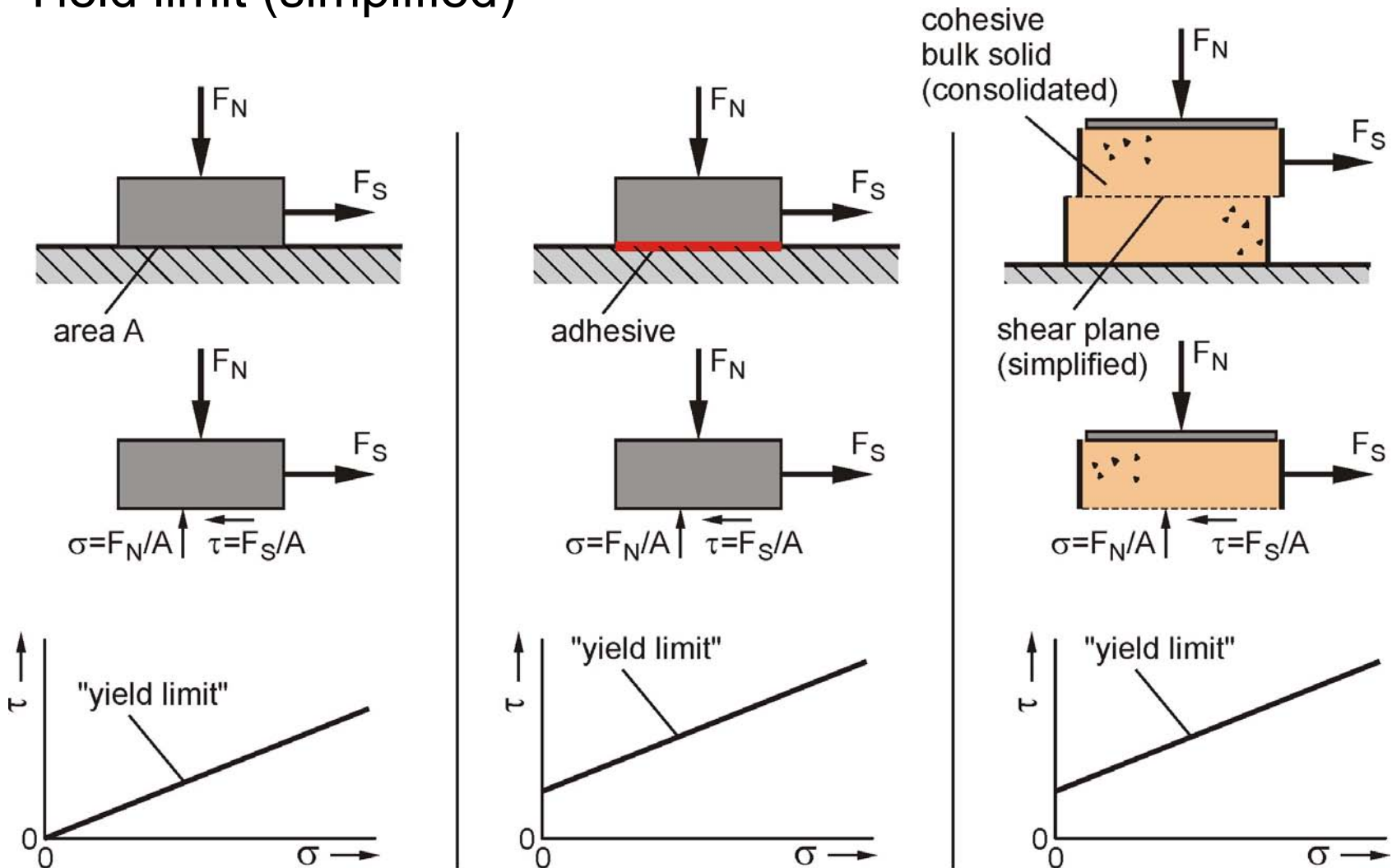
Unconfined yield strength (uniaxial compression test)
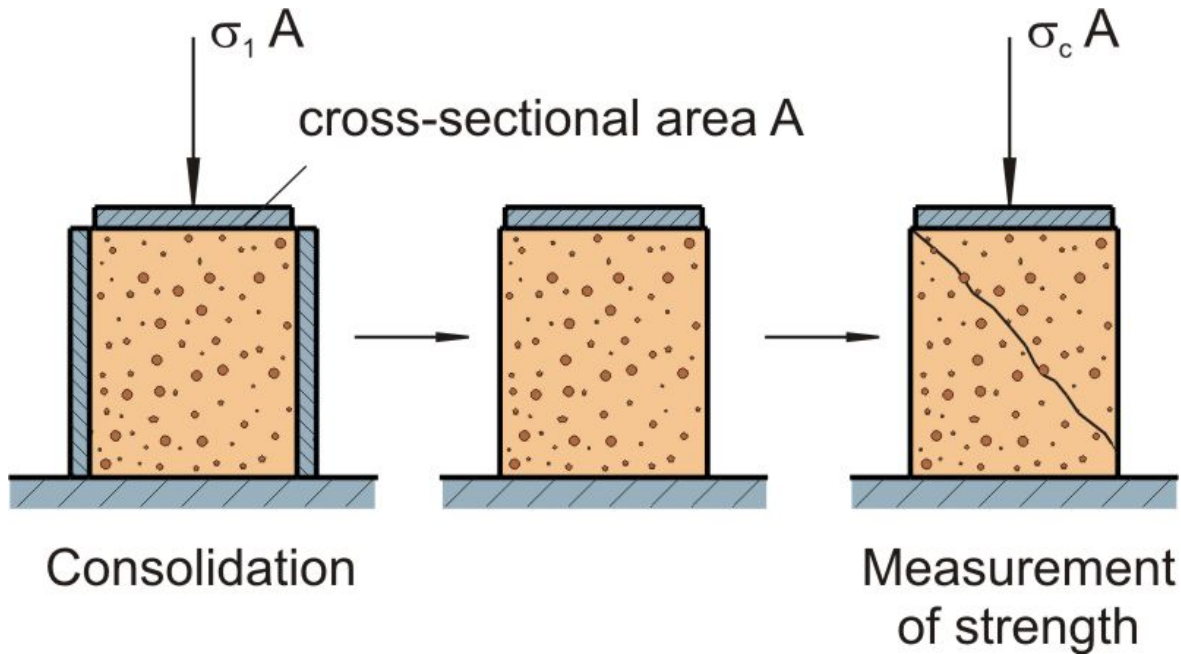
Consolidation with consolidation stress, σ_1.

Measurement of unconfined yield strength, σ_c (compressive strength).

The question is:
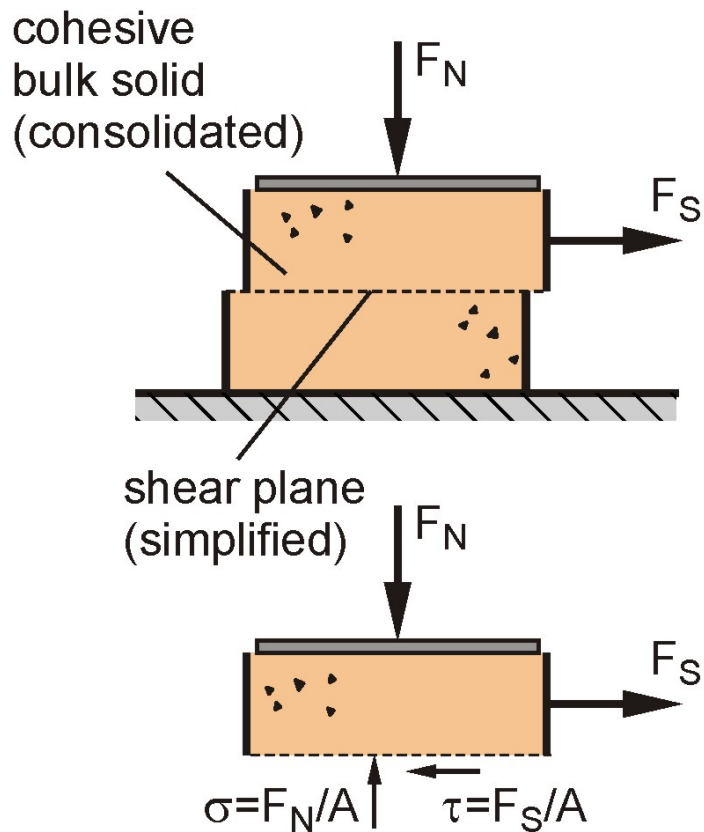# Where does flow or failure happen?
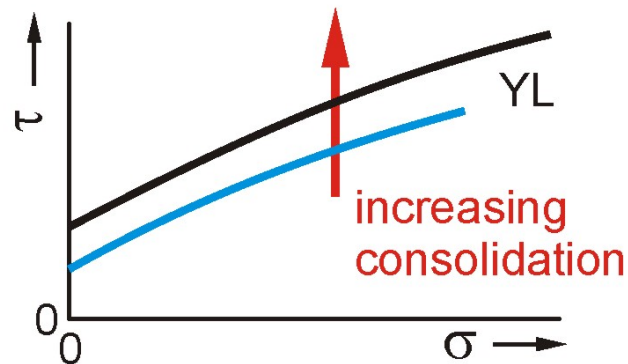# And why?

Yield limit (simplified)

A yield limit represents the shear stress which is required to initiate flow (failure) as a function of normal stress.

Yield limit of consolidated bulk solid → Yield locus

cohesive bulk solid (consolidated)

F_N

F_S

shear plane (simplified)

F_N

F_S

$\sigma = F_N/A$ $\tau = F_S/A$

Yield loci (YL) of a cohesive bulk solid:

τ

σ

YL

increasing consolidation

0

0

- A yield locus describes the "strength" of a bulk solid.
- Yield loci of cohesive bulk solids are shifted toward higher shear stresses with increasing consolidation stress.

Uniaxial compression test represented by stress circles
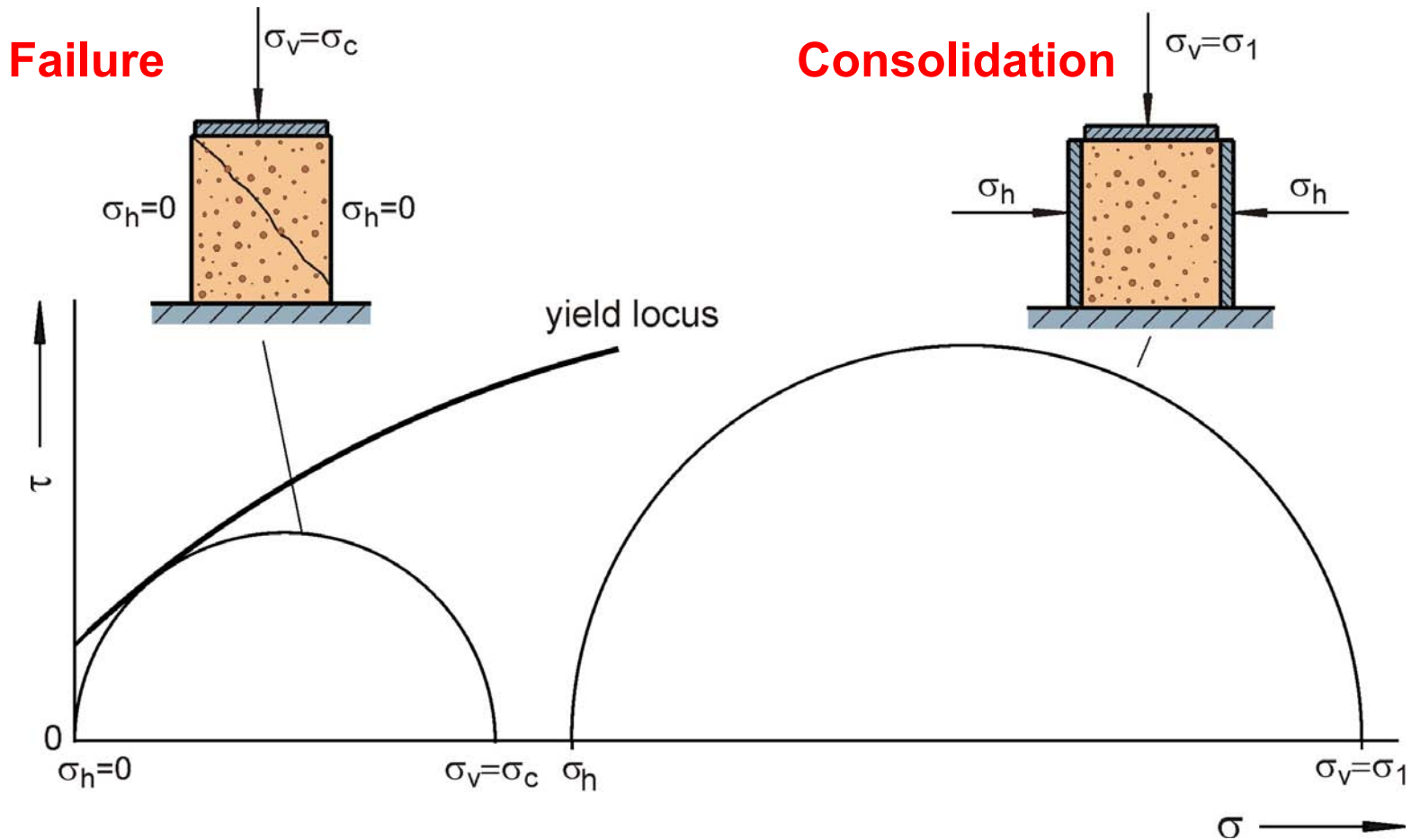
Stress circles represent stresses at the end of consolidation (right) and at failure (incipient flow, left).

Consolidation stress σ_1 is the greatest normal stress acting on the specimen (right point of intersection with σ-axis). It is also known as "major principal stress".

Determination of unconfined yield strength
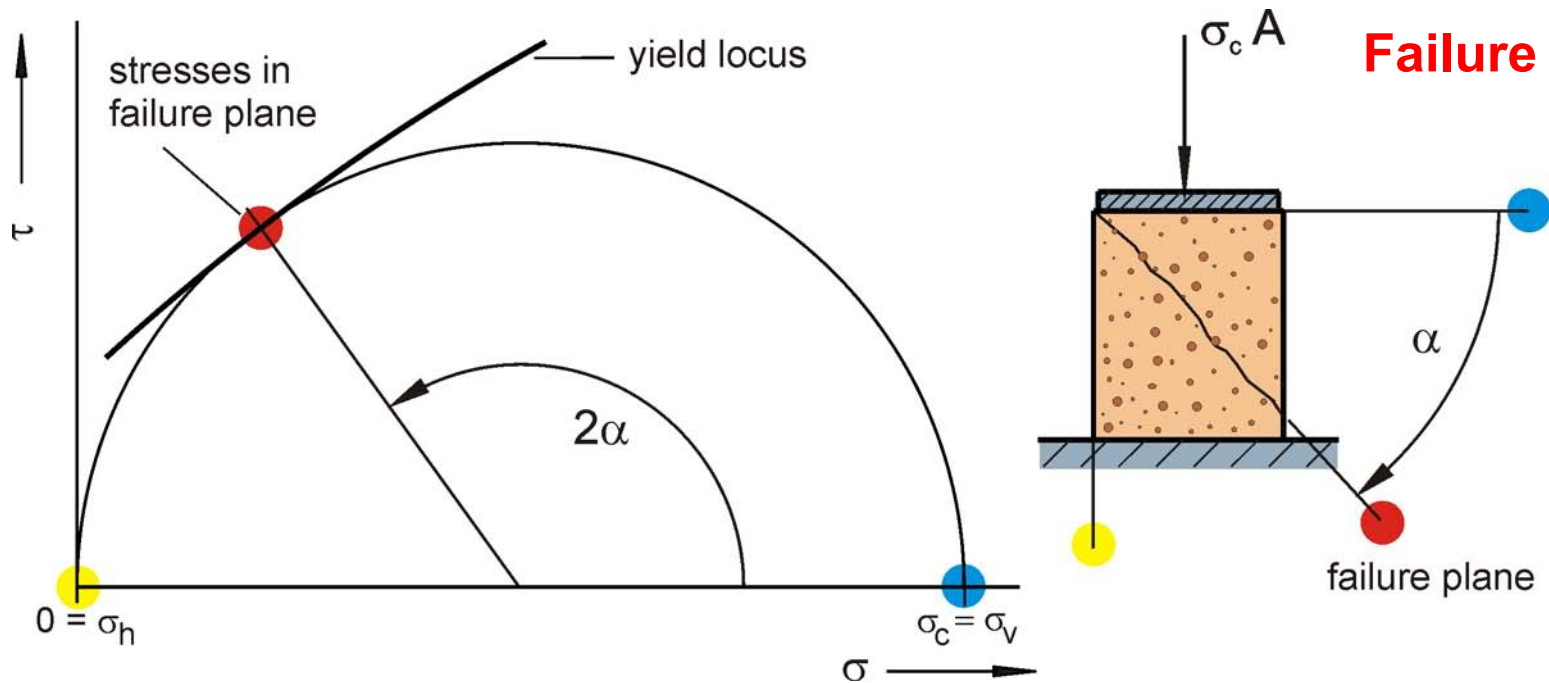
- To initiate flow, the failure criterion given by the yield locus must be matched: **Shear stress must be sufficiently large to move particles against each other.**

- This is the case in planes represented by the red point where the Mohr circle (representing the stresses) touches the yield locus.

- The orientation of the plane represented by the red point represents the orientation of failure planes.

3. Measurement of flow properties with shear testers

3.1 Yield locus test

Yield locus test (I)

Shear stress, τ, and bulk density, ρ_b, during a shear test.

preshear (consolidation)   shear to failure
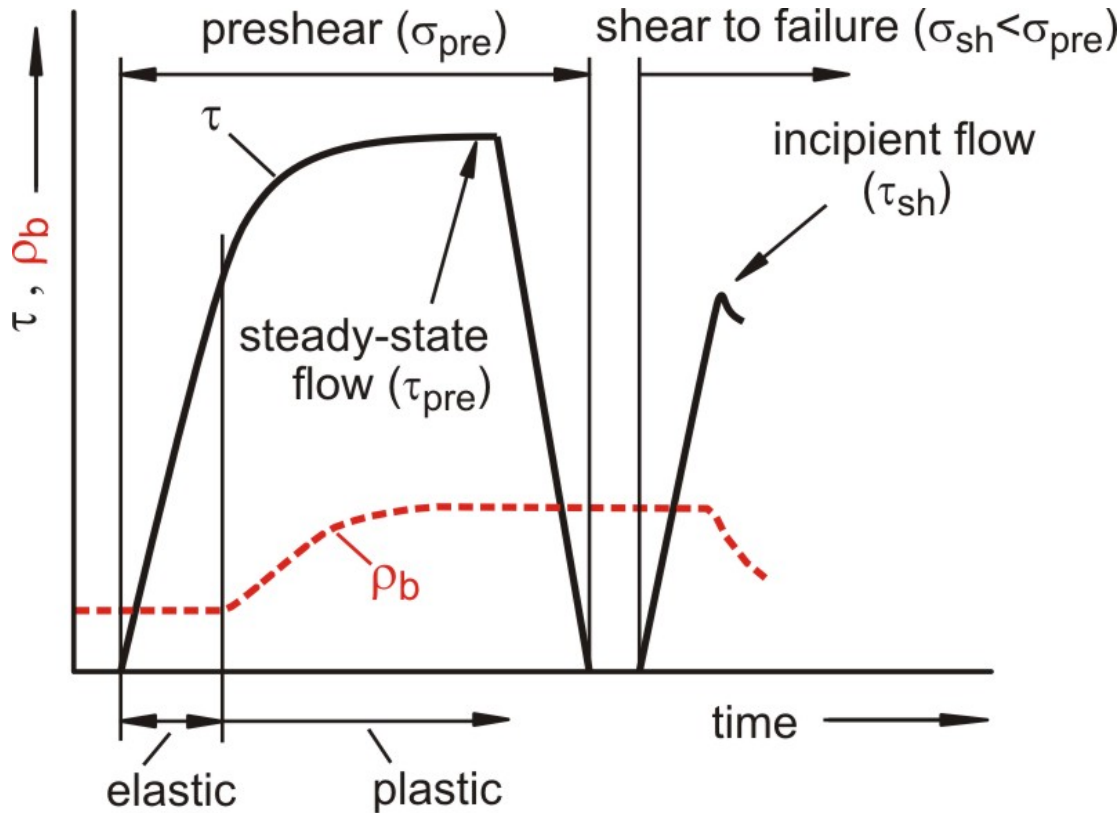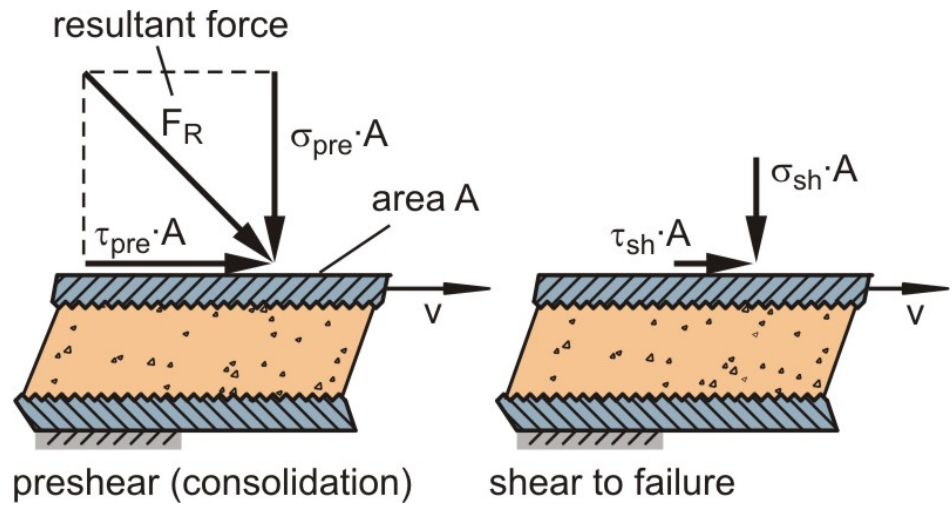
Preshear: Consolidation of a bulk solid specimen until steady-state flow is attained.
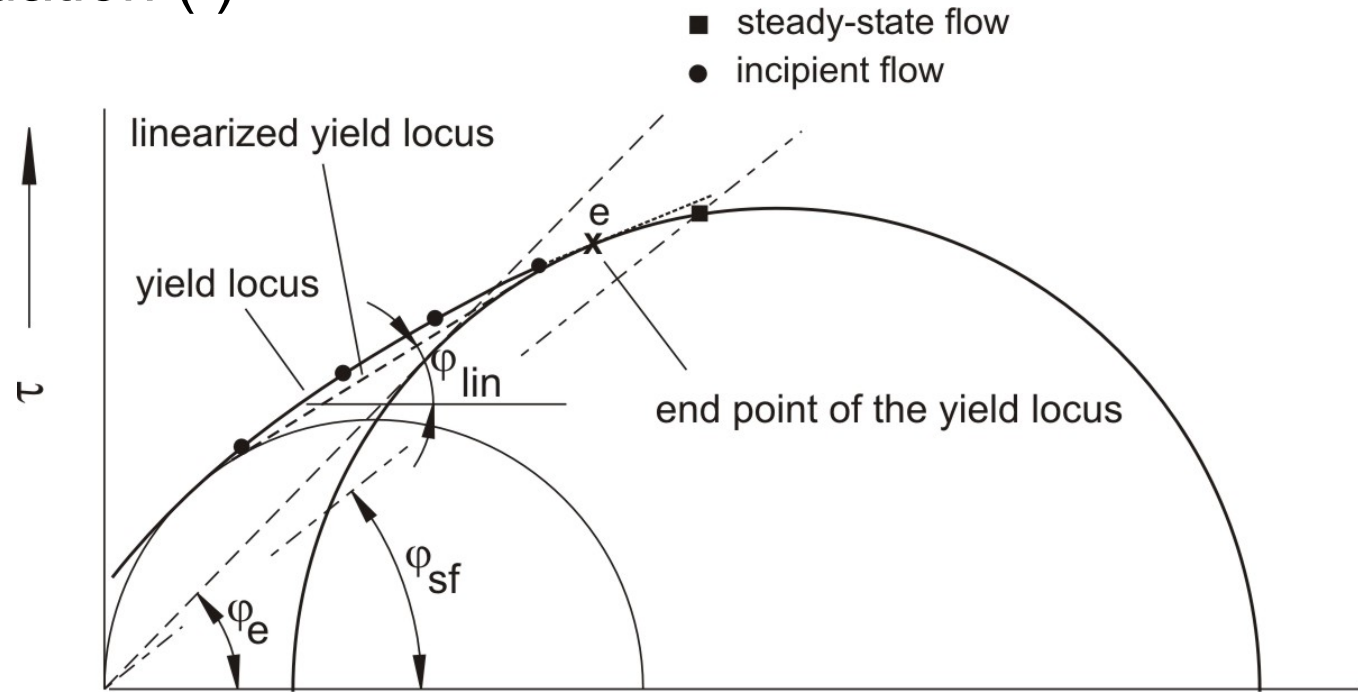
Shear (to failure): Measurement of the shear stress required to initiate flow.

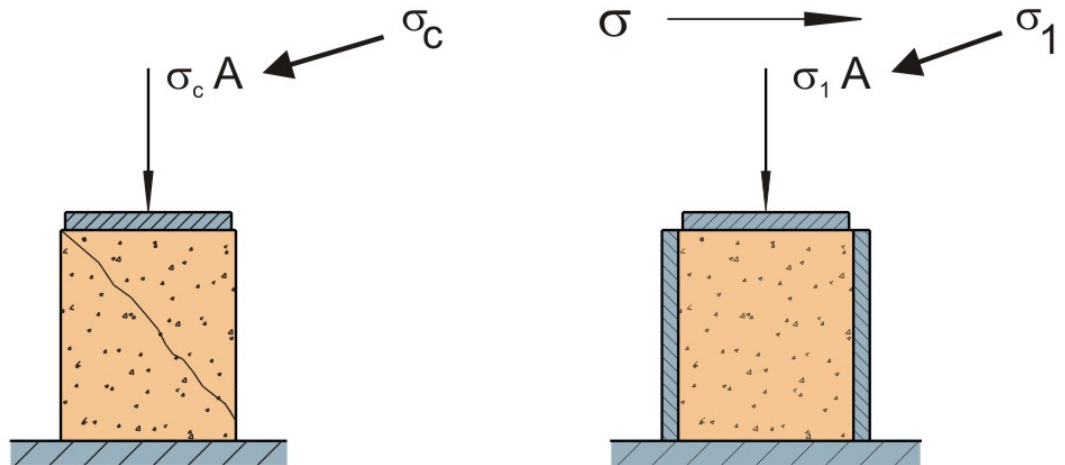# Yield locus test (II)
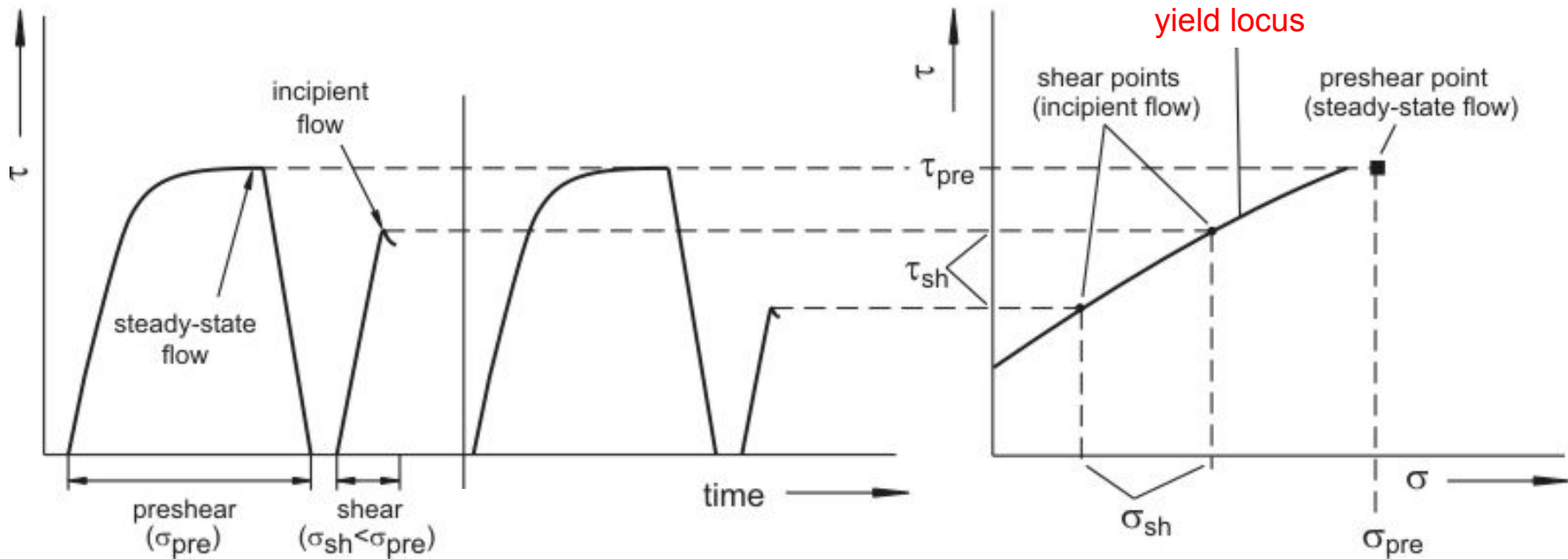
- Preshear at σ_{pre}, then shear (to failure) under normal stress $\sigma_{sh} < \sigma_{pre}$.
- This has to be repeated several times, whereby σ_{sh} is varied.
- Thus one obtains several points of the yield locus.

Yield locus evaluation (I)

Two Mohr stress circles are drawn:

- the left one defining the **unconfined yield strength, σ_c,**
- the right one defining the **consolidation stress, σ_1.**

- steady-state flow
- incipient flow

linearized yield locus

yield locus

φ_{lin}

end point of the yield locus

φ_{sf}

φ_e

e

τ

σ_c

σ

σ_1

$\sigma_c\, A$

$\sigma_1\, A$

Yield locus evaluation (II)

Most important parameters of the yield locus → flow properties:
- **consolidation stress, σ_1**
- **unconfined yield strength, σ_c**
- **various angles (φ_e, φ_{lin}, φ_{sf})** describing internal friction and YL slope
- **bulk density, ρ_b** (follows from specimen volume and mass)
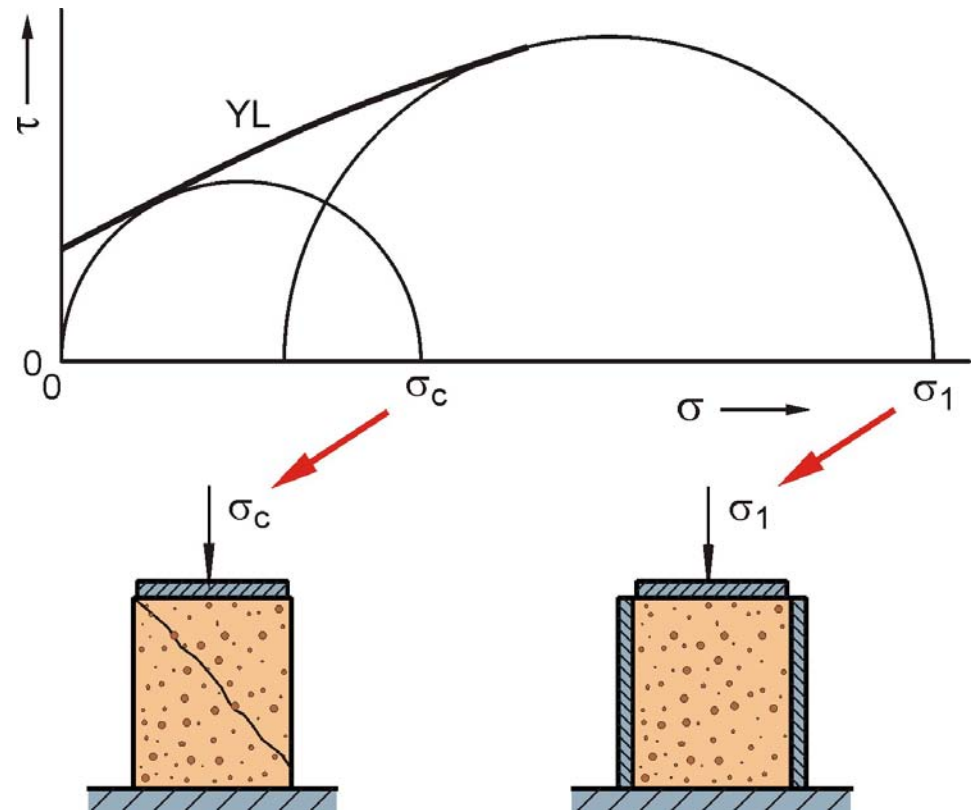- **flowability** →

Flowability ff_c ← most important for qualitative application

Definition:

$$ff_c = \frac{\sigma_1}{\sigma_c} = \frac{\text{consolidation stress}}{\text{unconfined yield strength}}$$
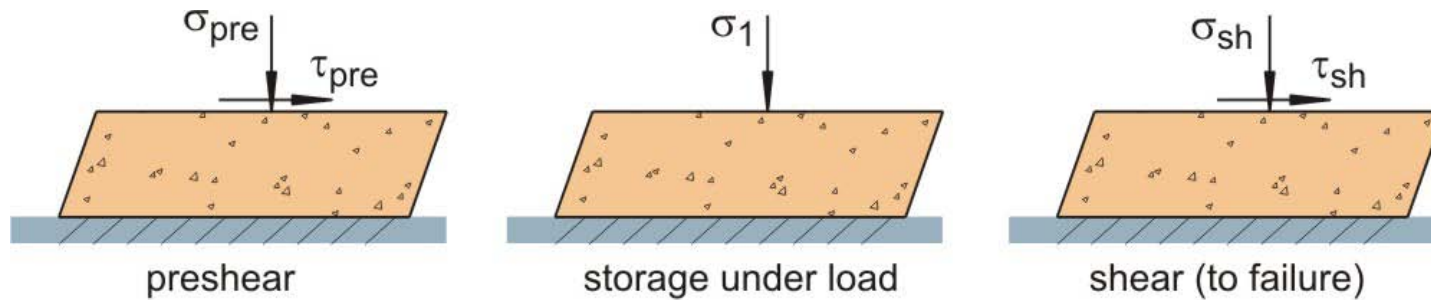
Classification of flow behaviour:

| ff_c	description
< 1	not flowing
1 .. 2	very cohesive
2 .. 4	cohesive
4 ..10	good-flowing
> 10	free-flowing

3.2 Time consolidation test

Time consolidation test

Procedure similar to yield locus test, but after preshear the specimen is stored under static load σ_1 over desired time interval, t.
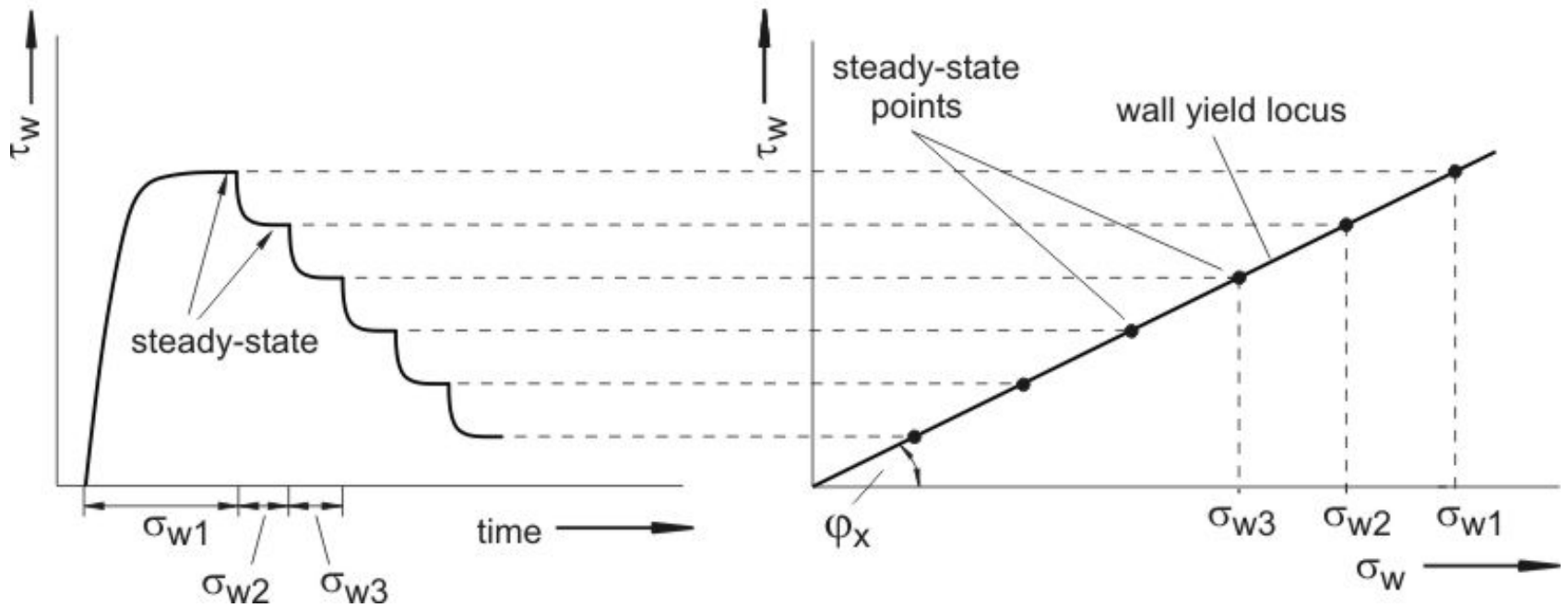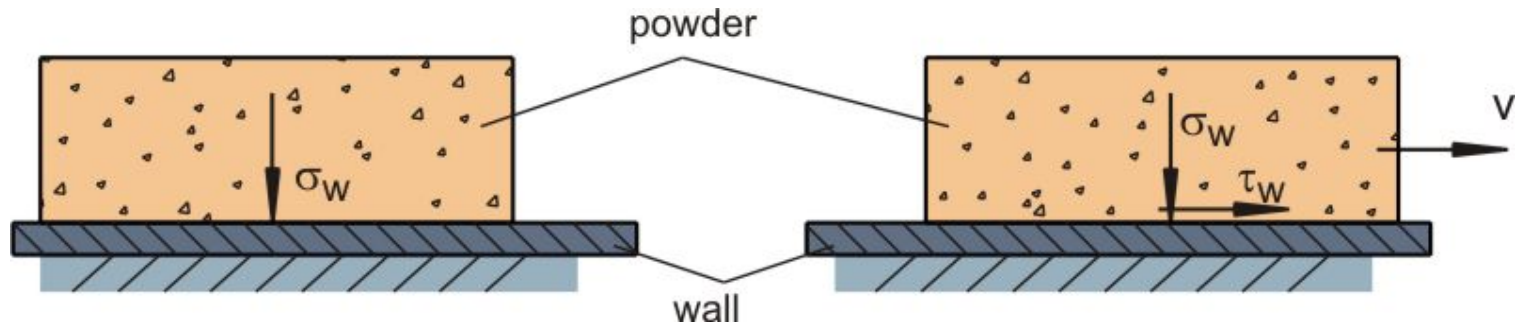
Subsequently a point of the time yield locus is measured.

Unconfined yield strength, σ_c, is determined from time yield locus.
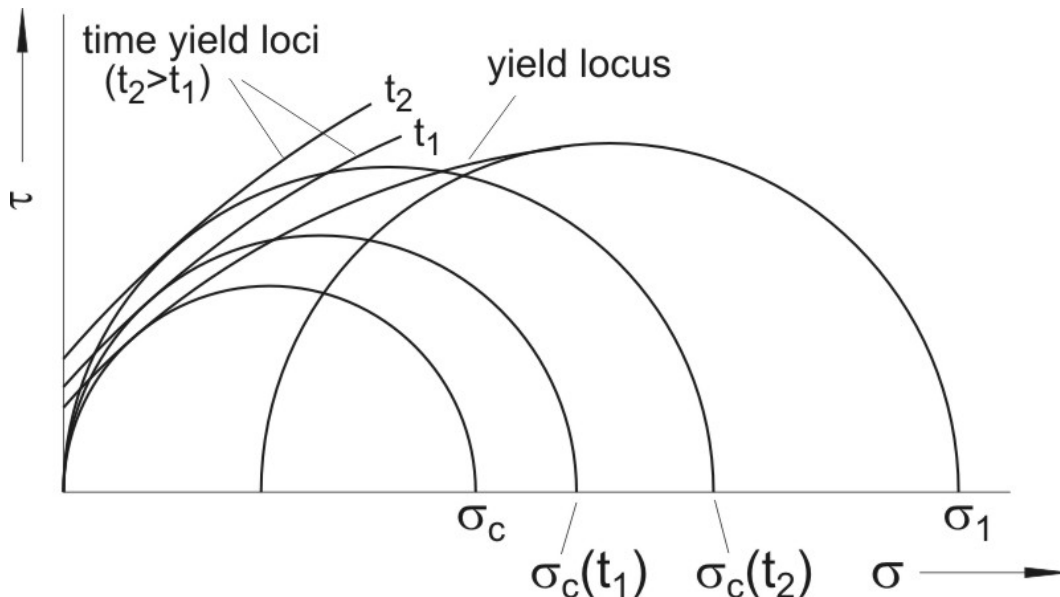
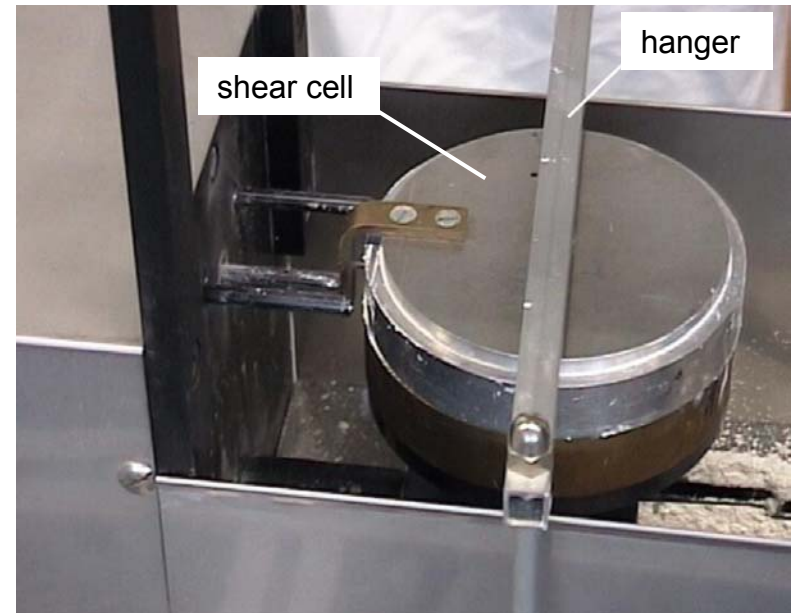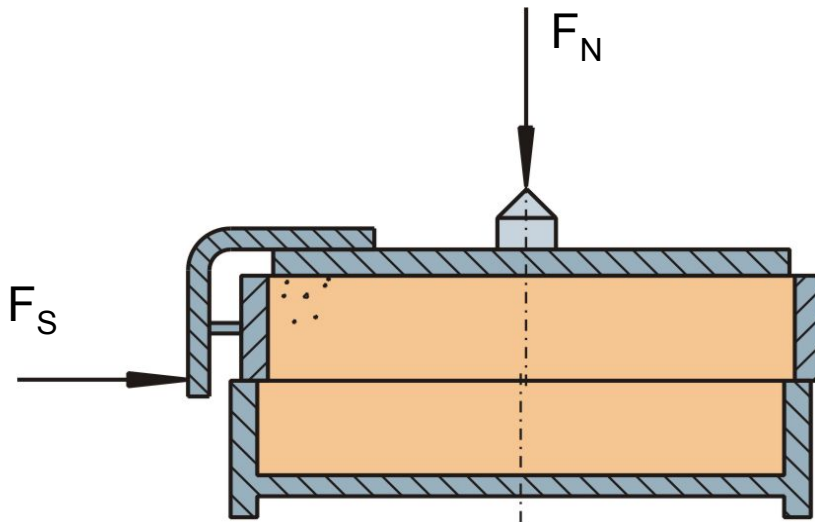# 3.3 Wall friction test

Wall friction test
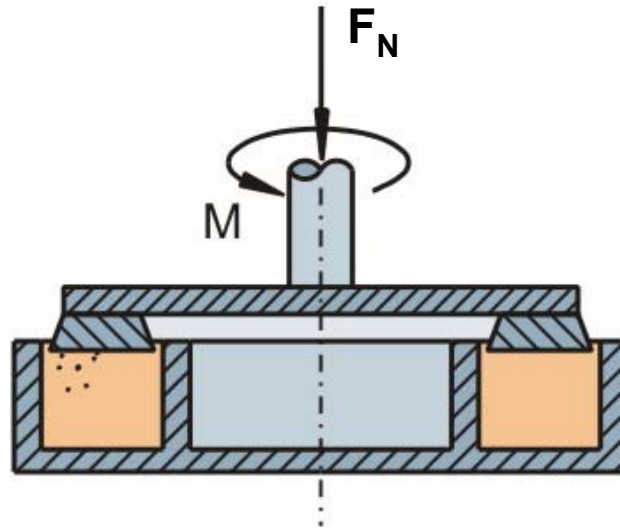

Determination of a wall yield locus

3.4 Shear testers

Jenike Shear Tester

- The first shear tester for powders and bulk solids.

- Manually operated, requires skill and time (1... 2 hours / YL)

- ASTM 6128 "Jenike shear cell"

Ring Shear Tester

F_N

M

- Applied in powder technology since about 1965 (Carr and Walker, "annular shear tester").

- New design developed in 1992/93: "Schulze Ring Shear Tester"
  - specimen volume from 85 cm^3 to 900 cm^3 (particles up to 10 mm)
  - automated test procedure (ca. 15 min / YL; operator time ca. 5 min).
  - ASTM 6772 "Schulze Ring Shear Tester"

- "Small volume tester" RST-XS since 2002 (3.5 cm^3 to 70 cm^3) →

Shear cell of Ring Shear Tester RST-XS (30 cm³ cell)
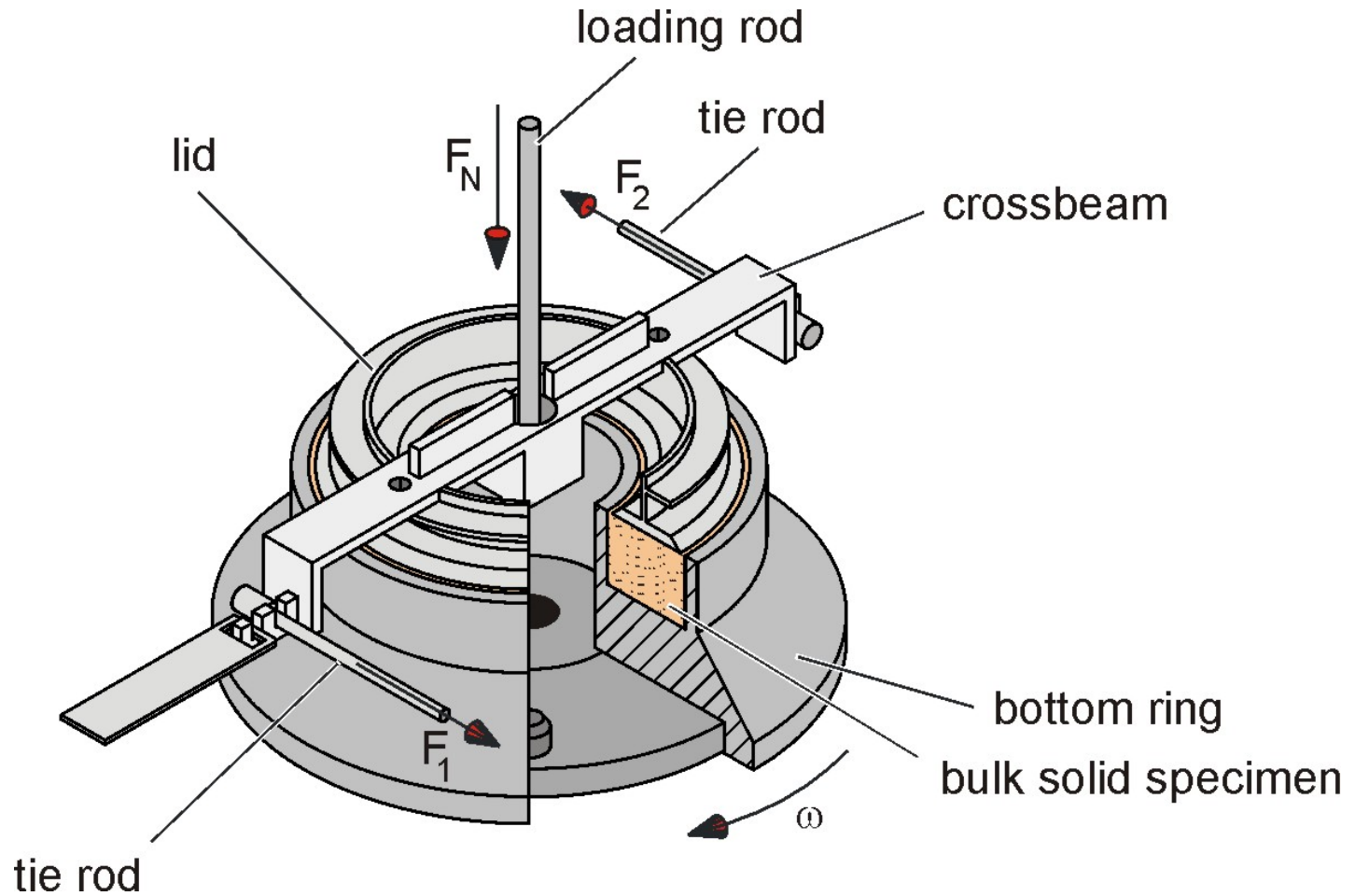
Computer-controlled Ring Shear Tester RST-XS

- Automatic Ring Shear Tester
- Shear cells with approx. 3.5 cm^3 to 70 cm^3 specimen volume

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4. Examples

Example: Results of a recent Round Robin project (I)

Testers used:
Ring Shear Testers RST-XS (30 cm^3 specimen volume) and
RST-01.pc (900 cm^3)

Powder: Standard limestone powder CRM-116 (available from BCR)

Results were compared with "certified" results of Jenike shear testers (BCR).

Test results have been supplied by following companies and institutions (in alphabetical order):
Amgen (USA), AstraZeneca (UK), BASF (D), Boehringer Ingelheim (D), Clariant (D), DSM (NL), Dupont (USA), F. Hoffmann - La Roche (CH), Glasgow Caledonian University (UK), GlaxoSmithKline (2xUK), Jenike & Johanson (USA), Kerneos (F), L'Oreal (F), Nestlé (D), Novartis (CH), Nycomed (D), Pfizer (USA, UK), Sanofi (D), Schwedes + Schulze Schüttguttechnik (D), Technical University Munich (D), Unilever (D), University of Applied Sciences Upper Austria (A), University of Birmingham (UK), Wyeth Healthcare (UK).

Example: Results of a recent Round Robin project (II)
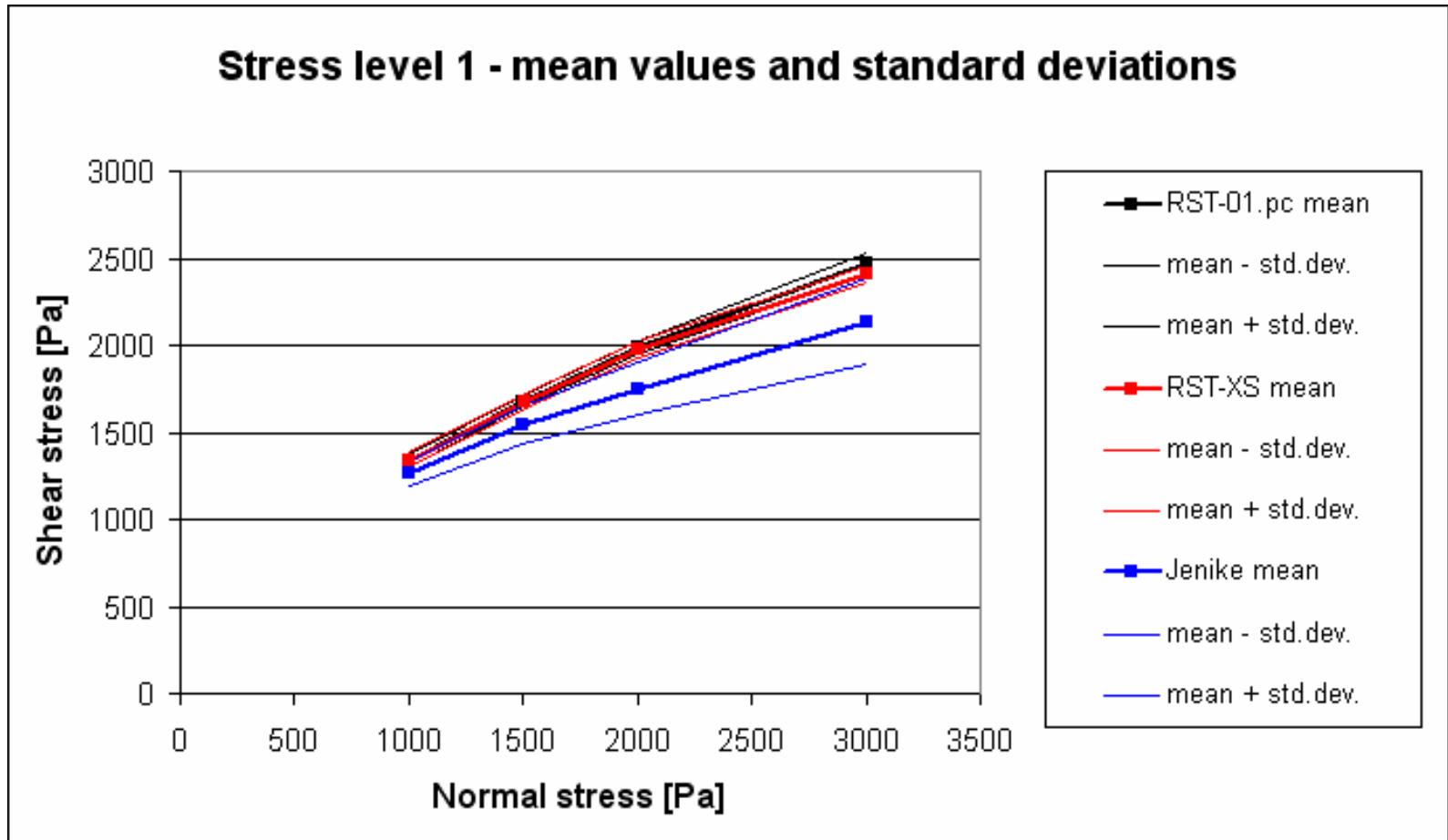
Yield locus test on limestone powder CRM-116

Example: Flowability in dependence on fines concentration


YIELD LOCUS OF:	CONCENTRATION:	SIGMA1 [Pa]	FC [Pa]	FFC [-]	RHOB [kg/m3]	PHIE [°]	PHILIN [°]	PHISF [°]
Mixture A/B	50% B	4288	1976	2.17	681	50	38	42
Mixture A/B	45% B	4158	1893	2.20	694	50	37	41
Mixture A/B	40% B	4261	1840	2.32	703	49	37	41
Mixture A/B	35% B	4232	1693	2.50	709	48	37	41
Mixture A/B	30% B	4210	1541	2.73	717	47	37	41
Mixture A/B	25% B	4208	1352	3.1	715	46	37	40
Mixture A/B	20% B	4090	1229	3.3	727	44	36	39
Mixture A/B	15% B	4015	1082	3.7	727	43	36	38
Mixture A/B	10% B	3991	914	4.4	743	41	35	37
Mixture A/B	5% B	3948	767	5.1	739	39	34	36

RSV 95 (c) Dietmar Schulze 2002

A, B: lactose; B is finer than A.

Measured with ring shear tester, shear cell 30 cm^3

# Example: Wall friction - material and moisture
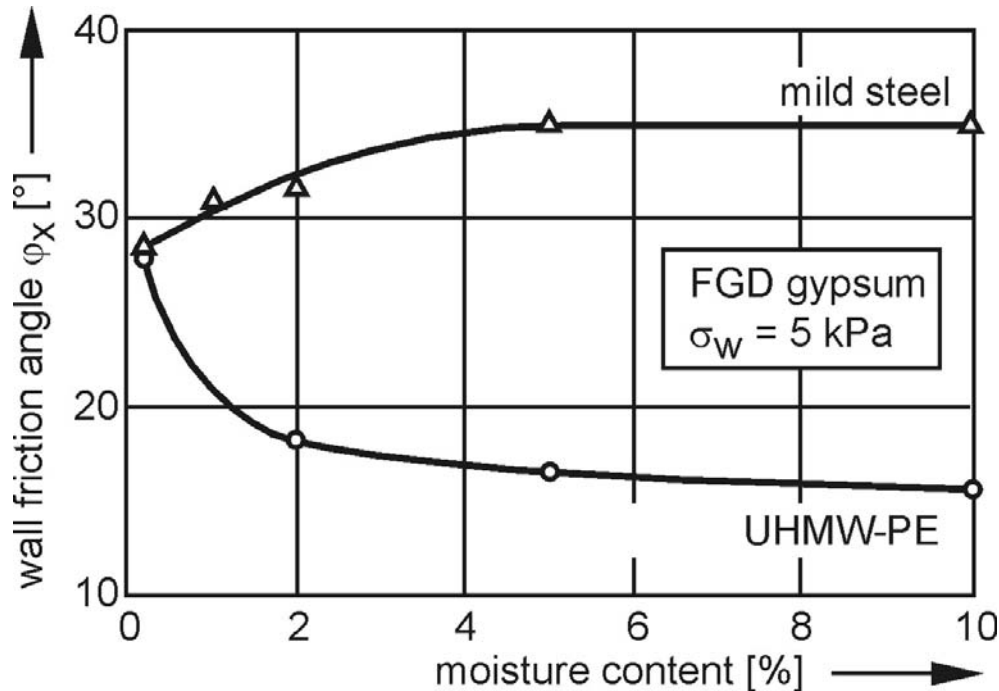
- Mild steel:
  Increase of wall friction angle with content moisture
  (high wettability; increasing adhesion due to liquid bridges)

- UHMW-PE:
  Wall friction decreases with increasing moisture content
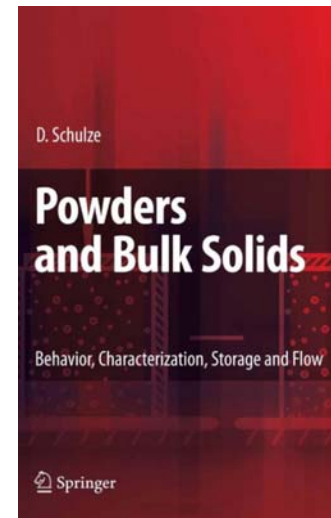 (low wettability).

Wall friction angles of Flue Gas Desulphurization (FGD) Gypsum

Example: Flow agent concentration

- Crystalline product with varied flow agent concentration.
- Diagram shows instantaneous flowability and flowability after 22 h storage at rest
- Best flowability is measured for concentration 0.55%.
- Decrease of flowability for concentrations > 0.55%.
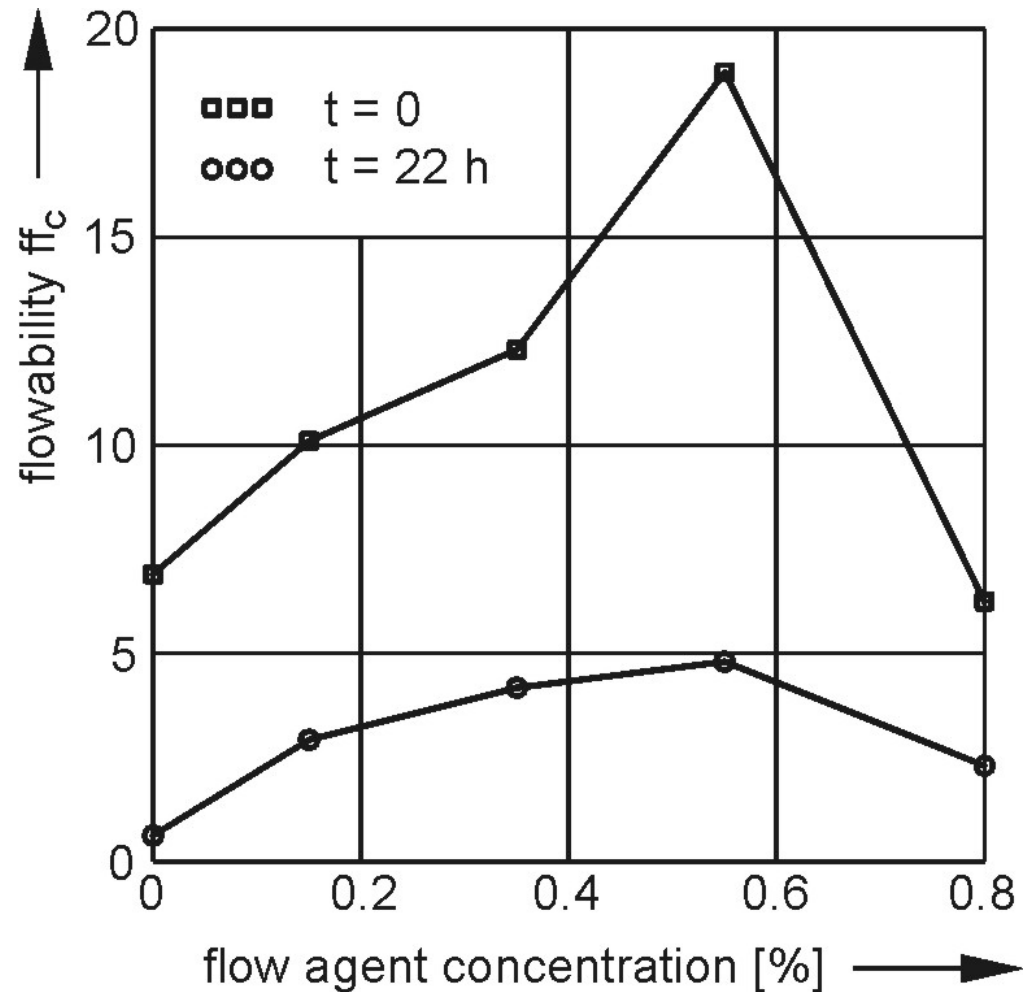
Thank you for your kind attention!