COMPRESSION BEHAVIOR OF DRY, MOIST AND WET ELASTO-PLASTIC GRANULES

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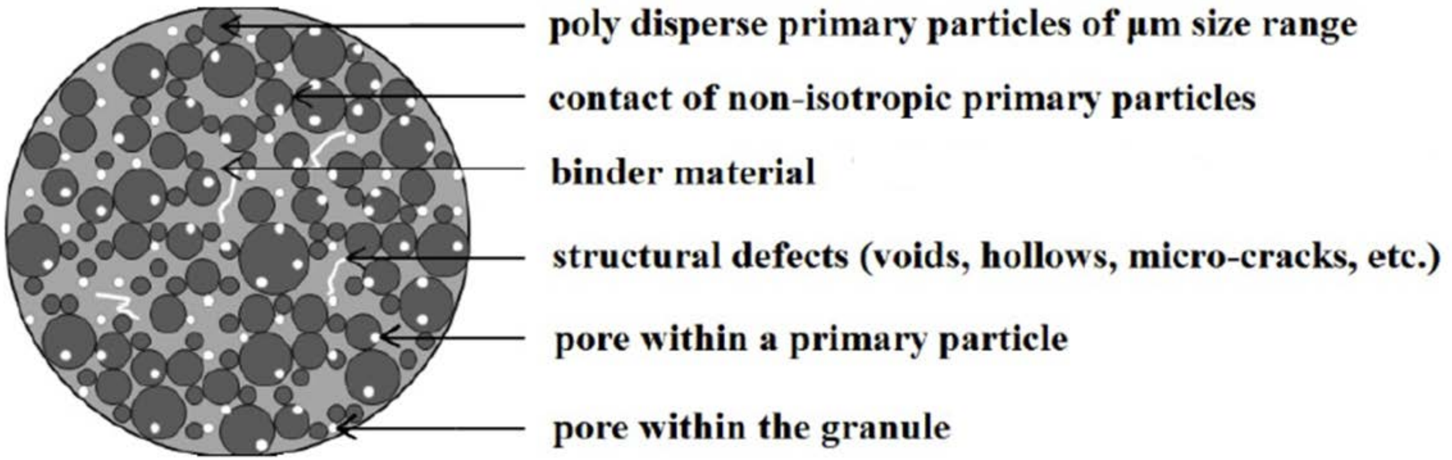
Handling of Granular Material

- fine inhomogeneous agglomerates (d > 100µm)
- consisting of discrete solid primary particles (10 µm < d < 100 µm)
- bonded by adhesive forces, liquid or solid bridges



Advantages:

- improved flowability
- reduced dust formation and segregation
- higher bulk density

Disadvantages:

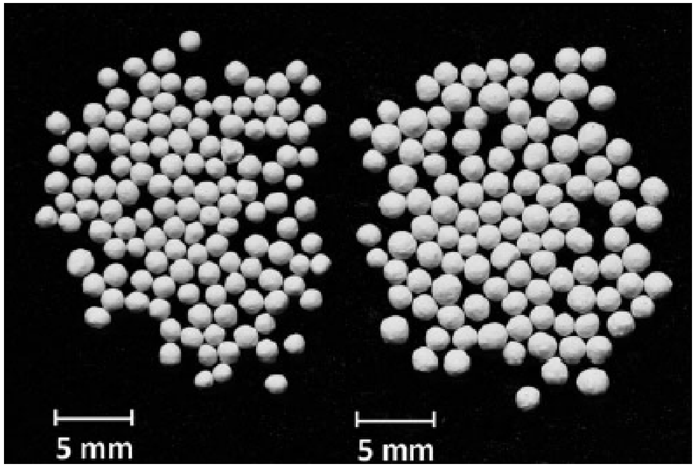
- inhomogeneous
- anisotropy
- random shape and size distributions

Source: Russell, A., Müller, P., Tomas, J., Chem. Eng. Sci. 114, 70–84 (2014)

Test Material – Zeolite 4AK

- water-insoluble, arguably spherical and highly hygroscopic granules
- regular pore size with a defined pore diameter of 4 Å (0.4 nm)
- 83% primary particles: $Na_2O \cdot Al_2O_3 \cdot 2SiO_2 \cdot nH_2O$
- 17% binder material: $(Mg,Al)_2 \cdot Si_4O_{10} \cdot (OH) \cdot 4(H_2O)$
- mechanical behavior: elasto-plastic deformation until brittle breakage
- adsorber for water and organic liquids

granule size d_{50} in mm	1.75	3.05
sphericity Ψ	0.98	0.98
granule density ρ_g in kg/m^3	1127.9	1102.0
solid density ρ_s in kg/m^3	2290.7	2374.7
porosity ε in %	50.8	53.6
specific surface area S_m in m^2/kg	38.13	44.25



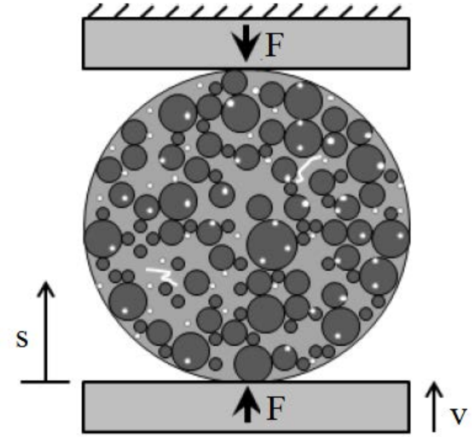
Source: Russell, A., Müller, P., Tomas, J., Chem. Eng. and Technol. 7, 1240–1248 (2013)

Test Method

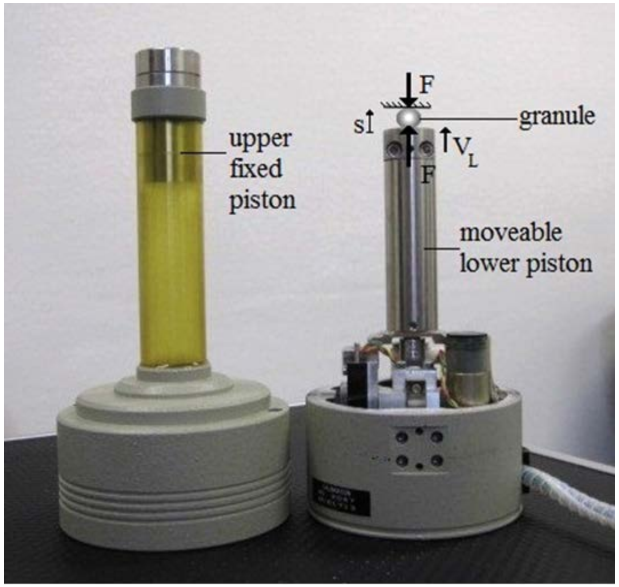
- uniaxial stressing between two flat and rigid contacts
- two test methods:

  - monotonic loading until primary breakage

  - cyclic loading (until primary breakage) with a load equal to 90% of the mean breakage force of fresh granules

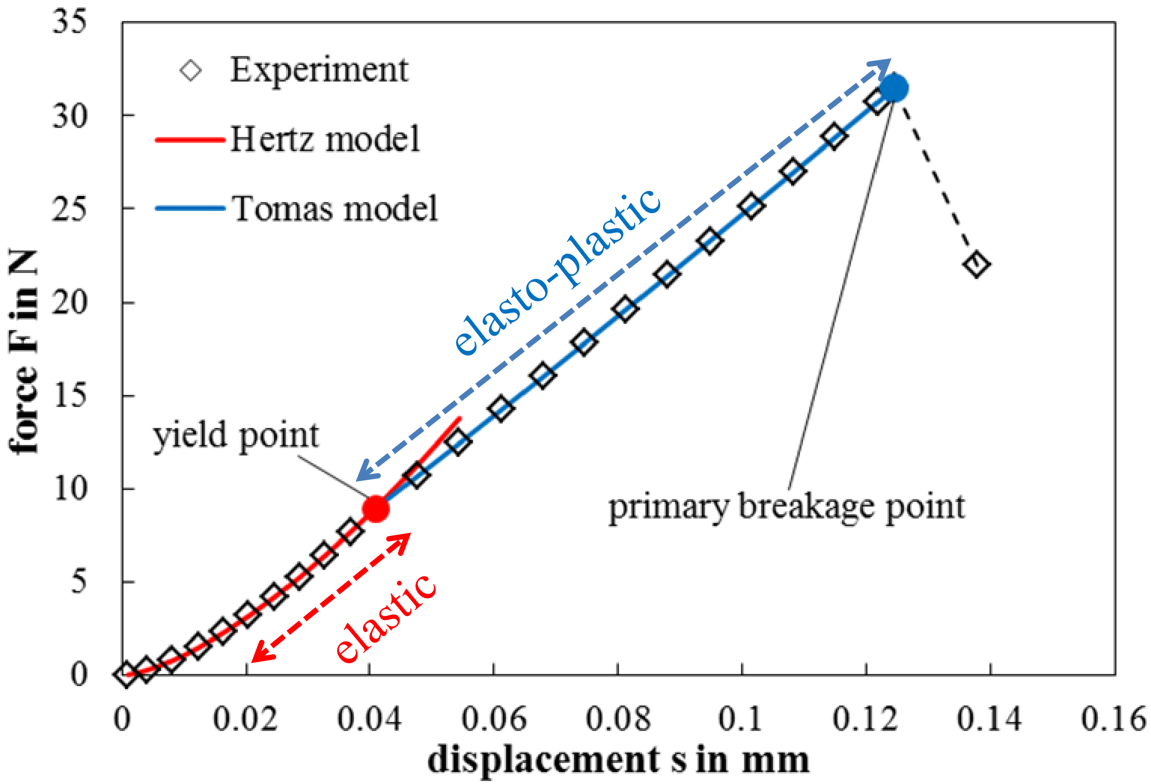


Granule size d_{50} in mm	1.75			3.05		
Loading velocity v in mm/min	0.2	1.5	2.0	0.2	1.5	2.0
Moisture content X_W in kg_W/kg_{DS}	0.00 0.16 0.45			0.00 0.14 0.49		



Source: Russell, A., Müller, P., Tomas, J., Chem. Eng. Sci. 114, 70–84 (2014)

Force-Displacement Behavior

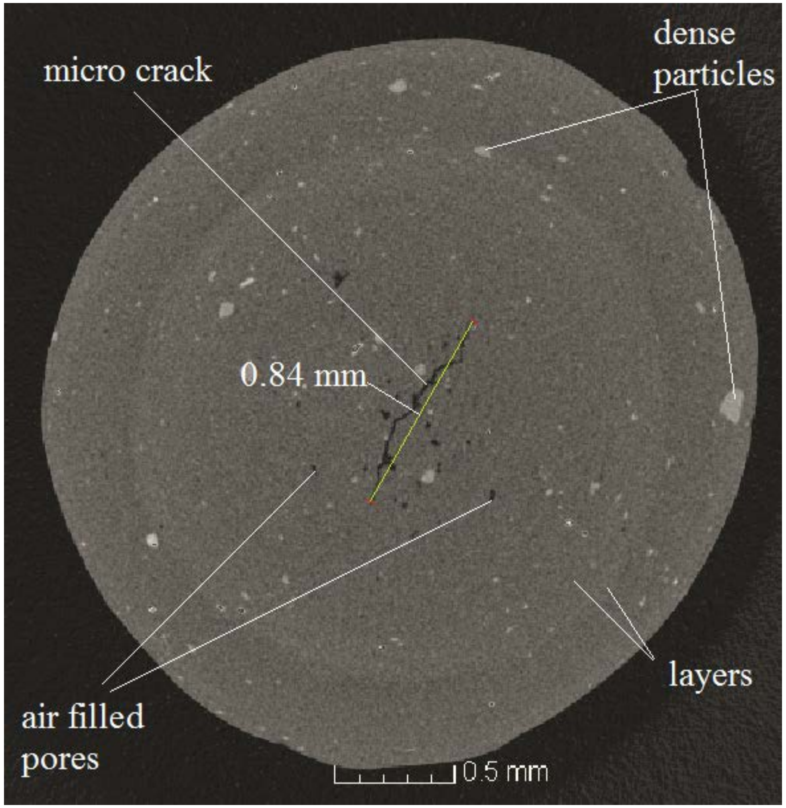


Hertz Model[1]	Tomas Model[2]
$F_{el,w-g-w} = \frac{1}{6}E^{*}\sqrt{d_1 s^3} = \frac{1}{3}\frac{1}{\left(1-\nu_1^2\right)}E_1\sqrt{d_1 s^3}$	$F_{el-pl,w-g-w} = \pi\lambda_{el-pl}R_1 p_y\left(1-\frac{1}{3}\sqrt[3]{\frac{s_y}{s}}\right)\frac{s}{2}$

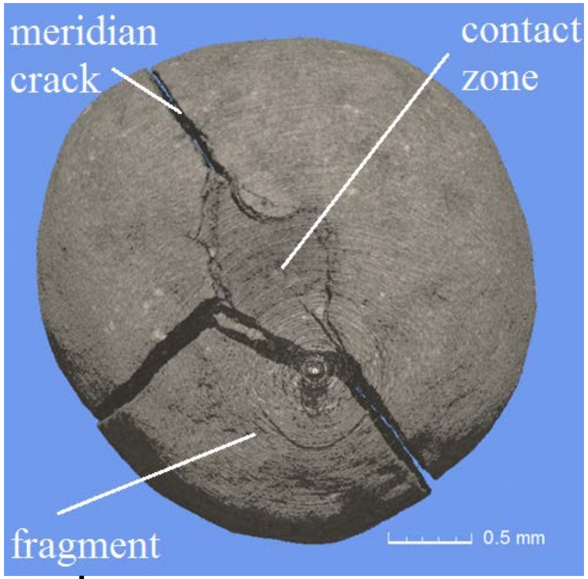
1: Hertz, H., Journal für die reine und angewandte Mathematik 92, 156–171 (1882)
2: Tomas, J., Chem. Eng. Sci. 62, 1997–2010 (2007)

Image Analysis

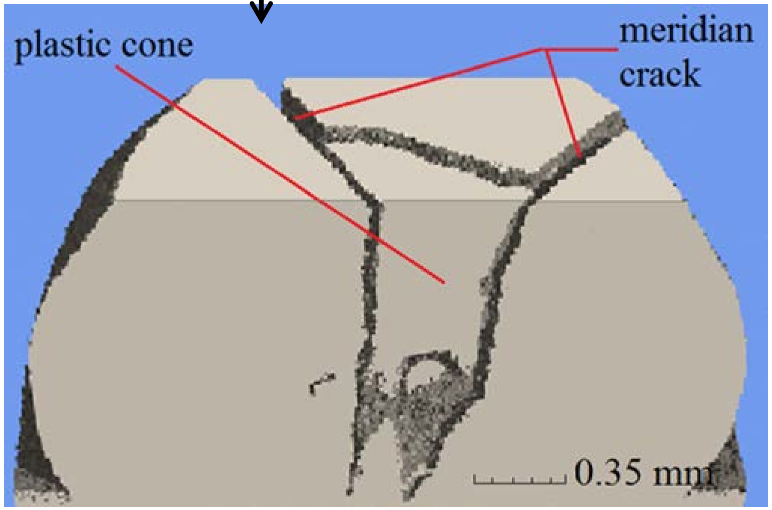
fresh and dry granule:



monotonic loading until primary breakage

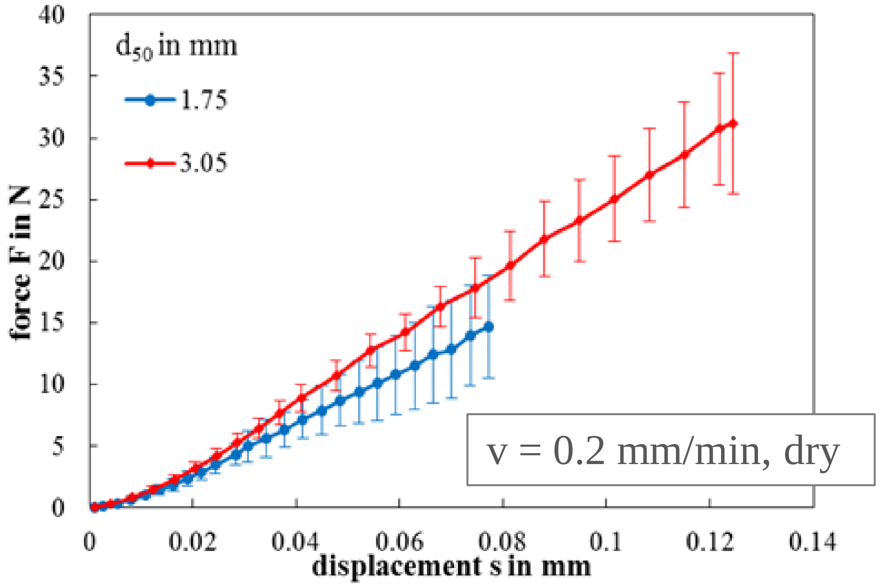


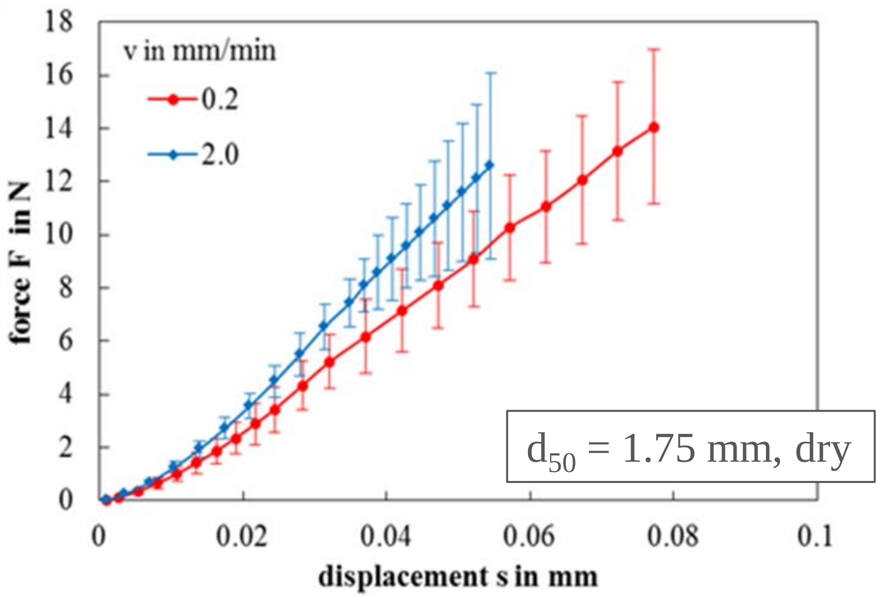
cross section



- micro cracks
- air filled pores
- layers
- unknown dense particles

Material Behavior of Fresh Granules





With increasing granule size:

- increase of k_{el} due to the Hertz law

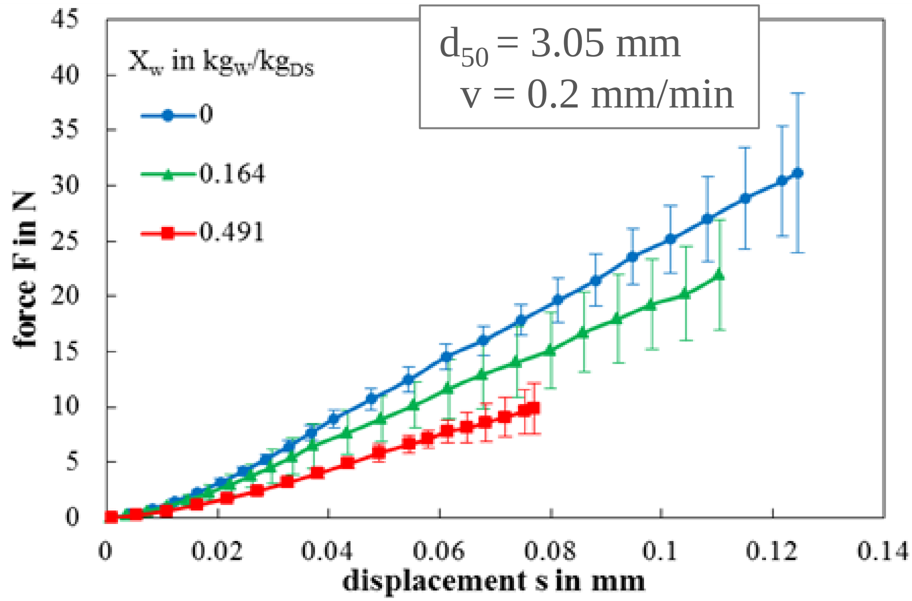
$$k_{el,w-g-w} = \frac{1}{2}\frac{E_1}{\left(1-v_1^2\right)}\sqrt{d_1 s}$$

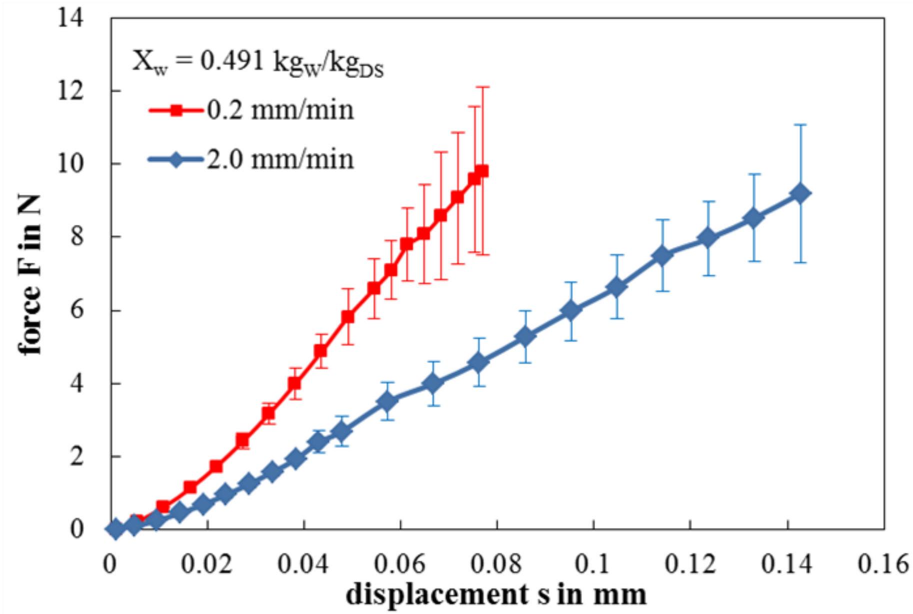
- decrease of E and p_y due to the higher influence of inhomogeneities

With increasing loading velocity:

- increase of k_{el}, E and p_y

Material Behavior of Fresh Granules





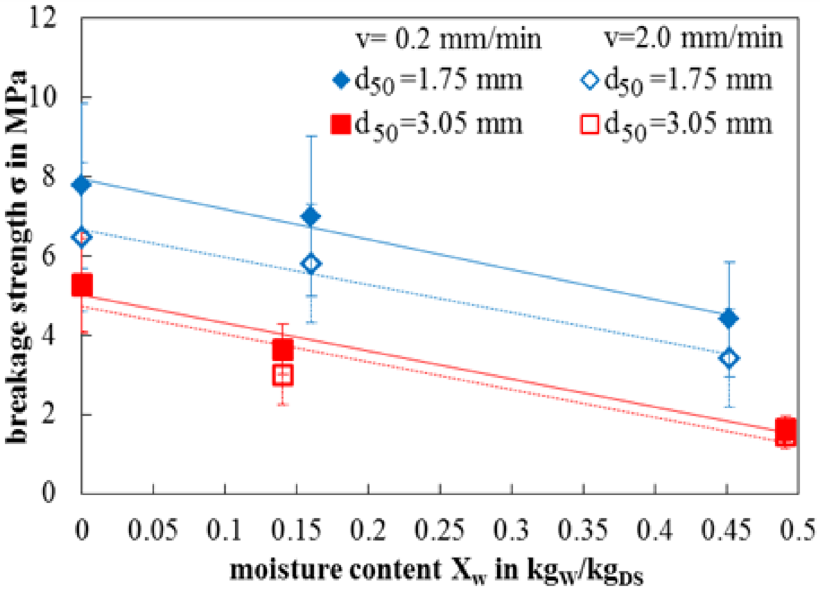
With increasing moisture content :

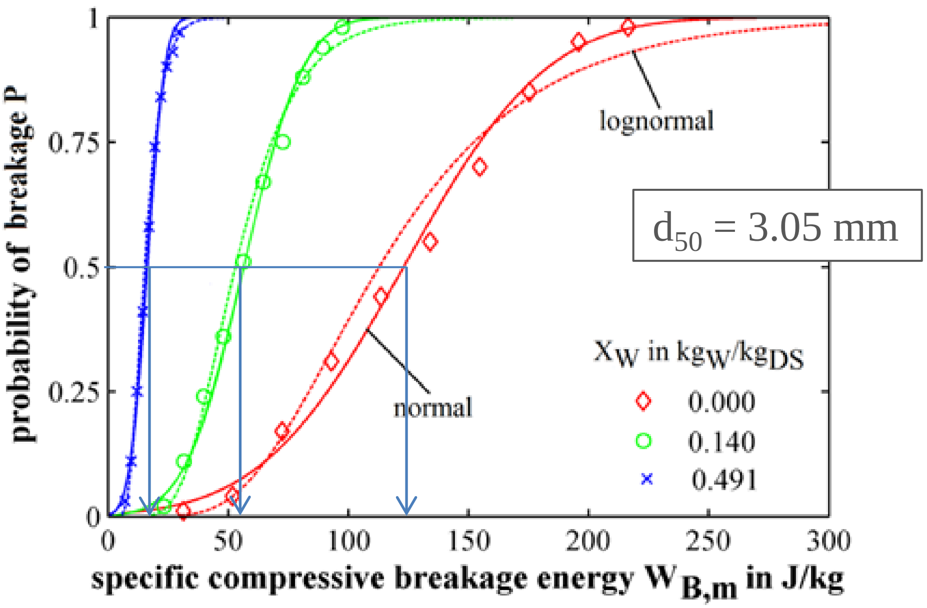
- significant decrease of k_{el}, E and p_y

Influence of loading velocity and moisture content:

- significant decrease of k_{el}, E and p_y with increasing loading velocity for moistened granules

Breakage Characteristics of Fresh Granules





$$\sigma = \frac{F_B}{A_g} = \frac{4F_B}{\pi d^2}$$

Decrease of strength σ with:

- increasing granule size
- increasing loading velocity
- increasing moisture content

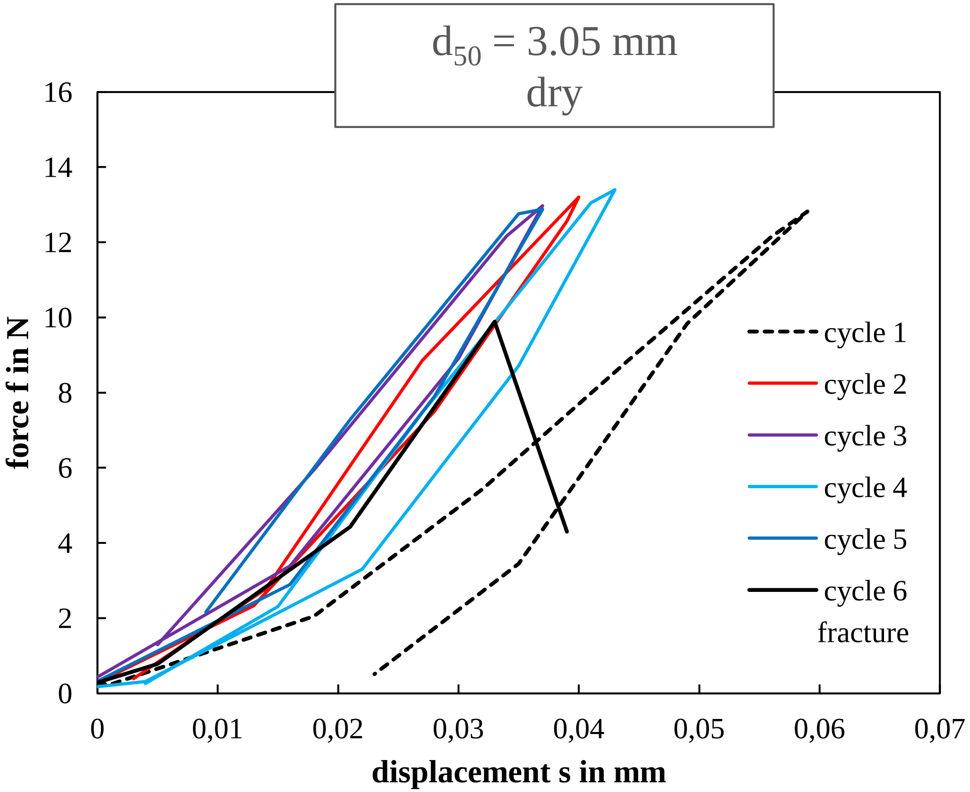
$$W_{B.m} = \frac{W_B}{m_g} = \frac{1}{m_g}\int_0^{s_B} F ds$$

d_{50} = 3.05 mm	$W_{B,m,50}$
dry	130 J/kg
moist	58 J/kg
wet	22 J/kg

Cyclic Loading - Hardening Effect

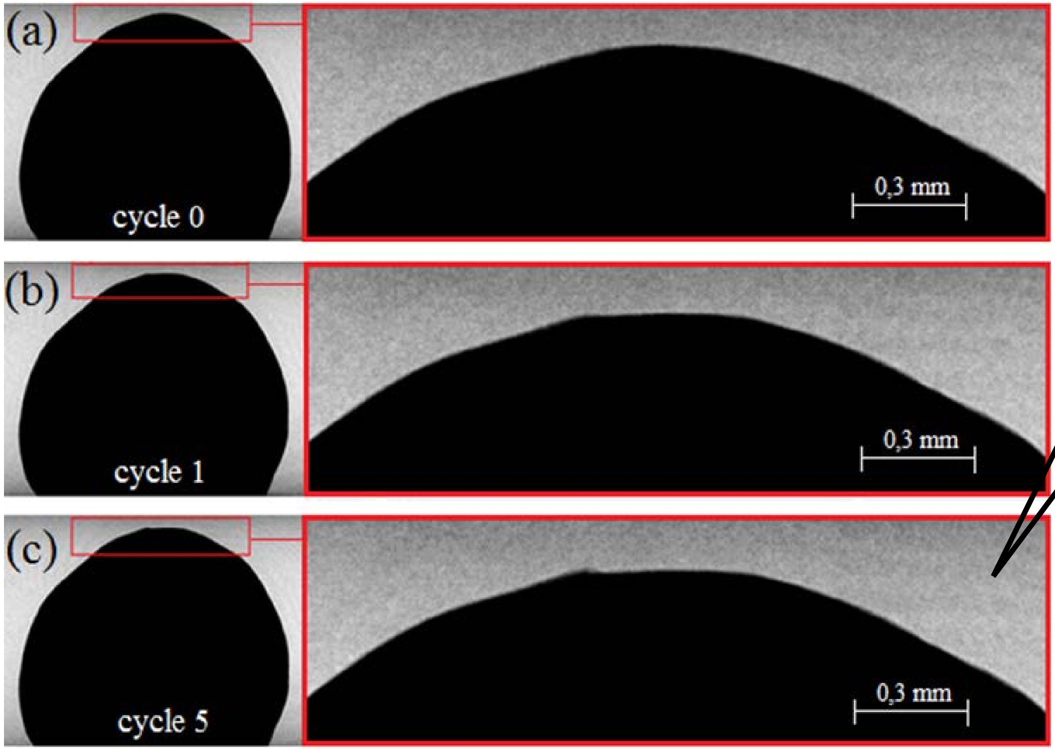
With increasing number of cycles:

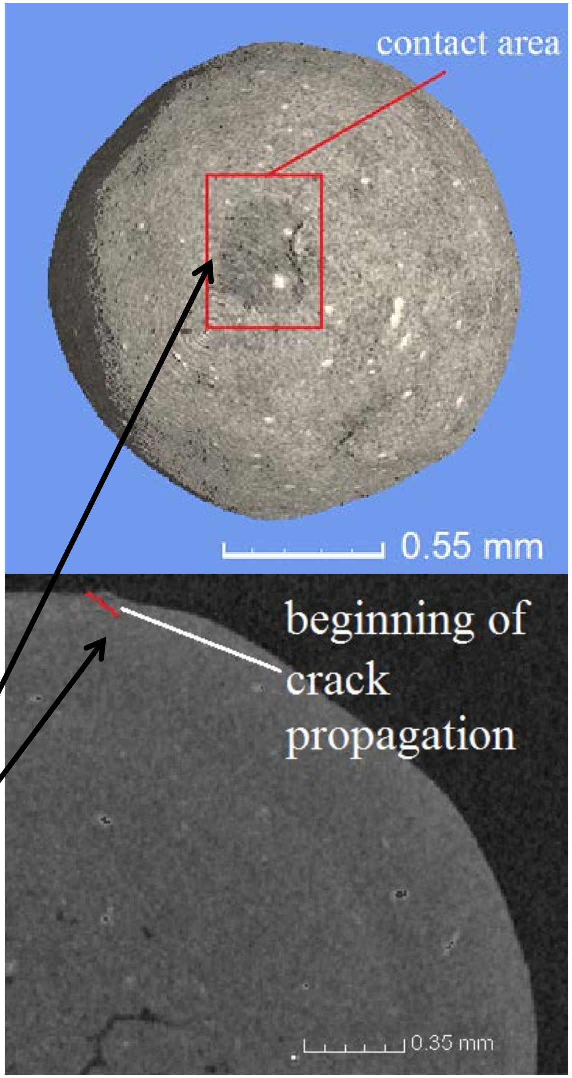
- decrease of plastic strain
- cyclic hardening effect:
  - increase of stiffness
  - significantly during first and second cycle
  - changes in microstructure promote breakage



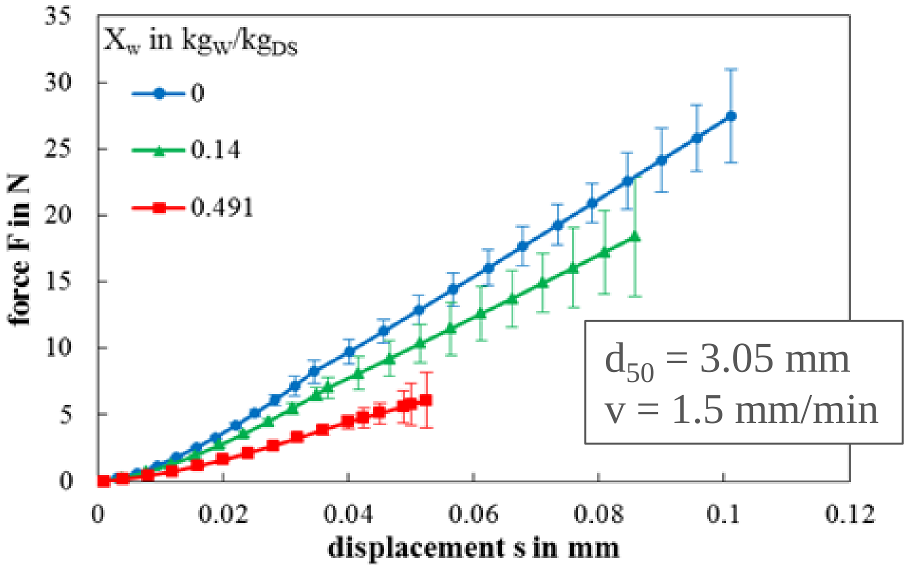
Contact Flattening during Cyclic Loading

- dominant flattening of the curvature with increasing number of cycles
- visible denser elasto-plastically deformed area at the contact zone



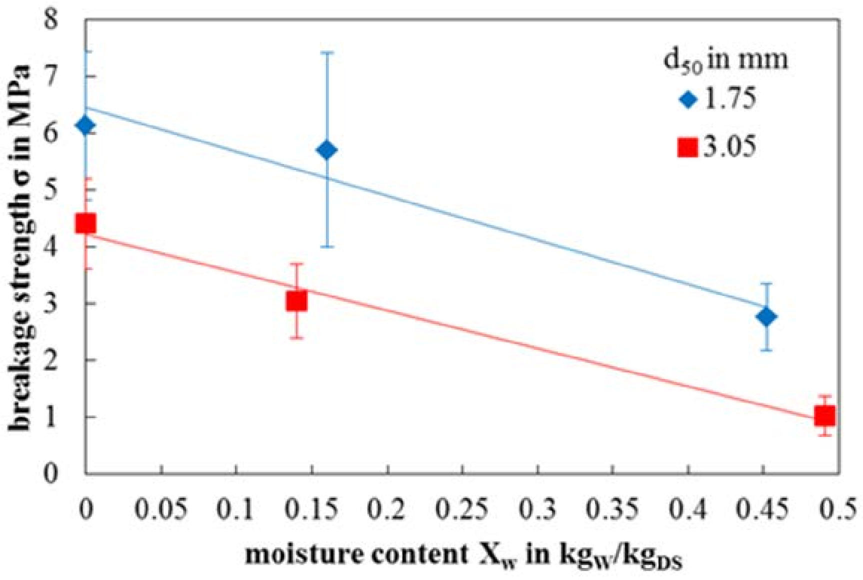


Material and Breakage Behavior of Preloaded Granules



With increasing moisture content:

- decrease of k_{el}, E and p_y
- decrease in displacement at breakage



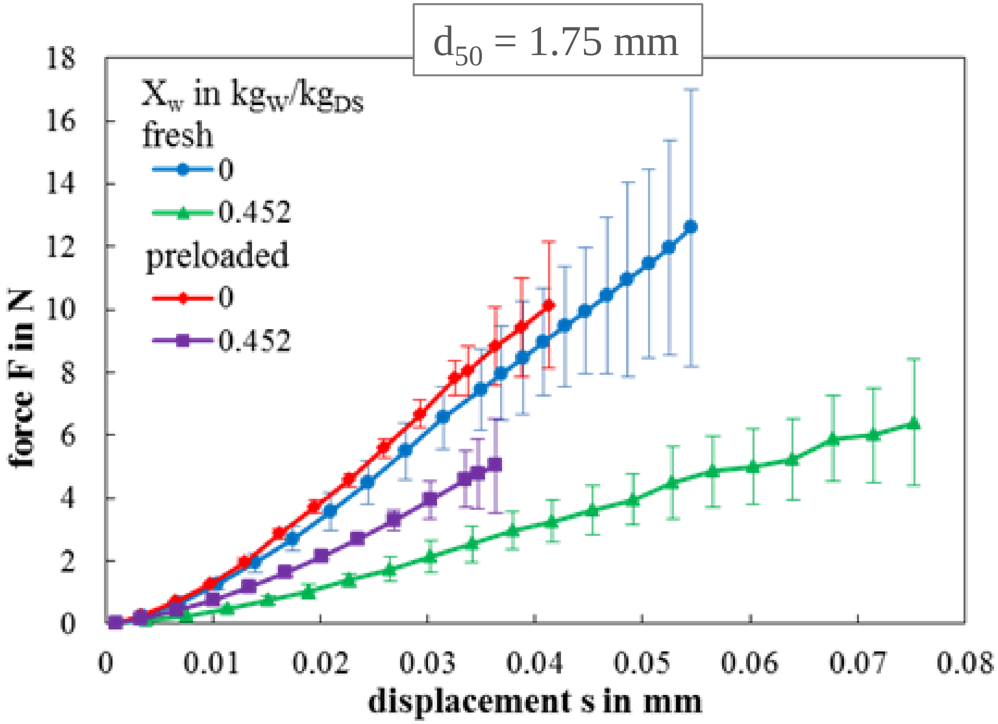
Breakage characteristics of preloaded granules:

Deacrease of strength σ with :

- increasing granule size
- increasing moisture content

→ due to the damage of the inner granular structure

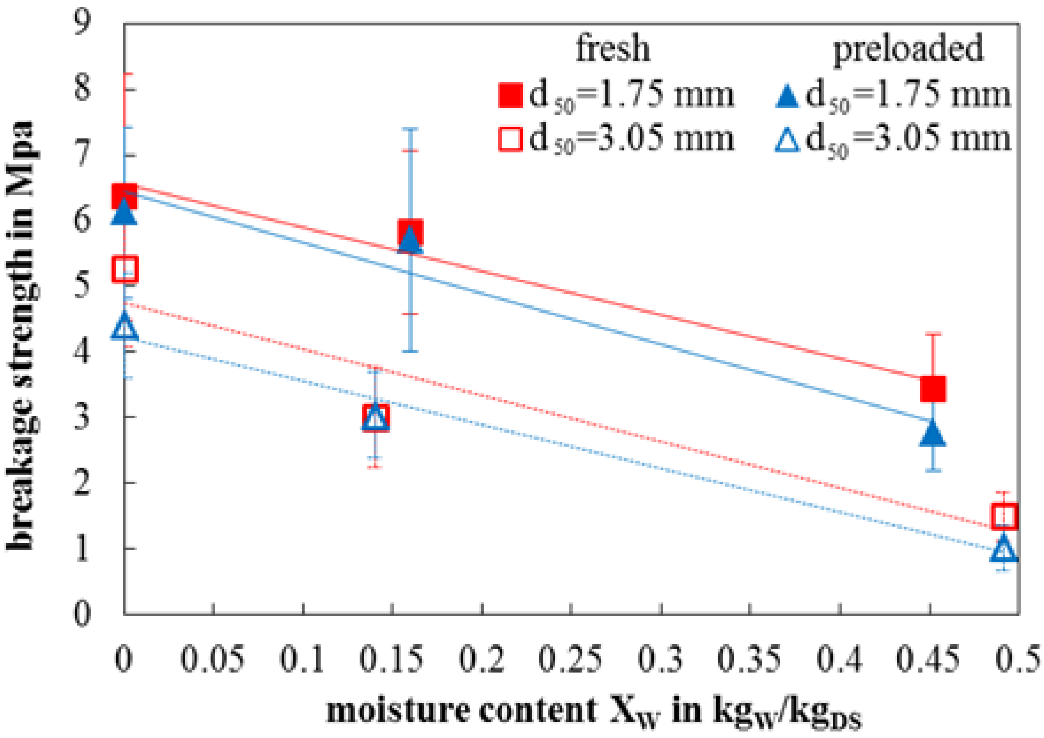
Comparison of the Material Behavior of Fresh and Preloaded Granules



Dry and completely wetted granules:

- increase of k_{el}, E and p_y with loading history (cyclic hardening effect)
- decrease in displacement at breakage, intensified with increasing moisture content

Comparison of Breakage Characteristics of Fresh and Preloaded Granuels



- decrease of breakage strength with loading history

Summary

State of the art before this thesis:

Influence of:

- granule size[1] larger ⟶ higher stiffness and lower strength
- loading velocity[2] faster ⟶ higher stiffness and lower strength
- moisture content[3] wetter ⟶ lower stiffness and lower strength

What we didn`t know:

- complex influence of loading velocity and moisture content
- influence of preloading
- crack propagation

1: Antonyuk, S.,Ph.D. Thesis, Otto von Guericke University of Magdeburg (2006)
2: Russell, A., Müller, P., Shi, H., Tomas, J., AIChE Journal (2014)
3: Müller, P., Ph.D. Thesis, Otto von Guericke University of Magdeburg (2011)

Summary

State of the art now:

- influence of loading velocity and moisture content

  → with increasing loading velocity and moisture content:

  lower stiffness and lower strength

- influence of preloading

  → higher stiffness

  → lower strength

- crack propagation according to Tomas[1,2]

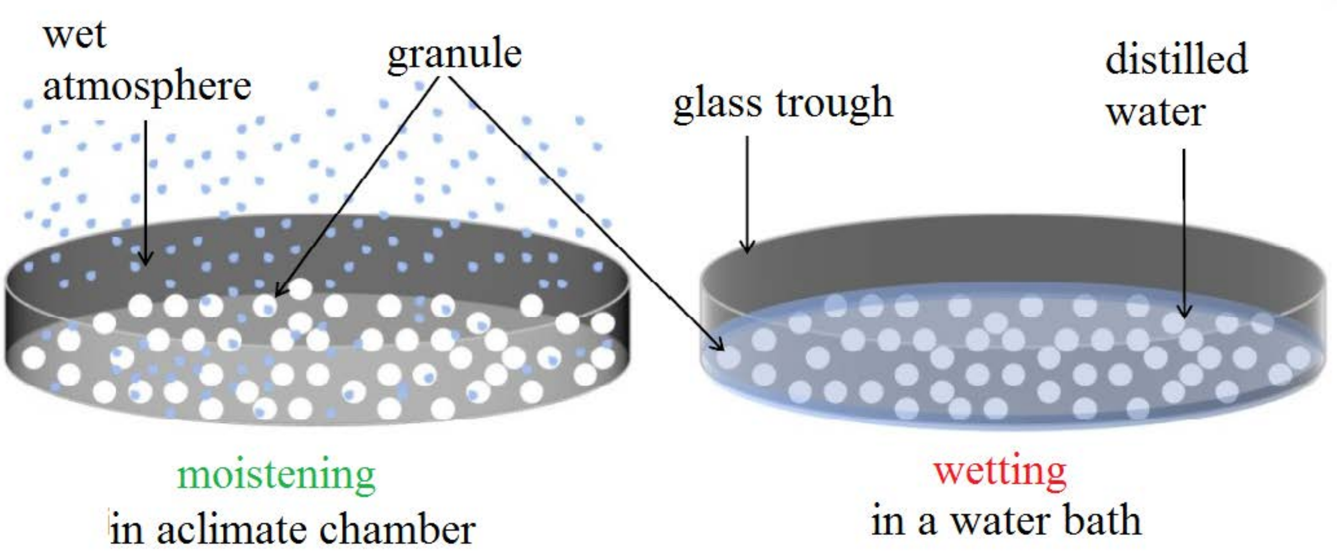
Future:

- creep tests
- influence of temperature

1: Tomas, J., Schreier, M., Gröger, T., Ehlers, S., Powder Technology 105, 39–51 (1999)
2: Tomas, J., Vorlesungsmanuskript, www.mvt.ovgu.de

Appendix

Zeolite 4AK



$$X_W = \frac{m_W}{m_{DS}} = \frac{m_{tot} - m_{DS}}{m_{DS}}$$

$$S = \frac{V_l}{V_{pore}} = \frac{(1-\varepsilon)V_l}{\varepsilon V_s} = \frac{(1-\varepsilon)\rho_s}{\varepsilon \rho_l} X_w$$

Source: Russell, A., WCPT7 2014 – 7th World Congress on Particle Technology

Micromechanical Model

The force-displacement curve was well-fitted by:

- using the Hertz model1 for non-linear non-adhesive elastic loading

$$F_{el,w-g-w} = \frac{1}{6}E^{*}\sqrt{d_1 s^3} = \frac{1}{3}\frac{1}{\left(1-\nu_1^2\right)}E_1\sqrt{d_1 s^3}$$

$$k_{el,w-g-w} = \frac{dF_{el,w-g-w}}{ds} = \frac{1}{4}E^{*}\sqrt{d_1 s} = \frac{1}{2}\frac{E_1}{\left(1-\nu_1^2\right)}\sqrt{d_1 s}$$

- using the Tomas model[2] for non-linear non-adhesive elasto-plastic loading

$$F_{el-pl,w-g-w} = \pi\lambda_{el-pl}R_1 p_y\left(1-\frac{1}{3}\sqrt[3]{\frac{s_y}{s}}\right)\frac{s}{2}$$

$$k_{el-pl,w-g-w} = \frac{dF_{el-pl,w-g-w}}{ds} = \pi\lambda_{el-pl}R_1 p_y\left(1-\frac{2}{9}\sqrt[3]{\frac{s_y}{s}}\right)$$

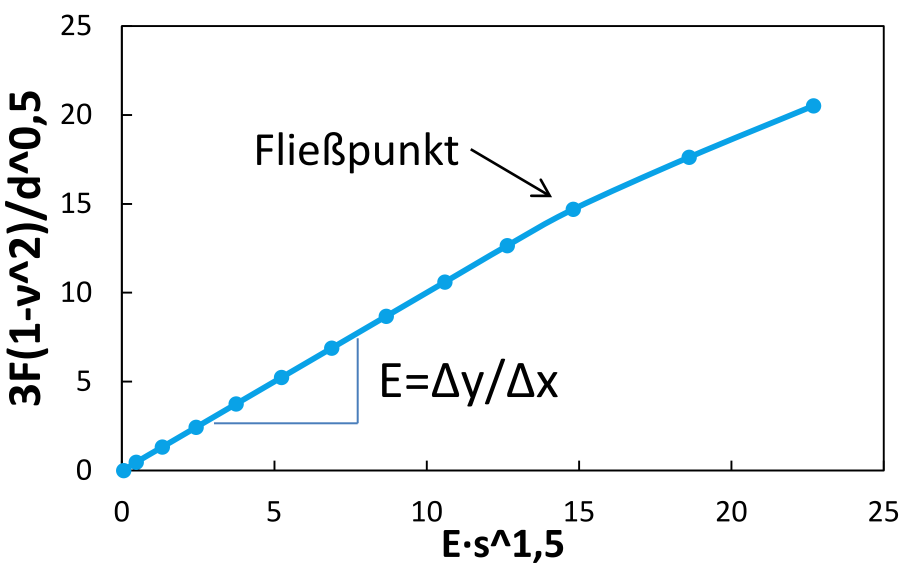
1: Hertz, H., Journal für die reine und angewandte Mathematik 92, 156–171 (1882)
2: Tomas, J., Chem. Eng. Sci. 62, 1997–2010 (2007)

E - Modul

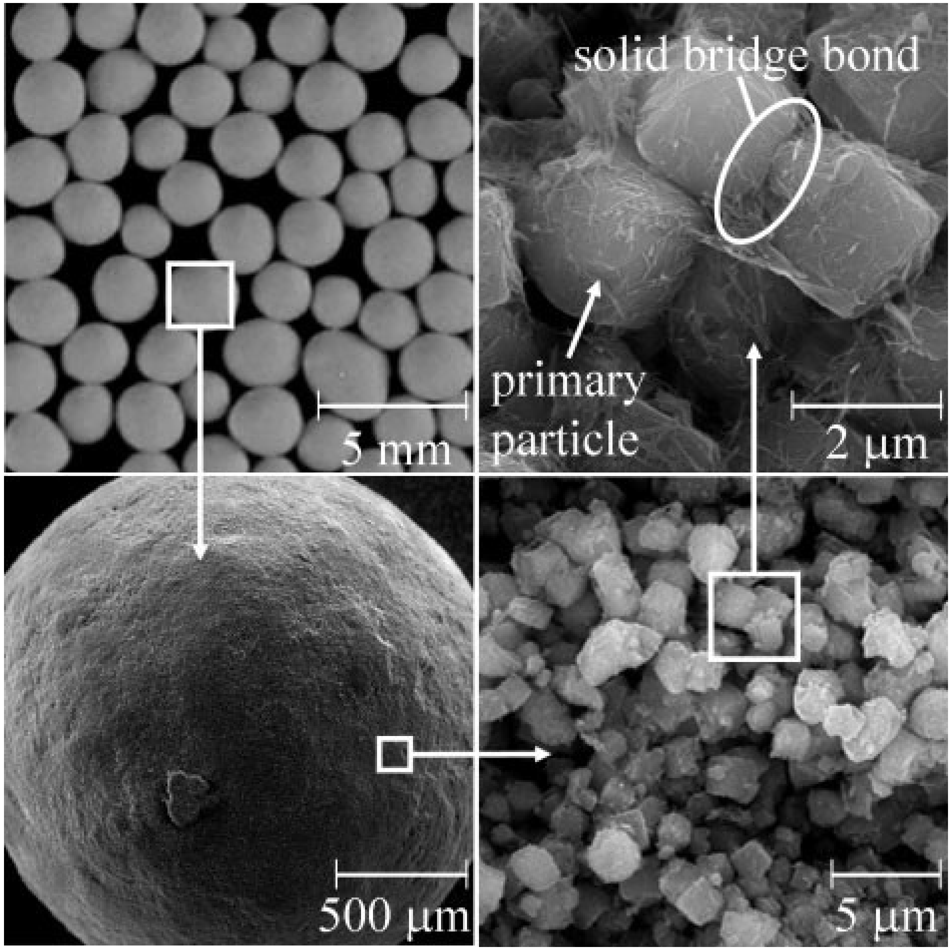
- Bestimmen des E – Modul durch Linearisierung des Hertz Modells

$$F_{el,w-g-w} = \frac{1}{3}\frac{1}{\left(1-\nu_1^2\right)} E_1 \sqrt{d_1 s^3} \xrightarrow{y = m \cdot x + n \nearrow 0} 3F_{el,w-g-w} \cdot \frac{\left(1-\nu_1^2\right)}{d^{\frac{1}{2}}} = E_1 \cdot s^{\frac{3}{2}}$$

m

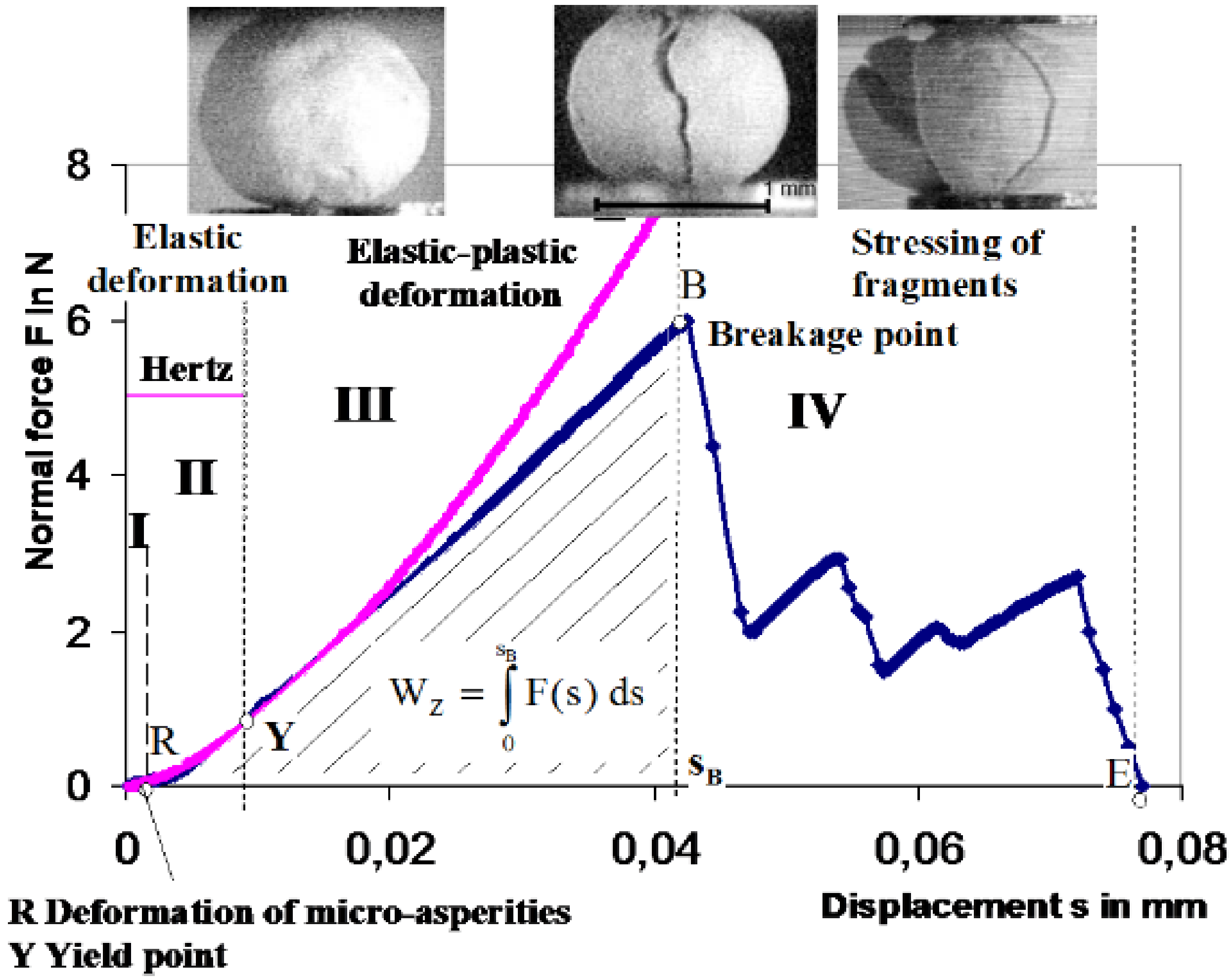


SEM Micrographs of the Granule Surface



Source: Müller, P., Tomas, J., Chem. Eng. Technol. 35, 1677–1684 (2012)

Crack Propagation



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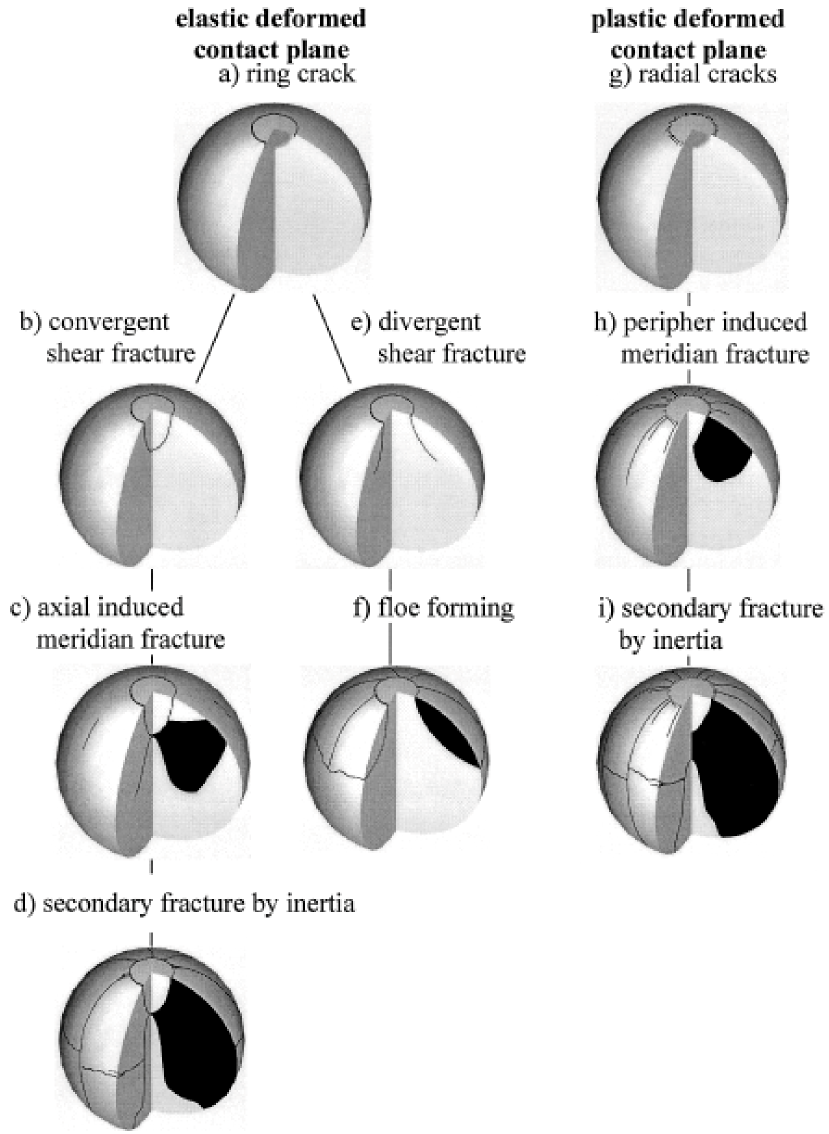
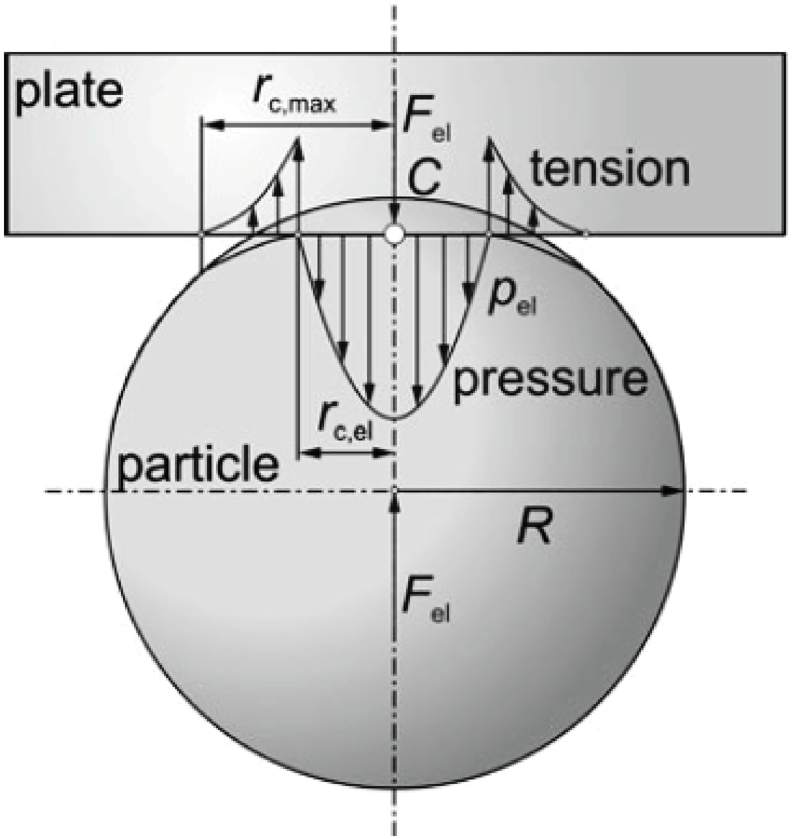
Tomas, J., Vorlessungsmanuskript, www.mvt.ovgu.de

Crack Propagation

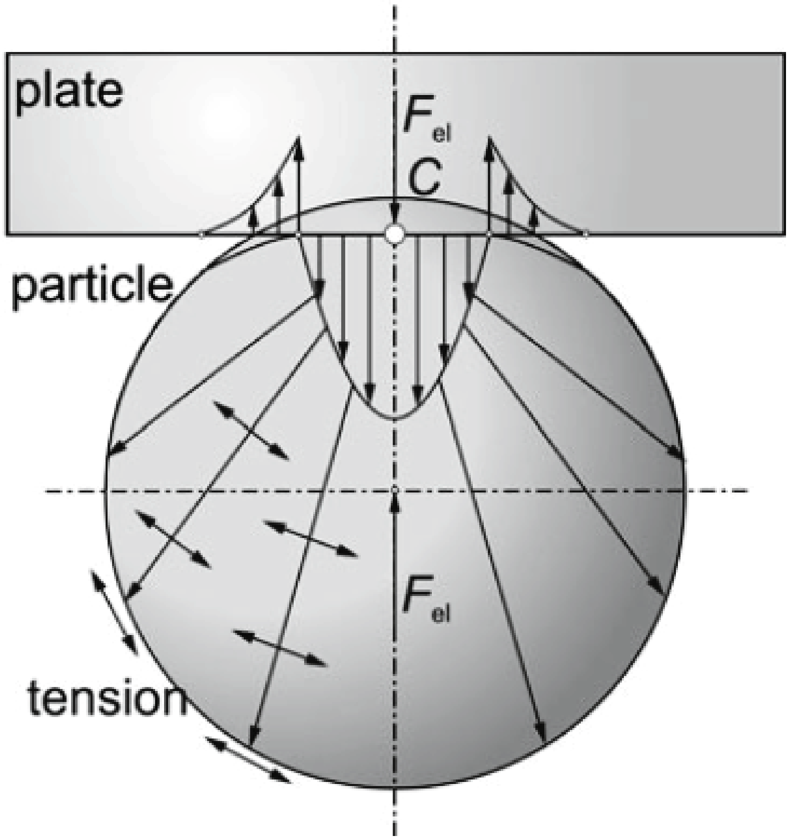


Source: J. Tomas, M. Schreier, T. Gröger and S. Ehlers, Powder Technol. 105, 39-51 (1999)

Comparison of the Stress State in Dry and Moist Granules



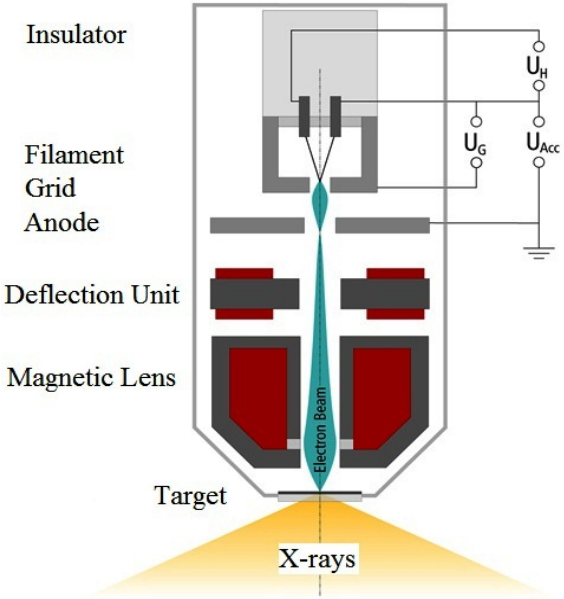
dry granule

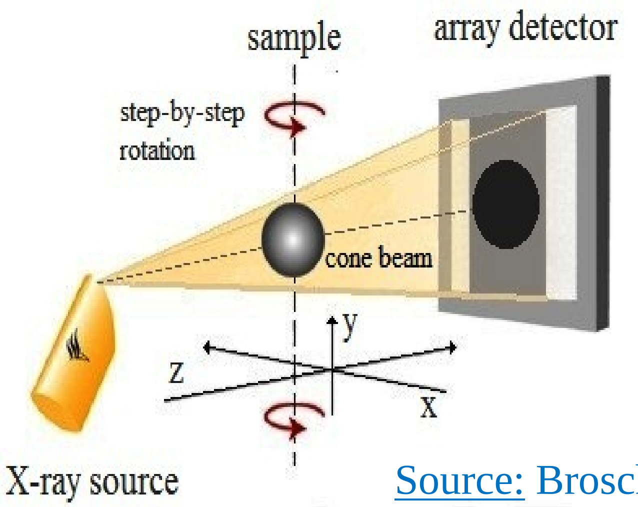


moist granule

Source: P. Müller, S. Aman, M. Stasiak and J. Tomas, Granul. Matter, Special Issue CHoPS (2013)

Micro CT and SEM





Source: Broschure for phoenix nanotom s, www.ge-mcs.com