UNIVERSITY OF TWENTE. FOM RUB

HYDRAULICAL & ACOUSTICAL PROPERTIES OF SINTERED GLASS BEADS

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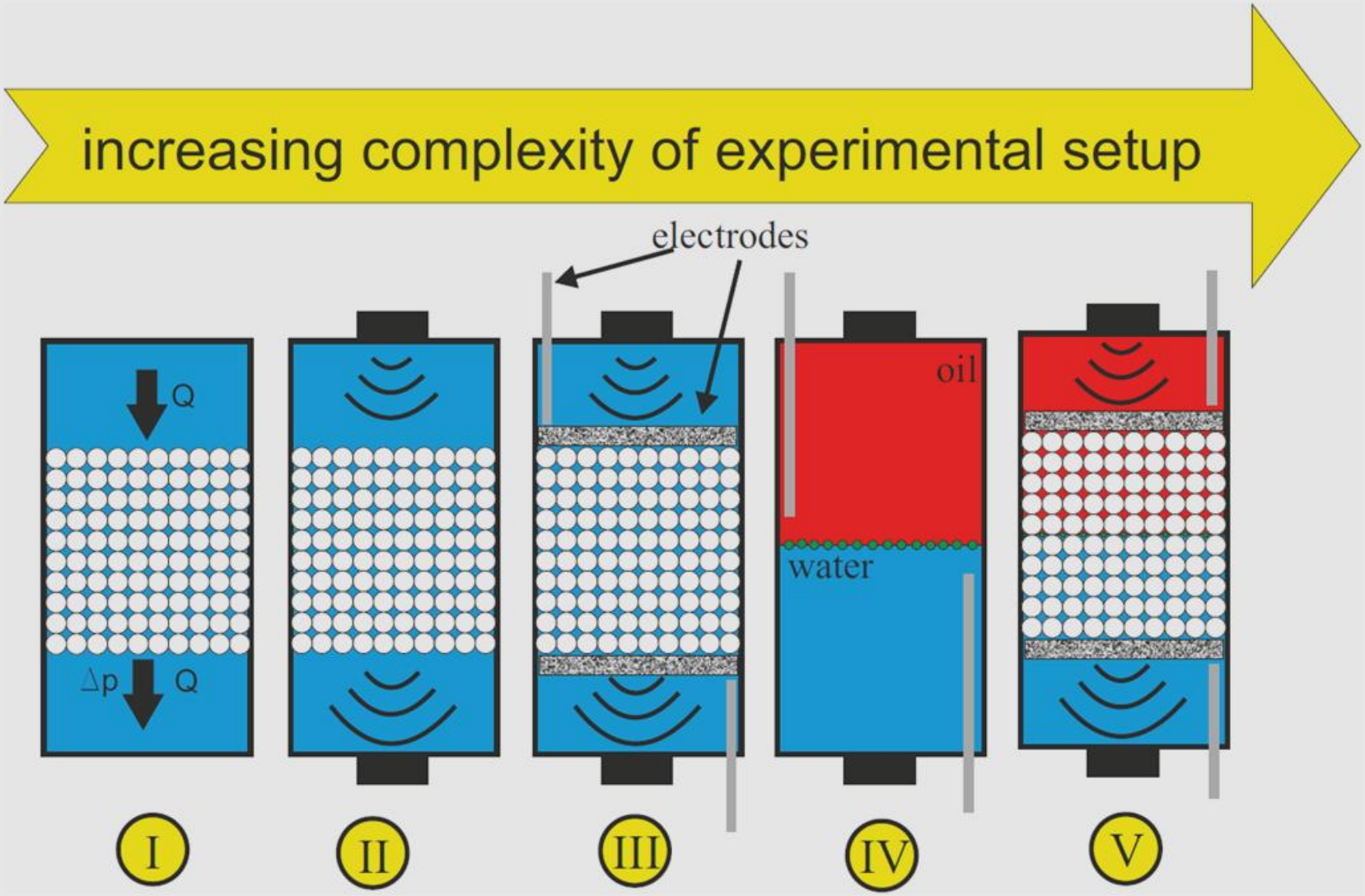
outline

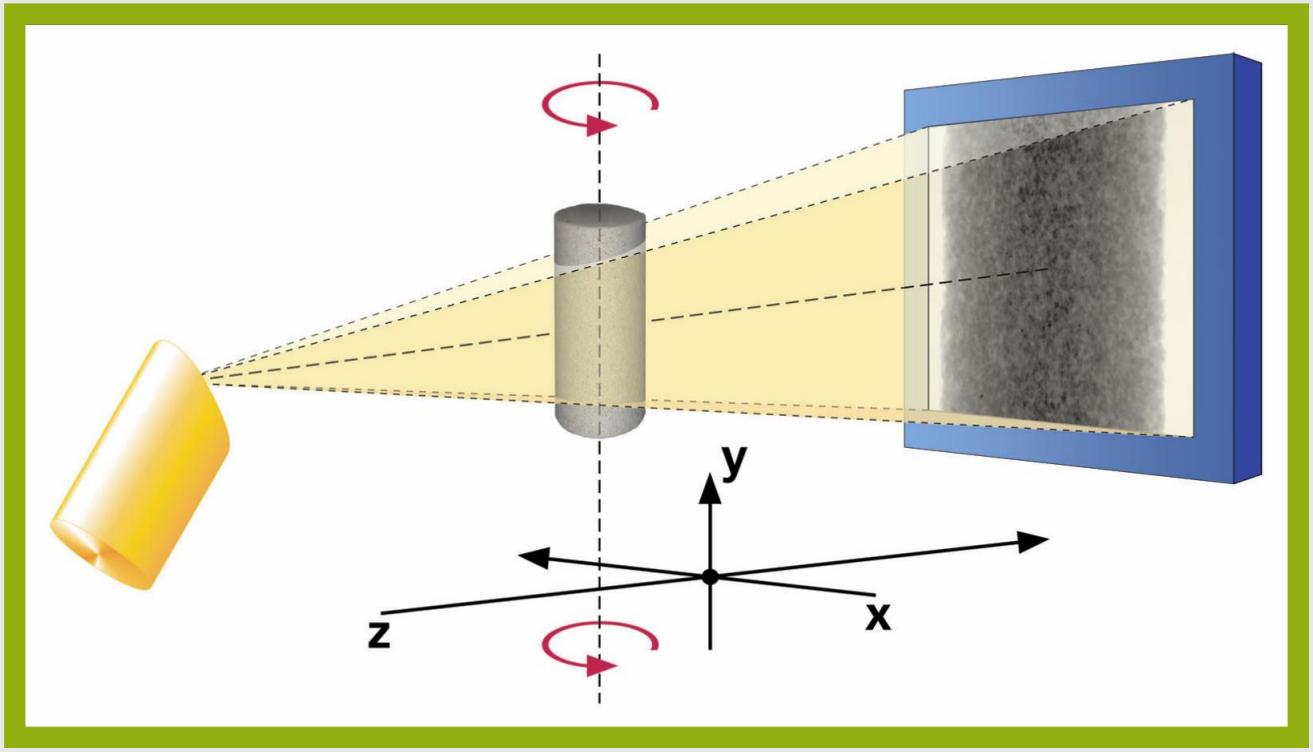
- overview
- sintering
- permeability experiments
- µCt data analysis
- ultrasound experiments
- conclusion

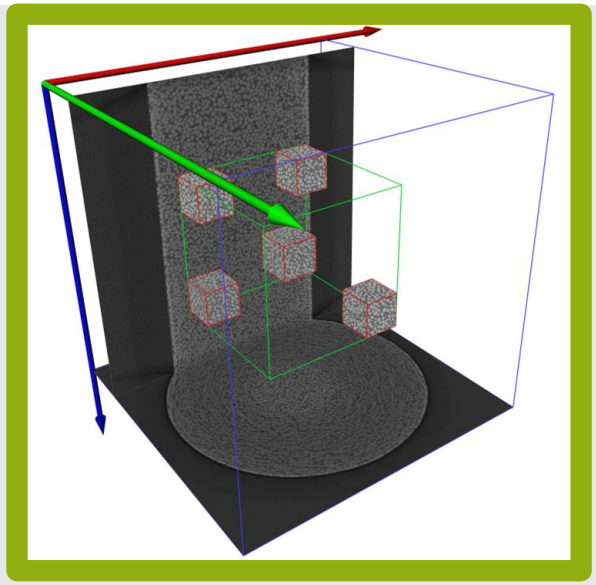
experimental setup – stages

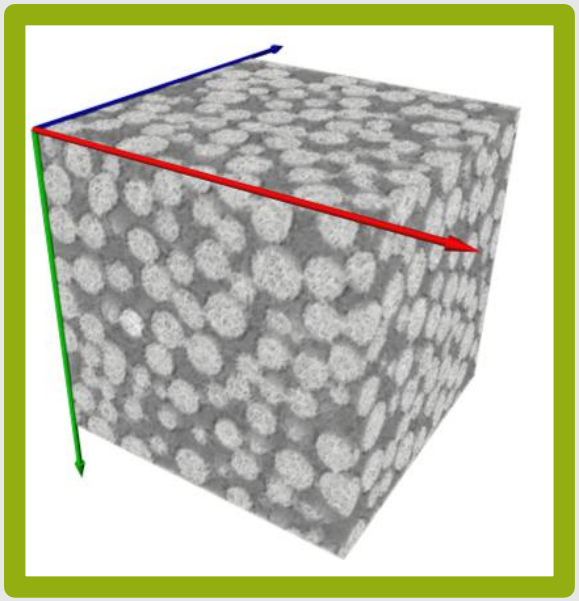


Different experimental stages with increasing complexity

experimental setup – stages

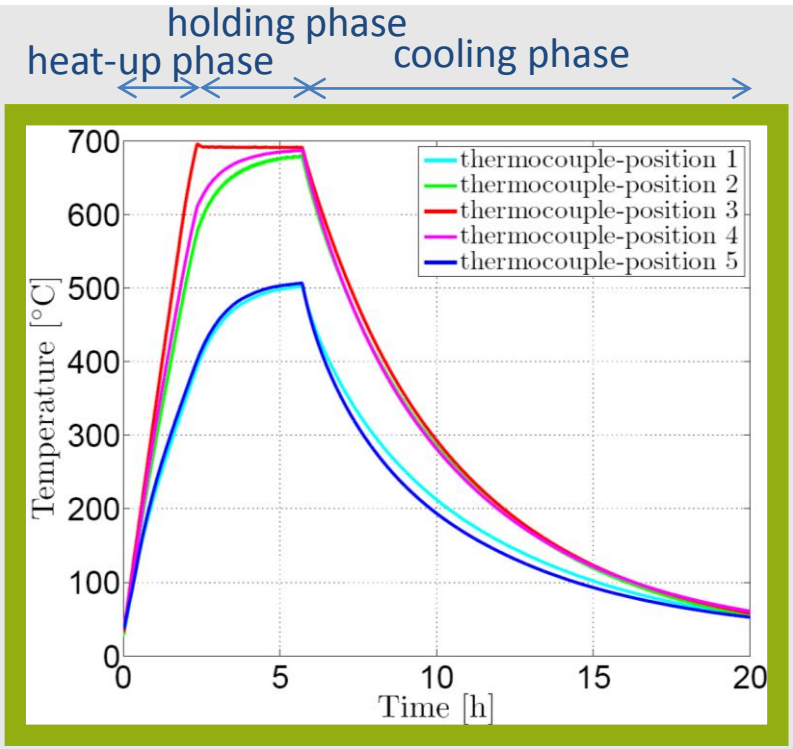






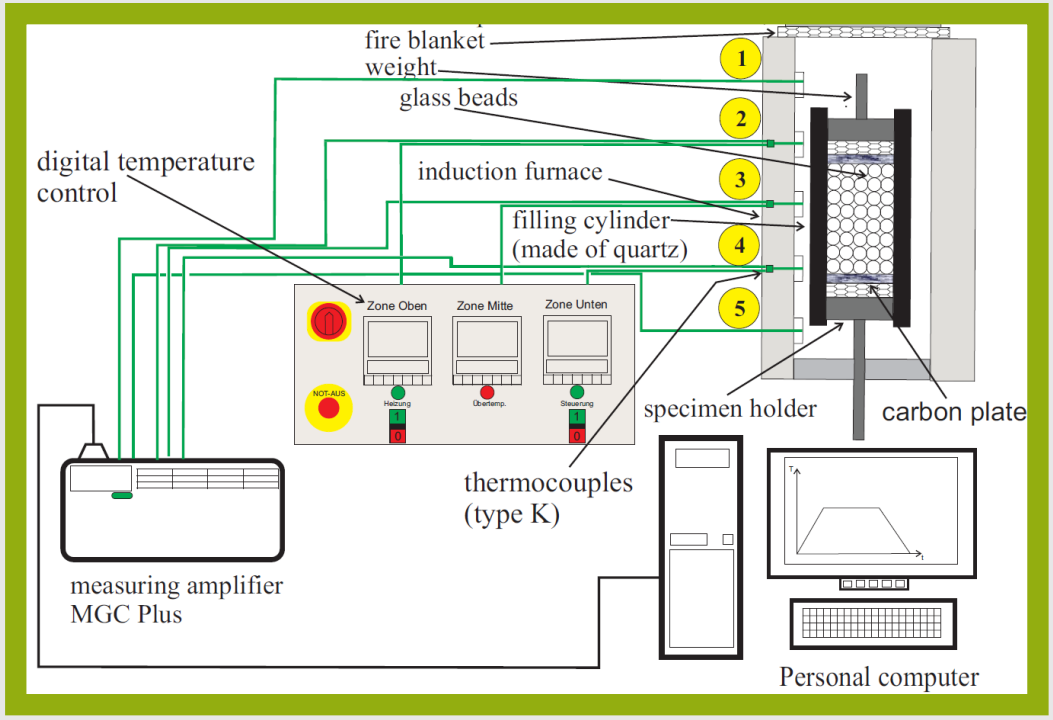
µCT scans of the produced sintered glass bead samples

sintering



Temperature curves in different furnace zones

Experimental set-up for sintering glass beads



- **tubular furnace with 0.7 kW heat output**
- **embedded in quartz glass cylinder**
- **averaged dead load/ half sample weight ratios (0.39, 0.32, 0.24)**
- **enclosed in carbon plates**
- **initial filling height: 58 – 90mm → shrinking during sintering process**

sintered glass bead samples

specimens

- **sintered mono- & weakly polydisperse glass beads:**
- **porosity: $\phi \approx 0.12 – 0.38$**
- **different glass bead diameters: $d_P = 0.4 – 8$ mm**
- **cylindrical samples with different diameters (d_b = 25, 30 & 50 mm)**

d_p = 0.6-0.8mm

d_p = 3.0mm

d_p = 5.0mm

d_p = 8.0 mm

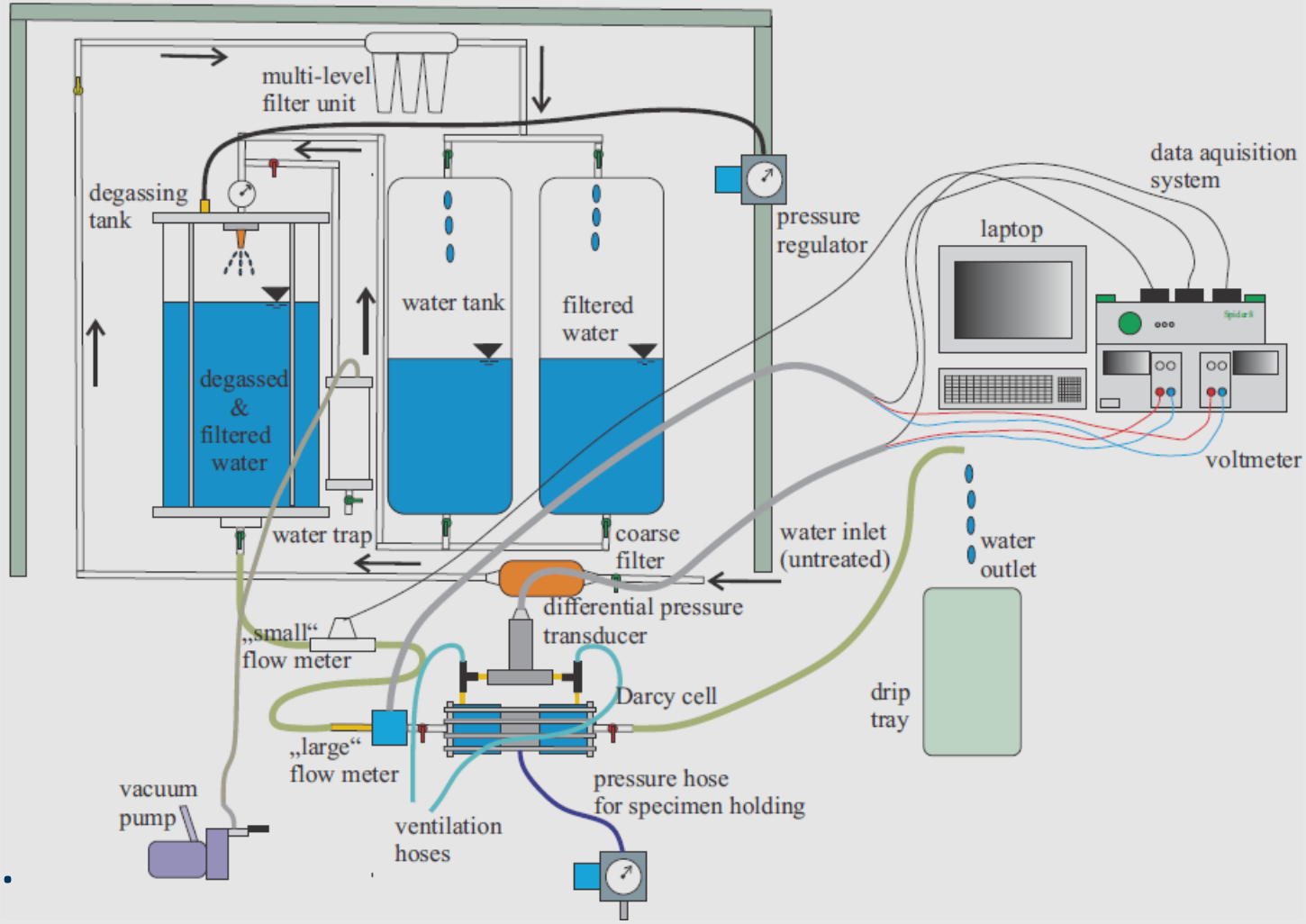
sintered glass bead samples

- **ADVANTAGES:**
- **chemically inertness**
- **relatively simple pore structure**
- **selective influencing of bulk properties (k^s, K_b, ϕ)**
- **high gray-scale contrast**

permeability experiments

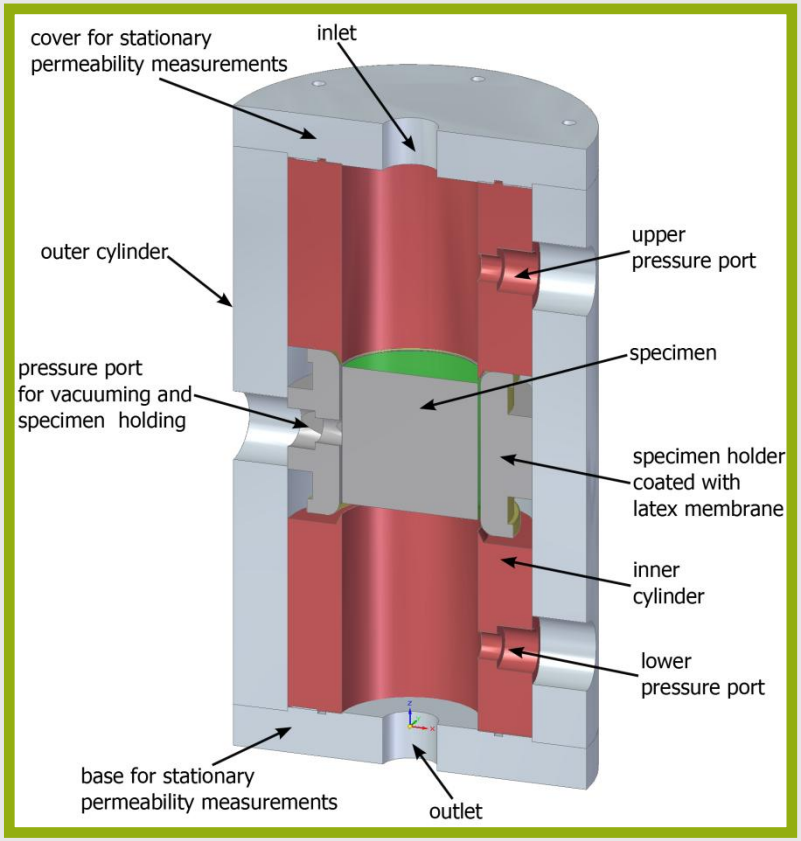
permeability experiments

- use of degassed & filtered water
- continous measured data aquisition (Q, Δp)
- Δp between the inlet & outlet chamber
- gradually increasing of water flow by increasing pressure in degassing tank
- flow-meter: 20-250ml/min.
- differential pressure transducer: 0-35mbar

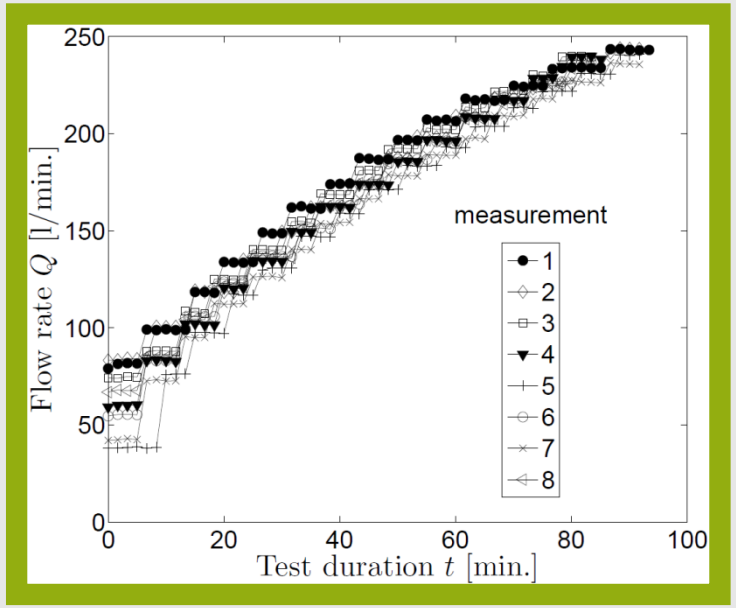


Principle set-up for stationary permeability measurements

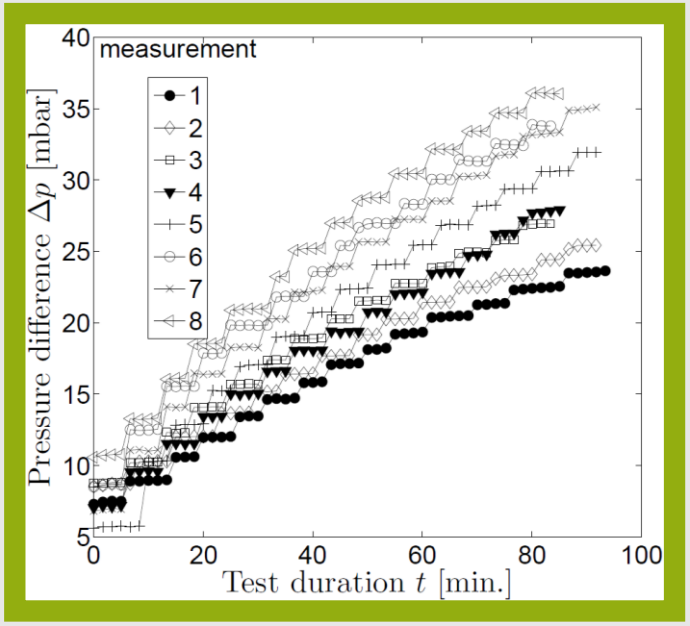
permeability experiments



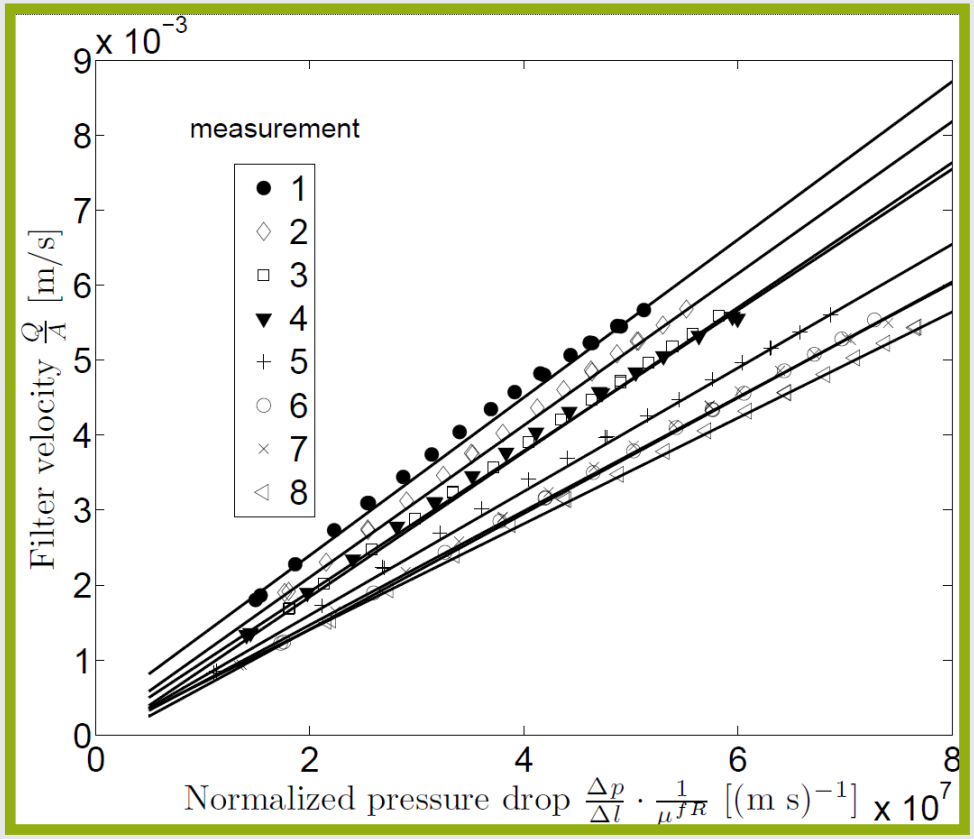
Three dimensional image of the measuring cell (half cut).



Stepwise increase of measured volume flux Q (left) and pressure difference Δp (bottom) in dependence of the measuring time t.
The sintered glass bead sample showed particle diameters between 0.4 and 0.6 mm.



permeability experiments



Filter velocity Q/A as a function of the pressure drop Δp/Δl (normalized to the viscosity of water μ^{fR}).

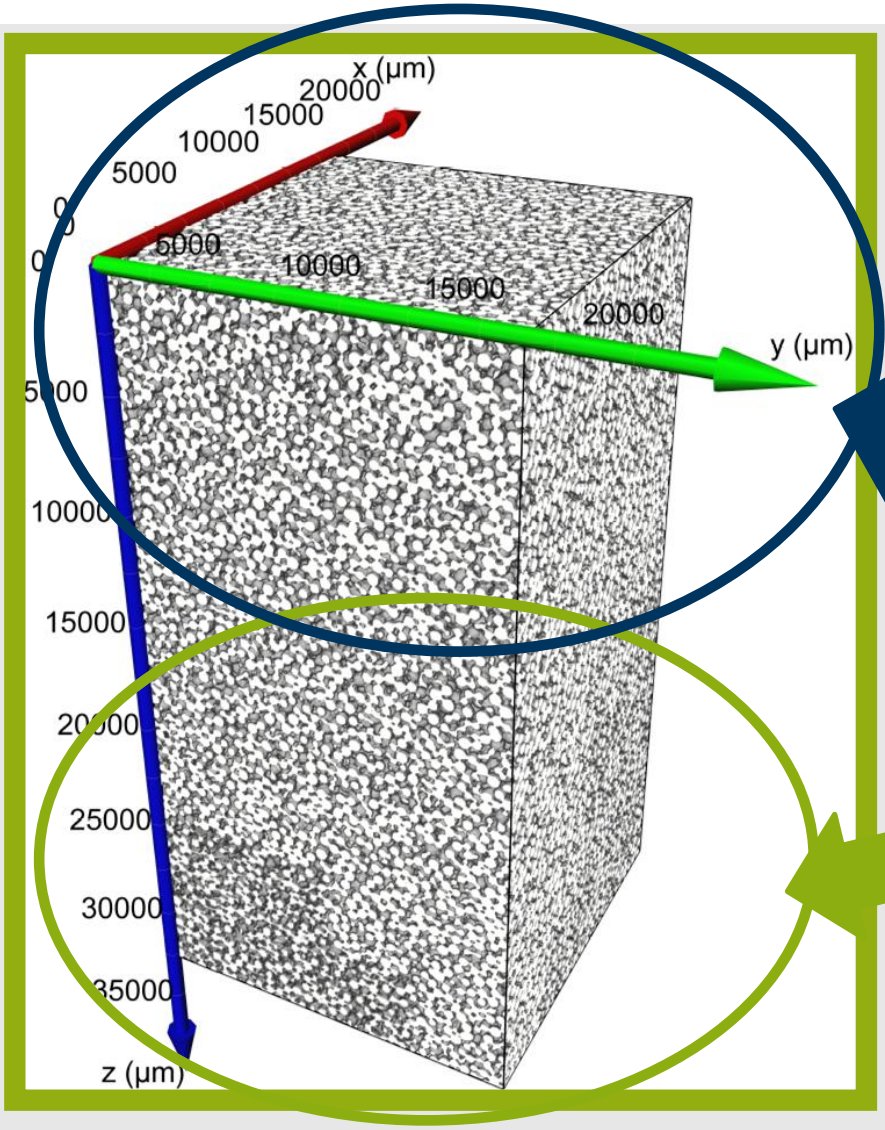
- **Darcy's law:**

$$\frac{Q}{A} = \frac{k^s}{\mu^{fR}} \frac{\Delta p}{\Delta l}$$

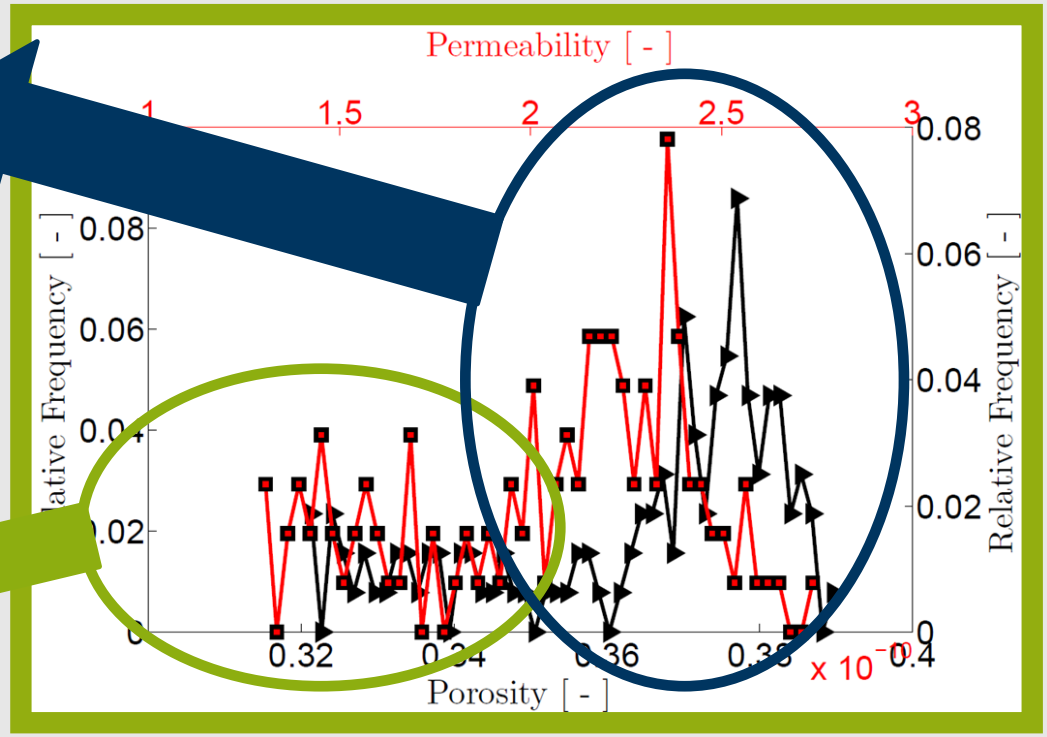
→ k^s: intrinsic permeability [m^2]
→ Q/A: superficial velocity [m/s]
→ μ^{fR}: fluid viscosity [Pa s]
→ Δp/Δl : pressure drop [Pa/m]

µCT data analysis

µCT analysis – porosity & permeability



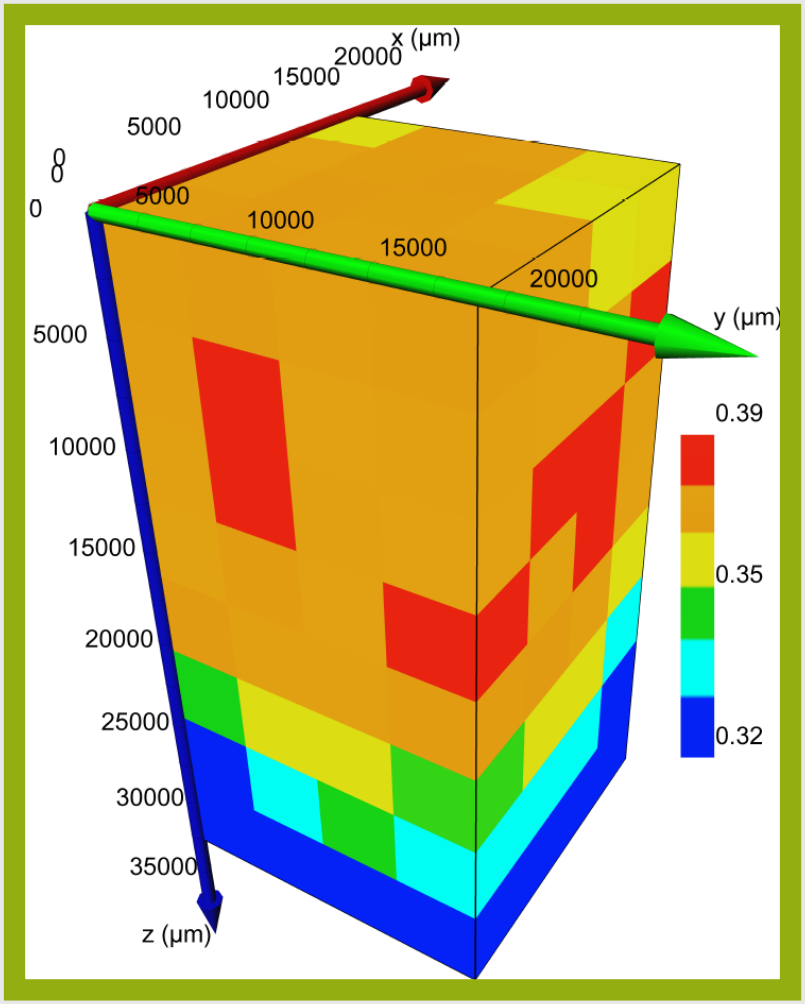
- voxel = 3D pixel
- voxel resolution: 16 µm/voxel



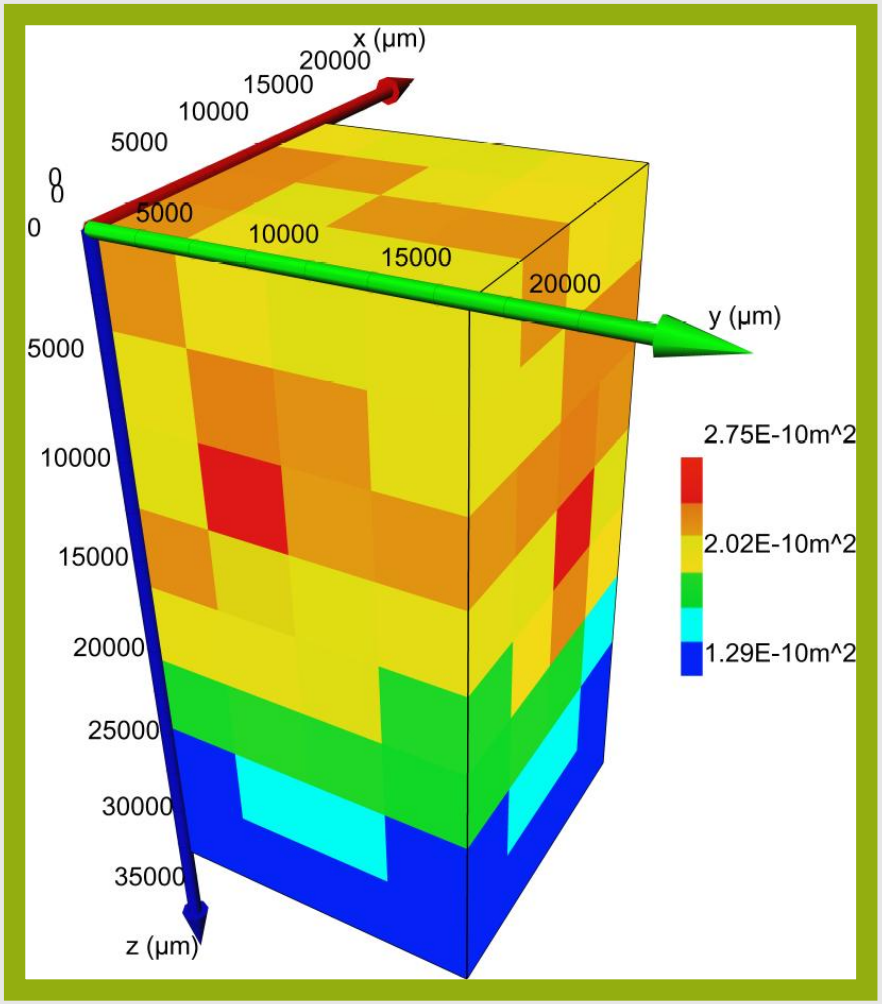
3D image data of the initial cube (left) with dimensions of 1024 (x) X 1024 (y) X 2048 (z) voxel^3 (left). Frequency distribution for porosity and permeability within the initial cuboid (right).

porosity & permeability

- **porosity**



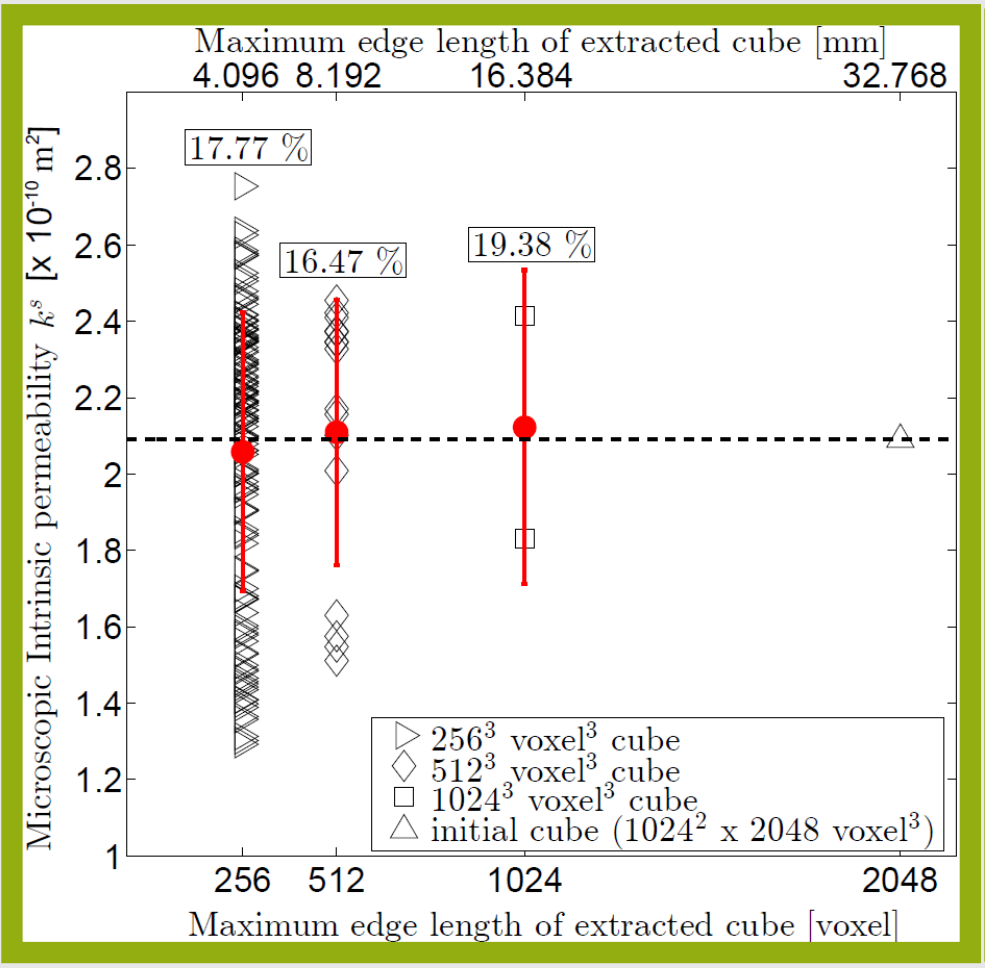
- **permeability**



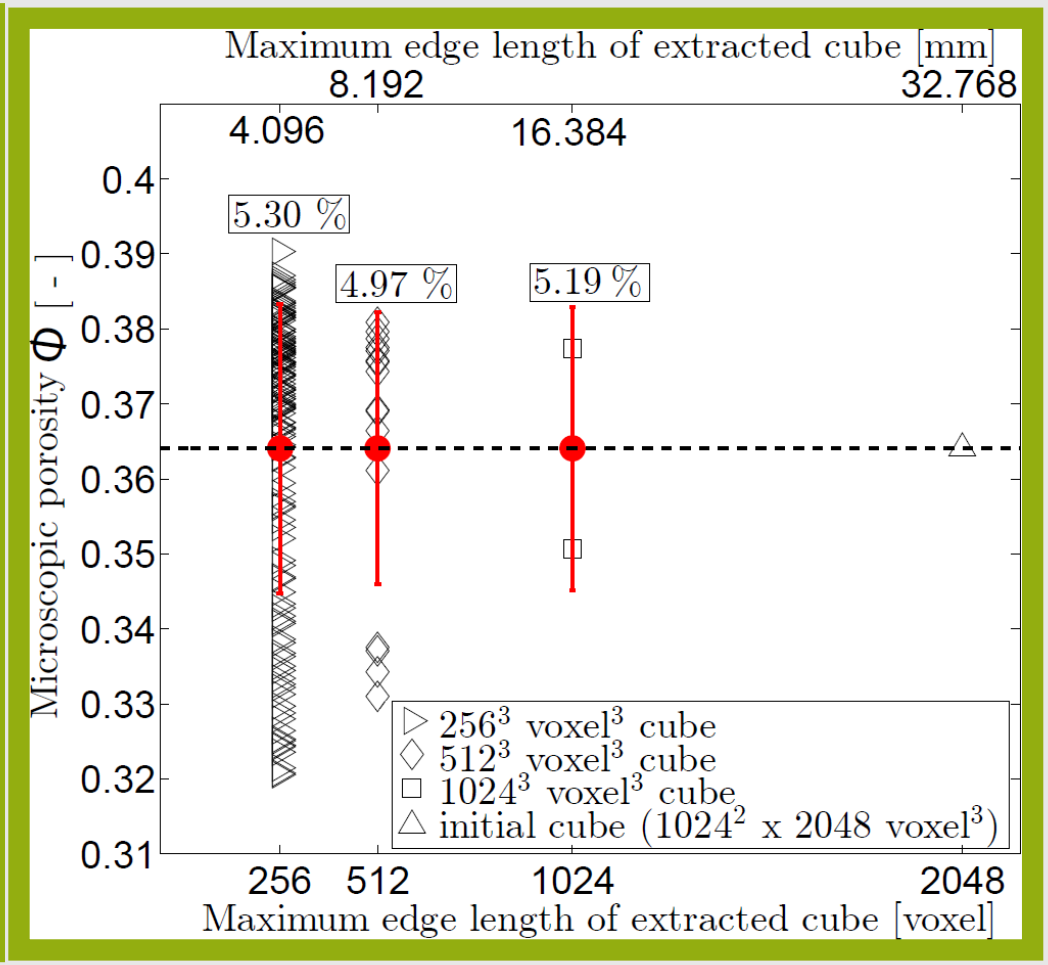
Porosity (left) and permeability (right) distributions within the initial investigated cuboid with dimensions of 1024 (x) X 1024 (y) x 2048 (z) voxel^3.

porosity & permeability

- **permeability**



- **porosity**



Conventional representative volume element (RVE) analysis for porosity (left) and permeability (right) are shown below.

porosity & permeability

Intrinsic permeabilities of different sized subsets as a function of porosity.

- lattice Bolltzmann simulations

  (Stefan Frijters/ Jens Harting)

  cf. NARVAEZ et al. (2010)

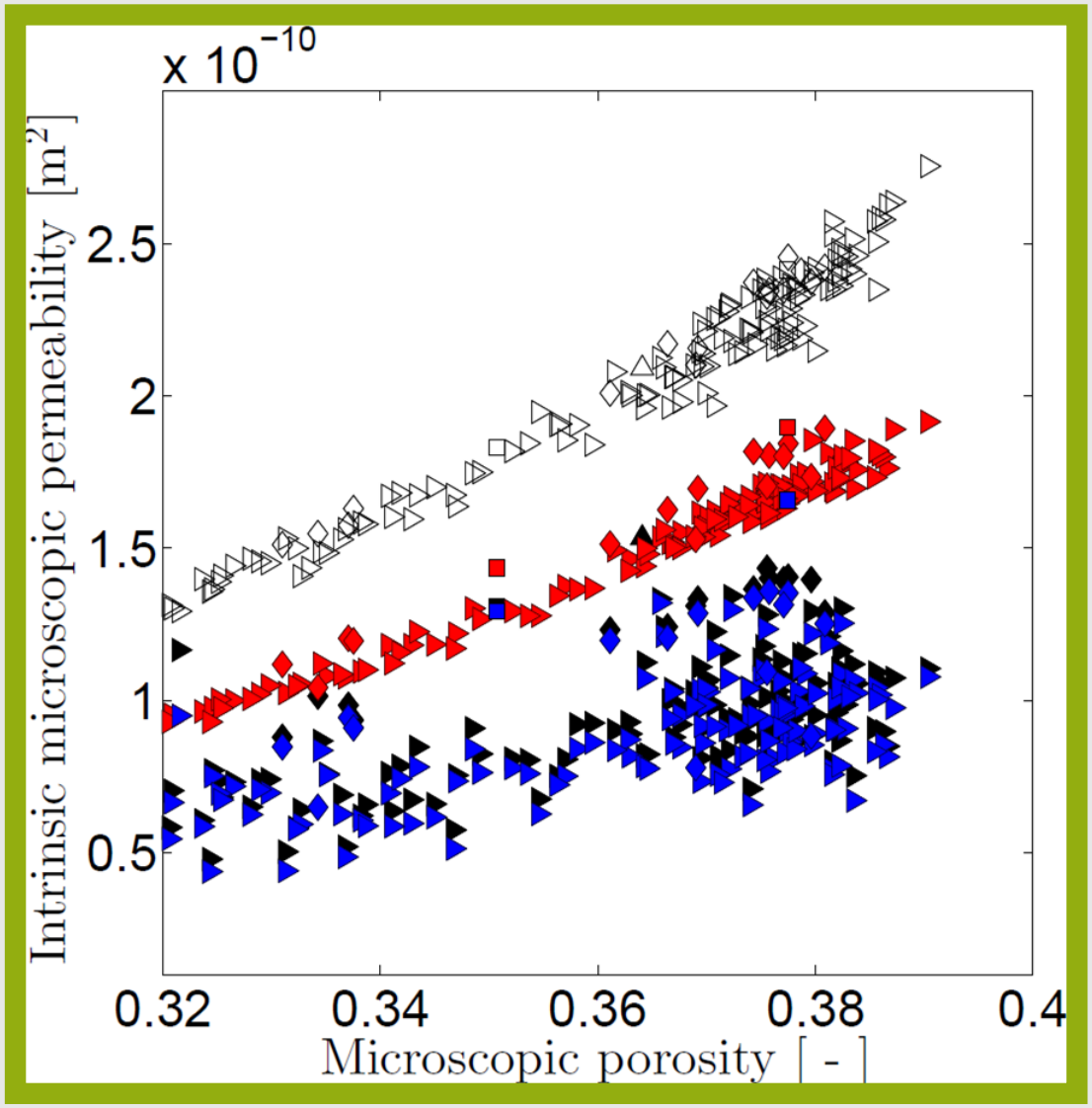
Kozeny-Carman equation:

$$k^s = \frac{1}{c_1} \frac{\phi^3}{(1-\phi)^2} d_p^2$$

with: $c_1 \approx 18\,\pi^2 \approx 178$

assumptions:

- monodisperse sphere packings
- no sintering effects



porosity & permeability

Dimensionless permeabilities of differently sized subsets as a function of porosity.

- **lattice Bolltzmann simulations**

  (Stefan Frijters/ Jens Harting)

  cf. NARVAEZ et al. (2010)

Kozeny-Carman equation:

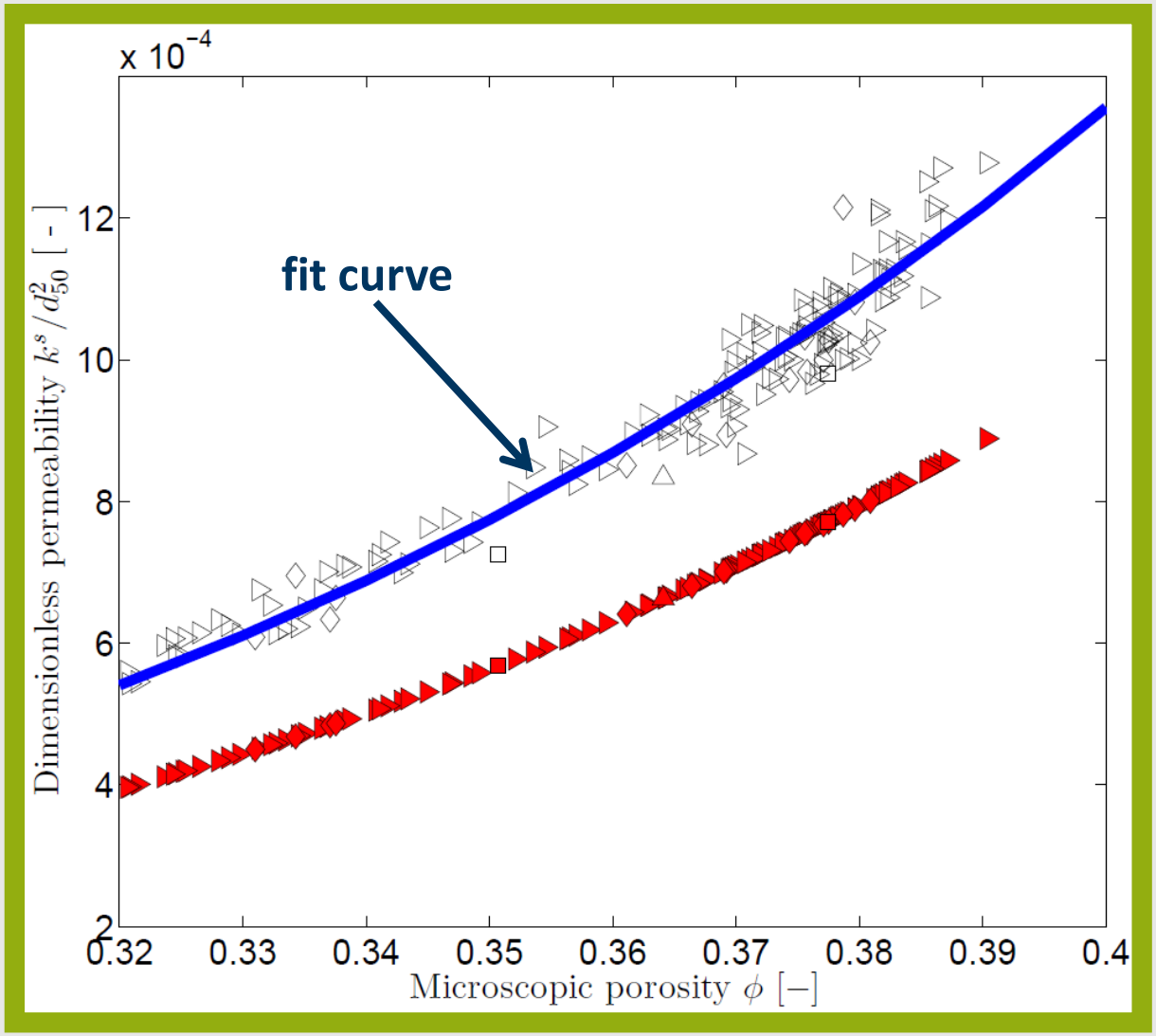
$$k^s = \frac{1}{c_1} \frac{\phi^3}{(1-\phi)^2} d_p^2$$

with: $c_1 \approx 18\,\pi^2 \approx 178$

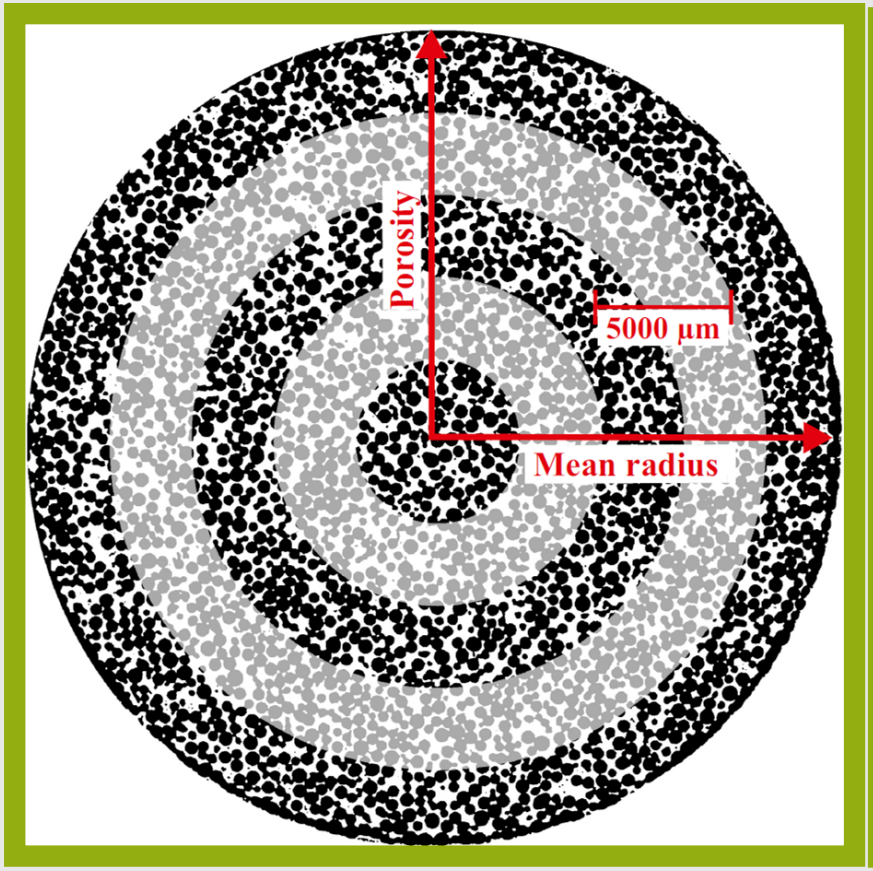
assumptions:

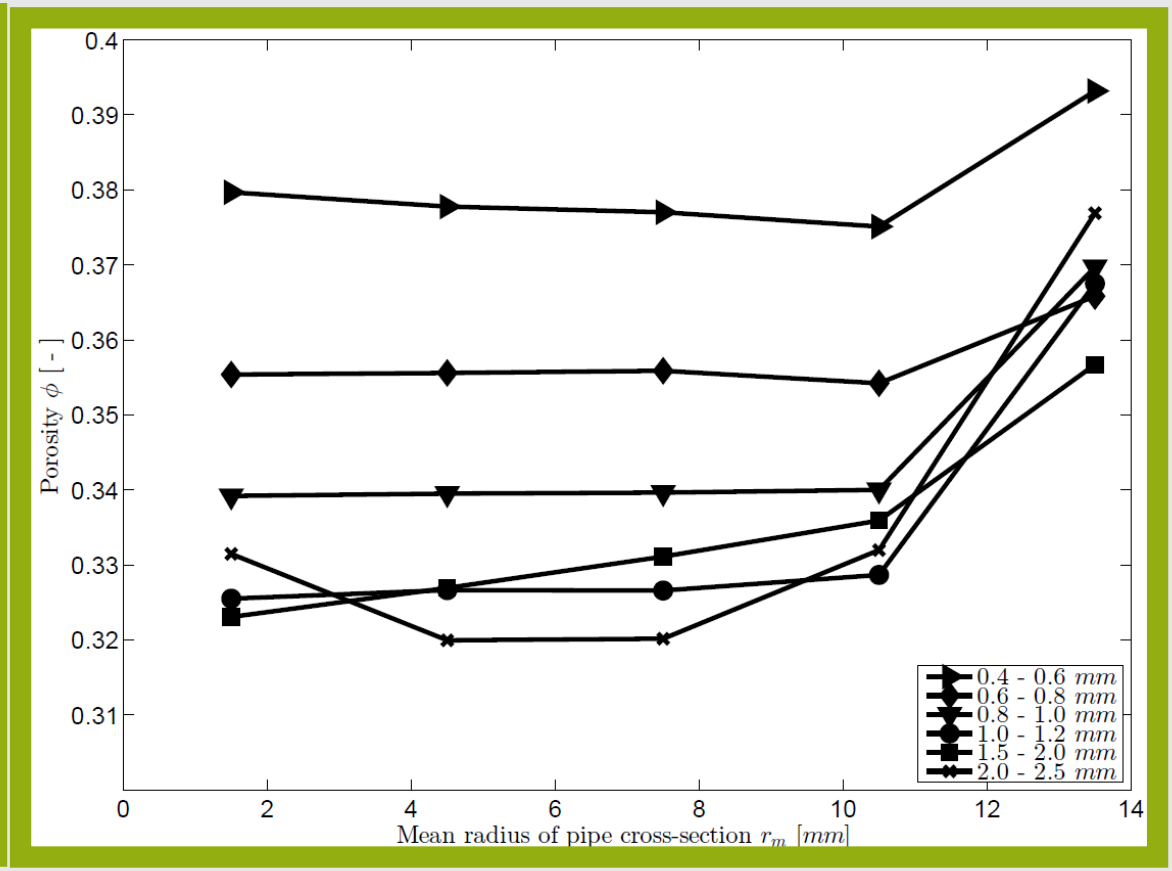
- **monodisperse sphere packings**
- **no sintering effects**

$$c_{1,fit} = 131$$



porosity

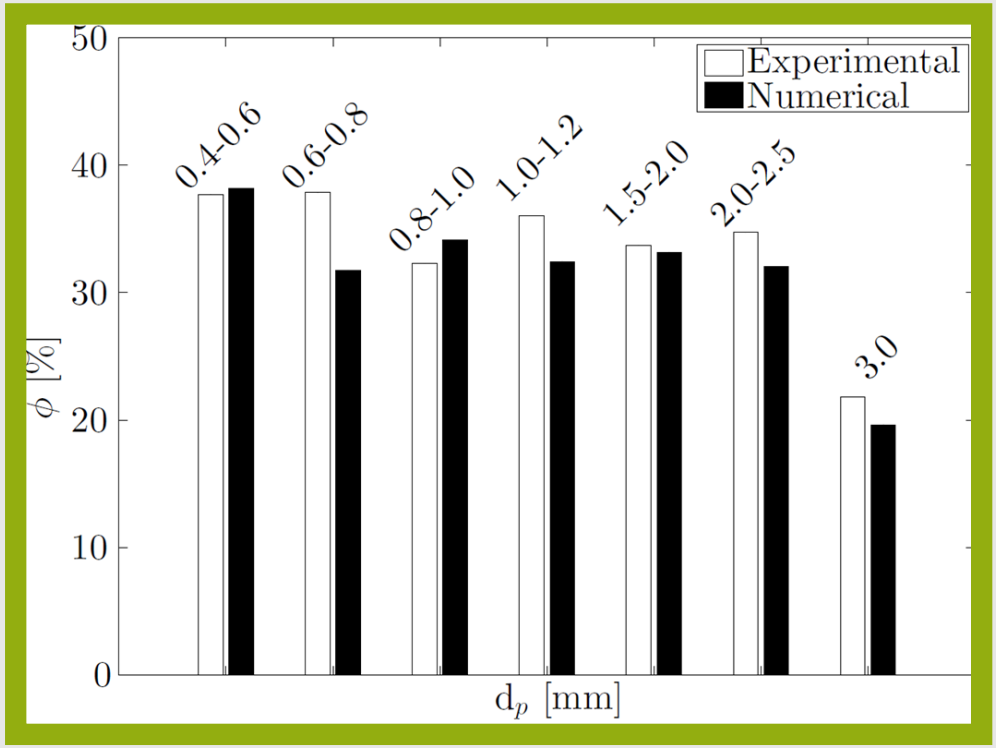




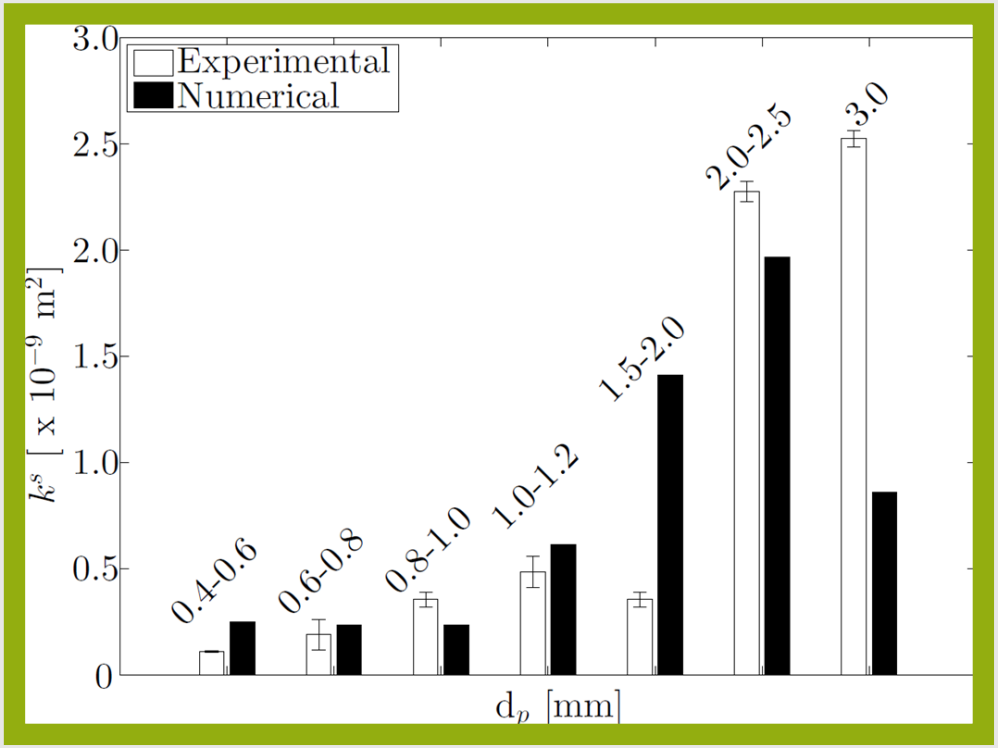
Cross section view after segmentation of tubes with different mean radii (left). Porosity distribution in dependence on the mean radius of pipe cross-section for different specimens (right).

porosity & permeability

❖ **porosity**



❖ **permeability**

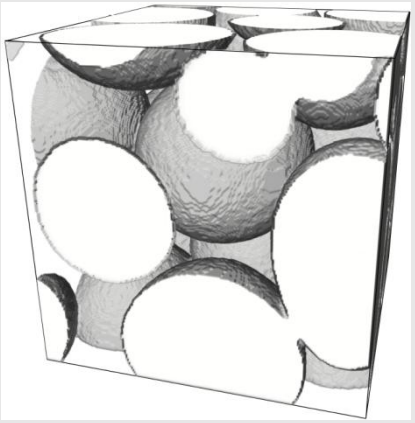


Numerical and experimental determined porosity (left) and permeability (right) values for sintered glass bead samples showing different particle diameter

permeability ← → pore throat from µCT

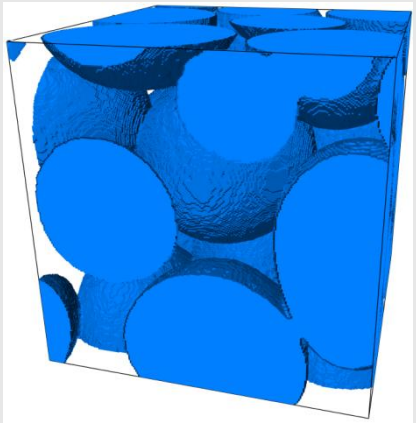
pore throat

- filtered grayscale image



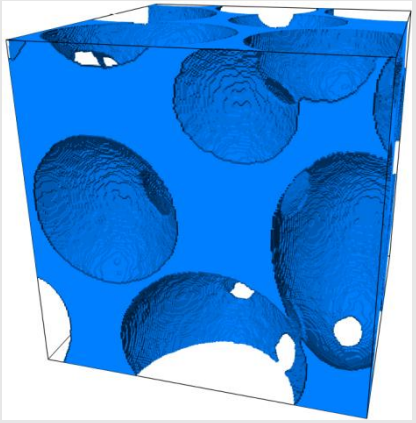
- interactive thresholding

- binarized image (beads = 1; pore space = 0)



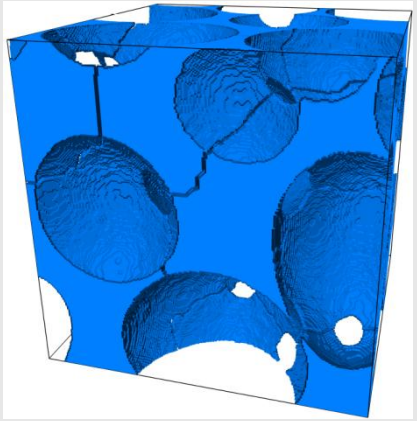
- inversion

- binarized image (beads = 0; pore space = 1)



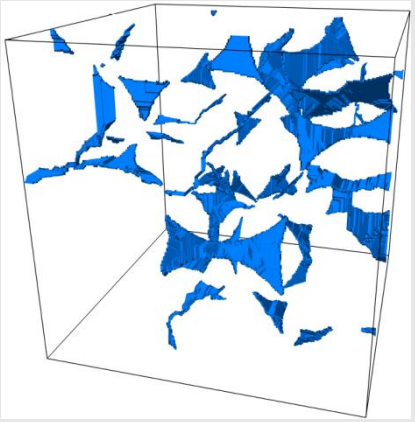
- separate objects

- separated pore space



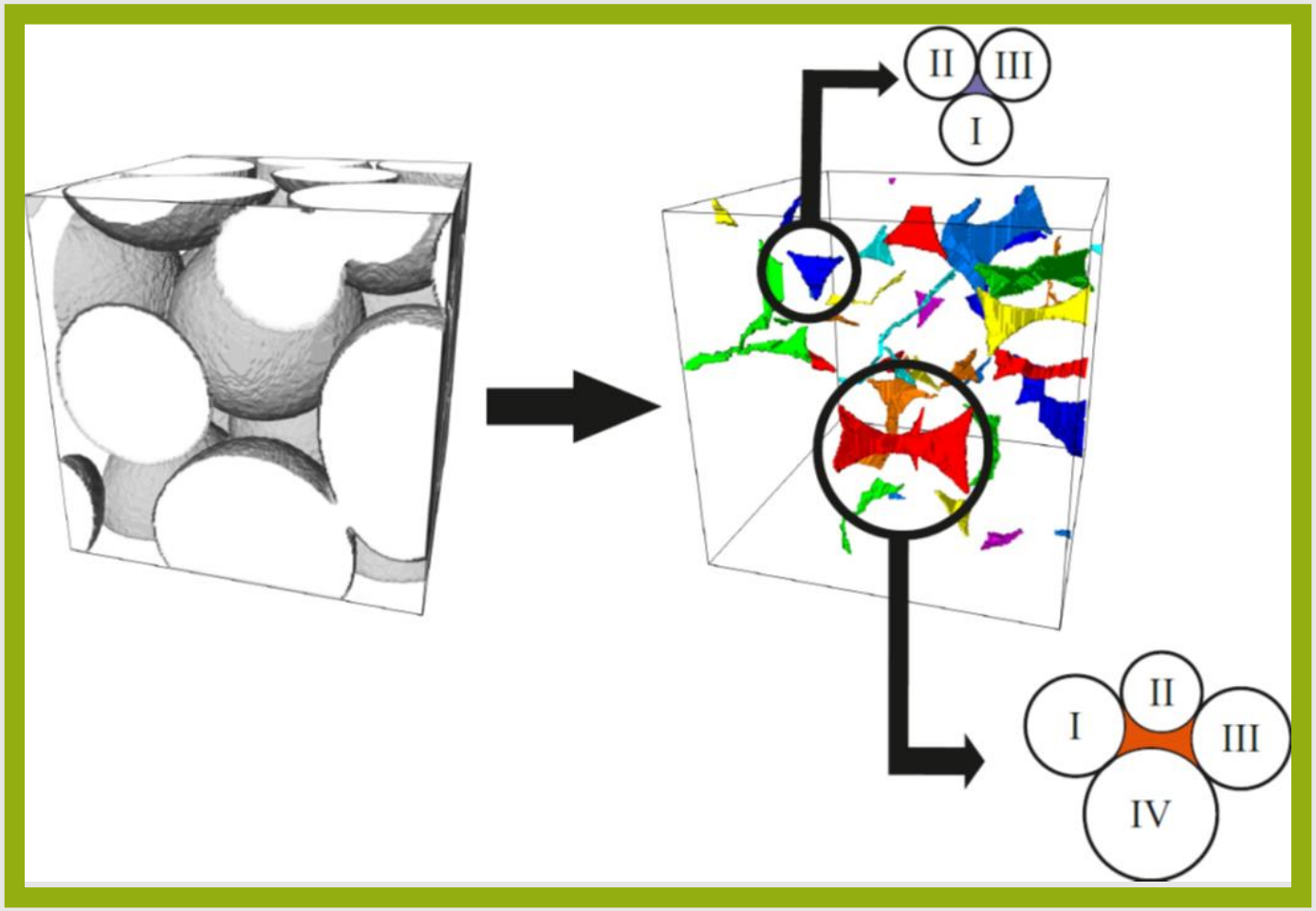
- arithmetic

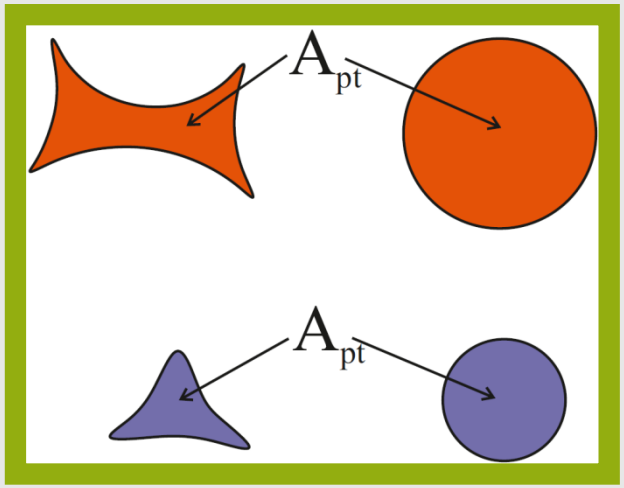
- cut surfaces



Depicting of main image processing steps for visualization and quantification of pore thoats in spherical systems.

pore throat



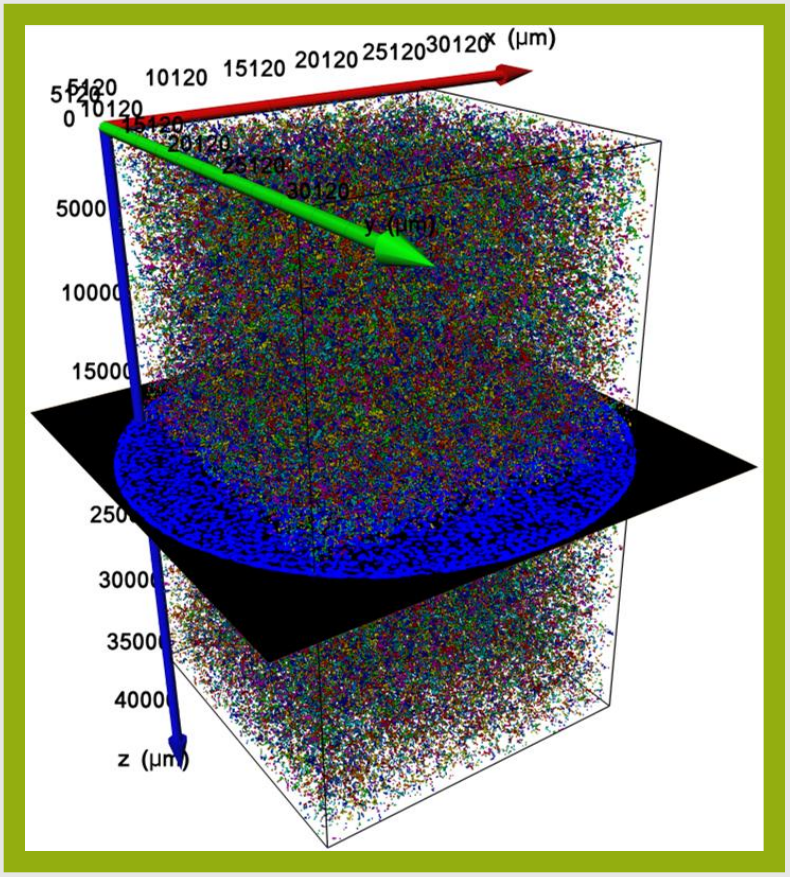


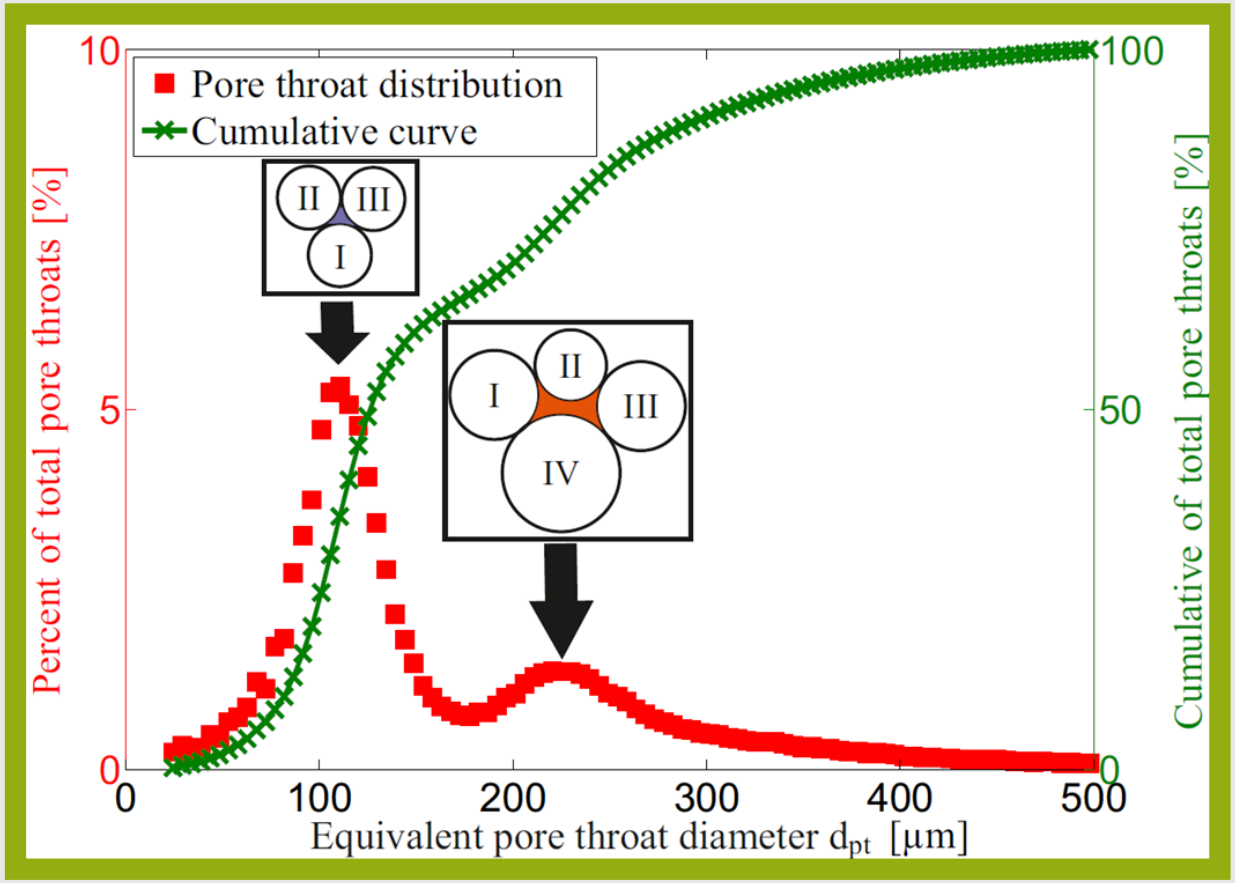
- **Equivalent pore throat diameter:**

$$d_{pt} = \sqrt{\frac{4\ A_{pt}}{\pi}}$$

Visualisation and quantification of pore throat areas in glass bead packages using AVIZO Fire 8.0.1. The results show typical pore throat areas which are formed by three or four particles. The pore throats strongly influence the intrinsic permeability, which significantly determines the coupling between fluid and solid in low-frequency range of acoustical wave propagation.

pore throat

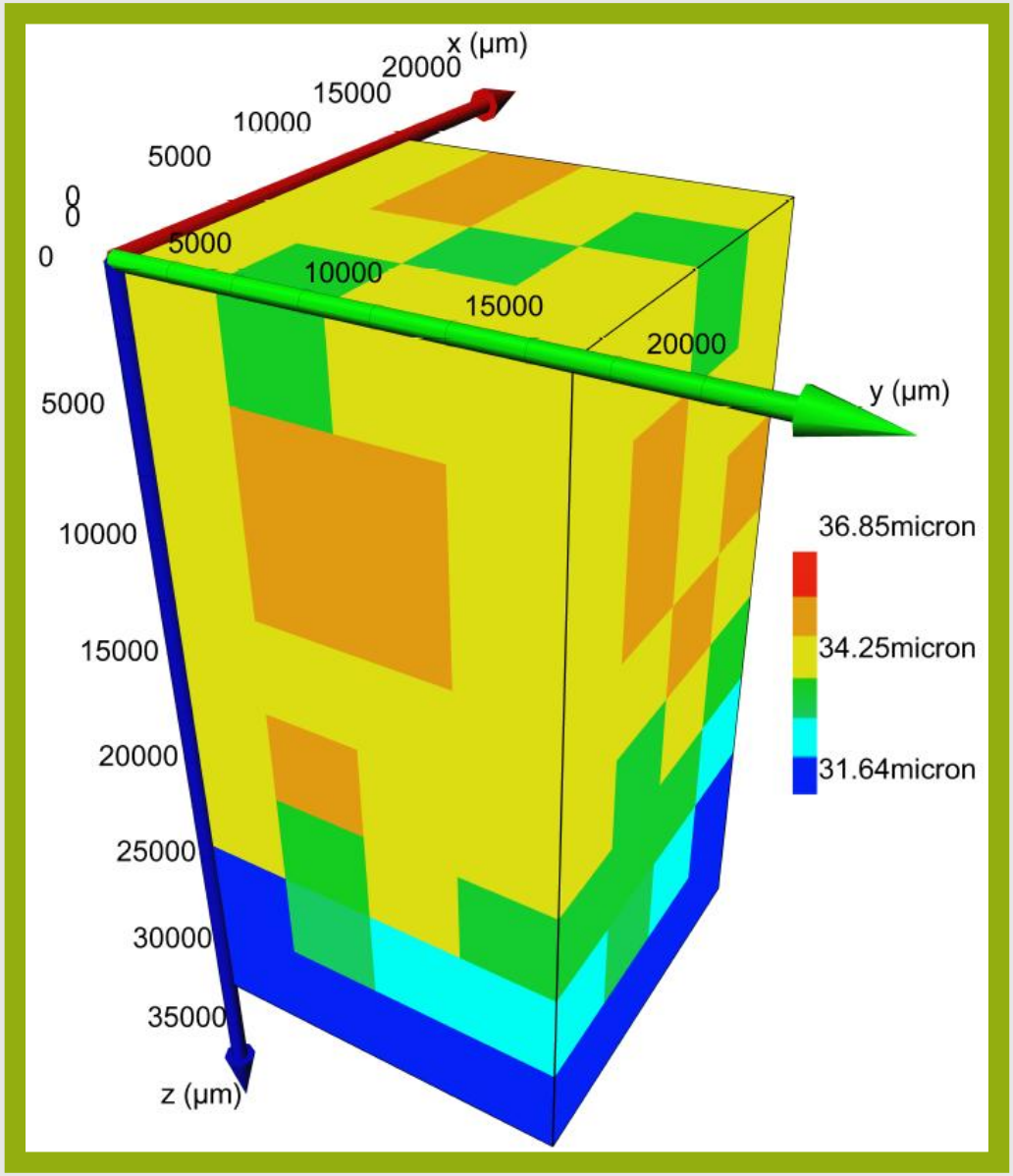




Investigated subvolume for pore throat analysis with dimensions of 1300 (x) X 1300 (y) X 2200 voxel^3 (left). The considered subset contained about 150,000 glass beads with particle diameters ranging mostly between 0.4 and 0.6 mm. The total number of pore throat areas within the considered cuboid was about 257,000. Resulting pore throat size distribution with corresponding cumulative curve (right).

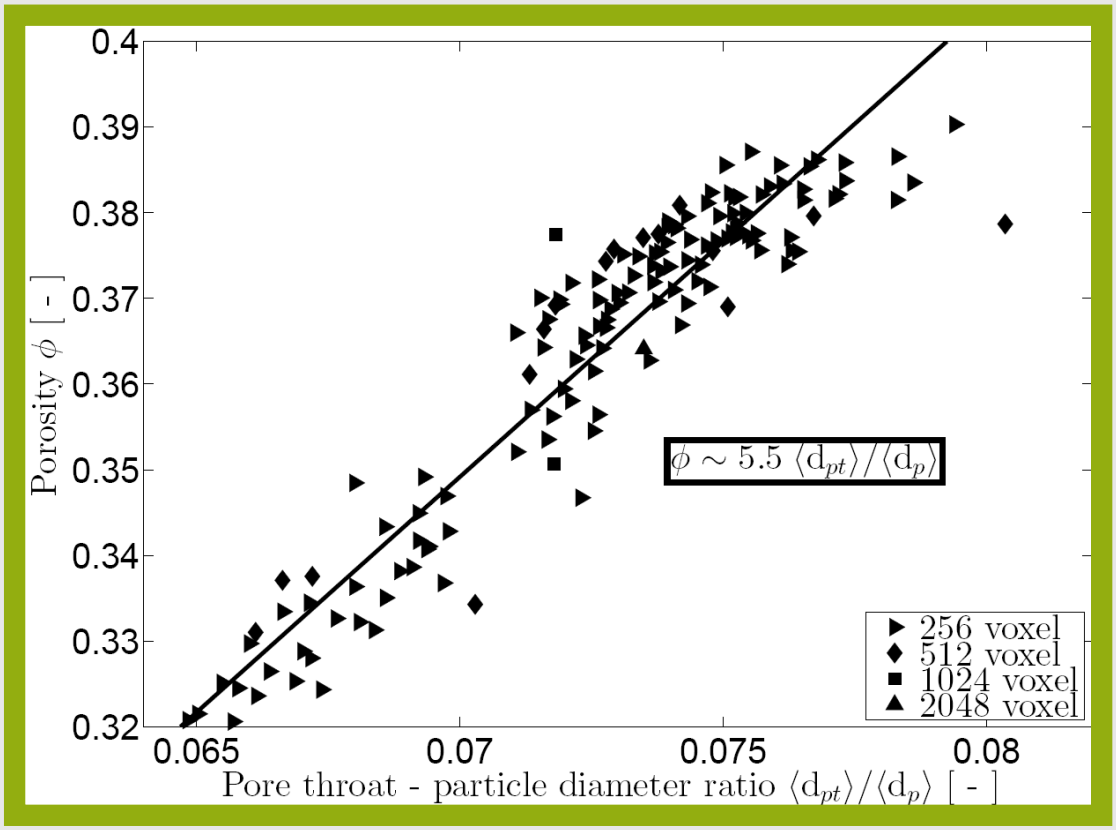
pore throat

Mean pore throat diameter distribution within the initial cuboid with dimensions of 1024 (x) X 1024 (y) X 2048 (z) voxel^3 **(right)**.

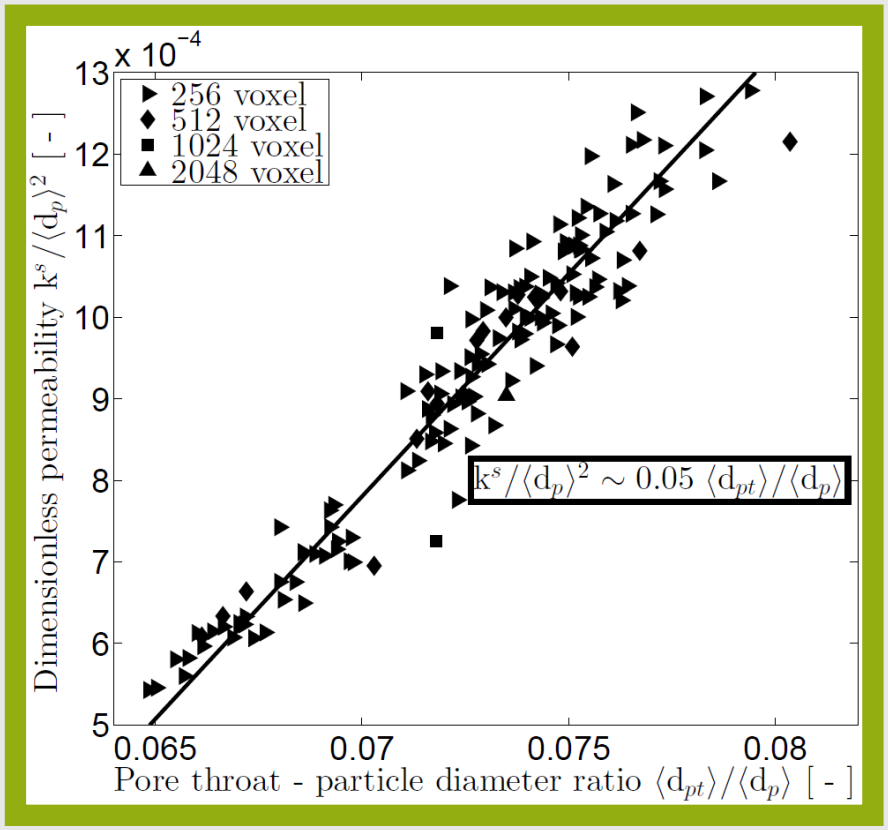


pore throat

- **porosity**



- **permeability**

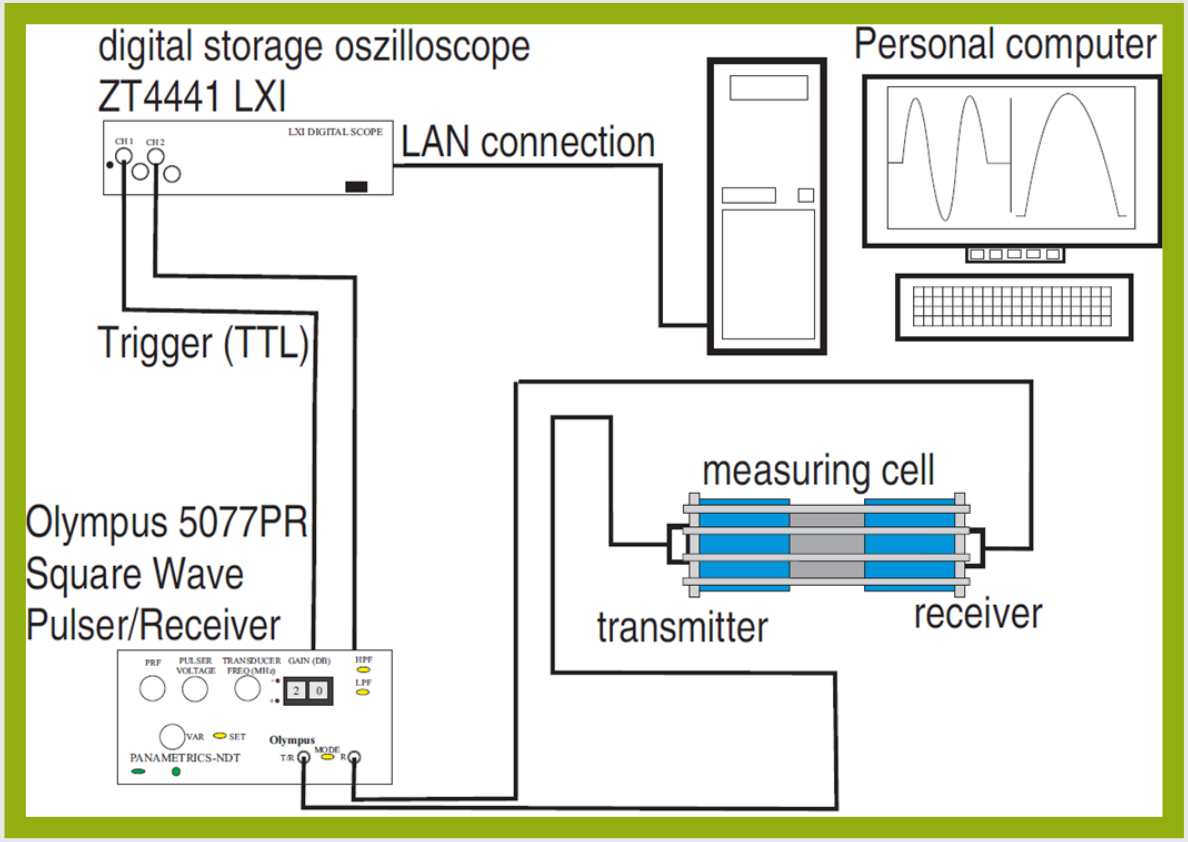


Porosity (left) and dimensionless permeability (right) in dependence on the mean pore throat diameter normalized to the mean particle diameter.

ultrasound experiments

ultrasound experiments

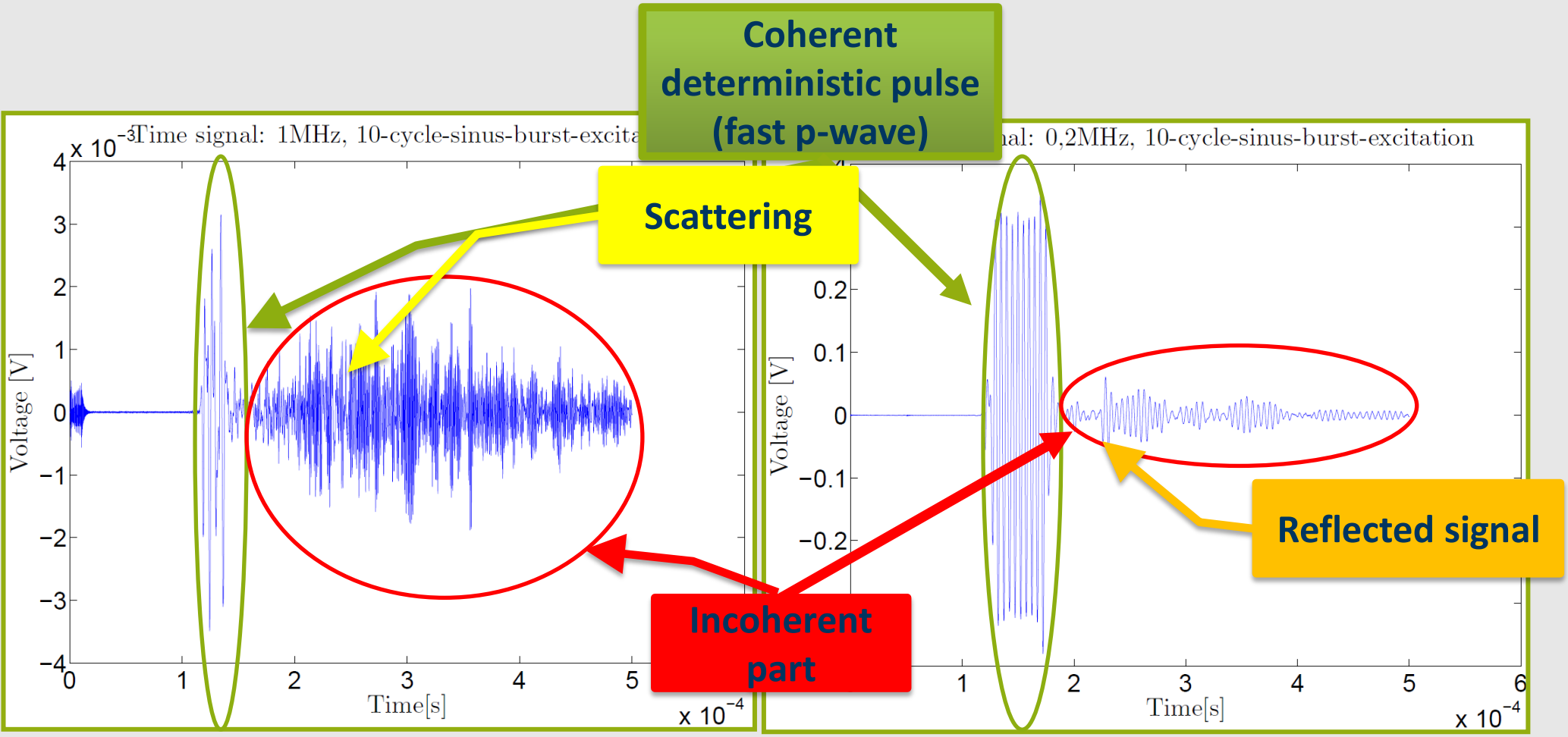
ultrasound experiments



Experimental arrangement for ultrasound measurements .

- **5 samples with same glass bead mixture (d_p = 1.0 – 1.2 mm & 3.0mm)**
- **different wave forms/frequencies**
- **<u>Goals</u>: 1. evaluation of intensity of scattered waves**
  2. theoretical description by diffusion model

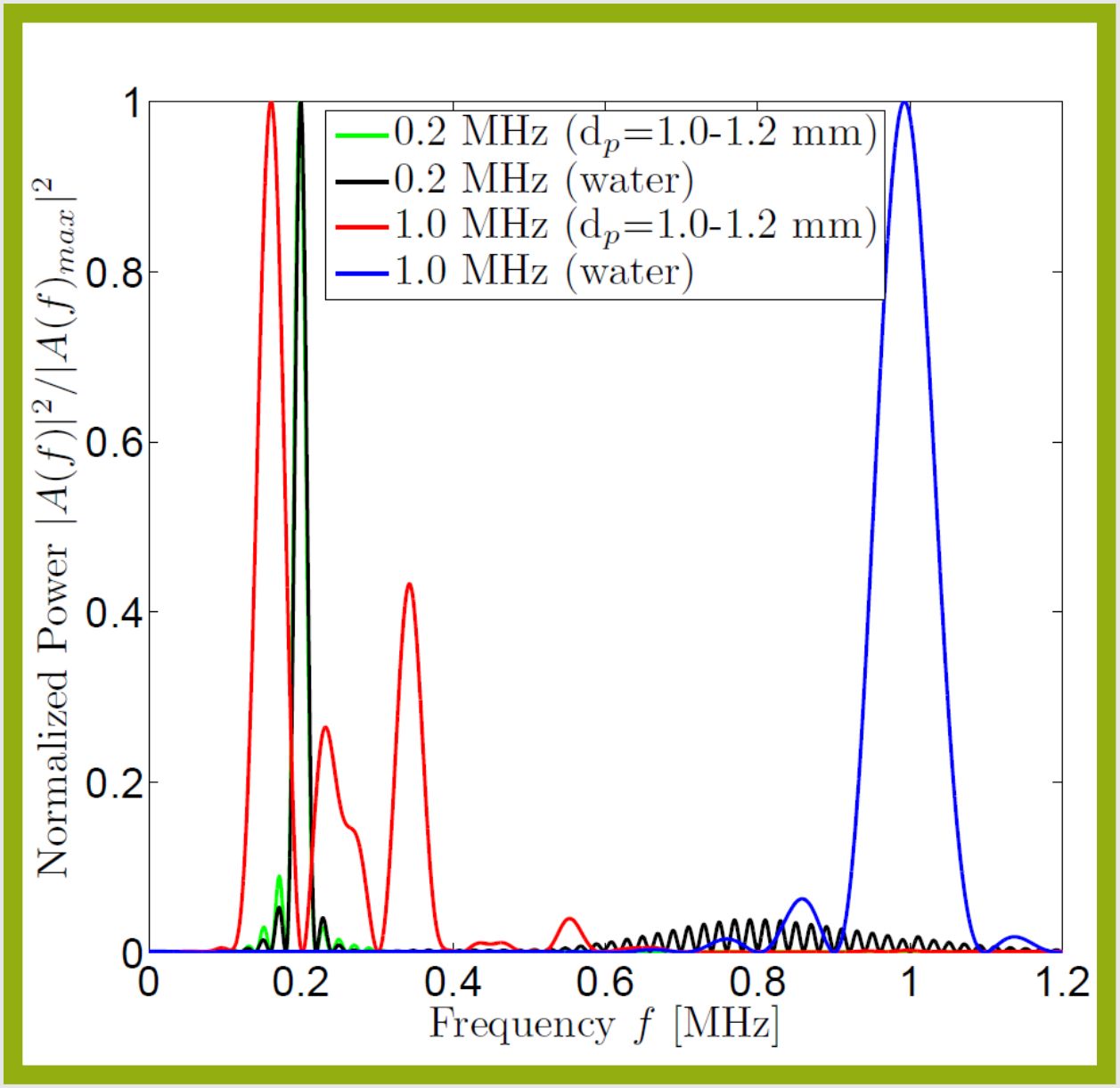
ultrasound – time signal



Time signals for water-saturated sintered glass bead sample consisting of glass beads with diameters between **1.0** and **1.2 mm** at different frequencies **1.0 MHz (left)** and **0.2 MHz (right)**.

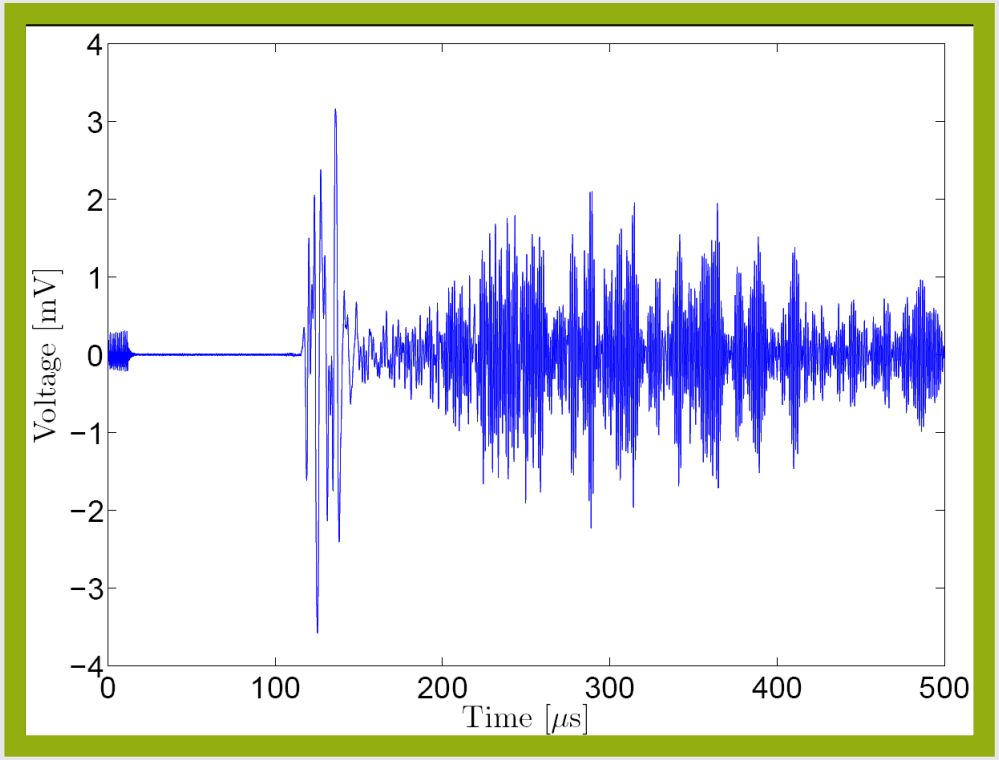
ultrasound – power spectrum

Normalized Power Spectra
of low- and high-frequency wave
through sintered sample and water.

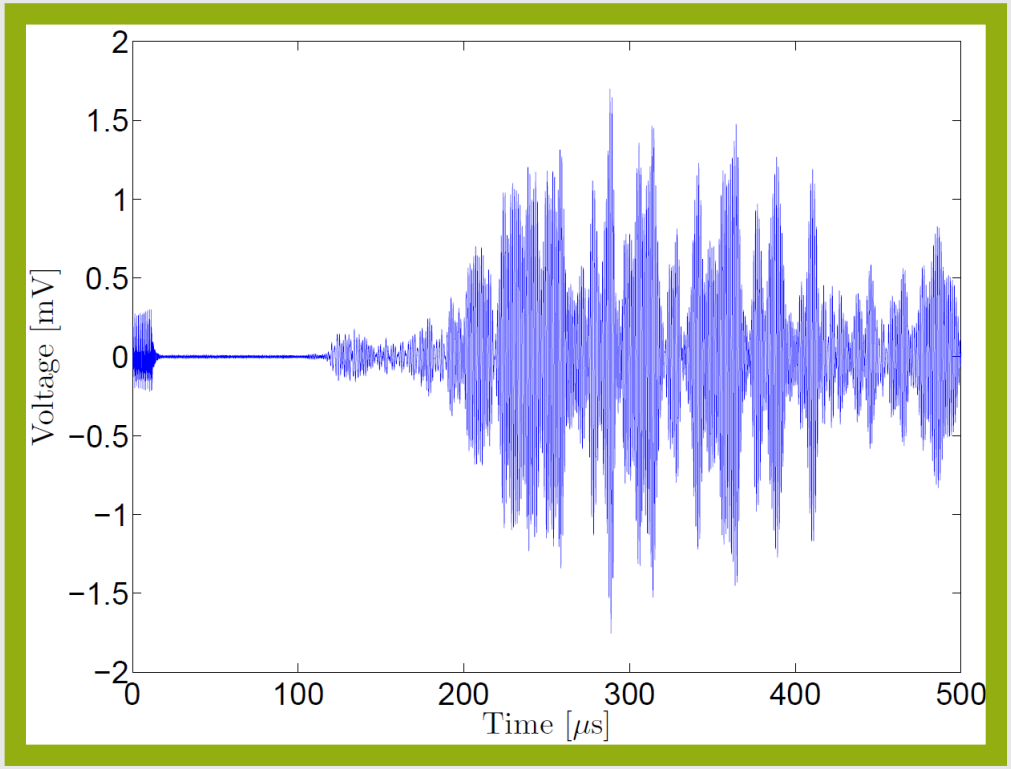


time signal

- raw signal

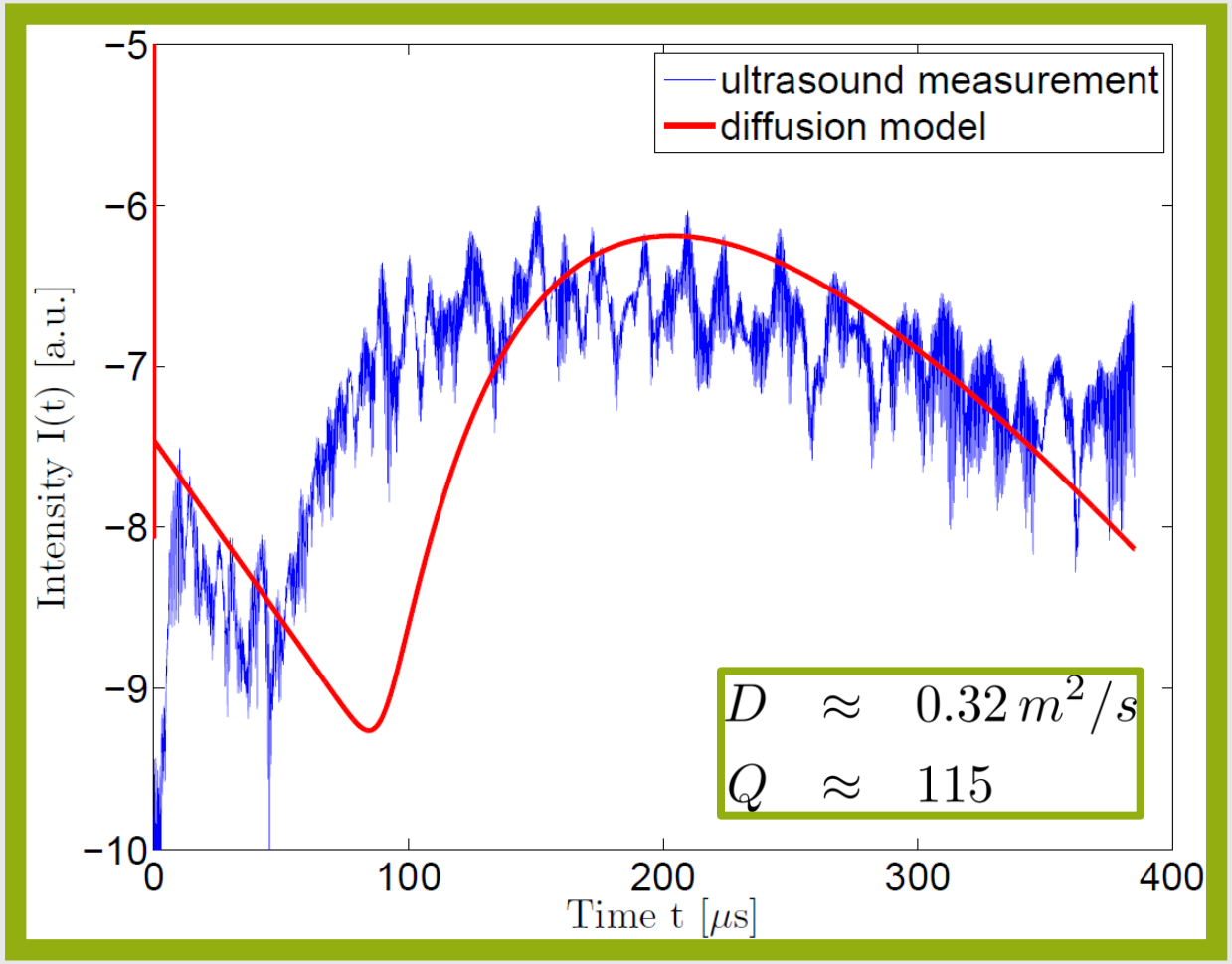


- highpass-filtered signal



Raw signal at **0.9 MHz** containing both the coherent pulse and incoherent scattered waves **(left)** . Highpass-filtered signal at a cut-off frequency of **0.7 MHz (right)**.

ultrasound – diffusion model



Comparison of the averaged intensity I of scattered waves from ultrasound measurements with fitting curve from diffusion approximation.

- **diffusion equation:**

$$\partial_t I - D \nabla^2 I + \frac{I}{\tau_\alpha} = \delta(z)\delta(t)$$

with: $D = \frac{1}{3} v_e l^\star$ **&** $\tau_\alpha = \frac{Q}{2\pi f}$

- Page et al, PRE 52 (1995)

→ D: diffusion coefficient [m^2/s]
→ τ_α: (inelastic) absorption time [s]
→ l^*: transport mean free path [m]
→ v_e: velocity of transported energy [m/s]
→ Q: Quality factor [-]
→ f: frequency [Hz]
→ δ(t),δ(z): source terms [-]

$$l^\star \approx 1 \text{ mm}$$

conclusion

- <u>hydraulical properties</u>:

  - holistic consideration of hydraulic influence parameter ←→ µCT data
  - spatial gradient of porosity, permeability and mean pore throat diameter ←→ rotational sintering
  - intrinsic permeability ←→ Kozeny – Carman model
  - empirical constant: $c_1 = 131$ (microstructural features!)
  - hydraulic conductivity is mainly influenced by pore throat and porosity , less by geometrical tortuosity
  - comparison between experimental & numerical results is difficult (exponential errors!)

- <u>acoustical properties</u>:

  - time signal = low-frequency coherent pulse + high-frequency incoherent part
  - low-frequency pulse → fast P-wave → Biot model
  - high-frequency incoherent part → (multiple-) scattered waves → diffusion model

Thank you for your attention!

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