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Characterization of microstructures

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Image analysis

The principle of this widely used method consists in obtaining quantitative information from an image (microscopic, photography, etc.). It can be performed either manually or automatically (after appropriate processing, such as thresholding). Higher quality images are needed for automatic measurements, because otherwise the errors caused by the software during evaluation can be exaggerated, leading to incorrect results. Traditional applications of microscopic image analysis include the quantification of microbial colonies, chromosome analysis, in the industry for example the analysis of particles of powder materials, determination of solution concentration from the color, detection of bottle defects, metallography etc.

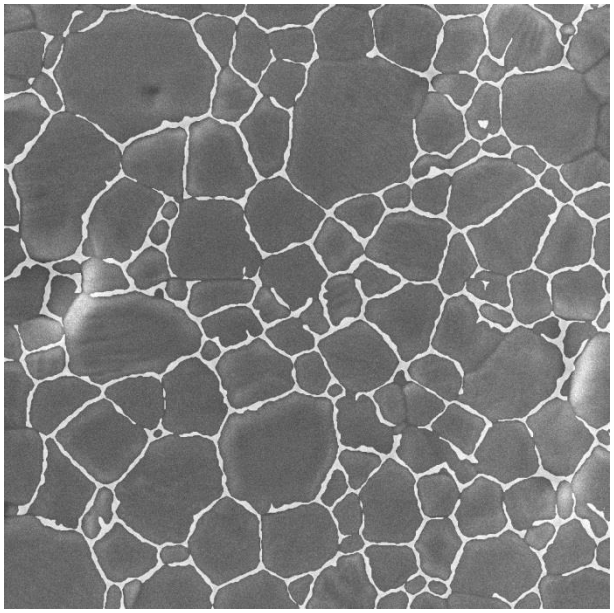


Fig. 1: Example of imperfect binarization (overlay of binarized image over the original), not all the edges are detected.

Particle, grain and pore characterization

Shape

Particles, grains and pores can be:

Isometric – approximately the same size in all directions – spherical shape, regular polyhedra (tetrahedron, cube, octahedron, etc.).

Anisometric – prolate (needles, cylinders, prisms) or oblate (plates, disks), the model shape (cylinder, rotational ellipsoid, …) and aspect ratio (the ratio of the largest to smallest dimension of the object or its projection or section) should be used in order to quantify the degree of anisometry.

Size

The size of spherical particles is defined as their diameter. Non-spherical particles, including irregular and anisometric ones, are characterized by an equivalent diameter, which is the diameter of sphere (or circle in case of a 2D projection of a 3D particle), which has the same property or behavior as the non-spherical particle of interest. Examples of equivalent diameters are the Stokes diameter – diameter of a sphere with the same settling velocity as the non-spherical particle (in slow laminar, so-called creeping, flow in the fluid with the same viscosity and density) and the Heywood diameter – diameter of a circle with the same area as the projection of the particle, also called area-equivalent diameter.

Apart from equivalent diameters there are other measures of size, sometimes called statistical diameters, in particular the chord lengths (also called intercept lengths) and caliper diameters. One of the most popular of these is the Feret diameter – the distance of two tangent parallel lines (usually maximum and minimum Feret diameter is used).

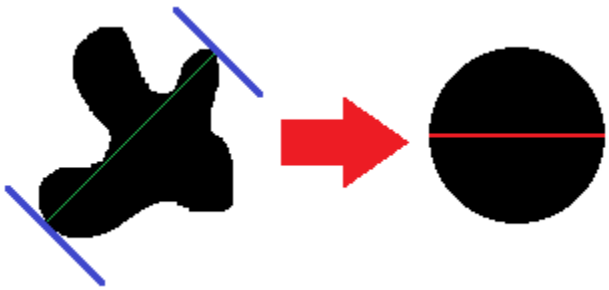


Fig. 2: Maximum Feret diameter (green thin line) and Heywood diameter (red thick) of a particle projection.

Size distribution

Particle systems (in a similar sense grains and pores or their sections in material microstructures) can consist of particles of only one size (monodisperse systems), but usually they consist of particle with different sizes (polydisperse systems). Polydisperse systems can be described by

distribution curves. Frequency curves show quantity (e.g. number or volume) of particles around a certain size; the size corresponding to the maximum on the frequency curve is called mode D_m. Discrete frequency curves are called histograms. Cumulative curves show the quantity (e.g. number or volume) of particles with size higher or lower than a certain size, e.g. $D_{30} = 7\ \mu$m means 30% of the particles (number or volume) is smaller than 7 µm. The most common quantile is the median D_{50}, which divides the population (e.g. in number or volume) of the particles into two halves with equal statistical weight. Another common quantiles are D_{10} and D_{90}, which together with the median can be used to calculate the span of the distribution $Span = (D_{90} - D_{10})/D_{50}$. Cumulative curves can be obtained by integrating frequency curves, frequency curves can be obtained by differentiating cumulative curves. Distribution curves can be number-weighted (f_0), length-weighted (f_1), surface-weighted (f_2) and volume-weighed (f_3). Number-weighted curves are the primary result of image analysis. It is also possible to obtain intensity-weighted (f_6) distributions (from dynamic light scattering / DLS). In order to compare the results of different methods, the same statistical weighting must be used.

When the particle shape is size-invariant, the recalculation from number-weighted to volume-weighted distributions is simply performed as follows

$$(q_3)_i = D_i^3 (q_0)_i \,,$$

where D is e.g. the area-equivalent diameter and the lower index (subscript) denotes the size class i.

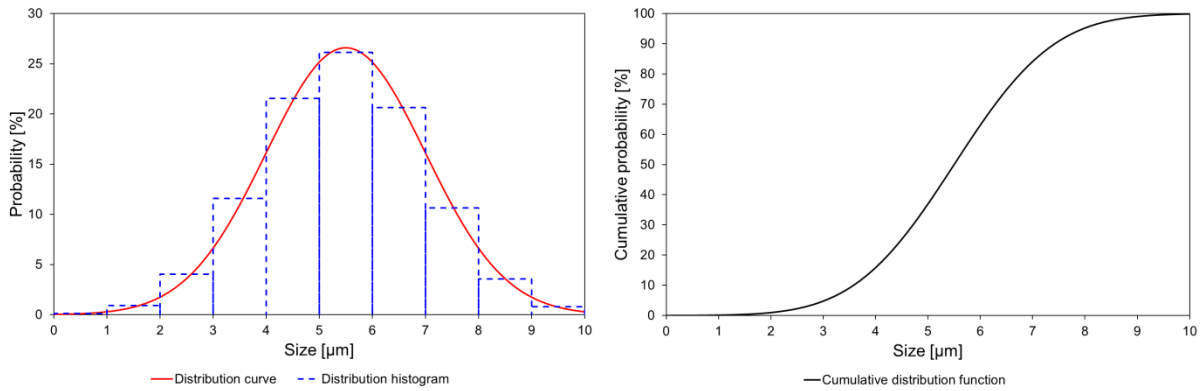


Fig.3: Comparison of distribution curve and histogram (left) and corresponding cumulative curve (right).

Microstructure analysis

Measurement of size distributions

There are two extreme types of microstructure – matrix-inclusion (inclusions are isolated and completely separated by matrix) and bicountinuous (phases have their own continuous space, f.e. foams prepared by replication method). Real microstructures can be also mix of these two extreme cases.

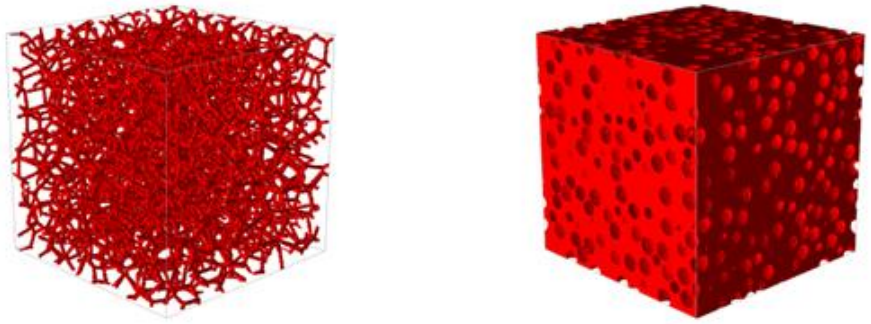


Fig.4: Ideally bicontinuous microstructure (left) and matrix-inclusion microstructure (right).

Only in matrix-inclusion microstructures it makes sense to determine size distributions (inclusions, pores or particles). In principal it is possible to measure it directly in 3D, if a spatial image or 3D model is available (e.g. obtained by computed tomography / CT), but most commonly it is performed on 2D sections by measuring areas of object sections and calculating the corresponding area-equivalent diameters. The problem is that the measurable 2D distribution on the image is usually not the same as the 3D distribution (the only exception being the special case of a Rayleigh distribution).

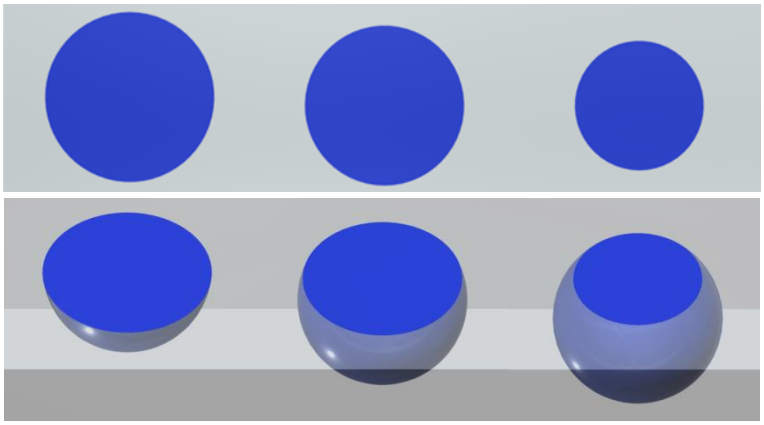


Fig.5: The 2D planar section (cut) through the microstructure shows sections with different sizes (top); however, the spatial image (3D model) shows that all spheres have the same size (bottom).

For spherical inclusions or pores it is possible to solve this problem (the so-called random section problem) by using Saltykov's matrix. This solution is based on the probability of section size which can be obtained from object of certain size and also on the probability that the section of that object will appear on the cut through the microstructure. Since the solution is based on the probability, the number of measured objects should be at least in order of thousands. The procedure itself is based on using a discretized distribution (histogram) of sphere section diameters per unit area n_j which is multiplied by Saltykov's matrix (Tab.1) A_{ij} and thus the distribution of sphere diameter per unit volume N_i is obtained.

$$N_i = A_{ij} n_j$$

Tab.1: Saltykov's matrix

	n_1	n_2	n_3	n_4	n_5	n_6	n_7	n_8	n_9	n_{10}	n_{11}	n_{12}	n_{13}	n_{14}	n_{15}
N_1	1	-0.1537	-0.036	-0.013	-0.0061	-0.0033	-0.002	-0.0013	-0.0009	-0.0006	-0.0005	-0.0004	-0.0003	-0.0002	-0.0001
N_2	0	0.5774	-0.1529	-0.042	-0.0171	-0.0087	-0.0051	-0.0031	-0.0021	-0.0015	-0.001	-0.0009	-0.0006	-0.0006	-0.0004
N_3	0	0	0.4472	-0.1382	-0.0408	-0.0178	-0.0093	-0.0057	-0.0037	-0.0026	-0.0018	-0.0013	-0.001	-0.0007	-0.0007
N_4	0	0	0	0.3779	-0.126	-0.0386	-0.0174	-0.0095	-0.0058	-0.0038	-0.0027	-0.002	-0.0016	-0.0012	-0.0009
N_5	0	0	0	0	0.3333	-0.1161	-0.0366	-0.0168	-0.0094	-0.0059	-0.004	-0.0028	-0.0021	-0.0016	-0.0013
N_6	0	0	0	0	0	0.3015	-0.1081	-0.0346	-0.0163	-0.0091	-0.0058	-0.0041	-0.0028	-0.0022	-0.0016
N_7	0	0	0	0	0	0	0.2773	-0.1016	-0.0329	-0.0155	-0.009	-0.0057	-0.004	-0.0029	-0.0022
N_8	0	0	0	0	0	0	0	0.2582	-0.0961	-0.0319	-0.0151	-0.0088	-0.0056	-0.0039	-0.0028
N_9	0	0	0	0	0	0	0	0	0.2425	-0.0913	-0.0301	-0.0146	-0.0085	-0.0055	-0.0039
N_{10}	0	0	0	0	0	0	0	0	0	0.2294	-0.0872	-0.029	-0.014	-0.0083	-0.0054
N_{11}	0	0	0	0	0	0	0	0	0	0	0.2182	-0.0836	-0.028	-0.0136	-0.008
N_{12}	0	0	0	0	0	0	0	0	0	0	0	0.2085	-0.0804	-0.027	-0.0132
N_{13}	0	0	0	0	0	0	0	0	0	0	0	0	0.2	-0.0776	-0.0261
N_{14}	0	0	0	0	0	0	0	0	0	0	0	0	0	0.1925	-0.075
N_{15}	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0.1857
N	1	0.4237	0.2583	0.1847	0.1433	0.117	0.0988	0.0856	0.0753	0.0672	0.061	0.0553	0.0511	0.0472	0.0441

Stereological measurements

Stereology can be described as a procedure to quantitatively describe 3D microstructures by using lower-dimensional measuring probes (i.e. area, length or point probes). Stereological descriptors that can be determined from planar sections are called metric, while those that require volumetric probing are called topological (e.g. connectivity). If the descriptors are valid for the whole microstructure, they are global, otherwise they are local.

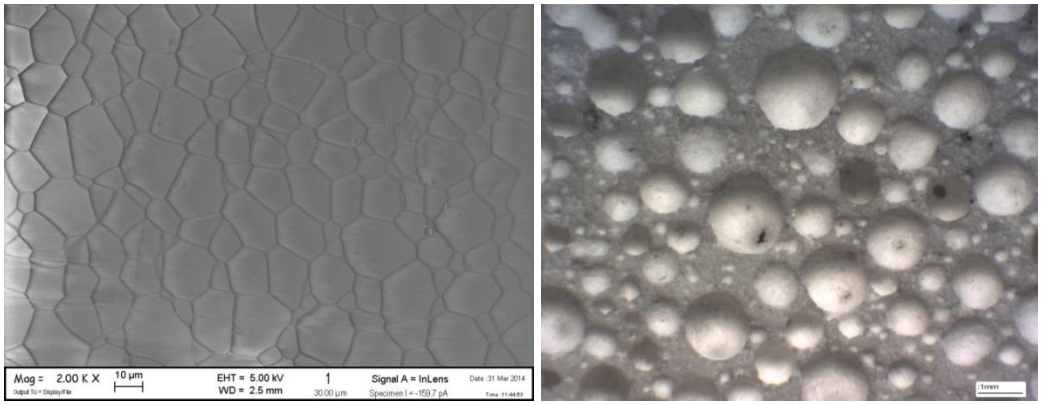


Fig. 6: Examples of microstructures: fully dense transparent yttrium aluminum garnet (left) and porous alumina (right).

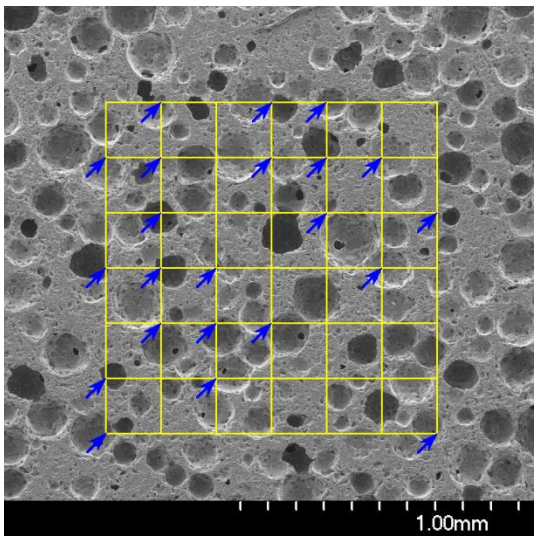
According to Hadwiger's theorem, there are only four independent Minkowski functionals: volume, surface, mean curvature and total curvature. Likewise for uniform isometric microstructures there are only four independent global descriptors – three metric: volume fraction V_V, interface density S_V, mean integral curvature density M_V; and one topological: total integral

curvature density K_V; all of the other parameters can be obtained by combining these independent global descriptors.

Only three independent global descriptors can be obtained from 2D section – V_V, S_V and M_V. If the microstructure of the sample is IUR – isotropic, uniform and random – it is possible to determine the volume fraction by using the Delesse-Rosiwal law

$$V_V = A_A = L_L = P_P \, .$$

Usually the point count method is the method of choice when manual measurement is used, because it allows a priori error predictions, but if the volume fractions are low, the measurement of line or area fractions is used instead. Automatic image analysis is usually based on area fractions.



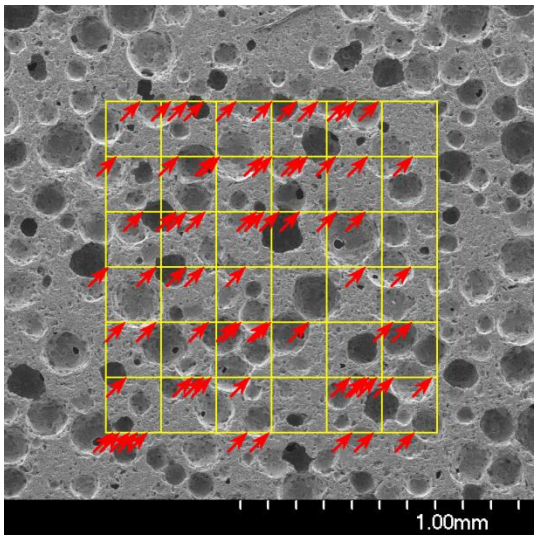
$P_P = \frac{22}{49} = 0{,}45$

The mean chord length $\bar{L}$ is generally connected with interface density and for multiphase materials it is defined as

$$\bar{L} = \frac{4\phi}{S_V} = \frac{\boldsymbol{2P_P}}{\boldsymbol{P_L}} .$$

The manual measurement consists of marking points, where the probe meets the interface and the point count is divided by the probe length - P_L. The point fraction P_P is the volume fraction as described above. For one-phase materials the mean chord length of grains is defined as

$$\bar{L} = \frac{2}{S_V} = \frac{\boldsymbol{1}}{\boldsymbol{P_L}} .$$



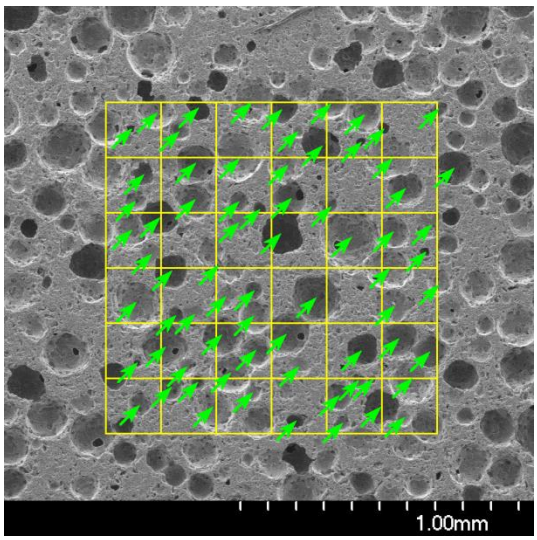
$P_L = \frac{70 \text{ points}}{8{,}4 \text{ mm}} = 8{,}33 \text{ mm}^{-1}$

The Jeffries size J is connected with mean integral curvature density:

$$J = \sqrt{\frac{2\pi P_P}{M_V}} = \sqrt{\frac{\boldsymbol{P_P}}{\boldsymbol{N_A}}}$$

The manual measurement consists of counting the objects in the area (with ignoring two neighboring sides and all three corresponding corners) and dividing the count by area - N_A. The Jeffries size for one-phase materials (Jeffries grain size) is

$$J = \sqrt{\frac{2\pi}{M_V}} = \sqrt{\frac{\boldsymbol{1}}{\boldsymbol{N_A}}}\,.$$



$N_A = \frac{70 \text{ objects}}{1{,}44 \text{ mm}^2} = 48{,}61 \text{ mm}^{-2}$

Experimental

1. Distribution of pore size
    a. Calibrate the image
    b. Try all available measuring shapes and select the most appropriate
    c. Split the results into size classes
    d. Use Saltykov's matrix to correct the random section problem
    e. Prepare graphs of number-weighted distribution (frequency and cumulative)
    f. Recalculate the results into volume-weighted distribution
    g. Prepare graphs of volume-weighted distribution (frequency and cumulative)
    h. Find modes, quantiles (D_{10}, D_{50} and D_{90}) and span of all distributions
2. Stereology of porous microstructure
    a. Calibrate the image
    b. Use grid with suitable grid parameter
    c. Determine volume fraction (= porosity)
    d. Determine mean chord length
    e. Determine Jeffries size
    f. Compare the two sizes with the results from part 1
3. Stereology of dense microstructure
    a. Calibrate the image
    b. Use grid with suitable grid parameter
    c. Determine mean chord length
    d. Determine Jeffries size
    e. Compare these two sizes

The laboratory report in MS Word should contain a short description of the measurement – principle and procedure, commented results and conclusions. The MS Excel file with source data is considered as part of the report. The report should be submitted within week via email to Tereza.Uhlirova@vscht.cz. The final grade takes into account the test results, quality of the work, quality of the report and its timely submission.

Recommended literature:

[1] Uhlířová T., Hostaša J., Pabst W.: Characterization of the microstructure of YAG ceramics via stereology-based image analysis, *Ceram. Silik.* 58 (3), 173-183 (2014).

[2] Uhlířová T., Gregorová E., Pabst W., Nečina V.: Preparation of cellular alumina ceramics via biological foaming with yeast and its microstructural characterization via stereological relations, *J. Eur. Ceram. Soc. 35* (1), 187–196 (2015).

[3] Pabst W., Gregorová E., Uhlířová T.: Microstructure characterization via stereological relations – a shortcut for beginners, *Mater. Charact.* 105 (1), 1–12 (2015).

[4] Uhlířová T., Pabst W., Gregorová E., Hostaša J.: Stereology of dense polycrystalline materials – from interface density and mean curvature integral density to Rayleigh distributions of grain sizes, *J. Eur. Ceram. Soc.* 36 (9), 2319–2328 (2016).

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[7] Gregorová E., Pabst W., Bohačenko I.: Characterization of different starch types for their application in ceramic processing, *J. Eur. Ceram. Soc.* ***26***, 1301–1309 (2006).

[8] Pabst W., Berthold C., Gregorová E.: Size and shape characterization of oblate and prolate particles, *J. Eur. Ceram. Soc.* ***27***, 1759–1762 (2007).

[9] Pabst W., Uhlířová E.: A generalized class of transformation matrices for the reconstruction of sphere size distributions from section circle size distributions, *Ceram. Silik.* **61** (2), 147-157 (2017).

[10] Gregorová E., Uhlířová T., Pabst W., Diblíková P., Sedlářová I.: Microstructure characterization of mullite foam by image analysis, mercury porosimetry and X-ray computed microtomography, *Ceram. Int.* **44** (11), 12315-12328 (2018).