Characterization of the microstructure of filter materials



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Filter materials

Non-woven, Fraunhofer Institut Zerstörungsfreie Prüfverfahren (IZFP), resolution 10µm

- fiber material (non-woven or felt, homogeneous or in layers)
- open foams (polymer, metal or ceramics)
- sinter material



Polyurethan foam, RJL Analytic, resolution 5µm



Sintered copper, Bundesanstalt für Materialprüfung, resolution 14µm



Types of microstructures and constituents

layers or spatially homogeneous



fiber felt, European Synchrotron Radiation Facility (ESRF), resolution 7µm



Non-woven, ESRF, resolution 1.4µm



Why 3d images?

Filter materials are often

- strongly porous (porosities > 95% are possible)
- weak (e.g. non-woven)







Nickelfoam, IZFP, resolution 10µm

Classical materialographics reaches its limits, as

- preparation of planar sections or cross sections is difficult or even impossible
- 2d images contain only a small fraction of the information





Goals of geometric analysis of microstructures



- geometric characterization for
 - o quality control,
 - o comparison and
 - o choice of materials



- preparation of image data for direct use in simulations (macroscopic materials properties)
- fitting of modells: parameters are deduced from geometric characteristics of the microstructure (virtual material design)



Goal: virtual material design

3d analysis opens new opportunities like

- fitting of three dimensional geometric models
- computing macroscopic material properties (permeability, filtration, stability...) using numeric simulations in the modell or the preprocessed 3d image
- determining the optimal material parameters for the desired properties



Flow of air through a non-woven, flow simulation A. Wiegmann, ITWM, visualisation C. Lojewski, ITWM modeling: GeoDict



Field features (Minkowski functionals)



 $L_v = 23.3 \text{mm}^{-2}$



 $\chi = 133$

Volume V, surface S, integral of mean curvature M, integral of total curvature K (Euler number $\chi = 4\pi K$)

Interpretation:

- fibers $L_V = \frac{M_V}{\pi(1 V_V)}$ (specific fiber length)
- isolated particles Euler number = number of particles
- open foam $\frac{S_V}{\pi L_V}$ mean thickness of edges

Theorem (Hadwiger, 1957): Every nonnegative, motion invariant, monotonously increasing, c-additive functional on the convex bodies is a linear combination of the Minkowski functionals. However, Minkowski functionals do not describe all features of a microstructure. (Baddeley, Averback, 1983): example with same Minkowski functionals, completely different tortuosity.



Crofton's intersection formulae

Minkowski functionals from intersections with planes/lines/points, measurement of the Minkowski functionals by counting

$$\begin{array}{rcl} d=3 & d=2 & d=1 & d=0 \\ \hline V(X) &=& \int A(X \cap E_{r,\omega_0}) \, dr &=& \int L(X \cap e_{y,\omega_0}) \, dy &=& \int \chi(X \cap \{x\}) \, dx \\ S(X) &=& \frac{4}{\pi} \int \int L(X \cap E_{r,\omega}) \, dr \, \mu(d\omega) &=& 4 \int \int \chi(X \cap e_{y,\omega}) \, dy \, \mu(d\omega) \\ \hline M(X) &=& 2\pi \int \int \chi(X \cap E_{r,\omega}) \, dr \, \mu(d\omega) \\ \hline E_{r,\omega} - \text{plane at distance } r \text{ from the origin, direction } \omega \\ e_{y,\omega} - \text{ line with intersection point } y \text{ in plane with direction } \omega \\ \mu - \text{ invariant measure on } \mathbb{S}^2 \end{array}$$



Analysis of open foams



- measurement of field features, compute e.g. mean length of edges per unit volume
- model based computation of quantities relevant for application (e.g. mean size of cells, ppi value)
- segmentation/reconstruction of the cells, measurement of pore sizes
- spectral analysis



Characteristics of the edge system from Minkowski functionals

Example: nickel foam

Estimated densities of the Minkowski functionals:

$$V_V = 14.5\%$$

 $S_V = 7.54 \text{mm}^{-1}$
 $M_V = 57.6 \text{mm}^{-2}$

Porosity: $p = 1 - V_V = 85.5\%$

Edge density (total edge length per unit volume): $L_V = \frac{M_V}{\pi (1 - V_V)} = 21.45 \text{mm}^{-2}$ Mean edge thickness: $\frac{S_V}{\pi M_V} = 111.9 \mu \text{m}$ Mean section area of the edges: $\frac{V_V}{L_V} = 6750 \mu \text{m}^2$



Open foams as edge system of a tessellation

Tessellation – partitioning of space in cells





Models for open foam should

- be macroscopically homogeneous, microscopically heterogeneous
- have predominantly pentagonal faces
- be analytically describable.



Random Voronoi tessellations

Step 1: Generate cell centres

Step 2: Centers grow uniformly. Growth stops when touching. Each center is assigned its influence region.

Poisson Voronoi: Centers are i.i.d. uniform.

Drawback: cells not sufficiently regular

Hard-core Voronoi: centers have minimal distance R

Drawback: no analytic description, too many faces with 4 vertices





Deterministic tessellations

Desired: tessellation by regular dodecahedra

Problem: pentagonal dodecahedra are not space filling





Alternatives: Kelvin foam – identical cells (truncated octahedra)

Weaire-Phelan foam

Drawback: microscopic heterogeneity not captured



The Weaire-Phelan foam

2 types of cells: pentagonal dodecahedra and polyhedra with 14 faces (2 hexagonal and 12 pentagonal)

Proportion of the 2 types: 2:6

A version of the Weaire-Phelan foam with slightly bend faces is the currently best known solution for Kelvin's problem – minimal surface of equal volume cells.



Images: Ken Brakke, www.susqu.edu/facstaff/b/brakke/



Measurements under model assumptions

Example: nickel foam, Weaire-Phelan model, in braces Poisson-Voronoi tessellation

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mean number of cells per unit volume8.1 \text{mm}^{-3} (7.0 mm^{-3})mean cell volume0.123 \text{mm}^3 (0.142 \text{mm}^3)ppi (pores per inch)67.5 (70.8)mean cell diameter0.741 \text{mm} (0.761 \text{mm})mean diameter of faces0.381 \text{mm} (0.374 \text{mm})mean area of faces0.099 \text{mm}^2 (0.102 \text{mm}^2)
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Segmentation/reconstruction of the cells

Step 1: binarization

Step 2: distance transformation – assigns each background pixel its distance to the edge system

Step 3: supression of "wrong" maxima

Step 4: watershed transform – interpretation of greyvalues as topographic depths, "flooding" of the catchment basins

Step 5: superposition with original edge system

Step 6: measuring features of cells (e.g. size)





Pore size distribution



Example: nickel foam

Pore size = diameter of ball of equal volume

Pores cutting the edge are not considered (size biased sample), therefore the volume weighted distribution is given.



Spectral analysis

Covariance of the open nickel foam, local maximum at 0.4mm corresponds to diameter of faces.







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Analysis of fiber material

Essential characteristics of the fiber system: specific fiber length and directional distribution





Directional distribution of the fibers

Directional information is contained in generalized **projections** when computing the integral of mean curvature (Crofton formulae)

$$\frac{M(X)}{2\pi} = \iint \chi(X \cap E_{r,\omega}) \, dr \mu(d\omega) = \int \wp_M^{\omega}(X \cap E_{r,\omega}) \, \mu(d\omega)$$

Directional distribution – distribution of the direction in the typical fiber point, connection to projections \mathbf{Y}_{1}

$$\wp_M^{\omega}(X) = \int_{S^2} |\langle \omega, u \rangle| \, \vartheta(du).$$

Discrete inversion of the cosine transformation: linear program (Kiderlen 2000, result discrete) or approximation by finite expansion in spherical harmonics (Legendre polynomials, result with smooth density)





Characterization of the pore space in fiber material

Problem: no clear definition for "pore"

Possibilities for characterization:

- 1. Spherical contact distribution distribution of the distance to the nearest fiber
- 2. Granulometry distribution sieving of pores with growing mesh size using morphological openings with growing structuring element
- 3. Percolation measure fraction of pore space being for balls of growing size
- 4. Tortuosity mean length of paths inside the pore space/thickness of the material



Modeling of Microstructures

modeling of microscopically heterogeneous but macroscopically homogeneous material

- measure geometric characteristics

 choose a model (basis: spatially homogeneous models from stochastic geometry)

-determine model parameters (fit the model)

- goal: optimal acoustic absorption







Modeling of a stacked fiber non-woven

Known: pressed non-woven, long fibers, fiber radia

Image data: confocal laser scanning microscopy, 2D sections

Model: cylinders, centres forming a Poisson line process with a one parametric directional distribution

Parameters to determine: length intensity, anisotropy parameter





Directional distribution

Isotropy: direction given by point uniformly distributed on sphere, that is longitude uniformly distributed, latitude weighted by $\sin \theta$

Pressing: directions near the equator preferred

Density: $\frac{1}{4\pi} \frac{\beta \sin\theta}{\left(1 + (\beta^2 - 1)\cos^2\theta\right)^{\frac{3}{2}}},$ $\theta \in [0, \pi), \phi \in [0, 2\pi).$







Estimation of the model parameters

Known: fiber section profiles, porosity

To estimate: anisotropy parameter from intensities of fibers in parallel and orthogonal planar sections

Estimation of the fiber intensity using an edge corrected component count

Result: 2.93 – intermediate degree of pressing

Preparation and light microscopic images, C. Maas, Institute for Materials Science, Universität des Saarlandes



Segmentation: Canny edge detection, region growing



Virtual material design

- 1. (3d) image of the microstructure
- 2. Fitting a geometry model
- 3. Numeric simulation of macroscopic materials properties (permeability, acoustic absorption, stability...)
- 4. Change of model parameters (e.g. fiber profiles or thickness)
- 5. Iteration of 3. and 4.



optimization





Products: current - a4iL (C library for Linux)

soon (10/2004) – MAVI, MAVI-OpenFoam (with GUI, system independent)



Challenges

Image aquisition: resolution, absorption contrast

Technical:

image/data sizes (2GB, soon 16GB), visualization

Mathematical/algorithmic:

difficulties/ambiguities when generalising 2d algorithms efficiency (due to image sizes) new tasks (e.g. pore size distribution in fiber materials) segmentation





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