Pore structure and effective permeability of metallic filters

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Abstract. The pore structures (microstructures) of two metallic filters were reconstructed using the stochastic reconstruction method based on simulated annealing. The following microstructural descriptors were included in the description of the real microstructures: the two-point probability function, the lineal-path functions for the void or solid phases, i.e. simulated annealing was constrained by all low-order statistical measures that were accessible through the analysis of images of polished sections. An effect of the microstructural descriptors on the course of reconstruction was controlled by modifying two parameters of the reconstruction procedure [1]. Their values resulted from repeated reconstruction of two-dimensional microstructures in such a way that the reference (experimental) and calculated two-point cluster functions deviated negligibly. It was tacitly assumed that the parameters adjusted during two-dimensional reconstruction had the same influence on the formation of the three-dimensional microstructures. Since connectivity of phases is a critical property of the stochastically reconstructed media, clusters of pore and solid voxels were determined using the Hoshen-Kopelman algorithm. It was found that the solid phase formed one large cluster in accordance with the physical feasibility. The void phase created one large cluster and a few small clusters representing the isolated porosity. The percolation properties were further characterised using the local porosity theory [2]. Effective permeability of the replicas was estimated by solving the Stokes equation for creeping flow of an incompressible liquid in pore space. Calculated permeability values matched well their experimental counterparts.

1. Introduction
The replicas like artificial representative 3D models of real microstructure of porous solid material are created by means of several classes of methods. The serial sectioning method [3] or focused ion beam lift-out technique [4], X-ray computed microtomography [5], process-based methods [6,7], and stochastic reconstruction methods can be categorized into basic list. The last considered class belongs to the group of statistical methods and appears as more economical alternative to the other reconstruction methods.

The stochastic reconstruction method based on the simulated annealing (SRSA) [8] generally provides quite realistic information on microstructure of porous materials. Nevertheless, the connectivity of pore space in some generated replicas is not reproduced in long-range scale sense so well; see [9,10]. This is the case of the metallic porous filters where the improved SRSA method was used to enhance long-range pore connectivity of their replicas.
2. Experimental

2.1. Porous metallic filters and preparation of samples
Porous metallic filters from Mott Corporation, USA, in the form of flat disc made from stainless steel were used in two variants differing in their textural properties [11]. The cross-sections in the appropriate various plane orientations were prepared from hardened epoxy resin blocks (vacuum impregnation method) by their cutting, lapping, grounding, and polishing.

2.2. Image acquisition and image processing
The resulting polished cross-sections were monitored in the scanning electron microscope (SEM JSM−5500LV, JEOL, Japan). Thereafter, series of back-scatter electron images from various locations of the considered cross-sections in uniform size and resolution were collected. The grey-scale images were underwent to the nonlinear filtering through the adaptive median and percentile filters. The Otsu’s segmentation algorithm of global thresholding was then applied to separate void (pore) and solid phases, respectively. Value of threshold was adjusted on the basis of information obtained from textural analysis (helium pycnometry, mercury porosimetry).

2.3. Microstructure and microstructural descriptors
Binary images of randomly selected parts of porous media represent 2D digitized medium in form of the indicator phase function; the void phase is labeled as 1 and the solid phase as 0. The relation between various shapes of both phases depicted on every 2D plane is captured in the microstructural descriptors which were derived from series of 2D binary images. The two-point probability function for the void phase, $S_2(u)$, the lineal-path function for both phases, $L^p(u)$, $L^m(u)$, respectively, were calculated for mentioned porous metallic filters. The detailed definition of all used microstructural descriptors can be found in [12]. The course of selected microstructural descriptors derived from the images of polished sections is common in 2D and 3D spaces. Therefore, they were used as starting data (reference descriptors having an origin from the real porous material) for the computational reconstruction algorithm.

2.4. Stochastic reconstruction of porous metallic filters
Stochastic reconstruction of porous material by means of simulated annealing method constrained by three microstructural descriptors, $S_2(u)$, $L^p(u)$, $L^m(u)$ was based on minimization of the objective function $E$, i.e. “energy” of the digitized system

$$E = e_0 + e_1 + e_2 \frac{\eta}{\eta + e_0 + e_1}$$

where $\eta$ is a given parameter to alter the weight of lineal-path function for solid phase during time of minimization (1). All used microstructural descriptors were calculated in principal and diagonal directions, under consideration of periodic boundary conditions.

In the principle, the random configuration of solid and void voxels (volume elements) was generated in 3D space on a simple cubic lattice (320×320×320). Consequently, this high-energy uncorrelated microstructure was gradually transformed into a more correlated microstructure with minimal energy content, see figure. The pair of voxels, the first voxel from one and the second from opposite phase, was selected randomly in each iteration step $k$ and their phases were mutually changed. The energy difference $\Delta E$ of two states of whole microstructure (before and after this change, respectively) was determined. Thereafter, the more suitable energetic state was accepted with
the probability resulting from Metropolis rule [13]. The decision to accept/reject is dependent on $\Delta E$ and system “temperature” $\Theta_k$. The considered temperature was kept constant, when sufficient number of new states had been generated. The temperature was decreased according to the annealing schedule. The two-point cluster function was used indirectly for adjustment of two special parameters $\eta, \zeta$ of our reconstruction procedure. The second parameter $\zeta$ controls the voxels selection. Other details on the modified method and on the optimal adjustment of parameters $\eta, \zeta$ are detailed described in [1].

\begin{figure}
\centering
\includegraphics[width=\textwidth]{figure}
\caption{Relation between the temperature of porous digitized system $\Theta_k$ and elapsed time during stochastic reconstruction on simulated annealing (increased correlation of voxels, cross-sections).}
\end{figure}

3. Results and discussion

Two real samples of the porous metallic filter (labeled as F1/F2) differed in total porosity (17.8 %, 20.1 %) and pore size distribution (median pore diameter 3.55/2.70 microns) were reconstructed by means of the modified SRSA method. Five replicas were generated for each porous material. Their percolation properties were characterized through the 3D descriptors defined in terms the local porosity theory [2]. Considered descriptors were local porosity distribution, the local percolation probability and the total fraction of percolating cells in the selected directions.

It can be summarize that both kinds of replicas formed large percolating clusters in the void (pore) and solid phases, respectively. The volume fraction of non-percolating void phase was very small (for instance, its value for the sample F1 is 0.0103 versus calculated total porosity 0.1849).

[9] who employed in their study the standard SRSA method, mentioned the existence of non-percolating clusters and poor connectivity of pores in sandstone rock samples as the result of artificial anisotropy in replicas. On the other hand, the adaptation of the objective function as well as the dynamics of the reconstruction of porous material (performance of this algorithm during time of reconstruction) played a significant role in the modified SRSA method. Additive constraint of the lineal-path function for metallic phase $L^\omega(u)$ into the objective function $E$ (see equation (1)) had a positive impact on the pore connectivity of both metallic replicas. These replicas became more penetrable without small isolated clusters in the metallic phase. The choice of microstructural descriptors calculation in the more orientations (principal and diagonal) supported the formation of metallic microstructures free of the rectangular-like shapes typical for using of descriptors evaluated only in principal directions. The next benefit for the improving of the percolation properties of reconstructed replicas consists in a deceleration of cooling schedule during time of the SRSA, already suggested by [9]. In the case of low-porosity porous material, above described modified SRSA method...
led to improving of percolation in all considered principal directions compared with the commonly used SRSA (parameter $\eta = 0$, thus the objective function $E = e_0 + e_1$).

The effective transport properties of the replicas of porous metallic filters were estimated for creeping flow of an incompressible liquid in pore space. Together with the balance (equation of continuity), no-slip boundary condition and considered pressure difference on the opposite walls of replica, the Stokes equation was solved with aid “permsolver”, the public domain software [14]. The effective permeability $\beta$ was derived from the mean survival time calculated as average time of random walker travelling in the pore space of metallic replica [1].

Calculated permeability values matched well their experimental counterparts obtained from permeation measurements with inert gases in Wicke-Kallenbach cell working under quasi-stationary conditions [11]. The correlation between pore connectivity and effective transport properties was clearly demonstrated on the replicas generated by means of the modified SRSA method. The results following from the local pore analysis and the cluster statistics were then confirmed.

4. Conclusion
The reconstruction of real porous metallic filters verified assumption of the pore connectivity improving in low-porosity materials. The both replicas constituted large clusters of the porous and metal phases, whereas non-percolating isolated clusters took only negligible fractions of both phases. Consequently, the modified SRSA method was capable to reproduce good connectivity. The 3D replicas generated from limited statistical information obtained from 2D micrographs can serve for evaluation of various effective properties included elastic modulus, fluid diffusivity, and permeability.

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