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Possibilities of porous-structure representation – an overview

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Abstract: Porous media can be found in all areas of scientific life, such as medicine, civil engineering, material science, fluid dynamics. Computing has achieved high efficiency and computational capacity – so far. However, three-dimensional Computational Fluid Dynamics (CFD) simulations of microstructure remain significant challenges. Pore-scale simulations can help understand the physical processes and determine macroscopic parameters such as the high-frequency limit of dynamic tortuosity, viscous, and thermal characteristic lengths. Independent of whether the computational problem is two or three-dimensional, the geometry as input parameter must be prepared. For this reason, geometry representation methods play a crucial role in the analysis at the pore-scale, especially in numerical simulations. In this article, an insight into microstructures' visualization capabilities is provided essentially for CFD simulations.

Keywords: Pore-scale simulation; Micro-structure, Porous-structure, Microscopy

1. Introduction

Researchers and engineers are mainly concerned with fluid flow and transport phenomena on a scale that is much larger than atomic; the pore-scale is commonly adopted in practice [1]. Fluid flows at pore-scale are detailed by the equation found on the law of Darcy [2] [3], together with the transport of mass by the advection-dispersion equation [4], in which bulk averaged fluxes satisfy the two equations [4] [5]. A correct and thorough understanding of larger-scale flow and transport processes requires knowledge of micro-scale processes, highly dependent on input porous media' geometrical details [6-9]. For example, simple stagnant zones can influence the transport phenomena and dispersive mixing [4]. Today, multiple flow and transport problems [4] in porous material necessary to be studied with the help of different numerical approaches, which allows the analysis of various processes and phenomena [2] in the microstructure. Moreover, their impacts on a larger scale (i.e., fluid flow behavior at the macro-scale) also must be studied using these methods. Therefore, the so-called "linking" between specific scales plays more and more significant role in the industrial field despite the reachable computational power, which heavily limits the spread of multi-scale methods [10] [11].

The natural pore materials have unique geometries, which are incredibly elaborate and enormously complicated. Therefore, solving the governing equations (Navier-Stokes or Stokes in case of Creeping flow) for actual pore materials is absolutely a challenging and exciting computational fluid dynamics (CFD) simulation task currently. Hence different numerical approaches are suited to handle real porous geometries. Therefore, it is needed to develop the simulation methods for pore-scale analysis [4]. With the continuous improvement of various visualization techniques, which can also imagine the internal structure [12] [13], and micro-fluidics experiments, more pore-scale physics and higher spatial resolution can be obtained [14-18]. The area of fluid flow and transport in porous materials [2] (micro-structures) has been completely changed by our ability to create images, in three dimensions, of porous media with modern microscopes at several resolution ranges [6] [9] [19]. Recent modern advances of various imaging techniques, which can represent the material's specific geometry with the complex inside-structure, can help in numerical simulation (e.g., CFD-simulations) with negligible loss in pore topologies and porous media [20-22]. It should be noted that the other imaging methods are also used for simulation, which cannot present an image of the inside structure of the material.

2. Theory of Porous Media

The pore-scale is characterized by the solid grains or body (black parts) of the porous material as well as the pore-spaces, which contain fluid (blue parts); Fig. 1. shows an example. If the pores are closed and not connected with each other, representing the whole solid by a macroscopic material law is ordinary practical. In a way, the material attributions are 'smeared' over the solid frame and its pores. However, a too 'coarse' definition might affect neglecting any probably critical effects [4].

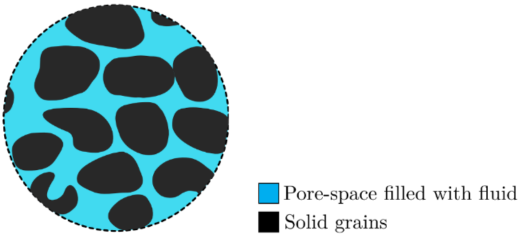

Figure 1. Schematic illustration of a pore-scale porous medium [4] [23]

Microscopic pores and cracks can have a significant influence on macroscopic behavior [20]. At all points of the present scale, either a fluid (marked with blue) or a solid phase (marked with black) has definite phase-boundaries. The fluid placed in the void spaces could be specified with several elemental quantities like density (ρ), kinetic (ν) and dynamic viscosity (η). Since the void boundaries' features are applicable, it is feasible to directly taking the particular void space geometries and merging with the correct boundary conditions in order to detail the flows of fluids – solution of the conservations of mass, momentum, and energy – in the pore space [4]. Fig. 2. shows two examples of porous media in the field of material science [1][20].

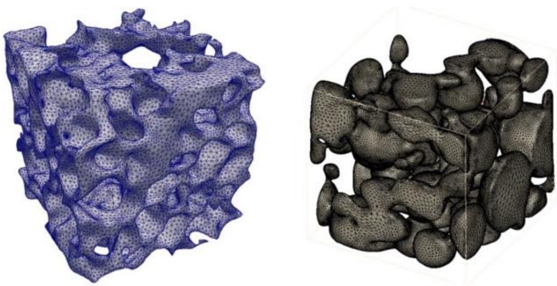

Figure 2. Two numerical models of porous media in the field of engineering [1] [20]

The porous medium simulation of fluid flow and transport could be divided into two types separated by scale. Simulating the fluid-transport inside the individual pores constituting the porous material's pore space is the first type. In this category, methods solve the so-called "governing equations of motion" inside the pore space where pores' wall treatment is a no-slip boundary. This first type is denoted as pore-scale simulations. The second type treats larger scales where the definition of averaged effective-properties such as porosity and permeability are possible. However, these are up-scaled properties from the smaller scales of porous media, and the physics and flow that take place on the pore-scale ultimately determine how larger-scale flows must be treated [1].

Generally, the porous material contains a solid skeleton or matrix within a massive number of microscopic voids/pores. The tiny porous inside the structure are commonly attached. For this reason, different flow and transport processes can happen inside. Natural substances are good examples, such as rocks, soils, sandstone, and artificial or industrial materials. Porous media is commonly applied in the field of engineering or sciences like biophysics, geosciences, or material science. Thus, it is incredibly critical in the research and application of fluid transport and flow through them. The flow and transport processes are affected by the pore structure, the matrix's physical characteristics, and the fluid-molecules (in the pore-space) [4].

These processes are treated as quite complicated due to the complexity of micro-structures. The pores mostly have irregular surfaces; therefore, fluid flow through them is genuinely problematic. Several pores make dead ends and have a severe effect on the flow and transport behavior. In single-phase flow through porous materials, just one fluid phase is flowing over the pores, e.g., water. Multi-phase flow or two-phase flow is in the case where multiple phases are flowing over the void spaces. Multi-phase flow can be present in unsaturated zones. Such as a petroleum recovery of hydrocarbons and various industrial contexts [4].

3. Porous-Structure Representation

New perspectives on the study of microstructures are being opened by modern three-dimensional imaging methods/tools capable of reconstructing porous materials' structure [6] [9] [19]. Different modern imaging techniques are able to provide high-fidelity visualization [24] and characterization of 2-D/3-D porous micro-structures [25] such as pore topologies with negligible loss, i.e., affordable computational resources. The digital micro-structure can be applied for a variety of simulation or diagnostic purposes [6] [9].

3.1. Two or Three-Dimensional Methods without Internal Structure

The commonly used techniques to provide 2-D (surface) or 3-D (without internal structure) visualizations of micro-structures are summarized in Table 1 [9] [14]. The methods are the Scanning Tunneling Microscopes (STM) [26], the Scanning Electron Microscopes (SEM) [27-28] (it has Backscattered Electron (BSE) mode [28]), the Optical Microscopes (OM) [14], the Scanning Probe Microscopes (SPM) [26] (Subtype: Atomic Force Microscope (AFM) [29]), and Confocal Laser Scanning Microscopes (CLSM) [30].

Table 1. 2-D (surface) and 3-D (without internal structure) geometry representation methods [6] [7] [9] [12-14] [19] [25-36] [38] [39]

Method	***Range***	***Resolution***	***Feature***
STM	500 × 500 nm	0.1 nm – 30 nm	• STM represents the surface, which makes it possible to analyze a considerable number of characteristics, e.g., surface defects, roughness, and inspecting the molecule's feature such as conformation and size;
SEM	10 × 10 ×10 μm	1 nm – 10 mm	• SEM is an efficient tool for characterization of particle size at the surface, but it does not fit for pore size; • Backscattered electrons produced at each point are collected, with practical electronics to imaging on the computer;
OM	50 × 50 μm	0.1 μm – 1.0 mm	• OM is the primary technique used for the analysis of the surface profile. • It is required for quantitative studies as it provides the base of image analysis systems illustrations/visual documentation;
SPM	10 × 10 μm	0.1 nm – 70 μm	• SPM has excellent resolution (spatial) and can imaging the structure and the surface topography also; • AFM is an effective technique of SPM for single-cell characterization;
CLSM	400 × 400 × 0.8 μm	180 nm – 1.5 mm	• CLSM has a unique ability to provide 3D images of the porous media with submicron resolution; • Although CLSM is greatly limited in depth (z-dimension);

Scanning Tunneling Microscopy (STM) is a type of electron microscopy. It is capable of producing an image of a sample's surface at the molecular scale [26]. The STM's fundamental is based on quantum tunneling theory, Fig. 3 shows the schematic representation. Unlike SPM techniques (such as AFM), this technique normally not requires contact with the specimen's surface, although some volts or millivolts are used between the specimen and the tip, while the current of tunneling is measured [40]. Just after the conducting tip is appropriately positioned, several angstroms from a semiconducting or metallic surface, a bias voltage is used among the surface. The probe tip makes it possible to tunnel electrons via the gap. When the tip scanning over the surface, the fluctuation in the tunneling current is registered. Therefore, it can create topographical images of the sample's surface. [26] The STM's regular depth and lateral resolutions are 0.1 and 0.01 nm. STM is a well-known technique, which is surface-sensitive, and unlike TEM, it requires a definitive clean surface, so it has a low success rate [26] [40] [41].

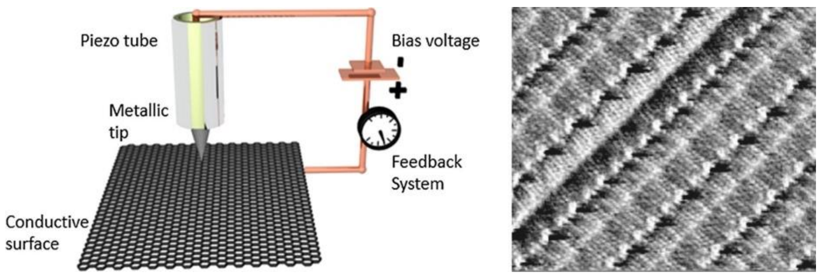

Figure 3. Schematic representation of an STM device and an STM-image [41]

Scanning Electron Microscope (SEM) is able to produce much information like surface (2D) structures, topographies, crystallines, and chemical compositions [28]. Present SEM-techniques are considered to fall within the general class of electron microscopes, which create 2D-images of different samples. In terms of its operation, illustrated in Fig. 4., the surface is scanned with a focused electron beam [42]. The electrons – used for scanning – have interaction with the sample's (investigated material) atoms. This process provides numerous data about the composition of the whole sample and the surface's topography (also see Fig. 4) [27]. The scanning process of the beam of electrons follows raster-scan-patterns, plus the beam's position relates to the intensity of the detected signal, which can produce two-dimensional images [38]. Backscattered Electron (BSE) operation mode has been commonly used to characterize phases with diverse chemical bases in various materials [28]. Moreover, BSE is generally used to study porous-structure substance in which differences in atomic number are expected [27]. Thus, backscattered images can be a proper method to characterize metallic phases [42].

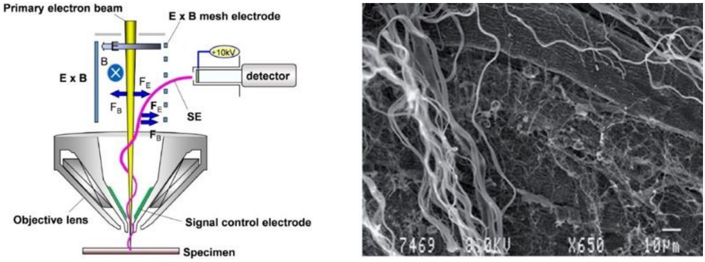

Figure 4. Schematic illustration of SEM signal detection and an SEM image [43-44]

Optical Microscopy possesses a wide field of view, ensuring a high throughput screening and the possibility to collect vast amounts of data (see Fig. 5) for post-processing and machine learning [35]. In the case of optical microscopy techniques, the preparation of the sample is not necessary [45]. These methods have low applicability in 3D-CAD modeling because it requires comprehensive data manipulation. For example, a high resolution of the sample requires to be split into fragile slides [14]. This method is profoundly labor-intensive and resulting 2D images, which need to be digitally processed to build the 3D images [46]. If the sample is inhomogeneous, this time-consuming process can provide the best solution to get a 3D image for CFD simulations. This method is hugely computation and memory demanding; therefore, it is incredibly challenging nowadays for computers to reach a level capable of identifying cells by optical characteristics [25].

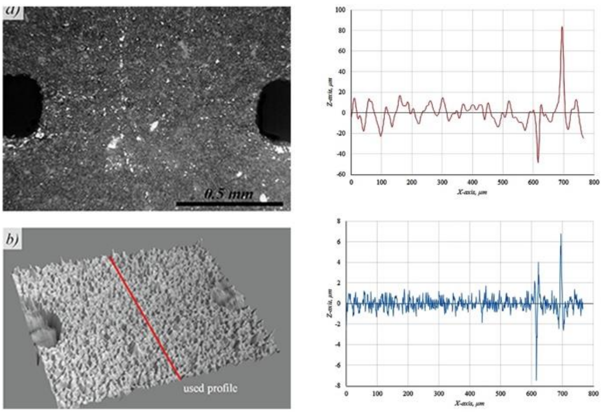

Figure 5. Fully focused optical image (a) and 3D rendering of surface morphology (surface profilometry) (b), the waviness profile (c) and roughness profile (d) taken from line profile across the image [47]

The scanning probe microscopes (SPM) can give several pieces of information about the material sample at the atomic scale. One of the SPMs is the atomic force microscopy (AFM) with extremely high resolution. Its application's main reasons are the following: the powerful piezoelectric actuation for the precise tip/sample driving; applying sharp tip; controlling with rapid feedback for tip-sample interaction defined by the user. The control of the movement of the tip/sample is possible at the picometer scale [26]. With this method, surface topology and intermolecular forces can be directly measured with a resolution of ≈ 0.1 nm vertically. AFM scans the surface of the sample with a sharp probe and collects information about the surface properties. This probe is assembled with a cantilever, substrate, and a sharp tip. AFM can provide both 2D and 3D images (similar to OM), which is represented in Fig. 6 [39].

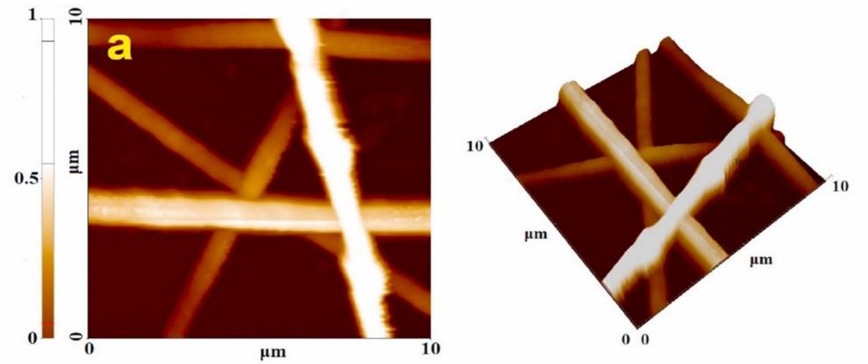

Figure 6. AFM 2D and 3D topography images [48]

Confocal Laser Scanning Microscopy (CLSM) is an effective device to visualize the porous materials [30] [49]. This method’s resolution is relatively lower than electron microscopy (e.g., SEM), but its most significant advantage is that remarkably fewer sample preparations are necessary, which could be intensely time-consuming [50]. Moreover, CLSMs make possible the 3D imaging of porous media (without internal structure) by collecting the fluorescence signals from the sample’s different planes and assembling the 2D-images (planar) into a 3D. Fig. 7 shows the process of imaging [51]. For this reason, CLSM is suitable for three-dimensional “live” imagine, allowing researchers to study both the biological and atomic processes [50]. Notably, that this image is the surface of the media. The maximum resolution is specified by the light’s diffraction limit, like in the case of OM [46] [52]. The compensation of light collection’s losses by raising the exciting laser light is not an appropriate solution because more laser power leads to more phototoxicity and photobleaching [25].

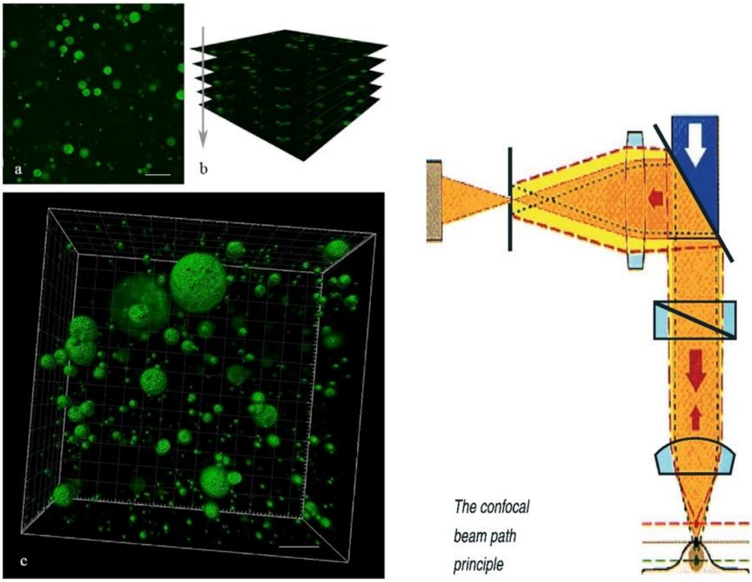

Figure 7. (a) 2D, (b) Z-stack, and (c) 3D CLSM images, and the principle of confocal (d) [53] [54]

3.2. Three-Dimensional Methods with Internal Structure

Three-dimensional microstructures or pore materials, including the whole internal structure, can be imagined with numerous techniques like Nuclear Magnetic Resonance (NMR) (one of NMR application: Magnetic Resonance Imaging (MRI) [34]), Focused Ion Beam technique (FIB) [7], Transmission Electron Microscopy (TEM) [12], X-ray Micro-Computed Tomography (μ-CT or XCT) [13]. Besides, these mentioned microscopy techniques are commonly combined, mentioning some effective methods of them: The Focused Ion-Beam combined with Scanning Electron Microscopes (FIB-SEM) [28] [37] and the Serial Block-Face combined with Scanning Electron Microscopes (SBF-SEM) [28]. These modern microscopy imaging techniques are able to represent a porous medium’s complex geometry with internal structure into a 2-D or 3-D image, which are listed and summarized in Table 2 [6] [9].

Table 2. 3-D (with internal structure) geometry representation methods [6] [7] [9] [12-14] [19] [26-36] [38] [39] [55]

Method	Range	Resolution	Feature
NMR	10 × 10 ×10 cm	100 µm – 1.5 cm	• Effective, non-invasive method, which is suitable for studying spatial resolved fluid saturation; • MRI is an application of NMR, which gives 3-D geometry with internal structure • However, it is less likely available to researchers and expensive;
FIB	5 × 5 ×5 µm	2 nm – 5 nm	• FIB operates in principle like the SEM technique; • FIB uses an ion (not electron) beam instead to create secondary electrons and ions for imaging;
TEM	10 × 10 ×10 µm	0.5 nm – 50 µm	• Suitable for an accurate representation of pore size and distribution with high resolution, it highlights the particle's center • Other 3-D imaging methods are required for a complete examination of porous structures;
Micro-CT	10 × 10 ×10 mm	5 µm – 150 µm	• XCT is capable of various in situ time-resolved investigations during imaging; • 3-D imagine within the inside structure is possible, but the sample size is limited and not suitable for nano-structures;
FIB-SEM	45 × 45 ×45 µm	4 nm – 16 nm	• Common FIB technique (using electrons and ions) in combination with simple SEM (using an ion column) • One of the most potent methods for 3-D imaging of porous microstructures;
SBF-SEM	80 × 80 ×80 µm	10 nm – 100 nm	• SBF-SEM technique is a useful tool for characterizing porous media with relatively high resolution;

Nuclear Magnetic Resonance (also known as NMR) is a widely used spectroscopic method for observation of local magnetic fields, which formed around the atomic nuclei. The investigated materials are put into a magnetic field. The signal of NMR is generated in the following way: radio waves excite the nuclei sample to nuclear magnetic resonance; this can be detected using a simple, sensitive radio receiver [34]. Examining a simple atom in a molecule, we find that the intramolecular magnetic field around it modifies the frequency of resonance. Consequently, it gives a report of the specific functional group of the molecule as well as its electric structure. This formed field is quite special and individual and specifies the compounds; therefore, these techniques are widely applied in chemistry for identifying monomolecular organic compounds [31]. Magnetic Resonance Imaging (MRI) techniques are found in the theory that with the main magnetic field, hydrogen (H_2) protons align themselves. Added a second, orthogonally magnetic field to the main field, which is oscillating at radio frequencies, then protons are pushed out of alignment. When these protons turn back to align with the main magnetic field, they are emitting a measurable signal at radio frequencies, which can be detected. Various material's protons have dissimilar realigning speeds. This attribution is suited to analyze different materials that specify the actual media. MRI-method is widely applied, especially in medical science or the food industry; an example is given in Fig. 8. The main reasons for its attractiveness are that MRIs are applying non-ionizing frequencies, and they are not destructive. As disadvantage can be mentioned that the imaging process is time-consuming, and the resolution of image is restricted [7].

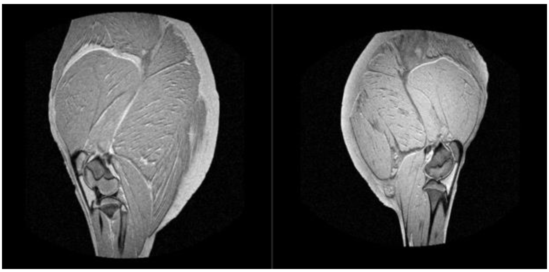

Figure 8. MRI image of different hams [31]

The fundamental of Focused Ion Beam (FIB) is genuinely like SEM [7], but it uses a focused beam of ions (see Fig. 9), not an electron beam. The diameter of the beam is down to about five nm. Using the FIB technique, internal structures can be imaged [33]. The equipment's primary (main) ion beam must be working at low currents for imaging of different materials. High currents give other alternative usages of FIB, like cutting, milling (see Fig. 9), drilling, or structuring at the atomic-scale, where critical material ablations are observable [7].

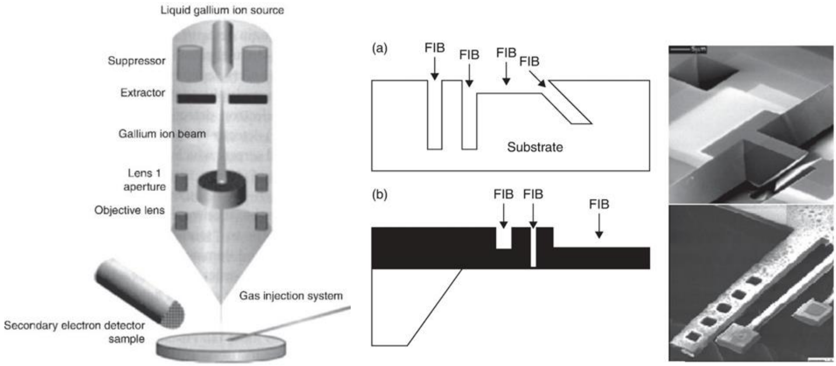

Figure 9. Schematic illustration of focused ion beam system and two examples for FIB milling: milling pattern (a) and milling structure (b) [56]

Transmission electron microscopy (TEM) is widely used and a viral technique in the characterization of nanomaterials [12]. For images of nanomaterials, the same spatial resolution as the atomic level can be achieved by using TEM [57]. The present device uses the same fundamental principles as Optical Microscopy (OM), but it utilizes not light but electrons. Fig. 10 shows a schema of the TEM technique with two different images. The wavelength of light is shorter than in the case of electrons. Consequently, TEM can offer high-resolution images compared to optical microscopy. Generally, TEM techniques allow researchers to investigate objects in the order of 10^{-10} m, reaching atomic levels [7].

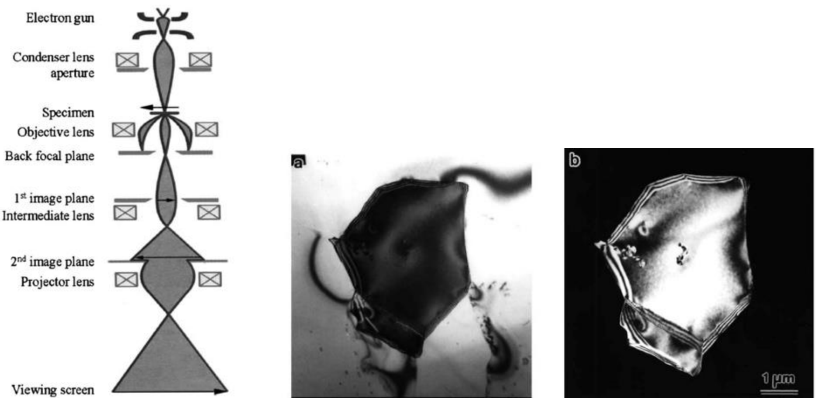

Figure 10. Schematic optical diagram of TEM (left), imagines with different objective aperture positions: bright- and dark-fields microscopy images (center and right, respectively) [58] [59]

X-ray Computed Tomography – also known μ-CT or XCT – makes it possible to enhance the applied resolution (approx. 7-8 orders of magnitude regarding the geometry volume), which strongly relates to the application [60]. This microscope's required resolution depends on the porous material's actual characteristic (especially shape and type) [7]. Micro-CTs are effective tools in the research of evaluating 3D-structures of the different porous scaffolds. Consequently, XCT can quantify important parameters, e.g., average pore size, pore interconnectivity, and porosity [19]. As both animal studies and cell culture are costly and time-consuming, advanced scaffold's optimal micro-structure needs to be verified in the early phase of development [61]. Computed Tomography imaging is an effective and powerful technique that is not destructive and utilizes thermal neutrons, but a nuclear reactor is needed [60]. A micro-CT scanner's direct output is commonly a grey-scale image (see Fig. 11) that is post-processed by filtering and applying segmentation techniques to identify discrete material phases in the image. Fig. 11 shows the whole process of 3D reconstruction of an XCT image [7] [19].

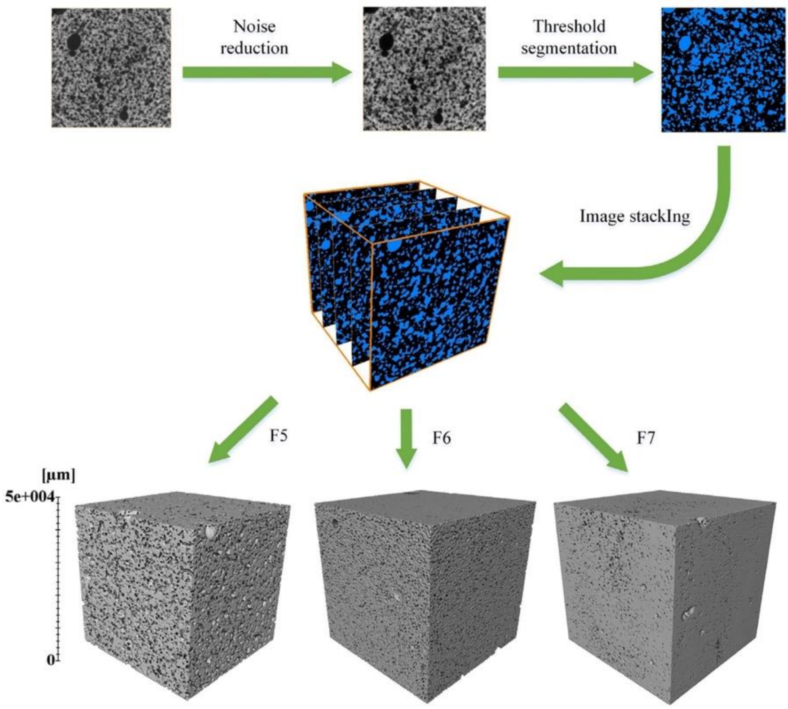

Figure 11. Process of XCT image 3D reconstruction [62]

Focused Ion-Beam – Scanning Electron Microscopies (FIB-SEM) are a combination of an ordinary SEM microscopy applying ion column (i.e., gallium) and the specific beams – namely, electrons and ions (FIB) – focused on one concrete point [32-33]. The latter purpose is to remove the unnecessary material and preparation of flat 2-D surfaces. A simple FIB can be realized based on SEM, which can be used to identify the location of homogeneous portions (in terms of X-ray attenuation and surface attributes) and, thus, the specific volumes for the process milling [32]. With the FIB-SEM methods, internal structures can also be mapped; an example is given for its images in Fig. 12 [33].

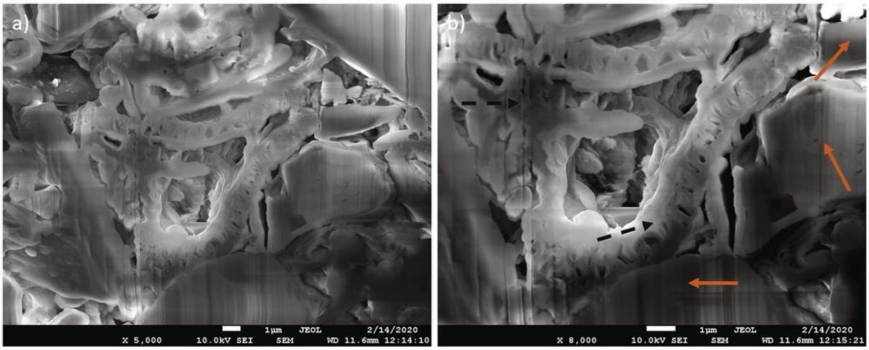

Figure 12. The imagine of FIB-SEM of an inner structure, using secondary (a) and back-scattering electrons (b) [32]

Serial Block-Face Scanning Electron Microscopes (SBF-SEM) are new and innovative techniques developed by Horstmann et al. [36] at the Max Planck Institute. Their commercialized microscopy is available as an accessory to several SEM models. Additionally, the microscope is operating with automatic serial sectioning and scanning of the sample embedded in SEM. Using the SBF-SEM technique, a three-dimensional image can be reconstructed from an aligned density map of the sample by choosing longer-term, extended processing of the sample (from 1 day to 30 days). The SEM’s image is similar to a conventional TEM, except that the resolution of the SEM is lower than the TEM at higher magnification ranges. At lower magnifications, the SEM has sufficient resolution to the competition with TEM used in the same range, illustrated in Fig. 13 [36].

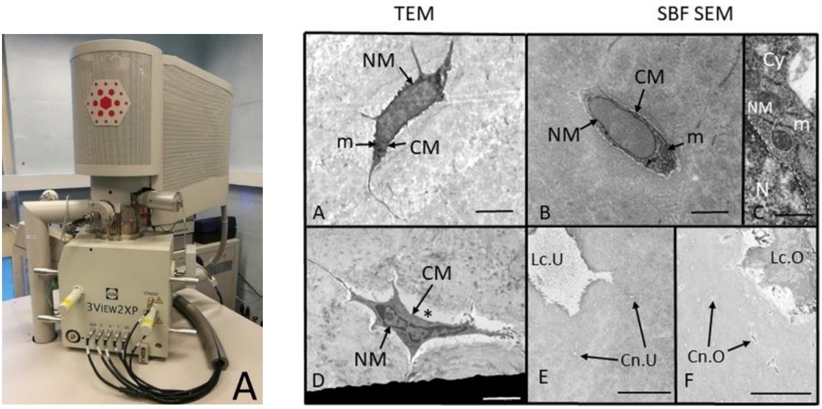

Figure 13.Modern SBF-SEM system and TEM and SBF SEM images comparison [63]

4. Summary

Geometry representation methods [25] [38] are especially critical because the two- or three-dimensional images are input parameters of numerical simulations. In complex cases, there are two different ways to produce the 3D geometry [38] for simulations as input: 3D imagines with the whole internal structure and reconstruction from numerous, separate 2D surfaces [6]. In this context, pore-scale simulations can improve the study and research process [64], as well as the comprehension of the physical processes at the pore-level and allow the determination of several macroscopic parameters. However, these parameters – obtained by simulation – can be applied in a continuity model, which could simply be determined from costly plus too consumptive of time field measurements or laboratory experiments. Besides, it is essential to note that it is not possible to implement experimental setups for various practical issues. Theoretical research in fluid dynamics supports simulation approaches' continuous improvement at different scales such as micro and continuum-scales. In addition to that, the practical application of these theoretical methods, especially in automotive engineering, requires in-depth technical knowledge of fluid behavior. Furthermore, it is necessary to extend them, involving multi-phase flows, fluid flow interactions with other physical phenomena – also known as multi-physics problems.

5. Outlook and Future Work

With the continuous development and spread of fluid flow simulations, the representation method of different materials is becoming increasingly important, especially in the field of microstructures. In the future, there will be a particular need to develop representation methods that can provide high-quality input with wide range for simulations without any further, time-consuming post-processing. The availability of such applicable methods would be of great benefit in material research.

In our subsequent research, we will scan a sample of porous material using a 3D representative method that can provide adequate input for CFD simulations. The sample size and the equipment availability also need to be considered to select the suitable/applicable method. At this time, the sample is still not determined. After scanning the material, the resulting geometry must be prepared for mesh generation. This future research aims to investigate the flow properties and characteristics of microstructures in a numerical simulation environment, e.g., viscous and thermal characteristic length, the high-frequency limit of the dynamic tortuosity, etc. Finally, the results will be compared to laboratory measurements and experiments to validate the simulation.

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