Hierarchical porous silica in monolithic form is used in chromatography and heterogeneous catalysis as an alternative to other fixed bed structures. Their distinctly bimodal pore size distribution is essential to the performance of silica monoliths as solid support. A continuous network of macropores (~2 µm) enables liquid transport at high flow rates without necessitating high pressures, while the nanometer-sized mesopores provide the high surface area for sufficient contact between chemicals and the stationary phase (for separation or catalysis) immobilized on the fixed bed.

Beyond a superficial characterization of the two pore categories, the monolith's disordered pore structure remained largely unknown until recently, when the macropore space has been accessed by confocal laser scanning microscopy [1]. For the much smaller mesopores nothing beyond pore size data from bulk methods, e.g. mercury intrusion porosimetry and nitrogen physisorption is available at present. However, an accurate and quantitative morphological characterization would be needed to tailor the morphological properties of mesoporous adsorbents for their intended use.

Here we demonstrate the reconstruction and characterization of the complete morphology of a silica monolith, from macropores to mesopores, by combining FIB slice&view techniques and high-resolution STEM tomography. Based on the three-dimensional reconstruction we performed a comprehensive statistical analysis to extract key structural parameters relevant to mass transport at the macropore and mesopore level. The reconstructed model is also the starting point for simulations of flow, mass transport [2], sorption and reaction which aim at a fundamental understanding of the morphology-transport relationships of hierarchically structured, disordered materials as a basis to improve morphological features responsible for separation efficiency and catalytic activity [3].

References
Fig. 1: Characterization of hierarchical structure combing FIB-SEM and HAADF-STEM tomographic techniques to build a 3D model.