Multiscale characterization of hierarchical particle–pore systems using correlative X-ray and electron tomography

Benjamin Apeleo Zubiri[1], Allison Götz[1], Silke Christiansen[2], Umair Sultan[3], Nicolas Vogel[3], Erdmann Spiecker[1], Johannes Boehmer[1], Matthias Thommes[4], Fabian Lutter[2], Dennis Possart[2], Daniel Augsburger[2], Arslan Usman[2], Sabrina Pechmann[2], Carmen Rubach[1], Moritz Buwen[1], Alexander Kichigin[1], Nora Vorlaufer[5], Peter Suter[6], Tor Hildebrand[6], Peter Felfer[5], Katharina Breininger[7]

[1] *Institute of Micro- and Nanostructure Research (IMN) & Center for Nanoanalysis and Electron Microscopy (CENEM), FAU Erlangen-Nürnberg, Germany*
[2] *Fraunhofer-Institute for Ceramic Technologies and Systems – Correlative Microscopy and Materials Data, Germany*
[3] *Institute of Particle Technology, FAU Erlangen-Nürnberg, Germany*
[4] *Institute of Separation Science and Technology, FAU Erlangen-Nürnberg, Germany*
[5] *Institute for General Materials Properties MSEI, FAU Erlangen-Nürnberg, Germany*
[6] *Lucid Concepts AG, Switzerland*
[7] *Professur für Informatik (Pattern Recognition), Center for AI and Data Science (CAIDAS), Julius-Maximilians-Universität Würzburg, Germany*

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Hierarchical porous, particulate materials underpin catalysts, batteries, electrolyzers, and separation media, with performance governed by coupled intra- and interparticle pore networks spanning multiple length scales. Precise multiscale characterization is therefore essential and requires correlative 3D microscopy to bridge the field-of-view–resolution trade-off of single modalities.

We present a lab-based, scale-bridging workflow that integrates electron tomography (ET), nano X-ray computed tomography (nanoCT), and micro X-ray computed tomography (microCT) to quantify particle and pore statistics from nanometers to millimeters. [1] A central challenge – simultaneously resolving fine internal structures and acquiring statistically robust particle populations – is addressed by coordinated sample design, multimodal imaging, and identical-location analysis.

As a model system, we study porous silica particles produced by polymerization-induced phase separation, featuring hierarchical meso- and macroporosity. [2] A customized tapered pillar of embedded particles is prepared by mechanical grinding and polishing to optimize the field of view (FOV) for each imaging technique. This geometry enables millimeter-scale microCT surveys for statistics on thousands of particles while preserving regions for high-resolution investigation by nanoCT at identical locations. MicroCT using a ZEISS Xradia 620 Versa captures ensembles and packed assemblies at down to 500 nm resolution on mm-scale specimens. Individual particles and their intraparticulate pore networks are imaged by nanoCT using a ZEISS Xradia 810 Ultra with 50 nm resolution at a (16 µm)^2 FOV and 150 nm at (64 µm)^2 FOV. Therein, Zernike phase contrast enhances structural details in weakly absorbing silica. To access smaller mesopores, we perform 360°-ET on a pillar extracted from a representative particle, mitigating the missing wedge and providing nanometer-scale ground truth. Cross-modal registration of corresponding regions of interest enables direct data correlation. Reconstructed volumes are segmented using supervised, neural-network–based methods to robustly delineate solid and pore space under phase-contrast conditions. The unified pipeline yields quantitative descriptors across scales, including particle size, pore size distributions (meso- to macropores), and porosity.

This work underscores the value of cross-scale, correlative 3D microscopy for a comprehensive understanding of hierarchical materials. Complementary imaging modalities, accurate registration, and machine-learning segmentation enable robust process–structure–property correlations and guide the optimization of functional porous architectures.

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References

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