Crystallographic and Defect Analysis of Colloidal Photonic Supraparticles Using Zone-Axis Wide-Field XRM and NanoCT

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Self-assembled colloidal supraparticles (SPs) are aggregates of primary particles that exhibit a wide range of structural configurations and associated optical properties [1]. For primary particle sizes on the order of 200–300 nm (e.g., polystyrene spheres), these assemblies can display photonic behavior in the visible spectral range, resulting in structural color. Such optical effects are most pronounced in highly-ordered, crystalline SPs, which can be fabricated, for instance, by ultraslow drying in spherical confinement. Typical structures include icosahedral, decahedral, and fully face-centered cubic (FCC) arrangements with octahedral symmetry. Scanning electron microscopy (SEM) is well suited to reveal surface morphology; however, access to the full internal 3D structure—including crystal defects and disordered regions—is essential to understand the origin and variation of structural color. Transmission-based imaging methods are therefore required. Due to their size, SPs lie at the upper limit of transmission electron microscopy and tomography (TEM/ET) [1], thereby motivating the use of X-ray microscopy (XRM) and tomography (nanoCT) as key techniques for volumetric characterization [2].

In this contribution, we investigate the 3D structure of crystalline SPs and their defects using a combination of electron and X-ray transmission microscopy/tomography. Drawing parallels to crystalline nanoparticles, which are commonly analyzed by high-resolution (S)TEM along low-index zone axes, we establish an analogous approach in XRM to access crystallographic information in larger assemblies. To this end, we employ a dedicated sample holder setup that enables precise and reproducible tilting of individual SPs, allowing controlled alignment along specific crystallographic directions. We demonstrate this capability by orienting a decahedral SP along its five-fold twin axis to analyze internal defect structures and compare them with simulations, as well as by imaging FCC SPs along different low-index zone axes. While high-resolution, 2D projections already allow the identification of planar defects, the detection and quantification of point defects necessitate volumetric imaging.

To further relate structure to optical response, we complement the experimental analysis with optical simulations based on the reconstructed particle configurations. These simulations enable systematic investigation of how structural motifs and defects influence scattering, color formation, and image contrast in optical microscopy. By linking three-dimensional structure, defect distribution, and optical signatures, this work provides deeper insight into structure–property relationships in colloidal supraparticles and supports their targeted design as photonic materials.

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References

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