

# Multiscale Reciprocal Space Mapping of Mesocrystals

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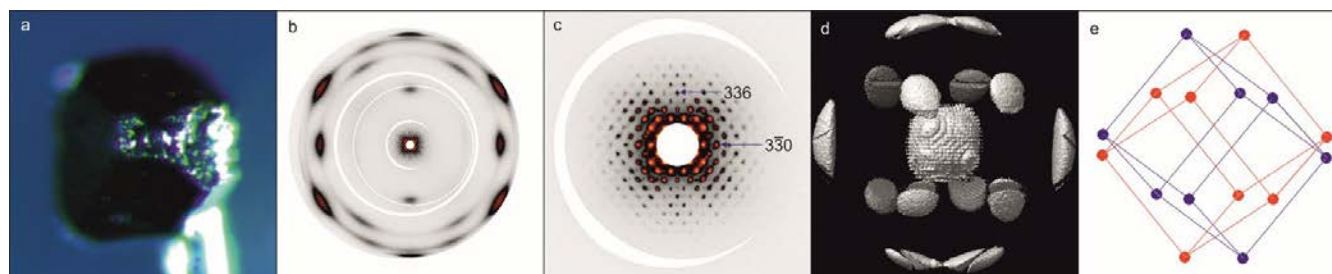
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Mesocrystals – a form of nanostructured material, usually defined as a nanocrystal superstructure with a common crystallographic orientation – exhibit a multiple-length-scale structure. Mesocrystals can occur naturally in abiotic and biogenic minerals or can be synthesized artificially [1]. The combination of small- and wide-angle X-ray scattering (SAXS and WAXS) techniques offers the possibility to non-destructively probe mesocrystalline structures simultaneously over multiple length scales to reveal their microscopic structure.

Compared to our previous work on biogenic calcite from sea urchins [2], the study of artificial magnetite mesocrystals was a major step forward. SAXS and WAXS regions could be fully separated since high orders of small-angle diffraction can merge with the low orders of diffraction from the atomic arrangement. WAXS data, beyond some momentum transfer, can be considered as an incoherent sum of individual diffraction patterns of nanoparticles. Thus, for sufficiently monodisperse mesocrystals, coherent diffraction imaging is naturally performed without isolating the individual nanoparticle. Combining electron microscopy with x-ray scattering, we were able to identify size and faceting of individual particles, long-range packing, packing defects and orientation distribution functions - and infer the mechanisms of growth symmetry choice of mesocrystals [3].

Large part of experiments was performed at the PILATUS-based ID28 ESRF diffractometer and expanded to the PETRA III ultra-SAXS P03 beamline [4].

Figure 1 exemplifies sometimes misleading faceting of as-grown mesocrystals – here cubooctahedral habitus hides regular twinning of body centred tetragonal structures, not following the macroscopic cubic-like symmetry.



**Figure 1.** (a) mesocrystal with cubooctahedral shape; (b) and (c) wide- and small-angle reconstruction of reciprocal space plane common for two twins, respectively; (d) 3D experimental isosurface showing spatial positions of (111)-type magnetite diffraction spots compared to the idealized scheme (d).

Magnetite mesocrystals are conveniently measured in diamond anvil cells, providing the information on the compressibility of hard crystalline cores and surfactant shells separately. It provides rather interesting possibility to test the models of scattering where mesocrystal scale joins atomic scale and SAXS and WAXS become inseparable.

[1] H. Cölfen, M. Antonietti, *Mesocrystals and Nonclassical Crystallization*, Wiley, Chichester, UK 2008

[2] H. Cölfen et al, *Acta Cryst.* 2022, B78, 356-358

[3] A. Chumakova et al., *Adv. Mater.* 2023, 35, 2207130

[4] A. Chumakova et al., *Crystals* 2023, 13(8), 1204