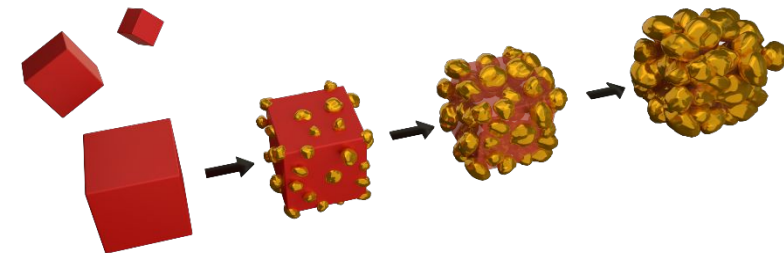
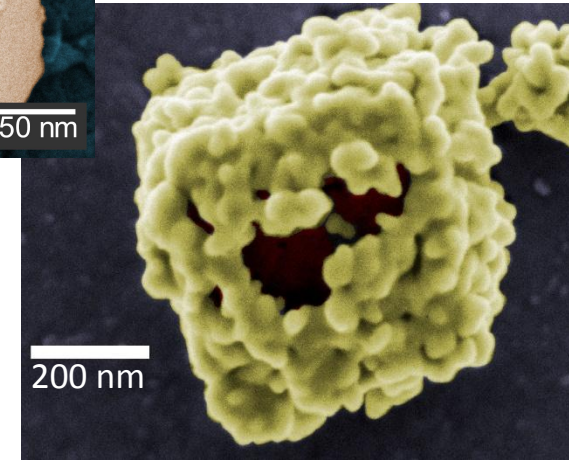
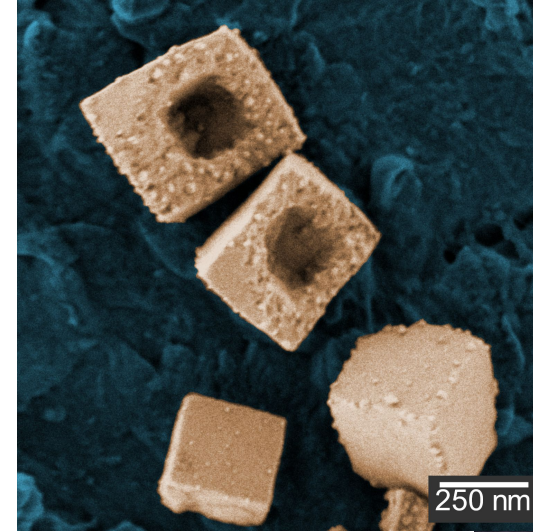


Imaging nanocube hollowing in solution with in situ X-ray ptychography

Lukas Grote

Research groups of Prof. Koziej and Prof. Schroer
University of Hamburg and DESY

03.05.2023



Understanding size and shape evolution



Humanity faces many global challenges.

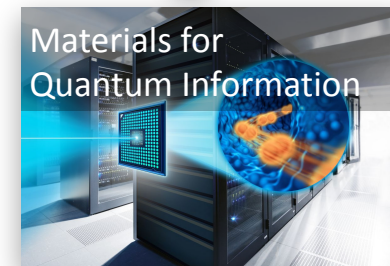
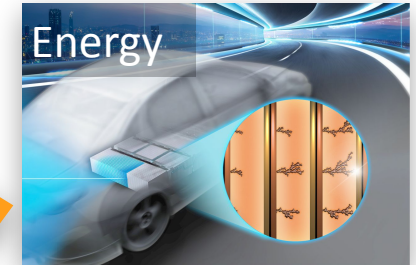
Sustainable technologies to address challenges require:

- novel materials with tailored functions
- sustainable processes

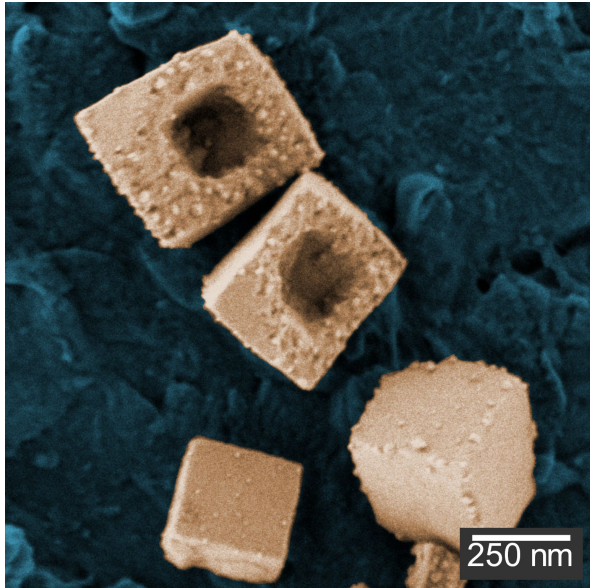
Sophisticated analytics needed:

- understand **structure and function** on all relevant **length and time scales** from atomic to macroscopic dimensions
- knowledge-based, rational design of materials, drugs, and processes

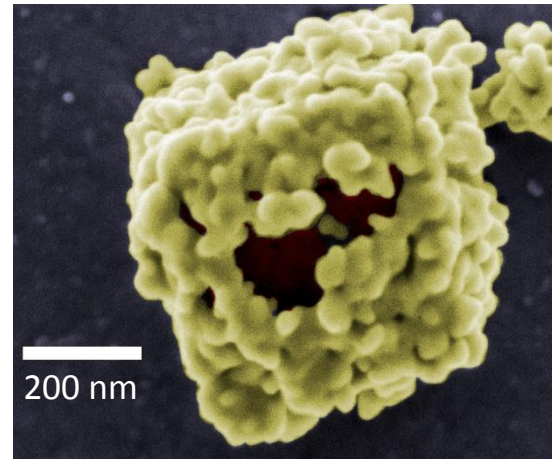
New paradigm: **observe** → **design** → **control**



Complex nanomaterial formation



hollow Cu nanocubes



$\text{Cu}_2\text{O}@\text{Au}$ nanocages

How do complex nanomaterials form in solution?

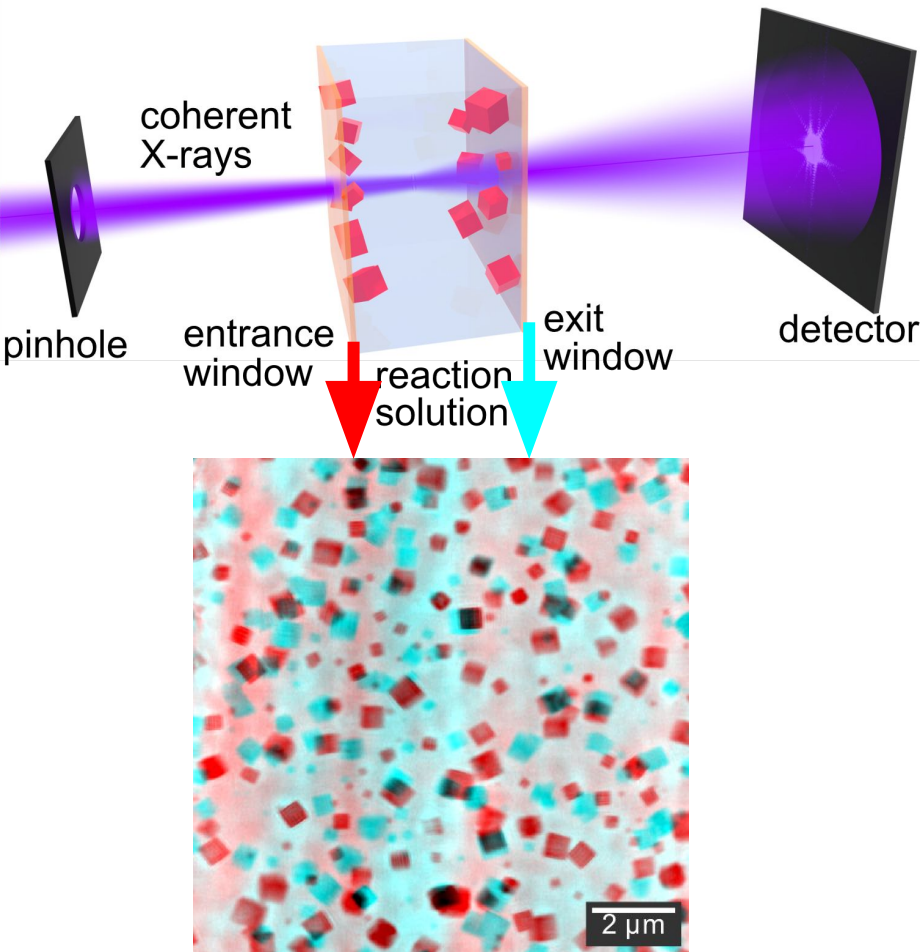
In situ X-ray microscopy enables direct visual observations!

- understand size and shape evolution
- unravel non-classical growth processes

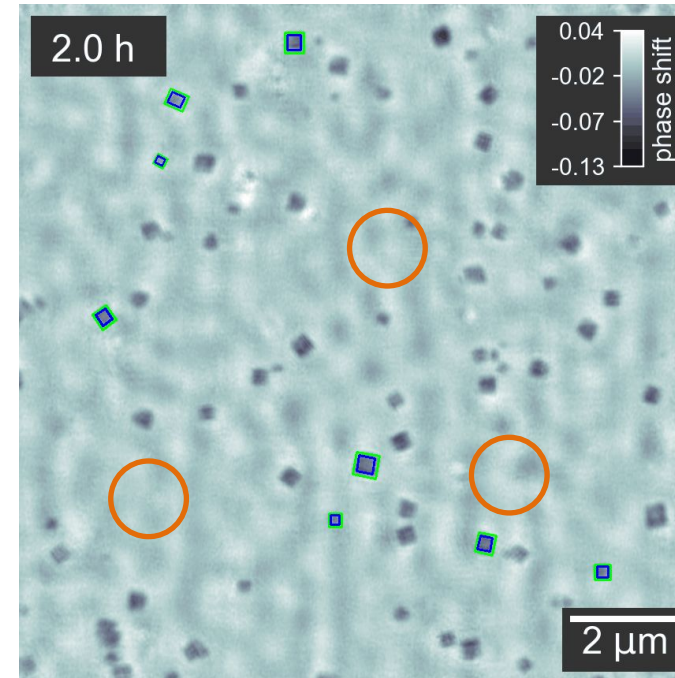


S. S. Mofarah et al., *J. Mater. Chem. A* **8**, 9 (2020)

Multi-slicing enables X-ray ptychography in solution

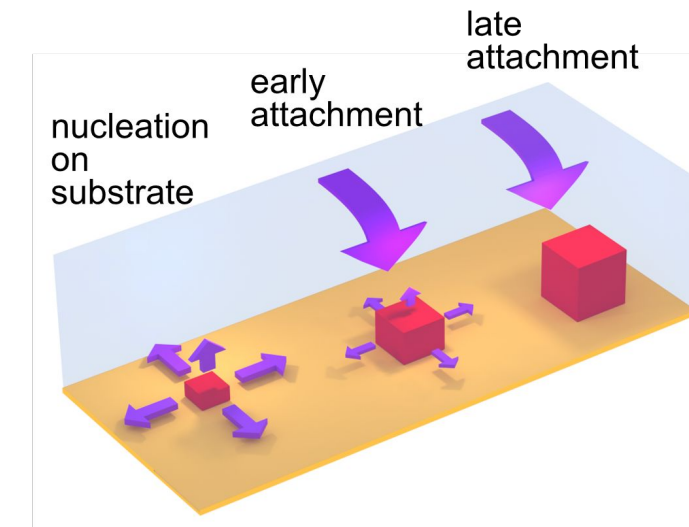


Separate high-resolution imaging of two reactor windows:



— in-plane size
— thickness estimation

quantitative phase shift:
Follow particle in 3D!
→ thickness evolution

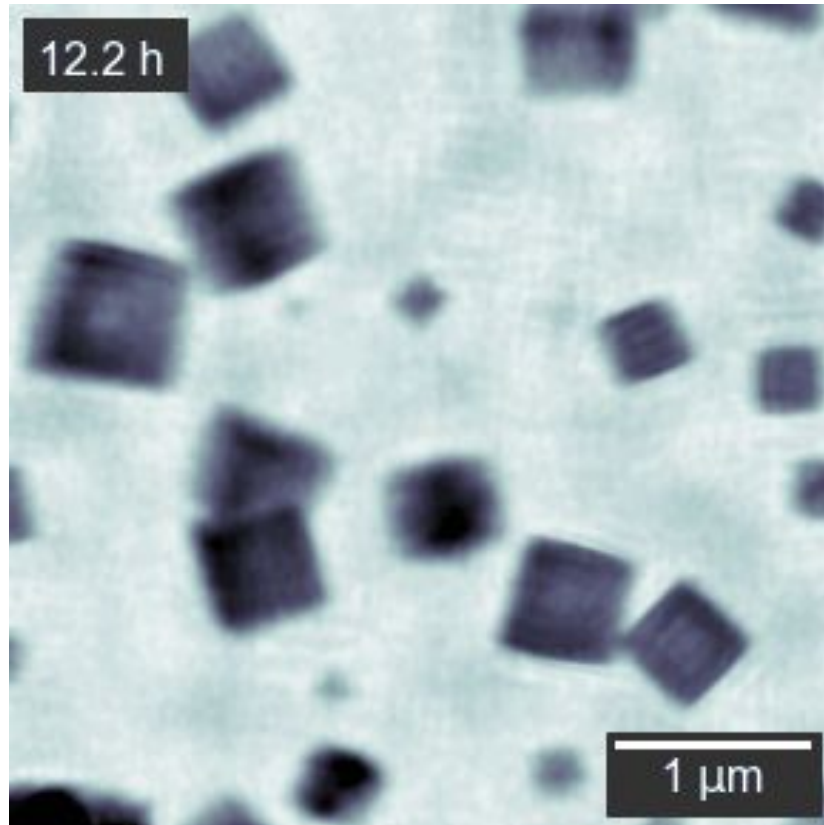


Multi-slice ptychography
→ follow growth in extended chemical reactor

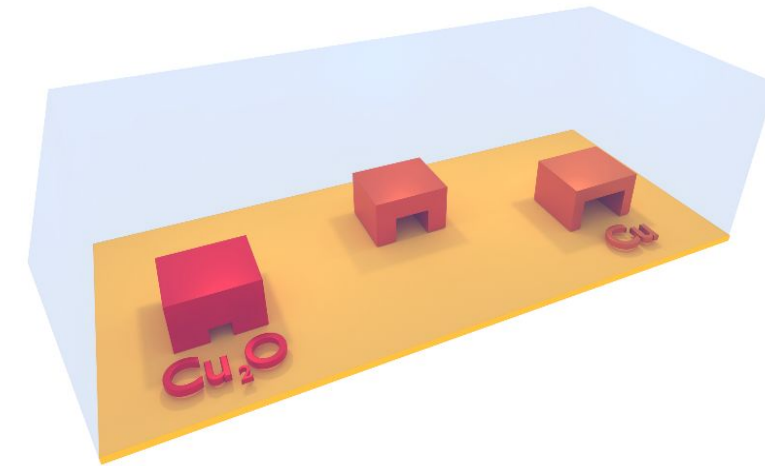
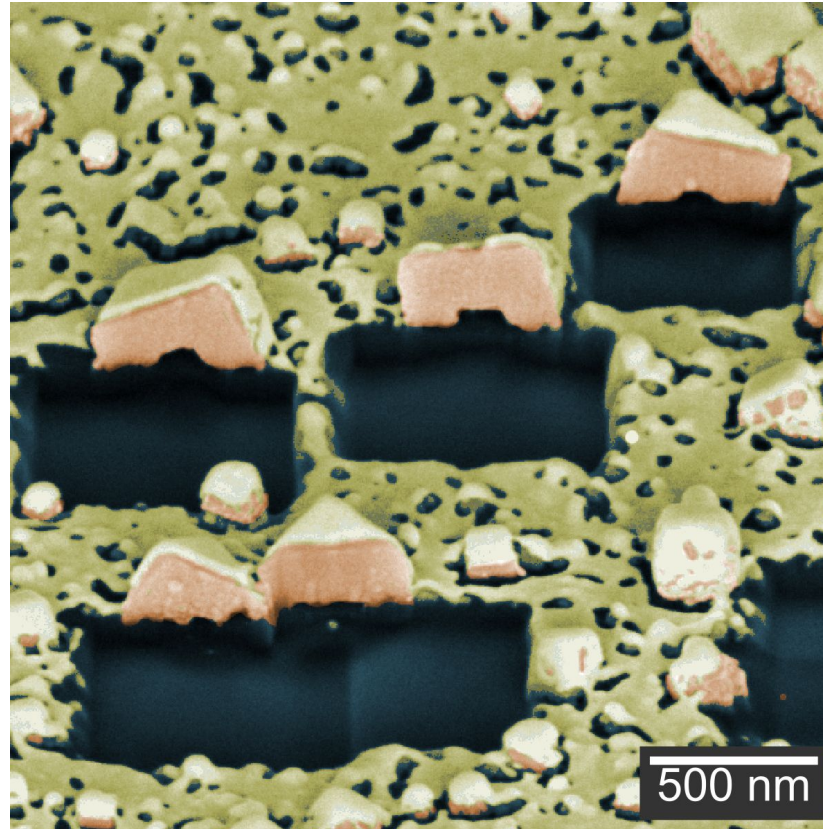
L. Grote et al. *Nature Communications* **13**, 4971 (2022); M. Kahnt et al. *Scientific Reports* **11**, 1500 (2021)

Void formation upon reduction to Cu

X-ray ptychographic phase images:



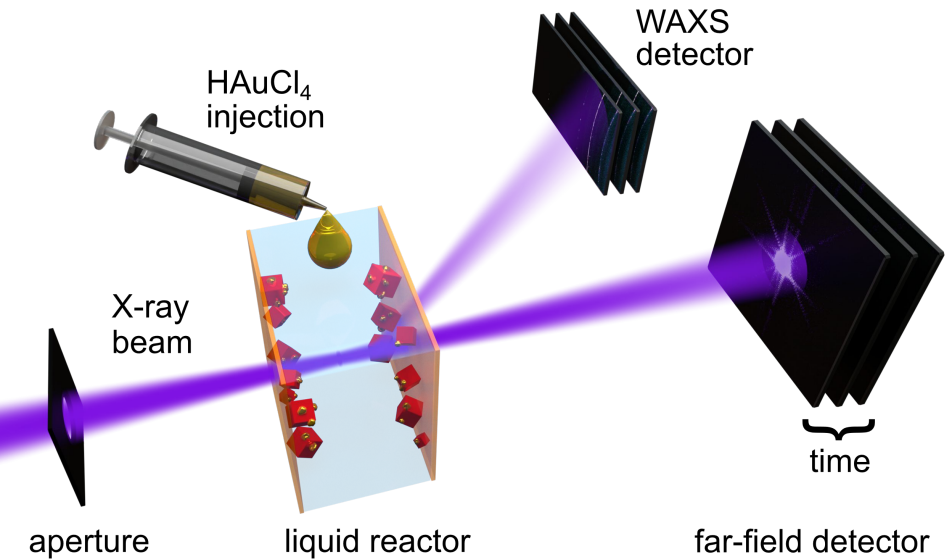
FIB-SEM:



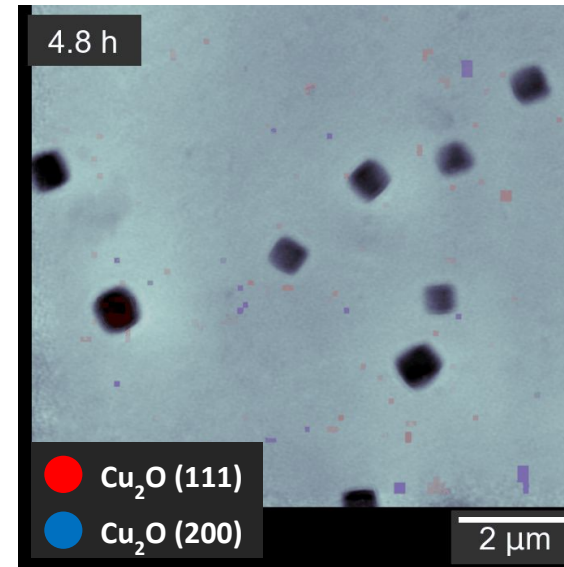
Strong binding between particles and substrate.

- mechanism:
surface-protected conversion
- volume shrinkage
compensated by void formation

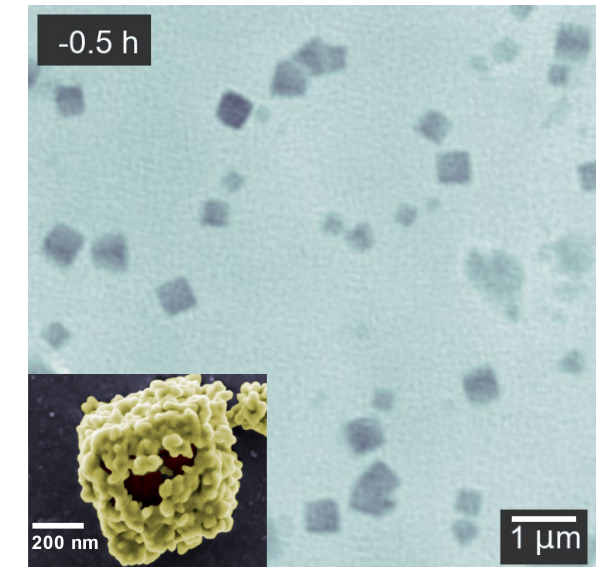
Multimodal imaging shows composition and crystallinity



Crystallinity and lattice orientation:



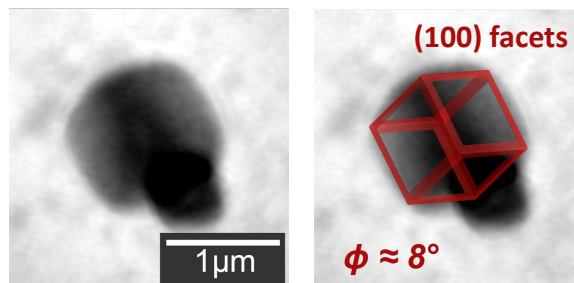
Composition of multi-material:



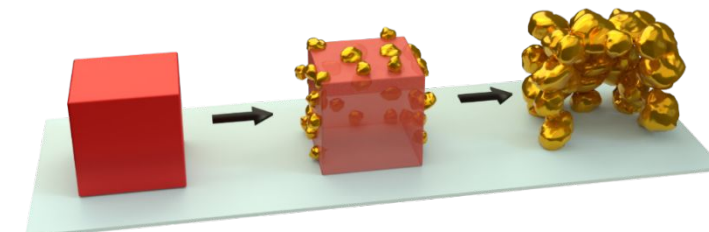
Simultaneous scanning WAXS and ptychography.

- crystallinity of particles
- estimate lattice orientation of single nanocube in situ
- composition of multi-material nanostructure

Nanocube has (100) facets.



Dissolution of particle interior occurs homogeneously.



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CUI: Advanced Imaging of Matter

EXC 2056 - project ID 390715994



The future is bright for materials science..



- in-solution X-ray ptychography
- visual proof of shape evolution
- multimodal ptychography and WAXS
- crystallinity and composition

Now we can: observe → design → control!

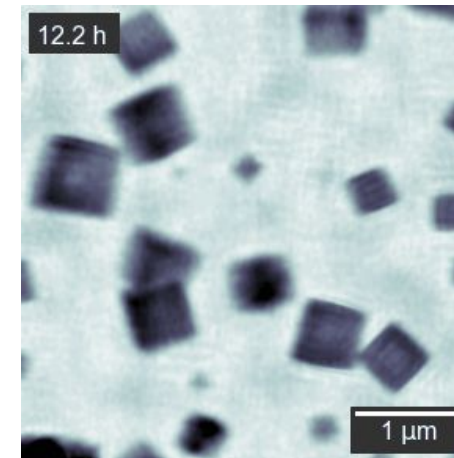
Future plans:

- 4th generation synchrotron sources (**PETRA IV**)
- 3D imaging with increased resolution
- single-pulse imaging at XFEL
- image dispersed nanoparticles

Observe materials in complex environments:

- battery electrodes in electrolytes
- catalysts in photochemical devices
- functionalized nanoparticles in cells

Cu₂O nanocube hollowing

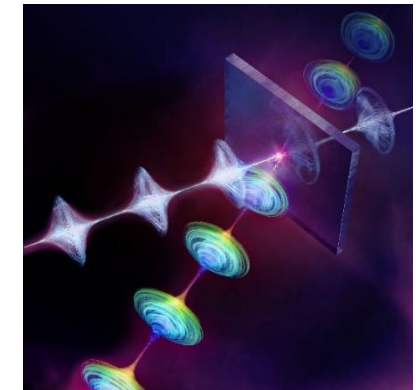


3D imaging at PETRA IV



<https://www.desy.de/news/>

single-pulse imaging at XFEL



https://www.xfel.eu/news_and_events/news