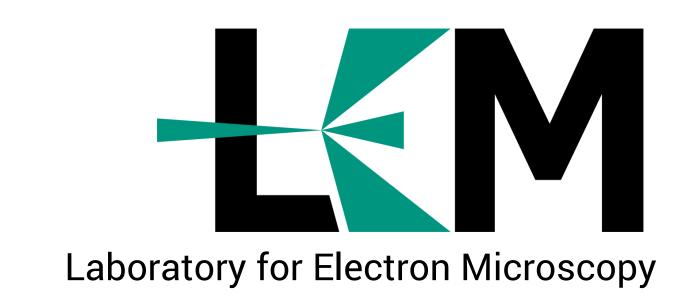


Direct Synthesis of ZIF-8 on Transmission Electron Microscopy Grids Allows Insights into the Growth Process



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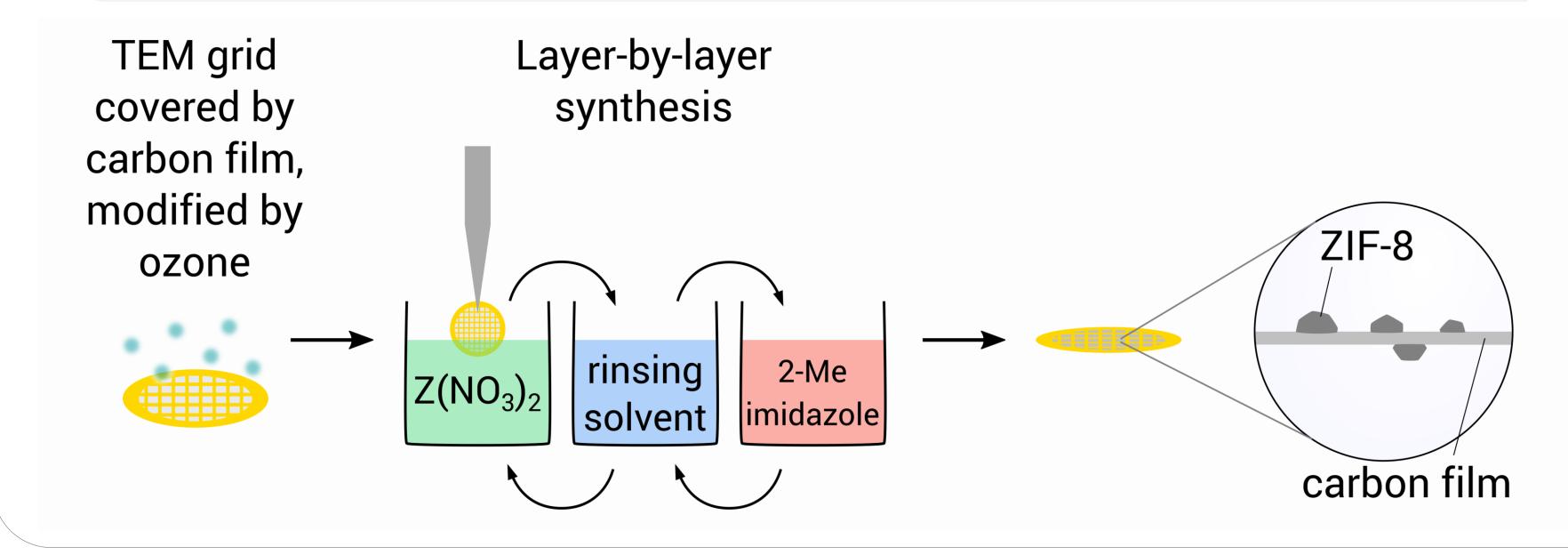
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Background and Goals

- ZIF-8 belongs to the class of zeolitic imidazolate frameworks
- Comparatively high thermal and chemical stability (humid atmosphere, solvents) [Par+06] but high sensitivity towards electron beam illumination [Zhu+17]
- Growth of surface-mounted thin film (SURMOF) in a layer-by-layer process [SE13]
- Crystal shape evolves from cubes to rhombic dodecahedra (RD) during growth when synthesized as powder [Cra+12]
- Challenge: Damage-free preparation of ZIF-8 SURMOF for TEM

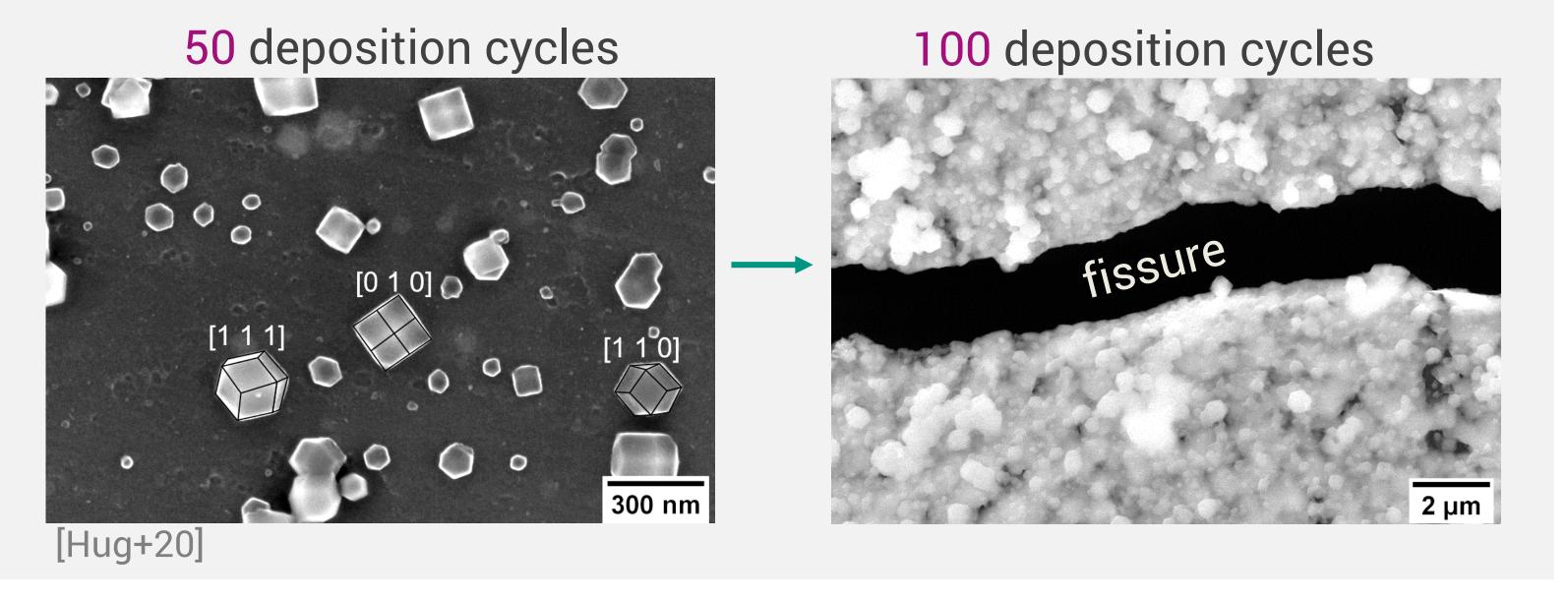
Layer-by-layer Synthesis

- Direct synthesis of ZIF-8 on a thin amorphous carbon (aC) film supported by a TEM grid
- → avoids damaging steps in preparation for TEM [Hug+20]
- Preparation of sample batch with different number of deposition cycles → study growth process



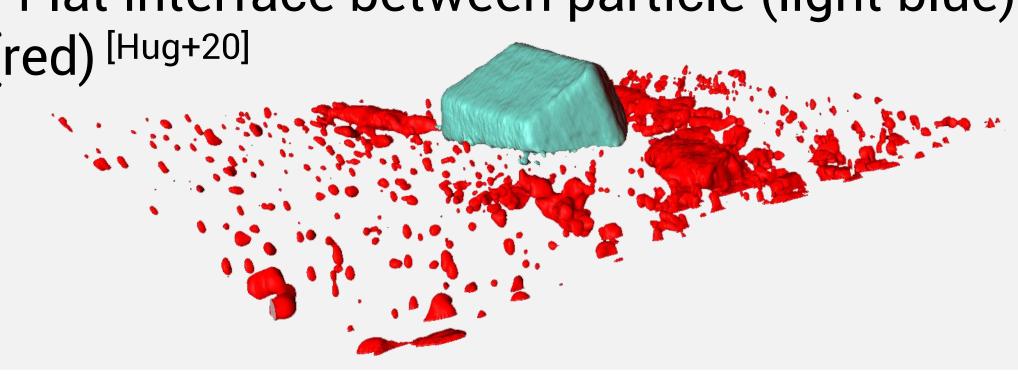
Film Formation

Individual particles form on the aC film, coalesce, and overgrow to form a closed layer



Interface Morphology between ZIF-8 and aC Film

■ TEM tomography: Flat interface between particle (light blue) and support film (red) [Hug+20]



Summary

- ✓ Dipping synthesis of ZIF-8 on aC thin film
 - → direct analysis by TEM and SEM
 - → promising technique for other MOFs
- ✓ Formation of polycrystalline ZIF-8 film

Particle Shape Evolution

ZIF-8 crystal shape evolves from cubes to rhombic dodecahedra (RD) during growth

39 deposition cycles

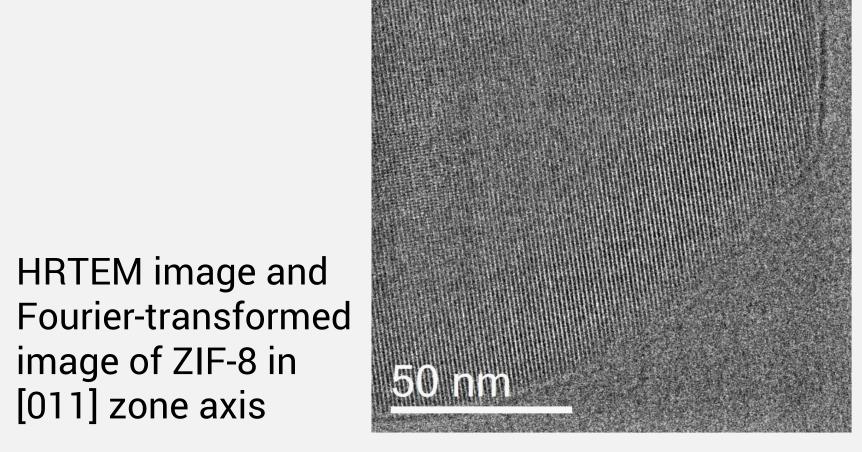
46 deposition cycles

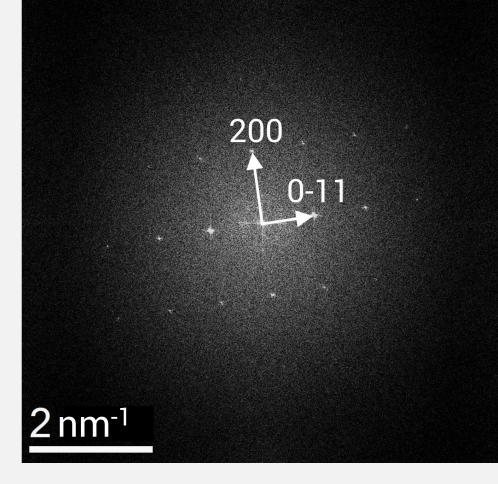
cubes

200 nm

Crystal Structure Analysis by HRTEM + Nanodiffraction

- Direct synthesis on aC film preserves crystal structure of ZIF-8
- Nanodiffraction yields crystal structure if sensitive camera is not available





[Cra+12] Cravillon et al., CrystEngComm 14, 492 (2012)

[Hug+20] Hugenschmidt et al., Part. Part. Syst. Charact. 37, 2000209 (2020)

[Par+06] Park et al., PNAS 103, 10186 (2006)

[SE13] Shekhah and Eddaoudi, Chem. Commun., 49, 10079 (2013)

[Zhu+17] Zhu et al., Nat. Mater. 16, 532 (2017)











