

Revealing Invisibles: Analysis of Complex Structures by 3D Electron Diffraction

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Single crystal X-ray diffraction (SCXRD) is currently the most predominant method for crystal structure determination at the atomic level, but requires large crystals with sufficient quality. Framework materials such as metal–organic frameworks (MOFs), and covalent organic frameworks (COFs) are often obtained as nanocrystals. During the past two decades, three-dimensional electron diffraction (3D ED) has been developed to overcome the barriers for structural analysis of nanocrystals, which drastically accelerated the development in the fields of functional materials.

Herein, I will give an overview of the development of low-dose 3D ED method for analyzing framework materials, where we overcome the challenges of electron beam damage to these compounds^[1,2]. Using 3D ED, I will give an example of taking the high-throughput advantage of 3D ED on discovery of new materials among phase mixtures^[3]. Furthermore, I will talk about using 3D ED to probe molecular motions in MOF nanocrystals^[4]. Last but not least, I will present using 3D ED to study host-guest interactions, including organic molecules^[5] and our recent development of studying CO₂ sorption process. We believe that using 3D ED as a powerful analytical tool for discovering new functional materials and revealing their unique properties at an atomic level would help to accelerate research in this community.

- [1] T. Yang, T. Willhammar, H. Xu, X. Zou, Z. Huang, *Nat. Protoc.* **2022**, *17*, 2389–2413.
- [2] Q. Chen, G. Zhou, Z. Huang, *Acc. Chem. Res.* **2024**, *57*, 2522–2531.
- [3] M. Ge, Y. Wang, F. Carraro, W. Liang, M. Roostaenia, S. Siahrostami, D. M. Proserpio, C. Doonan, P. Falcaro, H. Zheng, X. Zou, Z. Huang, *Angew. Chem. Int. Ed.* **2021**, *60*, 11391–11397.
- [4] L. Samperisi, A. Jaworski, G. Kaur, K. P. Lillerud, X. Zou, Z. Huang, *J. Am. Chem. Soc.* **2021**, *143*, 17947–17952.
- [5] M. Ge, T. Yang, H. Xu, X. Zou, Z. Huang, *J. Am. Chem. Soc.* **2022**, *144*, 15165–15174.

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