The Role of Cluster Analysis in Automated Serial Electron Crystallography

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Introduction

We are developing software and methodology to automate the collection of electron diffraction (ED) data.

Structure determination of (sub)micron-sized crystals

- With electron diffraction, high-resolution data can be collected on crystals orders of magnitude smaller than those needed for X-ray diffraction experiments

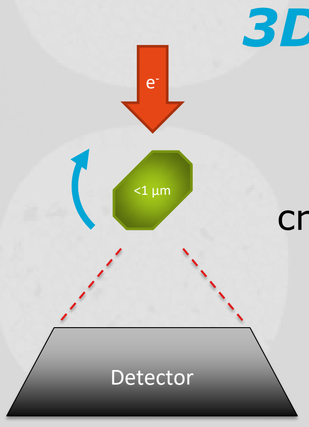
High-throughput serial electron diffraction (SerialED)

- Crystals are detected at low magnification. Snapshot (~1000 crystals/hour) and continuous rotation (~50 crystals/hour) data are collected automatically.

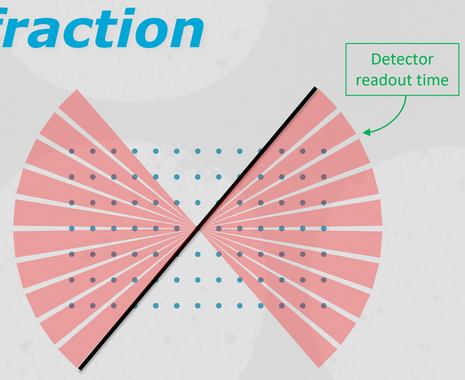
Hierarchical cluster analysis (HCA)

- HCA helps to sort through the 100s of data sets and selects the optimal ones for merging and structure analysis. HCA enables automated phase analysis of materials with known and unknown phases.

3D electron diffraction



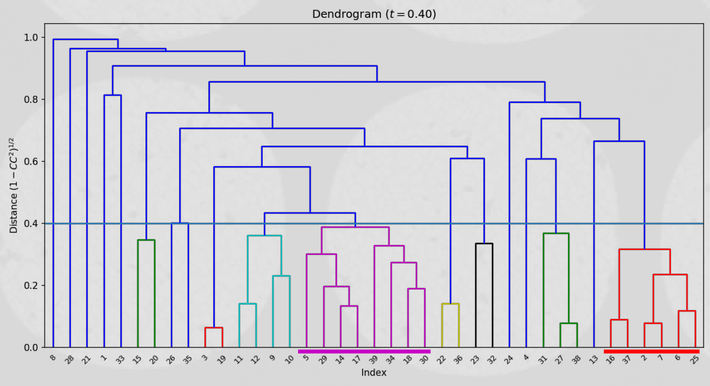
Continuously tilt crystal while reading out camera

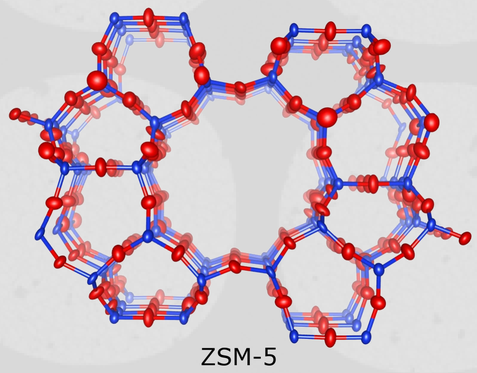


Tilt range: up to 150° (0.1-0.5°/frame)
Rotation speed: 0.5-3.0°/s

Hierarchical cluster analysis

- Cluster similar data sets by common reflection intensities
- Make optimal selection of data sets for merging
- Improve data precision and model accuracy



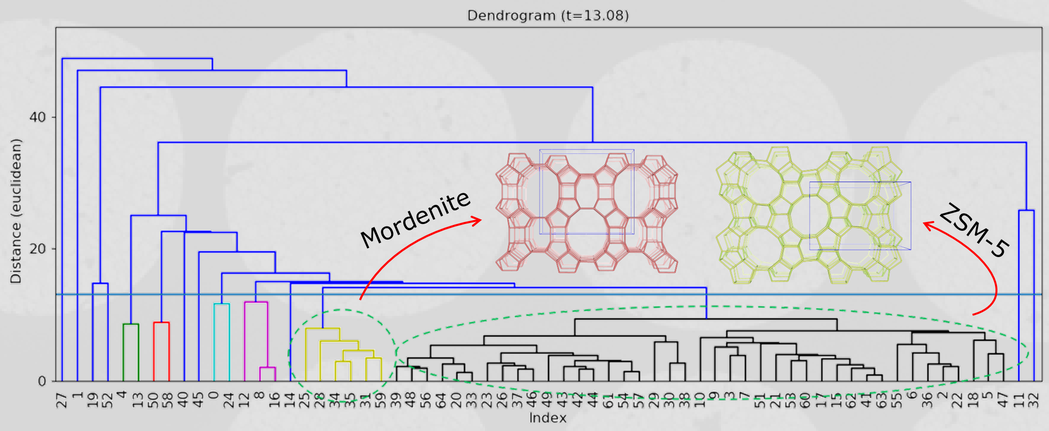


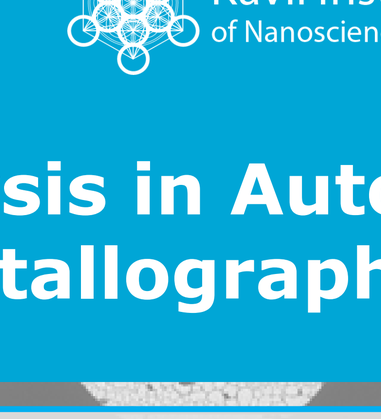
ZSM-5

Code: http://github.com/stefsmeets/edtools

Phase analysis

- Mix 2 crystalline zeolite phases
- Find lattice parameters from ED data
- Lattice-based clustering: group individual crystalline phases

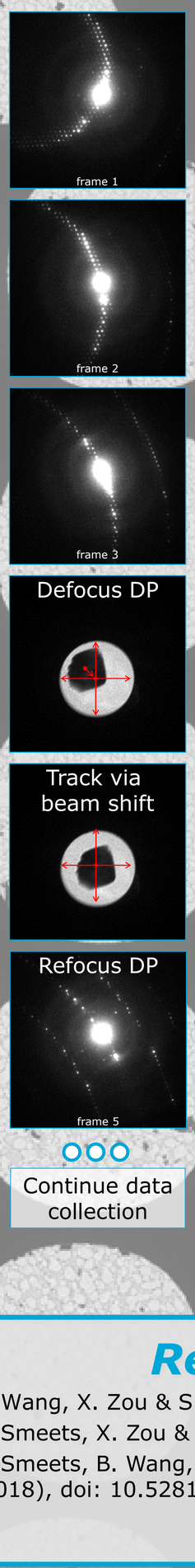




Probe size <1 µm

Screen for crystals

- Screen grid at low magnification
- Find crystals for 3D ED data collection
- Identify individual crystalline phases



Continue data collection

Automated crystal tracking

- Defocus every N^{th} crystal
- Find relative offset
- Adjust beam shift deflectors
- Apply descan (image shift)

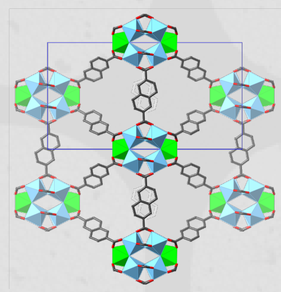
Software Instamatic (Python 3.6)

https://github.com/stefsmeets/instamatic

Sample	ZSM-5	PCN-416	ZSM-5 /mordenite	PST-20 /ZSM-25
Data collection time	6 h	2 h	2 h	4 h
Rotation (mean) /°	11.9	4.0	16.3	16.1
Rotation (max) /°	76.2	44.4	73.6	78.5
# suitable crystals	250	139	123	148
# data sets > 5°	126	66	89	99
# data sets > 20°	43	15	33	42
# indexed data sets	47	27	42/11	31/19
Resolution /Å	0.77	0.90	0.76/0.81	1.46/1.46

Serial vs. manual ED

Sample	Manual	SerialED
Space group	$I\bar{4}2m$	$I\bar{4}2m$
Cell /Å	16.7, 30.1	16.5, 29.8
# data sets	1	12
Resolution /Å	1.05	0.90
Completenes s	100%	97%
# uniq. refls	1918	2825
# obs. refls.	912	1254
***R*1**	0.258	0.216



MOF PCN-416

References & Funding

- B. Wang, X. Zou & S. Smeets, *IUCrJ* 6 (2019), 1-14.
- S. Smeets, X. Zou & W. Wan. *J. Appl. Crystallogr*. 51 (2018), 1262–73
- S. Smeets, B. Wang, M.O. Cichocka, J. Ångström & W. Wan, Instamatic 1.0. Zenodo (2018), doi: 10.5281/zenodo.1090388

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