Melk @ Crystallographic computing forum

2019-08-17

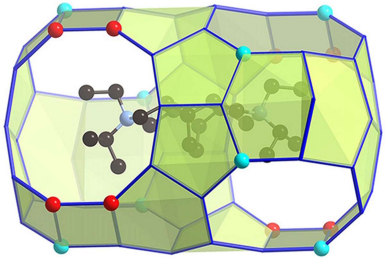
Glue it all together with Python: Automating electron diffraction data collection

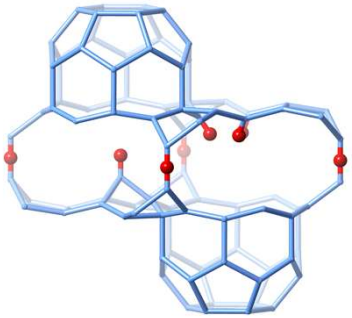
Stef Smeets

Kavli Institute of Nanoscience Delft

TU Delft

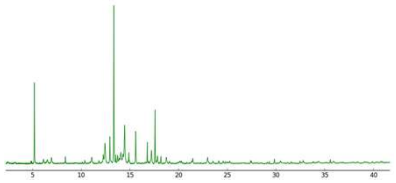
Zeolites



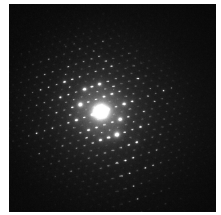


Structure determination and characterization

Method development



Powder diffraction



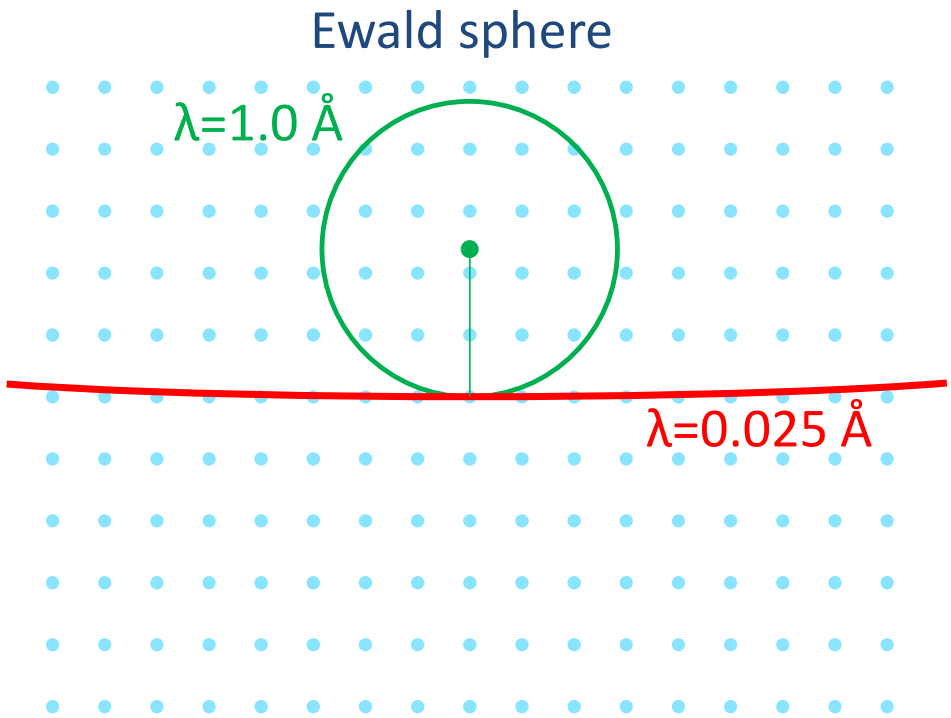
Electron diffraction

Crystallography

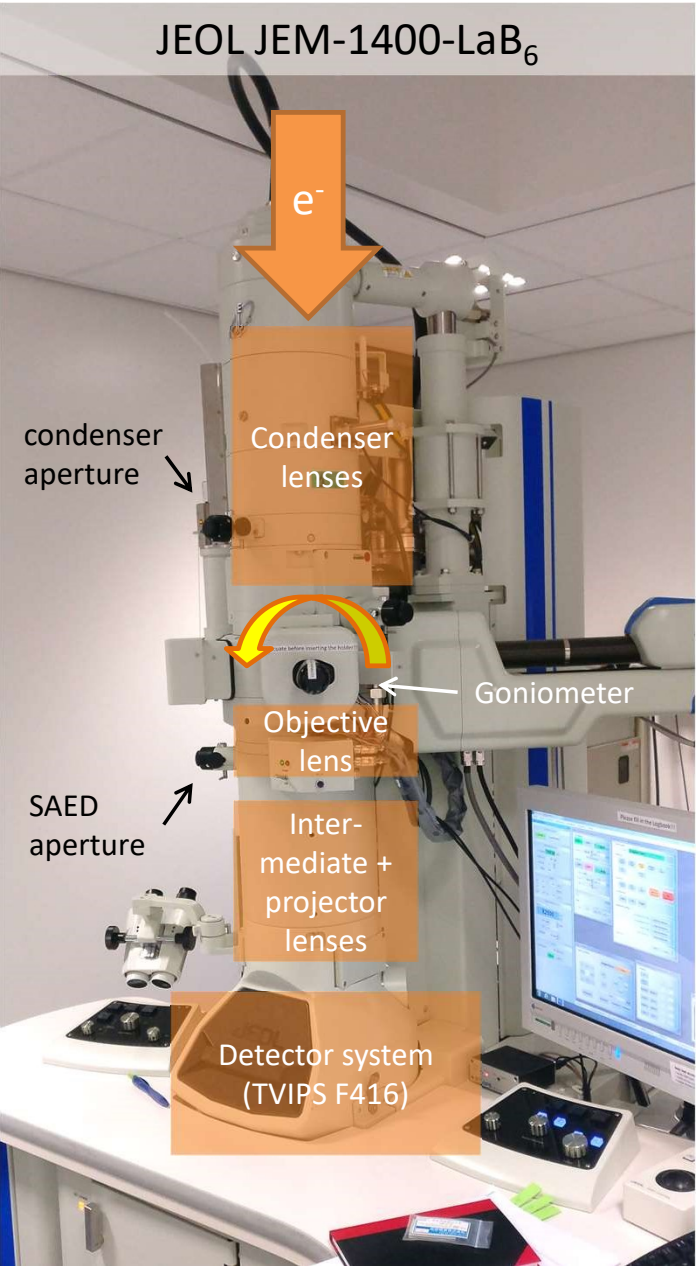
Programming

https://github.com/stefsmeets

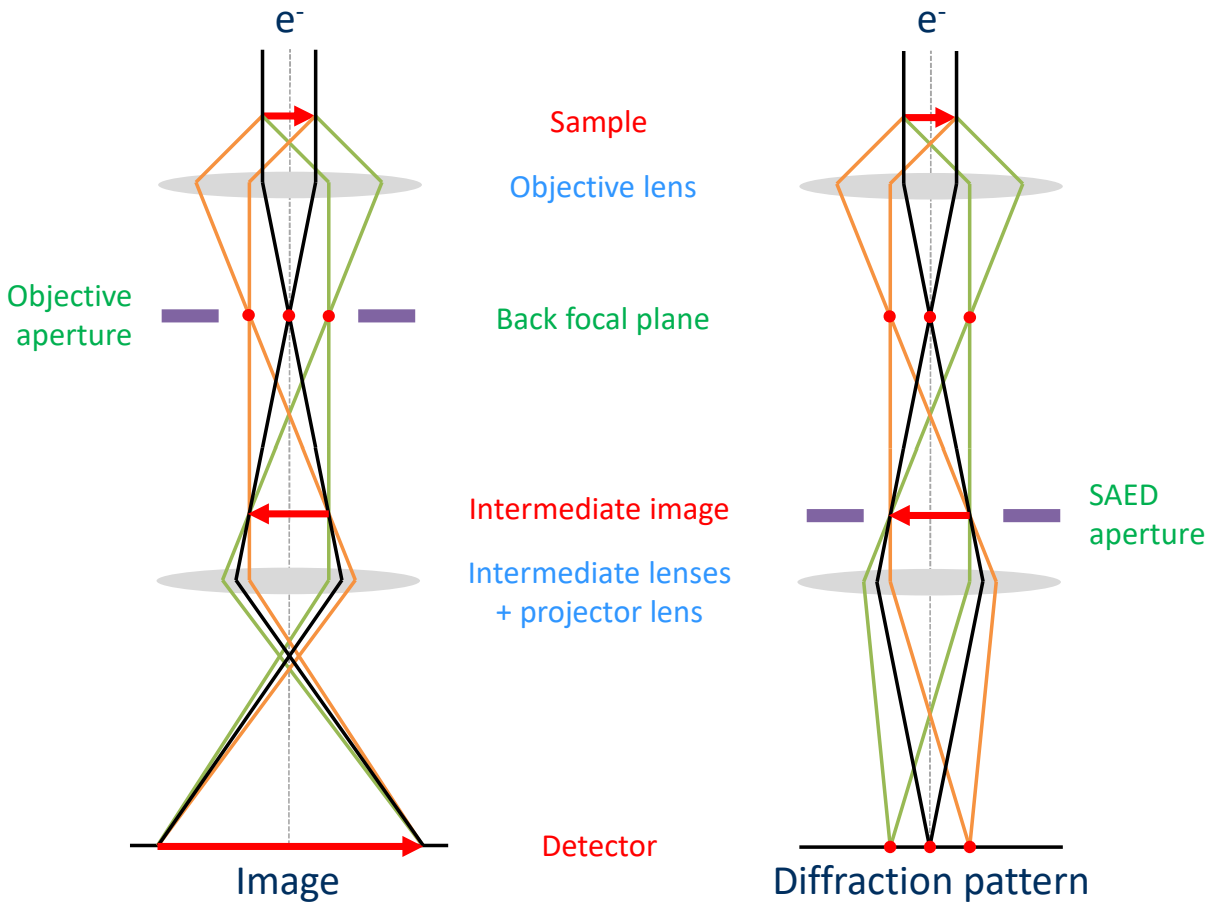
Electrons as a radiation source



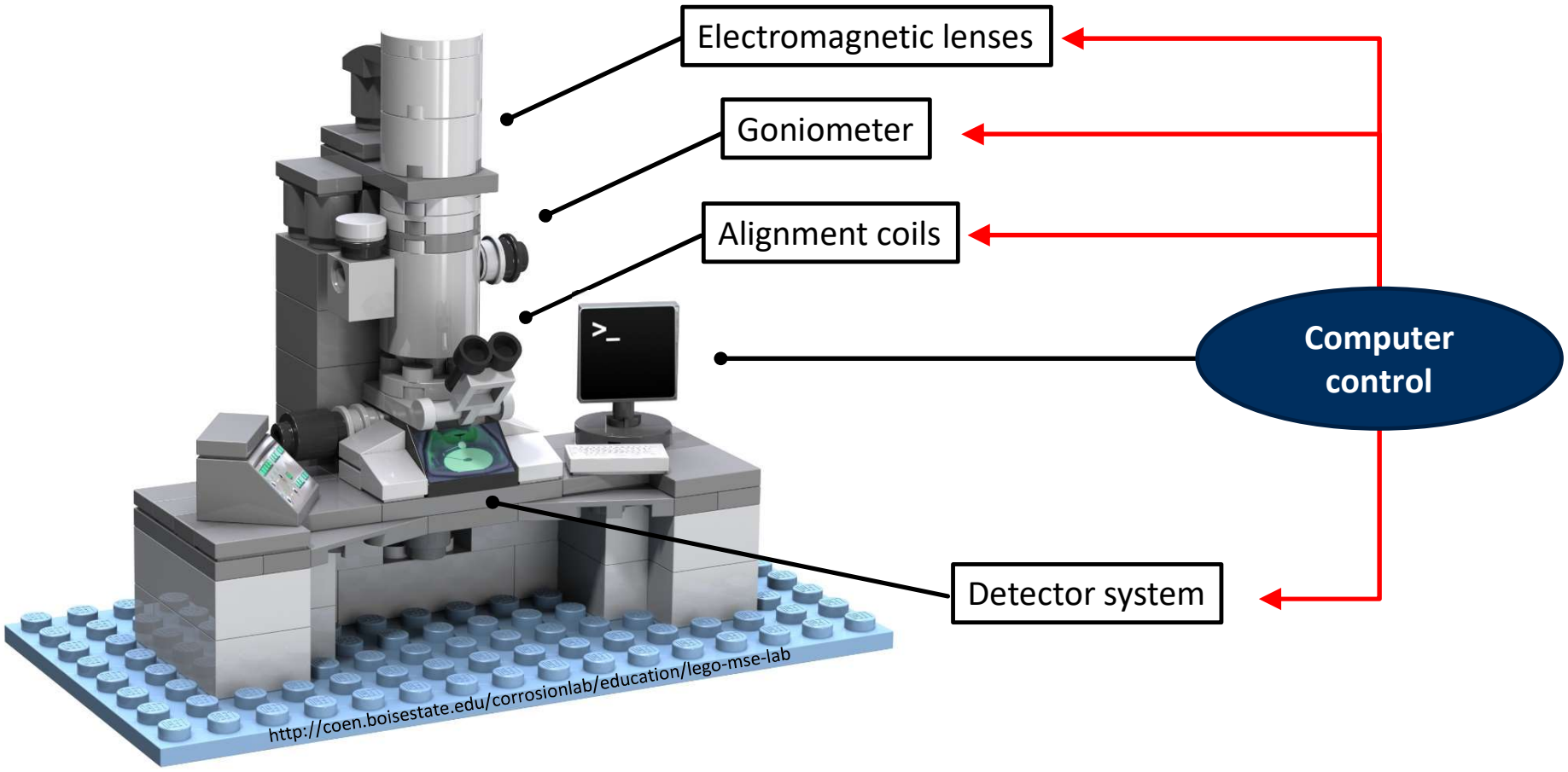
- Accelerating voltage: 100 to 300 keV
- Wavelength: 0.0251 Å @ 200 keV
- Probe electrostatic potential
- Strong interaction (10^6 stronger than X-rays)
- Require small samples (< 1 µm)
- High vacuum (<10^{-3} mbar)

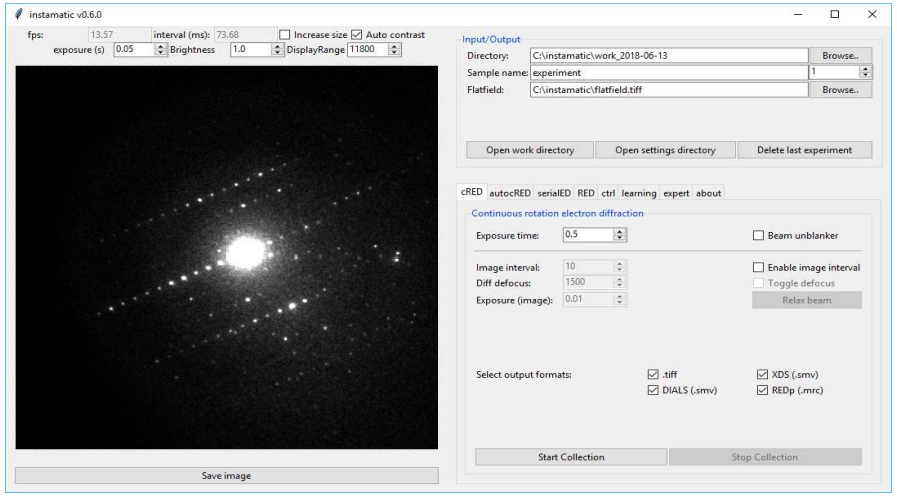


Electron ‘diffractometer’



The electron microscope as a giant toy for nanoscience





- Modular GUI
- Crystal finder
- Crystal tracking
- Neural network
- Calibrations

Instamatic **(Python3.6+)**

Serial rotation electron diffraction

Serial electron diffraction

Data reduction server

Continuous rotation electron diffraction

- .tiff (patterns + images)
- *REDp* (.ed3d, .mrc)
- *XDS* (XDS.INP, .smv)
- DIALS (.bat files)

- .hkl files
- Phase analysis
- Input files

Microscope control

TFS Titan/Themis Z

JEOL 1400/2100/3200

Simulated

Camera interface

ASI Cheetah

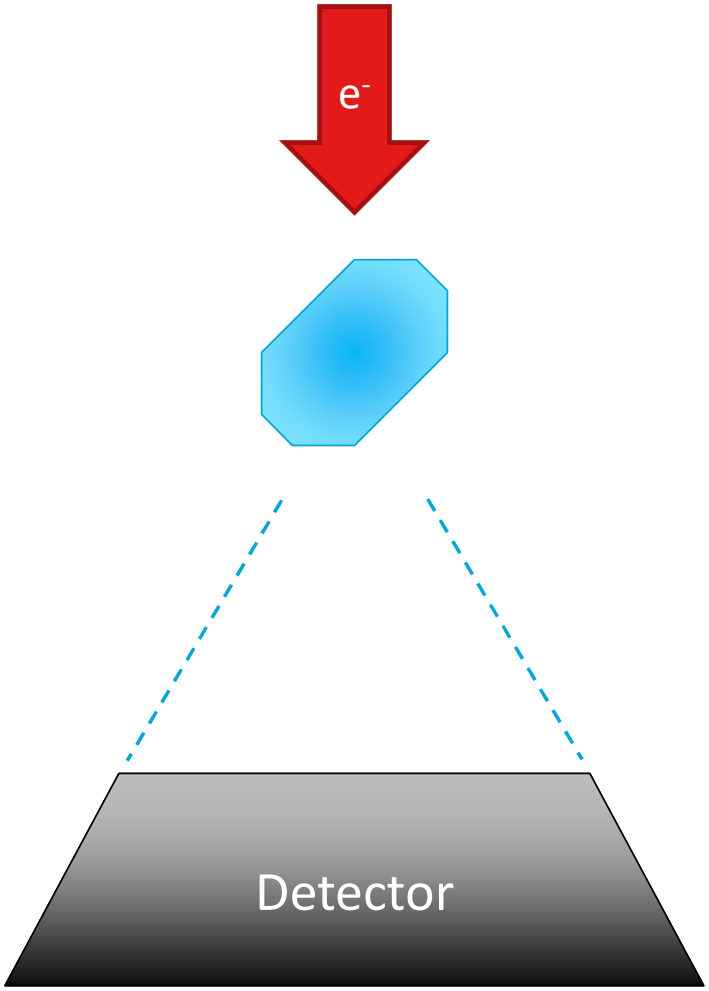
Gatan Orius

Simulated

TVIPS (X)F416

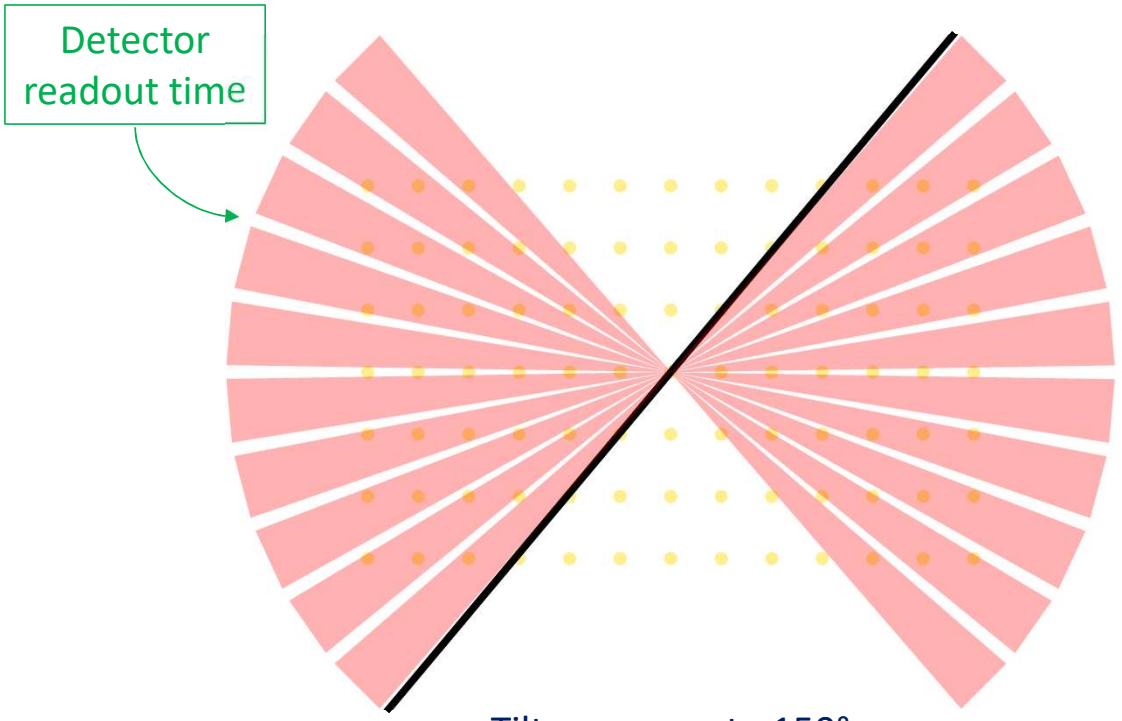
Source code:
http://github.com/stefsmeets/instamatic

3D Electron diffraction



Continuous rotation method

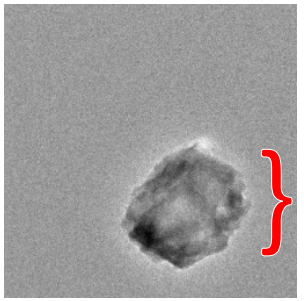
Nederlof *et al.*, Acta Cryst. D (2013), 69:1223
Nannenga *et al.*, Nat. Methods (2014), 11:927
Gemmi *et al.*, J. Appl. Cryst. (2015), 48:718
Cichocka *et al.*, J. Appl. Cryst. (2018), 51:1652

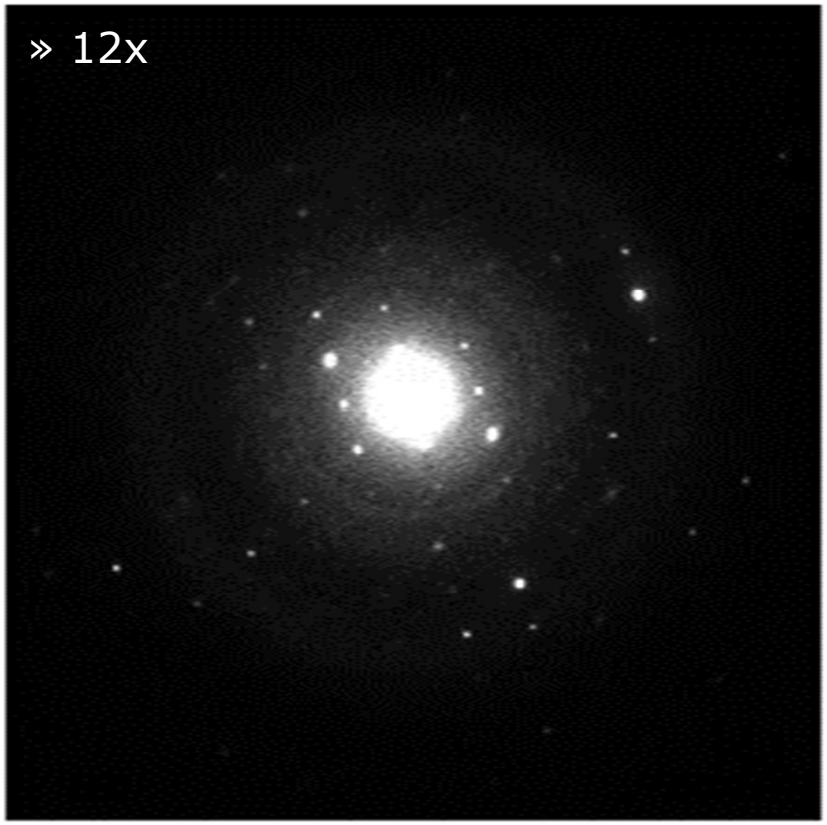


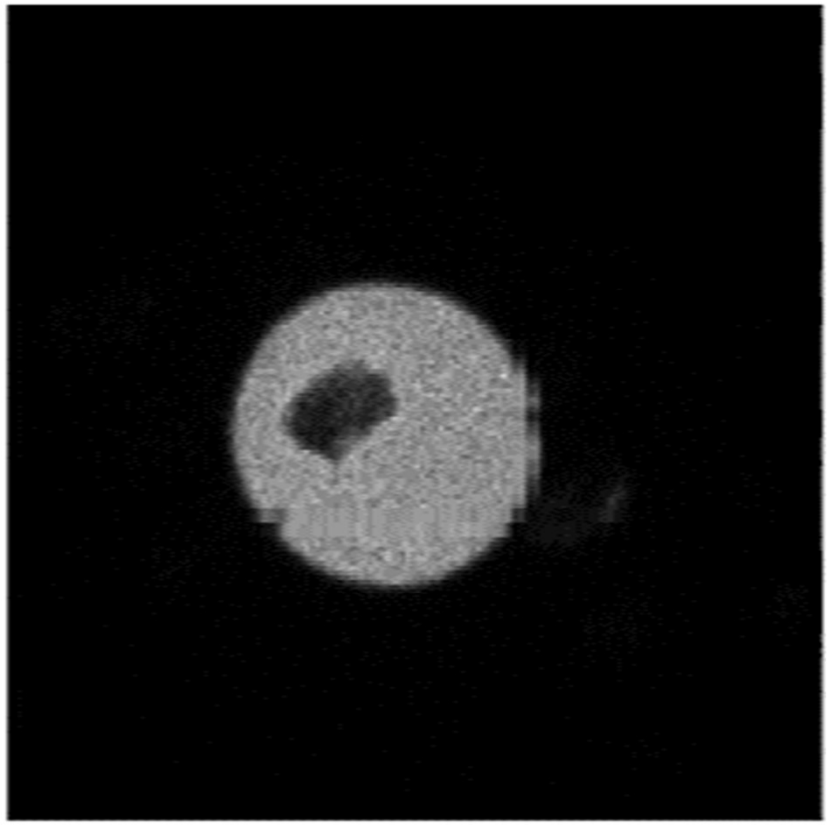
Tilt range: up to 150°
Oscillation angle: 0.1-0.5°
Rotation speed: 0.5-2.0°/s
<5 minutes

Zeolite mordenite

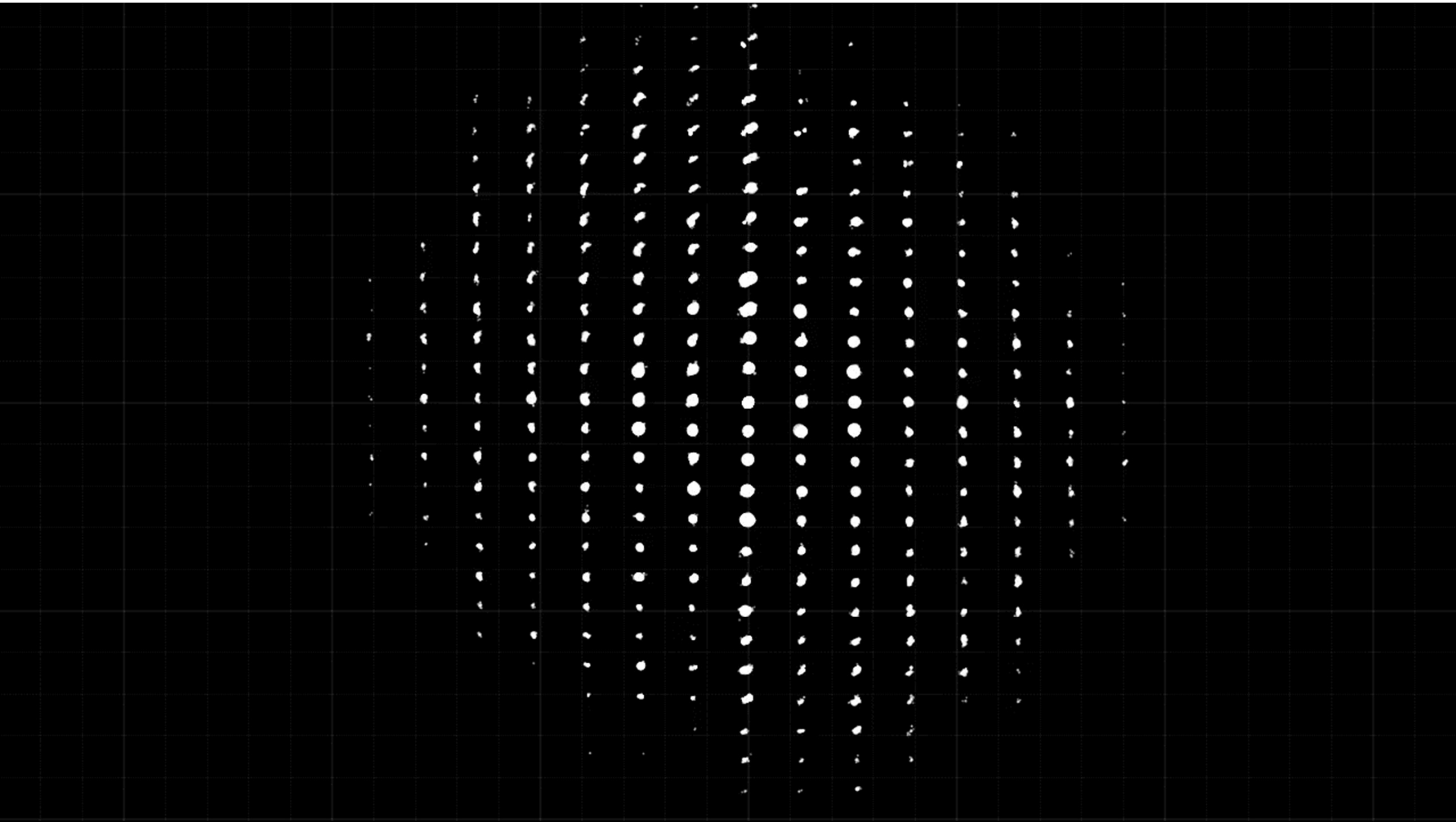
Rotate: -43.90° to 58.65° @ 0.45°/s (102.55°)
Exposure: 0.5 s, oscillation angle: 0.23°



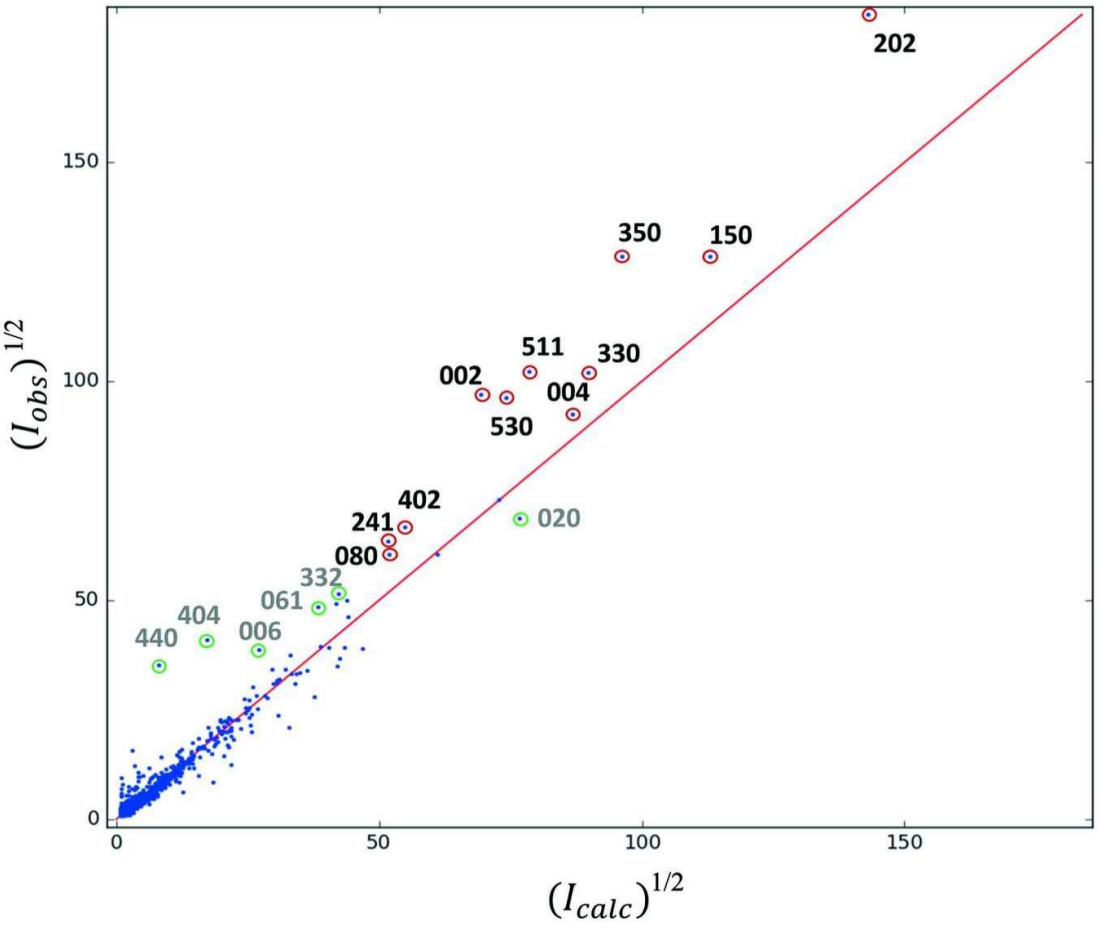




Cichocka *et al.*, *J. Appl. Crystallogr.* 51 (2018): 1652–61



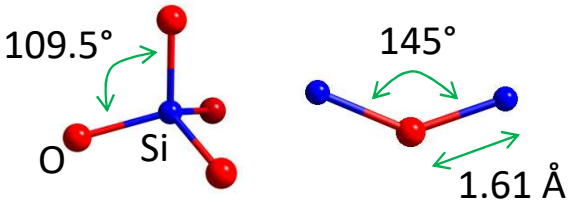
Refinement

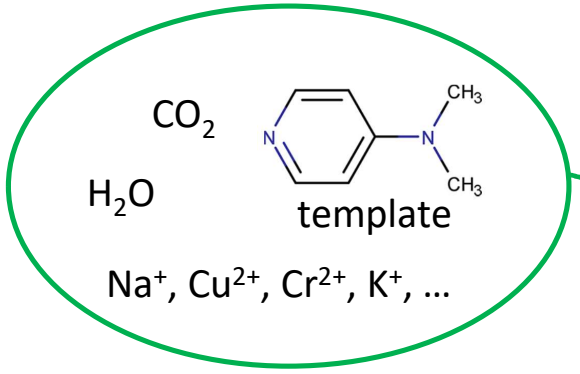


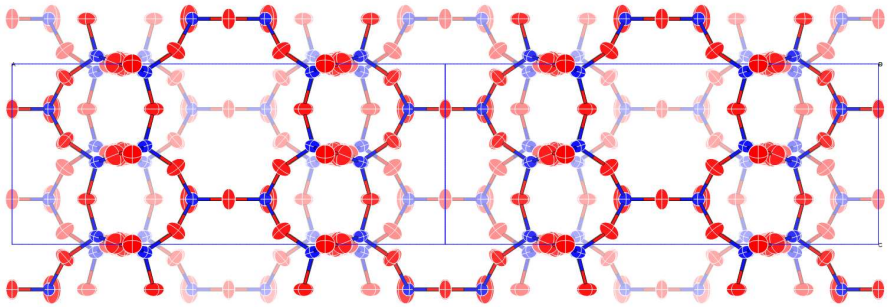
Chemical formula (refined)	$Si_{48}O_{96}$
Space group	*Cmcm* (63)
a (Å)	18.110
b (Å)	20.530
c (Å)	7.528
Resolution (Å)	0.80
No. of total reflections	5244
No. of unique reflections (all)	1585
No. of unique reflections [$F_o > 4\sigma(F_o)$]	1140
Refined parameters	96
Restraints	0
R_{int}	0.0878
$R1$ for $F_o > 4\sigma(F_o)$	0.1602
$R1$ for all data	0.1769
Goodness of fit	1.610

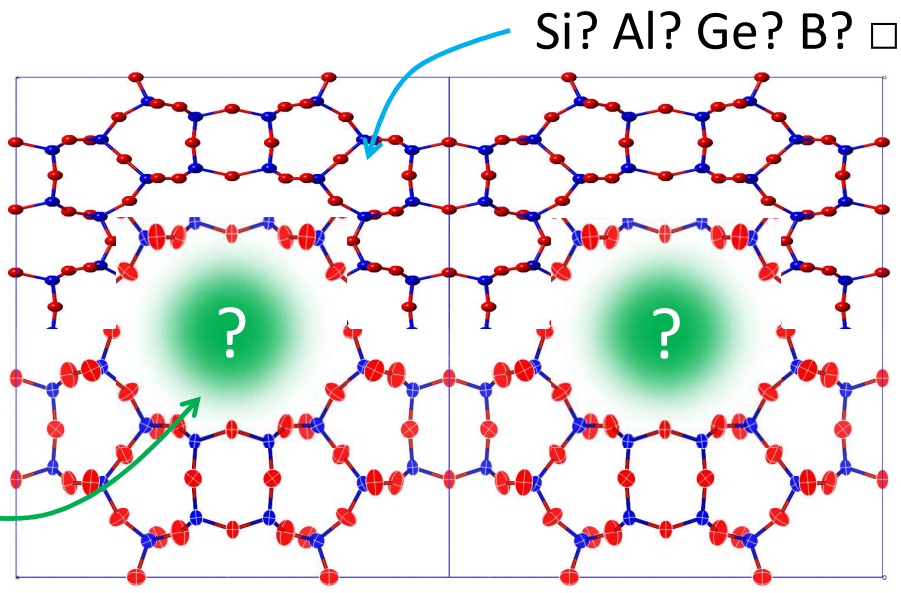
Framework structure

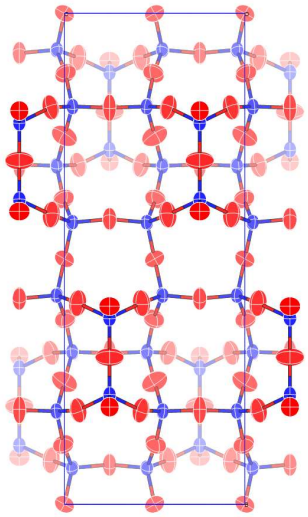
Si—O	1.614 ± 0.012 Å
Si—O—Si	109.5 ± 1.9°
O—Si—O	153.3 ± 12.0°





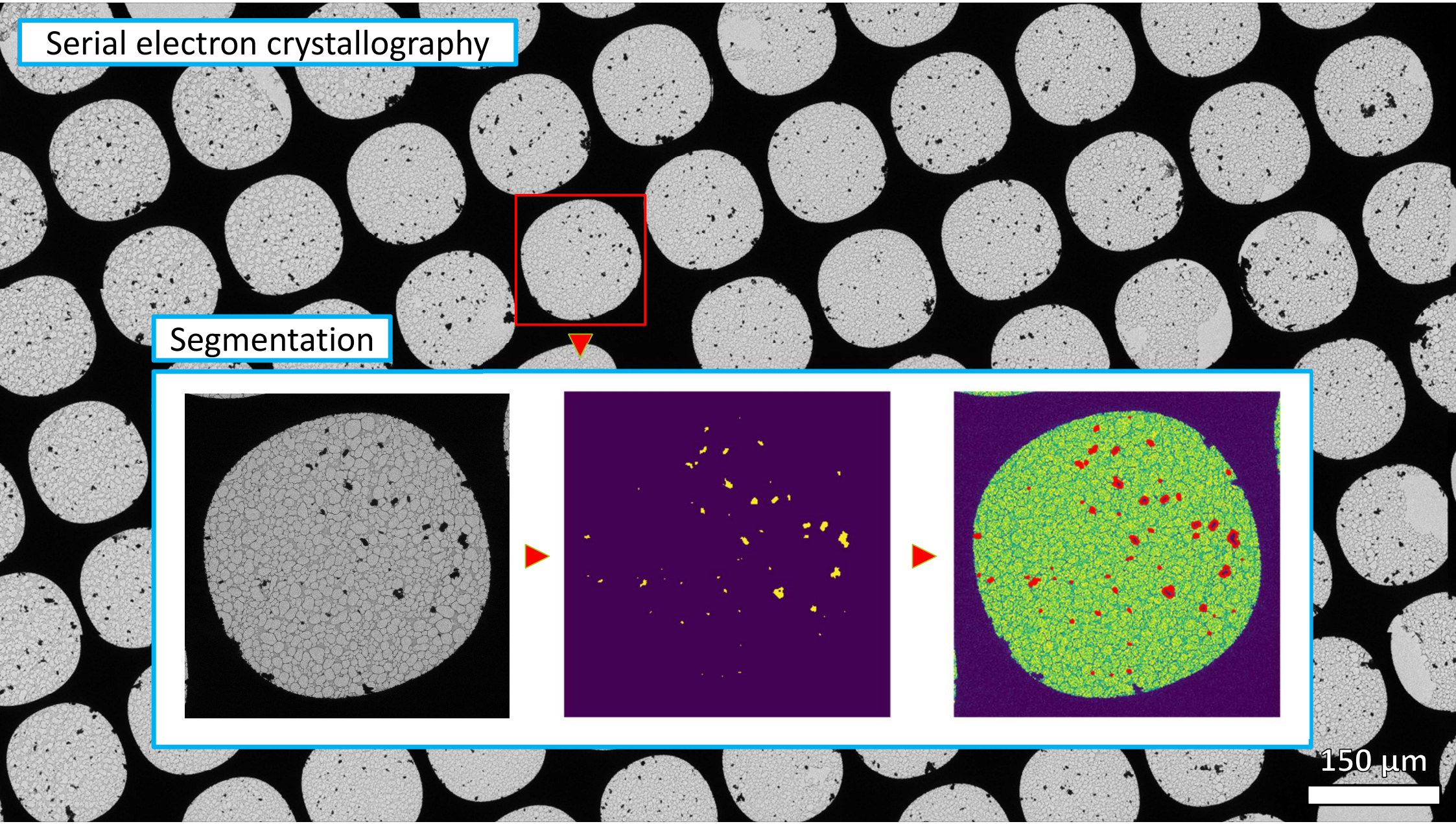


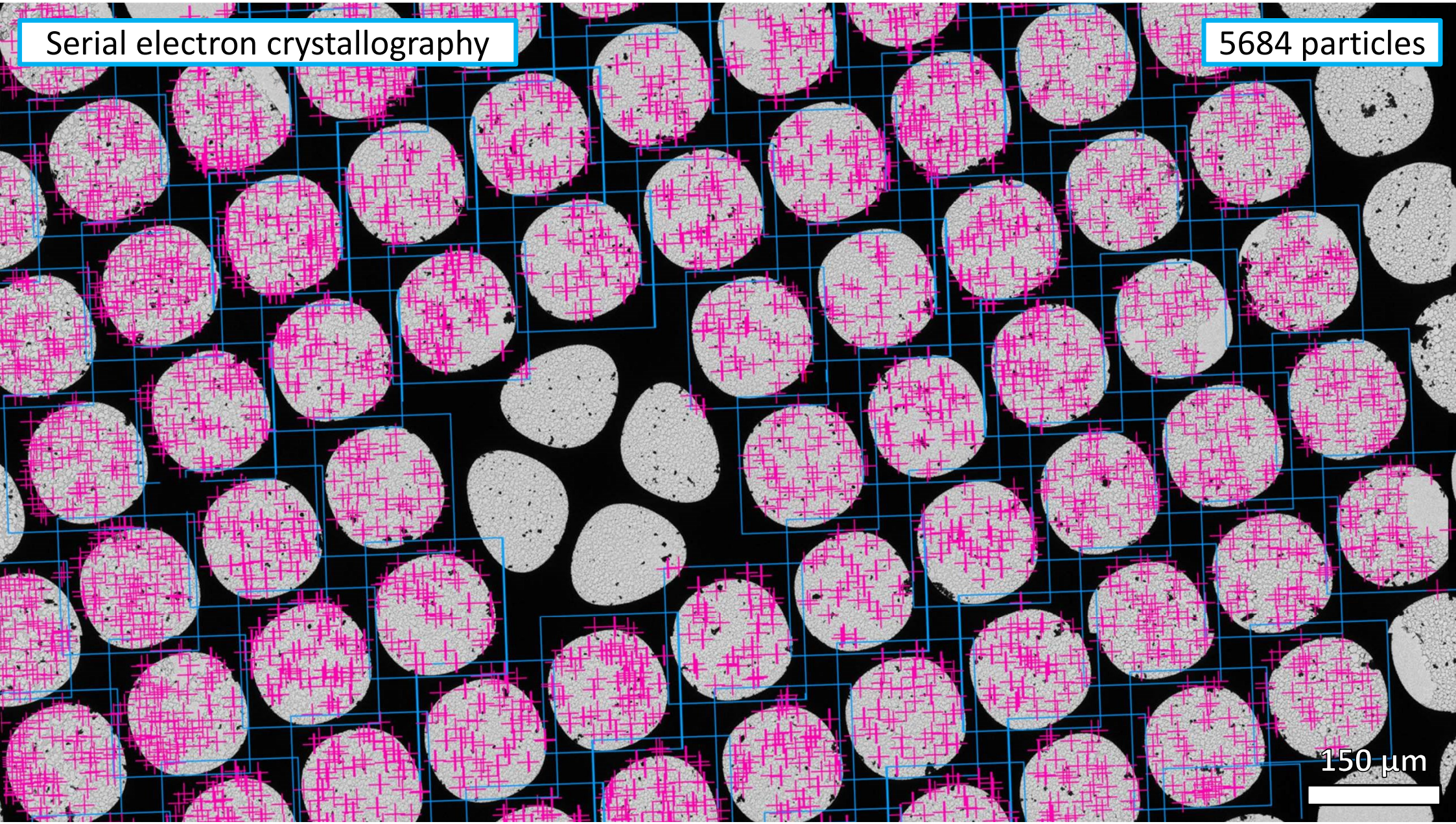




*R*1=0.160 (0.80 Å)

Cichocka *et al.*, *J. Appl. Crystallogr.* 51 (2018): 1652–61


Serial electron crystallography
Segmentation
150 µm

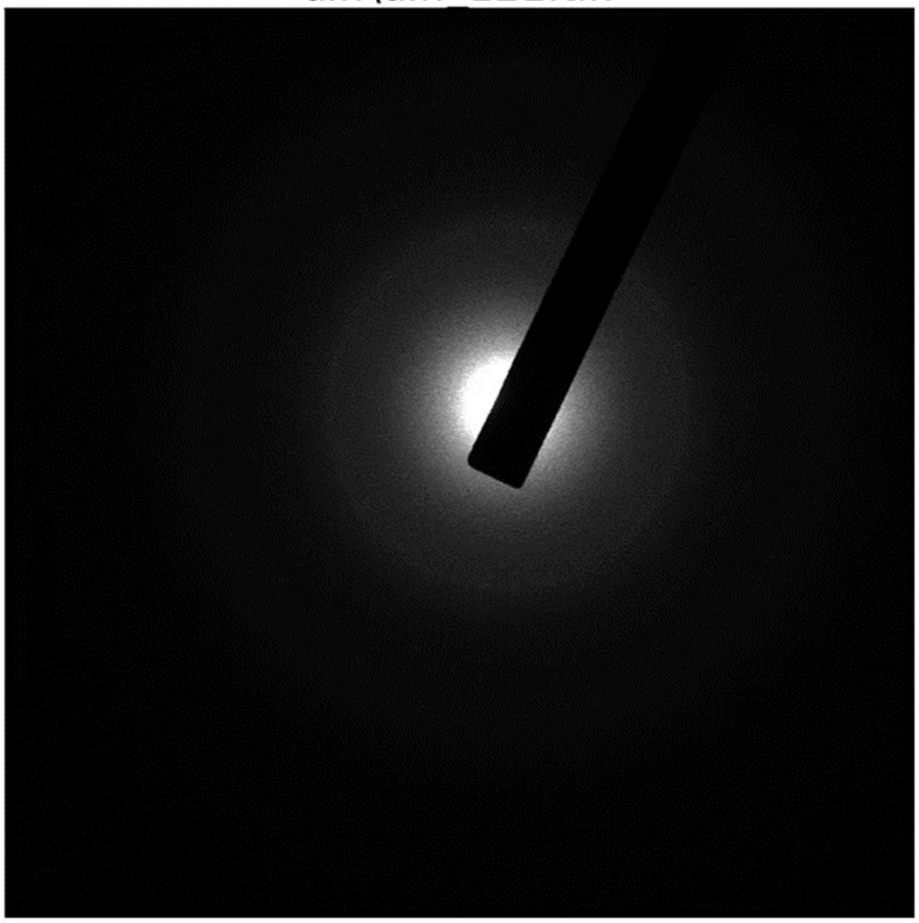

Serial electron crystallography
5684 particles
150 µm

Serial electron diffraction (SSZ-45)

JEOL 1400 LaB_6 @ 120 kV
401 images @ 400 ms/frame

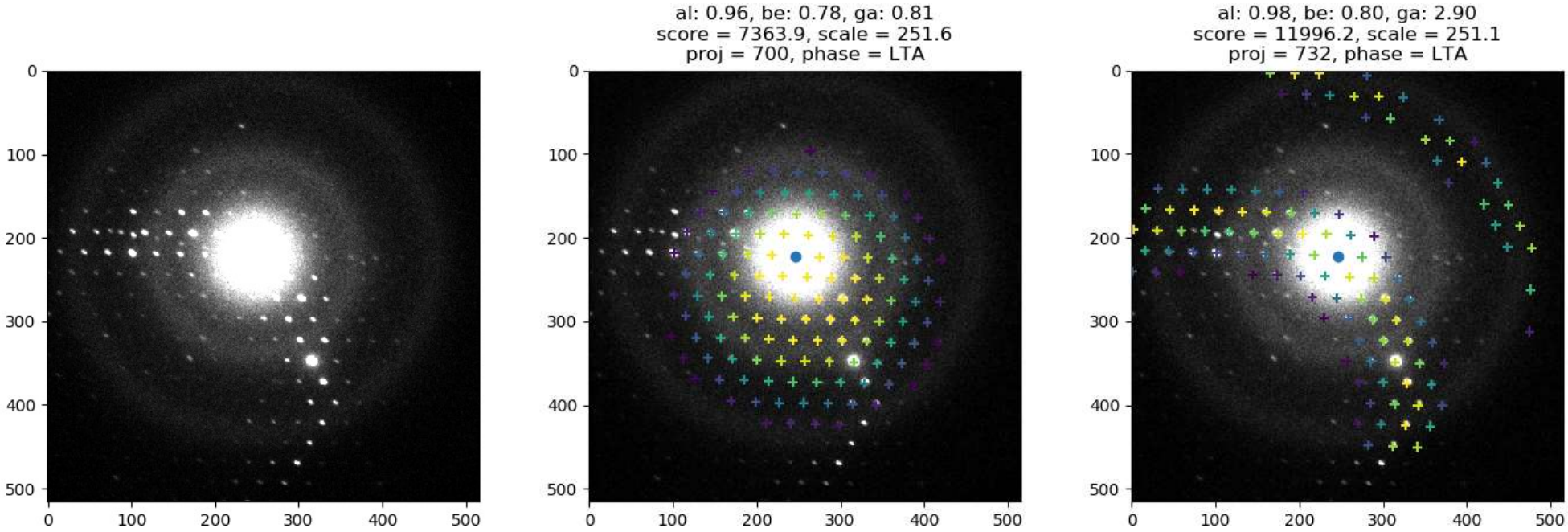
diff\image_121.tiff

diff\diff_121.tiff



Orientation finding

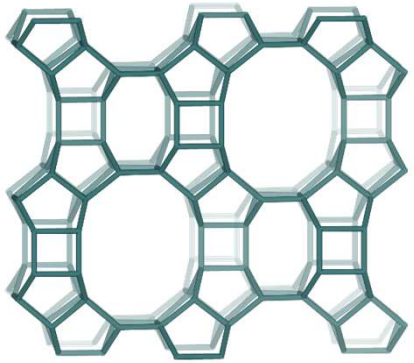
- Forward projection model using known lattice parameters
- Generate pattern library of all possible orientations (~1.5M in $P1$)
- Match best orientation and index data



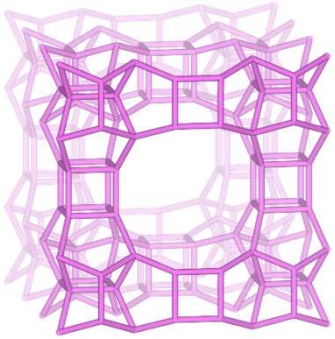
Smeets *et al.*, *J. Appl. Cryst.*, 2018, 51:1262

Structure determination

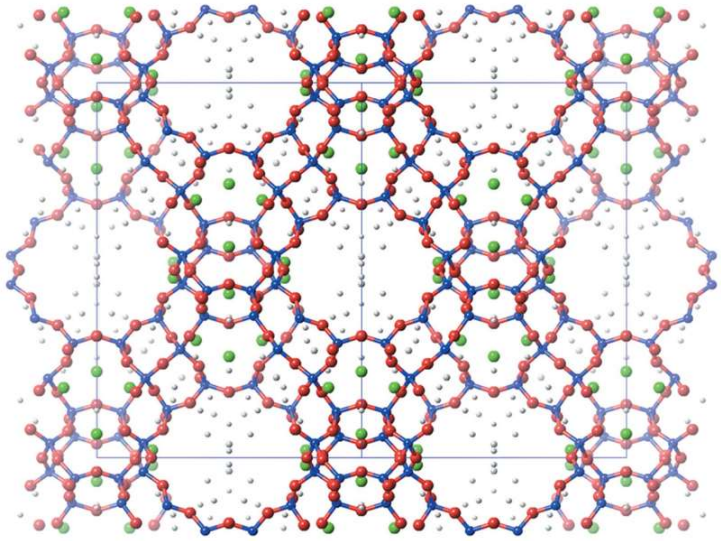
- Merge *hkl*-files using rank aggregation
- Combine data from many frames



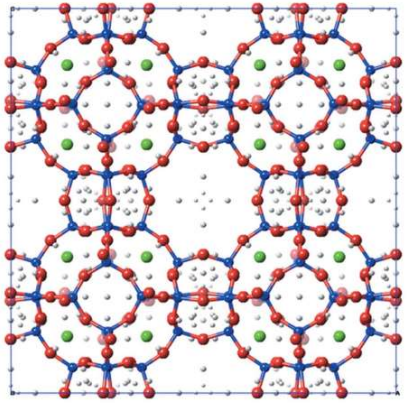
Mordenite



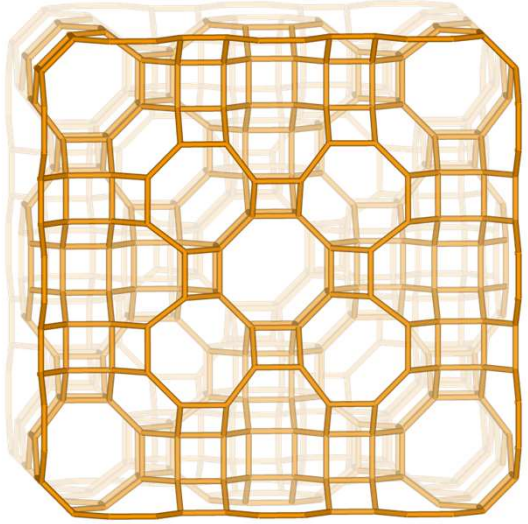
GeSi-BEC



Zeolite Y
(using 99 / 2506 frames)

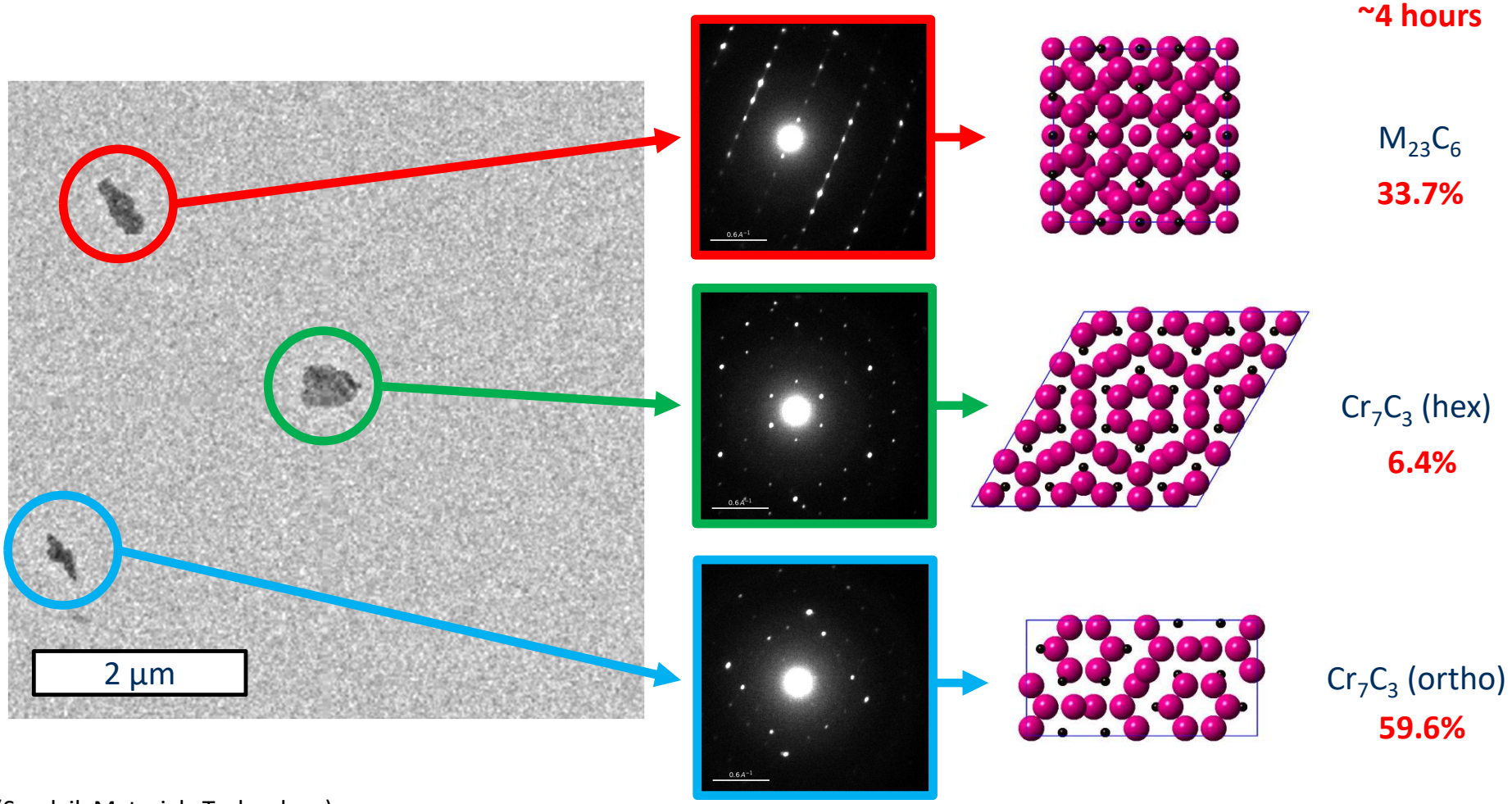


Zeolite A
(using 200 / 1107 frames)



ECR-18

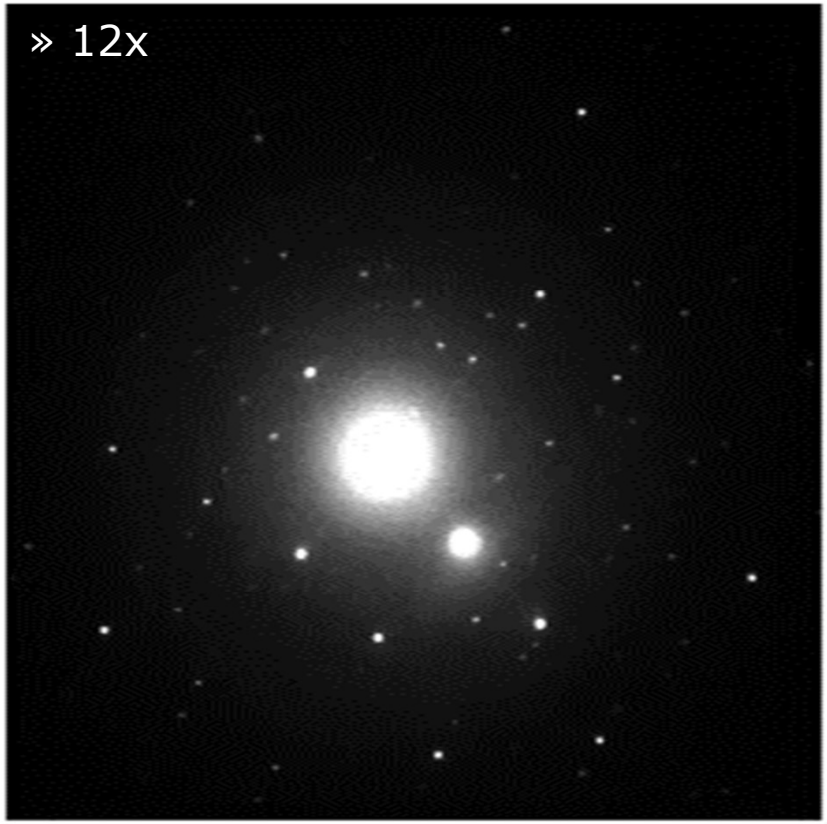
Quantitative phase analysis

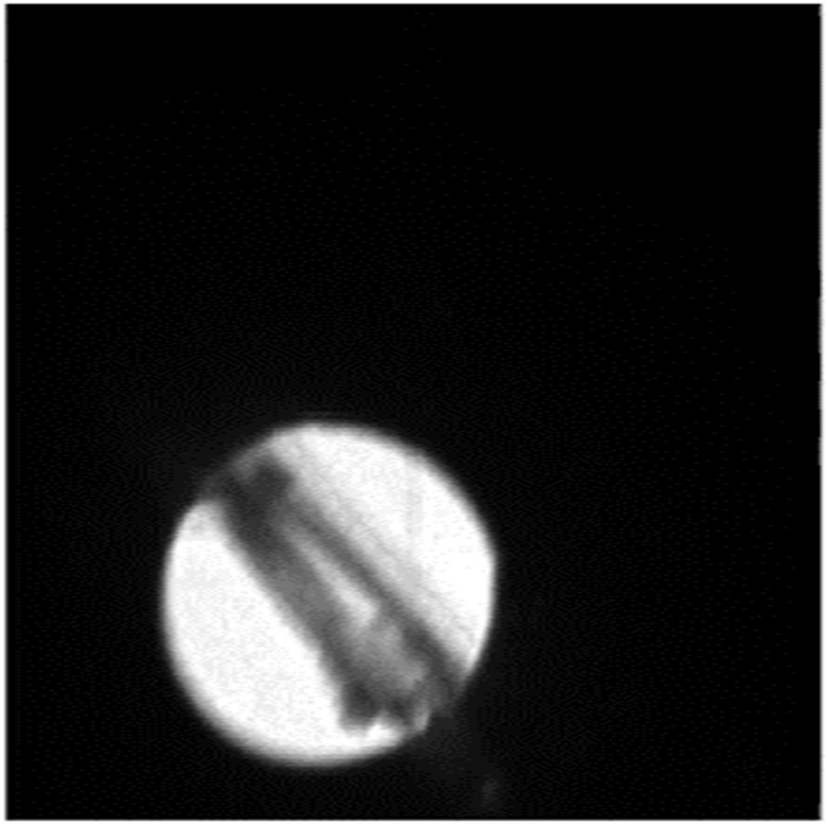


With Claes Olsson (Sandvik Materials Technology)
Smeets et al., *Steel Res. Int.* 90 (2019), 1800300

Automated crystal tracking

Rotation: -44.0 to 47.4° @ 0.76°/s (91.4°)
Exposure: 0.5 s, oscillation angle: 0.39°

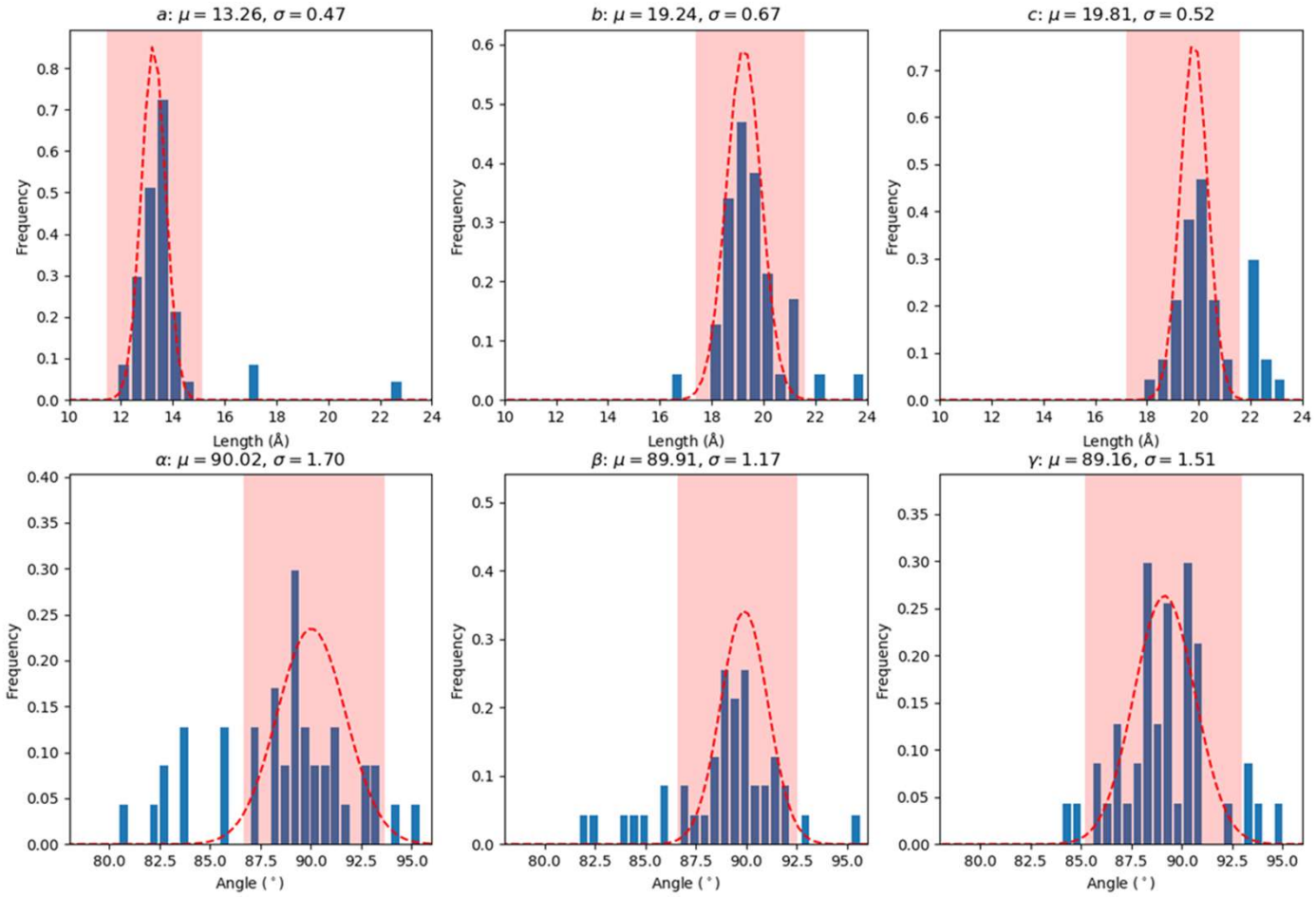




Bin Wang (Stockholm University)

Wang *et al.*, *IUCrJ* 6 (2019), doi: 10.1107/S2052252519007681

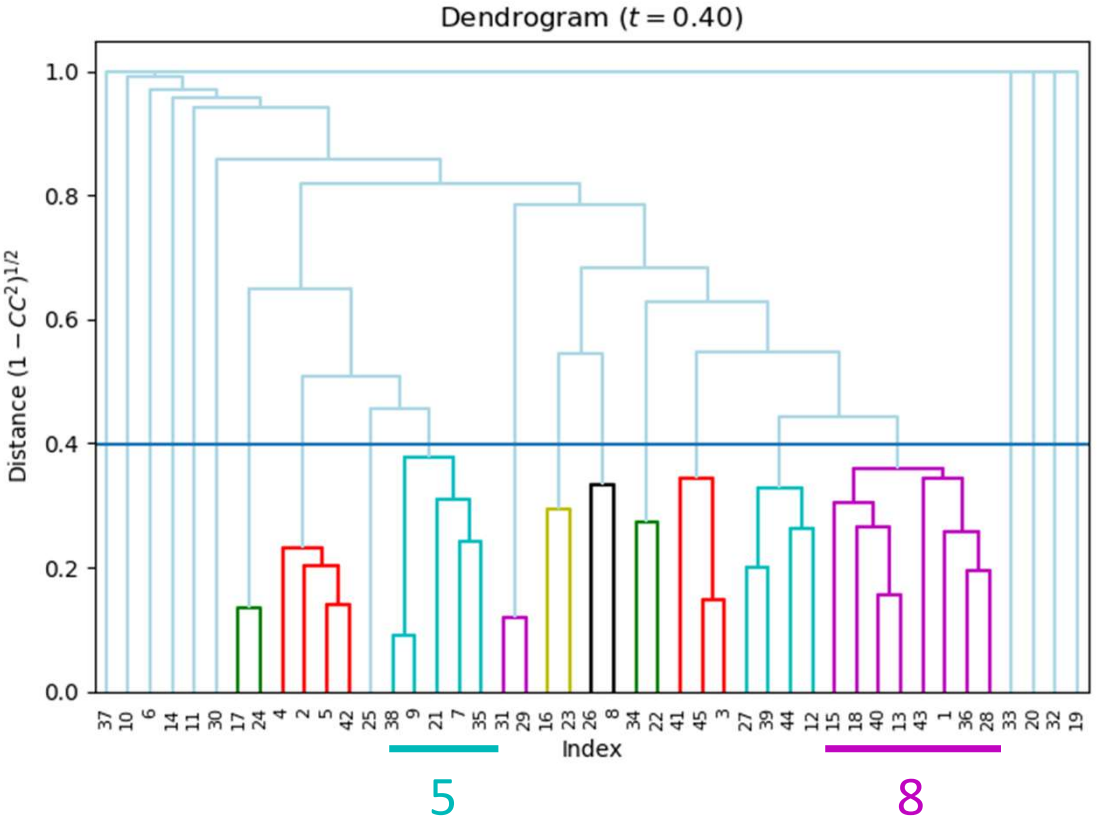
Find average unit cell (ZSM-5)

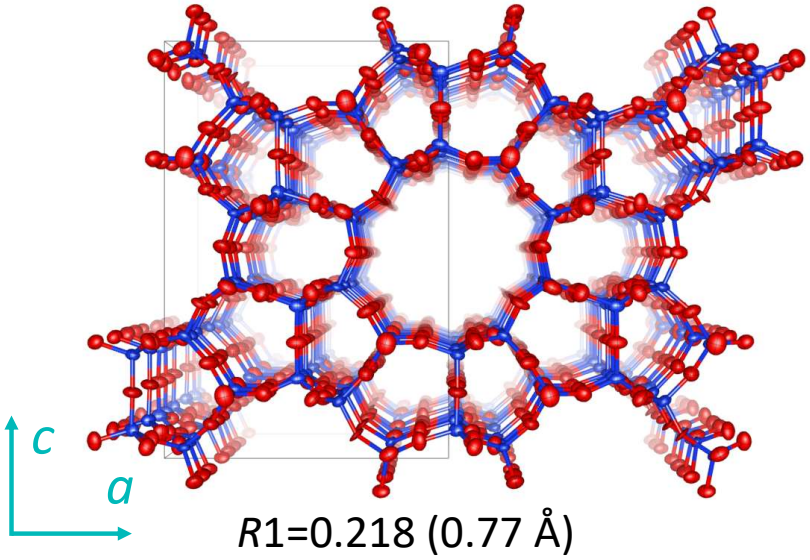


a=13.3(5) Å
b=19.2(7) Å
c=19.8(5) Å
α=90.0(1.7)°
β=89.9(1.2)°
γ=89.16(1.5)°

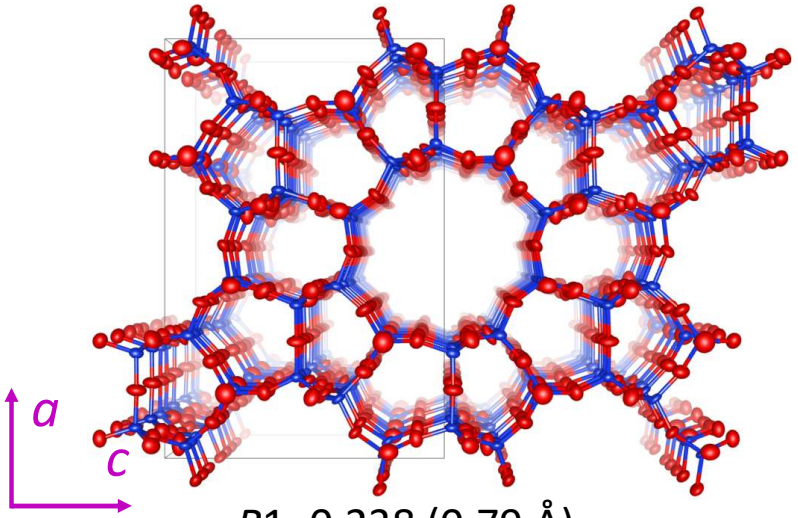
Orthorhombic
C-centered

Cluster analysis (intensities)

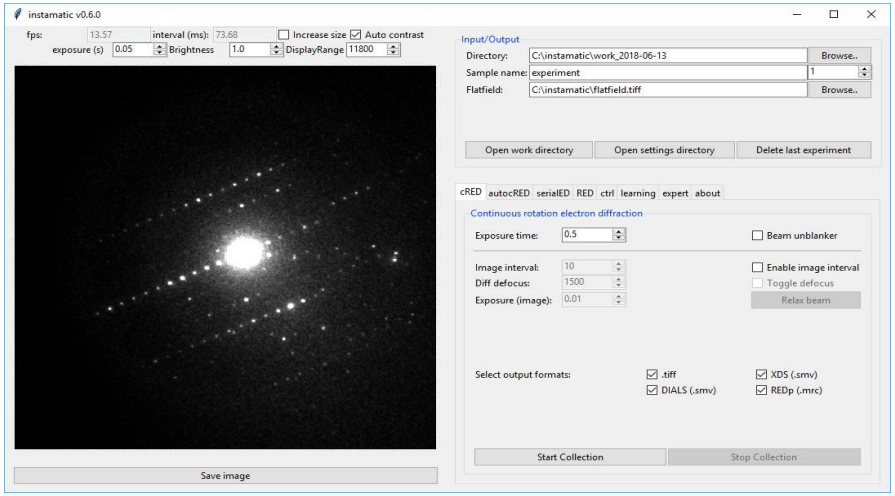




*R*1=0.218 (0.77 Å)



*R*1=0.238 (0.79 Å)



- Modular GUI
- Crystal finder/tracking
- Neural network
- Calibrations
- Automated experiments

Instamatic **(Python3.6+)**

Serial rotation electron diffraction

Serial electron diffraction

Continuous rotation electron diffraction

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TVIPS (X)F416

Source code:
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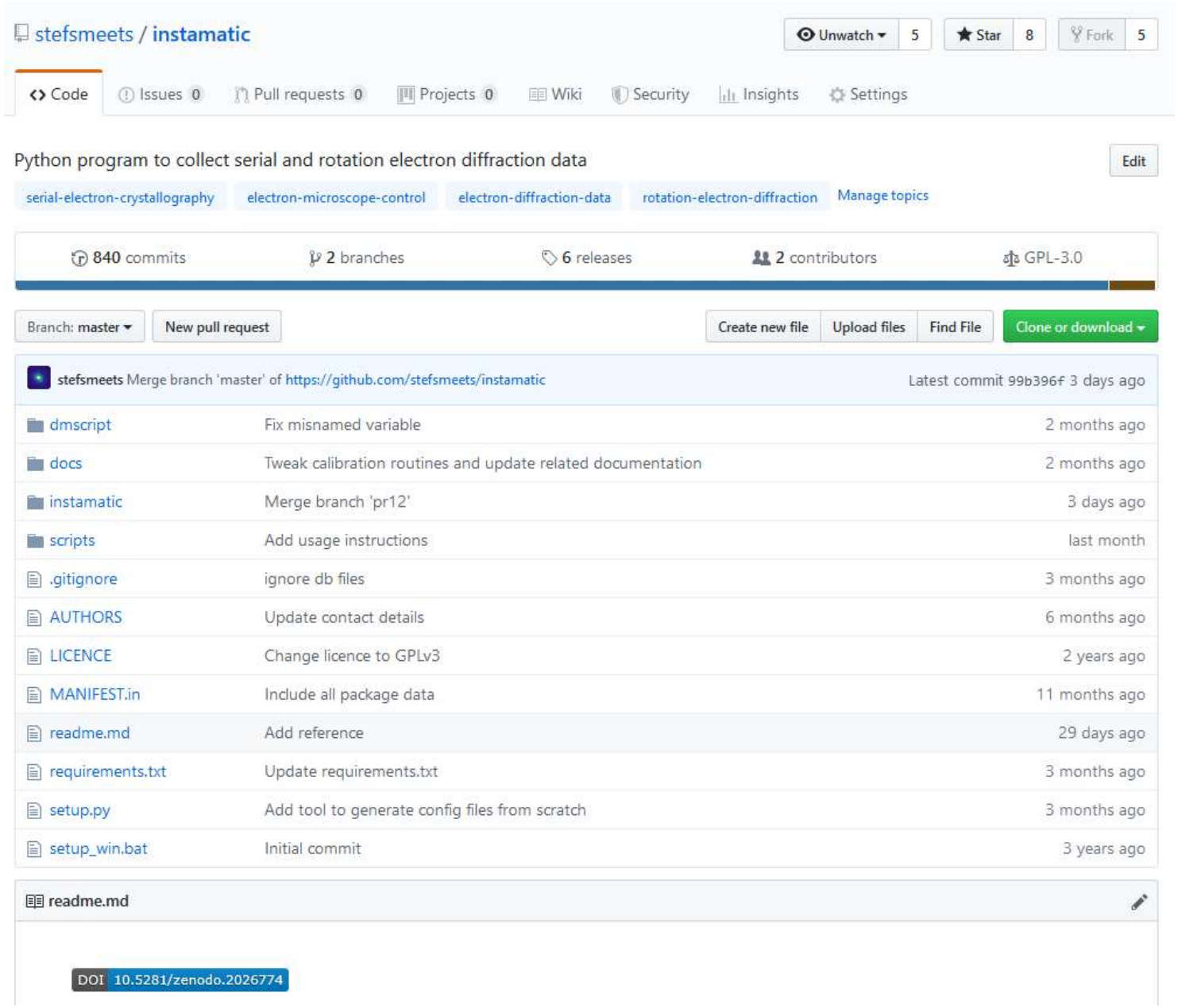
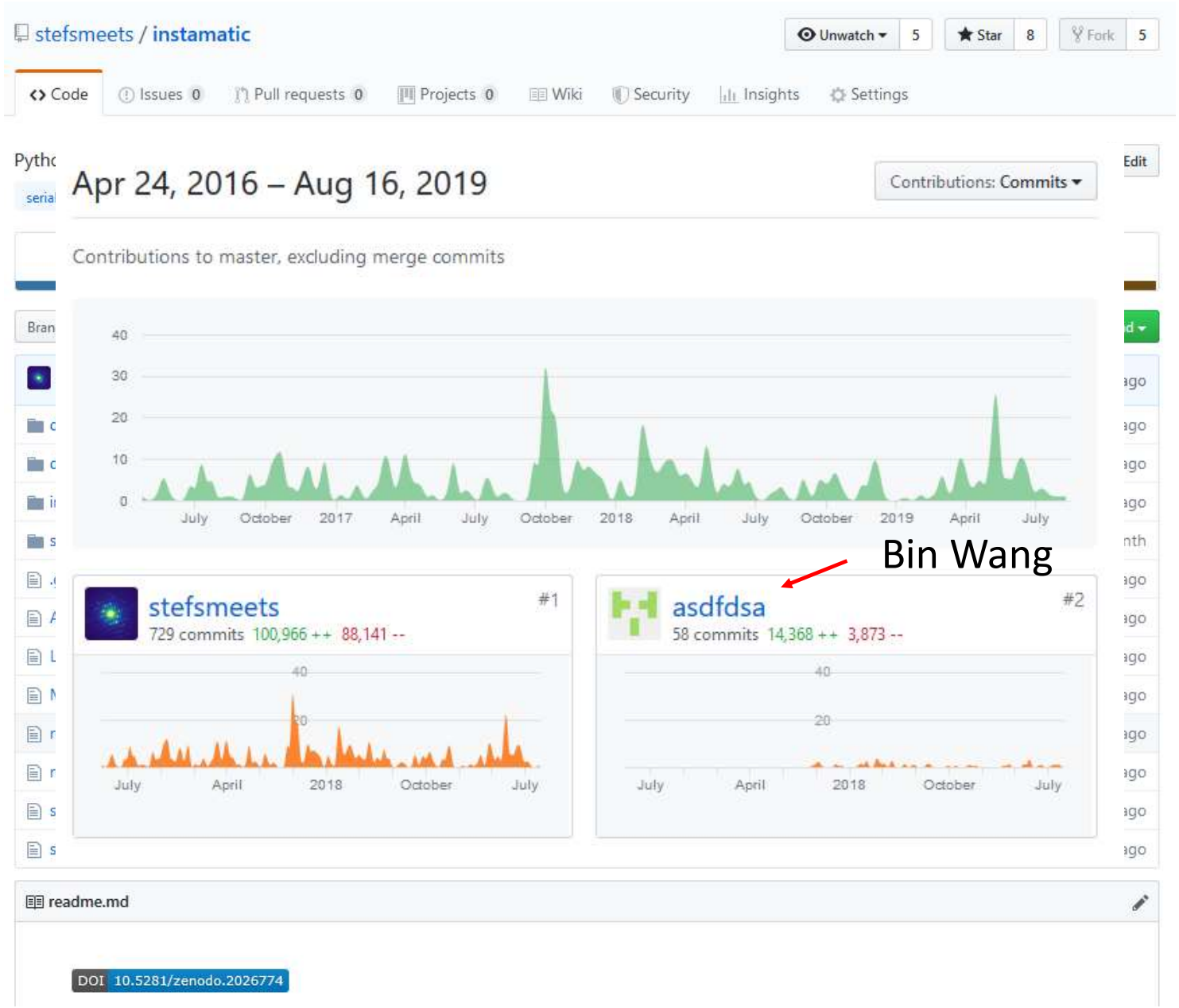

stefsmeets / instamatic
Unwatch 5
Star 8
Fork 5
Code
Issues 0
Pull requests 0
Projects 0
Wiki
Security
Insights
Settings
Python program to collect serial and rotation electron diffraction data
Edit
serial-electron-crystallography
electron-microscope-control
electron-diffraction-data
rotation-electron-diffraction
Manage topics
840 commits
2 branches
6 releases
2 contributors
GPL-3.0
Branch: master
New pull request
Create new file
Upload files
Find File
Clone or download
stefsmeets Merge branch 'master' of https://github.com/stefsmeets/instamatic
Latest commit 99b396f 3 days ago
dmscript Fix misnamed variable 2 months ago
docs Tweak calibration routines and update related documentation 2 months ago
instamatic Merge branch 'pr12' 3 days ago
scripts Add usage instructions last month
.gitignore ignore db files 3 months ago
AUTHORS Update contact details 6 months ago
LICENCE Change licence to GPLv3 2 years ago
MANIFEST.in Include all package data 11 months ago
readme.md Add reference 29 days ago
requirements.txt Update requirements.txt 3 months ago
setup.py Add tool to generate config files from scratch 3 months ago
setup_win.bat Initial commit 3 years ago
readme.md
DOI 10.5281/zenodo.2026774


stefsmeets / instamatic
Unwatch 5
Star 8
Fork 5
Code
Issues 0
Pull requests 0
Projects 0
Wiki
Security
Insights
Settings
Apr 24, 2016 – Aug 16, 2019
Contributions: Commits
Contributions to master, excluding merge commits
40
30
20
10
0
July
October
2017
April
July
October
2018
April
July
October
2019
April
July
Bin Wang
stefsmeets
#1
729 commits 100,966 ++ 88,141 --
asdfdsa
#2
58 commits 14,368 ++ 3,873 --
readme.md
DOI 10.5281/zenodo.2026774

Python as a glue language

> *Without Python, large amounts of C/C++ code often have to be written just to provide a flexible enough input mechanism so that scientists can feed the program its data, in all the variations that are required for reasons of experimental setup. Python can be used to write a much more flexible input mechanism in a much shorter time.*

Guido van Rossum (1998)
'*Python as a glue language*'
https://www.python.org/doc/essays/omg-darpa-mcc-position/

Python as a glue language

> *Python is ideal for oddball integration tasks.*

Guido van Rossum (1998)
'*Python as a glue language*'
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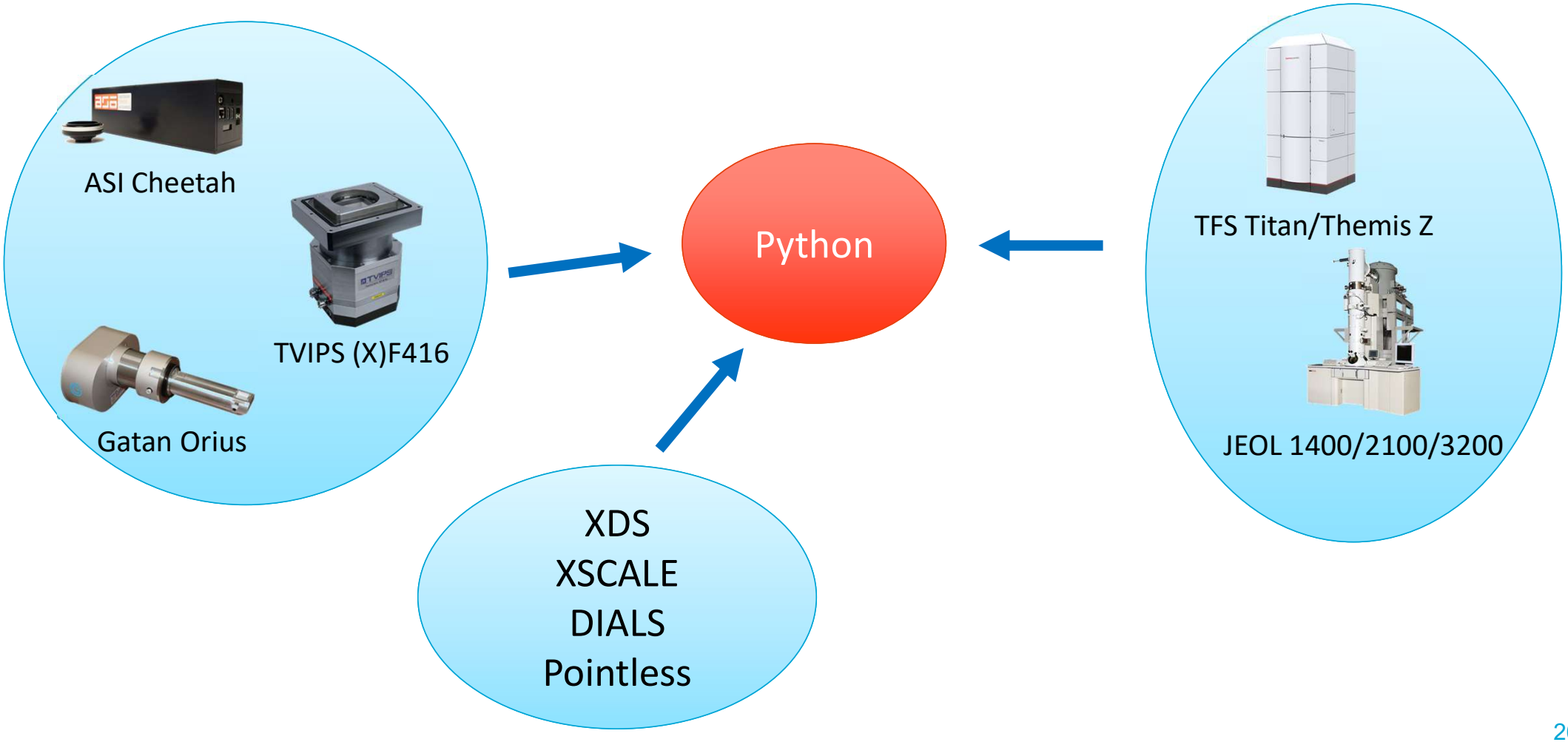
Python as a glue language



Python as a glue language

- Subprocess
- comtypes
- ctypes
  - call C functions from Python
  - Access Windows API
- Sockets
  - Netcat
  - Echo server
- Windows Subsystem for Linux
- Pyautogui

instamatic.camera.Camera

ASI Cheetah

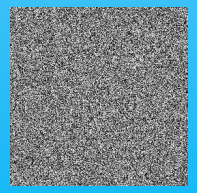
Through DLL (C++)
ctypes

TVIPS (X)F416

Through EMMENU
comtypes

Gatan Orius

Through DM plugin
COM -> DLL (C++) -> **ctypes**



Simulated

Python

import

Instamatic
main program

instamatic.server.cam_server (socket server)

instamatic.camera.Camera

ASI Cheetah

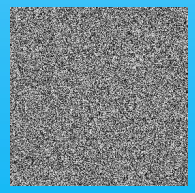
Through DLL (C++)
ctypes

TVIPS (X)F416

Through EMMENU
comtypes

Gatan Orius

Through DM plugin
COM -> DLL (C++) -> **ctypes**



Simulated

Python

TCP/UDP

(socket client)

Instamatic
main program

Instamatic
main program

import

instamatic.TEMController.Microscope

TFS Titan/Themis Z

TEMScripting (COM)
comtypes

JEOL 1400/2100/3200

TEMCOM (COM)
comtypes

Simulated

Python

(socket client)

Instamatic
main program

TCP/UDP

instamatic.server.tem_server (socket server)

instamatic.TEMController.Microscope

TFS Titan/Themis Z

TEMScripting (COM)
comtypes

JEOL 1400/2100/3200

TEMCOM (COM)
comtypes

Simulated

Python

3D electron diffraction (discrete rotation)

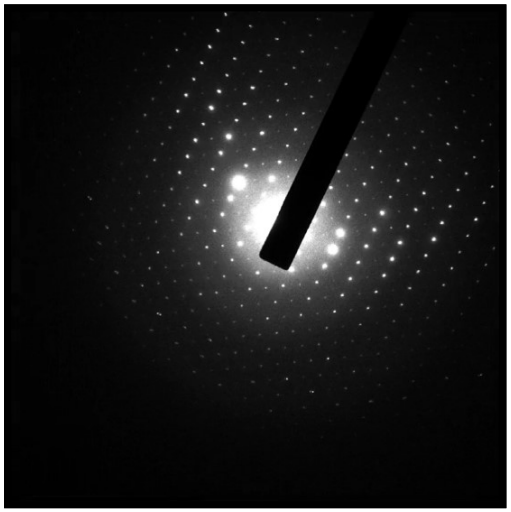
```python
from instamatic import TEMController
from instamatic.formats import write_tiff

ctrl = TEMController.initialize()

angles = range(-60, 60)

for i, angle in enumerate(angles):
    ctrl.stageposition.a = angle
    img, h = ctrl.getImage(exposure=0.5)
    write_tiff (f“diff_{i:4d}.tiff", img, header=h)
```

3D electron diffraction (continuous rotation)

```python
from instamatic import TEMController
from instamatic.formats import write_tiff

ctrl = TEMController.initialize()

start, end = -60, 60

ctrl.stageposition.set(a=start)
ctrl.stageposition.set(a=end, wait=False)

while ctrl.stageposition.a < end:
    img, h = ctrl.getImage(exposure=0.5)
    write_tiff (f"{i:4d}.tiff", img, header=h)
    print(f“Current angle: {ctrl.stageposition.a:.1f}”)
```

Serial electron diffraction

```python
from instamatic import TEMController
from instamatic.formats import write_tiff

ctrl = TEMController.initialize()

coords = get_list_of_coordinates()

for i, (x, y) in enumerate(coords):
    ctrl.stageposition.set_xy_with_backlash_correction(x=x, y=y)
    img, h = ctrl.getImage(exposure=0.5)
    write_tiff (f"{i:4d}.tiff", img, header=h)

    ctrl.difffocus.defocus(offset=1500)

    img2, h2 = ctrl.getImage()
    write_tiff(f"image_{i:4d}.tiff", img2, header=h2)

    ctrl.difffocus.refocus()
```



Data reduction server (Windows)

- **DIALS**: Socket server > subprocess > cmd > dials_script.bat >>> Python2.7

```
cmd = ["dials_script.bat", "./path/to/data"]
p = sp.Popen(cmd, stdout=sp.PIPE)
for line in p.stdout:
    parse(line)
```

- **XDS**: Socket server > subprocess > WSL > XDS

```
path = "./path/to/data"
p = sp.Popen("bash -ic xds_par 2>&1 >/dev/null", cwd=path)
p.wait()
```

Summary

- Python offers many options to interface other programs/libraries
  - The standard library (ctypes, subprocess, sockets, …)
  - Libraries (comtypes, pyautogui, …)

- Define common interface to access hardware
- Simplify and unify interaction through high-level interfaces
- Endless flexibility to design new experiments

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