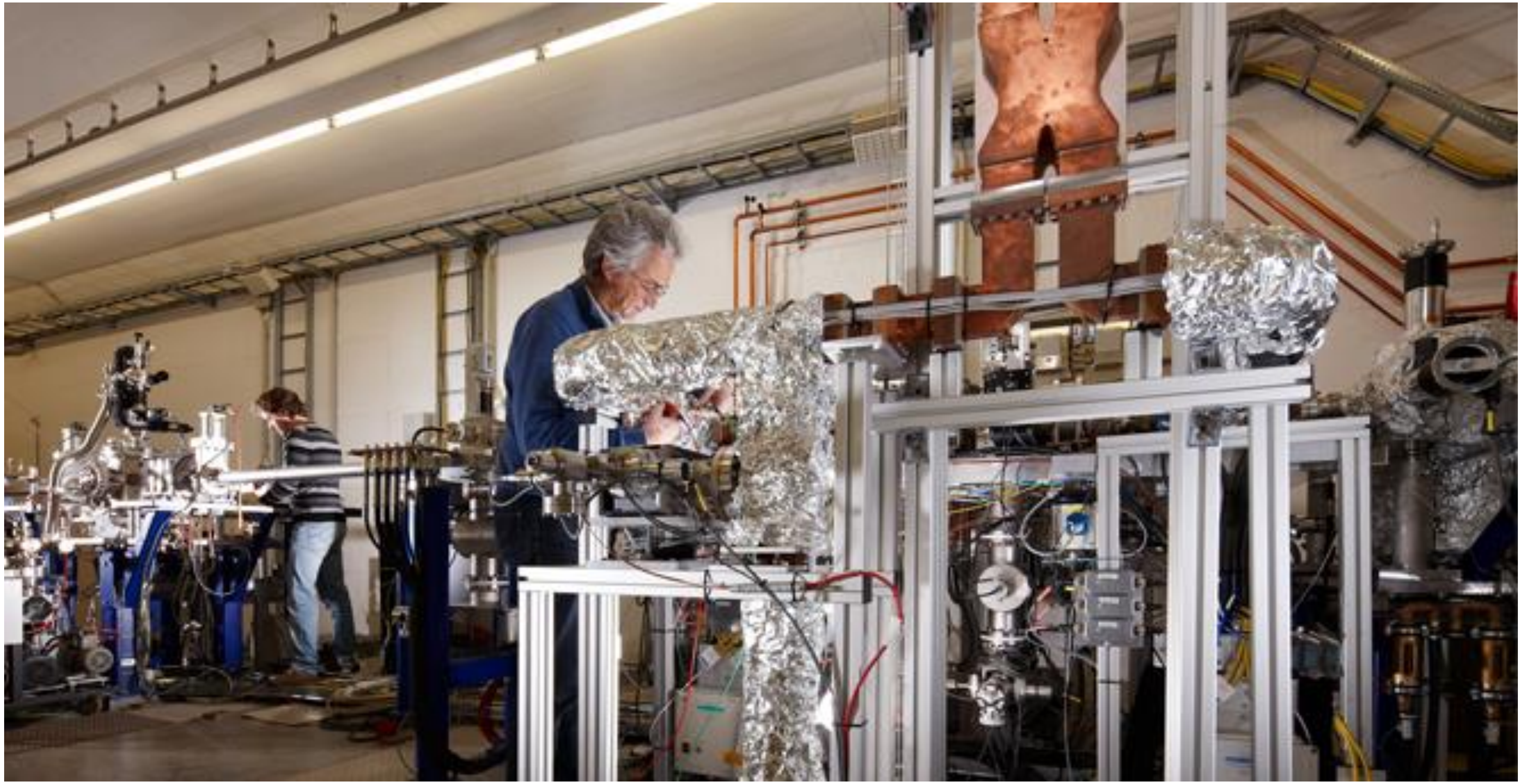
High energy electron difffravtion

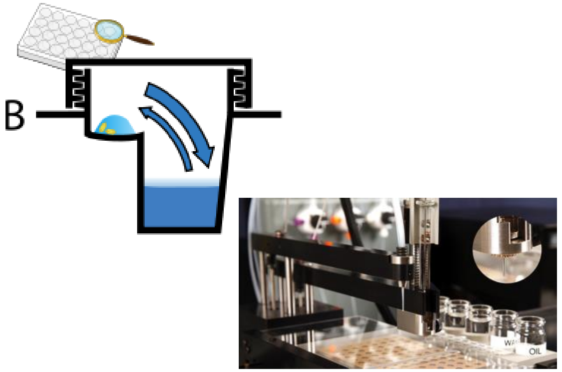
UED@REGAE



Workflow for High-throughput X-ray crystallography

High degree of automation opens new experimental capabilities

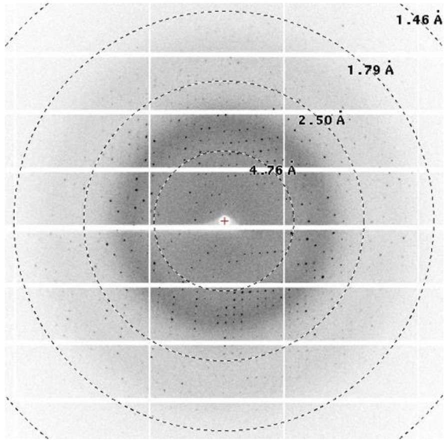
Protein production and purification



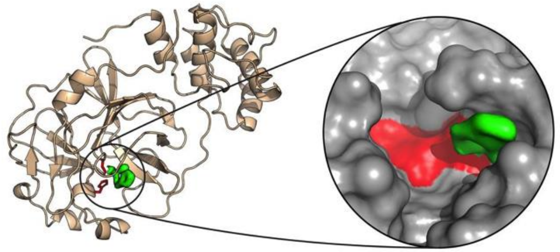
Protein crystallization

Crystal harvesting and flash-freezing

Synchrotron X-ray data collection



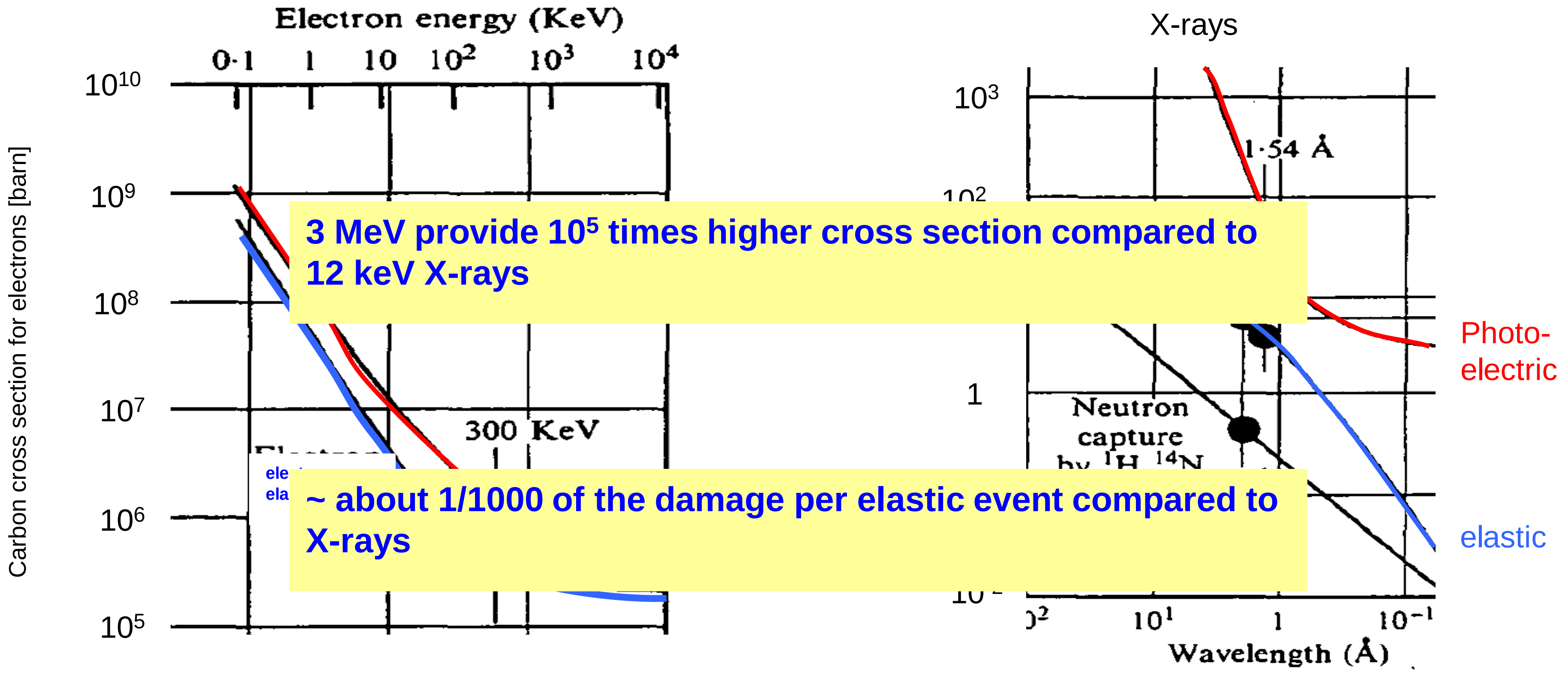
Data processing



Structure refinement and ligand identification

Comparison of X-rays with electrons

Stronger interaction with significantly reduced radiation damge



3 MeV provide 10^5 times higher cross section compared to 12 keV X-rays

~ about 1/1000 of the damage per elastic event compared to X-rays

Cross section for 3 MeV electrons:
~ 2 x 10^5 barn (10^{-24} cm^2)

Cross section for 12 keV X-rays
~ 3 barn (10^{-24} cm^2)

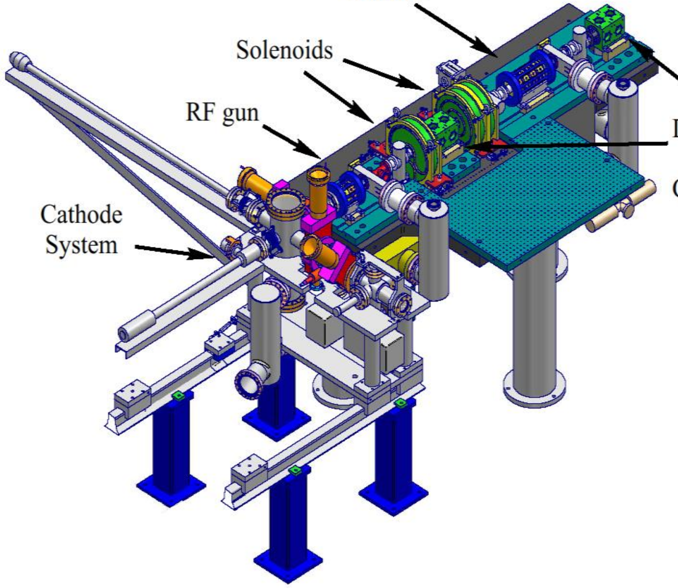
Henderson, R. *Q. Rev. Biophys.* **28**, 171–193 (1995).

Science case for REGAE

High-resolution time-resolved structural investigations of ultrathin low-Z samples.

REGAE´s electron beam parameters:

- Energy: 2.5 - 5 MeV
- Bunch charge: 100 fC
- Pulse duration: > 20 fs
- Beam size at sample position: 500 x 500 um^2
- Coherence lengths: ~ 1 nm (rms)
- Repetition rate: 50 Hz (in future: 100 Hz)



General advantage of 3 MeV electrons over X-rays

- ~10^5 x larger elastic cross section
- Radiation damage reduced by ~ 1000 times
- Better visibility of hydrogen atoms
- Photon equivalent of 100 fC electron pulse are about 10^{11} photons at 12 keV

-> ideally suited for structural investigations of ultrathin low-Z materials

Challenges with electron diffraction

- Bunch charge effects preventing microbeam experiments
- Sample thickness limited to < 1 µm
- Experiments require ultra high vacuum (UHV)
- Sample preparation and delivery is most challenging, in particular for liquids and hydrated biological samples

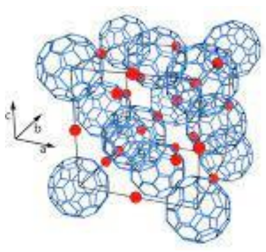
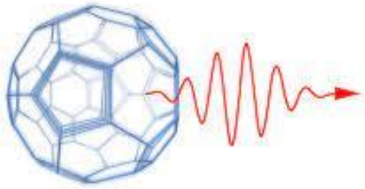
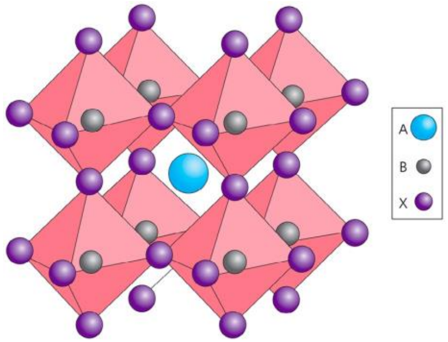
Science case for REGAE

Material Science applications

Quantum materials / Solar cells

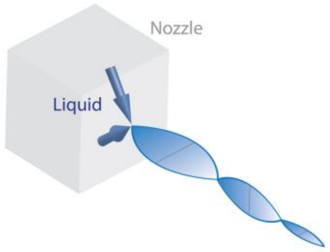
- fs time resolved 3D structure determinations of quantum materials
- Time resolved pair-distribution-function PDF experiments
- *Experimental methods: single crystal and powder diffraction with laser excitation*

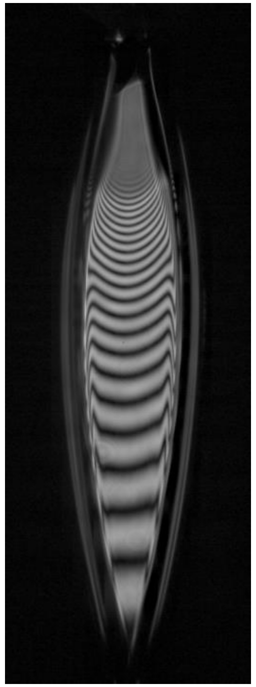
- Long term: *in-situ* studies of low-Z battery materials with MeV-electron reaction microscope

Molecular water Science

- Structural dynamics of water at different temperatures, e.g. super cooling, and with different laser excitation schemes (THz, IR, VIS, UV).
- Better visibility of hydrogen atoms compared to X-rays
- *Sample delivery as thin sheet liquid jets*





work by G. Esperenza / H. Chapman DESY

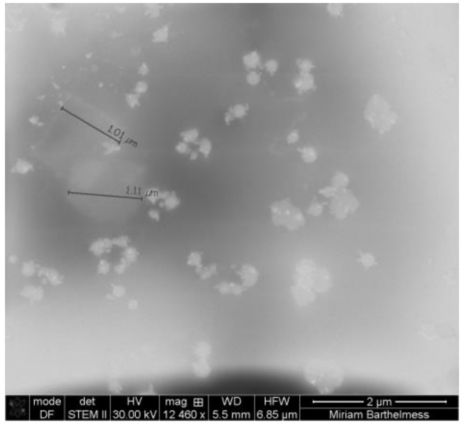
Science case for REGAE

Diffraction experiments from biological samples with microsecond time resolution

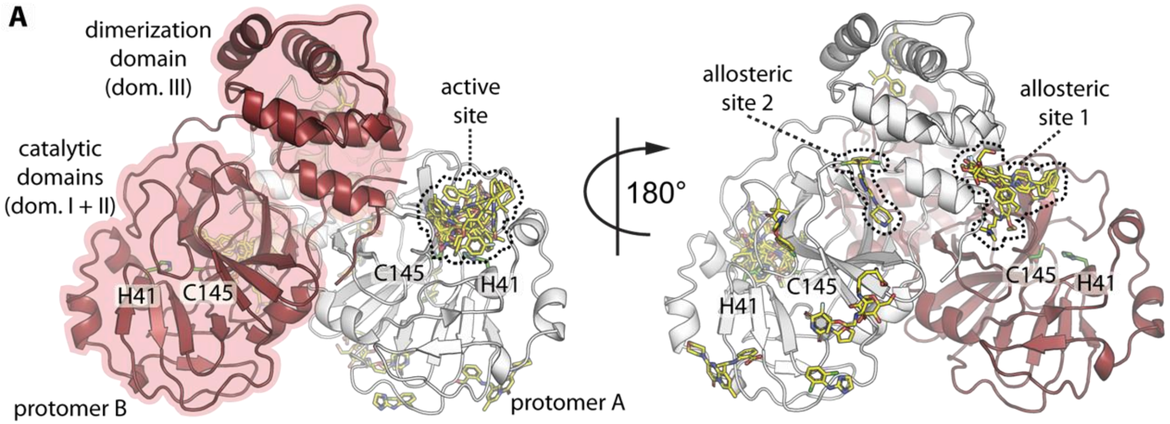
Life science applications:

- Time resolved diffraction experiments with laser excitation including THz with reduced radiation damage compared to X-rays
- High-throughput compound screening experiments for drug discovery with 1/1000 of sample required compared to X-rays
- Structure determinations from nano- and microcrystals benefitting from reduced radiation damage effects (damage free structures from metal-containing enzymes

-> these experiments require a micrometer size electron beam with a large coherence lengths.



SARS-CoV2 main-protease microcrystals on a SiN-membrane - Ready for micro-electron diffraction experiments at REGAE

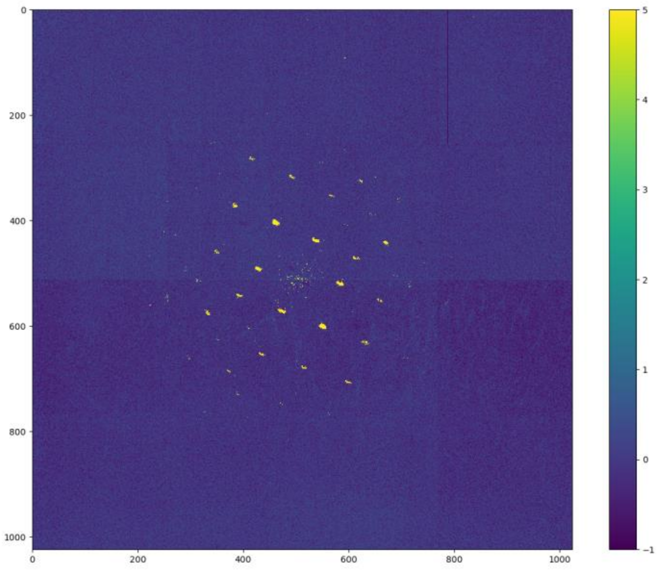


Recent improvements

significantly improved signal-to-noise ratio

Installation of a Jungfrau 1 M detector

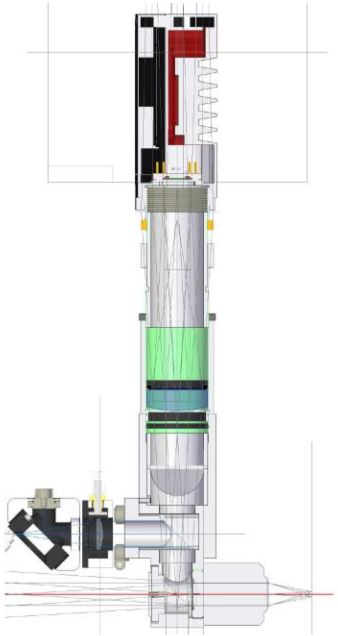
- So far: scintillator-based converting detectors in combination with a CCD camera
- Direct electron detection 1 Mega pixel detector
- UHV compatible, directly attached to the vacuum system. Collaboration with PSI, Switzerland.

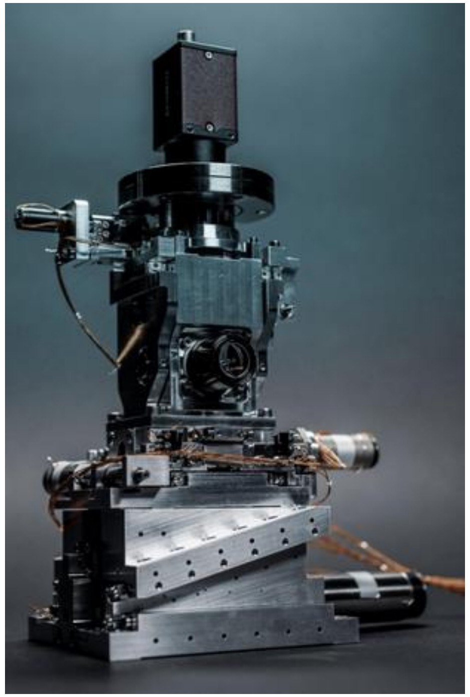


Single shot diffraction pattern from a 50 nm thick single crystalline gold crystal recorded with a Jungfrau 1M detector installed at REGAE.

Inline sample (on-axis) viewing microscope

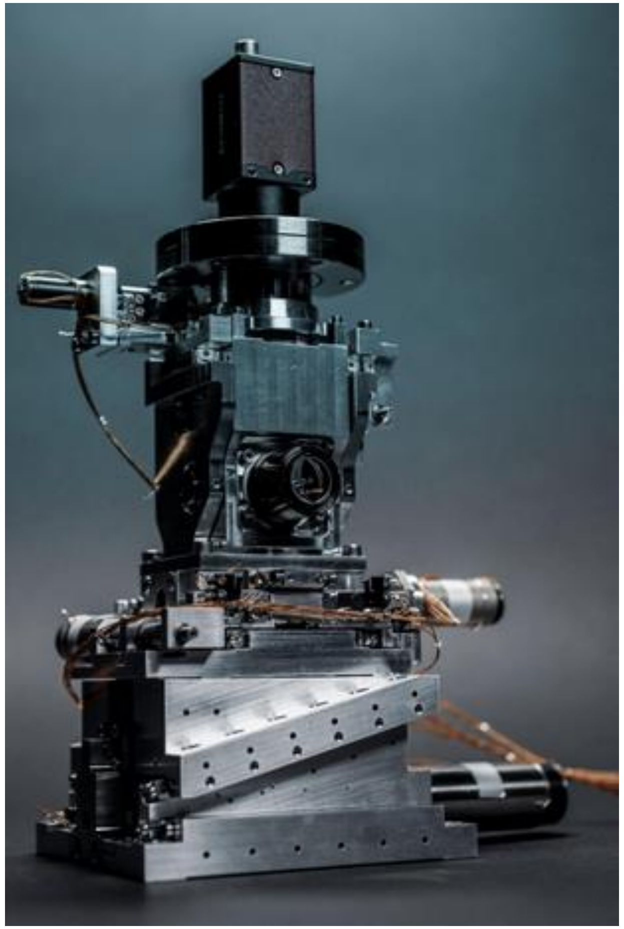
- High optical resolution for sample visualization
- Central drill-hole for electron beam along the optical axis
- Option for through-the-lense laser excitation

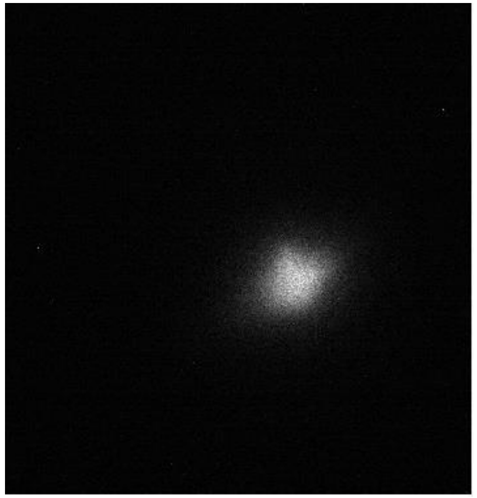




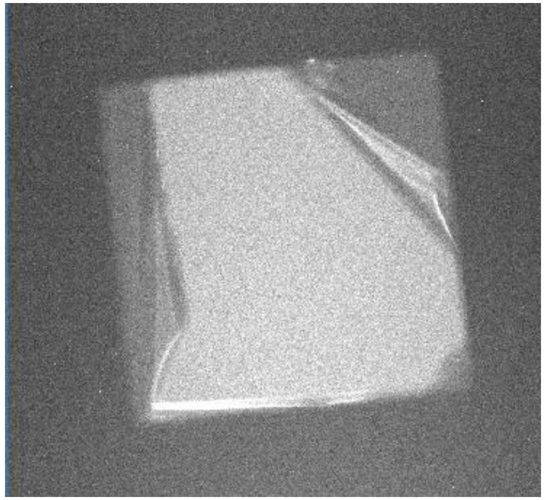
Beam and sample visualisation with UHV on-axis microscope

Simultaneous electron diffraction and VIS-microscopy

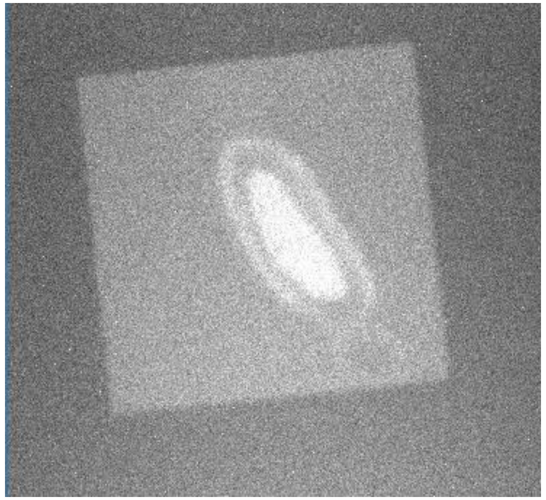




REGAE electron beam on YAG-screen

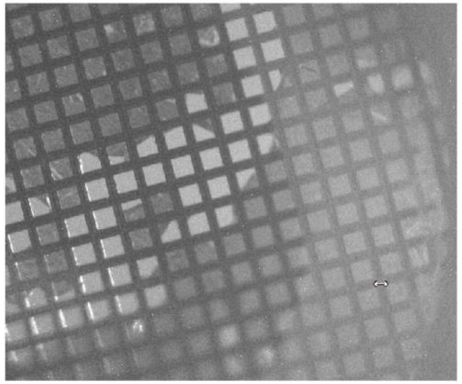


Au-coated SiN membrane damaged by pump laser beam



Laser induced melting zone on a Au-coated SiN membrane

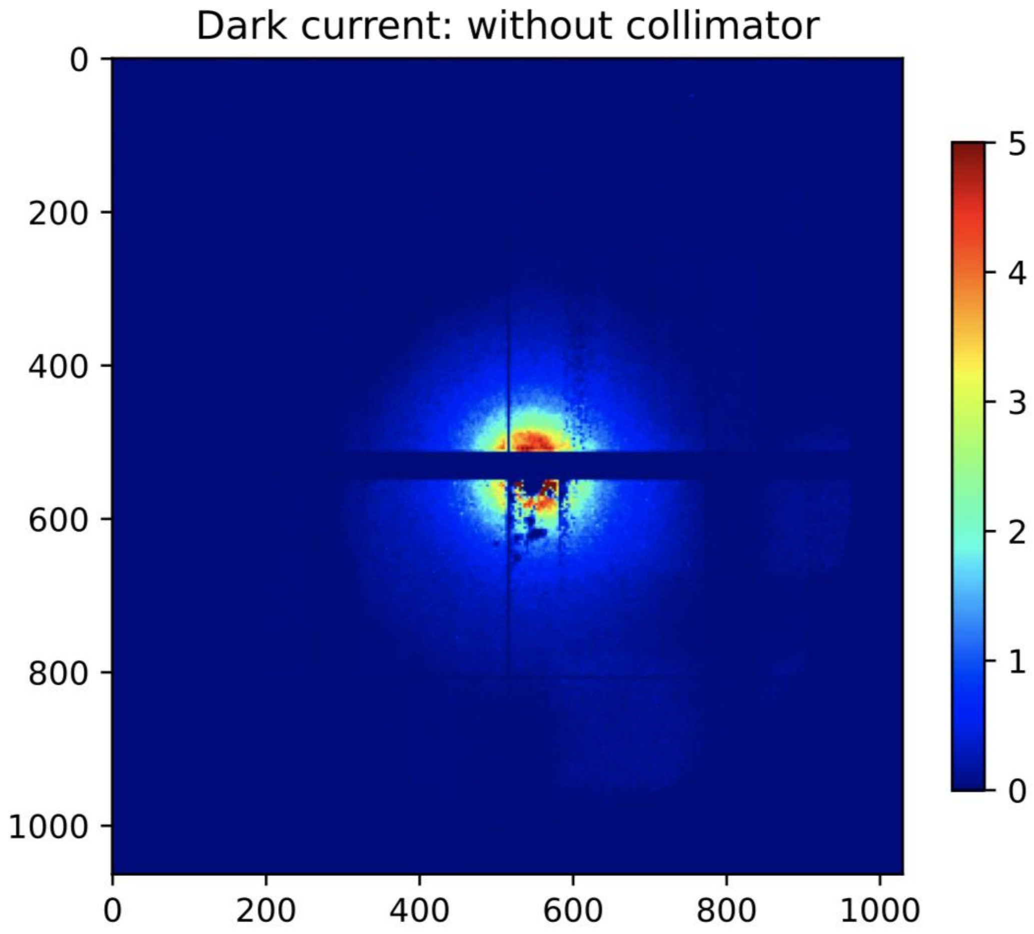
field of view: ~ 1,1 x 1,1 mm

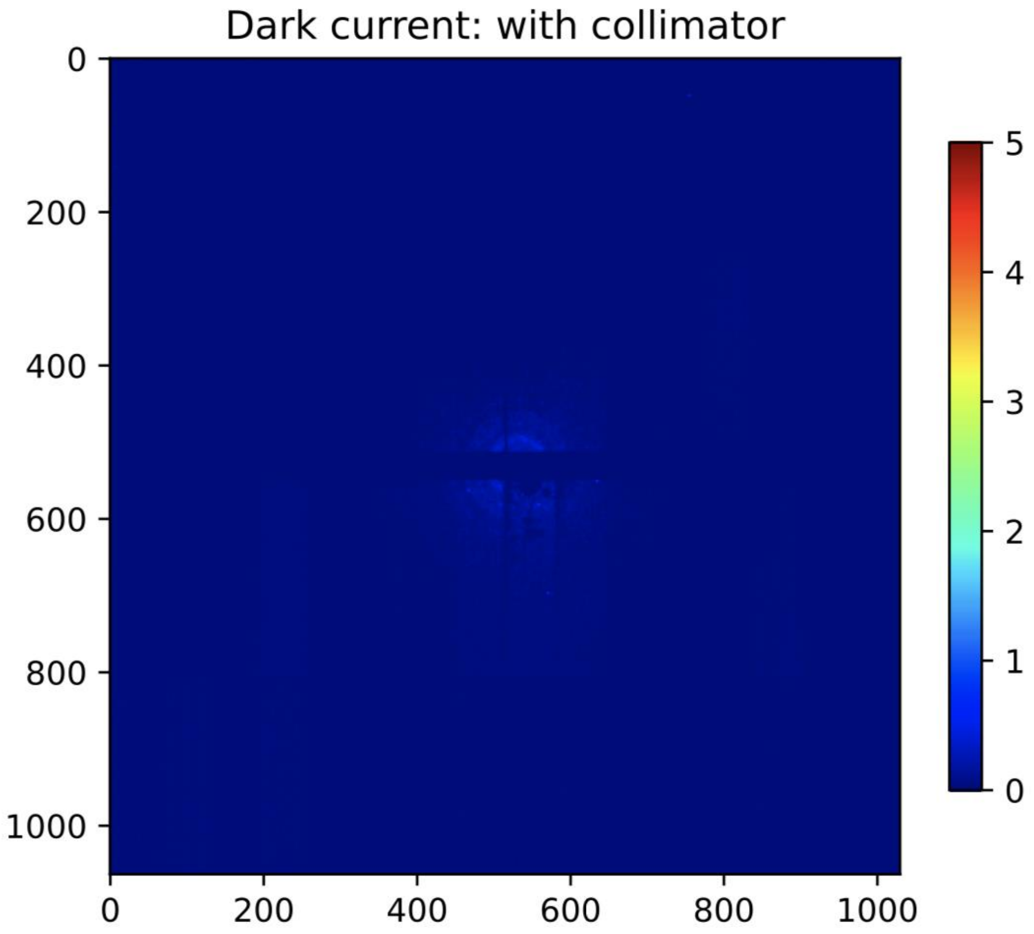


TEM-grid microscope image

Reduced dark current with collimator setup

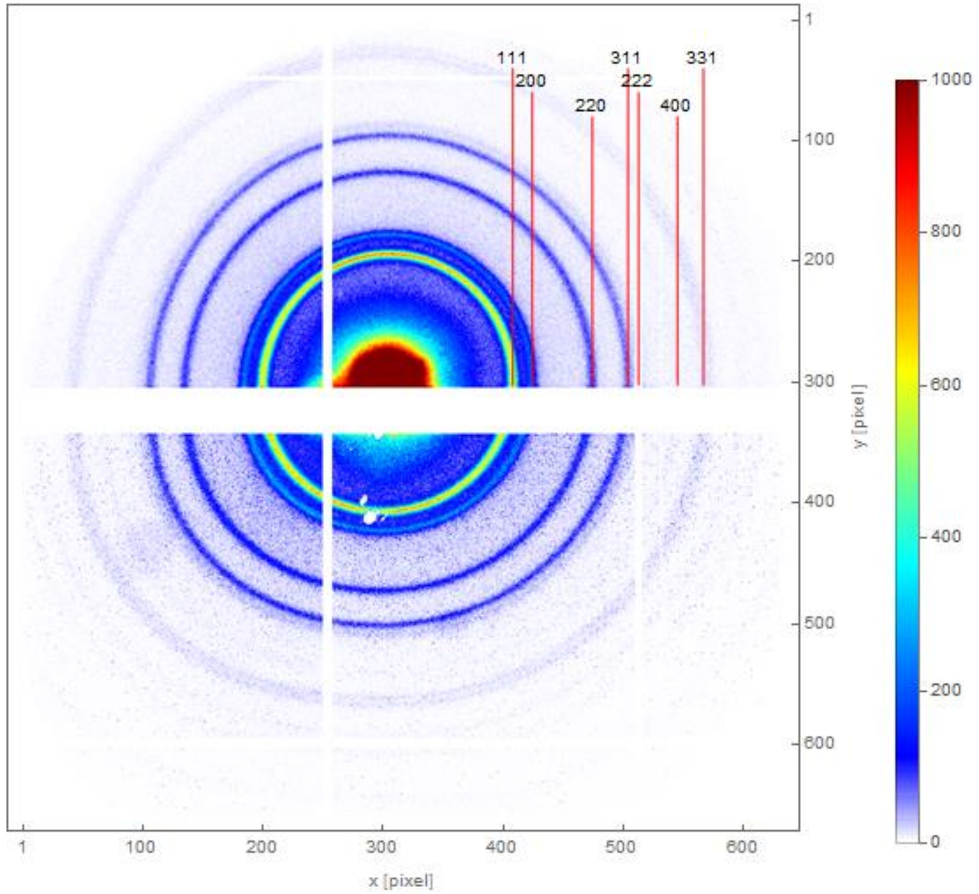
Significant improvement in particuar at small diffraction angles

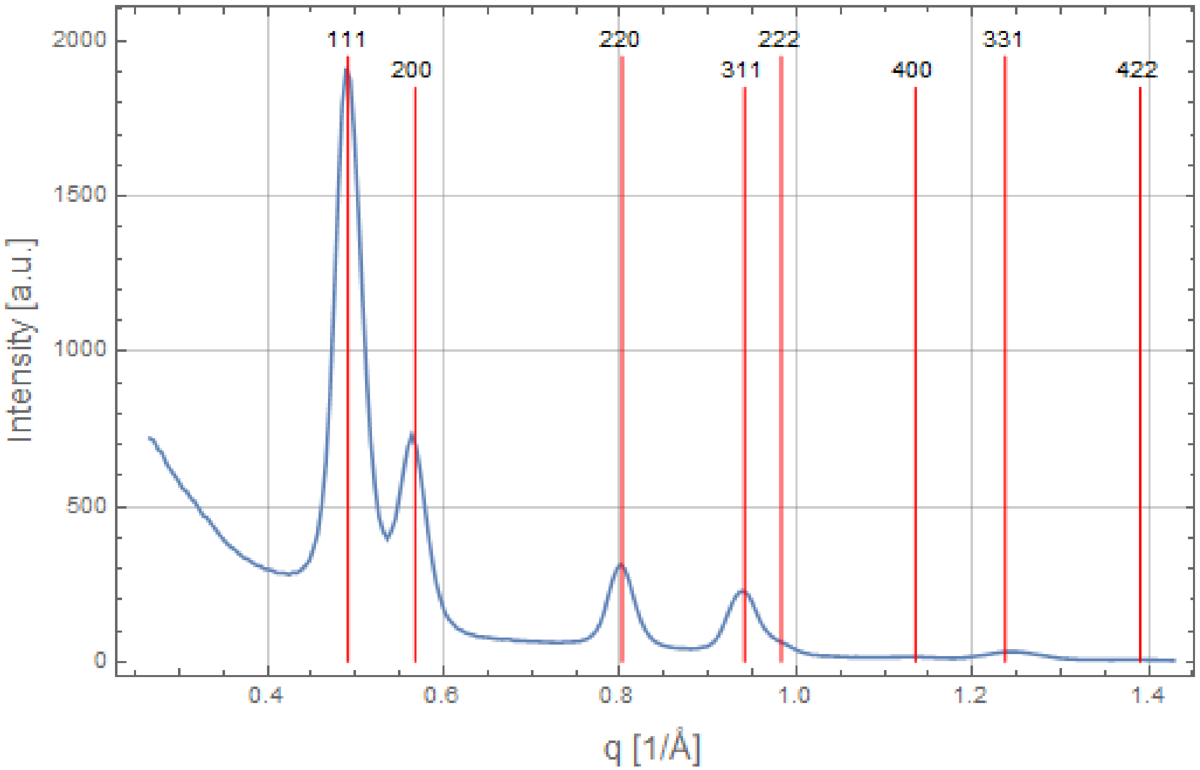




Powder diffraction experiments

Significantly improved signal to-noise-ratio with Jungfrau 1M detector

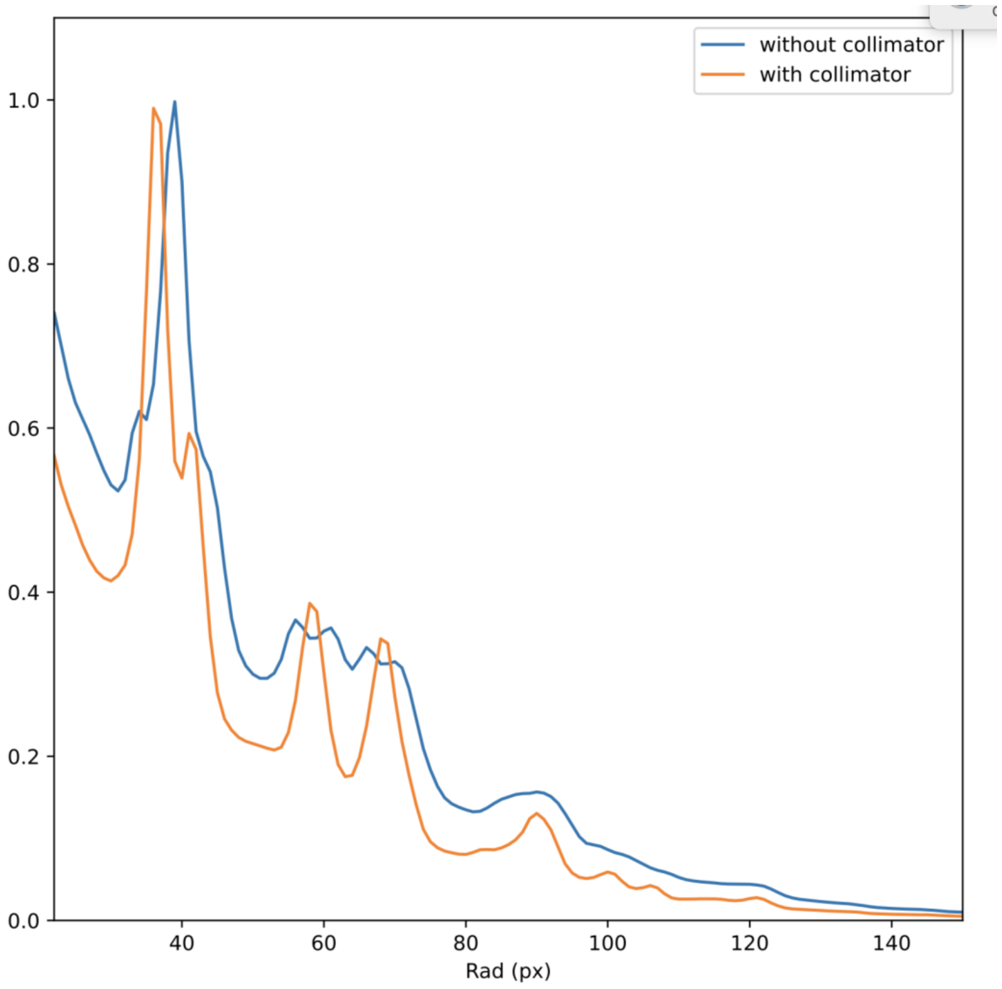




250 averaged diffraction patterns recorded from a 100 nm Ni foil with 50 Hz repetition rate on the Jungfrau detector

improved q-resolution with collimator

Improved signal-to-noise-ratio caused by reduction of beam jitter?

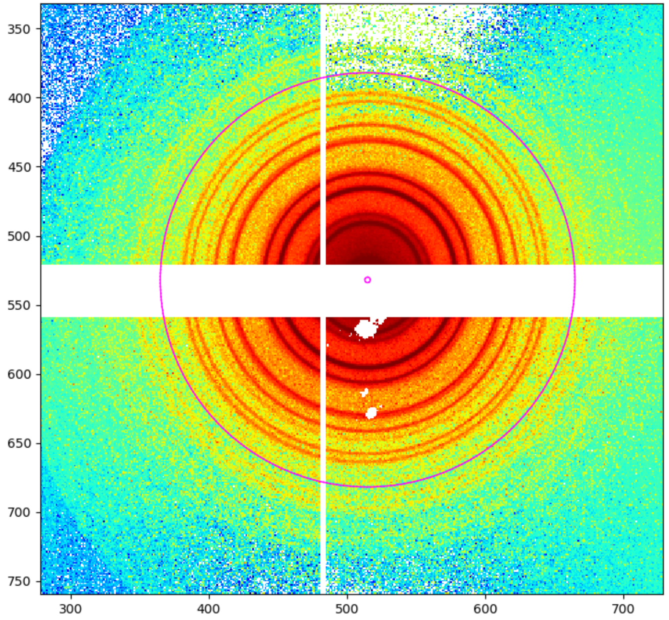


- 1000 averaged diffraction patterns from a 100 nm Ni foil
- recorded with Jungfrau 1M detector at 50 Hz frame rate
- Background scattering mainly originating from 70 SiN membrane

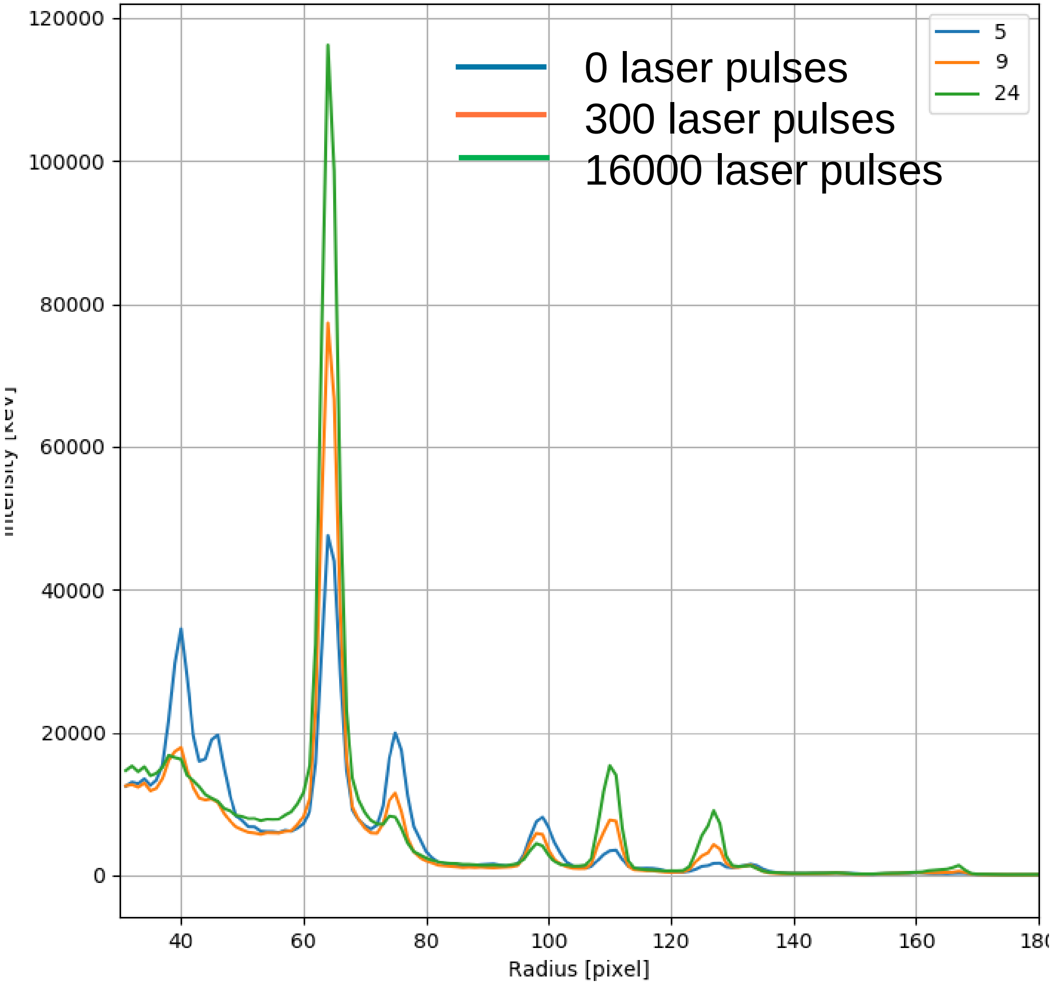
Laser-induced recrystalization of gold

Preperation for time-resolved diffraction experiments

- **Sample: 30 nm Au foil on SiN membrane**
- Electron beam parameters:
  - energy: 3 MeV
  - pulse length: 600 fs
- Laser parameters:
  - length: 600 fs
  - wavewength: 400 nm
  - laser power per shot: 12 uJ



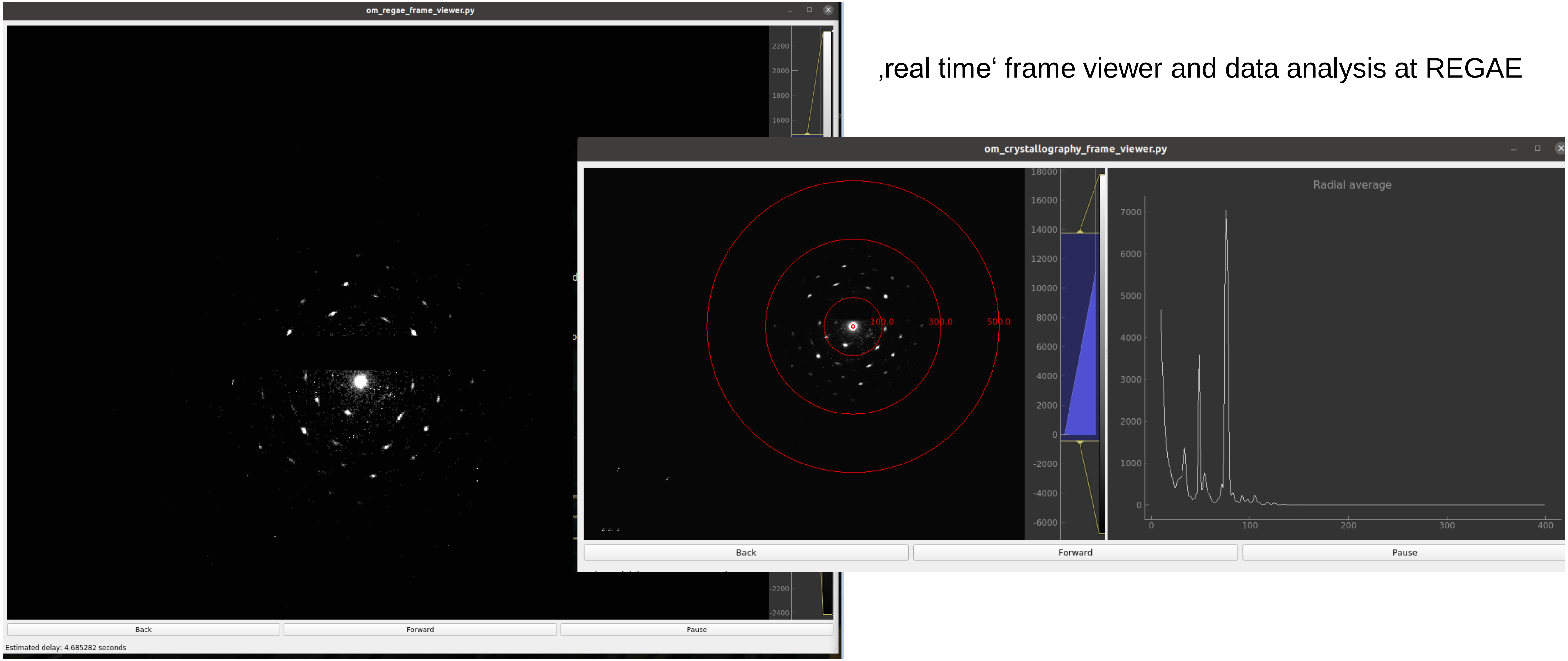
Sum of 100 sinlge shot electron diffraction images



Radially averaged diffraction patterns (based on 100 individual diffraction images)

Single crystal diffraction

50 nm single crystalline gold foil – single shot diffraction pattern @ 50 Hz



‚real time‘ frame viewer and data analysis at REGAE

New sample preparation laboratory

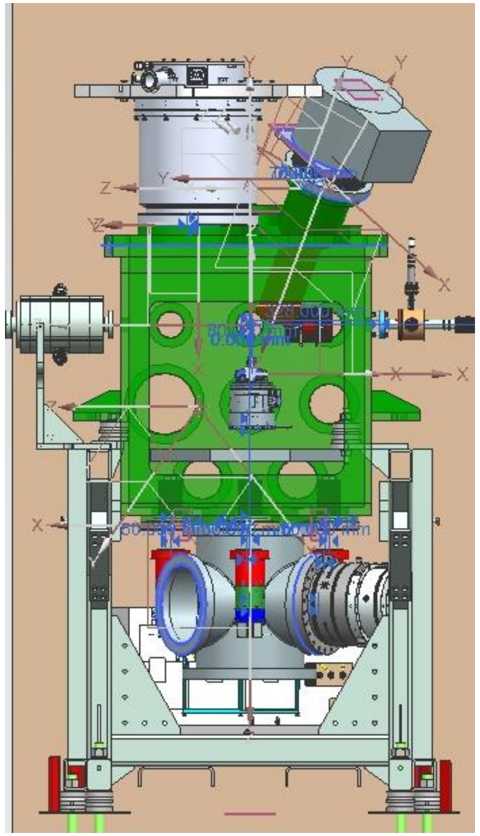
Advanced sample preparation in direct proximity to the experiment

Planned hardware upgrades of REGAE

New diffraction setup with high-precision goniometer, cryogenic sample cooling, and robotic arm

Hardware upgrades:

- New sample chamber with extended experimental capabilities (design finished)
- High-precision goniometer for diffraction experiments (first hardware components received)
- Option for cryogenic sample cooling (bio-sample and quantum materials)
- Load-lock system with robotic arm for fast exchange of cryogenically cooled samples
- Integration of a liquid jet setup
- Long term: Installation of a microscopy setup for time-resolved MeV electron tomography.



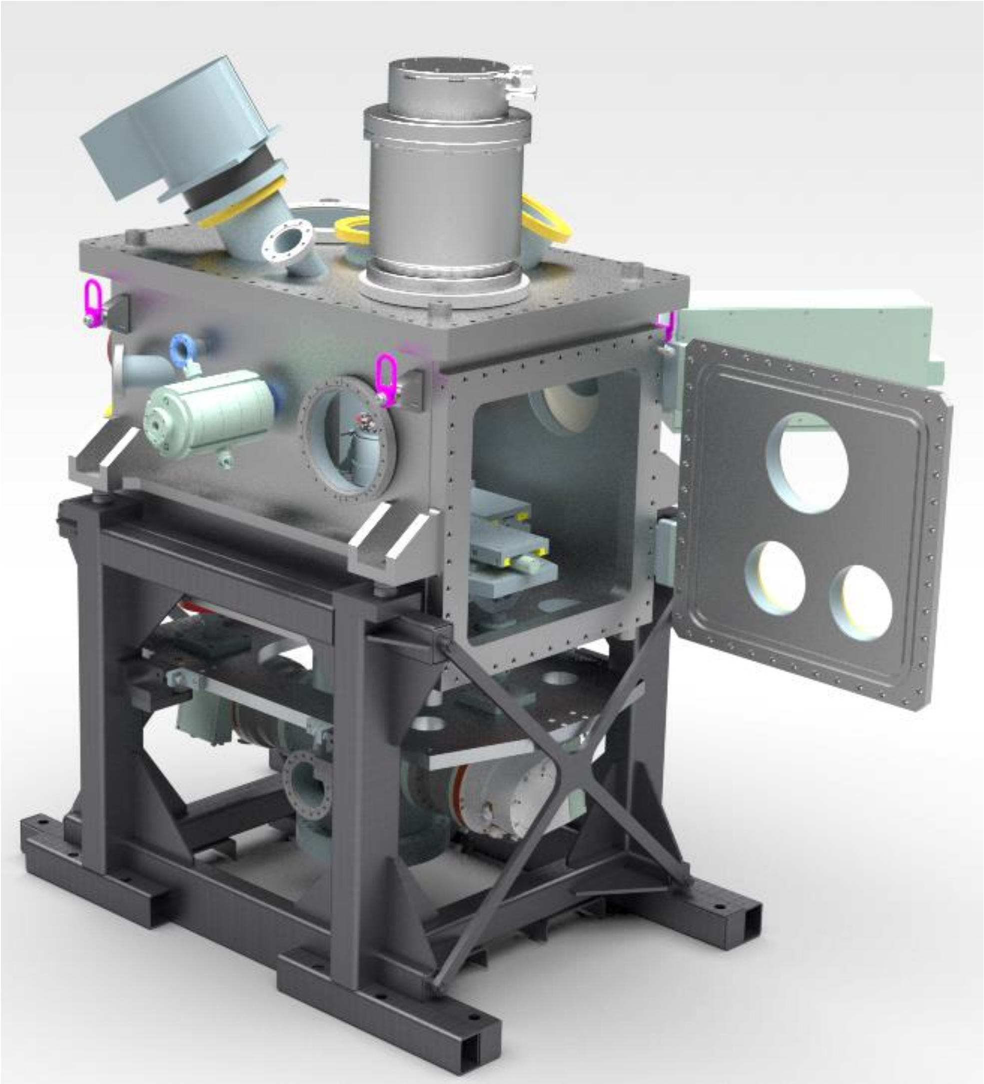
New sample chamber for REGAE
for different experimental setups

Universal robots UR5E robotic arm
for fully automatic sample exchange

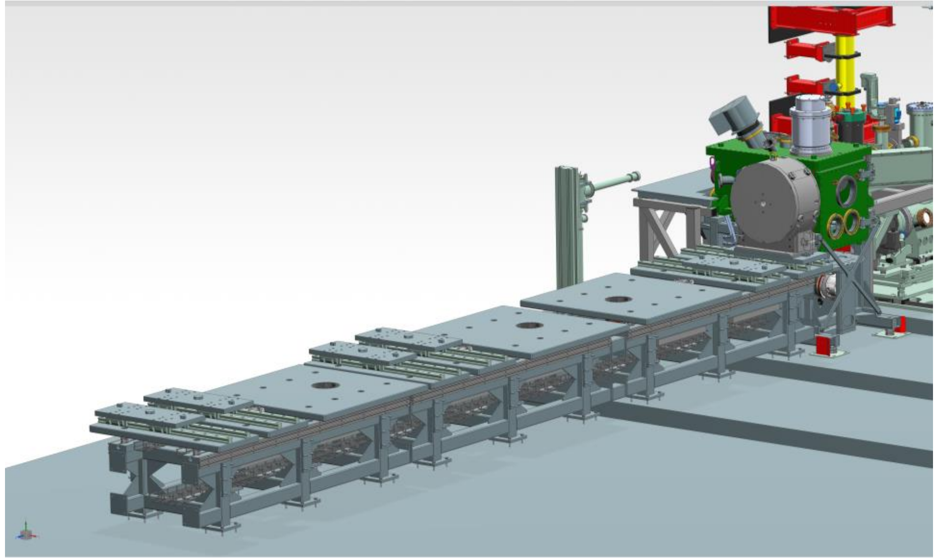
e^--Roadrunner UHV compatible goniometer

New sample chamber for REGAE

Space for three experimental setups



- Two standard setups:
  - For solid sampes (crystallography)
  - For liquid samples (sheet jet)
- Space for one user specific setup
- reduced vibration due to decoupling of vacuum system and inner parts
- Vacuum system capable for handling of liquid jets
- Different laser pump options
- Cryostat for sample colling down to 10K



New diffraction setup for solid state samples

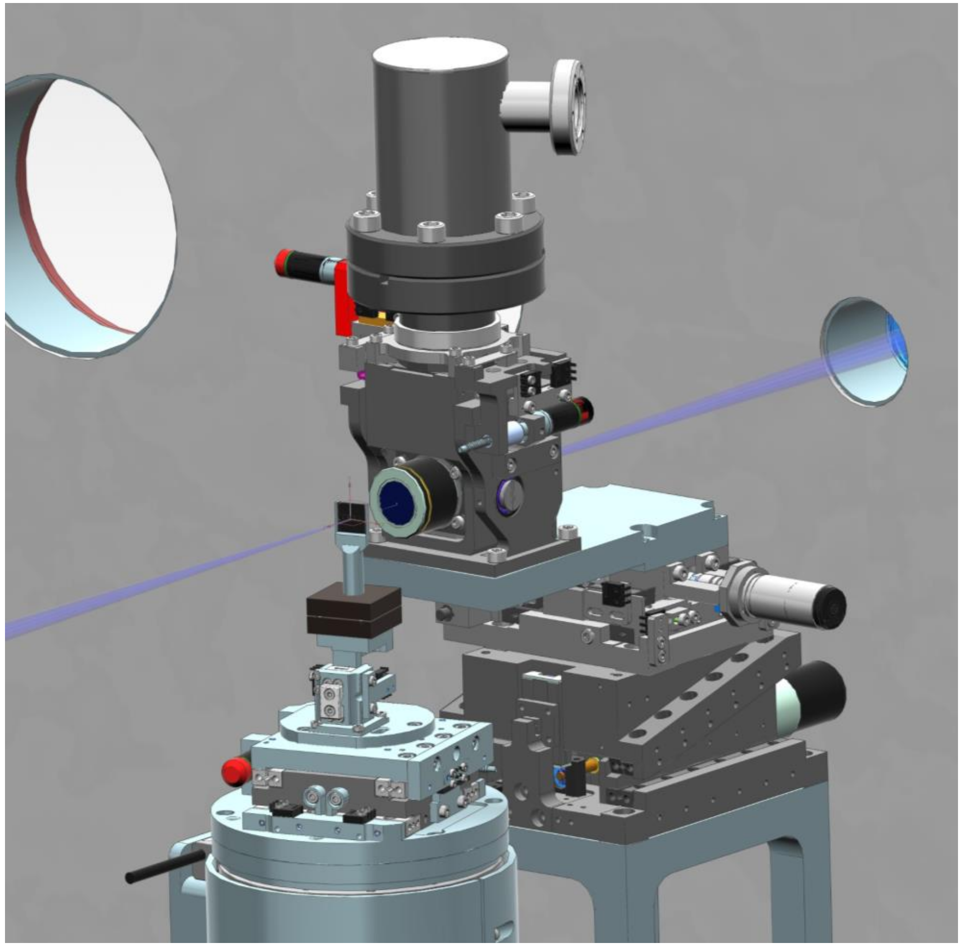
High-precision eRoadrunner goniometer

- **Fully UHV compatible**

- **High-precision rotation axis:**
  - Full 360 degree rotation
  - Servo motor operated
  - SOC: 1 µm
  - Angular resolution: 0.0001 degree

- **Centering stage:**
  - 12 mm travel range in x,y,z
  - Positiiong accuracy: 1 µm
  - Through-the-lense laser excitation for pump-probe experiments

  Future upgrade plans:
  - Cryogenic sample cooling to 10 K
  - Robotic sample loading through load-lock system

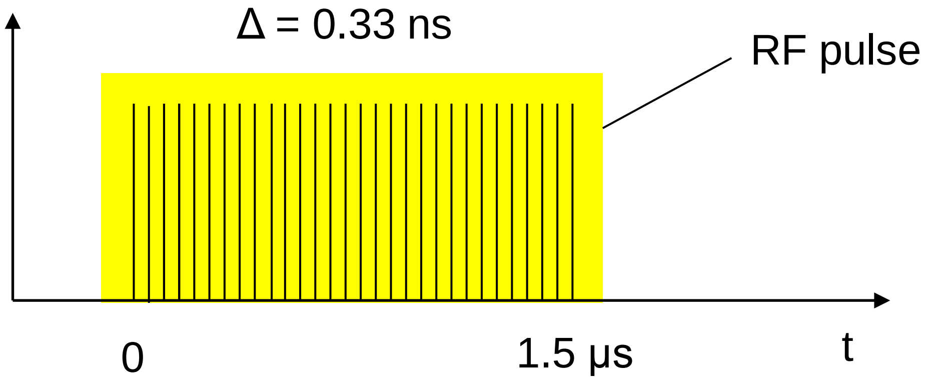


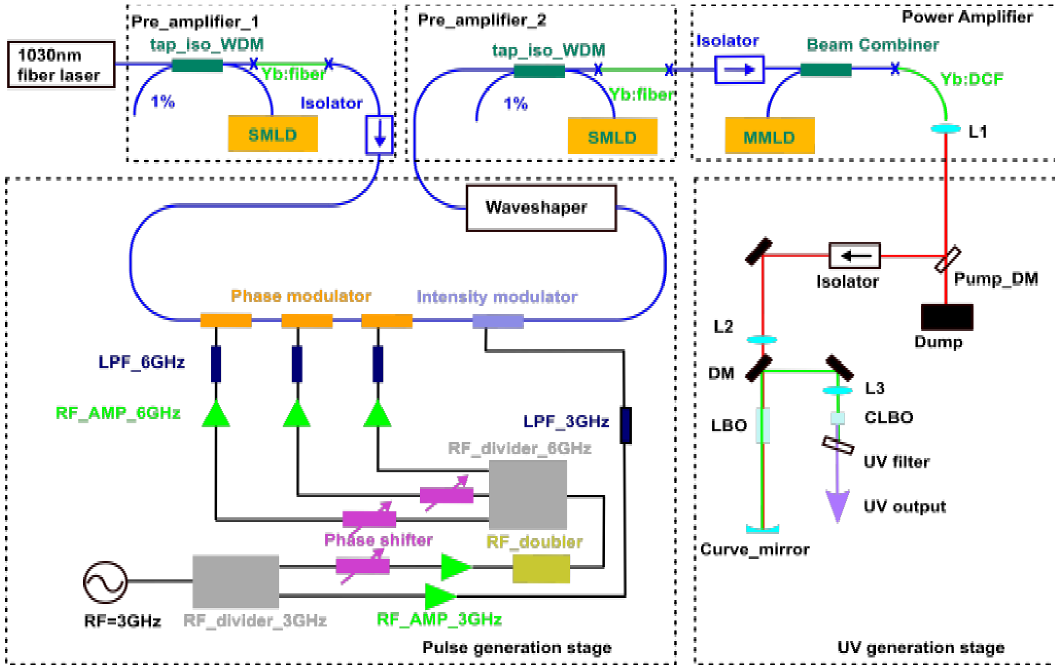
3 GHz laser for coherent micro-beam experiments

1.5 microsecond bunch trains with 100 fC

Coherent micro-beam mode with 3 GHz laser

- For electron diffraction with protein crystals and electron microscopy requiring a coherent (> 10 nm) micrometer size electron beam (~1 µm)
- 1.5 µsec bunch trains with 100 fC consisting of ~4500 low charge micro bunches avid bunch charge effects
- in collaboration with Ingmar Hartl (FS-LA), to be ready for installation in October 2021 Δ

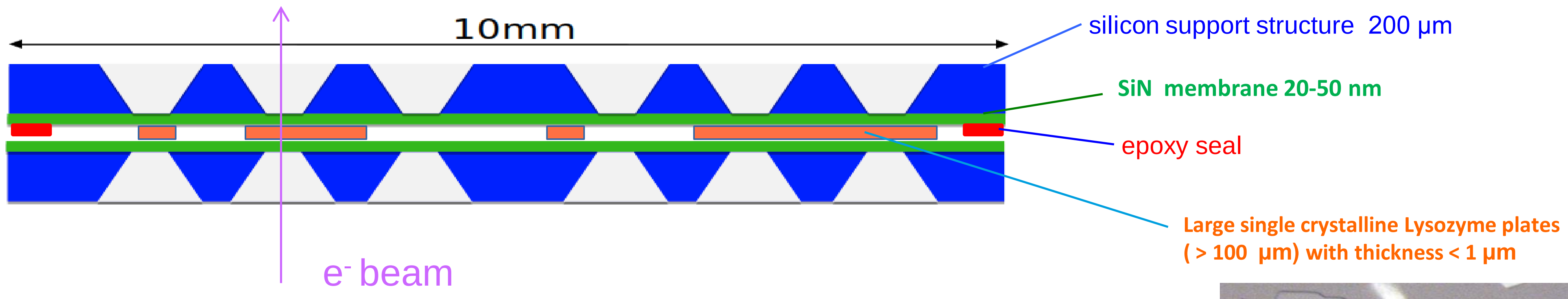




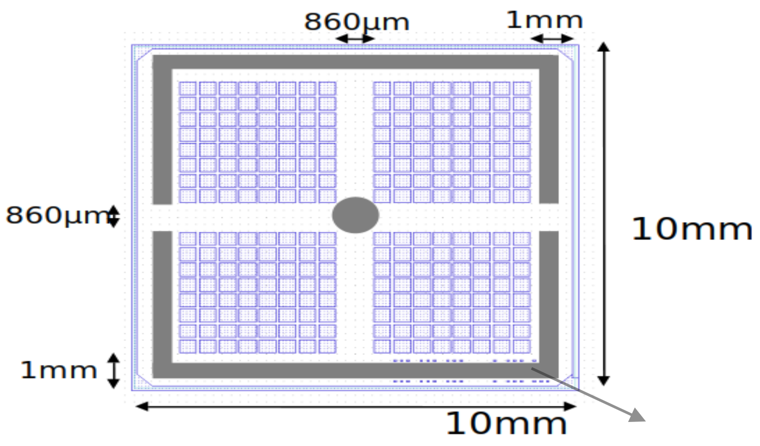
Crystalline protein samples for MeV-UED experiment

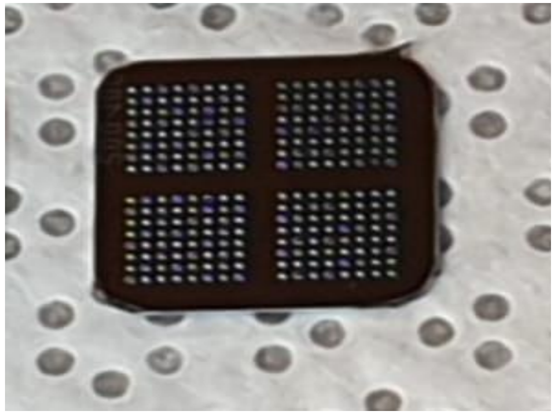
Sealed sandwich structure: keeps crystals hydrated

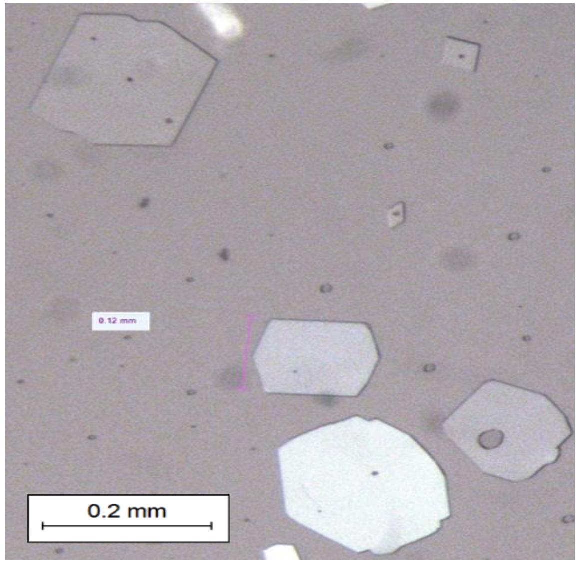
Side view:



Top view:







REGAE Time plan

First friendly user experiments in 2023

• First time resolved diffraction experiments with laser excitation	April 2022
• Implementation of 3 GHz laser	May 2021
• First diffraction experiments with biological samples	June 2022
• Installation of new experimental chamber and related hardware upgrades	August 2022
• First friendly user experiments	spring 2023
• Implementation of cryogenic sample and robotic sample exchange	spring 2023
• Implementation of microscopy setup	planned for 2024

People involved & Tanks to …

Joined effort between DESY's M and FS divisions

Vincent Hennicke	mechanical engineer
Max Hachmann	scientist (machine operation)
Ana Rodriguez	scientist (real time data analysis)
Sadegh Bakhtiarzadeh	scientist (laser operation)
Klaus Flöttmann	scientist (machine operation)
Hossein Delsim-Hashemi	scientist (machine operation)
Tim Pakendorf	mechanical engineer
Lars Gumprecht	mechanical engineer
Pontus Fischer	software engineer
Jan Meyer.	software engineer
Alke Meents	scientist (diffraction experiments)
Miriam Barthelmess	scientist (sample preparation)
Leticia de Melo Costa	scientist (sample preparation)
Patrick Reinke	scientist (sample preparation, diffraction)
Christoph Mahnke	FS-LA scientist (laser operation)
Ingmar Hartl	FS-LA
Henry Chapman	FS-CFEL
Wim Leemanns	M

Support from many other M- and FS groups