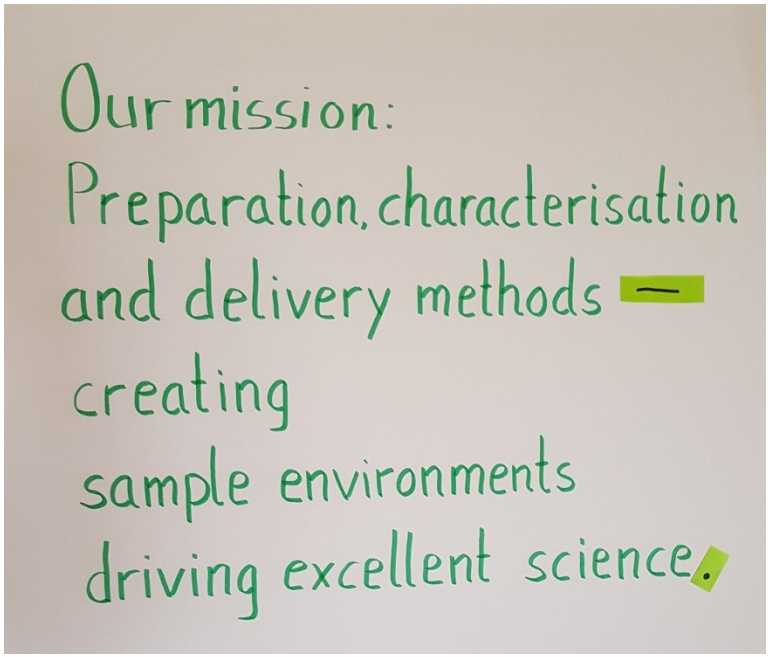
User Laboratories at the European XFEL

How we can help you getting your sample into the beam

Kristina Lorenzen
Sample Environment and Characterization

XBI User Consortium

Sample Environment and Characterization Group:



Scope

- developing new sample environments at all six beamlines
  - Jets
  - Gases
  - Magnetic fields
  - Solid support
- **Supporting Users with their sample preparation needs at the European XFEL**
  - Biological samples
  - Chemistry
  - Solid samples
- **Running the USER Laboratories**
- **Driving science forward**

European XFEL User Laboratories – Your Home away from Home

User Laboratories at the European XFEL

- 560 m² biology wet lab
  - Predominantly financed by the XBI user consortium
- 30 m² preparation lab at the SPB/SFX instrument
- 50 m² chemistry labs
- 150 m² physics and microscopy labs

Why do we need a user laboratory?

- Beamtime is precious and expensive, you want the best pre preparation and characterization for your sample
- Ability to adapt quickly and produce more sample/ alter the quality, find and solve problems related to your sample
- XFELs due to their brilliance are able to provide diffraction patterns of much smaller crystals compared to third generation synchrotron sources. The labs on site allow to test your xtals size and properties and jetting before your allocated beamtime.
- Special properties of the beam structure, allowing to monitor fast reactions require special properties during sample preparation (dark, anaerobic…)
- Molecular Movies – small crystalls allow for diffusion rates short enough to make kinetic measurements feasible
- Single Particle Imaging - Other high resolution methods like electron microscopy rely on forms of sample fixation and do not image living cells or organisms.

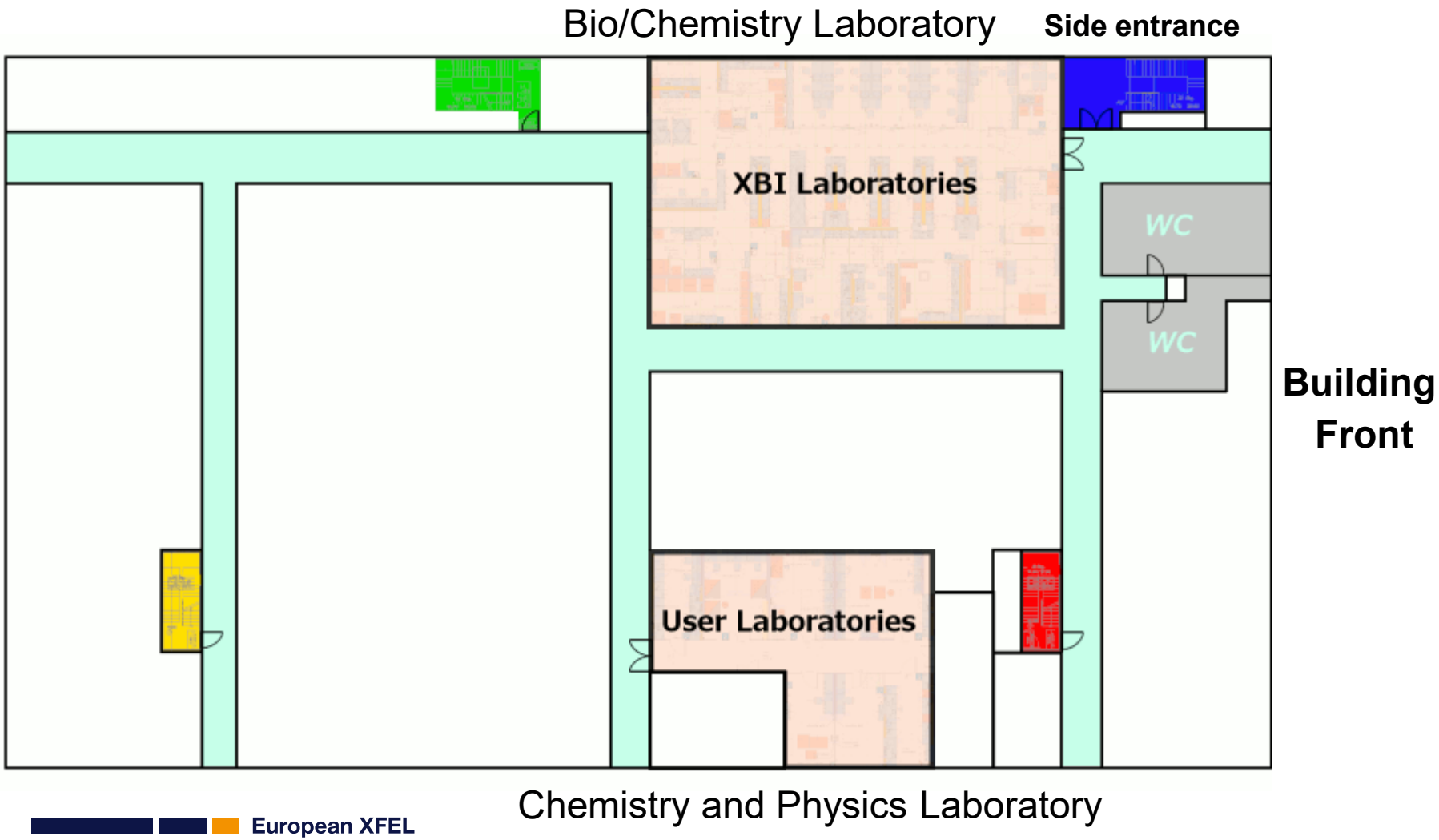
Concept for User Support

- Bio lab usage is requested in the application for beamtime
- Webpage and Alfresco site (users with exfel.eu account)
  - https://www.xfel.eu/users/experiment_support/user_labs
  - https://docs.xfel.eu/share/page/site/userlabs/dashboard
- Three weeks before the users come they need to fill out a "labwork description form"
- The User will have three contacts
  - Local contact
  - Sample contact
  - Lab contact
    - Does check in and check out with users
    - Helps to fill out "labwork description form"
- XBI Staff Support
  - Mon - Fri from 9am till 6pm
  - Users are allowed to work outside these hours
  - OnCallDuty (contacted by beamline scientist)

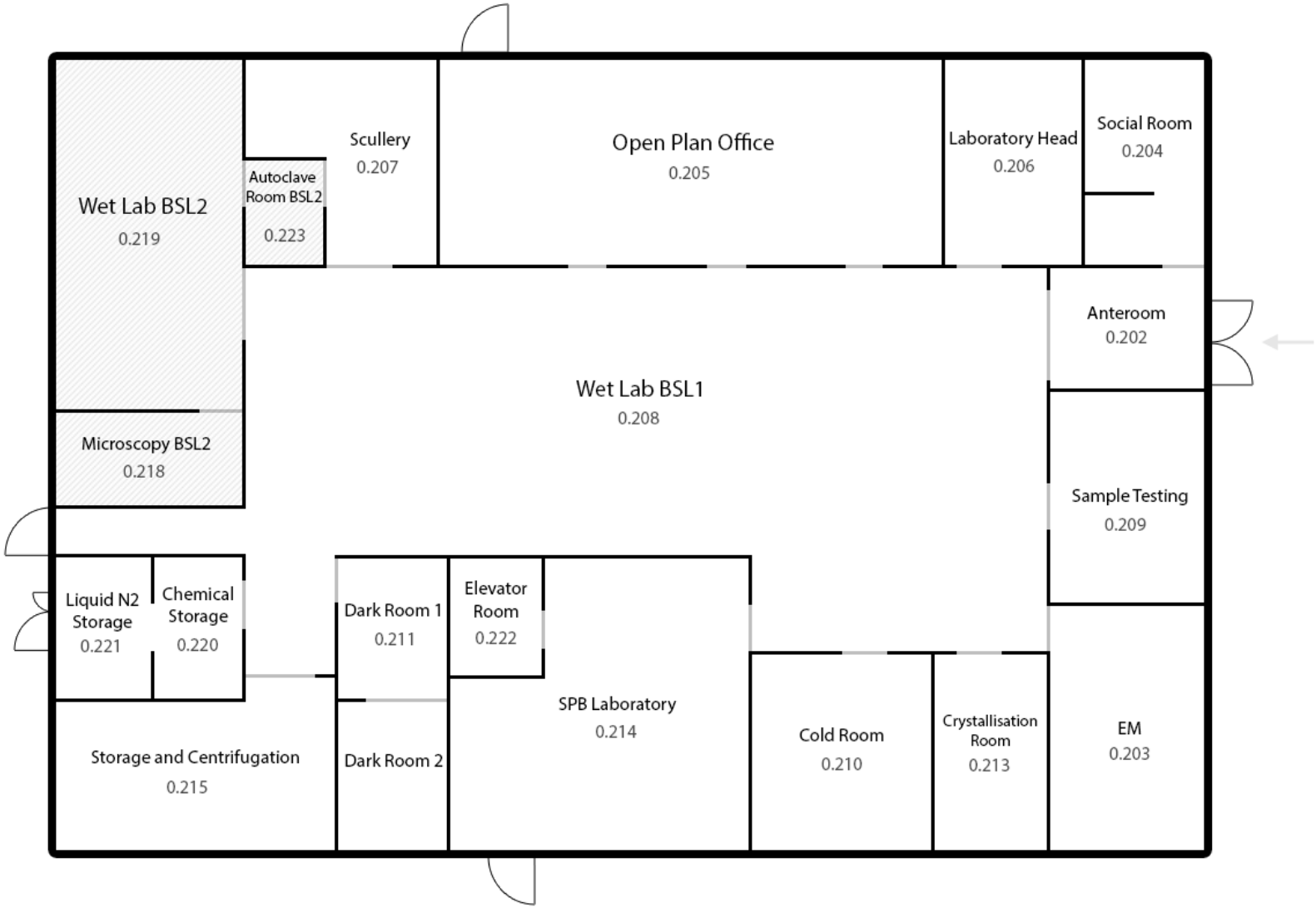
How to contact us

Sample.environment@xfel.eu

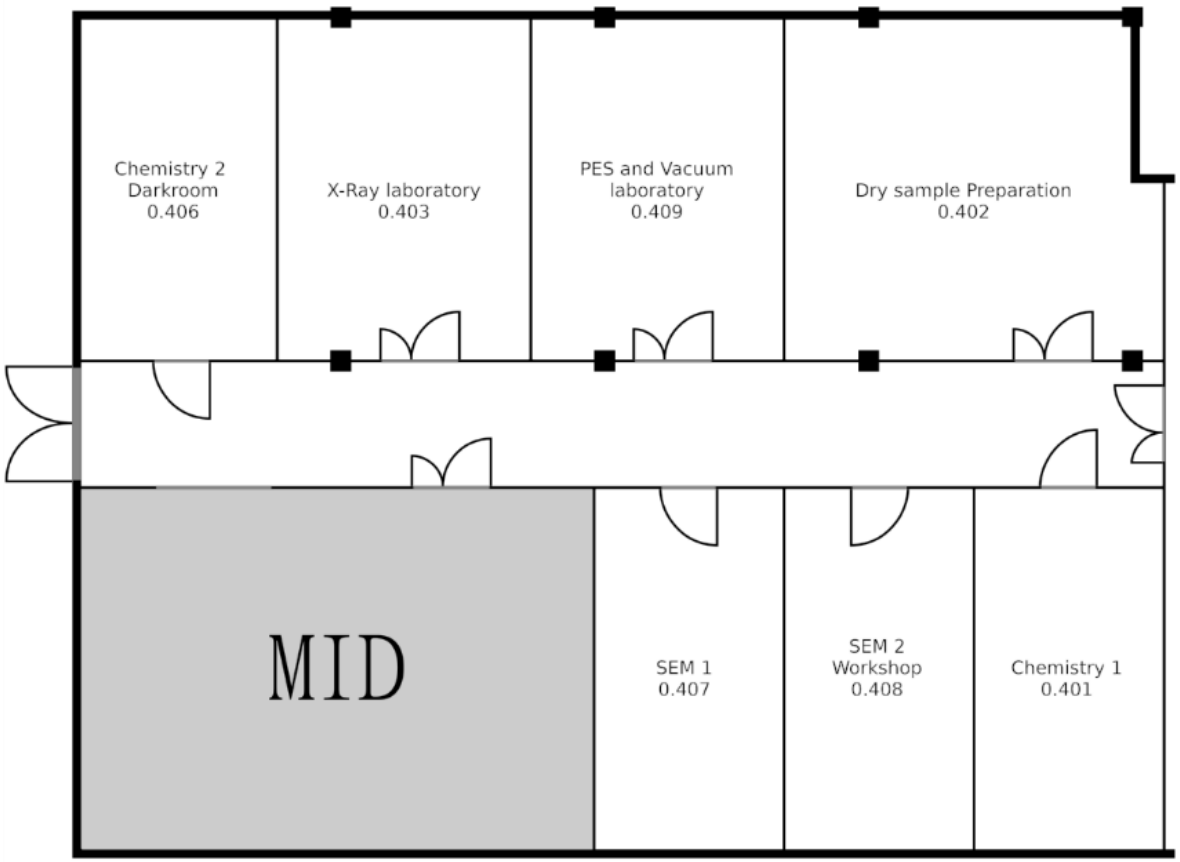
User Laboratories



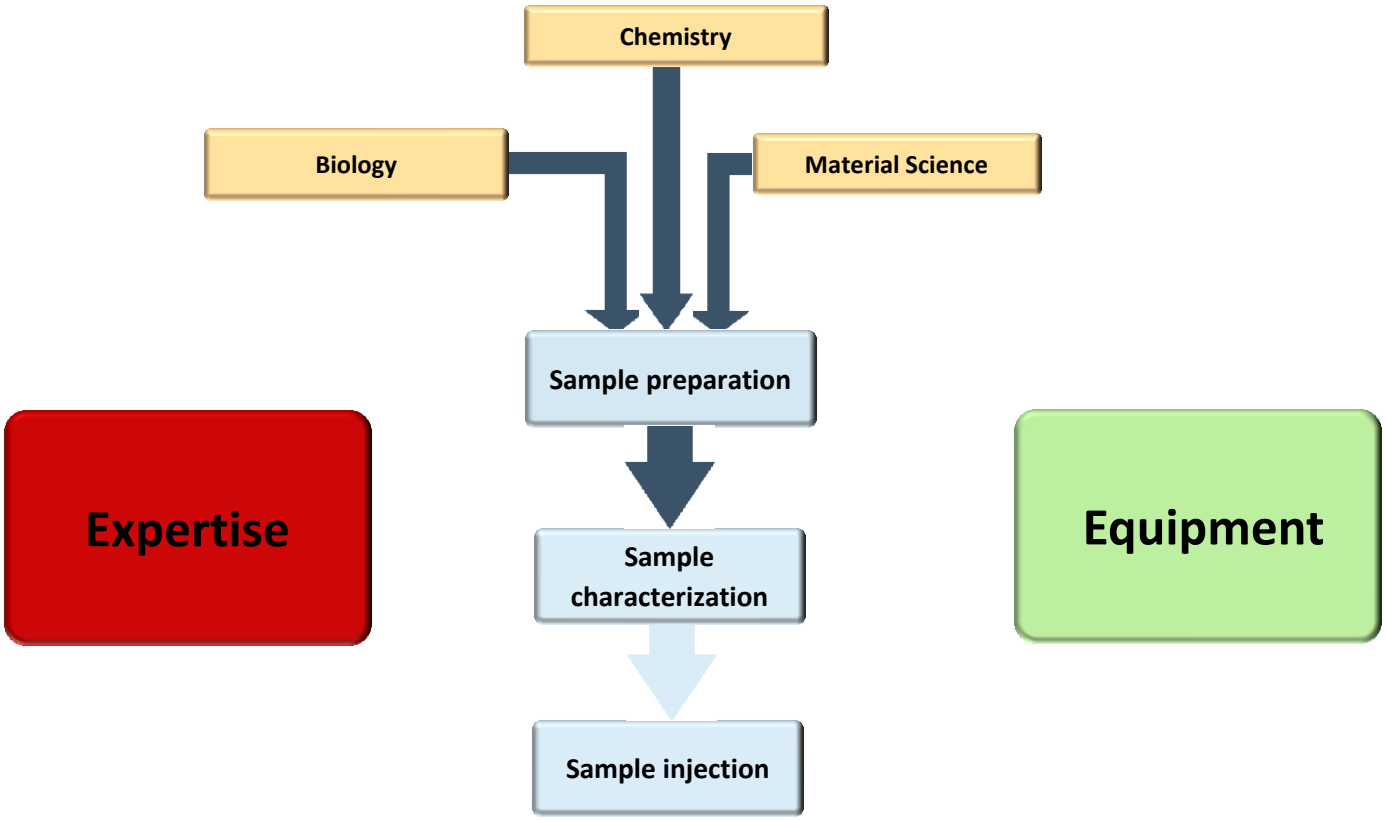
Biological Labs



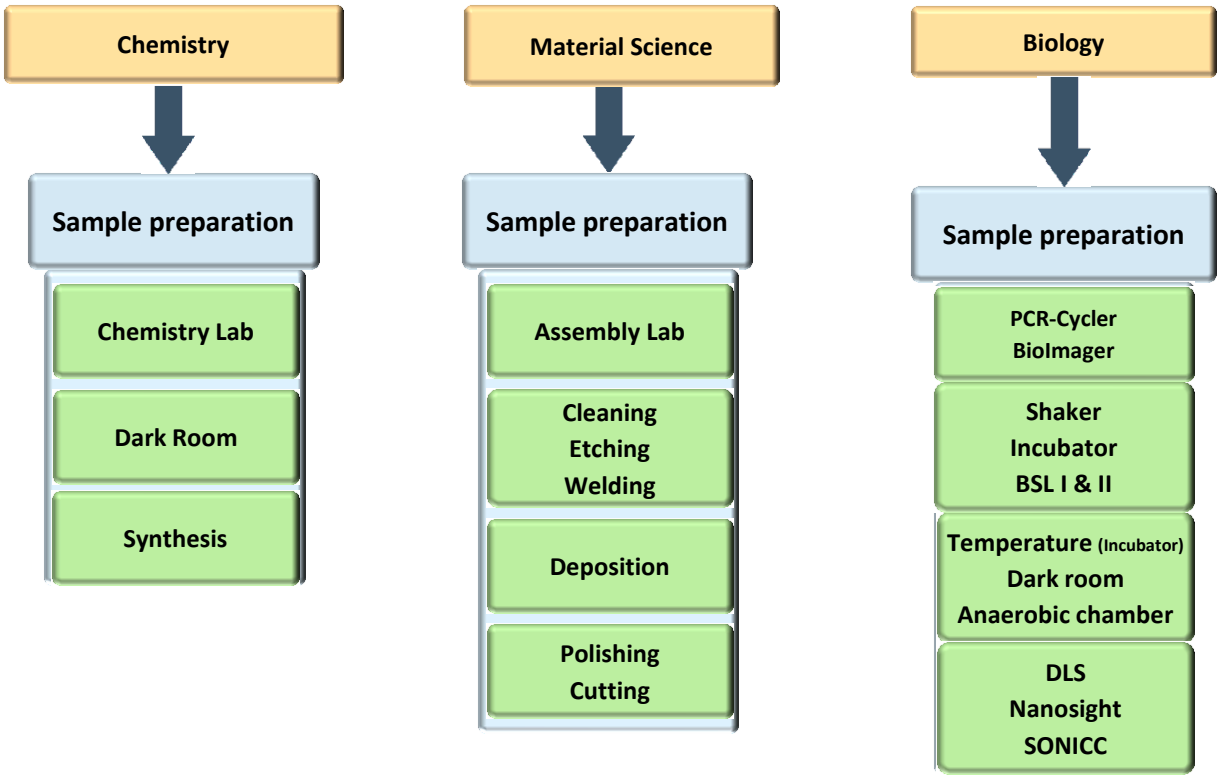
Chemistry and Physics Laboratories



How we can help you getting your sample into the beam



Sample Preparation



Expertise - Chemistry

Vasilii Bazhenov

Sample Preparation - Chemistry

- Sample preparation
  - Chemistry Lab
  - Dark Room
  - Synthesis

- **Wet Lab**
  - Fumehood
  - Stirrer
  - Shaker
  - Heating Plate

Sample Preparation - Chemistry

- Sample preparation
  - Chemistry Lab
  - Dark Room
  - Synthesis

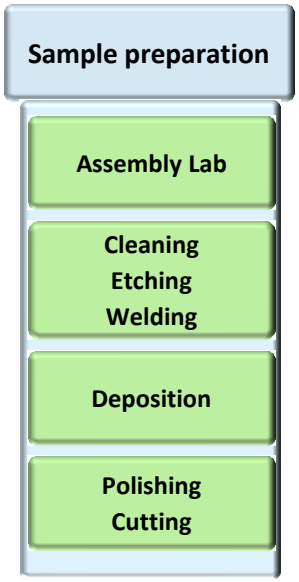
- **Wet Lab**
  - Bench space
  - Fumehood
  - Stirrer
  - Shaker
  - Heating Plate

- **Fume Hood**
  - Schlenk Line
  - Rotary Evaporator (including Vacuum Pump)

Expertise – Material Science

Carsten Deiter James Moore Manuel Izquierdo

Sample Preparation - Material Science/ Solid Samples



- **Assembly Lab**
  - Tools
  - Laminar Flow Clean Tent
  - Optics

Material Science/ Solid Samples

Sample preparation

- Assembly Lab
- Cleaning
  Etching
  Welding
- Deposition
- Polishing
  Cutting

- **Spinncoater**
  - thin film sample deposition
  - Up to 6" (150mm) Ø Wafers
  - Up to 4" x 4" (100mm) Substrates
  - Speed range: 1-12.000 RPM (±0.1 RPM)

Material Science/ Solid Samples

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- **Vacuum Oven**
  - w/wo inert gas
  - Up to 200°C
  - Ex-proof models for safe drying of samples containing flammable solvents

Material Science/ Solid Samples

Sample preparation

- Assembly Lab
- Cleaning
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- **Plasma Cleaner**
  - removal of impurities and contaminants

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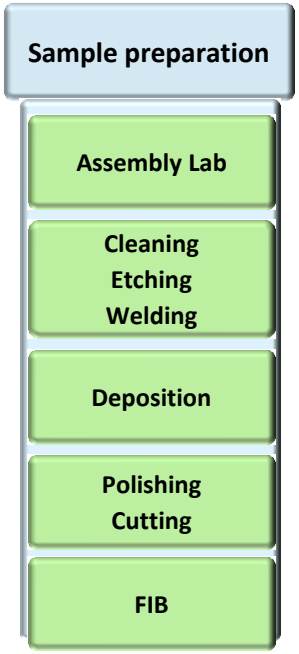
- **Plasma Cleaner**
  - removal of impurities and contaminants

- **Etching**
  - Including hydrofluoric acid

Material Science/ Solid Samples



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  - Ex-proof models for safe drying of samples containing flammable solvents
- **Plasma Cleaner**
  - removal of impurities and contaminants
- **Etching**
  - Including hydrofluoric acid
- **Welding**
  - Including spot welder

Material Science/ Solid Samples

Sample preparation

- Assembly Lab
- Cleaning
  Etching
  Welding
- Deposition
- Polishing
  Cutting
- FIB

- **Spinncoater**
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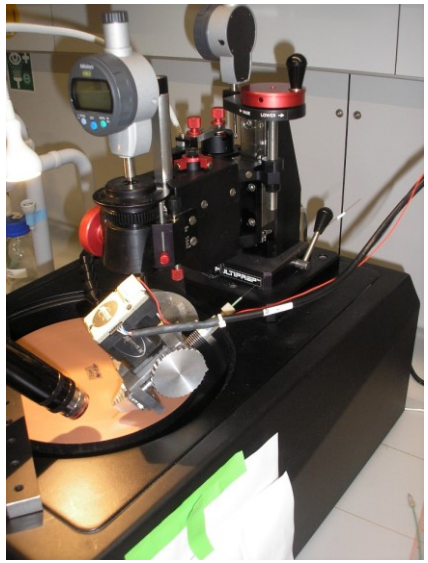
- **Vacuum Oven**
  - w/wo inert gas
  - Up to 200°C
  - Ex-proof models for safe drying of samples containing flammable solvents
- **Plasma Cleaner**
  - removal of impurities and contaminants
- **Etching**
  - Including hydrofluoric acid
- **Welding**
  - Including spot welder
- **Polishing and cutting**
  - Grinder
  - Microtome



Material Science/ Solid Samples

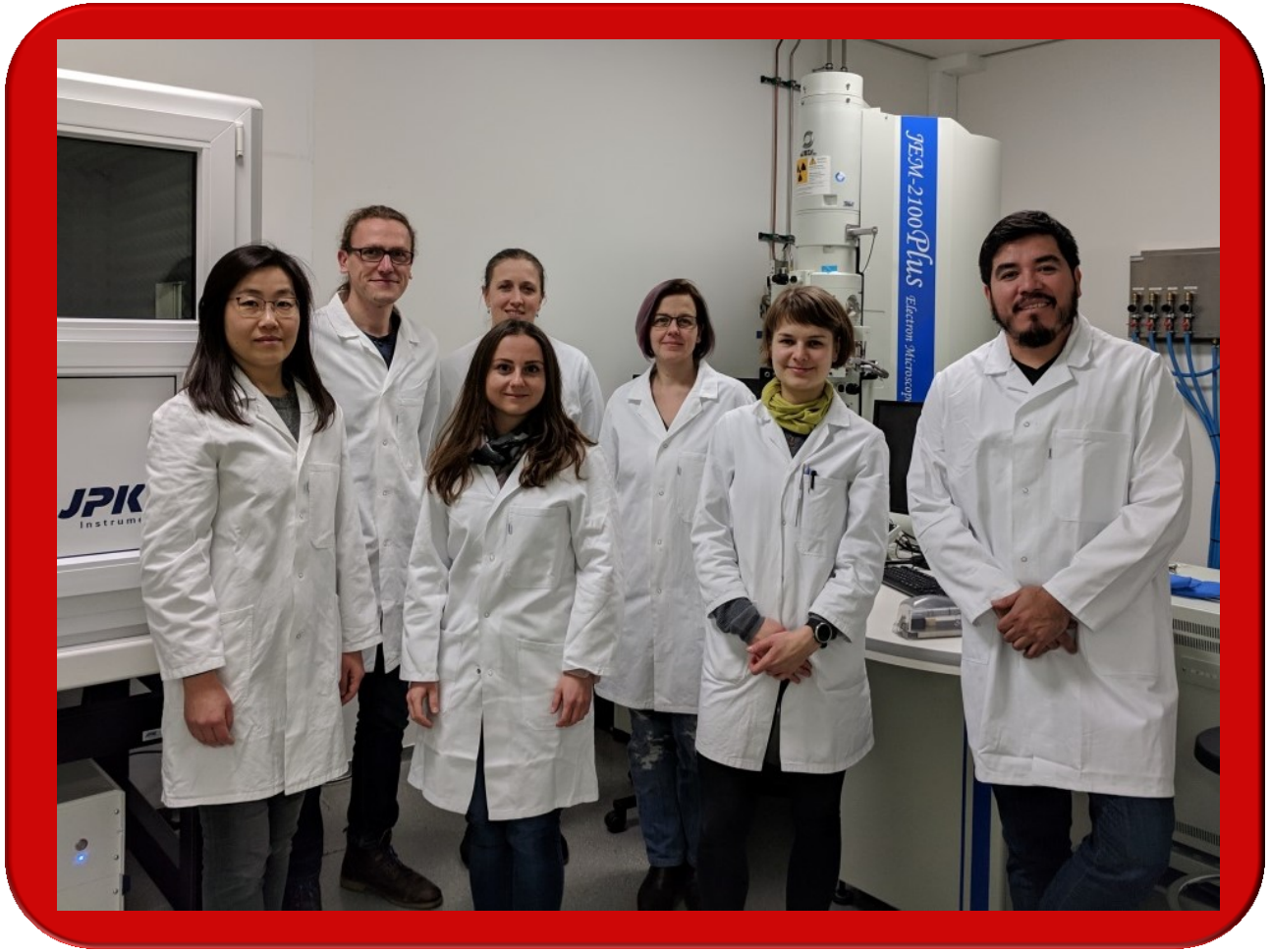
Sample preparation

- **Assembly Lab**
- **Cleaning**
  Etching
  Welding
- **Deposition**
- **Polishing**
  Cutting
- **FIB**

FEI Helios G4 UC

- Available in late 2019
- TEM lamella preparation
- Nano-patterning via ion-milling
- Pt and C nano-deposition

Expertise - Biological Sample Preparation



Huijong Han
Robin Schubert
Yasmin Gül
Ekaterina Round
Kristina Lorenzen
Jana Makroczyova
Domingo Meza

Biological Sample Preparation

Safety cabinets FACS BSL I & II

- Insect cell culture

Insect cells

Expression

E. coli

- Bacterial cell culture

Shaker Incubator BSL I & II

- 2 large **shaker** and 1 **incubator** in BSL I and BSL II

- 3 **Microbiological safety cabinets** in BSL II , 1 in BSLI
  - Type B2

- **Fermenter**
  - Infors Labfors 5

- **Large Volume Centrifuge**
  - Sorvall Lynx 6000
- **Sonicator**
  - Qsonica Q700
- **Homogenizer**
  - Avestin EmulsiFlex-C3

Safety cabinets FACS BSL I & II

- Insect cell culture

Insect cells

Expression

E. coli

- Bacterial cell culture

Shaker Incubator BSL I & II

Protein purification

- Cell disruption

Homogenizer Sonicator

- Affinity chromatography
- Size exclusion chrom.
- Precipitation

Chromatography Ultracentrifuge

- Verify protein purity

SDS-PAGE

- **Chromatography**
  - GE Healthcare Äkta Pure
  - 4 °C and 20 °C
    - ► Size exclusion chromatography
    - ► Affinity chromatography
      - MonoQ/MonoS 5/50 GL
      - Superdex 75/100 increase 10/300 GL
      - Superdex 75/100 HiLoad 16/600 and Superdex 200 26/600

- **Ultracentrifuge**
  - Beckman Coulter Optima XPN-90
  - Beckman Coulter Optima MAX-XP
    - ► Up to 500k g
    - ► Fixed angle Typ 45 Ti and Typ 70 Ti
    - ► Swinging bucket SW 60 Ti

Protein crystallization

- Choose crystallization environment

Temperature (Incubator)
Dark room
Anaerobic chamber

- Screen and optimize crystallization conditions

Crystallization robot
SONICC

- Prepare seed-stocks

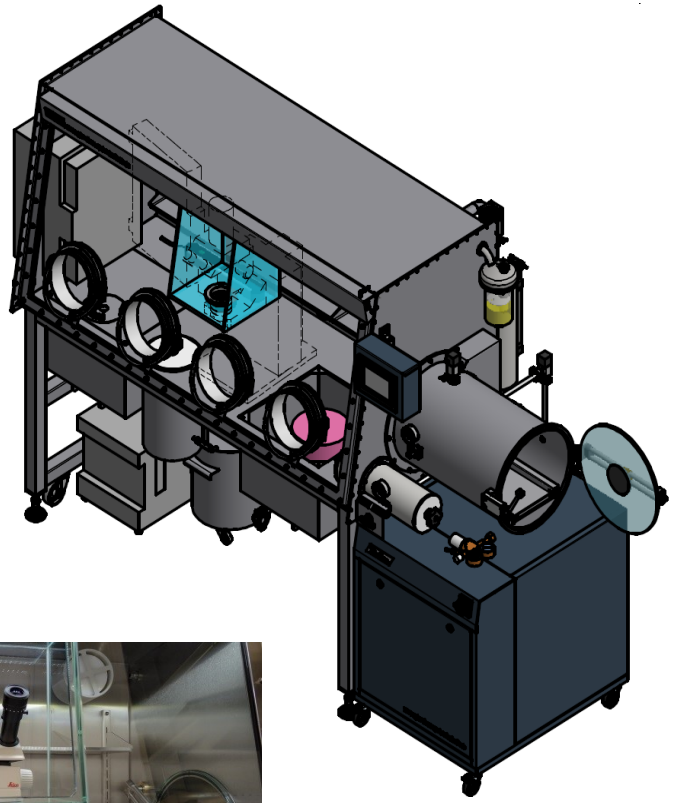
DLS
Nanosight

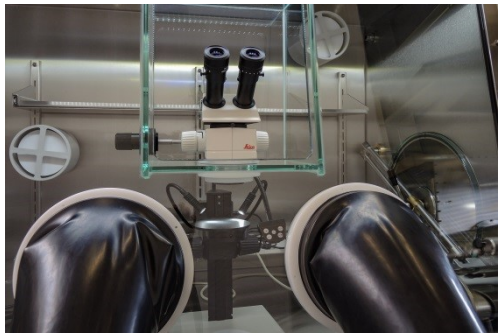
- Batch-crystallization

Anaerobic chamber

Glovebox Systemtechnik

- 4-glove glovebox
- N_2 or Ar, oxygen free atmosphere (<0.5 ppm)
- Over and underpressure operation
- 4° C fridge
- Humidifier
- Solvent adsorber
- Microscope in front glass (Leica M205)
- Flask storage and retractable shelfs
- Spin coater (Polos SPIN150i)





Protein crystallization

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Temperature (Incubator)
Dark room
Anaerobic chamber

- Screen and optimize crystallization conditions

Crystallization robot
SONICC

- Prepare seed-stocks

DLS
Nanosight

- Batch-crystallization

Crystallization robot (coming soon)

- ▶ Small volume pipetting
- ▶ LCP-dispensing

Crystallization plate imager

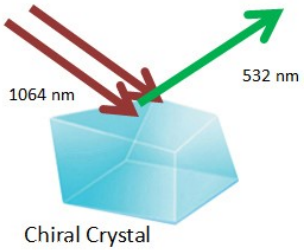
Formulatrix SONICC

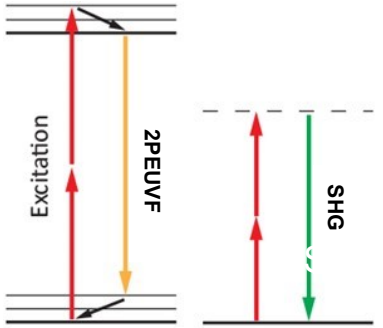
- ▶ Visible imaging
- ▶ SHG imaging
- ▶ UP-TPEV imaging

Crystallization incubator

Sanyo

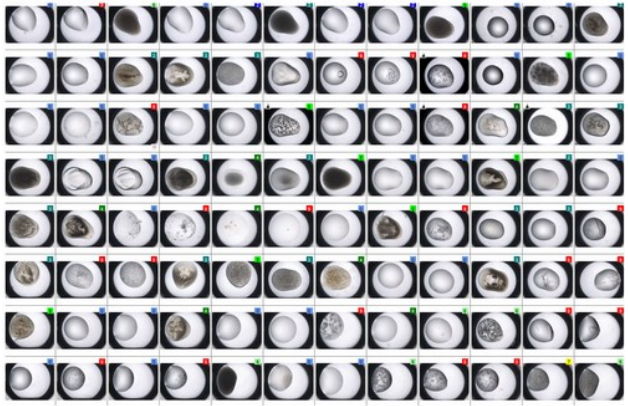
- ▶ 2 incubators
- ▶ 1 incubator in dark room



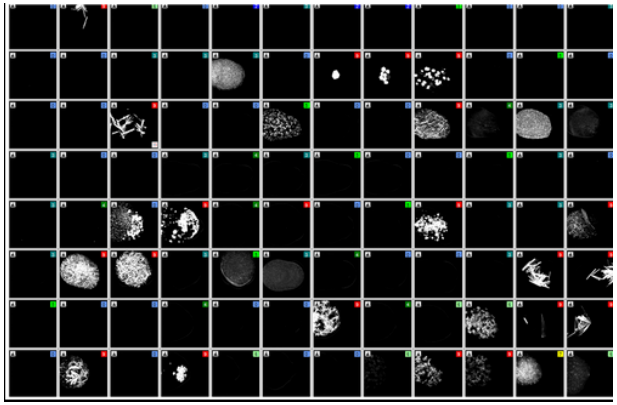


© semrock.com (modified)

visible



SHG



© formulatrix.com

- **Crystallization robot (coming soon)**
  - ▶ Small volume pipetting
  - ▶ LCP-dispensing
- **Crystallization plate imager**
  - Formulatrix SONICC
    - ▶ Visible imaging
    - ▶ SHG imaging
    - ▶ UP-TPEV imaging

Protein crystallization

- Choose crystallization environment

Temperature (Incubator)
Dark room
Anaerobic chamber

- Screen and optimize crystallization conditions

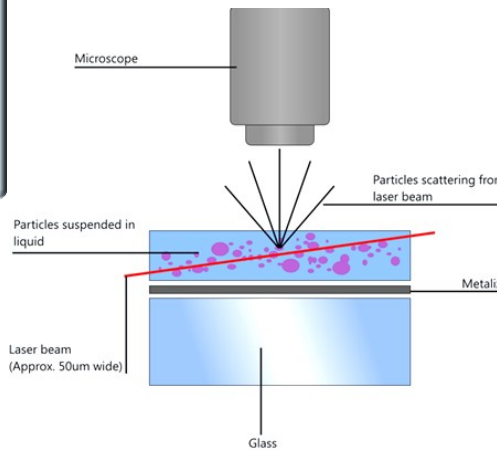
Crystallization robot
SONICC

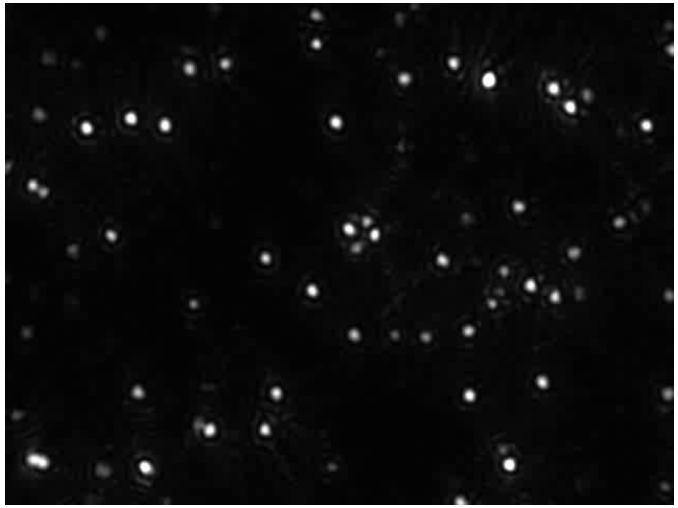
- Prepare seed-stocks

DLS
Nanosight

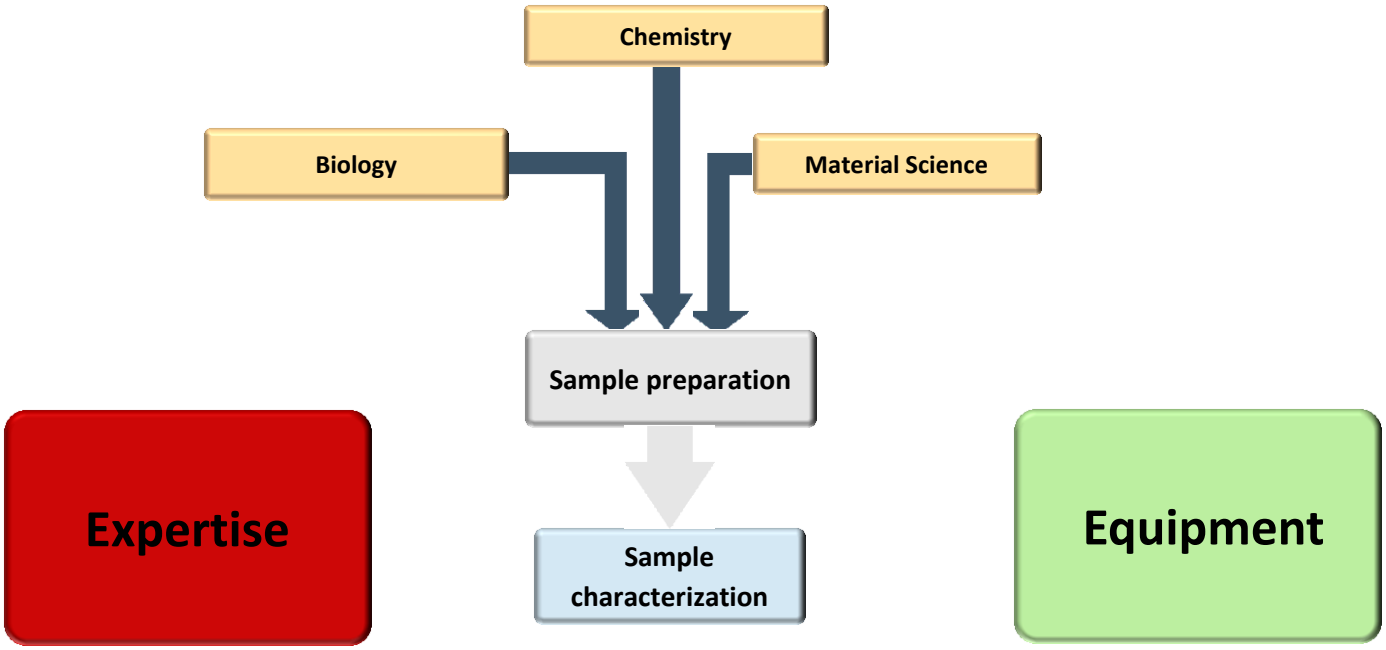
- Batch-crystallization

- **Crystallization incubator**
  - Sanyo
    - ▶ 2 incubators
    - ▶ 1 incubator in dark room
- **Single Particle Tracking**
  - Malvern NS300 Nanosight
    - ▶ Hydrodynamic radius (20 nm – 2 µm)
    - ▶ Concentration measurements (10^6 – 10^9 ml^{-1})
    - ▶ 480 nm laser

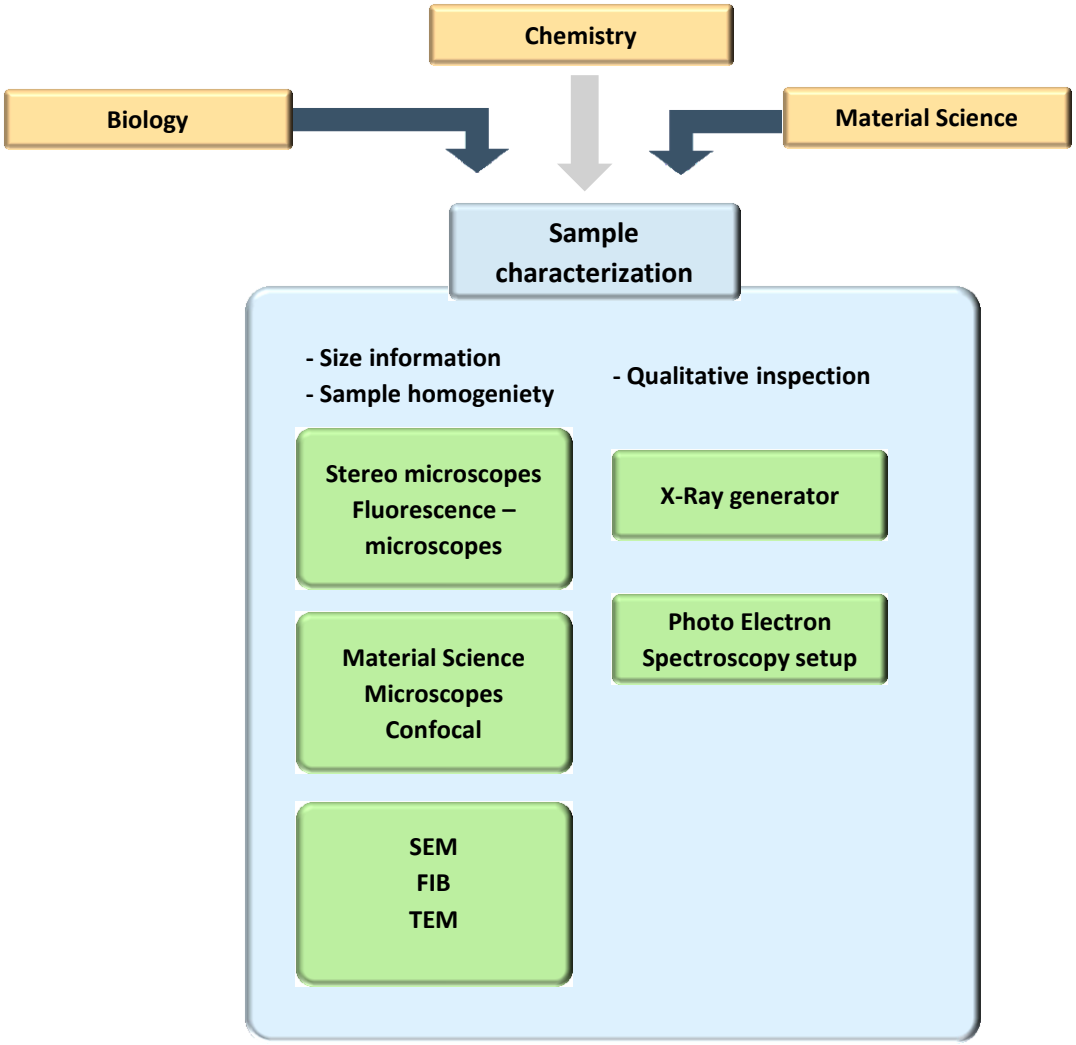




How we can help you getting your sample into the beam



Sample Characterization



Stereo Microscopes

- Leica M205 FCA
- Nikon SMZ18
- Olympus SZX16

Sample characterization

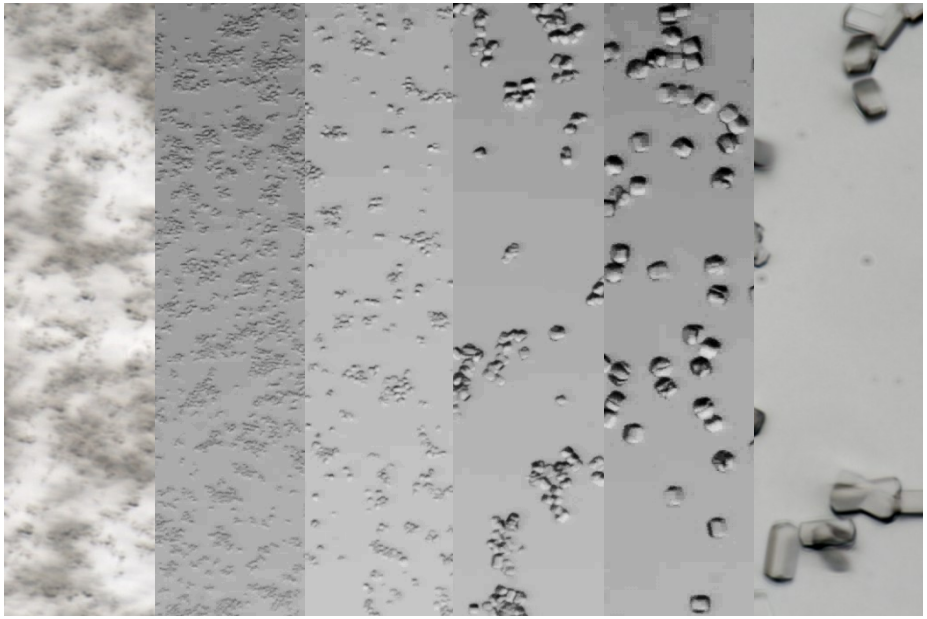
- Size information
- Sample homogeniety

Stereo microscopes
Fluorescence – microscopes

Material Science
Microscopes
Confocal

SEM
FIB
TEM

Lysozyme (XBI)



Sample characterization

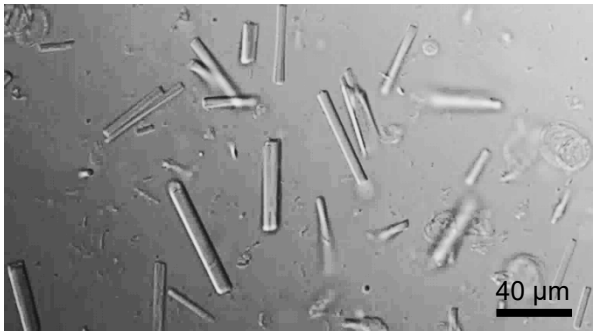
- Size information
- Sample homogeniety

Stereo microscopes Fluorescence – microscopes

Material Science Microscopes Confocal

SEM
FIB
TEM

- PAK4 (Betzel group)



- **Fluorescence Microscopes**
  - Nikon Fluorescence NI-E (upright)
  - Nikon Fluorescence TI2-E (inverted)

- **Material Science Microscopes**
  - Keyence VHX-6000

- **Confocal Microscopes**
  - Nano Focus Custom

Sample characterization

- Size information
- Sample homogeniety

Stereo microscopes
Fluorescence – microscopes

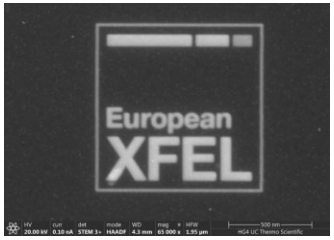
Material Science
Microscopes
Confocal

SEM
FIB
TEM

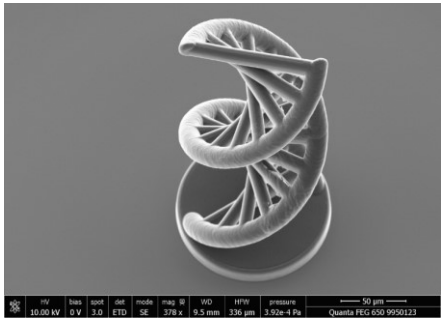
Scanning Electron Microscope (SEC lab)

- FEI Quanta FEG 650
  - ▶ SE, BS, ETD
  - ▶ WetSTEM detector
  - ▶ EDS detector
- Leica sputter coater
  - ▶ Carbon, gold, iridium coating
  - ▶ Glow discharging

- STEM image of XFEL logo
  - Platinum deposited on Si_3N_4-membrane



- SEM image of 3D printed structure
  - Aim to print nozzles in photoresin



Sample characterization

- Size information
- Sample homogeniety

Stereo microscopes
Fluorescence – microscopes

Material Science
Microscopes
Confocal

SEM
FIB
TEM

FEI Helios G4 UC

- STEM imaging of lamellas
- EDS detector

Sample characterization

- Size information
- Sample homogeniety

Stereo microscopes
Fluorescence – microscopes

Material Science
Microscopes
Confocal

SEM
FIB
TEM

Transmission Electron Microscope

- Jeol JEM-2100 Plus
  - ▶ 200 keV
  - ▶ Cryo pole piece
  - ▶ Fishione cryo holder
  - ▶ Negative staining with non-radioactive dyes
- Leica UC7 Ultramicrotome
  - ▶ Glass and diamond knife
  - ▶ Thin sections down to 50 nm
  - ▶ Cutting at RT and cryo conditions
- Leica GP2 Cryo plunge freezer
  - ▶ Grid freezing in liquid ethene

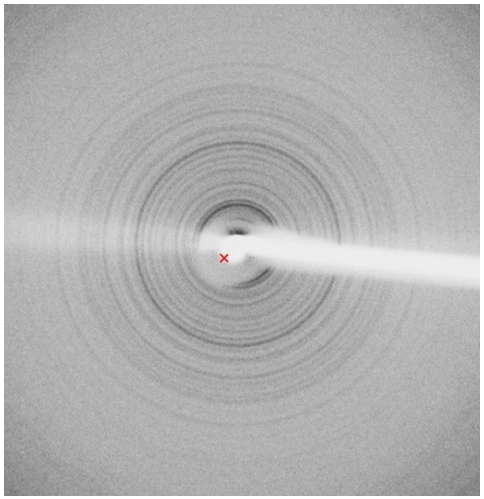
Sample characterization

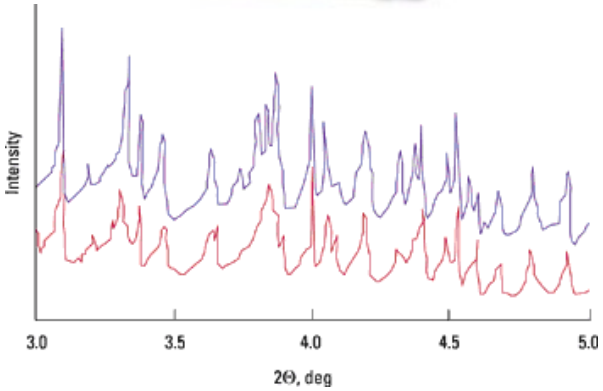
- Qualitative inspection

X-Ray generator

Photo Electron Spectroscopy setup

- **X-Ray generator (SEC lab)**
  - Bruker D8 Advance
    - ► Powder diffraction analysis
    - ► Obtain 1D diffraction spectrum





Robert Von Dreele, 2003

Photoelectron spectrometer

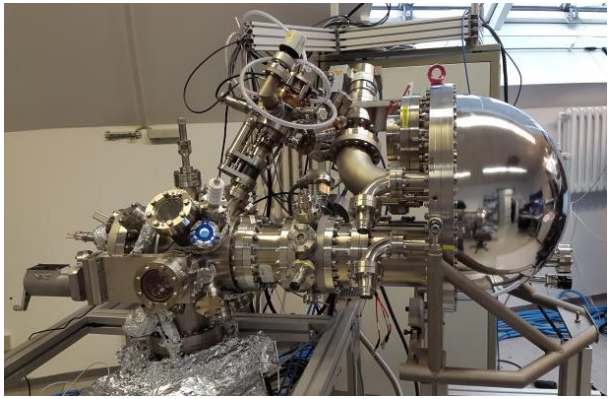
Sample characterization

- Qualitative inspection

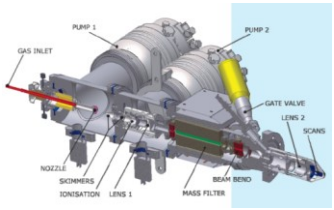
X-Ray generator

Photo Electron Spectroscopy setup

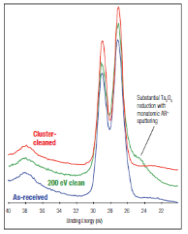
- Electronic structure
  - Band dispersion
  - Fermi surface
- Chemical analysis
  - Alloying
  - Chemical segregation
- Magnetic properties
  - Spin resolved PE
  - MCD/MLD in PE
- Sample characterization
  - Single crystals
  - Thin films
  - Clusters

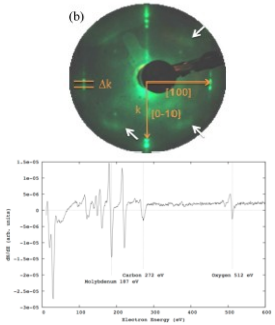


Cluster source



LEED/Auger





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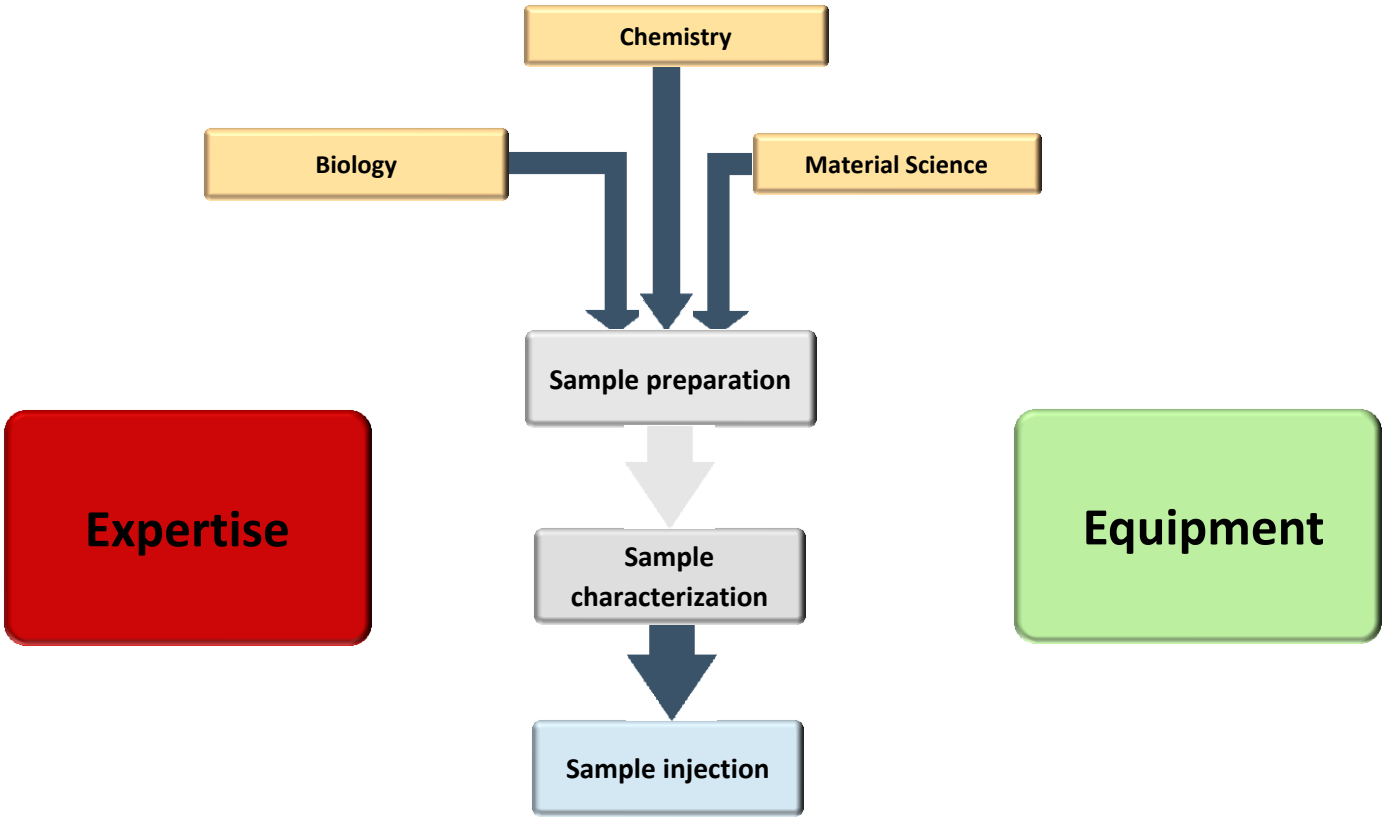


Expertise – Sample Injection

Katerina Dörner Johan Bielecki

- **Nozzle fabrication**
  - GDVN or Microfluidic nozzels, flat sheets, Rayleigh Jets
    - ► Grinder
    - ► Plasma cleaner

Sample injection

- Nozzle assembly
- Rapid prototyping
- Microfluidics

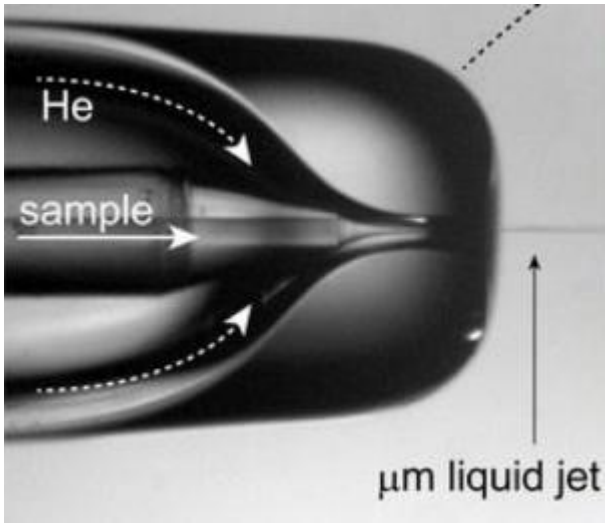
Nozzle fabrication Nano-3D printer

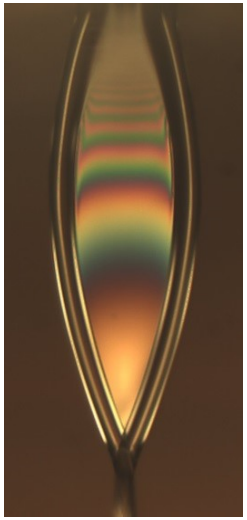
- Test sample injection
- Adjust concentration

Liquid-jet test-chamber
Aerosol-jet test-chamber

Fast Solid Sample Scanner

Magnetic Fields





https://lcls.slac.stanford.edu/sed/equipment

Sample injection

- Nozzle assembly
- Rapid prototyping
- Microfluidics

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Liquid-jet test-chamber Aerosol-jet test-chamber

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- GDVN or Microfluidic nozzels, flat sheets, Rayleigh Jets
  - ▶ Grinder
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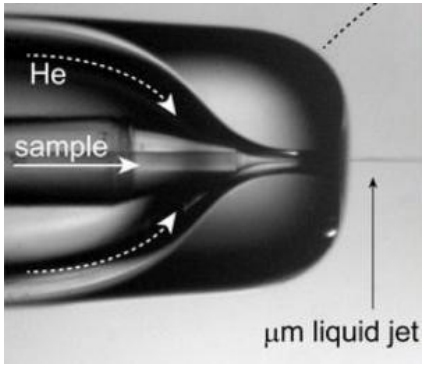

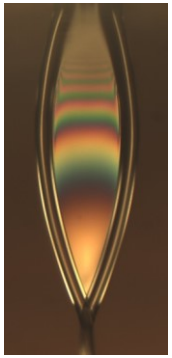

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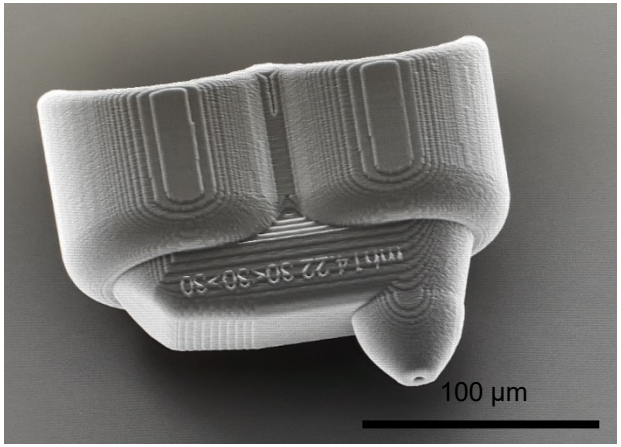
Nano 3D printer (SEC lab)

- Nanoscribe Photonic Professional GT2
  - ▶ Two-photon polymerization printing
  - ▶ Lateral feature sizes 160 nm
  - ▶ Printing area of up to 100 × 100 mm^2



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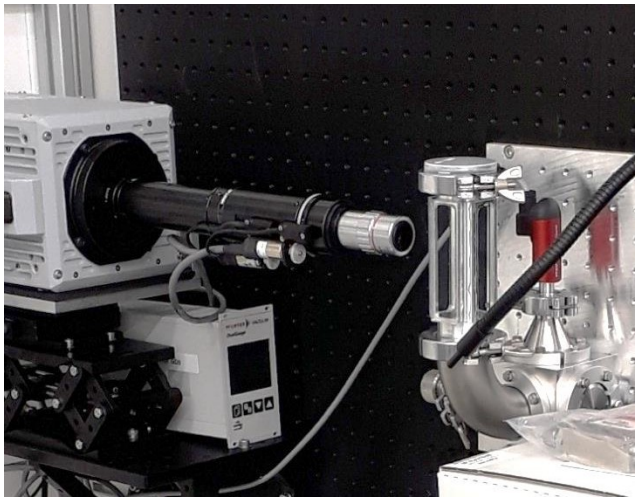

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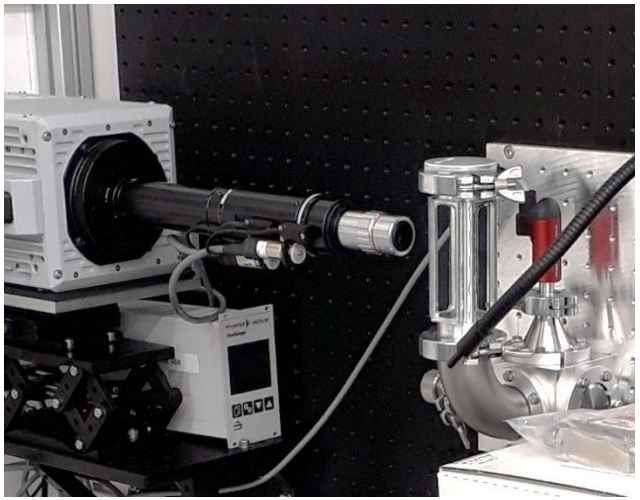

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  - ▶ GDVN
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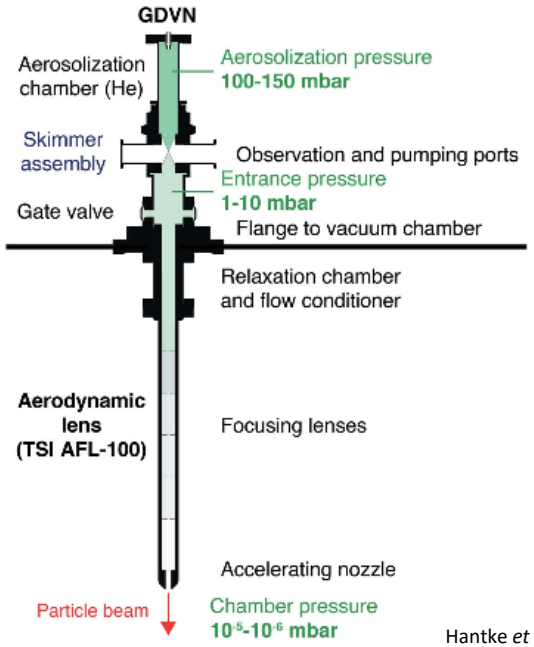

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- SCS, MID and HED planned
  - ▶ Pre-characterization and localization
  - ▶ Load Lock with sample changer

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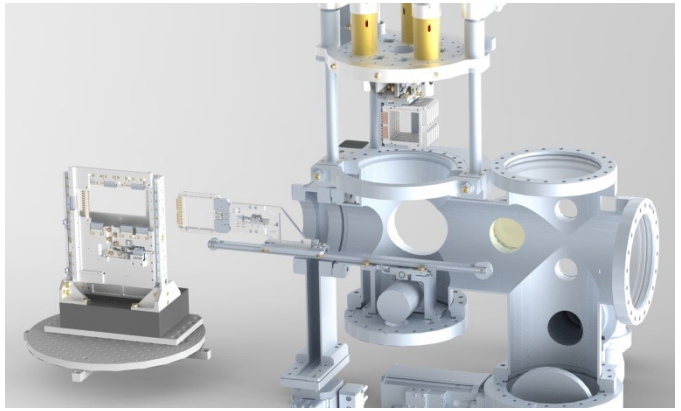

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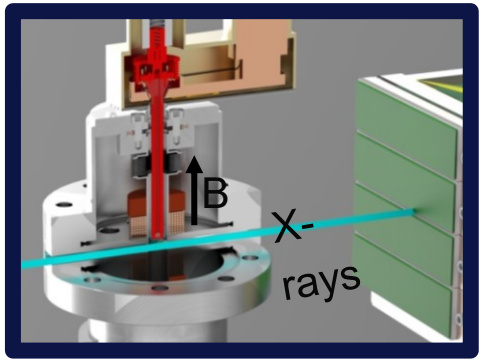
- Home built miniature coils with cryostat



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… moving science forward

- Providing knowledge and support for sample preparation
- Support users with sample testing and injection
- Develop new injection methods

- Help potential future users with sample characterization for beamline proposals
- Scientific collaboration projects
- Spread knowledge (schools, courses, workshops)

Acknowledgements

Sample Environment Group

All European XFEL Staff

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ASU EMBL European XFEL

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UH

UKE HAMBURG

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UPPSALA UNIVERSITET

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