Freie Universität Berlin | HZB Helmholtz Zentrum Berlin | TECHNISCHE UNIVERSITÄT DRESDEN | TU BERGAKADEMIE FREIBERG

Kick-Off-Meeting

German-Eastern-European-Laboratory for Energy Materials Research (GELEM)

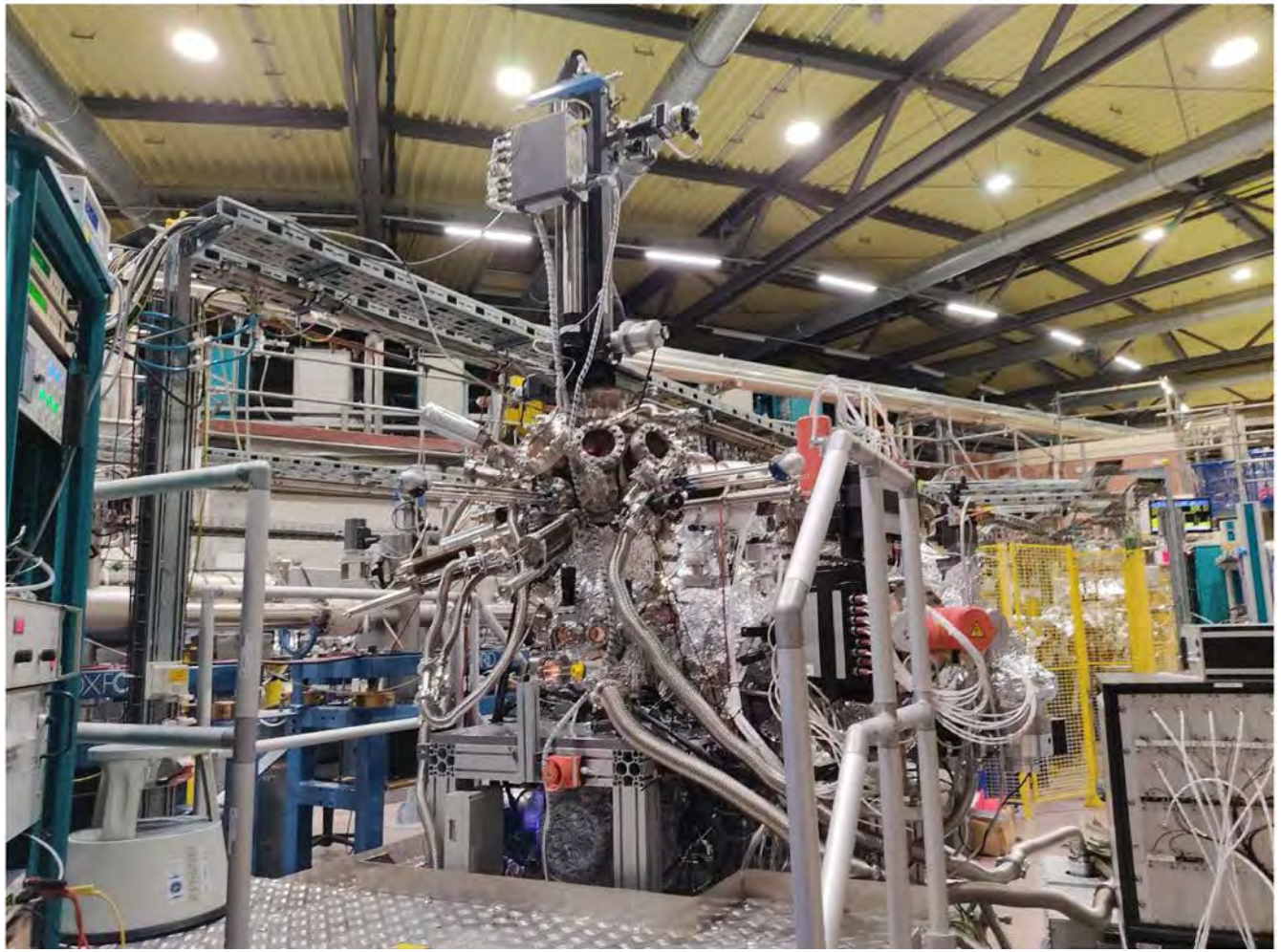
July 07, 2023 - 14:00 (CEST)

Agenda

1. Welcome	Eckart Rühl (FU Berlin)
2. Research Opportunities at the GELEM Laboratory	Clemens Laubschat (TU Dresden / Martin Weinelt, FU Berlin)
3. Overview on the Experimental Resources and Research Highlights	
(a) Dipole Beamline	Anna Makarova (FU Berlin)
(b) Undulator Beamline	Oliver Rader (HZB)
4. Beam Time Proposals at BESSY II and their Evaluation	Antje Vollmer (HZB)
5. Practical Aspects of Beam Times	Anna Makarova (FU Berlin)
6. Future Prospects of GELEM	Martin Weinelt (FU Berlin)
7. Questions and Answers	Serguei Molodtsov (TU Freiberg)
8. Summary and Conclusions	Eckart Rühl (FU Berlin)

2. Research Opportunities @ U125_PGM

Spin- and angle-resolved photoemission



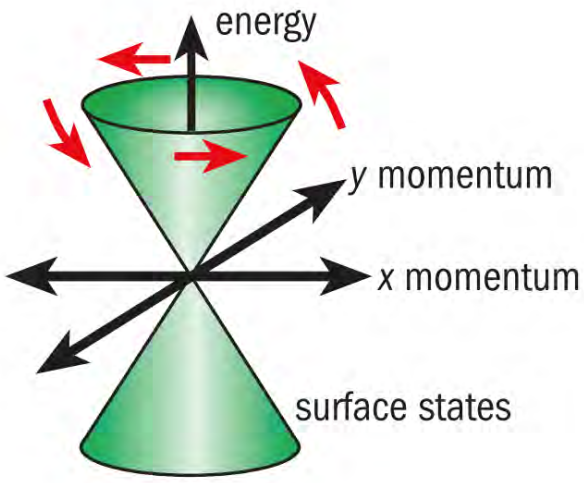
Scope of previous users on electronic and spin structure

- Topological insulators
- Weyl nodes and spin texture of Fermi arcs in Weyl semimetals
- Magnetic and spintronic materials
- Metal intercalation in graphene
- Conversion of fullerenes into graphenes

Courtesy of Oliver Rader !

Motivation: New materials for next generation spin and electronic applications

- Linear (massless) dispersion
- High carrier mobility
- Large transversal magnetoresistance
- Negative longitudinal magnetoresistance



Showcase projects:

- Topological insulator & magnetic field → Opening of band gap
- Dirac semimetal with tunable Dirac point
- Dirac semimetal & magnetic field → Weyl semimetal, appearance of Fermi arcs

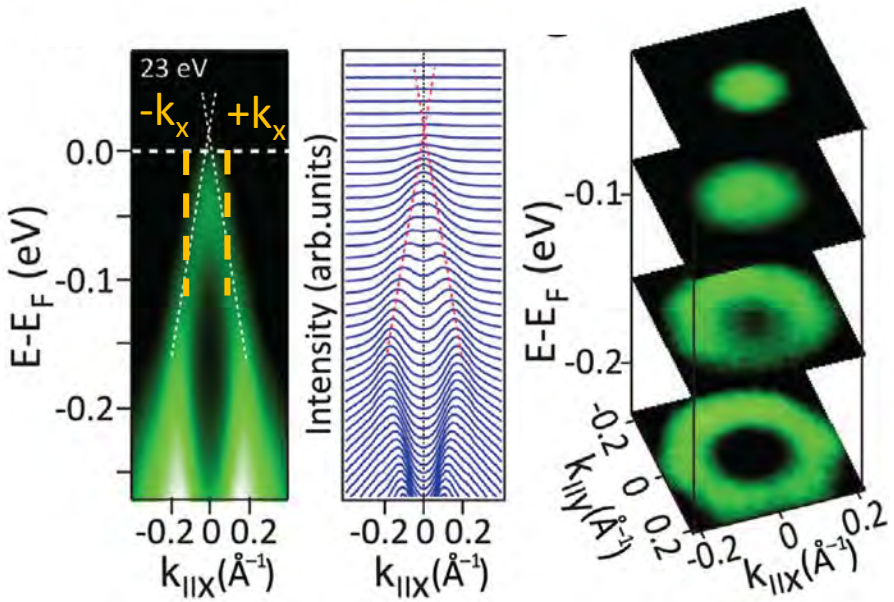
- Lead halide perovskites

Wimmer et al., Advanced Materials **33** (2021) 2102935.

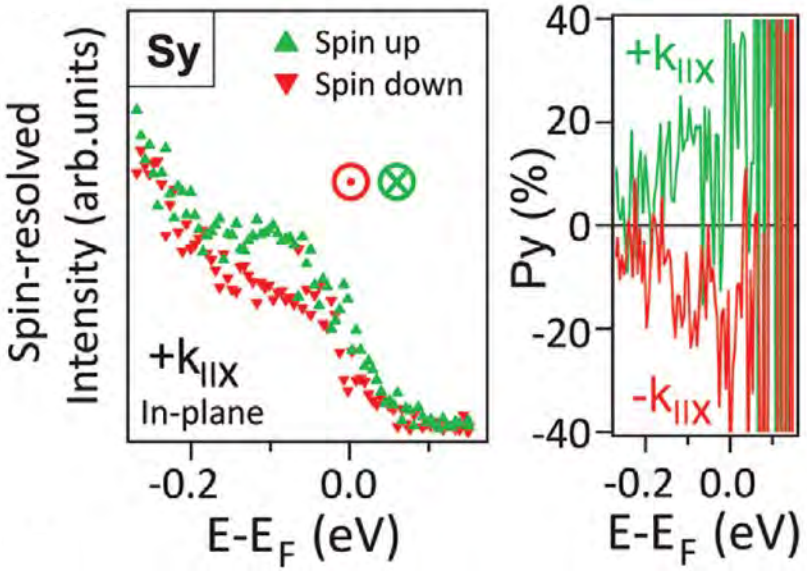
Ferromagnetic topological insulator: p-type $MnSb_2Te_4$

- 2D Dirac cone with Dirac point close to the Fermi level
- Mn stoichiometric → ferromagnetic, high Curie temperature of 50 K
- Out-of-plane magnetic anisotropy
- PES proves out-of-plane spin polarization
- STS reveals bandgap closing at the Curie temperature

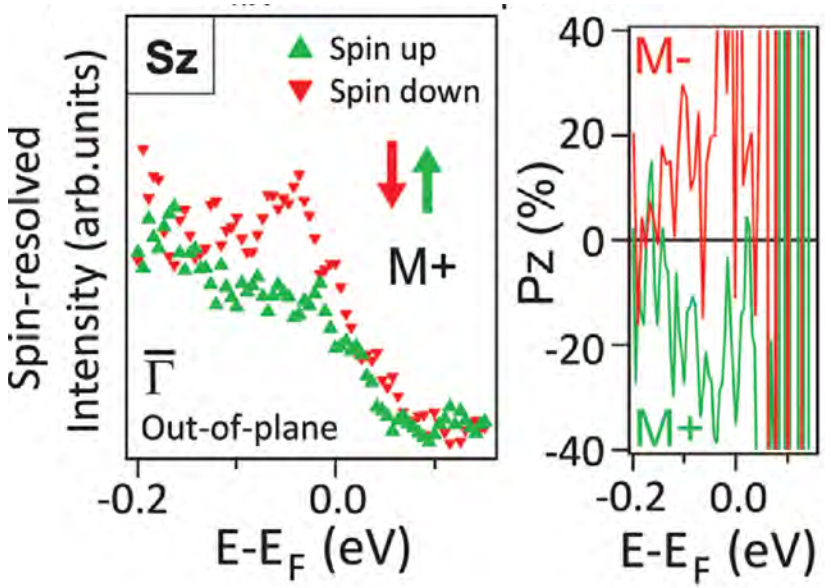
Dirac cone with linear dispersion



Helical spin texture



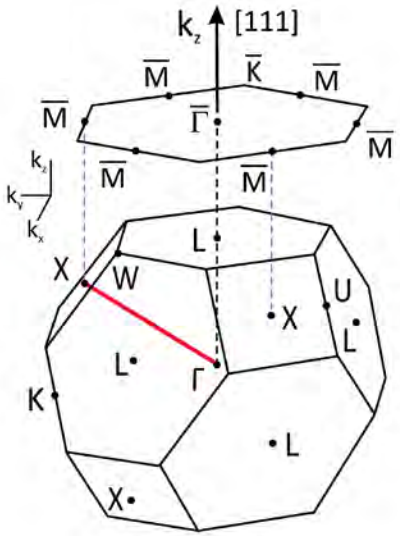
Out of plane spin component

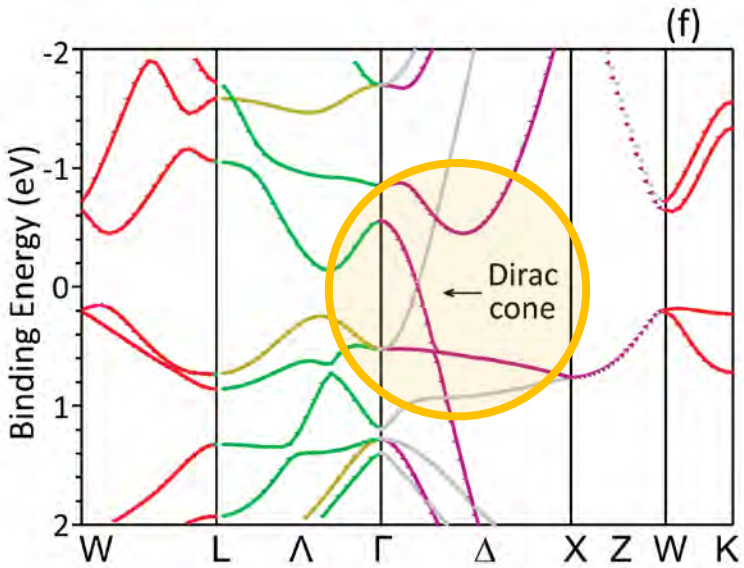


➢ Gap closing at T_C confirmed by STS

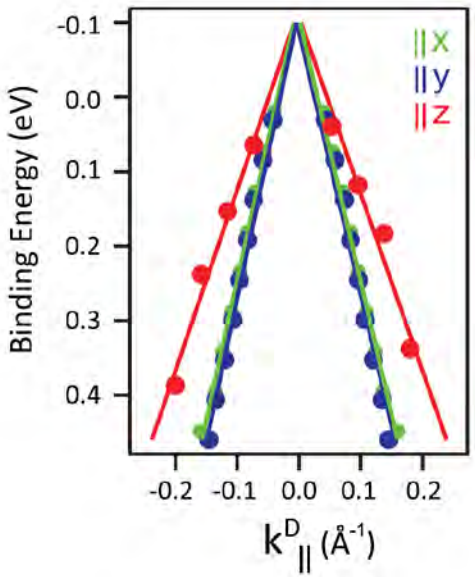
Sánchez-Barriga et al., Phys. Rev. Lett. 130 (2023) 236402

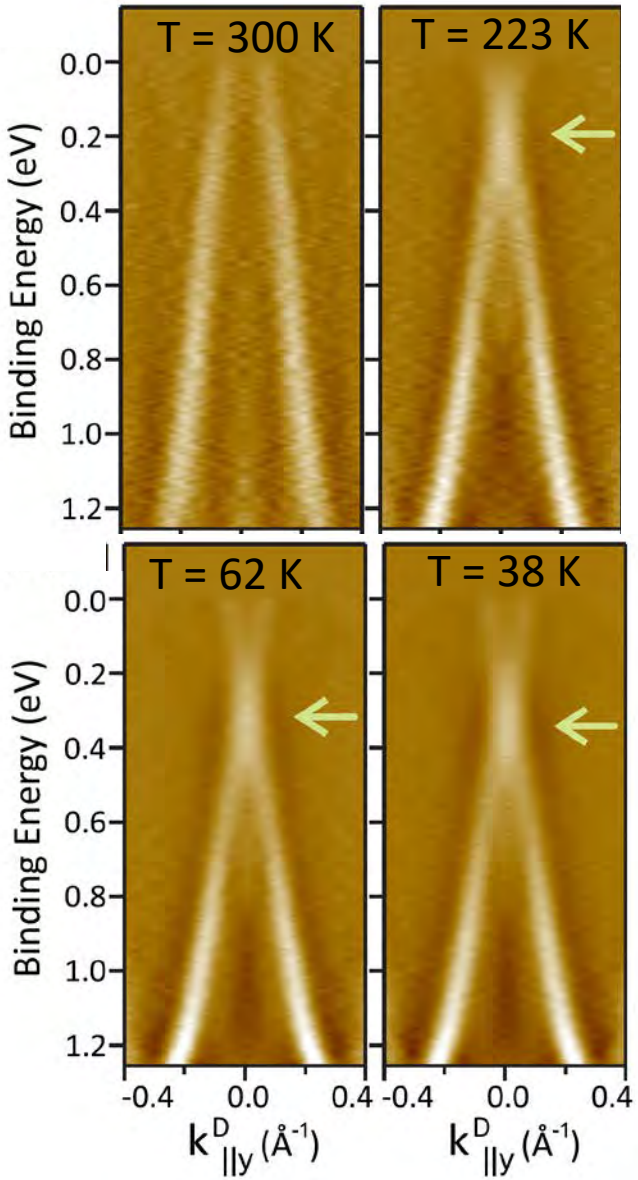
3D Dirac semimetal Au_2Pb with tunable Lifshitz transition





3D Dirac cone protected by C_{4v} symmetry & SOC

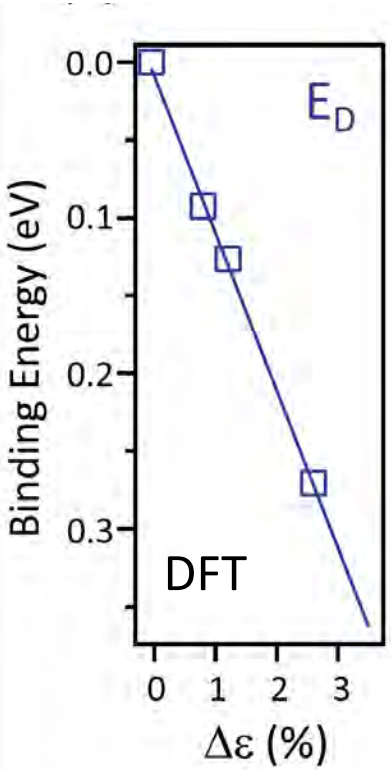




450 meV shift of Dirac cone:

Transition from hole-type to electron-type Fermi surface

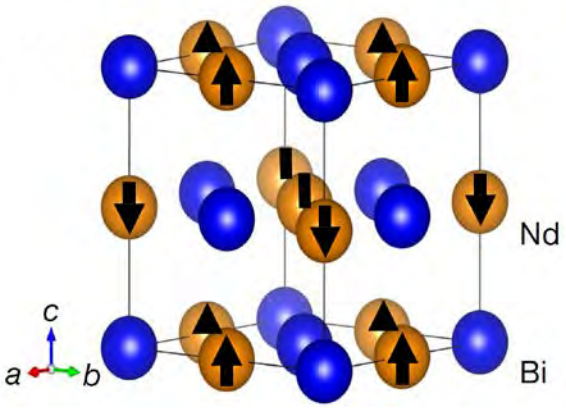
T-dependent lattice compression

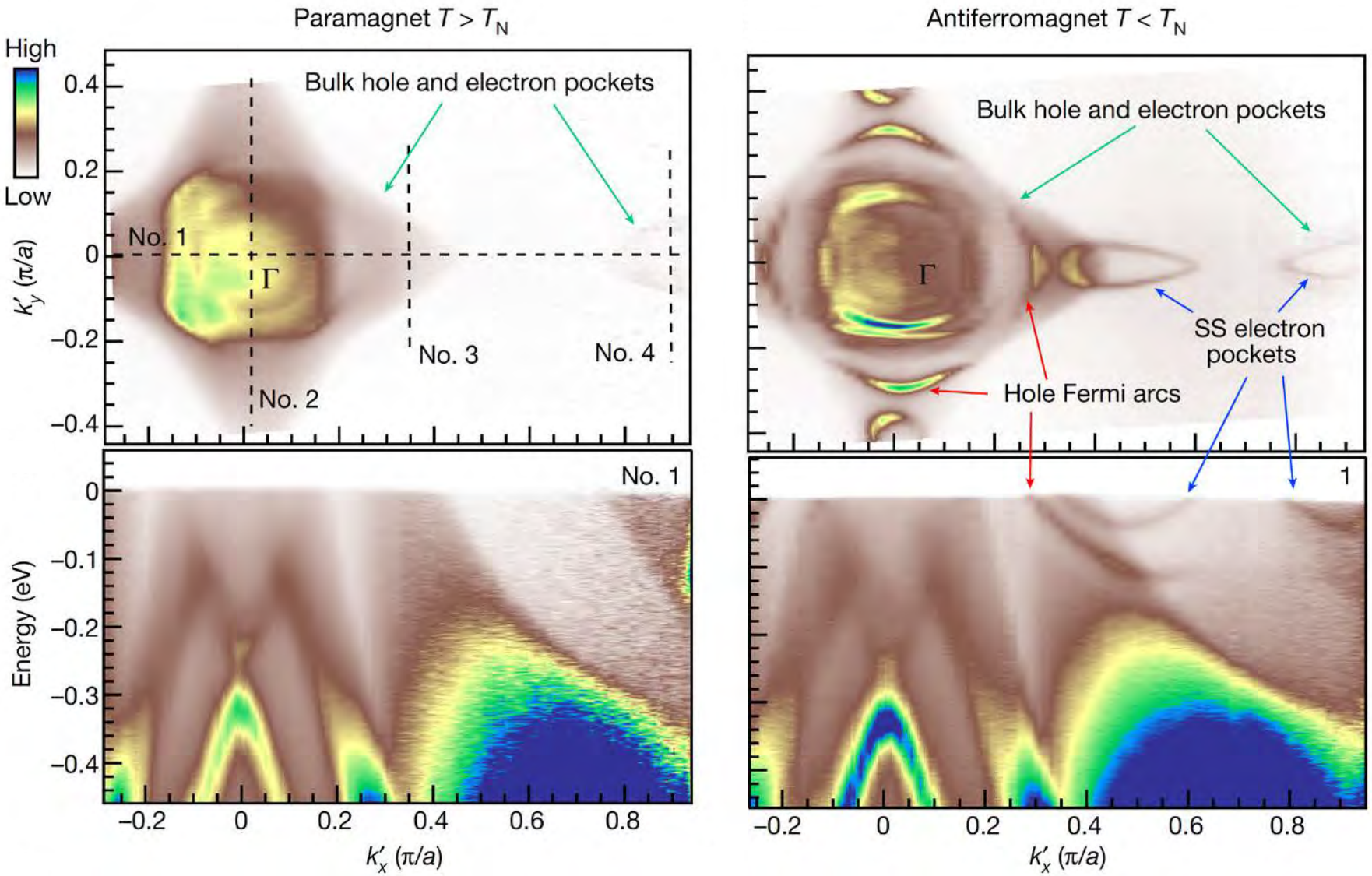


Schrunk et al., Nature 603 (2022) 610.

Fermi arcs in an antiferromagnet

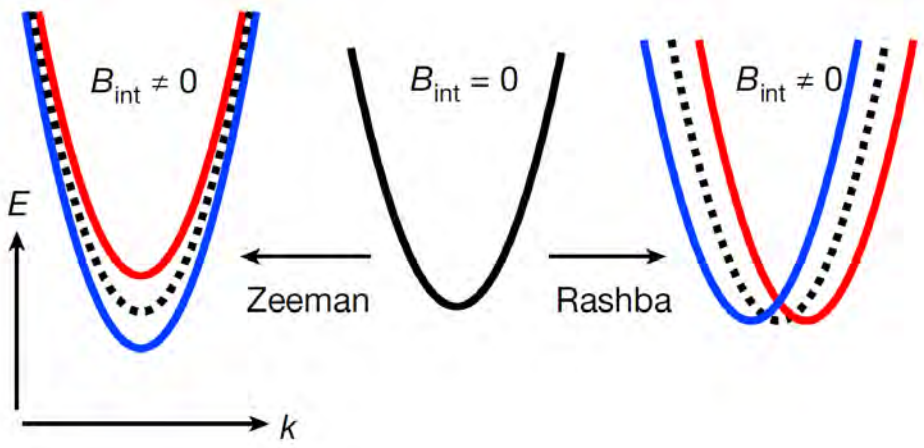
- Cubic monopnictide NdBi
- Antiferromagnetic below 24 K
- Fermi arcs below T_N
- Unusual magnetic splitting
- Complex dichroic response / signature of spin texture



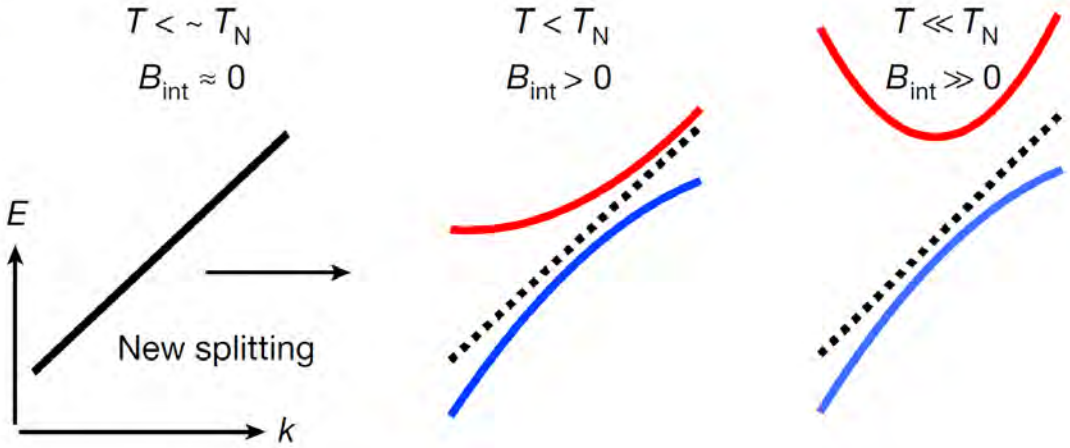


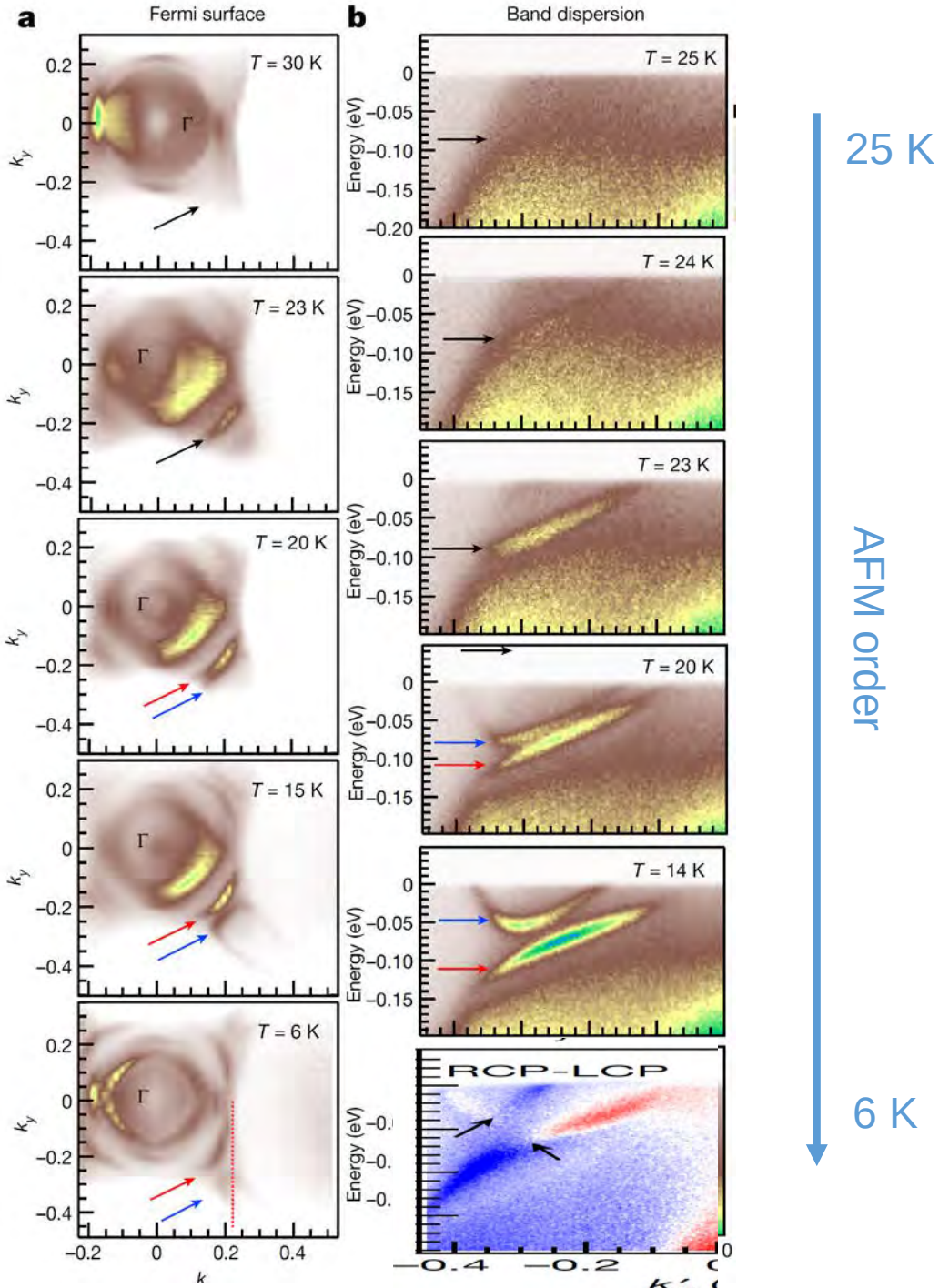
Fermi arcs in NdBi

Usual band splitting (band curvature conserved)



New band splitting (opposite band curvature)





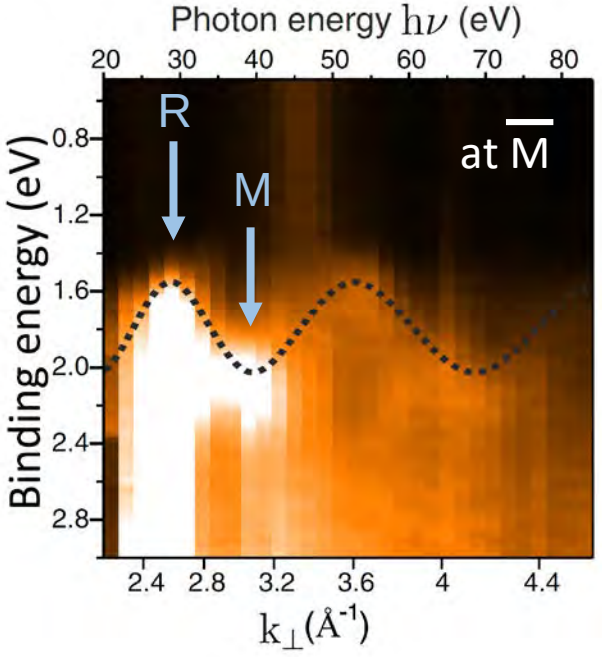
Energy materials: lead halide perovskites

- Tandem-cell efficiency of 30%
- High defect tolerance, low mobility,
  low charge carrier trapping, low recombination rates

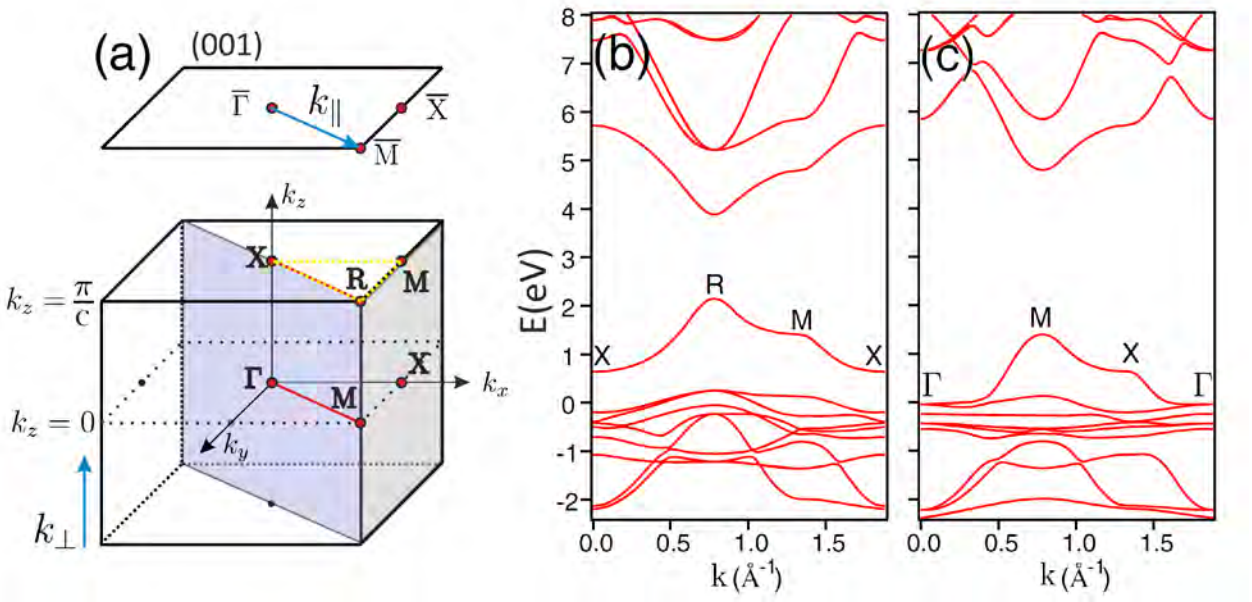
Large polaron formation ?

- Mass enhancement
- Satellites in spectral function

Sajedi et al., Phys. Rev. Lett. **128** (2022) 176405.

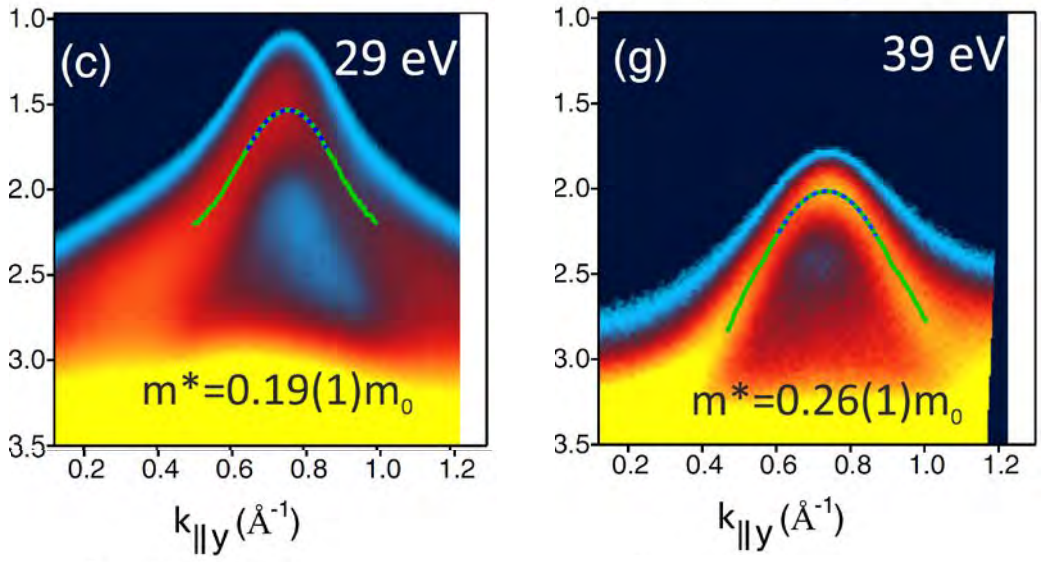


Brillouin zone of $CsPbBr_3$ (DFT calculation)



R - point

M - point



No large polarons in ARPES !

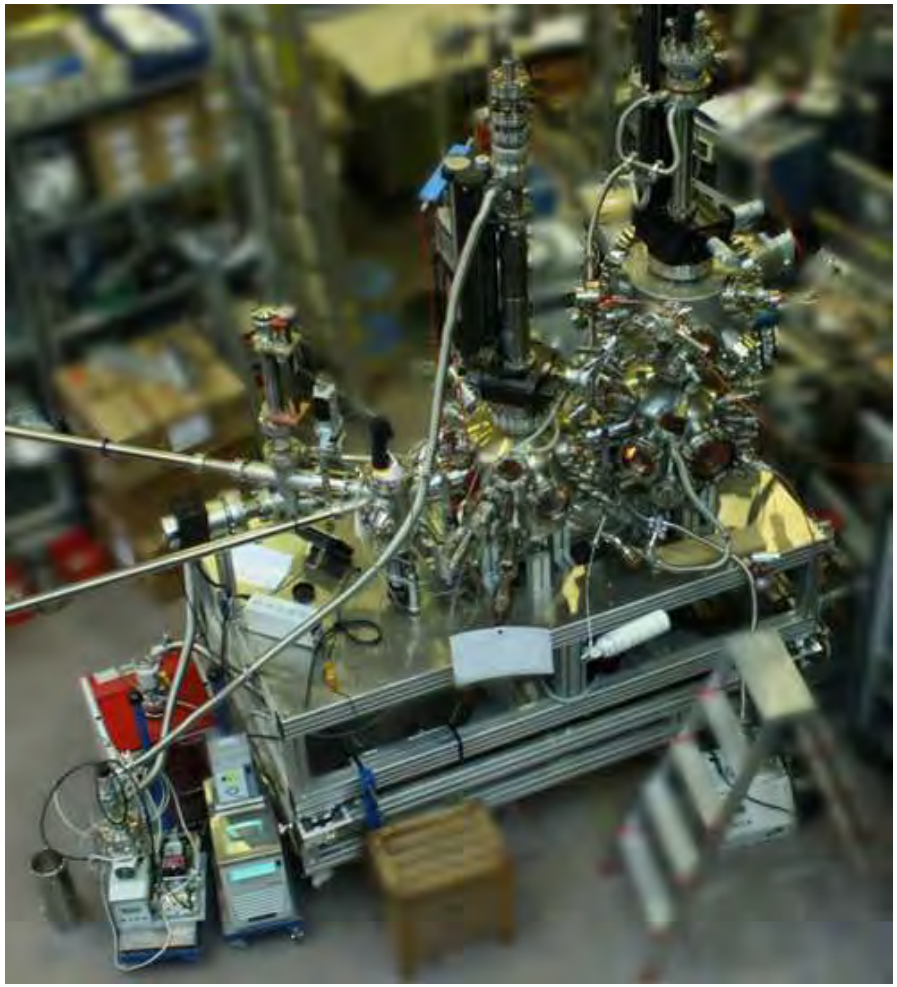
Thanks to Oliver Rader !

&

Thank you for your attention !

3(a)

Dipole beamline: experimental resources and recent highlights



Anna Makarova

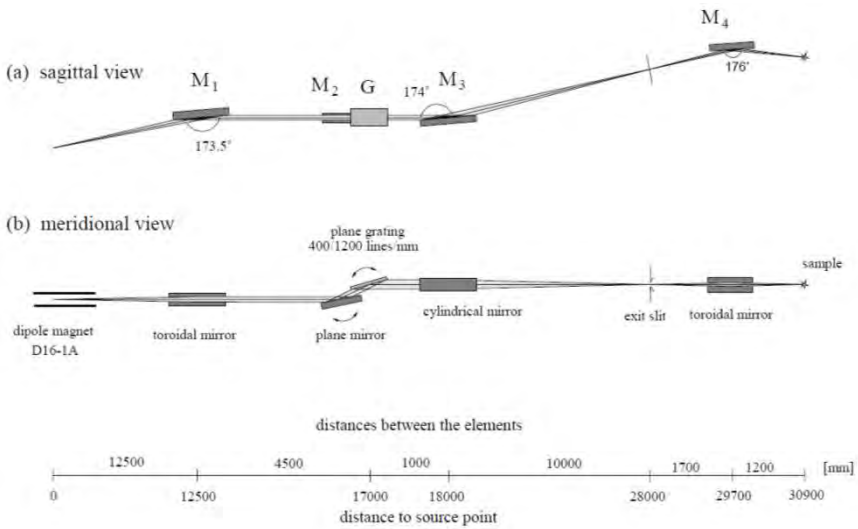
Freie Universität Berlin

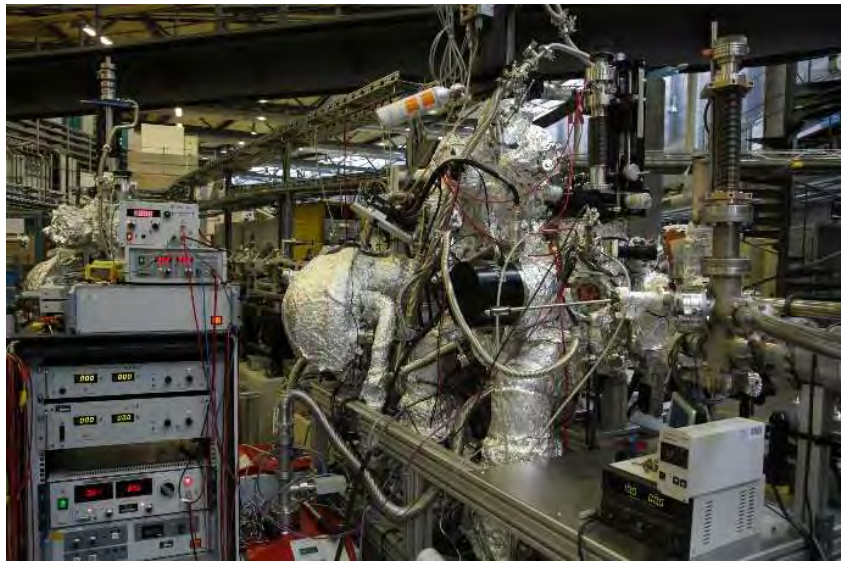
Bundesministerium für Bildung und Forschung

Dipole beamline

Characteristics of the beamline

Energy range	80 - 1500 eV
Flux	up to 10^{11} photons/s/100 mA
Resolving power	up to 11000 at ~400eV
Polarisation	linear
Experimental station	fixed, RGBL-PES

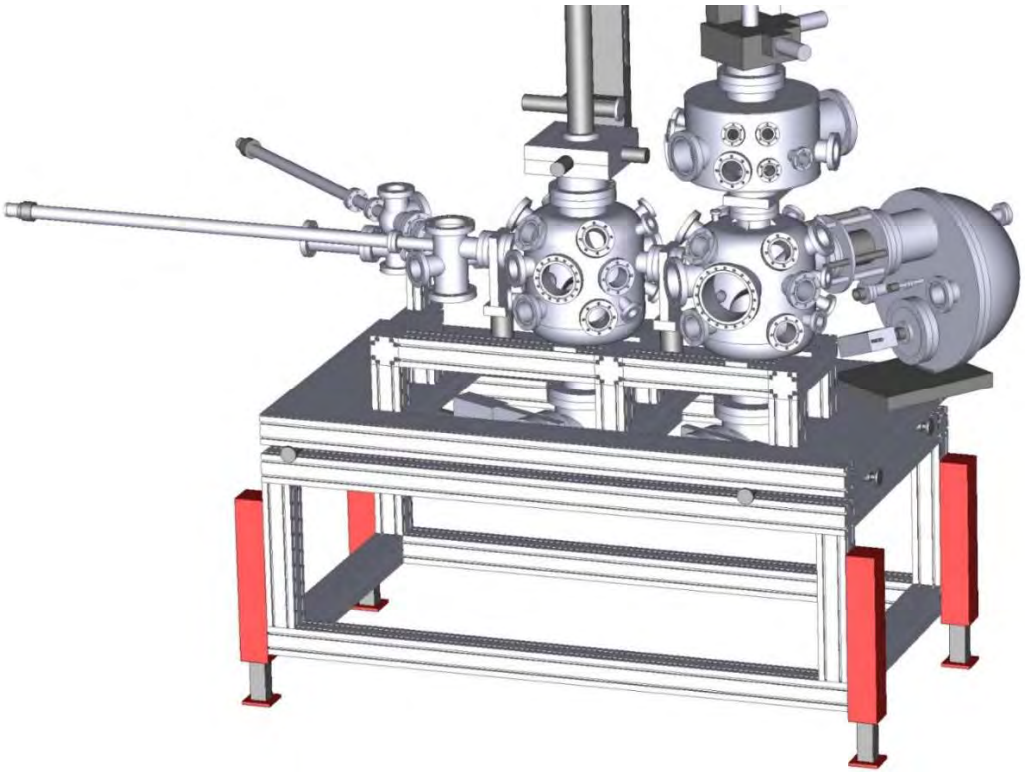




S.I. Fedoseenko et al. / Nuclear Instruments and Methods in Physics Research A 505 (2003) 718–728

Experimental station @ dipole beamline

„Maximum flexibility for user‘s experiments“



Design: D. Marchenko, D, Vyalikh, S. Fedoseenko, C. Laubschat.
Installation and implementation: O. Vilkov

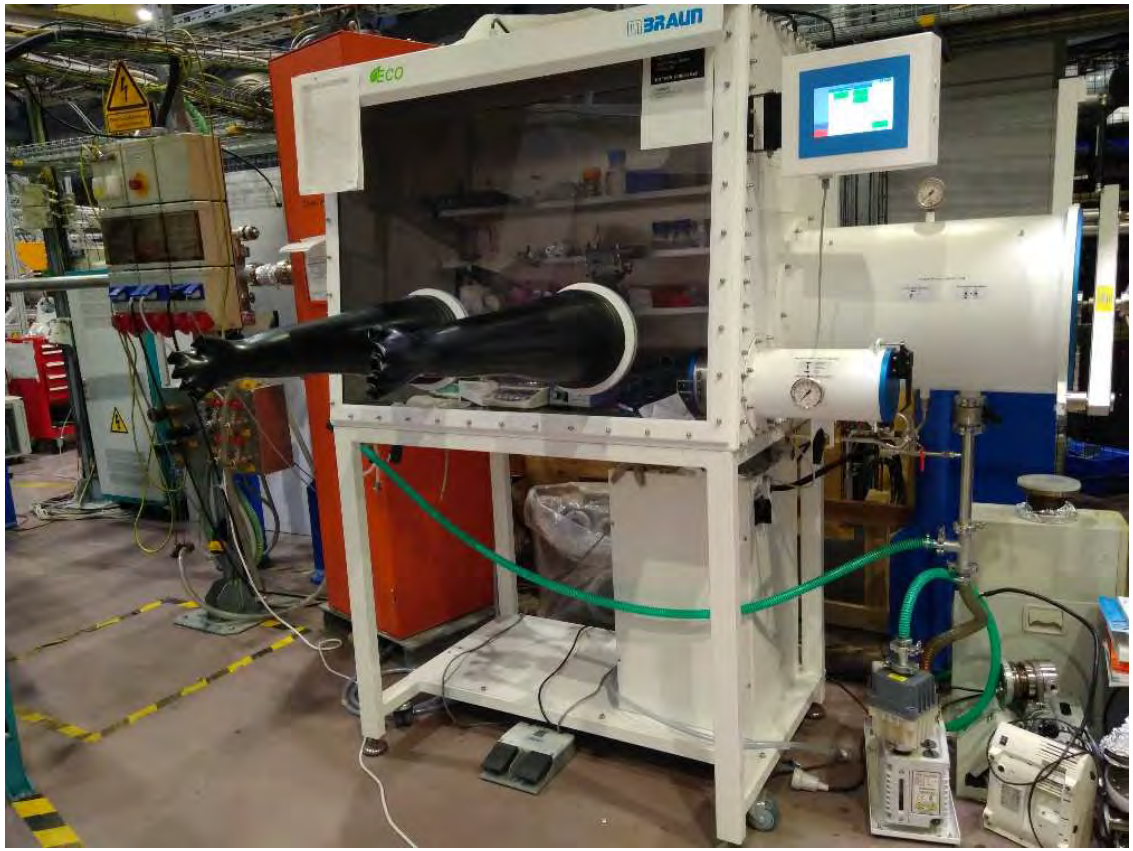
<u>**U**ltra-**H**igh **V**acuum station</u>

- fast-entry load-lock chamber (simultaneous load of up to 8 sample holders)
- 2 preparation chambers:
  - Flash-machine
  - heating stage
  - ion gun
  - evaporators (metals, organics, etc)
  - gas inlet systems,
  - atomic hydrogen source

  etc.

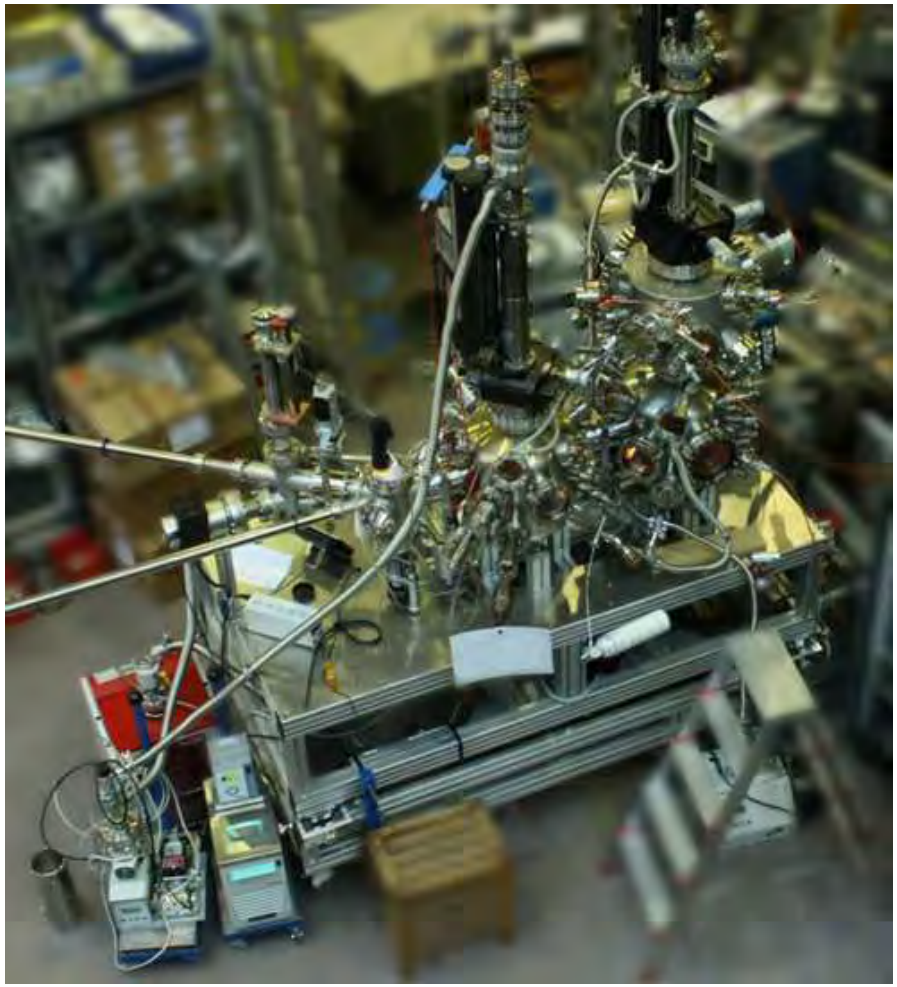
✓ **cleaning of the *ex situ samples***
✓ **synthesis *in situ***
✓ **post-growth modification: study on the interactions with gases, metals, influence of heating etc.**

Sample Environment



Argon glove box allows transfer of the samples to the experimental station without contact with air

Experimental station @ dipole beamline



in operation since 2011

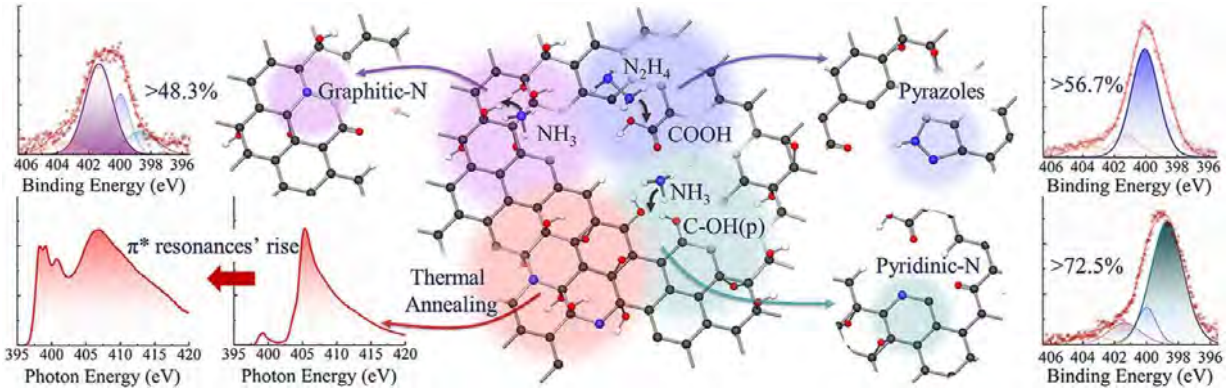
Methods available:

- Photoelectron Spectroscopy (PHOIBOS 150 Electron Energy Analyzer)
- Near Edge X-ray Absorption Fine Structure (NEXAFS):
  - *total electron yield, TEY (ampermeter Keithley)*
  - *partial electron yield, PEY (MCP-detector)*
  - *fluorescence yield, FY (installation of external fluorescence detector possible)*
- Low Energy Electron Diffraction (in analysis and preparation chambers)
- Mass-spectroscopy (in preparation chamber)

Functionalization of 2D materials

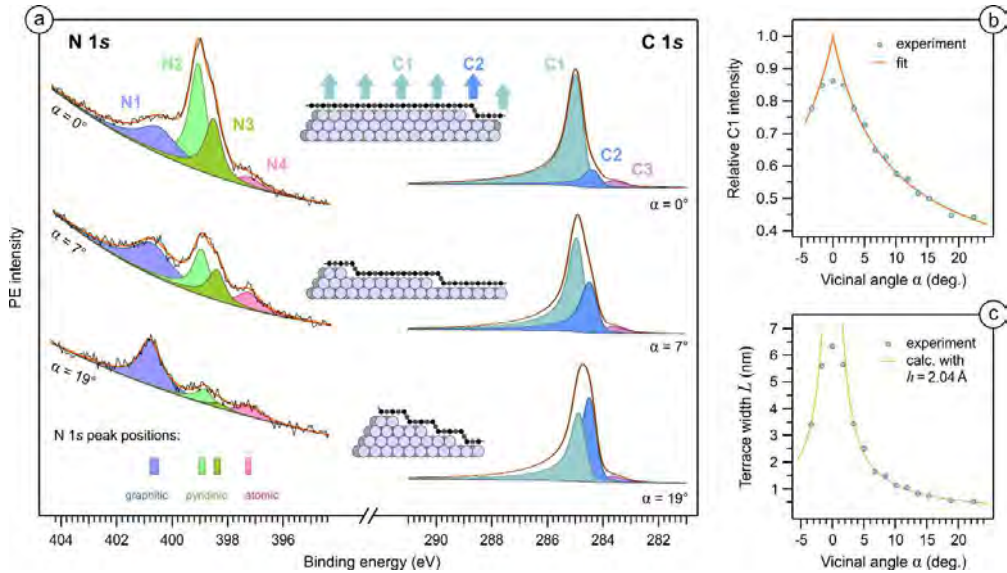
- Doping of graphene with heteroatoms

Modulating nitrogen species via N-doping and post annealing of graphene derivatives: XPS and XAS examination



M. K. Rabchinskii, et al., Carbon 182 (2021) 593-604

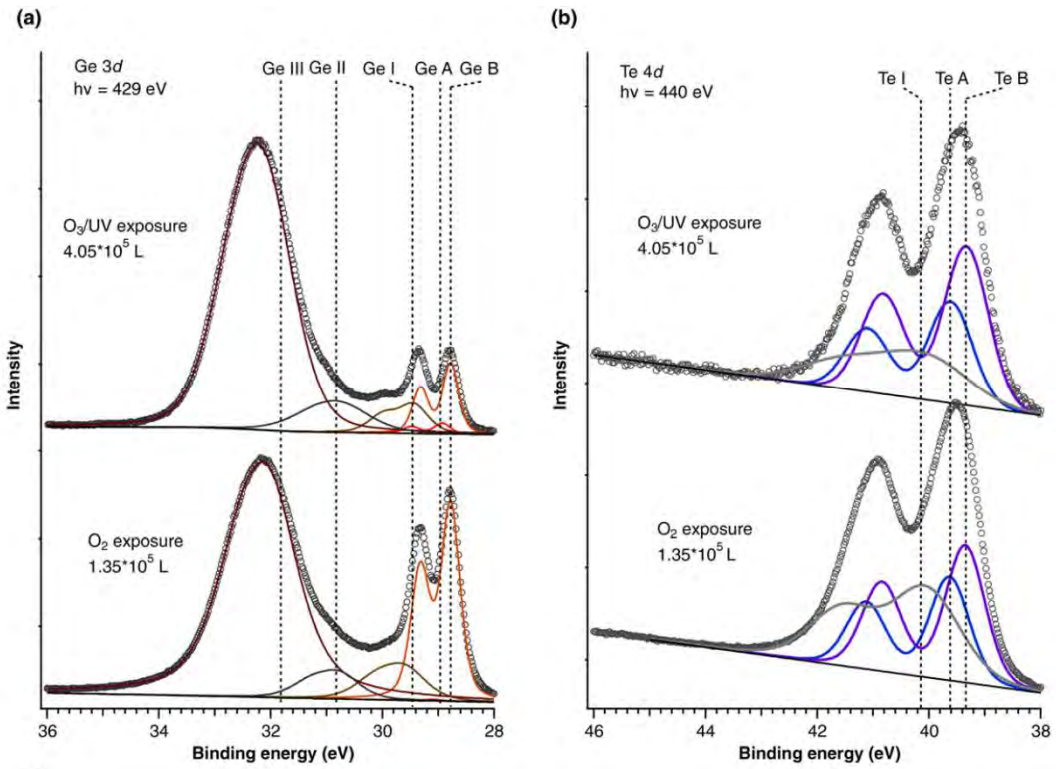
Nitrogen-doped graphene on a curved nickel surface



O. Yu. Vilkov, et al., Carbon 183 (2021) 711-720

Stability of materials

Nanoscale phase separation in the oxide layer at GeTe (111) surfaces



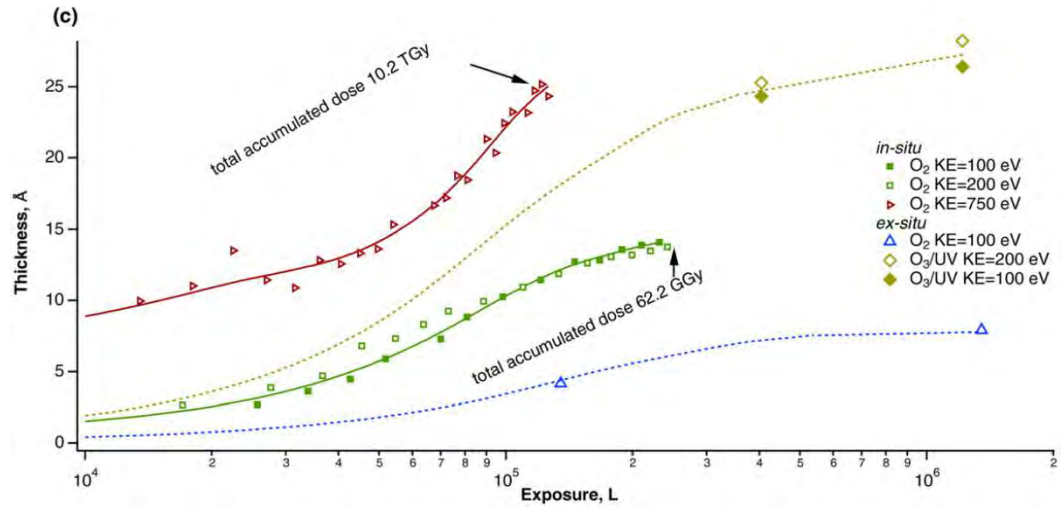
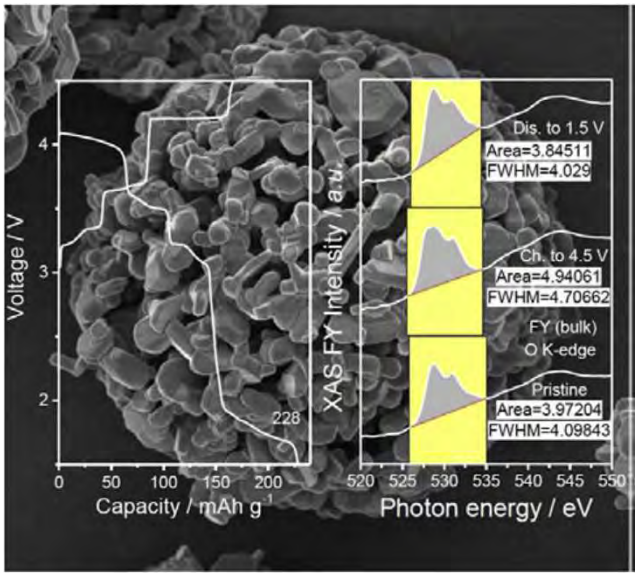


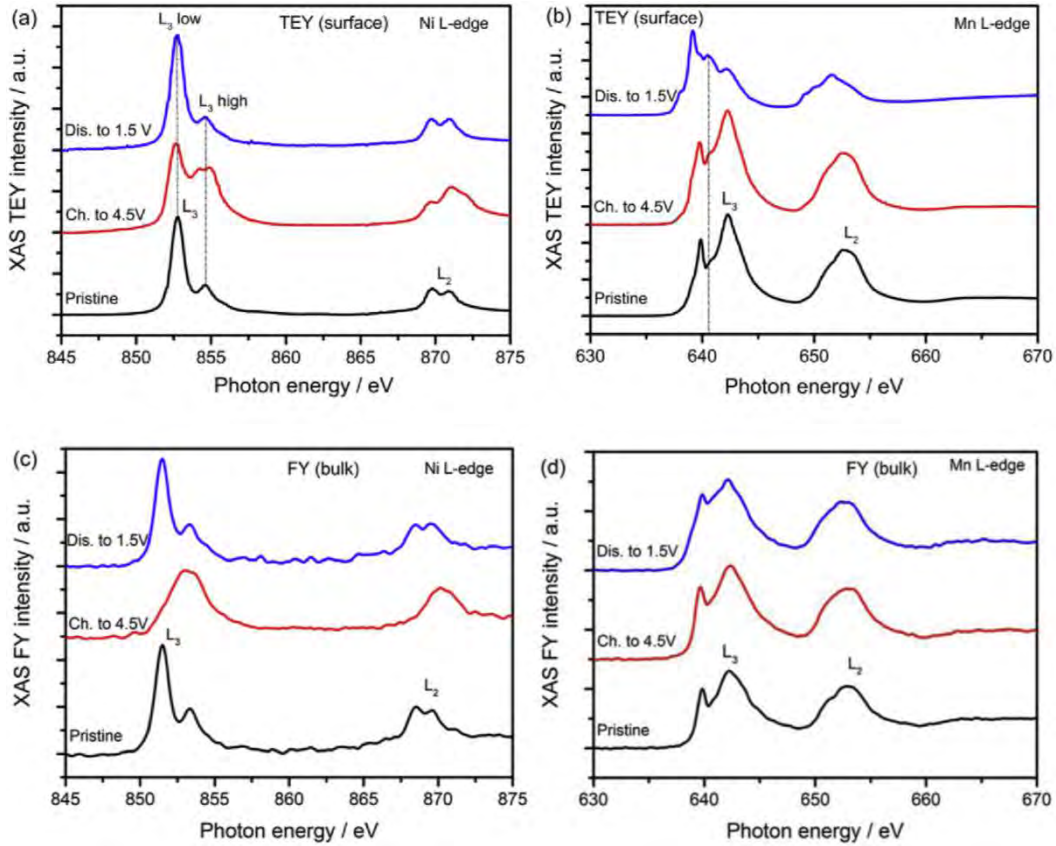
Figure: comparison of the *in situ* and *ex situ* kinetics: (a) and (b) ex situ Ge 3d and Te 4d for a GeTe (111) surface treated with molecular and atomic oxygen; (c) Reaction kinetics for the in situ data obtained at low (green) and high photon flux (brown), ex situ data obtained for molecular oxygen (blue) and in the presence of atomic oxygen (dark yellow).

A. S. Frolov et al. Nanoscale 2022, 14, 12918

Energy storage materials

A high-capacity P2 $Na_{2/3}Ni_{1/3}Mn_{2/3}O_2$ cathode material for sodium ion batteries with oxygen activity



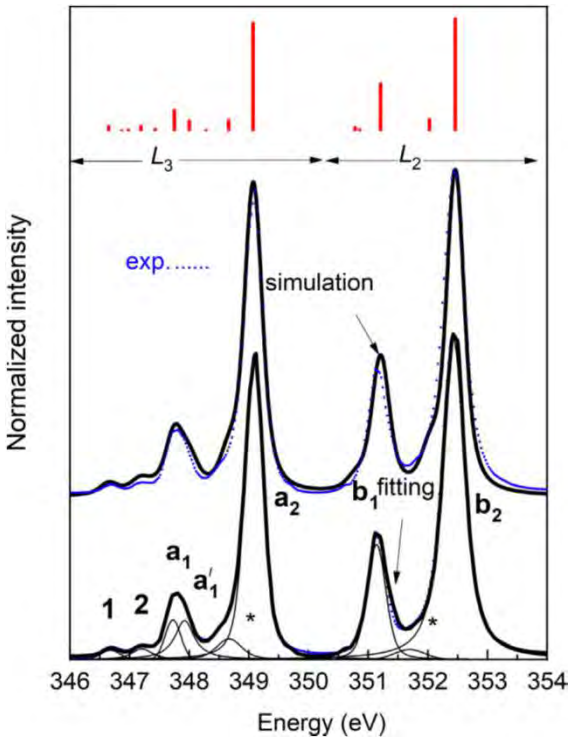


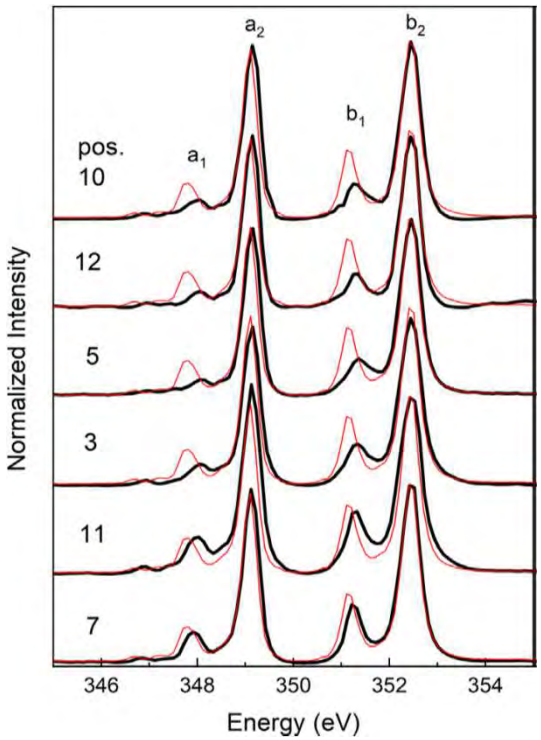
P2-Na_xTMO_2 materials

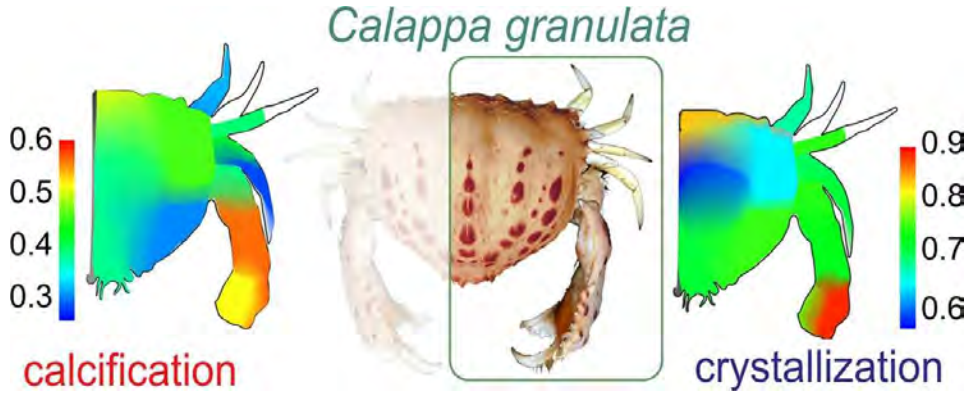
T. Risthaus, et al., Journal of Power Sources 395 (2018) 16

Materials of biological origin

Crystalline and amorphous calcium carbonate as structural components of the *Calappa granulata* exoskeleton







Left: Fitting and simulation of the Ca L3,2-edge NEXAFS spectrum of a crystalline calcite sample. Right: Ca L3,2-edge NEXAFS spectra recorded from various parts of the exoskeleton – black, compared with the spectrum of a calcite reference sample - red.

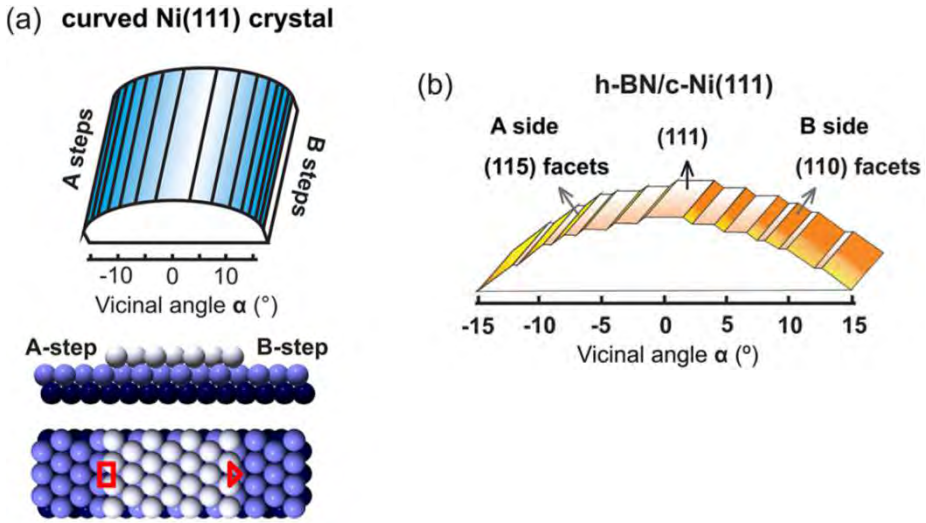
M. Katsikini et al., Journal of Structural Biology, 211, 3, 2020

Example of a complete study

Study on the interaction of h-BN grown on c-Ni(111) with molecular oxygen

Step1. Synthesis

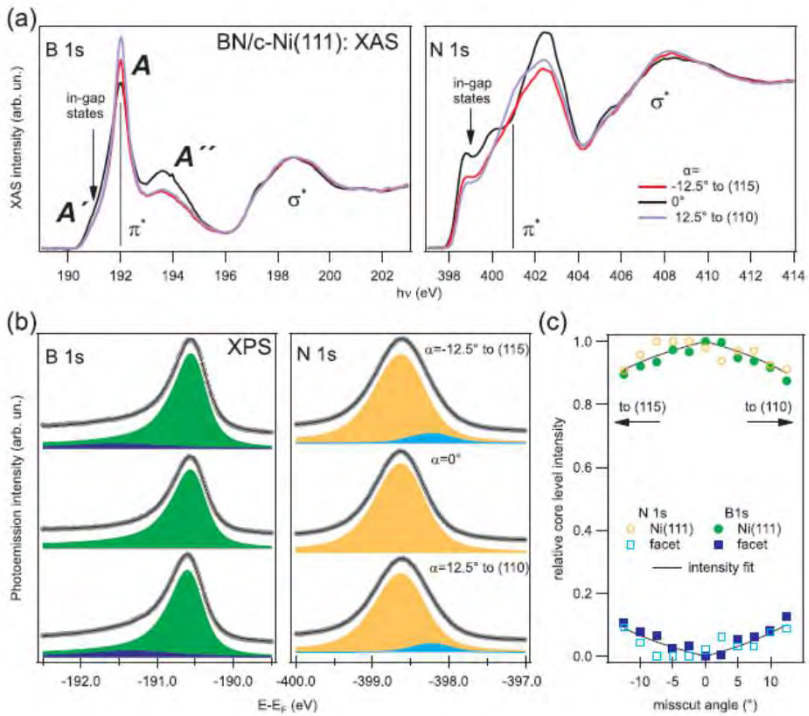
h-BN growth: CVD synthesis at 2×10^{-7} mbar of borazine ($B_3N_3H_6$) at 750°C for 12 min on c-Ni(111) surface



L. Fernandez et al, 2D Mater. 6 (2019) 025013

A. Makarova et al., JPCC 123 (2019) 593-602

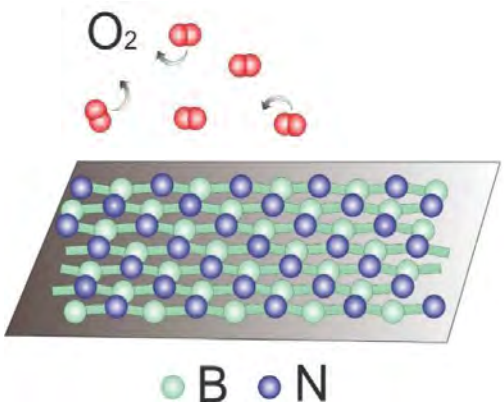
Step 2. Characterization

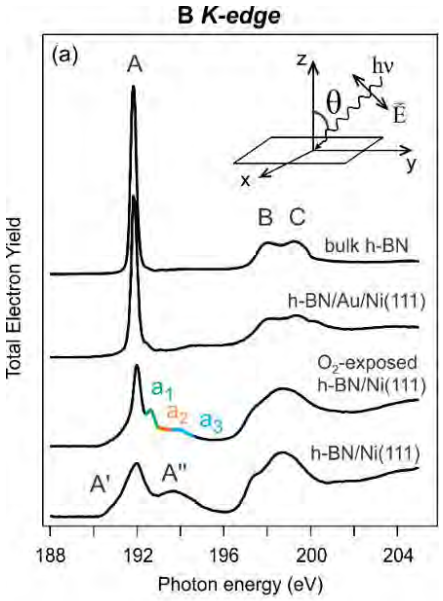


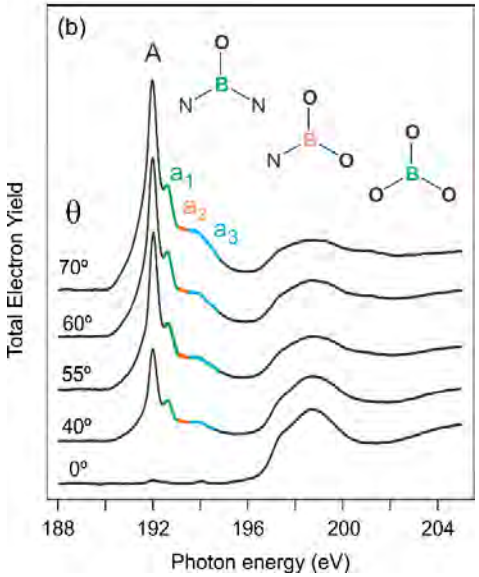
Example of a complete study

Step 3. Sample treatment

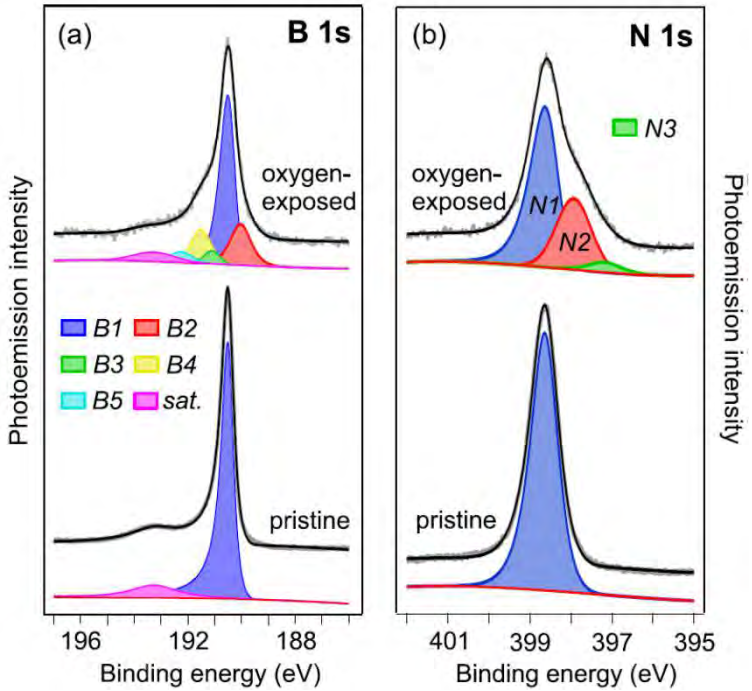
Thermal oxygen exposure: 10^{-5} mbar of molecular oxygen at 300°C for 10 min







Step 4. Further characterization



A. Makarova et al., JPCC 123 (2019) 593-602

Future capabilities

New Manipulator for *in operando* and *in situ* studies

Key features:

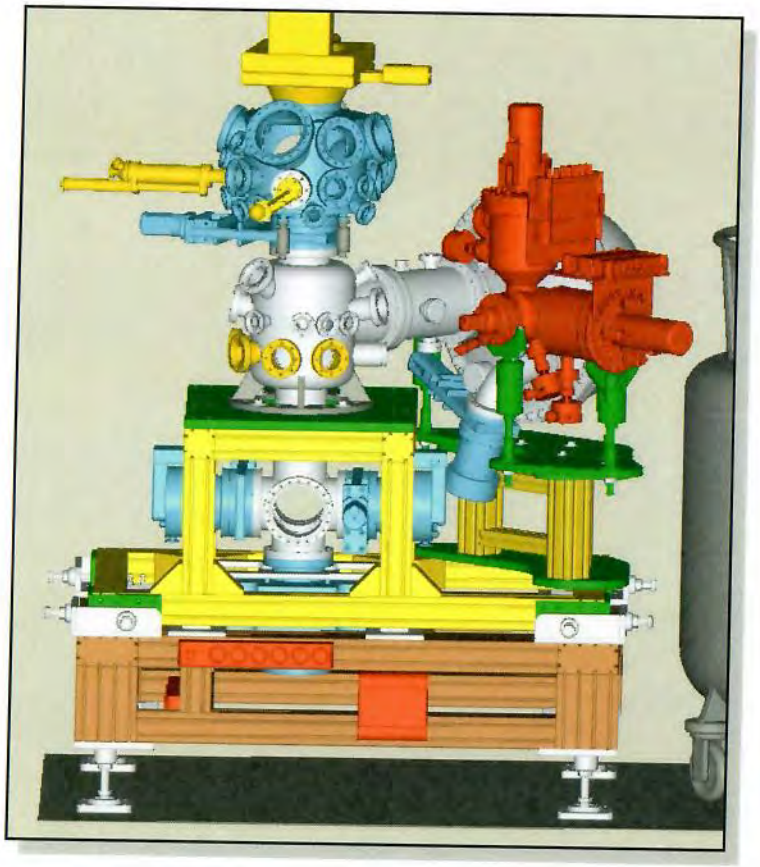
- wide temperature range 100-1000K → temperature programmed XPS and XAS:
  - ✓ temperature-dependent physicochemical processes in 2D materials (doping, recrystallization, intercalation, etc.)
  - ✓ self-organization of low-dimensional systems
  - ✓ phase transitions
  - ✓ interface formation
  - ✓ thermal stability
  - ✓ chemical reactions on the surfaces, etc.
- electrical contacts on manipulator
  - ✓ connection of potentiostat – electrochemistry, all-solid-state batteries
  - ✓ gas sensing devices
  - ✓ electric field induced phenomena (e.g. superconductivity)

3(b)

Spin- and angle-resolved photoemission

➢ Spin-ARPES Endstation

- Preparation chamber
  - e^--beam evaporators
  - Noble-gas sputter gun
  - Heating stage up to 2000°C
  - precise heating stage up to 500°C
  - Coil for pulses up to 0.5 T in 3D
  - Load lock



Staff scientist : **J. Sánchez-Barriga** (*HZB*)
Technical design by **A. Varykhalov** (*HZB*)

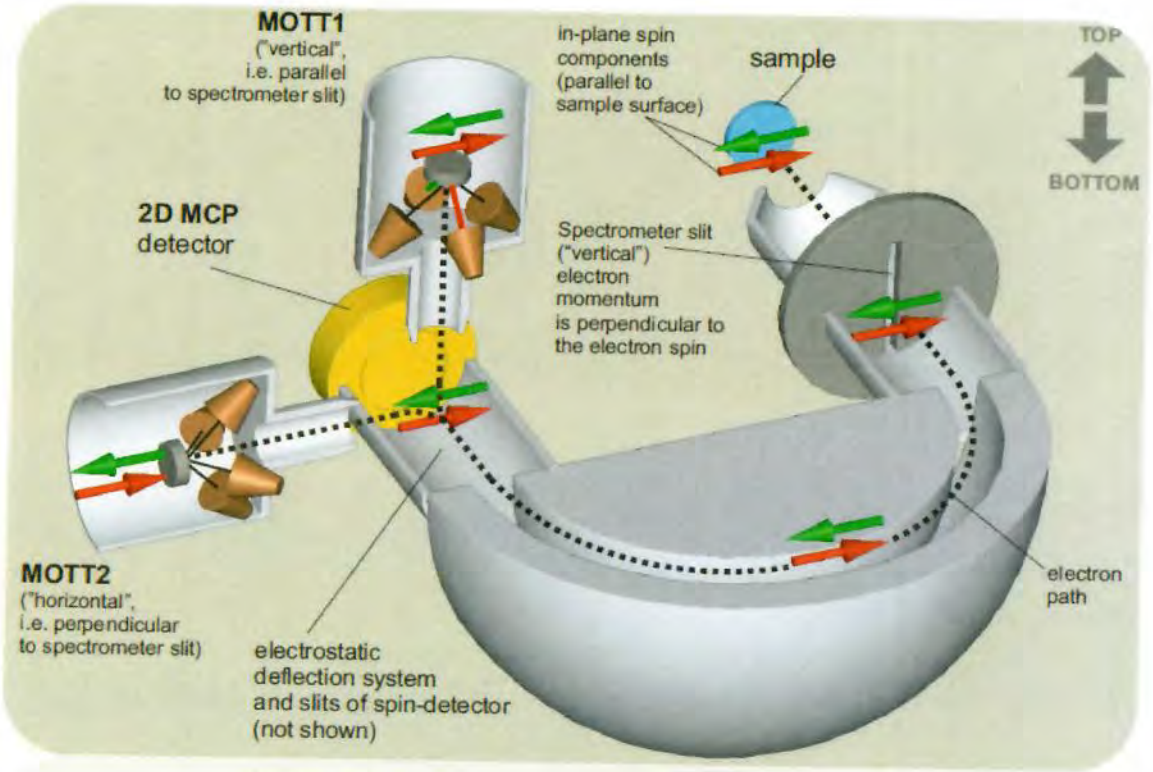
Construction & Commissioning: **J. Sánchez-Barriga, A. Varykhalov and D. Marchenko** (*HZB*)

Funding BMBF 05K100 DC (**C. Laubschat, D. Vyalikh**, TU Dresden)

➢ Spin-ARPES Endstation

- SCIENTA R8000 hemispherical analyzer
- combined 2D MCP and two Mott-type spin detectors
- $E(\underline{k}, \sigma_x, \sigma_y, \sigma_z)$
- Angle/energy resolution 0.3°, 10 meV
- Angle/energy resolution 0.8°, 30 meV in spin-resolved mode
- Six-axes manipulator, T = 35-40 K to 300 K
- Automatic sample positioning for band structure measurements
- Omicron sample plates
- Analysis chamber
  - In-situ cleaveage stage
  - Port for UHV suitcase

Spin- and angle-resolved photoemission



Spin- and angle-resolved photoemission

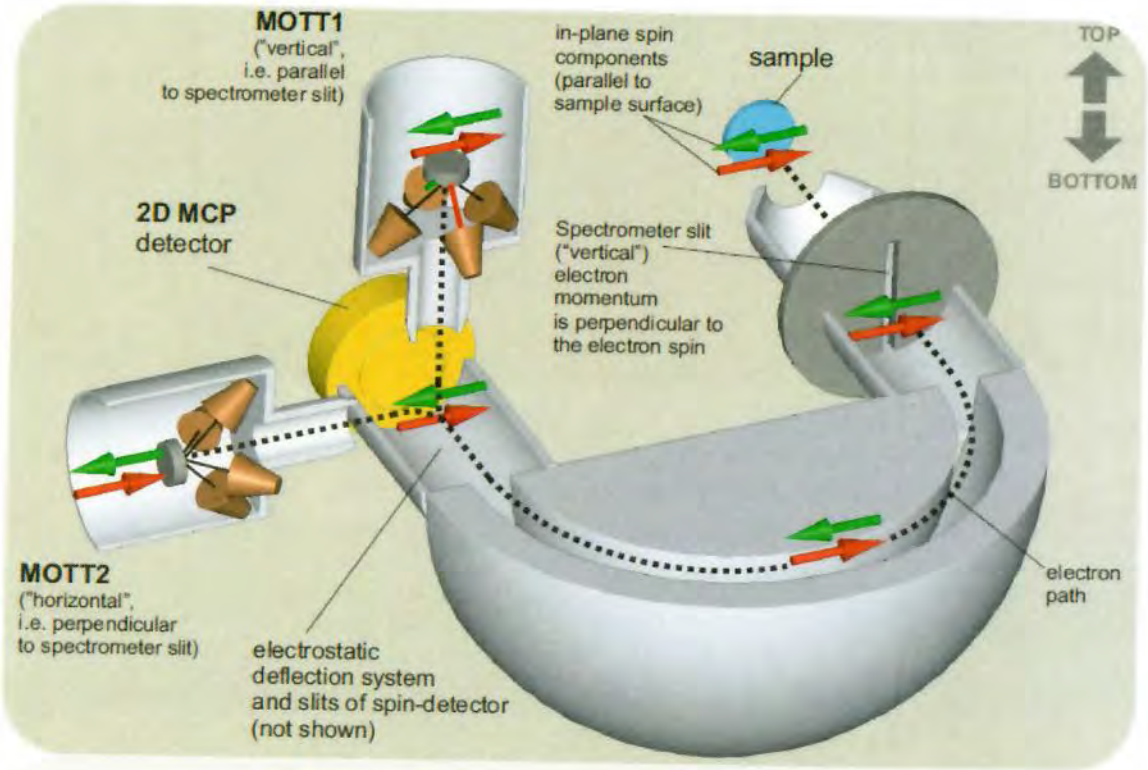
➢ Undulator beamline U125_PGM

- Quasiperiodic undulator
- 2 gratings (600 l/mm and 1000 l/mm)
- $h\nu$ = 15 - 200 eV (optimal range < 100 eV)
- ΔE < 1 meV for $h\nu$ < 100 eV
- linear horizontally polarized (fixed)
- Beamspot: 68 µm x 25 µm
- photon flux: > 10^{13} Photons/s/0.1%BW/100mA

Spin- and angle-resolved photoemission

➢ Spin-ARPES Endstation

- SCIENTA R8000 hemispherical analyzer
- combined 2D MCP and two Mott-type spin detectors
- $E(\underline{k}, \sigma_x, \sigma_y, \sigma_z)$
- Angle/energy resolution 0.3°, 10 meV
- Angle/energy resolution 0.8°, 30 meV in spin-resolved mode
- Six-axes manipulator, T = 35-40 K to 300 K
- Automatic sample positioning for band structure measurements
- Omicron sample plates
- Analysis chamber
  - In-situ cleaveage stage
  - Port for UHV suitcase



Spin- and angle-resolved photoemission

➢ Undulator beamline U125_PGM

- Quasiperiodic undulator
- 2 gratings (600 l/mm and 1000 l/mm)
- $h\nu$ = 15 - 200 eV (optimal range < 100 eV)
- ΔE < 1 meV for $h\nu$ < 100 eV
- linear horizontally polarized (fixed)
- Beamspot: 68 µm x 25 µm
- photon flux: > 10^{13} Photons/s/0.1%BW/100mA

4.

HZB BESSY II Light Source

HOW TO WRITE A GOOD PROPOSAL...

WELCOME TO BESSY II

48 beamlines

38 in user service

(27 simultanously)

Advantages of synchrotron radiation

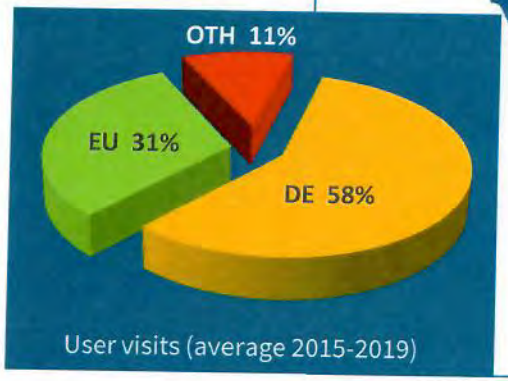
- Broad spectrum
- High flux / brilliance / stability
- Polarization - both linear and circular
- Pulsed time structure (down to tenths of ps)

THE WORLD @ BESSY II

Proposal deadlines:
1 March
and
1 September

70% of beamtime available to external user groups

(peer review process)



On average per year at BESSY II

1200 submitted proposals, 800 executed BT campaigns, 2700 user visits, 500 certified publications

Beamtime is free of charge!

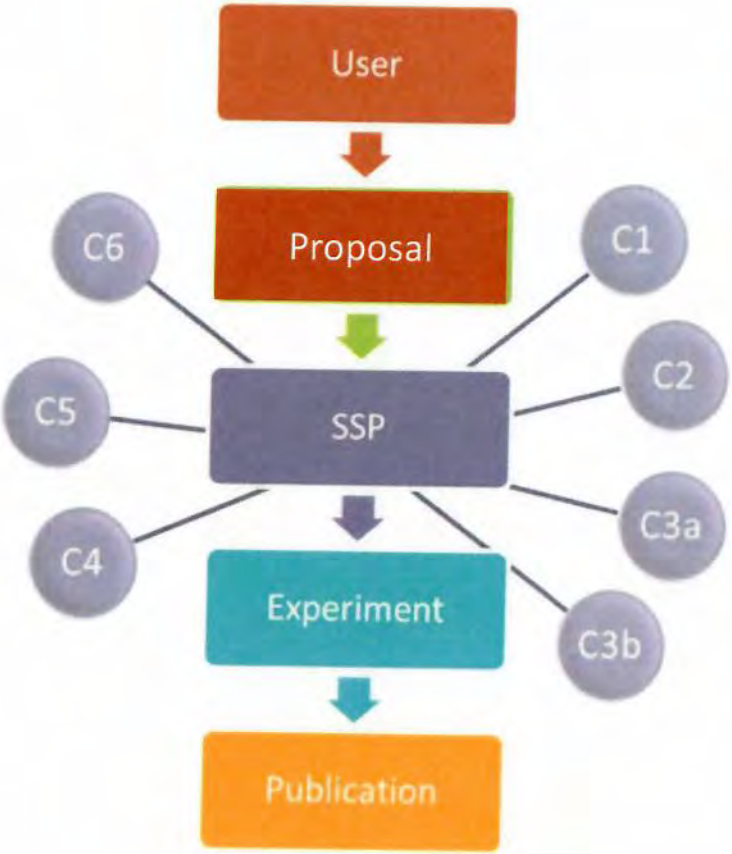
under the condition that

- proposer aggrees to open science (HZB data policy) (*public access to data after 5 years embargo)*
- results are published in international journals in good time
- use of HZB facilities is properly mentioned in the paper

HZB would really appreciate if the local contact is part of the authors list
(at least the local contact should be mentioned by name in the acknowledgements)

Industrial applicants or users not agreeing to open science **have to pay for the use of HZB facilities**


HZB GATE
Oracle Database
IGAMA
Usertool
Calendar
Referee tools
Admintool
Database Access



User

Proposals

Preparation of the Evaluation

Evaluation Process

Allocation and Scheduling

Feedbacks

FROM IDEA TO PROPOSAL

- **The good idea**

  new science, new systems, new questions to old systems, …

  Beware of stamp collections

- **Find the right beamline**

  Starting point – the webpage of the HZB User Coordination: **hz-b.de/user**

THE RIGHT BEAMLINE

BESSY II Experimental Stations

full operation
limited operation
Test/Inhouse
under construction

Station		Remote Access	Methods	Beamline(s)	Contact
ALICE	Diffractometer/reflectometer for resonant magnetic x-ray scattering applications	depends on experiment - please discuss with Instrument Scientist	XMCD, NEXAFS, EXAFS, XPS	UE56-2_PGM-2, UE112_PGM-1, UE52_SGM, U49-2_PGM-1, PM3	Radu Abrudan
ARPES One-Cube	1^3-ARPES ultra high resolution photoemission station	depends on experiment - please discuss with Instrument Scientist	UPS, ARPES, XPS	UE112_PGM-2b-1^3	Emile Rienks Alexander Fedorov
ARPES One-Square	1^2 ARPES ultra high resolution photoemission station	not possible	XPS, UPS, ARPES	UE112_PGM-2a-1^2	Andrei Varykhalov

ARPES One-Square

1^2 ARPES ultra high resolution photoemission station

ARPES One-Square is dedicated to high-resolution angle-resolved photoelectron spectroscopy. It is equipped with a very high resolution electron energy analyzer and attached to a beamline providing photons in the energy range from 10 – 250 eV with high resolution and full control over the polarization. A precise 6-axes cryomanipulator combined with an electron analyzer Scienta R8000, operated in parallel angular detection mode, allows for accurate band structure mapping. The versatility of the system is extended by exerting a very broad spectrum of in-situ sample treatment techniques.

Selected Applications:

- 2D materials and graphene
- Topological insulators, Weyl semimetals, Dirac nodal line materials
- Metallic single crystals with flat and vicinal surfaces
- High-temperature superconductors
- Metal halide perovskites (inorganic)

Model of ARPES One-Square

Instrument Scientist

Dr. Andrei Varykhalov

Instrument Scientist

Dr. Maxim Krivenkov

UE112_PGM-2a-1^2

UE112_PGM-2a-1^2

Station data	
Temperature range	Measurement: 15–400K, Preparation up to 2300K
Pressure range	1E-10 mbar
More details	ARPES One-Square
Beamline data	
Segment	H13
Location (Pillar)	14.2
Source	UE112 (Elliptical Undulator)
Monochromator	PGM 2

Instrument Scientist

Dr. Andrei Varykhalov

Instrument Scientist

Dr. Maxim Krivenkov

Publication list

Reference

All publications based on experiments using UE112_PGM-2a-1^2 should cite the following reference.

YOU CAN ADD ADDITIONAL INFRASTRUCTURE

User Lab Cluster

HZB hosts a number of state-of-the-art on-site laboratories and user facilities. All these research facilities are designed to serve researchers from universities, foreign research institutions and industry.

Prerequisite for the use of the User Lab Cluster is an application for beamtime at the synchrotron radiation source BESSY II.

User Labs@HZB Wannsee

- DEGAS - GasLab
- 3D Data Analytics Lab
- Micro-CT Lab
- Crystallography Lab (preparation and characterization)
- Bulk Properties Lab (former LaMMB)

CoreLabs@HZB

- X-Ray CoreLab
- CCMS (Correlative Microscopy and Spectroscopy)
- CoreLab QM (Quantum Materials)
- EMIL (Energy Materials In-Situ Laboratory Berlin)

User Labs@HZB Adlershof

- MX BioLab
- Chemistry Lab

Additional User Facilities@HZB Adlershof

- Mechanical Workshop
- Sputter Lab

Sample Environment Infrastructure at BESSY-II

The listed infrastructures can be booked by all BESSY-II users and beamline managers.

Name and Description		Contact
Nitrogen-Glovebox (ENERGIZE) Specifications	Glovebox with integrated spin coater	Bastian Klemke Matthias Neeb
Argon-Glovebox (KMC-2) Specifications	Glovebox for dry and oxygenfree sample preparations	Dirk Wallacher Nico Grimm
UV-VIS 2700	UV-VIS Spectrophotometer Shimadzu UV-2700	Anna-Marie Runge
Differential pumping stage	differential pumping stage	Matthias Neeb
Nitrogen Generator	Nitrogen generator PARKER NitroFlow Lab	Dirk Wallacher Nico Grimm
Drop Shape Analyzer	Drop shape analyzer KRÜSS DSA25 for wetting investigations	Beatria-Kamelia Menzel Tristan Petit Anna-Marie Runge Matthias Neeb Bastian Klemke

Beamtime applications (BESSY , CoreLabs, VIPERlab...) via the general access tool GATE
A guide for beamtime application is available online

Talk to us

Do not hesitate to contact us if you need help !

- **User Coordination** – helps to find the right partners for the good ideas — **photons@helmholtz-berlin.de**
- **BL scientists** – clarify what is possible at the beamline — **hz-b.de/instruments**
- **Sample Environment** – helps to make things possible — **hz-b.de/samenv**

Talking before writing ensures that the proposed exeperiment is feasible

GATE (HZB)

PREPARING EXPERIMENT TIME
Guest visits (BEObern, PTB, Lab@emsh)
Guest visits - handbook

AFTER THE EXPERIMENT TIME
Submit experiment time feedback
Submit report

MY PROPOSAL
New proposal
Proposal review feedback
Proposal list
Publication list

REFEREE SECTION
Review proposals
Review proposals offline
Proposals per call
Proposals
Reports
Referee handbook
Your fields of expertise

PERSONAL SECTION
Personal data
Change password
Change username
Online warnings
Disable / delete account
KFS

HANDBOOK

LOGOUT

GATE ADMIN

GATE (HZB) - Antje Vollmer

New Proposal

Choose the HZB research facility to submit a new proposal

- BESSY II
- Macromolecular Crystallography
- X-Ray CoreLab

Behind: Abstract, scientific case, experimental plan, safety, funding and more

BESSY II

HZB has decided to drop the unrevised resubmission of unchanged proposals completely. Resubmissions are no longer possible.

Applications for experiment time using the facilities of HZB

- BESSY II
- EMIL

Submission closed. Deadline for round 2022 / 1 was 05 September 2021

Macromolecular Crystallography

Applications for experiment time using the MX-beamlines@BESSY II

Submission closed. Deadline for round 2022 / 1 was 05 September 2021

X-Ray CoreLab

Applications for experiment time using the different X-Ray diffraction methods at HZB location Adlershof (WCRC).

- start submission
- new proposal from XML

https://www.helmholtz-berlin.de/user/apply-for-beamtime/index_en.html

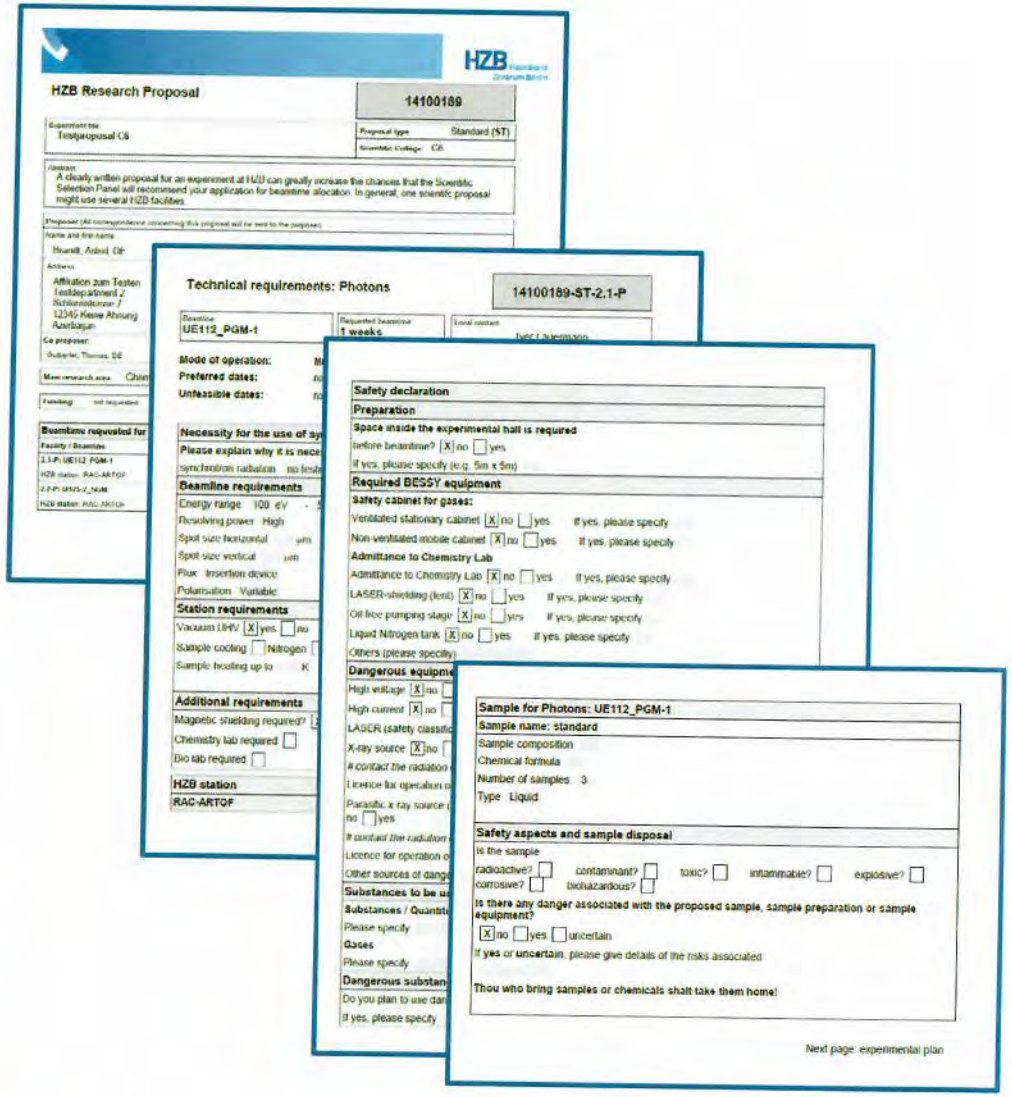
HZB BESSY II Light Source

THE PROPOSAL

A clearly written proposal can greatly increase the chance that the Scientific Selection Panel will recommend the application for beamtime allocation.

Proposal submission: **hz-b.de/gate**

- **Abstract**
- **Scientific part**
- **Previous results**
- **Necessity for the use of synchrotron radiation**
- **Expected results**
- **Technical part**
- **Safety declaration**
- **Sample declaration**
- **Experimental plan**



ABSTRACT AND SCIENTIFIC CASE

Abstract (max. 1000 characters)
A short but meaningful summary of the proposed experiment.

Scientific case *(written in English, no longer than two A4 pages; readable font-size, readable figures !!!)*

- **Scientific context**
  You should give a clear account of the aims of the experiment and set it within the broader scientific context. Keep in mind that not all review panel members are experts in the respective scientific field.
- **Choice of specific instrument/station**
  Give reasons for your choice of instrument. Justify why you need to use this particular instrument and why HZB is important.
- **Preliminary work**
  If possible, give results of preliminary work carried out (e.g. NMR, other scattering methods, magnetic properties, ...) in support of your proposed experiment and to demonstrate sample quality.
- **Results expected**
  Describe the expected results and how you intend to treat the data.
- **References**
  List the 5 most important publications in this field of science in order to show that you are aware of the work of others.

PREVIOUS RESULTS / NECESSITY FOR THE USE OF SYNCHROTRON RADIATION

Previous results

- **Publications** based on former HZB experiments (if available)
- **Reports** on former HZB experiments related to the new one

Necessity for the use of synchrotron radiation

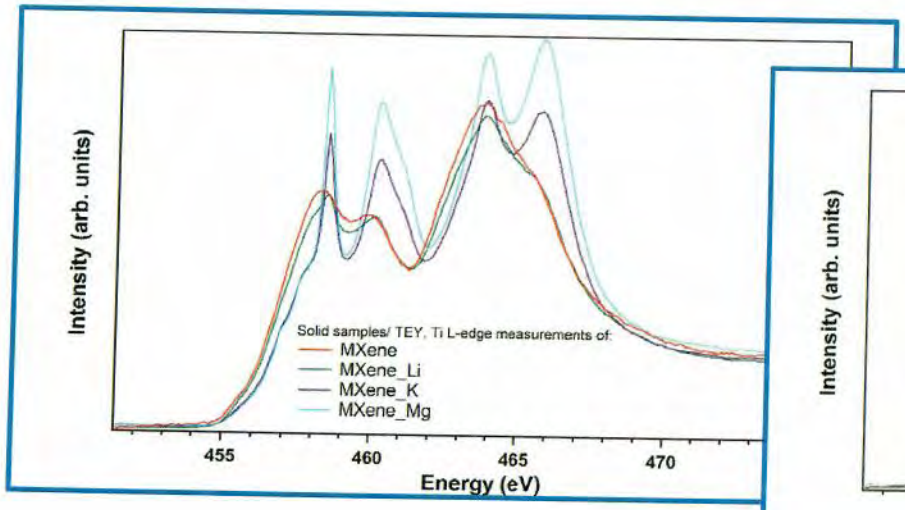
Explain why synchrotron radiation is needed, i.e. why laboratory methods are not sufficient

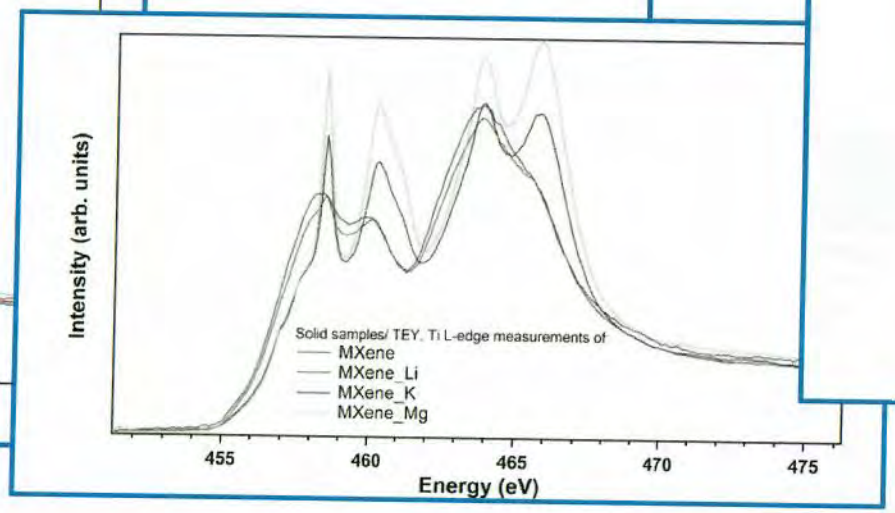
Expected results

Describe which results you expect from the experiment and how you will treat/evaluate the data

THE SCIENTIFIC CASE – GONE WRONG

- Proof read your proposal text – too many typos annoy the referees
- Do not shrink the font size to increase the amount of content on the two allowed pages
- Do not be too brief either
- Figures – make sure they are decipherable for the referee. Do not rely on colours !





Detailed experimental plan

The experimental plan should justify the amount of requested beamtime.

Please state

- number of samples
- number of measuring points per sample
- sample conditions (magnetic fields, temperatures, any number of combination of the before mentioned, intended changes in sample environment, …)

Do not forget to account for sample preparation, sample change, condition change etc.

For a complex series of experiments, please show a breakdown of how you calculated your final beamtime request.
Example: 2 samples at 3 pressures and 4 temperatures for 5 hours each = 2 x 3 x 4 x 5 = 120 hours of beamtime

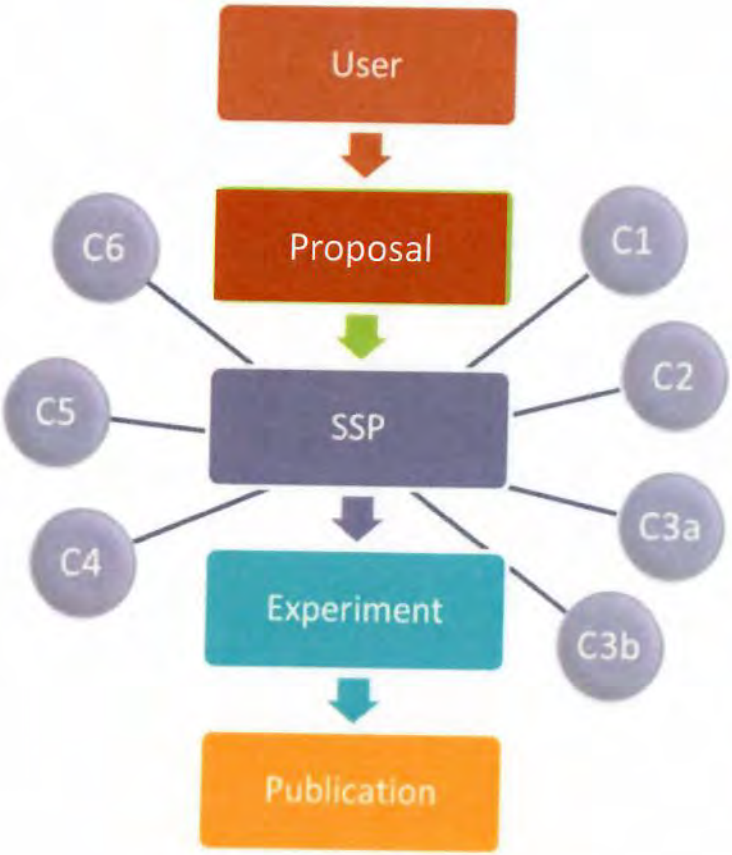
Ask the sample environment group or the instrument scientist/station manager for help if needed.

CONCLUSION

!!! Talk to us !!!

- **User Coordination : photons@helmholtz-berlin.de**
- **BL scientists: hz-b.de/instruments**
- **Sample Environment: hz-b.de/samenv**

We are happy to help you!



User

Proposals

Preparation of the Evaluation

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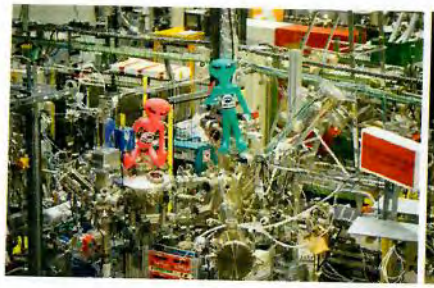
Feedbacks

After the evaluation by an international panel, you will be informed whether beamtime is granted




6 month later

Hurrah, it's beamtime!

Stopp, sorry not yet… don't forget your:
user registration, access cards, safety instructions,
guesthouse reservation, …

Now it's beamtime ☺



Thank you for your kind attention

https://www.lindau-nobel.org/a-driving-force-towards-peace-scientific-cooperation/ Picture: iStock.com/Rawpixel Ltd

6. Future Prospects of GELEM

TO DO:

- Establish a new Eastern European User Community using the Laboratory for Energy Materials Research at BESSY II

Will be an essential prerequisite to:

- Allocate / renew funding for instrumental support (German ministry, BESSY)
- Allocate funding for beamline development (German ministry, calls)
- Organize travel support (EU - pending)
- ➢ Continuation of successful beamtimes and excellent publication record

PROSPECTS:

- Establish and be part of an international steering committee
- Dedicated share of beamtime at GELEM including independent evaluation of beamtime proposals
- Meetings and scientific exchange of GELEM members
- Establish new collaborations and common beamtimes
- State-of-the-art instruments / dedicated user support
- GELEM at BESSY III …

8. Summary and Conclusions

- This was the GELEM Kick-Off Meeting providing first information
- Subsequent meetings of this newly shaped community can be scheduled upon request
- Virtual seminars can be organized
- We request your strong user proposals for the use of GELEM
- Beyond: YOUR Support for being able to host user groups so that support can be requested by the German BMBF
  We need from the user community evidence for a strategic need for such support expressed by LoI
  (needed latest by Fall 2023)
- In summary: We look forward to strong user proposals for GELEM and expect competition for the limited beam time and a high scientific productivity of the best proposals AND dedication of groups who want to do more, i.e, expressing Letters of Intent for contributing to the future as being partner in GELEM so that we can develop GELEM as successful as possible.