







Kick-Off-Meeting

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

July 07, 2023 - 14:00 (CEST)

Agenda

- 1. Welcome
- 2. Research Opportunities at the GELEM Laboratory
- 3. Overview on the Experimental Resources and Research Highlights

(a) Dipole Beamline

(b) Undulator Beamline

- 4. Beam Time Proposals at BESSY II and their Evaluation
- 5. Practical Aspects of Beam Times
- 6. Future Prospects of GELEM
- 7. Questions and Answers
- 8. Summary and Conclusions

Eckart Rühl (FU Berlin)

Clemens Laubschat (TU Dresden / Martin Weinelt, FU Berlin)

Anna Makarova (FU Berlin) Oliver Rader (HZB) Antje Vollmer (HZB) Anna Makarova (FU Berlin) Martin Weinelt (FU Berlin) Serguei Molodtsov (TU Freiberg) Eckart Rühl (FU Berlin)

²Research Opportunities @ U125_PGM

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

Spin- and angle-resolved photoemission



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 \rightarrow Opening of band gap
- Dirac semimetal with tunable Dirac point
- Dirac semimetal & magnetic field \rightarrow Weyl semimetal, appearance of Fermi arcs
- Lead halide perovskites

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

Ferromagnetic topological insulator: p-type MnSb₂Te₄

- 2D Dirac cone with Dirac point close to the Fermi level
- Mn stoichiometric \rightarrow ferromagnetic, high Curie temperature of 50 K
- Out-of-plane magnetic anisotropy

Helical spin texture

- PES proves out-of-plane spin polarization
- STS reveals bandgap closing at the Curie temperature





Out of plane spin component



Gap closing at T_C confirmed by STS

3D Dirac semimetal Au₂Pb with tunable Lifshitz transition



450 meV shift of Dirac cone:

Transition from hole-type to electron-type Fermi surface

T-dependent lattice compression

DFT

ED

3

Δε (%)

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)





AFM order

25 K

6 K

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 CsPbBr₃ (DFT calculation)







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



Dipole beamline

Characteristics of the beamline





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

Ultra-High Vacuum station

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



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)

in operation since 2011

Functionalization of 2D materials

Doping of graphene with heteroatoms •



Nitrogen-doped graphene on a curved nickel surface

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

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

experimen

0 5 10 15 20

Vicinal angle a (deg.

0 5 10 15 20

Vicinal angle a (deg.)

experiment

calc. with

h=2.04A

Stability of materials

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



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



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).

Energy storage materials

A high-capacity P2 Na_{2/3}Ni_{1/3}Mn_{2/3}O₂ cathode material for sodium ion batteries with oxygen activity



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



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 (B₃N₃H₆) at 750°C for 12 min on c-Ni(111) surface

(a) curved Ni(111) crystal



Step 2. Characterization



- L. Fernandez et al, 2D Mater. 6 (2019) 025013
- A. Makarova et al., JPCC 123 (2019) 593-602

Example of a complete study

Step 3. Sample treatment

Step 4. Further characterization



Future capabilities

New Manipulator for in operando and in situ studies

Key features:

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

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



Spin- and angle-resolved photoemission





A 40.842k B5. Under 5 80.000k 0% Off

Staff scientist : J. Sánchez-Barriga (HZB)Construction & Commissioning: J. Sánchez-Barriga, A. VarykhalovTechnical design by A. Varykhalov (HZB)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(<u>k</u>, σ_x, σ_y, σ_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



Undulator beamline U125_PGM

- Quasiperiodic undulator
- 2 gratings (600 l/mm and 1000 l/mm)
- hv = 15 200 eV (optimal range < 100 eV)
- $\Delta E < 1 \text{ meV}$ for hv < 100 eV
- linear horizontally polarized (fixed)
- Beamspot: 68 μm x 25 μm
- photon flux: > 10¹³ Photons/s/0.1%BW/100mA



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HOW TO WRITE A GOOD PROPOSAL...

4.



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)





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







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



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

A0045.0 me Square is dealcated to high resolution angle-resolved photoelectron spectroscepy. It is equipped with a very high resolution electron every analyzer and attached to a beamline providing photoes in the carry anegric new 10–20 eV with the resolution and bit control user the polerination. A precise 6 axes cryomanipulater combined with an electron analyzer Scienta 80006, overaded in panial engular detection mode, alicons for accurate band structure mapping. The versatility of the system is attended by event and avait modal opercision of submit simple transmit techniques.

Selected Applications:

- JD materials and graphene
- Topological invalators, Weyl semimetals, Dirax nodal line materials
 Metallic single crystals with flat and visinal surfaces
- · High temperature superconductors
- · Metal halide perovakites (inorganic)







Instrument Scientist

Dr. Andrei Varykhalov

1030 0041 - 14802

Instrument Scientist

Storage Rigg Ha

Dr. Asidzei Waryktudiow

States of \$2225 (see Square

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	AIGHES One Separa
Beamline data	
Segment	H13
location (Pillar)	14.2
iource	UE112 (Elliptical Undulator)
Monochromator	PGM 2
	in the set



Reference All publications based on experiments using 0110 Pluk 24-1*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	CoreLabs@HZB
DEGAS - GasLab <u>3D Data Analytics Lab</u> <u>Micro-CT Lab</u> <u>Crystallography Lab (preparation and characterization)</u> <u>Bulk Properties Lab (former LaMMB)</u>	X-Ray CoreLab CCMS (Correlative Microscopy and Spectroscopy) CoreLab QM (Quantum Materials) EMIL (Energy Materials In-Situ Laboratory Berlin)
User Labs@HZB Adlershof	Additional User Facilities@H2B Adtershof
<u>MX BioLab</u> <u>Chemistry Lab</u>	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 Klenike Matthias Nees
Argon-Glovebox (KMC-2) Specifications	Glovebox for dry and oxygenfree sample preparations	Dirk Walacher Nico Grimm
UV-VIS 2700	UV-VIS Spectrophotometer Shimadzii UV-2700	Anna-Marie Runge
Differential pumping stage	differential pumping stage	Matthias Neeb
Nitrogen Generator	Nitrogen generator PARKER NitroFlow Leb	Dirk Wallacher Nico Grimm
Drop Shape Analyzer	Drop shape analyzer KRUSS DSA25 for wetting investigations	Beatria-Kamelia Menzel Tristan Petit Anna-Marie Runge Matthias Neeto Bastian Klemice

Beamtime applications (BESSY, CoreLabs, VIPERIab...) via the general access tool <u>GATE</u> A guide for beamtime application is available online

FROM IDEA TO PROPOSAL

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
- BL scientists clarify what is possible at the beamline
- Sample Environment helps to make things possible

photons@helmholtz-berlin.de

hz-b.de/instruments

hz-b.de/samenv

Talking before writing ensures that the proposed exeperiment is feasible

GATE (HZB)

PREPARING EXPERIMENT TIME Guest visits (BEX'sem PTB) Latiersh

Groest works, Handbook

AFTER THE EXPERIMENT TIME Superior experiment umebreddas k Sabina report

MY PROPOSAL

New proposal

Proposal review feedback Proposel Inc.

Public ation ini

REFERCE SECTION

Review proposals

Review propriods offline

Proposals per call

Proposals Reports

Referee Nandboon

Your Neural of expense

PERSONAL SECTION

Personal data Change paraword

Change overname

Griline Mainings

Disable / delete accours

RES

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

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 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







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

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Sample	heating up to K	Others (please specify)	
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		Substances / Quantitie Please specify	equipment?
		Ques	X no yes uncertain
		Please specity	If yes or uncertain, please give delais of the risks associated
		Dangerous substan	Thou who bring samples or chemicals shall take them home!
		Do you plan to use dan	and the second second second reve mant tickle



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 EXPERIMENTAL PLAN

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.









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





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



^{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 ...

- 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.