Scientific Facility Nanostructuring Lab https://www.fkf.mpg.de/NSL

> hot plate AB004

hot plate

spin coater AB201

AB 13

From Brochure: FORSCHUNG LEBEN, Uni Stuttgart (Photo: Max Kovalenko)



Scientific Facility Nanostructuring Lab

@ Max Planck Institute for Solid State Research

Jürgen Weis

http://www.fkf.mpg.de/NSL

Few words on

Max Planck Institute for Solid State Research

Max-Planck-Institut für Festkörperforschung (MPI-FKF) belongs to:



Max Planck Society

- Max Planck Institutes (about 84) carry out basic research in the life sciences, natural sciences and the social and human sciences.
- Harnack Principle:

Identify and hire world's leading researchers as 'Director':

They themselves define their research subjects and are given the best working conditions, as well as free reign in selecting their staff.

- Society is independent, self-organized by the Scientific Members (about 240 ,Directors')
- Funded by tax payer (50% Bund, 50% 16 Bundesländer) (2 Mrd € per year)

Few examples:

Max Planck Institute for Plasma Physics Max Planck Institute for Empirical Aesthetics Max Planck Institute for Biology of Ageing Max Planck Institute for Brain Research Max Planck Institute for the Study of Crime, Security and Law Max Planck Institute for Demographic Research Max Planck Institute for Evolutionary Anthropology Max Planck Institute of Quantum Optics Max Planck Institute for Software Systems

Campus Stuttgart-Büsnau



Campus Stuttgart-Büsnau:

before 03/2011:

- Max Planck Institute for Metal Research (* 1921 as KWI)
- Max Planck Institute for Solid State Research (* 1969)
- Material Science of University Stuttgart

Campus Stuttgart-Büsnau:

after 03/2011:

- Max Planck Institute for Intelligent Systems (* 2011) (Stuttgart/Tübingen) (part of ,Cyber Valley')
- Max Planck Institute for Solid State Research (* 1969)
- Material Science of University Stuttgart

Campus Stuttgart-Büsnau



Precision Lab

removed

MPI for Solid State Research

MPI for Intelligent Systems

Understanding and designing electronic/ionic properties of (crystaline) materials (electron-, ion-, phonon transport, superconductivity (@ RT ?), magnetism, spin textures)

Pioneering various epitaxial growth techniques: Molecular Beam Epitaxy (MBE), Pulsed Laser Deposition (PLD), Thermal Laser Epitaxy (TLE), bulk crystal growth

Started in 1970s: MBE of III-V Compound Semiconductors



- Pioneering various epitaxial growth techniques: Molecular Beam Epitaxy (MBE), Pulsed Laser Deposition (PLD), Thermal Laser Epitaxy (TLE)
- Pioneering analytic tools and theoretical models (neutron scattering spectrometer, probing ultrafast dynamics, scanning tunneling spectroscopy)

Understanding and designing electronic/ionic properties of (crystaline) materials (electron-, ion-, phonon transport, superconductivity (@ RT ?), magnetism, spin textures)

- Pioneering various epitaxial growth techniques: Molecular Beam Epitaxy (MBE), Pulsed Laser Deposition (PLD), Thermal Laser Epitaxy (TLE)
- Pioneering analytic tools and theoretical models (neutron scattering spectrometer, probing ultrafast dynamics, scanning tunneling spectroscopy)
- Design functionality at Interfaces / Surfaces

Chemical Bonding, Mechanical Strain, Topology of Band Structure

- Pioneering various epitaxial growth techniques: Molecular Beam Epitaxy (MBE), Pulsed Laser Deposition (PLD), Thermal Laser Epitaxy (TLE)
- Pioneering analytic tools and theoretical models (neutron scattering spectrometer, probing ultrafast dynamics, scanning tunneling spectroscopy)
- Design functionality at Interfaces / Surfaces (LaAIO₃/SrTiO₃ Interface) Chemical Bonding, Mechanical Strain, Topology of Band Structure

- Pioneering various epitaxial growth techniques: Molecular Beam Epitaxy (MBE), Pulsed Laser Deposition (PLD), Thermal Laser Epitaxy (TLE)
- Pioneering analytic tools and theoretical models
 (neutron scattering spectrometer, probing ultrafast dynamics, scanning tunneling spectroscopy)
- Design functionality at Interfaces / Surfaces
- Confined to reduce spatial dimensions (quantum Hall effect, quantum dots)

- Pioneering various epitaxial growth techniques: Molecular Beam Epitaxy (MBE), Pulsed Laser Deposition (PLD), Thermal Laser Epitaxy (TLE)
- Pioneering analytic tools and theoretical models
 (neutron scattering spectrometer, probing ultrafast dynamics, scanning tunneling spectroscopy)
- Design functionality at Interfaces / Surfaces
- Confined to reduce spatial dimensions (quantum Hall effect, quantum dots)
- Superpose another periodicity (,superlattice')

- Pioneering various epitaxial growth techniques: Molecular Beam Epitaxy (MBE), Pulsed Laser Deposition (PLD), Thermal Laser Epitaxy (TLE)
- Pioneering analytic tools and theoretical models
 (neutron scattering spectrometer, probing ultrafast dynamics, scanning tunneling spectroscopy)
- Design functionality at Interfaces / Surfaces
- Confined to reduce spatial dimensions (quantum Hall effect, quantum dots)
- Superpose another periodicity (,superlattice')
- Oxides of transition metals (d shell)

- Pioneering various epitaxial growth techniques: Molecular Beam Epitaxy (MBE), Pulsed Laser Deposition (PLD), Thermal Laser Epitaxy (TLE)
- Pioneering analytic tools and theoretical models (neutron scattering spectrometer, probing ultrafast dynamics, scanning tunneling spectroscopy)
- Design functionality at Interfaces / Surfaces
- Confined to reduce spatial dimensions (quantum Hall effect, Quantum dots)
- Superpose another periodicity (,superlattice')
- Oxides of transition metals (d shell)
- Strong electron-electron correlations

Understanding and designing electronic/ionic properties of (crystaline) materials (electron-, ion-, phonon transport, superconductivity (@ RT ?), magnetism, spin textures)

- Pioneering various epitaxial growth techniques: Molecular Beam Epitaxy (MBE), Pulsed Laser Deposition (PLD), Thermal Laser Epitaxy (TLE)
- Pioneering analytic tools and theoretical models (neutron scattering spectrometer, probing ultrafast dynamics, scanning tunneling spectroscopy)
- Design functionality at Interfaces / Surfaces
- Confined to reduce spatial dimensions (quantum Hall effect, Quantum dots)
- Superpose another periodicity (,superlattice')
- Oxides of transition metals (d shell)
- Strong electron-electron correlations

,New Quantum Materials'

Departments (Physics, Chemistry, Theory)

https://www.fkf.mpg.de



480 persons (220 scientists including about 100 PhD students)

Institute offers Unique Infrastructure ...

Precision Lab MPI FKF

ALL TYPE LEADER I AND THE AND THE ATTEMPT IN A TYPE AND THE ADDRESS OF THE

lillan treas

1

Ultra-low floor vibrations Electro-magnetic and acoustic shielding

WALT

Group Weis: Scanning Probe Microscopy below 50 MilliKelvin latest generation of

Transmission Electron Microscopes

Stuttgart Center for Electron Microscopy (StEM)



Scientific Facility Nanostructuring Lab https://www.fkf.mpg.de/NSL

> hot plate AB004

hot plate

spin coater AB201

AB 13

From Brochure: FORSCHUNG LEBEN, Uni Stuttgart (Photo: Max Kovalenko)

Scientific Facility Nanostructuring Lab



Mission:

To support Scientists of the Institute in their needs on sample processing and characterization down to nm scale. Materials which are processed:

III-V Semiconductors Graphene, various 2D flakes Carbon nanotubes, Nanowires, usually deposit on glass or Si /SiO₂ substrate Organic films, Metal Oxide heterostructures Diamond Silicon SiC, BN Glass Li and Na based materials



Source: Wikipedia



Handling small pieces up to wafer



Tools / Techniques ?



Electron Beam Lithography JEOL JBX6300FS (100 kV) Raith eLine, Raith eLine plus (< 30 kV)



Electron Beam Lithography JEOL JBX6300FS Raith eLine, Raith eLine plus

Optical Lithography (Mask aligner, Laser Direct Writer)



Electron Beam Lithography JEOL JBX6300FS Raith eLine, Raith eLine plus

Optical Lithography

Scanning Electron Microscopy Zeiss Merlin, Zeiss SEM 500

Scanning Electron Microscopy

Imaging + Analytics + In-situ Complementary Techniques

High-resolution SEM imaging



EBSD: Kikuchi pattern on thermally treated ALD-HfO_x





Electron Beam Induced Current (EBIC) signal overlaid on SEM image of pn diode







Electron Beam Lithography JEOL JBX6300FS Raith eLine, Raith eLine plus

Optical Lithography

Scanning Electron Microscopy Zeiss Merlin, Zeiss SEM 500

,Swiss Knife for the Nanoworld' Zeiss Crossbeam

- Focused Ga Ion Beam
- Scanning Electron Microscope
- Gas Injection (,Glue')
- Micromanipulators
- EDX

•

Example: Focussed Ion Beam Preparation (for Dept. Takagi)



A. Bangura (Dept. Takagi), B. Fenk

Example: Focussed Ion Beam Preparation (for Dept. Takagi)



A. Bangura (Dept. Takagi), B. Fenk


A. Bangura (Dept. Takagi), B. Fenk



Vacuum / Oxygen-free Shuttle between Systems



Electron Beam Lithography JEOL JBX6300FS Raith eLine, Raith eLine plus

Optical Lithography

Scanning Electron Microscopy Zeiss Merlin, Zeiss SEM 500

,Swiss Knife for the Nanoworld' Zeiss Crossbeam

Material Deposition/Sputtering in Vacuum



Electron Beam Lithography JEOL JBX6300FX Raith eLine, Raith eLine plus

Optical Lithography

Scanning Electron Microscopy Zeiss Merlin, Zeiss SEM 500

,Swiss Knife for the Nanoworld' Zeiss Crossbeam

Material Deposition/Sputtering in Vacuum

Wet and Dry Etching

Atomic Layer Deposition



Dry Etching / Reactive Ion Etching Oxford Instruments Plasma Pro100 Cobra (ICP-RIE)



- installed 2016
- up to 16 process gases

```
Ar, He, N_2

O_2

H_2, CH_4

SF_6

CI_2, BCI_3, SiCI_4

HBr

C_4H_8, CHF_3, CF_4
```

- Sample Electrode -150 °C 300 °C
- ,Bosch Process' for deep Si etching

Dry Etching / Reactive Ion Etching Oxford Instruments Plasma Pro100 Cobra (ICP-RIE)



- installed 2016
- up to 16 process gases

```
Ar, He, N_2

O_2

H_2, CH_4

SF_6

CI_2, BCI_3, SiCI_4

HBr

C_4H_8, CHF_3, CF_4
```

- Sample Electrode -150 °C 300 °C
- ,Bosch Process' for deep Si etching

fluorine, chorine, bromine chemistry

Thermal Atomic Layer Deposition (ALD)

Cambridge Nanotechnolgy Shavanna 100



Materials deposited: AI_2O_3 HfO_x TiO_2

Atomic Layer Deposition (ALD): Resist Profiles (?)



Sentech-Cluster



Plasma Processing Cluster



Plasma Processing Cluster with

Induced Coupled Plasma Reactive Ion Etching (ICP RIE) (fluorine, chorine, bromine chemistry)

Plasma Enhanced Atomic Layer Deposition (PEALD) with in-situ Laser Ellipsometry (oxides, nitrides, sulfides, metal)

Surface Treatment and Surface Sealing without breaking vacuum Broad Spectrum of Processing and Material Options



Electron Beam Lithography JEOL JBX6300FX Raith eLine, Raith eLine plus

Optical Lithography

Scanning Electron Microscopy Zeiss Merlin, Zeiss SEM 500

,Swiss Knife for the Nanoworld' Zeiss Crossbeam

Material Deposition/Sputtering in Vacuum

Wet and Dry Etching

Atomic Layer Deposition

Wire Bonding

Clean Chemical Working Places (,Wet benches') (including HF etching with safety infrastructure)

Service offered from the NSL Team

- You get trained / take ,driver license' and use equipment on your own.
- We process samples / make SEM analysis / ... for you.
- We take care of the equipment / infrastructure.
- We make a common process development.
- We push common research projects.
- We develop processing techniques and infrastructure further.
- We keep knowledge about processing techniques.
- We evaluate new tools for possible use in NSL.



NSL Customers are Scientists of

MPI-FKF, MPI-IS, University of Stuttgart

+ Collaborations (like PTB)

Electron-Beam Lithography

SEM-based Electron Beam Lithography Raith eLine

Established in Summer 2006

Meanwhile, more than 150 persons have been trained.

In the past, more than 40 persons had access in parallel.



Electron Beam Lithography Raith eline plus (installed in 09/2021)



Examples for samples processed by users on the Raith eLine

Split–Gate Technique:



Started 1990s

Quantum Dot System by Etching Grooves



Armin Welker, MPI-FKF (2005)

Electron Interference via Two Quantum Dots



Pre-structured Substrate (AI,Ga)As Heterostructure with 2DES









Two Quantum Dots in an Interferometric Arrangement



Leonhard Schulz, MPI-FKF (2008)

Aligment Accuracy of Three E-beam Steps



200 nm

Leonhard Schulz, MPI-FKF (2008)

Examples for Contacting Randomly Distributed Nanowires / Flakes



Examples for Contacting Randomly Distributed Nanowires / Flakes

Before : define a coordinate system on the substrate by e-beam lithography



Simple efficient coordinate markers for investigating synthetic nanofibers'
Gyu-Tae Kim, Ulrike Waizmann, Siegmar Roth
Appl. Phys. Lett. 79, 3497–3499 (2001)



10	μm

Eleonora Storace, MPI-FKF (2007)



InAs wire contacted by metal Eleonora Storace (2007)







300 nm

NMOS inverter with level-shift stage realized on a single ZnO nanowire

Daniel Kälblein (2009)

Examples for Plasmonic Structures (Optical Meta Materials)



.... -----.... and the ------.... --.....

5 µm

Plasmonic molecule Septamere

Mario Hentschel (2009)
Two layers of plasmonic structures



Two layers of plasmonic structures



Gold (Triangle) – Palladium (Dot)



Gold (Triangle) – Palladium (Dot)



Example for Double-layer Resist with large Undercut for Shadow Evaporation

Single–Electron–Transistor (SET)



Proposed: D. Averin, K.K. Likharev, IEEE Trans. Magn. 23, 1142 (1987) First Realisation: T.A. Fulton and G.J. Dolan, Phys. Rev. Lett. 59,109 (1987)

Single–Electron Transistor Made of Aluminum Two–Angle Shadow Evaporation Process:

after Dolan 1987



Single–Electron Transistor Made of Aluminum Two–Angle Shadow Evaporation Process:









Two electrostatically coupled metal single-electron transistors Ingmar Bruder / Jochen Weber (2007)

1 µm

Scanning-Probe Experiments on Quantum Hall Samples at 50 mK

Array of metal single-electron transistors



Several PhD projects: Jochen Weber, Kostas Panos, Marcel Mausser, Andreas Gauss, Maximilian Kühn, Lukas Freund

Hall sensor



Evolution of compressible/incompressible landscape versus magnetic field within the 2DES bulk



100 kV Electron Beam Lithography System Jeol JBX 6300 FS (installed 2009)

with special environmental conditions



Enviromental Requirements for Dedicated Electron Beam Lithography System

- Temperature Stability: < 0.1 °C
- Magnetic Field Stability: $< 0.1 \mu$ T peak-to-peak during writing time
- Low Floor Vibrations
- Cleanroom Class 100

Not fulfilling? This means about 10 nm shift



Enviromental Requirements for Dedicated Electron Beam Lithography System

- Temperature Stability: < 0.1 °C
- Magnetic Field Stability: $< 0.1 \mu$ T peak-to-peak during writing time
- Low Floor Vibrations
- Cleanroom Class 100

Not fulfilling? This means about 10 nm shift

Stability of the earth magnetic field over a year

(close to Munich in 2002)



Strong magnetic fields everywhere !

Cryostat with superconducting magnet



Magnetic field variations by moving parts





Concept:



Laminar air flow to obtain constant temperature and cleanroom conditions

Passive magnetic field shielding and active compensation (Helmholtz coils)

Active floor vibration isolation



Jeol JBX6300FS in Shielded Cleanroom in 2B24





Dedicated 100 kV Electron Beam Lithography System ?

 Acceleration Voltage: Interaction with resist/substrate (Proximity Effect, Damage)















Radical diffusion? Catalytic Reactions?

Electron Scattering: Monte-Carlo-Simulation

An example:



From Kyser and Viswanathan (1975)

Point Spread Function: (Energy density deposited radially vs. Distance)



Marcus Rommel Monte-Carlo-Code ,Penelope

Split-gate structure:



Split-gate structure:





Jeol JBX6300FS (2009) (no fracturing and dose variation)

Split-gate structure:





Even smaller:


Interdigital Metal Structure for generating Surface-Acoustic Waves



Single-Photon Detector Based on NbN Superconductor



Eric Reutter, MPI-FKF (2022)

following Nature Photonics 2, 302 (2008)

Interaction of Electron Beam with Sample



Electron Beam Lithography on a Thin Membrane





Diffraction pattern for use in a TEM (Koch, van Aken, MPI IS)

1 µm

Gold Structure on 15 nm thin SiN_x Membrane



Gold Structure on 15 nm thin SiN_x Membrane



- Acceleration Voltage: Interaction with substrate (Proximity Effect, Damage)
- Writing speed:

typically 1 nA (Jeol) versus 5 pA (Raith eline)

Millions of Transistor Structures based on Oxide Heterostructures



Why Large Area ? An Example: Optics / Metamaterials

Small area sample

Large area sample



No well defined k for light

Allows angle dependent reflection / transmission measurements FIR to Visible











Marcus Rommel (2011)



μm	 WD = 3.8 mm EHT = 5.00 kV	Mag = 1.00 K X Width = 114.3 μm	Signal A = InLens Signal B = InLens	File Name = RD10D10Ce_157.tif Date :7 Jan 2013	

















with Dr. Gompf (University of Stuttgart)

1 µm

- Acceleration Voltage: Interaction with substrate (Proximity Effect, Damage)
- Writing speed:

typically 1 nA (Jeol) versus 5 pA (Raith eline)

- Enables patterning thick resists
- Allows defined large undercut in double-layer resists



Electron Beam Lithography on a Thin Membrane



Electron Beam Lithography on a Thin Membrane













Array of Metal Single-Electron Transistors as Primary Thermometer on 30 nm thin Membrane for Heat Capacity Measurements

T. Reindl et al. with Andreas Rost (St. Andrews, Schottland)



- Acceleration Voltage: Interaction with substrate (Proximity Effect, Damage)
- Writing speed:

typically 1 nA (Jeol) versus 5 pA (Raith eline)

- Enables patterning thick resists
- Allows defined large undercut in double-layer resists
- Reproducibility, Stability, Precision

- Acceleration Voltage: Interaction with substrate (Proximity Effect, Damage)
- Writing speed:

typically 1 nA (Jeol) versus 5 pA (Raith eline)

- Enables patterning thick resists
- Allows defined large undercut in double-layer resists
- Reproducibility, Stability, Precision

- Acceleration Voltage: Interaction with substrate (Proximity Effect, Damage)
- Writing speed:

typically 1 nA (Jeol) versus 5 pA (Raith eline)

- Enables patterning thick resists
- Allows defined large undercut in double-layer resists
- Reproducibility, Stability, Precision

Facilitating and Enabling certain Lithography !
Dedicated 100 kV Electron Beam Lithography System ?

- Acceleration Voltage: Interaction with substrate (Proximity Effect, Damage)
- Writing speed:
 - typically 1 nA (Jeol) versus 5 pA (Raith eline)
- Enables patterning thick resists
- Allows defined large undercut in double-layer resists
- Reproducibility, Stability, Precision
- Alignment requires good metal marks
- High investment / high running costs
- Flexible ? (sample size, alignment) requires well developed work flow

Dedicated 100 kV EBL system

versus SEM-based Raith eLine ?

They are partly complementary.

We need both.

Thank you for your attention !

https://www.fkf.mpg.de/NSL