KNMF Installations

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Facilitating innovation in advanced multimaterial micro and nanotechnologies

Thomas Schaller
KNMF User Office

KIT – University of the State of Baden-Wuerttemberg and National Research Center of the Helmholtz Association
The Karlsruhe Nano Micro Facility (KNMF) is a high-tech innovation platform for structuring, functionalising and characterising a multitude of materials at the micro- and nanoscale.

KNMF provides users from industry and academia open and, in case of public work, no cost access to an integrated set of multimaterial state-of-the-art micro and nanotechnologies.

KNMF is operated by the Karlsruhe Institute of Technology as a Helmholtz Research Infrastructure.

KNMF possesses a unique technology portfolio and leading expertise which can be combined to provide individual solutions to challenging user requests.

An on-going investment programme is enabling an enhancement of our facilities.

Visit our website for up to date information and establish your first personal contact with our experts.

Contact

KNMF User Office
Karlsruhe Nano Micro Facility (KNMF)
Karlsruhe Institute of Technology
Hermann-von-Helmholtz-Platz 1
76344 Eggenstein-Leopoldshafen
Germany

Phone: +49 721 608-23123
Fax: +49 721 608-26273
E-mail: knmf-useroffice@kit.edu
Web: www.knmf.kit.edu
Technologies

KNMF Laboratory for Micro- and Nanostructuring

- 3D Direct Laser Writing (3D-DLW)
- Atomic Layer Deposition (ALD)
- Deep X-ray Lithography (XRL)
- Dip-Pen Nanolithography (DPN) & Polymer Pen Lithography
- Direct Laser Writing (DLW)
- Dry Etching Cluster (DRIE)
- Electron Beam Lithography (EBL)
- Focused Ion Beam (FIB)
- Hot Embossing (HE)
- Injection Moulding (IM)
- Laser Material Processing (LMP)
- Thin Film Technologies (TFT)

KNMF Laboratory for Microscopy and Spectroscopy

- 3D Atom Probe Tomography (APT)
- Atomic Force Microscopy (AFM)
- Auger Electron Spectroscopy (AES)
- Bulk and Trace Analysis (BTA) of Nanomaterials
- Helium Ion Microscope (HIM)
- Laser Ablation ICPMS (LA-ICPMS)
- Single Crystal X-ray Diffractometry (SCXD)
- Soft X-ray Spectroscopy, Microscopy, and Spectroscopy (WERA)
- Thin Film Characterisation (TFC) Methods
- Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS)
- Transmission Electron Microscopy (TEM)
- Travelling Wave Ion-Mobility Time-of-Flight Mass Spectrometry (ESI-/MALDI-TOF)
- X-Ray Photoelectron Spectroscopy (XPS)
The Nanoscribe Photonic Professional Two-Photon Lithography is preferentially used for 3D structuring and allows nanometer patterning. But beyond that 2D and 2.5D structures with nano dimensions are also possible. This system uses a nonlinear two-photon absorption process to modify a photosensitive medium at a specific point in the resist. By scanning the photoresist with a piezo stage over this point a 3D-structure with dimensions in the submicron scale can be written.

Contact

Stefan Hengsbach  
Phone +49 721 608-24441, fax +49 721 608-24331, email stefan.hengsbach@kit.edu
Dr. Klaus Bade  
Phone +49 721 608-24431, fax +49 721 608-24331, email klaus.bade@kit.edu

Institute of Microstructure Technology (IMT) - www.imt.kit.edu/english

Features

- Resolution: 200 nm (3D) or 180 nm (2D)
- Feature size: max. 300 x 300 x 300 µm³
- Writing area: 100 x 100 mm²
- Writing speed: between 10 µm/s and 500 µm/s depending on structure
- Feature accuracy: 50 nm depending on the aspect ratio

Limitations/constraints

- The realizable structure size depends on the structural stability of the design
- The best results can be reached by using the IP-resists

Materials

Resists: IP-L, IP-G, IP-Dip, SU8 and similar resists that are photosensitive at a wavelength of 380nm
The full-fledged research atomic-layer-deposition (ALD) system, model Picosun SUNALE R-200 Advanced, is featuring a hot wall reactor for temperatures of up to 500 °C, an ozone generator, a plasma generator as well as the capability to handle three liquid and three solid precursor materials at the same time. The “Picoflow” mode allows for coating of highly irregular surfaces with high aspect ratios of up to 1:2000. A powder holder enables coating of micropowders. Sensitive samples can be handled in an argon filled glove-box and loaded into the process chamber through a load-lock. For this purpose the carrier gas can be switched from N₂ to Ar.

Contact
Dr. Christian Reitz
Phone +49 721 608-28923, fax +49 721 608-28976, email christian.reitz@kit.edu
Dr. Philipp Leufke
Phone +49 721 608-28687, fax +49 721 608-28976, email philipp.leufke@kit.edu

Features
- conformal 3D coatings (maximum aspect ratio 1:2000)
- 2D thin films
- multilayers
- graded coatings, powder coatings
- ALD is attached to an argon filled glove-box
- Process gasses:
  - N₂ or Ar as carrier gas / purge gas
  - H₂O / O₃ for thermal / ozone assisted ALD
  - NH₃ for thermal ALD of nitrides
  - N₂, O₂, H₂/Ar (5/95), H₂/N₂ (5/95), H₂/Ar (5/95)
  - gases for plasma assisted ALD processes
- Processes available: Al₂O₃ and AlN, TiO₂ and TiN, TaN, HfO₂, SiO₂, Ag, Nb₂O₅

Design rules
- Max. sample height 15 mm
- Max. diameter 200 mm (8” Si-Wafer)
- Max. 28 cm³ volume for powder coatings (max. 300 m²/g specific surface area)

Materials
metals, ceramics, nitrides

Typical structure
SEM image of an approx. 75 nm thick TiO₂ thin film coating on a (100)-oriented Si-Wafer
Deep X-ray lithography uses synchrotron radiation to pattern thick PMMA layers (thickness: several microns up to several millimetres) in order to achieve high aspect ratio microstructures (aspect ratio up to 50). The structures are characterised by very steep sidewalls (slope angle better than 1 mrad) and sidewall roughness in the range of 20 to 30 nm. For optical applications usually microoptical benches with cylinder lenses, prisms and fixing structures for other optical components are fabricated. The structures are either used as prototypes, as lost form for metal replication or as moulds to fabricate mould inserts.

Features
- Aspect ratios up to 50
- Structural height up to several millimetres
- Structural details less than 1 μm
- Slope angle better than 1 mrad

Limitations/constraints
- Only PMMA and SU8 (in case of prototyping)
- Time consuming process for prototyping due to mask fabrication (4 to 6 weeks)

Design rules
- Rounding of structural edges (radius > 5 μm)

Contact
Dr. Martin Börner
Phone +49 721 608-24437, fax +49 721 608-24331, email martin.boerner@kit.edu

Dr. Jürgen Mohr
Phone +49 721 608-24433, fax +49 721 608-24331, email juergen.mohr@kit.edu

Institute of Microstructure Technology (IMT) - www.imt.kit.edu/english

Typical structures and designs

Fig. 1: 500 μm high PMMA structure (width of the small bar: 5 μm)

Fig. 2: Gear wheels and anchors made out of Au (99%) and Ni/Co alloy

Fig. 3: Crossed X-ray lenses (SU-8)

Fig. 4: Microoptical bench with cylindrical mirrors and fixing structures
Dip Pen Nanolithography (DPN) uses the tip of an Atomic Force Microscope (AFM) to deliver molecular inks to a surface. Being a constructive (bottom-up) approach to lithography, DPN has several unique capabilities. First, it can be readily carried out using parallel tip arrays enabling both high throughput and high areal resolution. Second, since no etching or post-processing is typically required, prepatterned surfaces composed of a variety of materials can be used. Finally, DPN is capable of integrating of multiple materials (or inks) with both high resolution and high throughput [1]. In particular, the use of lipid-based inks developed at the Karlsruhe Institute of Technology takes advantage of these DPN aspects. DPN with lipids was used in diverse fields from sub-cellular arraying [2,3] to sensors [4,5].

The related technique of Polymer Pen Lithography (PPL) combines the strengths of microcontact printing (large area parallel printing, inexpensive stamp materials) with the advantages of DPN (pattern flexibility, multiplexing) [6].

Features

- 20 nm resolution for thiols on gold, or 100 nm for phospholipids
- Throughput on the order of cm²/min using massively parallel arrays
- Compatible with biological molecules (e.g. DNA, protein & phospholipids)
- Phospholipid based inks can write on a variety of surfaces – metals, insulators, hydrophobic, hydrophilic, etc.
- Capability of integrating multiple ink materials on a single substrate
- Compatible with pre-structured surfaces
- No undercuts
- No hollow parts
- A one step fabrication process

Materials

- Alkanethiols on gold
- Phospholipids with
  – fluorescent headgroups
  – biotinylated headgroups
  – NTA-headgroups
  – other lipids suitable for liposomes
- Azides on alkyne-functionalized surfaces ("Click-Chemistry")
- Substrates for lipid patterning:
  – glass, silicon
  – PMMA, polystyrene
  – Metals (e.g. Au, Ti)

Limitations/constraints

- There must be a driving force for the ink to flow from the tip to the sample
- Parallel integration of different inks requires that the different inks have similar transport properties
- Each tip in a passive parallel array draws the same structure
- A high throughput quality control method must be used for massively parallel fabrication
- 80 x 80 micron scan area (per tip) for DPN 5000 system
- Alignment marks must be used to align with pre-patterned substrates
- Tips are typically spaced 35 µm in a 1D array or 20 x 90 µm² in a 2D array.
- Custom arrays available
Typical structures and designs

Fig. 1: Functional phospholipids patterned on a glass surface are used to template two proteins at sub-cellular scales [2]

Fig. 2: Functionalization of pre-existing sensor structures by DPN with lipids [5]

Fig. 3: Multi-color micro patterns generated by PPL with fluorescent lipids [6]

Fig. 4: Covalently linked fluorescently labeled azide ink written on an alkyne functionalized CVD coating [7]
References

The system is able to write directly onto a substrate without the need of an additional mask. A laser is focused on the substrate and a high precision stage system scans the writing area. The writehead allows feature sizes down to 2.5 µm with a substrate size up to 6". The machine complements E-Beam lithography and is a less expensive and faster alternative for structures without nano sized features.

Contact
Stefan Hengsbach
Phone +49 721 608-24441, fax +49 721 608-24331, email stefan.hengsbach@kit.edu
Dr. Klaus Bade
Phone +49 721 608-24431, fax +49 721 608-24331, email klaus.bade@kit.edu

Features
- No mask needed
- The wavelength of the Laser (355 nm) allows to expose SU-8-Resist up to 300µm thickness and AZ-Resists up to 7µm thickness
- Writing speed is 35 mm²/min. A 4” wafer will be written in round about 3 hours

Limitations/constraints
- The structure sidewalls are not vertical
- Aspect ratios up to 4 are possible depending on structures and/ or resists

Materials
Resists: AZ1505, AZ4533, SU8 and similar resists
Dry Etching Cluster

Our Dry Etching Cluster consists of the Oxford RIE Plasmalab System 100 with ICP 380 source and the Oxford RIBE Ionfab 300. (RIE: Reactive Ion Etching, ICP: Inductively Coupled Plasma, RIBE: Reactive Ion Beam Etching). The Dry Etching Cluster is an advanced tool for micro- and nanomachining of various materials. The basic feature is a high frequency generator (RF) working at 13.56 Mhz, combined with a high vacuum chamber for wafers with a diameter of 4”. The power varies in the range of 1-2500 W. Available process gases are SF₆ and O₂ for silicon etching; Cl₂, He, Ar and O₂ for chromium and other metals.

Contact

Mechanical Engineer Alban Muslija
Phone +49 721 608-23011, fax +49 721 608-26667, email alban.muslija@kit.edu
PD Dr. Manfred Kohl
Phone +49 721 608-22798, fax +49 721 608-26667, email manfred.kohl@kit.edu
Institute of Microstructure Technology (IMT) - www.imt.kit.edu/english

Features

- Silicon etching via the cryo process (process temperatures are between -80 and -150 °C)
- Production of highly vertical, highly parallel and smooth sidewalls
- Critical lateral dimensions down to the range of 100 nm
- Aspect ratios (ARs) up to 6 are possible.
- Laser end point detection
- Metal etching via RIBE

Limitations/constraints

Silicon:
- Min. lateral dimensions: 100 nm
- Min. depth: 50 nm
- Max. aspect ratio at critical dimensions: 4
- Total max. depth: 40 µm

Chromium:
- Min. dimensions in lateral: 100 nm
- Selectivity over resist: 1:1
- Etch rate: 25...35 nm/min

Materials

- Mask material: PMMA, SiO₂, ma-N 2401
- Structures on Si fragments or complete 4” Si wafers

Notice: Only silicon and chromium substrates can be processed reproducibly with standard processes at the moment.

Design rules

- Explicit and unambiguous layout according to the mentioned limitations.
- Markers for the better localization of the structures, e.g. in the SEM
- If the micro/nano structure is already written onto the substrate, the mask material has to be PMMA, SiO₂ or ma-N 2401
- If combined with the KNMF e-beam, specific limitations concerning the e-beam design rules have to be considered

Typical structures and designs

Deep etched silicon gratings
Typical structures and designs (continued)

Silicon nanopillars with high aspect ratio

Freestanding cantilevers in silicon

Cantilever structures in chromium
Electron Beam Lithography

E-Beam VB6 UHR-EWF
- Substrate: 4” and 6” wafer; special piece parts (on request, minimum size 20 mm x 20 mm)
- High voltage: 100 kV
- Main field: ≤ 1310 μm
- Resolution: < 1 nm (depends on main field size)

E-Beam lithography in extremely thick PMMA (3200 nm) with structural details in submicron range (~ 200 nm)
E-Beam lithography down to 20 nm scale in PMMA (resist thickness < 100 nm).

Contact
Prof. Dr. Christian Koos
Phone +49 721 608-22770, fax +49 721 608-23928, email christian.koos@kit.edu

Dr. Lothar Hahn
Phone +49 721 608-23852, fax +49 721 608-24331, email lothar.hahn@kit.edu

Institute of Microstructure Technology (IMT) - www.imt.kit.edu/english

Features
- Aspect ratio up to 10 depending on geometry
- Structural details ≥ 20 nm
- Resist thickness up to 3200 nm
  (e.g. for electroplating of high aspect ratio gold structures required for X-ray lithography)

Limitations/constraints
- Standard Resist: PMMA

Design rules
- Rounding of structural edges
- Design of dummy structures for stress reduction
- Homogeneous structure allocation (in case of subsequent electroplating)

Materials
Substrate materials: silicon, glass, metal
Other resist and substrate materials on request
The FEI Strata 400S and the Zeiss Auriga 60 Dual Beam FIB are both a combination of a scanning electron microscope (SEM) and a focused ion beam (FIB) system, which allows imaging and structuring of materials at the nanoscale. The focused gallium ion beam can either be used for ion imaging or to cut predefined patterns or images in the surface of a solid. At the same time, the SEM can be used to image the nanostructures generated by FIB. In addition, it is possible to locally deposit C, Pt or W from precursor gases using the electron or ion beam. Additionally, Insulator Enhanced Etching (IEE) using XeF₂ is available.

Using this combined approach it is possible to
- perform cross-sectional structural analysis of surfaces
- extend the cross-sectional analysis by slice and view techniques to image a complete 3D volume
- pattern surface at the nanoscale
- electrically contact selected structures on a sample
- target preparation of TEM samples and in-situ lift-out

Features

**FEI Strata 400 STEM**
- Electron Optics 0.8 nm at 30 kV STEM
- 1.0 nm at 15 kV SEM
- 1.9 nm at 1 kV SEM
- Voltage 200 V–30 kV
- Gallium Ion optics 7.0 nm at 30 kV
- Voltage 2–30 kV
- Detection: TLD SE, ETD, BSE, STEM, CDEM
- Analytical: EDX
- Omniprobe 200 micromanipulator
- GIS for C, Pt and W deposition
- GIS for XeF₂ etched enhance
- Flip-stage

**Zeiss Auriga 60**
- Electron Optics 1.0 nm at 15 kV SEM
- 1.9 nm at 1 kV SEM
- Voltage 100 V–30 kV
- Gallium Ion optics 2.5 nm at 30 kV
- Voltage 0.5–30 kV
- Detection: In-lens SE, ETD, EsB, 4QBSD, SESI, segmented STEM
- Analytical: EDX+EBSD
- Omniprobe 400 micromanipulator
- GIS for C, Pt, W and Si deposition
- Gas injection for charge compensation
- Vacuum transfer system

Materials

Depending on the material, typically a volume of up to 30 x 30 x 10 μm³ can be removed in a reasonable processing time, volumes of up to 100 x 100 x 50 μm³ are possible to remove.

Limitations/constraints
- Sample has to be a solid at RT and stable under vacuum conditions
- Maximum sample dimensions restricted to 5 cm diameter

Typical structures and designs

*Fig. 1: SEM image of the photonic structures on a butterfly wing with a FIB prepared cross-section in the inset.*

In collaboration with R. Siddique and R. Prang, KIT.
Fig. 2: Cross-sectional analysis of a photonic crystal generated by 3D direct laser writing.
In collaboration with NanoScribe and T. Scherer, KIT.

Fig. 3: FIB generated photonic structure in a thin gold film.
In collaboration with Y. Yu, IMTEK and D. Chaissing, KIT.

Fig. 4: Electric contacting of a silver nanowire for 4-point conductivity measurements.

Fig. 5: 3D nanoscale morphology analysis of micro- and macroporous silica for application in HPLC: segmented digital slices through the 3D volume and volume rendering of a small area.
In collaboration with D. Stöckel, B. Smarsley, Univ. Gießen and C. Kübel, R. Prang, KIT.

Fig. 6: TEM cross-section target preparation of a nanoindent in an Ag nanowire.
In collaboration with A. Kobler, T. Beuth and R. Prang, KIT.
Hot embossing is a replication process especially suited for the replication of delicate micro- and nanostructures structures with high aspect ratios on thin layers. Because of the short flow paths and the low shear velocities during molding the replicated structures are characterized by low inner stress. The process is very flexible, because both mold inserts and the type of polymer can be exchanged quickly, which is why hot embossing machines are very popular for laboratory use and for the replication of small series.

Process:
The hot embossing process is an open tool technique, where a semi-finished polymer sheet is put in between the upper and the lower molding tool. The complete tool is evacuated in order to ensure complete filling of the cavities of the microstructured tool, and the polymer is heated up above its softening temperature (melting temperature or glass transition temperature, depending on the polymer class). The softened polymer is pressed into the microstructured cavities. After mold filling, the polymer is cooled down below the softening temperature, while maintaining the applied force in order to avoid shrinkage and sinking marks. Finally, the machine is opened and the microstructured part can be demolded.

Contact
Dr. Matthias Worgull
Phone +49 721 608-26828, fax +49 721 608-24331, email matthias.worgull@kit.edu
Dr. Markus Guttmann
Phone +49 721 608-23850, fax +49 721 608-24331, email markus.guttmann@kit.edu
Institute of Microstructure Technology (IMT) - www.imt.kit.edu/english

Features
- Cycle times 6–20 min
- Molding area up to 8 inch
- Double sided molding (alignment)
- Molding of through holes
- In general molding of all thermoplastic polymers, including high temperature polymers
- Structure size down to the nano range (nanoimprint)
- Quick change of mold insert and polymer – small series and prototypes with different polymers

Limitations/constraints
- Cycle times determined by heating and cooling times
- Max. molding area of 8 inch depends on the available molding tool, larger sizes requires a new development
- Fixation of mold inserts refers to standardized mold inserts and clamping units; other formats require further modifications of the clamping unit

Materials
- Polymer

Design rules
- Side wall draft angle if possible
- Low roughness of the mold surface
- No undercuts
Injection moulding allows the high economic mass fabrication of complex-shaped nano and micro components. These can be singular items or large bodies with nano- or microstructures on the surface, respectively. In both cases very high geometric accuracies and smallest tolerances can be achieved using e.g. LIGA-fabricated mould inserts. Besides the replication of polymers powder injection moulding (MicroPIM) allows for the micro fabrication of components made of a large variety of metals or ceramics. Having reached a reliable status, two-component injection moulding and inmould-labelling reveal strong advantages with respect to reduced mounting expenditures and the capability to produce multifunctional devices.

Contact

Dr. Volker Piotter
Phone +49 721 608-26463, fax +49 721 608-22095, email Volker.piotter@kit.edu

Institute for Applied Materials (IAM-WPT) - www.iam.kit.edu/wpt/english

Features

- Cycle times < 3 s – 6 min
- Largest replicated aspect ratio:
  - 17 for free standing structure (height: 2000 μm; width: 115 μm)
  - 25 for buried structure (height: 250 μm; width: 10 μm)
- Smallest replicated structural detail:
  - < 100 nm for aspect ratio 1, in case of lower aspect ratio replication minima decrease correspondingly
- Special variants like compression injection moulding for enhanced accuracies
- Fabrication of metal and ceramic parts via powder injection moulding
- Multifunctional parts by two-component or inmould-labelling powder injection moulding
- Special equipment for designing / developing feedstock compositions
- Special equipment for thermal treatment available, e. g. hot isostatic pressing (HIP) applying temperature and pressure parameters on a worldwide unique high level

Limitations/constraints

- Relatively large efforts for tooling necessary
- Replication process very sensitive to mould insert’s surface roughness
- Side wall draft angle or ejector slope is recommended for larger aspect ratios depending on the mould insert’s roughness
- Limited undercuts
- No hollow parts in one step fabrication possible

Materials

- 1- and 2-component injection moulding with polymers, metals, and ceramics
- Polymers: nearly all thermoplastics and thermoplastic elastomers
- Functional polymer-based nanocomposites with improved optical, dielectric or conductive properties (e. g. PMMA/CNT, PC/Al₂O₃ a. o.)
- Feedstock development for customer-specific materials using e. g. nano-sized powders
- Metals: PM steels like 17-4PH and 316L, Cu, W and W-alloys, hard metals
- Ceramics: oxide ceramics like ZrO₂ and Al₂O₃, Si₃N₄, mixture ceramics like TiN-Al₂O₃
- with defined material properties, e. g. electrical conductivity
- Subsequent densification and reduction of porosity by HIP

Typical structures and designs

Fig. 1: Smallest puzzle of the world whose pieces have been made of PMMA using singular LIGA mould inserts.
Typical structures and designs

(continued)

Fig. 2: SEM figure of polymer part (PMMA) with nano-sized structures made by injection moulding. (Länge = length).

Fig. 3: SEM-picture of pure tungsten after sintering (above), the same material after additional HIP densification showing significantly reduced porosity (below).

Fig. 4: Gear wheel/shaft sample made by two-component injection moulding of alumina (shaft) and zirconia ceramic (gear wheel). Combined sintered part (left) and green body (right).

Fig. 5: Ring gear of planetary gear set, 1.4542.
An appropriate choice of laser and process parameters is used to control the interaction between laser radiation and material on micrometer and nanometer scale. The types of materials processing which can be carried out are micro- and nanostructuring, microdrilling, cutting, laser transmission welding and surface modification, respectively. The actual smallest structure size which can be achieved, each according to the process and material, is in the range of 200 nm to 400 nm. Aspect ratios up to a maximum of 50 can be realized. Ultraviolet laser radiation as well as ultrafast lasers (femtosecond and picosecond pulse duration) have a particularly high potential for precise micro- and nanoablation, due to its selective material removal and very low thermal load.

Contact
Dr. Wilhelm Pfleging
Phone +49 721 608-22889, fax +49 721 6089-22889, email wilhelm.pfleging@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Processes/limitations
- Structuring of polymer materials and thin films with excimer lasers or high repetition rate and ultrafast laser radiation: structure size 200 nm
- Aspect Ratio:
  - 50 for drilling, 10 for ablation and cutting
- Structuring of metals and ceramics:
  - resolution 1 µm - 10 µm
- Deposition and structuring of tape cast battery materials: 5-50 µm
- Cutting of metals, ceramics, polymers, and battery materials: cutting width 5 µm - 50 µm
- Laser LIGA:
  - surface roughness Rₐ =60 nm, edge radius 1 µm, aspect ratio 5
- Surface modification of polymers and thin films / adjustment of wettability / surface energy / biocompatibility with structure width <1 µm
- Laser-assisted moulding of polymers for the rapid generation of micrometer and nanometer-sized surface structures

Materials
- Structuring:
  - PMMA, PS, PEEK, PI, PSU, thin films (amorphous carbon, SnO₂, LiCoO₂,...), standard battery materials (LiCoO₂, LiMn₂O₄, NMC, LiFePO₄), steel, nickel, brass, WC, Al₂O₃, ZrO₂, SiC
- Cutting:
  - steel, Ti, NiTi, quartz, Al₂O₃, PMMA, PI, PS
- Laser-LIGA: nickel
- Surface modification:
  - PS, PC, PMMA, amorphous carbon thin films

Fig. 1: Micro-machining workstation (PS450-TO, Optec) equipped with a tunable ultrafast laser (Tangerine, Amplitude SYSTEMES) and a tunable short pulse fiber laser (IPG photonics)
Fig. 2: Laser structured mould insert made of steel for replication of microfluidic chips (channel structure width 50 μm)

Fig. 3: Laser microstructured SnO₂ anode material

Fig. 4: Laser generated microgrooves in polystyrene

Fig. 5: Local control of wetting behaviour due to laser-assisted surface modification of PDMS

Fig. 6: SEM image of conical microstructures in NMC cathode material formed by excimer laser radiation

Fig. 7: Specific discharge capacities of an unstructured and laser structured cathode material for lithium-ion batteries (Swagelok® design)
By reactive and non-reactive magnetron sputtering 2-dimensional samples can be coated with metallic or ceramic films in a thickness of 10 nm to 5 μm in order to improve surface properties of the substrates or to allow complete new properties of the system. The deposition process runs in a noble or reactive gas atmosphere at a pressure of Pa in a dc or r.f. plasma process. Thin films can be realized for protective and functional applications. Multilayer or sandwich coatings with up to four different materials can be realized without breaking vacuum.

Contact
Dr. Harald Leiste
Phone +49 721 22541-22889, fax +49 721 608-22567, email harald.leiste@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Features
- Single and multilayer modus
- Reactive gas components N₂, CH₄, O₂
- Different coating concepts like nanocomposites, multilayer and graded coatings are possible
- Typical targets: metals, ceramics, glasses
- Protective (wear resistant) and functional (ferromagnetic) coatings

Limitations/constraints
- Max. sample height 20 mm
- Max. four targets
- Target size Ø 75 mm and Ø 150 mm
- No conductive limitations of material
- Directed coating; no coating inside of tubes or holes
- Surface topography and roughness will be reproduced

Materials
Metals, ceramic, glass

Typical structures and designs
Fig. 1: PVD Thin film deposition facility Leybold Z550
Fig. 2: Fracture surface of a TiN/ZrN multilayer coating
By reactive and non-reactive magnetron sputtering two- or three-dimensional samples can be coated at temperatures between 200 °C and 400 °C with metallic or ceramic films in a thickness of 100 nm to 5 μm in order to improve surface properties of the substrates or to allow complete new properties of the system. The deposition process with balanced and non-balanced magnetron regime runs in a noble or reactive gas atmosphere in a pressure range of Pa in a plasma dc process. Plasma cleaning process before coating is mandatory. Rotating of samples is commonly used.

Contact
Dr. Harald Leiste
Phone +49 721 22541-22889, fax +49 721 608-22567, email harald.leiste@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Features
- Three fold rotation
- Stop and go modus (two fold rotation in front of one target)
- Reactive gas components N₂, CH₄, O₂
- Multilayer and graded coatings possible
- Typical targets: metals electrical, conductive ceramics

Limitations/constraints
- Max. sample height 350 mm
- Temperature range 200–400 °C
- Electrical conductive substrate
- Max. two conductive targets
- Directed coating; no inside coating of tubes or holes

Materials
Metal, ceramic

Typical structures and designs

Fig. 3: PVD Thin film deposition facility Hauzer HTC625

Fig. 4: Coated tools and components
By r.f. or dc reactive and non-reactive magnetron sputtering as well as microwave plasma source deposition two-dimensional samples can be coated at temperatures between 100 °C and 900 °C with metallic or ceramic films in a thickness of 100 nm to 5 μm to improve surface properties of the substrates or to allow complete new properties of the system. The deposition processes are running in gas atmosphere consisting of Ar, N2, O2, CH4 and/or C2H2 in a pressure range of 0.1 Pa and 10 Pa. Plasma cleaning process before coating is mandatory. The samples are fixed on the substrate holder. A dc or r.f. substrate bias can be applied.

Contact

Dr. Harald Leiste
Phone +49 721 22541-22889, fax +49 721 608-22567, email harald.leiste@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Features

- Using of max. 3 magnetrons and 2 microwave plasma sources simultaneously
- Reactive gas components N2, C2H2, CH4, O2
- Multilayer and graded coatings and nanocomposites possible
- Typical targets: metals electrical, conductive ceramics
- No substrate rotation

Limitations/constraints

- Max. sample height 10 mm, max. sample diameter 75 mm
- Temperature range 100–900 °C
- Electrical conductive or non-conductive substrates
- Max. 3 conductive or non-conductive targets
- Directed coating; no inside coating of tubes or holes

Materials

Metal, ceramic

Typical structures and designs

Fig. 5: CVD/PVD-hybrid coating apparatus

Fig. 6: 3D-CAD sketch of a patented high performance plasma source
Atom Probe Tomography allows the three-dimensional imaging of a tip-shaped specimen atom by atom. Additionally, APT enables to determine the chemical composition on the atomic scale in an arbitrary analysis volume within the imaged sample volume. Typical APT specimens are sharp needles, which have to have apex diameters of about 100 nm or less, so that the surface atoms may get evaporated under a static high voltage (HV = 5-20 kV) and an additionally applied high-frequency HV or laser pulse. The time resolved and position sensitive detector analyses the type of the evaporated ions by Time-of-Flight mass spectroscopy. Combining the detector information, the specimen can be reconstructed three dimensionally with almost atomic resolution. Fields of applications include e.g. metals and semiconductors.

Contact

Dr. Julia Wagner  
Phone +49 721 608-26960, fax +49 721 608-26429, email julia.wagner@kit.edu  
Institute for Applied Materials IAM/WK - www.iam.kit.edu/wk/english

Features

- APT type: LEAP 4000X HR
- FOV: up to 250 nm

**Voltage Atom Probe**

- High Voltage: up to 20 kV
- Pulse frequency: up to 200 kHz

**Laser Atom Probe**

- Laser Wavelength: 355 nm
- Spot size: < 3 μm
- Pulsing frequencies: up to 250 kHz

**Available preparation techniques**

- FIB
- Electro Polishing (only conductive materials)

Limitations/constraints

- Spatial resolution (depth): 0.1 - 0.3 nm
- Spatial resolution (lateral): 0.3 - 0.5 nm
- Specimen dimensions:
  - sharp needles: length a few μm,
  - tip apex: < 100 nm.

Materials

- Metal, Semiconductors, Silicon
Since its invention in 1986 the atomic force microscope (AFM) is nowadays widely used for the inspection of sample surface down to the atomic-scale. The experimental set-up of an AFM is based on a simple idea. It detects forces acting between a sample surface and a sharp tip that is mounted on a soft leaf spring (the so-called cantilever). A feedback system, which controls the vertical z-position of the tip on the sample surface, keeps the deflection of the cantilever (and thus the force between tip and sample) constant. Moving the tip relative to the sample in the x–y-plane of the surface by means of piezoelectric drives, the actual z-position of the tip is recorded as a function of the lateral x–y-position with very high precision. The obtained data represent a map of equal forces which can be interpreted as the surface topography. The lateral resolution (x–y) depends on the radius of the tip which is between 10–20 nm. The vertical resolution (z) is typically better than 1 nm.

Contact
Dr. Hendrik Hölscher
Phone +49 721 608-22779, fax +49 721 608-24331, email hendrik.hoelscher@kit.edu
DI (FH) Richard Thelen
Phone +49 721 608-22727, fax +49 721 608-24331, email richard.thelen@kit.edu

Features
- Measurements in different modes and techniques
  - Contact mode
  - Dynamic modes (tapping, AM-mode, ...)
  - Friction force microscopy (FFM)
  - Magnetic force microscopy (MFM)
  - Electrostatic force microscopy (EFM)
  - Kelvon probe force microscopy (KPFM)
  - Nanolithography option
  - Nanoindentation option
- Large sample sizes (up to 8” wafer)
- Large scan ranges (up to 800 μm x 800 μm)
- Measurement in ambient conditions and fluids
- Combination of AFM and optical microscopy
- Sample heating and cooling during scanning (-30°C up to 200°C)

Limitations/constraints
- Sample should be smooth and flat
- Maximal sample height is restricted to 2 cm

Typical structures and designs
Nanolithography on a PMMA substrate
Magnetic signal measured on a 1.44 MB floppy disk
Auger electron spectroscopy (AES) is used to determine the elemental composition and, in many cases, the chemical state of the atoms in the surface region of a solid, vacuum stable, not insulating material. AES has found widespread use in an extensive variety of materials applications, especially those requiring surface specificity and high spatial resolution. The method is based on the Auger effect which is resulting from inter- and intrastate transitions of electrons in an excited atom. Because of the relatively low kinetic energy of the Auger electrons they can only escape from the uppermost few monolayers of a specimen surface. This is the reason why this technique is such surface sensitive. In combination with Argon ion sputtering depth profiles to 1000 nm are available without prior sample preparation. In many cases there is no complex and time-consuming sample preparation needed.

Contact
Tobias Weingärtner
Phone +49 721 608-22913, fax +49 721 608-922913, email tobias.weingaertner@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Features
- Resolution:
  - e-beam spot size at 10kV and 20nA < 24nm
  - depth resolution 0.5–5nm (depending on Auger electron energy)
  - energy resolution 0.5 to 0.1 %
- Floating column ion gun:
  - spot size 0.5 mm,
  - 10 - 3000eV Ar+ ion energy
  - charge neutralization possible
- Semi-quantitative analysis of Li to U; quantitative analysis with standards possible
- Practical detection limit 0.5 to 5 at% (depending on elements)
- Multi-point and area analysis, line scans, element maps, depth profiles
- Coaxial electron gun and analyzer geometry
- Compucentric Zalar RotationTM for better interface resolution
- Fracture stage with liquid N₂ cooling for in situ fractures (grain boundary analysis)

Equipment

Fig. 1: PHI 680 Xi Field Emission Scanning Auger Nanoprobe

Limitations/constraints
- Maximum sample size Ø 60 mm
- Sample has to be solid at RT and stable under vacuum conditions (10⁻⁹Torr)
- Depending on the chemical composition samples might be sensitive to the electron beam
Typical measurements

Fig. 2: Auger survey and SE image of Cu nano wires

Fig. 3: SE image

Fig. 4: AES element mapping

Fig. 5: AES line scan

Fig. 6: AES spectrum

Fig. 7: AES depth profile
For the chemical characterization of the bulk material for micro and nano materials five different analytical instruments are operated for the KNMF by the analytical group of the Institute of Applied Materials - Applied Material Physics (IAM-AWP):

- **X-Ray Fluorescence Spectrometry, XRF** (S4 Pioneer, Bruker-AXS)
- **Atomic Emission Spectrometry by Inductively Coupled Plasma, ICP-AES** (OPTIMA 4300 DV, Perkin-Elmer)
- **Mass Spectrometry by Inductively Coupled Plasma, ICP-MS** (7500ce, Agilent)
- **Carrier Gas Heat Extraction, CGHE** (TC 600, LECO)
- **Carbon-Sulfur-Analyzer** (CS 600, LECO)

**Contact**

Dr. Thomas Bergfeldt  
Phone +49 721 608-22914, fax +49 721 608-27715, email thomas.bergfeldt@kit.edu  
**Institute for Applied Materials (IAM-AWP)** - www.iam.kit.edu/awp/english

**Equipment**

**X-Ray Fluorescence Spectrometry, XRF**  
(S4 Pioneer, Bruker-AXS)

**Specification**
- Sequential wavelength dispersive X-ray spectrometer
- Detectable elements: (B) F to U in the concentration range from ppm to 100%
- Non-destructive analysis for qualitative and semi-quantitative determinations
- Sample forms like powder, solid, paste, film, liquid with size of 10 to 50000 μm
- Precise quantitative determination of samples prepared in fused borate beads or with polished surface

**Accessories**
- Grinding, pelletizing, fusion machines

**Typical samples**
- Precise determination of main compounds like Si, Ti, Al and other minor elements in nano powders of SiO₂, TiO₂, Al₂O₃, glass, other nano oxides/carbides/nitrides

*Fig. 1: S KA1. Analysis from a filter cake on a paper filter. Concentration range 0.01 – 3.0 mass %.*
Atomic Emission Spectrometry by Inductively Coupled Plasma, ICP-AES (OPTIMA 4300 DV, Perkin-Elmer)

**Specification**
- Echelle grating optical system combined with prisms and two segmented charge coupled device (SCD) detectors enables simultaneous measurement of all elements except noble gas, halogens, hydrogen, oxygen and nitrogen
- Element concentrations ranging from below 1 μg/g (depending on sensitivity) to 50% in solids and < 0.001 to 100 mg/L in liquids

**Accessories**
For solid samples all kind of dissolution techniques, i.e. microwave assisted digestion

**Typical samples**
Widely used, one of the most versatile methods of inorganic and organic analysis: liquids, electrolytes, dissolved solids of metals, oxides, nitrides, carbides

Fig. 2: Au with the ICP-OES by 242.795 nm. Chemical digestion from a mixture of Au and TiO₂ NM with aqua regia. Concentration range 0.005 – 0.20 mg/l

Mass Spectrometry by Inductively Coupled Plasma, ICP-MS (7500ce, Agilent)

**Specification**
- Quadrupole mass spectrometer with off-axis Omega lenses and Octopole
- Reaction System (ORS) to eliminate polyatomic interferences
- Mass range: 6–260 amu, He–U
- Ultra trace analysis ranging from below 1 ng/g (depending on sensitivity) to 1000 μg/g in solids and < 0.001 to 100 μg/L in liquids

**Accessories**
For ultra-trace analysis: sub boiling point distillation, laminar flow bench

Fig. 3: Consistent interference reduction in a variable, complex matrix using He mode. Comparison plots showing Std mode (no cell gas - red) and He mode (green) spike recovery data for 5 ppb Cr in a variable matrix (up to 1% each of HCl, H₂SO₄ and Butanol). Potential interferences on 52 Cr include Ar₂C, ClO₂H and SO.
Carrier Gas Heat Extraction, CGHE  
(TC 600, LECO)

**Specification**
- Simultaneous Nitrogen and Oxygen determination using IR and thermal conductivity by melting the sample in a graphite crucible in a metal bath at 2600°C with He as carrier gas
- Analysis range: < 0.00001 to 50%

**Typical samples**
Solids: metals, inorganic materials like oxides, nitrides etc.

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Carbon-Sulfur-Analyzer  
(CS 600, LECO)

**Specification**
- Simultaneous Carbon and Sulfur determination by combustion in a high frequency furnace in oxygen flow using IR-detection of CO₂ and SO₂
- Analysis range: < 0.0005 to 100%

**Typical samples**
Solids: metals, inorganic and organic materials

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**Fig. 4:**  
Different kind of oxygen bonds in steel

**Fig. 5:**  
C in WC

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**Sauerstoff ppm**  
1382.605

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**Kohlenstoff %**  
0.22273
The Zeiss Orion Plus allows high-resolution imaging with high surface sensitivity and a depth-of-field 5-10 times higher than in a modern FE-SEM. The focused helium ion beam can also be used for machining at the nanoscale with feature sizes 10-20 times smaller than achievable using a Focused Ion Beam (FIB). The tool is well suited to imaging challenging samples such as polymer-based systems and biological specimens without additional sample coating. For electrically insulating samples, positive charge resulting from the ion beam is compensated by using an electron flood gun directed at the sample. In addition, it is possible to locally deposit Pt, W, or SiO₂ from precursor gases using the helium ion beam.

**Contact**

Dr. Ruth Schwaiger  
Phone +49 721 608-24878, fax +49 721 608-22347, email ruth.schwaiger@kit.edu  
Institute for Applied Materials (IAM-WBM) - www.iam.kit.edu/wbm/

**Features**

- Resolution: ≤ 0.35 nm
- Magnification: 100 X – 1 000 000 X
- Field of view: 1 mm – 100 nm
- Beam current: 1 fA – 100 pA
- Acceleration voltage: 10 kV – 30 kV
- Detectors: Everhart-Thornley Secondary Electron Detector (ETD), Rutherford Backscattered Ion Detector (RBI)
- Charge compensation: Electron flood gun
- Base pressure: Chamber 2 x 10⁻⁶ Torr
- Stage: Tilt 0-45°, maximum travel in x and y ±25 mm from center
- Gas injection system for Pt, W, TEOS
- NanoPatterning and Visualization Engine (NPVE) for advanced patterning

**Application examples**

- 3D SURMOF (plan view and cross-sectional view, collaboration with H. Gliemann, IFG, KIT)
- Biofilms E. faecalis DSM 2570 (24 h incubation at 37°C) on Calgary Biofilm Device (CBD) (collaboration with P. Sanyal and A. Ulrich, IBG, KIT)
- Nanostructuring of Au nanowires (collaboration with O. Kraft, IAM-WBM, KIT)

**Limitations/constraints**

- Sample has to be a solid at room temperature, dry and stable under high vacuum conditions
- Maximum sample height 20 mm
- Samples should be very clean prior to imaging to minimize carbon contamination
In LA-ICP-MS conductive or non-conductive samples are directly analysed by ablating the material with an excimer laser. The aerosol is transported by Ar or He into the ICP from where the generated ions are extracted through an optional collision cell into a quadrupole mass spectrometer.

Contact
Dr. Thomas Bergfeldt
Phone +49 721 608-22914, fax +49 721 608-27715, email thomas.bergfeldt@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Equipment
- Excimer Laser (UP 193 FX) + Quadrupole
- ICP-MS (Agilent 7500ce)

Features
Excimer Laser UP 193 FX
- Spot size 2–150 μm, 1–100 Hz repetition rate, < 5 nsec pulse duration, > 3GW/cm²
- Point and multi-point analysis, linescans
- Depth resolution typically 1 μm
- Samples size > 60 mm

ICP-MS (Agilent 7500 CE)
- Li–Bk (6–260 amu), semi-quantitative, quantitative analysis by standards
- Concentration range: < 1 μg to 100 %
- Independent of sample geometry, < 0.005 to Ø 60 mm sample size

Typical use
- Major, minor and trace level compositional analysis of conductive, semi-conductive and non-conductive materials
- Contamination of plastics, pharmaceuticals, organics or biological materials
- Failure, contamination and inclusion analysis
- Mineral, glass and ceramics sample analysis

Typical results
Fig. 1: Example for a spot diagram. Laser start after 30 sec with 0.4 L/min He and 0.84 ml/min Argon. Material Li₂B₄O₇ and Mn₃O₄.
Single crystal X-ray diffraction is a method of determining the arrangement of atoms within a crystal, in which a beam of X-rays strikes a crystal and causes the beam of light to spread into many specific directions. From the angles and intensities of these diffracted beams, a crystallographer can produce a three-dimensional picture of the density of electrons within the crystal. From this electron density, the mean positions of the atoms in the crystal can be determined, as well as their chemical bonds, their disorder and various other information.

The data will be measured with a new STOE StadiVari goniometer which is equipped with an ultra-fast and sensitive DECTRIS PILATUS pixel detector (300 K) and two microfoci X-ray sources (Cu- and Mo-radiation). The special characteristics of the detector (ultrafast readout and almost zero background) in combination with high power X-ray sources offer new dimensions in time and data quality especially in the field of molecule crystallography. The attached low temperature device allows for measurements of the crystals from 120 to 350 K in a stream of nitrogen. Required size of the single crystals: between 0.02 and 0.2 mm. The smallest dimension of the crystal should not go below 0.02 mm.

Contact

Dr. Andreas Eichhöfer
Phone +49 721 608-26371, email andreas.eichhoefer@kit.edu
Institute of Nanotechnology (INT) - www.int.kit.edu/english
IFP’s soft x-ray analytics facility WERA at ANKA provides a coherent combination of electron spectrosopies and microscopies for studying in detail the chemical (electronic) and magnetic structure of bulk materials, thin films, and micro- and nanostructured objects.

Contact
Dr. Stefan Schuppler
Phone +49 721 608- 24631, email stefan.schuppler@kit.edu
Dr. Peter Nagel
Phone +49 721 608- 26560, email peter.nagel@kit.edu
Dr. Michael Merz
Phone +49 721 608- 26635, email michael.merz@kit.edu


Equipment for Electron spectroscopy and spectromicroscopy:
XAS, PES (XPS), SXMCD, µ-XAS, µ-PES (±-XAS), µ-SXMCD, topography

- Three experimental stations at WERA equipped with PEEM, electron energy analyzer, detectors, cryostats, etc.
- Sample preparation chambers, loadlocks, in-vacuo sample transfer

Features
The excitation (photon) energies are in the soft x-ray range from 100 –1500 eV, which is especially well suited for studying the light elements (like oxygen), the 3d transition metals, and the 4f rare-earth elements at their particularly informative K, L, and M edges, respectively. In the soft x-ray range, radiation-induced damage in, e.g., carbonaceous materials is orders of magnitude less than with electron bombardment (like for EELS in TEM). The photon energy resolution $\Delta E/E$ can be chosen as low as $10^{-4}$.

The main instrument for KNMF applications is the photoemission electron microscope (PEEM). Using electron optics with great magnification, the lateral distribution of electrons emitted from the illuminated spot on the sample is imaged to an intensifier and CCD camera. The lateral resolution in PEEM can be better than 30 nm (100 nm in spectromicroscopy), and this as well as the variable probing depth of <1 to about 10 nm, depending on technique, is well matched to many nano- and microstructured materials and their typical length scales. Generally, the methods are element-specific.

Fig. 1:
Part of the cluster of experimental stations, preparation chambers, and loadlocks at WERA. The PEEM is located near the center.
Two main modes are used:

Imaging of chemical (electronic), magnetic, and topographic contrast. The field of view can be chosen from 250 µm down to about 20 µm. Using the sample translation stage, a total sample area of up to about 10 mm diameter can be studied.

Spectromicroscopy: this is an especially powerful PEEM application: by taking stacks of images while tuning the photon energy or the kinetic energy of the detected electrons, laterally resolved sets of x-ray absorption (µ-XAS) and photoemission (µ-PES, µ-XAS) spectra, resp., are efficiently obtained and can be further analyzed, see figure 2. Field of view and accessible sample area are the same as in (1). The polarization of the synchrotron radiation is a further useful variable: with circular polarization, for instance, ferromagnetic domains become visible ("magnetic dichroism", µ-SXMCD), see figure 3, and the associated spectromicroscopy gives element-specific magnetic information such as on spin and orbital magnetic moments. Linear polarization of the incident light makes the experiment sensitive to parameters like molecular and bond orientation.

In addition, WERA comprises further, complementary stations for XAS, PES (XPS), and SXCMCD. There, the signal is averaged over the illuminated spot on the sample (typically 1 x 0.5 mm2). This allows higher energy resolution and even greater flexibility in detection methods, polarization, and sampling depth (including fluorescence detection for bulk-sensitive XAS measurements). The sample environment includes temperatures between 15 and 800 K and high magnetic field (currently 2 T).

A number of sample preparation chambers and loadlocks are part of WERA. All are interconnected with the experimental chambers by an in-vacuo sample transfer system enabling the combined investigation with methods in different chambers without the need to break the vacuum. The available preparation and characterization methods include pulsed-laser deposition, evaporation (Knudsen cell), sputtering, annealing in vacuum or gases including oxygen, LEED, and RHEED. Compatibility with ANKA’s NanoLab and the future KNMF laboratory at ANKA will ensure an even wider range of possibilities for preparation and characterization.

All methods at WERA are embedded in both the KNMF and the ANKA user landscape. The WERA personnel has a strong commitment to user support. Please discuss your application with us.

WERA is constantly being upgraded and expanded. Major upgrades include: (i) an undulator as a second, alternative light source for experiments needing higher photon flux density on the sample; and (ii) expanding the magnetic fields available for SXCMCD to 7 T.

Limitations/constraints

All experiments are performed in ultrahigh vacuum at pressures in the 10⁻¹⁰ mbar range. Samples should be compatible with this.
Typical samples

Fig. 2:
Nanolithography of phospholipids by the dip-pen method (see KNMF Laboratory for Micro- and Nanostructuring). The written triangles are 10 µm wide. The increasing admixture of nickel-chelating lipids (with a single Ni atom per lipid macromolecule) to the carrier appears in this Ni-sensitive, spectroscopic image as the increasingly red-yellow colors visible from bottom to top. Quantitative analysis reveals that for the lower four triangles, the admixture ratios observed in the written patterns directly track the original values. For the top triangle, some demixing seems to set in, leading to a smaller than-expected fraction of Ni-chelating lipids actually arriving in the written pattern. (Cf. S. Sekula et al., Small, 2008).

Fig. 3:
Magnetic domains and possibly defect-related subdomains with various orientation are visible in this magnetic dichroism image of thin film permalloy squares.
Thin Film Characterisation Methods

(1) Hardness/adhesion

The film hardness can be determined by Vickers indentation with a diamond tip. The geometry of the indentation is measured by an attached optical microscope and delivers the dimensions of indentation and therefore the value of hardness.

A diamond stylus (Rockwell geometry) is scratching with linear increasing load over the coated surface. The load value at which the film begins to fail can be defined. This value is a criterion for the film adhesion. In order to optimize the substrate-film system the origin of the failure can be evaluated by the type of failure, film failure or interface failure.

Contact

Dr. Harald Leiste
Phone +49 721 22541-22889, fax +49 721 608-22567, email harald.leiste@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Features

- Vickers hardness material independent
- Scratch test – for brittle thin film material

Limitations/constraints

- Microhardness indentation load 5 g > L > 400 g
- Critical load of failure Load 0 < L < 200g
- For hardness determination the indentation depth must be 1/10 or lower of the film thickness

Materials

Metals, alloys, ceramic, oxides, nitrides, carbides, glass
Thin Film Characterisation Methods

(1) Hardness/adhesion (continued)

Typical structures and results

Indentation for the determination of the hardness (load 50 g)

Trace of the scratch test for the determination of the critical load of failure
Thin Film Characterisation Methods

(2) Nanoindentation

Nanoindentation is the most important method for the nanoscale measurement of mechanical properties (hardness, elastic modulus, fracture toughness) of surfaces, thin films and small volumes of material. A pyramidal diamond tip is pressed into the specimen as an indenter and removed again after reaching a maximum load. During this process, the load and the penetration depth of the indenter are recorded. This load/penetration depth curve represents a “fingerprint” of the mechanical properties averaged over a certain volume area that increases with increasing load. The curve fitting allows the extraction of the mechanical properties.

Contact

Dr. Harald Leiste
Phone +49 721 22541-22889, fax +49 721 608-22567, email harald.leiste@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Features

- Force range: 10mN – 10N
- Optical system max magnification 2000x
- Min distance between indents < 250nm
- Recording of depth profiles of hardness and elastic modulus possible after special sample preparation by nanogrinding

Materials

- Polymer, metal, ceramic, glass, silicon, organic

Limitations/constraints

- The surface roughness is a crucial parameter and should be < 10nm
- Indentor geometry Vickers
- Depth resolution 0.3nm
- For thin films on substrates the substrate influence on the hardness can be neglected if the indentation depth is < 1/10 of the film thickness
- For thin films on substrates there is practically no minimum indentation depth to fully exclude the substrate influence on the reduced elastic modulus
**Thin Film Characterisation Methods**

(1) Hardness/adhesion
(2) Nanoindentation
(3) Plasma diagnostics and particle flux analysis
(4) Raman spectroscopy
(5) Film thickness
(6) Magnetometer (VSM) and high frequency permeameter
(7) X-ray diffraction XRD/XRR

**Typical structures and results**

- **Hardness depth profile of a 20-layer TiN/ZrN nanolaminated composite coating recorded at 2 mN load by small-angle cross section method (SACS)**

- **Dependence of hardness and reduced elastic modulus of 100 nm thick c-BN:O films on the oxygen flow rate measured at 700 μN load**

- **AFM topography image of eutectic Al₂O₃-ZrO₂ region in a laser-modified alumina ceramic showing nanoindents (maximum load: 1 mN; M = Al₂O₃ matrix, A =Al₂O₃ lamella, Z = ZrO₂ lamella)**

- **Load–displacement curve and AFM image (insert) of the cracking of a TiAIN single-layered coating for the calculation of fracture toughness**
(3) Plasma diagnostics and particle flux analysis

Low pressure plasmas can be characterized by electrical single and double probes and optical emission spectroscopy. The particle fluxes onto the substrates can be determined by Faraday cup as well as retarding field analyzer. Based on the measurements the following physical quantities are calculated: electron temperature Te, plasma density ne, ion and electron current density jion and je, plasma potential Upl, ion energy Eion, energy distribution of ions f(Eion).

Contact
Dr. Harald Leiste
Phone +49 721 22541-22889, fax +49 721 608-22567, email harald.leiste@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Features
- Faraday cup/retarding field analyzer: 0–1000 eV
- Single- and double probe: plane and cylindrical
- Optical emission spectrometer: 200 –800 nm

Limitations/constraints
- Height: 50 mm, diameter: 80 mm
- Temperature range: RT–200 °C
- KF40 flange utilizable
- The whole system is transportable

Typical results
Current–voltage-characteristic of a double probe measurement (r.f. magnetron argon plasma)

Energy distribution of Ar-ions during film growth by magnetron sputtering
(3) Plasma diagnostics and particle flux analysis (continued)

Typical setups

Retarding field analyzer for measurement of plasma potential, ion energy and energy distribution ions

Micropole mass spectrometer mounted on KF16 flange
(4) Raman spectroscopy

By laser excited Stokes Raman scattering the chemical bonding and microstructure of compact, powder and thin film materials are evaluated. In solid state physics, Raman spectroscopy has become an important method to investigate carbon species and a-C: metal nanocomposites. Raman spectroscopy is also suitable for the microscopic examination of minerals, polymers and ceramics, but also of cell and proteins. Recently, the Raman spectroscopy could be used for measuring concentration profiles within micro channels.

Contact

Dr. Harald Leiste
Phone +49 721 22541-22889, fax +49 721 608-22567, email harald.leiste@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Features

- Backscattering geometry
- Excitation with Argon-Ion-Laser (514.5 nm)
- Excitation with Helium-Cadmium-Laser (325 nm)
- Holographic grating 2400 grooves mm\(^{-1}\)
- UV-enhanced CCD detector with thermo-electric cooling

Limitations/constraints

- Spectral resolution 1–2 cm\(^{-1}\)
- Spectral range (Raman shift) < 200–4000 cm\(^{-1}\)
- Spatial resolution 2 μm lateral (× 50 objective)
- Confocal mode possible
- Typical sample: flat, thin film, fine powder

Materials

Metal, ceramic, glass, silicon, polymer

Typical setup

Optical arrangement for Micro Raman spectroscopy
(4) **Raman spectroscopy** (continued)

**Typical results**

Raman spectra of six samples with different Ti:C ratios deposited using -150 V substrate bias voltage

Characterisation of LiCoO₂ phases
Thin Film Characterisation Methods

(5) Film thickness

The film thickness can be determined by calo-test, where a spherical calotte will be polished into the surface. The sectional view diameters provide, together with the geometry of the grinding sphere, the value of the film thickness. By the use of a surface profilometer (Type Tencor P10), the roughness, waviness, step size of uncoated/coated regions and curvature of bending can be measured by the stylus method with a diamond tip.

Features

- Calo-test is a local destructive method
- Roughness values like Ra, Rt, Rz will be given

Limitations/constraints

- Calo-test film thickness 500 nm < t < 5 μm; min. film area 1mm
- Profilometer film thickness 20 nm < t < 5 μm; max. scan length 150 mm
- Resolution and accuracy depends on roughness

Materials

Metal, ceramic, glass, polymer

Contact

Dr. Harald Leiste
Phone +49 721 22541-22889, fax +49 721 608-22567, email harald.leiste@kit.edu

Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Typical results

A calotte polished into a coated substrate

Surface profile of a partial coated substrate
Thin Film Characterisation Methods

(6) Magnetometer (VSM) and high frequency permeameter

For the characterisation of ferromagnetic thin films properties especially the determination of magnetic hysteresis behaviour a vibrating sample magnetometer can be used to determine the saturation magnetisation $M_s$, the coercivity $H_c$, permeability $\mu$ and anisotropy $H_a$. The high frequency permeability with real and imaginary (damping) part can be determined by the use of a coplanar measuring head in a frequency range of $30 \text{ kHz} < f < 6 \text{ GHz}$.

Contact
Dr. Harald Leiste
Phone +49 721 22541-22889, fax +49 721 608-22567, email harald.leiste@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Features
- Vibrating sample magnetometer for thin film samples (max field 600 mT)
- High frequency permeameter $30 \text{ kHz} < f < 6 \text{ GHz}$

Limitations/constraints
- Vibrating sample magnetometer: Min film thickness min 50 nm
- Vibrating sample magnetometer typical sample: flat, thin film, size $5 \times 5 \times 0.5 \text{ mm}^3$
- High frequency permeameter, sample size $5 \times 5 \times 0.3 \text{ mm}^3$

Materials
Ferromagnetic thin films on silicon, ceramic, glass
(6) Magnetometer (VSM) and high frequency permeameter

(continued)

Typical setups and results

Vibrating sample magnetometer (VSM)

The high frequency permeameter consists of a strip-line measuring head on an attached vector network analyzer

Thin Film Characterisation Methods

1. Hardness/adhesion
2. Nanoindentation
3. Plasma diagnostics and particle flux analysis
4. Raman spectroscopy
5. Film thickness
6. Magnetometer (VSM) and high frequency permeameter
7. X-ray diffraction XRD/XRR

Magnetic hysteresis of a thin ferromagnetic thin film with uniaxial anisotropy (easy axis of magnetisation (black) and hard axis of magnetisation (red))

Frequency dependent permeability; real part (black) and imaginary part (blue)
(7) X-ray diffraction XRD/XRR

By means of X-ray radiation (Cu-cathode) the microstructure of powder, compact and thin film material can be measured and evaluated. Different geometries, and circle movements (Bragg-Brentano-geometry, GID, texture, X-ray reflectivity) can be realized. It can be decided if the material is amorphous or crystalline, and the lattice parameters can be determined from the line positions.

Contact
Dr. Harald Leiste
Phone +49 721 22541-22889, fax +49 721 608-22567, email harald.leiste@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Features
• Bragg-Brentano
• Texture
• XRR-characterisation
• RSM
• Cu-X-ray tube
• Optional monochromator
• Compact material, thin films, powder samples

Limitations/constraints
• Resolution 1/1000°
• Max. sample load 5 kg
• Min. beam Ø 2mm
• XRR typical sample: flat, thin film, size 20x20x1mm³
• Min sample size 5x5mm²
• Max sample size Ø 150mm x 20mm

Materials
Metal, ceramic, bulk, powder, thin films

Typical setup
4 circle goniometer for XRD characterization (Seifert 3003 HR)
(7) X-ray diffraction XRD/XRR
(continued)

Typical results

Bragg-Brentano diagram of Al2O3-sample

XRR-diagram of a thin film for density determination

Texture diagram of TiN thin film

Thin Film Characterisation Methods

(1) Hardness/adhesion
(2) Nanoindentation
(3) Plasma diagnostics and particle flux analysis
(4) Raman spectroscopy
(5) Film thickness
(6) Magnetometer (VSM) and high frequency permeameter
(7) X-ray diffraction XRD/XRR
Time-of-Flight Secondary Ion Mass Spectrometry is available only in some 100 industrial and academic laboratories worldwide. It is – complementary to XPS – a surface analysis technique providing chemical and molecular information of topmost layers at high spatial resolution. A focused high energy ion beam is rastered across the surface of the sample releasing characteristic fragments of the material to be analyzed. Secondary ions are mass separated and counted resulting in a mass spectrum of the sample (information depth approx. 1-2 nm). The lateral distribution of chemical functionalities can be obtained by rastering the primary beam and the sample itself. ToF-SIMS is ideally suited for the analysis of polymers, organosilanes or thiol self assembled monolayers, as well as surfaces from technical applications and environmental studies. Depth profiling and 3D imaging is performed by applying a sputter ion source eroding the sample with Cesium, Oxygen, or C_{60} ions. Charge compensation on insulating samples is facilitated by an electron flood gun.

Several data processing tools allow for the analysis of complex sample chemistries. These approaches include principal component analysis of the multidimensional spectra or images and “gentle-SIMS” to correct for some fragmentation effects.

Contact

Dr. Michael Bruns
Phone +49 721 608-22641, fax +49 721 608-922641, email michael.bruns@kit.edu
Institute for Applied Materials (IAM-ESS) - www.iam.kit.edu

Dr. Alexander Welle
Email alexander.welle@kit.edu
Institute of Functional Interfaces (IFG) - www.ifg.kit.edu

Equipment

ToF.SIMS\textsuperscript{5}-100, ION-TOF GmbH, equipped with a liquid metal cluster ion source, and several sputter sources.

Features

- Bi/Mn Source (Bi\textsuperscript{+}, Bi\textsuperscript{3+}, Bi\textsuperscript{5+}, Mn\textsuperscript{+})
- Mass resolution: up to 11000 m/\Delta m @ 29 amu (bunched mode)
- Spatial resolution < 150 nm (collimated mode)
- Surface sensitivity < 1 nm
- Cs thermion source and O\textsubscript{2} EI source for sputter depth profiling, Zalar-rotation possible
- C\textsubscript{60} EI source for analysis and sputter depth profiling of organic samples
- Transfer vessel for atmosphere contact free sample transport from glove boxes to the spectrometer
- Sample heating and cooling in UHV
- Max sample size: 6×7 cm

Limitations/constraints

All elements and isotopes are detectable, the sample has to be solid at RT and stable under vacuum conditions, powders are possible. Most biological samples require fixation, (freeze-) drying or other preparations. Quantification requires standards or calibration based on complementary techniques available within the KNMF. Detection limits: ppm of a monolayer for elements, sub-fmol for molecules. Dynamic SIMS is destructive because particles are removed from the surface.
Typical results

High mass range of a positive polarity secondary ion spectrum of a porphyrin derivative immobilized via silanol groups onto a silicon wafer (Bi$^{+}$).

The multipletts reproduce the isotope distribution of this molecule and can be unambiguously assigned to the porphyrine headgroup, C$_{54}$H$_{58}$N$_{5}$, etc.

Imaging of C$_{2}$H$_{3}$O$_{2}^{-}$ groups demonstrating the local UV light induced photo oxidation of poly(9,9-dioctylfluorenyl-2,7-diyl) used a light emitting polymer for OLED devices. Stage scanning for large field of view.

Dual beam depth profiling (C$_{60}^{+}$/Bi$^{+}$) of a polymer sample produced according to merrifield peptide synthesis. The depth distribution of triphenylmethyl protecting groups is shown by the 243 m/z signal (blue).
Transmission electron microscopy (TEM) enables characterization of powders and thin films (which can be prepared in a target preparation from bulk materials) by direct imaging with up to atomic resolution. The image information can be locally correlated with spectroscopic techniques (EELS/EFTEM and EDX) to provide semi-quantitative elemental composition/maps with sub-nanometer resolution. All of these techniques can also be performed in-situ, e.g. during heating, electrical biasing or straining to directly correlate structural changes and materials properties. For complex three-dimensional structures, electron tomography can be used to generate a 3D representation of the material with a spatial resolution of 1–2 nm.

Contact
Dr. Christian Kübel
Phone +49 721 608-28970, fax +49 721 608-28976, email christian.kuebel@kit.edu
Institute of Nanotechnology (INT) - www.int.kit.edu/english

Features
- FEI Titan 80–300 (aberration corrected TEM)
- Resolution:
  - 0.08 nm information limit TEM
  - 0.14 nm resolution in STEM
  - 0.7 eV energy resolution EELS
- Imaging and Analysis Techniques:
  - BF-TEM, aberration corrected HRTEM
  - HAADF-STEM, HRSTEM
  - EFTEM, EELS, EDX
  - (S)TEM tomography
  - electron diffraction, electron precession
  - orientation mapping
  - Lorentz imaging
  - low-dose techniques & cryo imaging
- In-situ Techniques:
  - Heating (Protochips Aduro: RT-1200 °C; Gatan 652: RT-800 °C)
  - Cooling (Gatan 915: LN2-80 °C)
  - Straining (Hysitron Picoindenter PI 95 and Gatan 654)
  - Electrical Biasing (Protochips Aduro)
  - Electro chemistry (Protochips Poseidon 500)
- Sample preparation:
  - Thin films or nano powders can be directly analyzed without additional preparation
  - Target preparation by FIB lift-out (for details see FIB description) with final polishing by low-voltage Argon ion beam (Fischione 1040 NanoMill)
  - Electro polishing
  - Classical preparation by cutting, grinding, argon ion milling or microtomy

Limitations/constraints
- Sample has to be a solid at LN2 temperatures and stable under high vacuum conditions
- Maximum sample thickness: 10–2000 nm (depending on resolution and technique)
- Depending on the structure and chemical composition, the sample might be sensitive to the electron beam resulting in changes during analysis
- Except in tomography, TEM always provides an image/analysis of the projected structure of a sample
- H, He und Li cannot be detected by our analytical techniques
Typical results

Fig. 1: HAADF-STEM image (filtered by NAD) of a La$_x$Sr$_{1-x}$MnO$_3$/SrTiO$_3$ interface with the individual atomic columns well resolved across the interface. Overlaid is an EELS/EDX intensity profile across this interface. P.M. Leufke and D. Wang et al., Thin solid films, 2012, 520, 5521-5527.

Fig. 2: Atomic resolution TEM image of a triple and a quadruple line at the interface between $\Sigma_3$ boundaries and a $\Sigma_9$ boundary in nanocrystalline palladium. H. Rösner and C. Kübel et al., Acta Mat., 2011, 59, 7380-7387.

Fig. 3: Geometric phase analysis reveals the local strain distribution around the triple line in the image above. H. Rösner and C. Kübel et al., Acta Mat., 2011, 59, 7380-7387.

Fig. 4: In-situ orientation mapping (different color correspond to different crystal orientations) of the grain structure changes in nanocrystalline gold during straining – selected images of the straining series showing anomalous grain growth. A. Kobler and C. Kübel et al., Ultramicroscopy, 2013, 128, 68-81.
Typical results (continued)

Fig. 5: EFTEM mapping (Si-blue, C-red) and HRTEM image of nanocrystalline silicon particles with a covalently bound C18 shell. The EFTEM maps reveal the ~1.2 nm wide carbon shell around the silicon core. Sample provided by G. Ozin, University of Toronto.

Fig. 7: HRTEM image of nano graphene with the corresponding low-loss EELS spectrum showing the characteristic $\pi$ and $\pi+\sigma$ plasmon losses. J. Biener and D. Wang et al., Adv. Mater. 2012, 24, 5083–5087.

Fig. 6: HAADF-STEM image with EDX compositional mapping of the different layers in a silicon quantum dot based organic LED (SiLED). F. Maier-Flaig and C. Kübel et al., Nano Letters, 2013, online; DOI: 10.1021/nl400975u.

Fig. 8: HRTEM image of a Fe/LiF/C anode for lithium ion batteries revealing $\alpha$-iron nanoparticles each surrounded by a few graphene layers. R. Prakash and C. Kübel et al., J. Power Sources, 2011, 196, 5936-5944.
Typical results (continued)

Fig. 9: Electron tomographic reconstruction of a self-assembled CdS nano cluster superlattice (with additional 5 nm gold particles in yellow). The two digital slices, one unit cell apart, show a single vacancy, an extended vacancy and dislocations in 3D. T. Levchenko and C. Kübel et al., Chem. Eur. J., 2011, 17, 14394-14398.

Fig. 10: HAADF-STEM and HRTEM imaging of uniform ThO$_2$ nanorods. D. Hudry and E. Courtois et al., Chem. Eur. J, 2013, 19(17), 5297–5305.
Matrix assisted laser desorption ionization (MALDI) or electrospray ionization (ESI) allows the soft ionization and transfer of analytes to the gas phase. Time-of-flight (TOF) mass spectrometric analysis provides high-resolution, exact mass measurement and accurate isotope distributions of positive or negative ions for identification. This allows for chemical identification of intact molecular and cluster species, transferred from solution (ESI) or a solid matrix (MALDI).

Recent advances in technology have led to the efficient coupling of ion-mobility analysis with high resolution mass spectrometers. Ion mobility spectrometry (IMS) provides a means to separate ions based on their shape and size, providing complementary information to that obtained via standard mass spectrometry. The Synapt-G2 HDMS is the first commercial instrument of its kind, offering flexibility in terms of ion source (MALDI, nanoESI, ESI) and sample analysis, coupling a high resolution TOF mass analyzer with a travelling wave ion mobility (TWIMS) separation cell. Thus, the instrument has both the capability to obtain high resolution ESI-/MALDI-TOF mass spectra, or, with application of IMS, to obtain 2D structure-mass correlation maps. Ions of interest may also be mass selected in a quadrupole mass filter and have their individual chemistry (e.g. via collision induced dissociation) probed.

Contact
Dr. Nicole Rijs
Phone +49 721 608-26416, email nicole.rijs@kit.edu
Dr. Jean-François Greisch
Phone +49 721 608-26975, email jean-francois.greisch@kit.edu
Institute of Nanotechnology (INT) - www.int.kit.edu

Features
- ESI/nanoESI/1kHz MALDI ion sources
- High resolution mass spectra
- 32kDa expanded mass range
- Analysis of positive and negative ions, ion chemistry
- TWIMS separation cell

Limitations/constraints
- Lower mass detection limit of: 100
- Spatial resolution of MALDI: currently 100 µm
- nanoESI/ESI: samples must be soluble.
- Air sensitive samples- the ion source is open to atmospheric conditions

Typical results
Negative-ion electrospray mass spectrum obtained from a solution of Na-Au₄Pd₈ in H₂O/DMSO. The two insets highlight peaks B and B’ and compare their isotope distribution to the simulated ones for the molecular ions as labelled.
Typical results (continued)

(a) Schematic structures of the two possible stacking configurations for $\text{Tb}_2(\text{A}_3\text{B})_2\text{Pc}$ complex: a symmetric one (on the left) and an asymmetric one (on the right).

(b) Calculated structure of $(\text{A}_3\text{B})_2\text{PcTb}_2$ (symmetric stacking)

(c) IMS arrival time distribution of the anion of $\text{Tb}_2(\text{A}_3\text{B})_2\text{Pc}$ indicating the presence of two different isomers.
X-ray photoelectron spectroscopy (XPS) is the most widely used surface analysis technique to provide both quantitative atomic concentration and chemical state information of the detected elements. X-ray irradiation of surfaces results in the emission of photoelectrons whose energies are characteristic of the elements. The information depth is approximately 5–7 nm. Angle-resolved XPS offers non-destructive resolution of structures within the XPS sampling depth, e. g. layer ordering, composition and thickness can be determined. Moreover, XPS can be utilized for sputter depth profiling to characterize thin films and multi layer systems by quantifying matrix-level elements as a function of depth.

Contact
Dr. Michael Bruns
Phone +49 721 608-22641, fax +49 721 608-922641, email michael.bruns@kit.edu
Institute for Applied Materials (IAM-ESS) - www.iam.kit.edu

Features
K-Alpha XPS Spectrometer
- Mono AlKα X-ray source, spot size 30–400 μm (spatial resolution)
- Energy resolution < 0.5 eV FWHM Ag 3d5/2
- Ion gun for sputter depth profiling, 100–3000 eV Ar+ ion energy
- Charge neutralisation system
- 50 x 60 mm² sample stage, sample height max. 15 mm
- Tilt stage enabling ARXPS
- Glove-box for atmosphere-contact free sample transfer: O2 < 1ppm, H2O < 1ppm

ESCA 5 / Alpha 110 analyser
- MgKα/AlKα dual anode X-ray source
- Energy resolution < 0.85 eV FWHM Ag 3d5/2 (MgKα)
- Ion gun for sputter depth profiling, 300–3500 eV Ar+ ion energy
- Max. sample dimensions 15 x 15 x 2 mm²
- In-situ sample cooling and heating (-190–500 °C)
- Fracture stage and T-peeler
- Additional methods:
  – UPS (UV photoelectron spectroscopy)
  – RGA (residual gas analysis, mass range 1–300 amu)

Limitations/constraints
- All elements are detectable except for H and He
- Sample has to be a solid at RT and stable under vacuum conditions, powders are possible
- Depending on the chemical composition samples might be sensitive to X-ray irradiation

Typical results

Non-destructive ARXPS concentration depth profile of an Al-Si-oxide membrane

C 1s XPS spectra from PS surfaces after laser modification in O2 atmosphere