Karlsruhe Institute of Technology

KNMF Installations

August 2019

Facilitating innovation
in advanced multimaterial
micro and nanotechnologies

Thomas Schaller
KNMF User Office

KIT – The Research University in 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.

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Karlsruhe Institute of Technology

Technologies

KNMF Laboratory for Micro- and Nanostructuring

- 3D Direct Laser Writing (3D-DLW)
- 3D Printing (3DP)
- 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)
- Surface-Anchored Metal-Organic Frameworks (SURMOFs)

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)
- Single Crystal X-ray Diffraction (SCXD)
- Soft X-ray Spectroscopy, Microscopy, and Spectromicroscopy (WERA)
- 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)

3D Direct Laser Writing

3D Direct Laser Writing is a tool to fabricate 3D freeform structures down sub µm. It is based on *Two-Photon Lithography* 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, *e.g.* polymerize, a photosensitive medium at a specific point in the resist. By scanning the photoresist with a stage over this point a 3D-structure with dimensions in the submicron scale or greater can be written.

Contact

See KNMF website or contact the KNMF User Office.

Equipment

- Nanoscribe Photonic Professional GT
  - Several sample holders
  - Galvoscan unit
  - Hybrid stage for large accurate travel distances
- Critical point dryer (supercritical CO_2)
- Coming soon: UV flood exposure (shell writing mode for large structures)

Features

- Resolution:
  3D:200 nm lateral, 750 nm normal
  2D: 180 nm
- Feature Structure size: max. 600 x 600 x 3700 µm³ depending on filling factor
- Writing modes:
  piezo (100x DIP), galvoscan (63x GT, 25x GT)
- Corresponding writing fields: 300 x 300 x 300 µm³; 140 x 140 µm²; 280 x 280 µm²
- Writing times: piezo slow, galvoscan fast
- Larger areas have to be stitched
- Accessible writing area: 100 x 100 mm² where structures could be placed

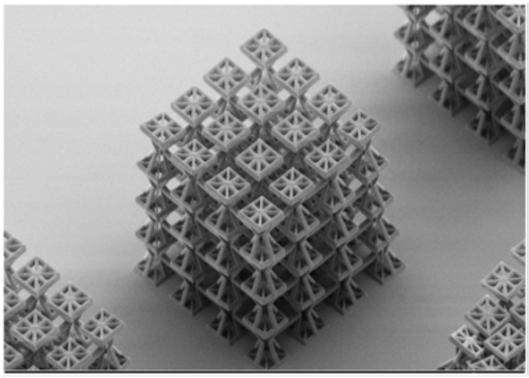
Limitations/constraints

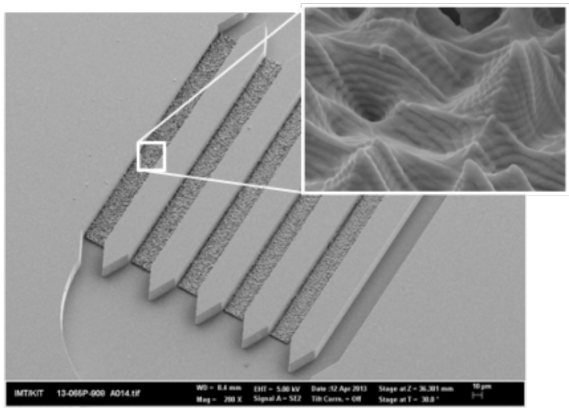
- The realizable structure size depends on the structural stability of the design
- The best results can be reached by using the IP-resists

Materials

- Negative / positive / experimental resists
  Details and substrates see next page

Sample structures





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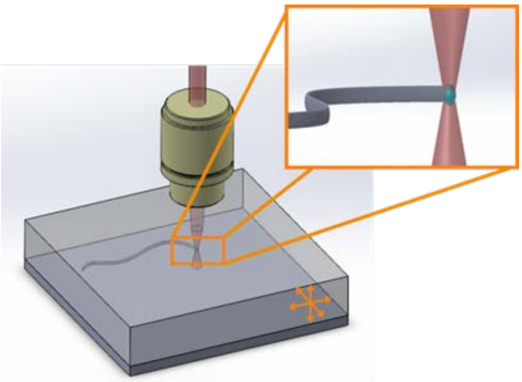
3D Direct Laser Writing

Writing principle



Sketch of the writing of a line in 3D-space inside a resist layer. The inset shows the modification in the voxel (blue) at the focal position. Only in the voxel two-photon absorption occurs.

Writing modes

- structure height
- Fixed thin substrate

- any resist usable

- Higher AR of voxel
- Bigger voxel
- Bigger processing effort

- Only solid resist

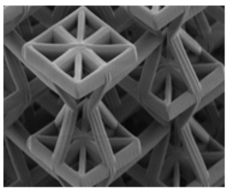
- no transparent substrate needed
- High working distance

- Not every resist usable
- Special glass substrates
- Small working distance

- structure height
- Very small voxel

- Special glass substrates
- no vertical lines possible

- 100x faster than other writing modes



Resists

- Negative Resists: IP-L, IP-G, IP-Dip, IP-S, and similar resists that are photosensitive at a wavelength of 380nm
- Positive Resist: AZ9260 (in preparation)
- Experimental Resists are possible, but only the Air mode objective or oil-immersion Objective are applied. Dip-in techniques can only be used with proven compatibility.

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3D Direct Laser Writing

Substrates

- 25 x 25 x 0.7 mm Glass, glass covered with ITO, cover slides 22 x 22 x 0.17 mm
- Si-wafer 4'' (100 mm)
- Si/SiO2-wafer 4'' (100 mm)
- Metallized Si-Wafer (Cr/Au)
- Other substrates have to be provided by the user.

Data

- Stl-format
- CAD data can be exported in stl-format:
- Sometimes CAD programs export erroneous stl-files. Solid works and similar programs used in mechanical engineering produce correct stl.files.
- To avoid errors within stl-files the following rules can help:
  - Use correct units during contruction (µm), otherwise resolution could be bad
  - Use volumes, no areas, these objects have to be closed
  - Avoid Boolean operations
  - Avoid duplexes (use snaps)
  - Think about plane orientation. Normals are used to define interior or exterior. Therefore contruct plane consistently.
  - Avoid additional structures not necessary (not written) to your part. Correction of the stl-file is nearly impossible
  - In case of periodic structures a single unit cell is sufficient.

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3D Printing

3D Printing (3DP) provides a wide range of possibilities for the realization of freeform 3D structures. Recent developments allow for a wide range of materials to be processed, from pure polymer materials to highly filled composites for creating metal parts. In the FDM process, the material of choice is provided as a filament and molten in a hot end before deposition on the building platform layer by layer.

Contact

See KNMF website or contact the KNMF User Office.

Equipment

FDM-Printers

- Stratasys uPrint SE Plus
- Leapfrog Xeed
- Leapfrog Bolt
- zMorph 2.0 SX
- Makerbot Replicator 2x

3D Scanning

- Einscan Pro (Accuracy up to 0.05 mm)

Filament fabrication

- Filafab Extruder & Winding

Coming soon: 3D Inkjet Printing with dual printheads (multimaterial inkjet)

Features

- Resolution: 50 µm (z-axis), 200 µm (x/y-axis)
- Accessible printing area: 300 mm x 320 mm x 205 mm
- Dual printheads (two build materials or one build/one support material)
- Printhead temperature up to 250 °C
- Graded structures possible (see Samples)

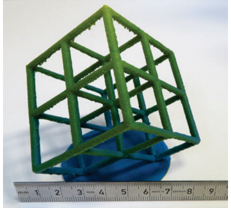
Limitations/constraints

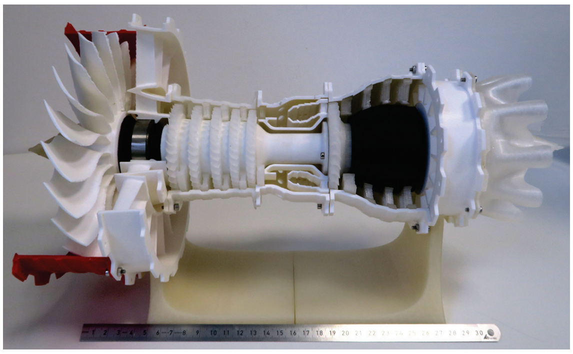
- Depending on object size & geometry: long build time
- No transparent objects possible
- Currently no materials with melting temperature >250 °C possible

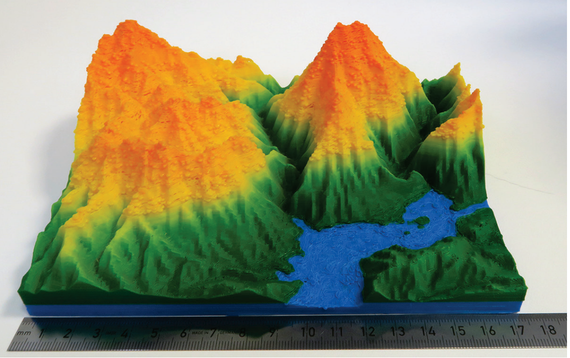
Materials

- ABS, PLA, PVA, Nylon
- Filled materials (optical effects, magnetic, electrically conductive)
- Custom materials

Samples







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3D Printing

Data

- STL format
- Various CAD files can be exported to STL format
- Wall thicknesses should be multiples of x/y resolution for better accuracy
- Use correct units during designing the structures
- Avoid duplexes (use snaps)
- Overhangs with degrees >30° need support
- Models must be solid bodies, not surfaces. No open faces, models must be watertight
- In case of periodic structures a single unit cell is sufficient

Atomic-Layer-Deposition (ALD)

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 N_2 to Ar.

Contact

See KNMF website or contact the KNMF User Office.

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:
  - N_2 or Ar as carrier gas / purge gas
  - H_2O / O_3 for thermal / ozone assisted ALD
  - NH_3 for thermal ALD of nitrides
  - N_2, O_2 , H_2/Ar (5/95), H_2/N_2 (5/95), H_2/Ar (5/95) gases for plasma assisted ALD processes
- Processes available: Al_2O_3 and AlN, TiO_2 and TiN, TaN, HfO_2, SiO_2, Ag, Nb_2O_5

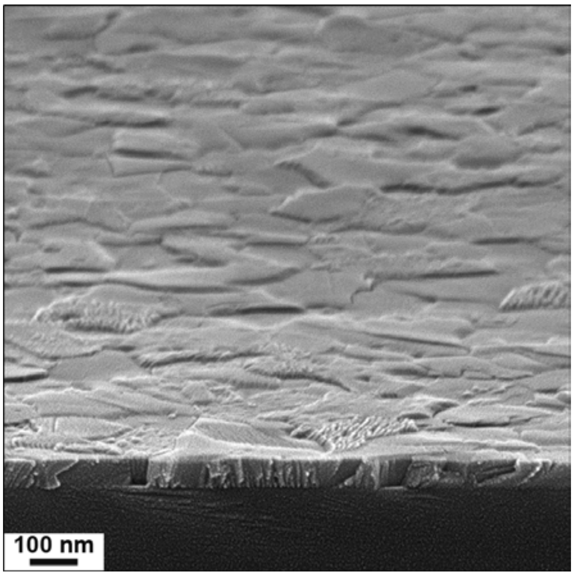
Design rules

- Max. sample height 15 mm
- Max. diameter 200 mm (8" Si-Wafer)
- Max. 28 cm^3 volume for powder coatings (max. 300 m^2/g specific surface area)

Materials

metals, ceramics, nitrides

Typical structure



SEM image of an approx. 75 nm thick TiO_2 thin film coating on a (100)-oriented Si-Wafer

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Deep X-Ray Lithography

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.

Contact

See KNMF website or contact the KNMF User Office.

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)

Typical structures and designs

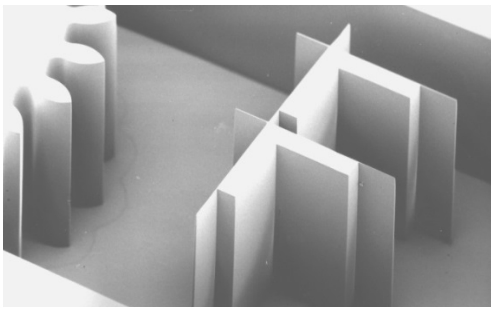

Fig. 1: 500 µm high PMMA structure (width of the small bar: 5 µm)

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

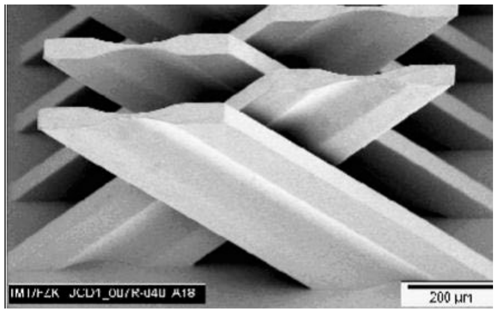

Fig. 3: Crossed X-ray lenses (SU-8)

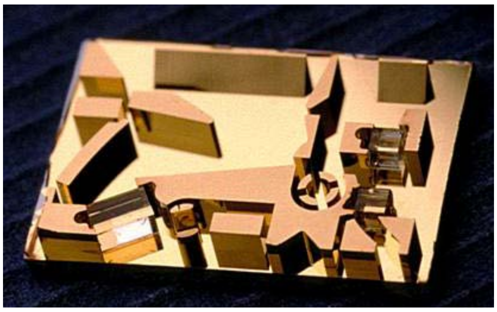

Fig. 4: Microoptical bench with cylindrical mirrors and fixing structures

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Dip-Pen Nanolithography & Polymer Pen Lithography

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].

Contact

See KNMF website or contact the KNMF User Office.

Features

- 20 nm resolution for thiols on gold, or 100 nm for phospholipids
- Throughput on the order of cm^2/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 μm^2 in a 2D array.
- Custom arrays available

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Typical structures and designs

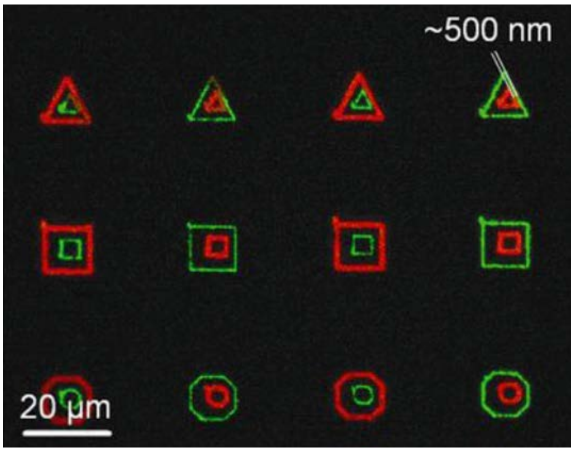

Fig. 1: Functional phospholipids patterned on a glass surface are used to template two proteins at sub-cellular scales [2]

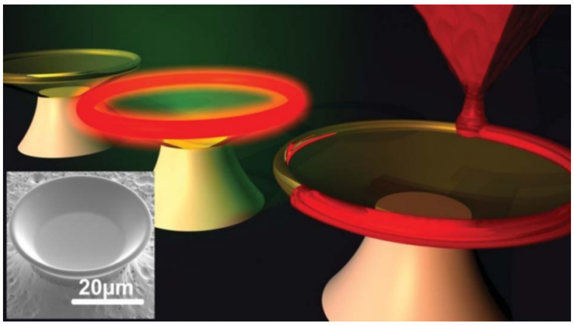

Fig. 2: Functionalization of pre-existing sensor structures by DPN with lipids [5]

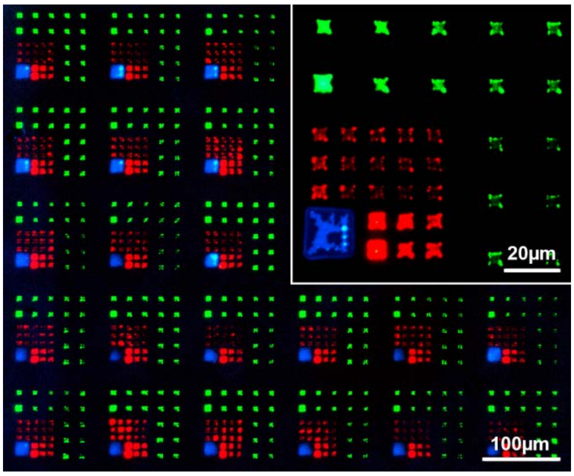

Fig. 3: Multi-color micro patterns generated by PPL with fluorescent lipids [6]

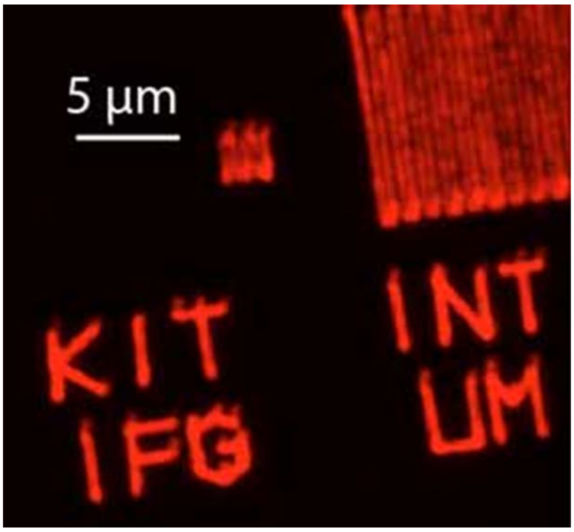

Fig. 4: Covalently linked fluorescently labeled azide ink written on an alkyne functionalized CVD coating [7]

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References

[1] S. Lenhert, H. Fuchs & C. A. Mirkin in Nanoprobes (ed. H. Fuchs), 171–196 (Wiley-VCH, Weinheim, 2009)
[2] S. Sekula, J. Fuchs, S. Weg-Remers, P. Nagel, S. Schuppler, J. Fragala, N. Theilacker, M. Franzreb, C. Wingren, P. Ellmark, C. A. K. Borrebaeck, C. A. Mirkin, H. Fuchs, S. Lenhert. Multiplexed lipid dip-pen nanolithography on subcellular scales for the templating of functional proteins and cell culture. Small 4, 1785-93 (2008)
[3] S. Sekula-Neuner, J. Maier, E. Oppong, E.; A. C. B. Cato, M. Hirtz, H. Fuchs. Allergen arrays for antibody screening and immune cell activation profiling generated by parallel lipid dip-pen nanolithography. Small 8, 585-591 (2012)
[4] S. Lenhert, F. Brinkmann, T. Laue, S. Walheim, C. Vannahme, S. Klinkhammer, M. Xu, S. Sekula, T. Mappes, T. Schimmel, H. Fuchs. Lipid multilayer gratings. Nature Nanotechnology 5, 275-9 (2010)
[5] U. Bog, T. Laue, T. Grossmann, T. Beck, T. Wienhold, B. Richter, M. Hirtz, H. Fuchs, H. Kalt, T. Mappes. On-chip microlasers for biomolecular detection via highly localized deposition of a multifunctional phospholipid ink. Lab Chip 13, 2701-2707 (2013)
[6] F. Brinkmann, M. Hirtz, A. M. Greiner, M. Weschenfelder, B. Waterkotte, M. Bastmeyer, H. Fuchs. Interdigitated Multicolored Bioink Micropatterns by Multiplexed Polymer Pen Lithography. Small (2013), DOI:10.1002/smll.201203183
[7] H. Y. Chen, M. Hirtz, X. Deng, T. Laue, H. Fuchs, J. Lahann. Substrate-independent dip-pen nanolithography based on reactive coatings. JACS 132, 18023-5 (2010)

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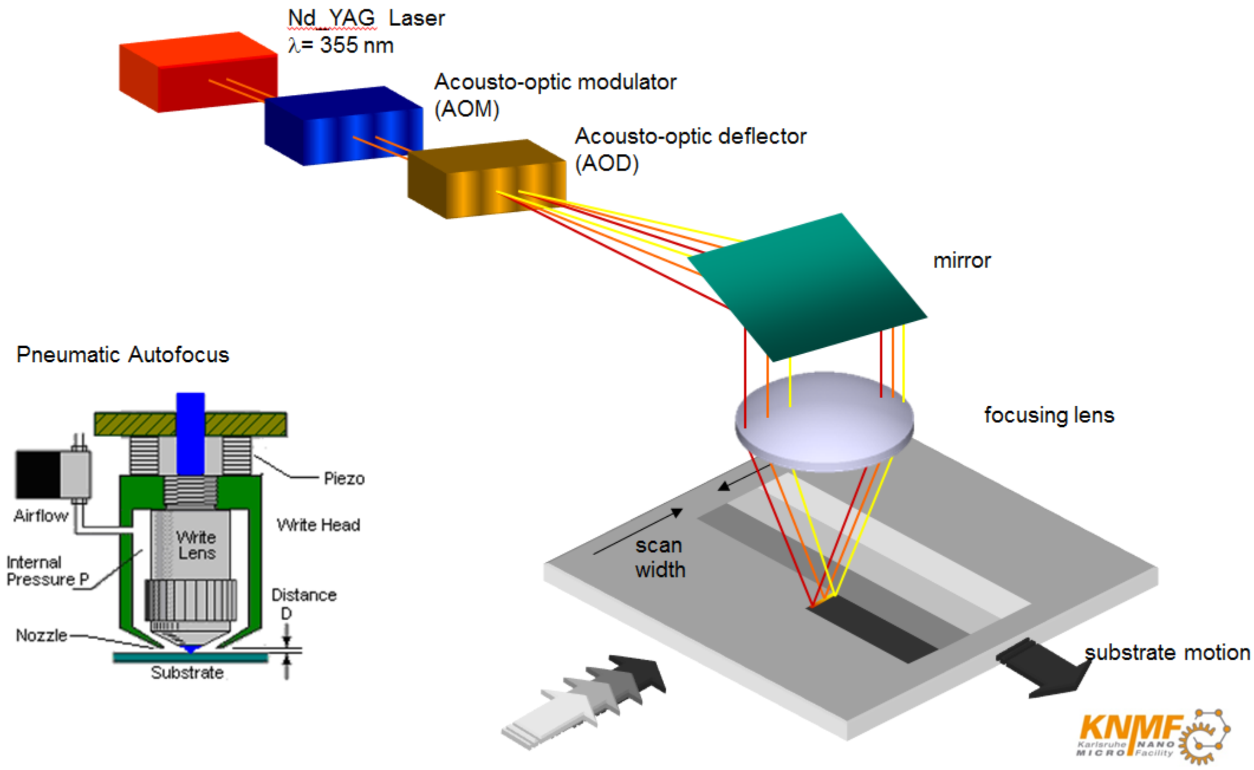
Direct Laser Writing

With DLW resist 2D-patterns in the µm-range are written in a photo resist layer on a substrate with a focused laser beam. No mask is needed as in conventional photo lithography.



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. Typically this method is applied to generate first prototypes and allows a faster iteration.

Due to the used wavelength of 355 nm it is possible to pattern thicker resist layers (*e.g.* SU-8 type resists). The focusing of the laser beam leads in this case to sloped sidewalls (< 5°).

In case of reflective substrates a bottom antireflection coating (BARC) has to be applied, which may be considered in the whole process layout.

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Direct Laser Writing

Equipment

- Heidelberg Instruments DLW66 fs
- Writehead 10 mm and 4 mm (lower and higher resolution)

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 in case of thicker resist layers
- Aspect ratios up to 4 are possible depending on structures and/ or resists

Materials

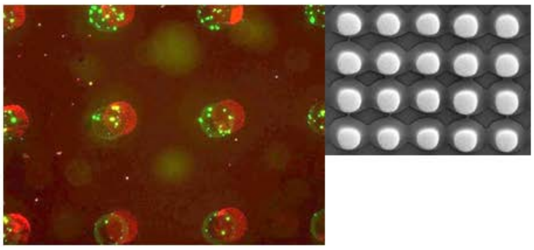
- Resists: AZ1505, AZ4533, SU8 and similar resists
- BARC: BARLI-II
- Substrate properties:
  - flat
  - roughness peak to peak < 80 µm
  - 100 µm < thickness < 2500 µm

Data

- Data File format: gds-II, dxf (2D), cif
- To work with good files the following hints are very useful:
  - closed polylines should be used
  - layers should not be named "main"
  - define proper scale before designing your pattern
  - use appropriate number of points in polygon approximation

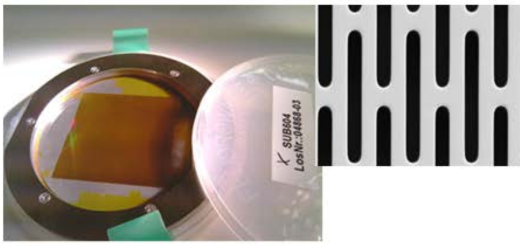
Structure examples

- Biological applications



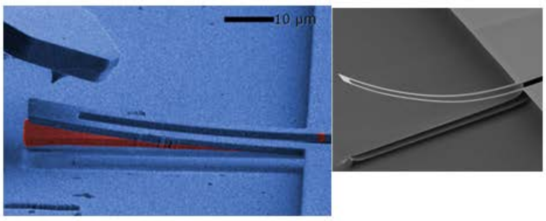
Stamps for peptides

- X-Ray masks

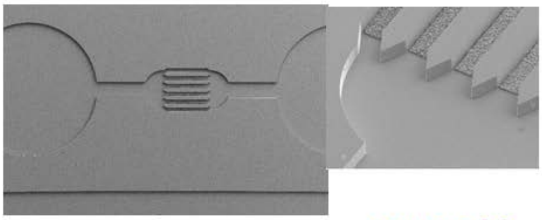


Grid structures; period 10 µm

- actuators



- Microfluidics



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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 SF_6 and O_2 for silicon etching; Cl_2, He, Ar and O_2 for chromium and other metals.

Contact

See KNMF website or contact the KNMF User Office.

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

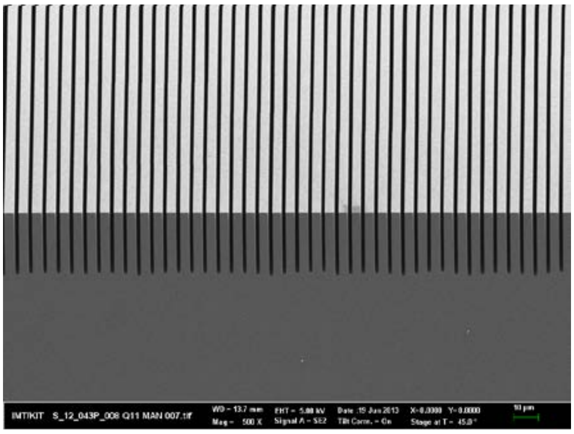
- Mask material: PMMA, SiO_2, ma-N 2401
- Structures on Si fragments or complete 4” Si wafers

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, SiO_2 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

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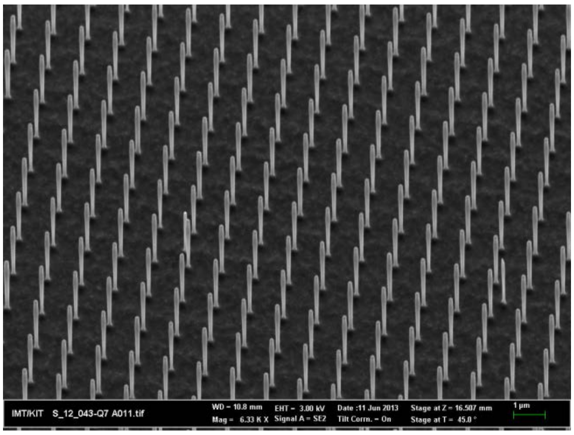
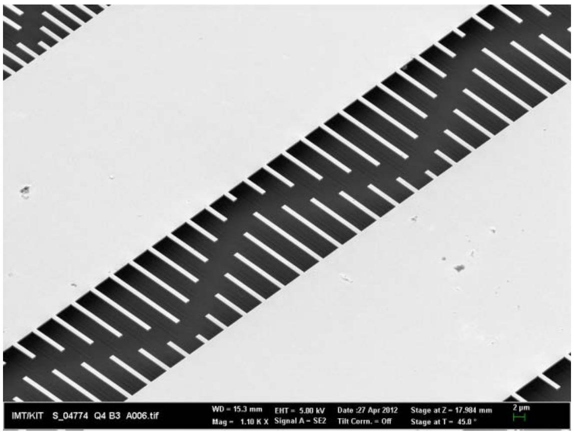
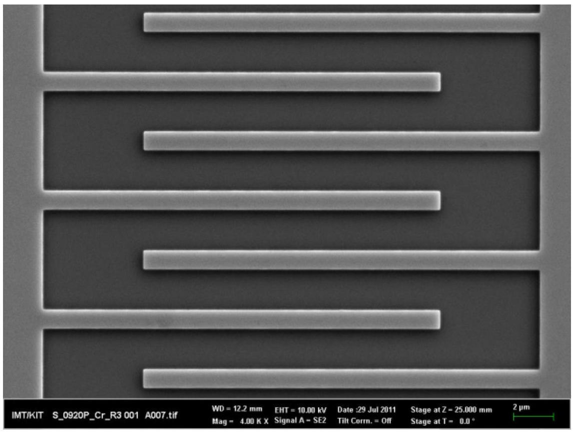
Typical structures and designs
(continued)



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

See KNMF website or contact the KNMF User Office.

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

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Focused Ion Beam

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 XeF_2 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**

Contact

See KNMF website or contact the KNMF User Office.

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 XeF_2 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 μm^3 can be removed in a reasonable processing time, volumes of up to 100 x 100 x 50 μm^3 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

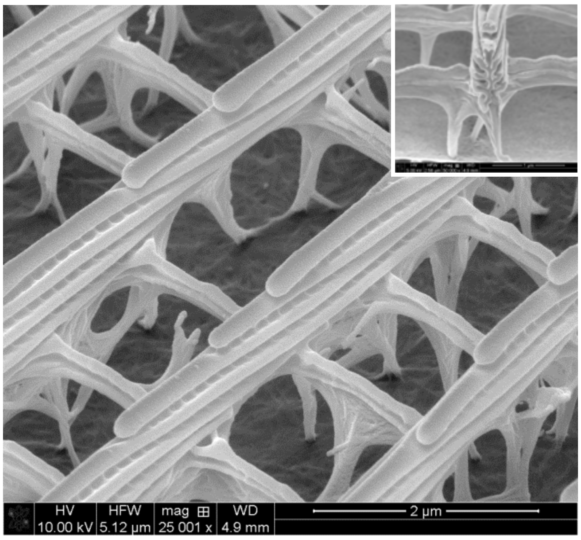

Fig. 1: SEM image of the photonic structures on a butterfly wing with a FIB prepared cross-section in the inset.
In collaboration with R. Siddique and R. Prang, KIT.

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Focused Ion Beam

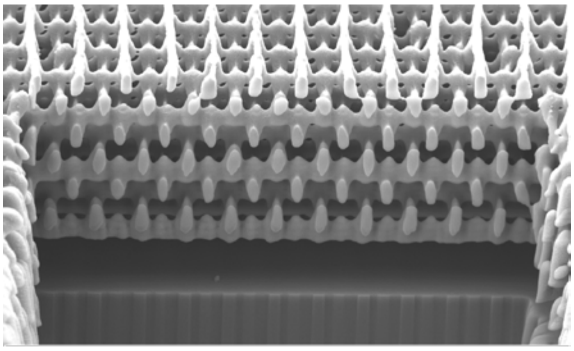

Fig. 2: Cross-sectional analysis of a photonic crystal generated by 3D direct laser writing.
In collaboration with NanoScribe and T. Scherer, KIT.

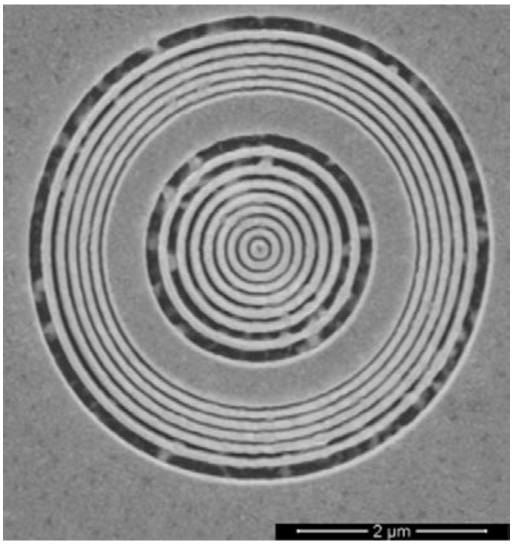

Fig. 3: FIB generated photonic structure in a thin gold film.
In collaboration with Y. Yu, IMTEK and D. Chaissing, KIT.

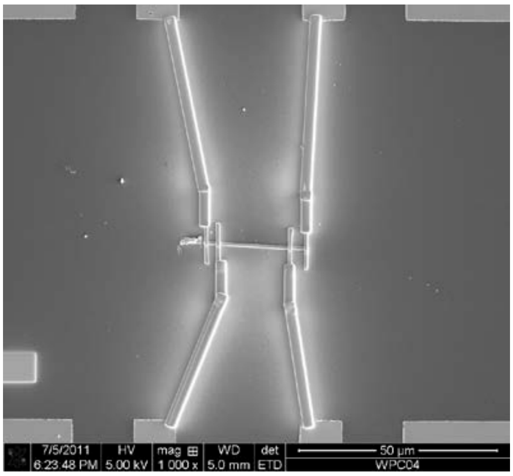

Fig. 4: Electric contacting of a silver nanowire for 4--point conductivity measurements.

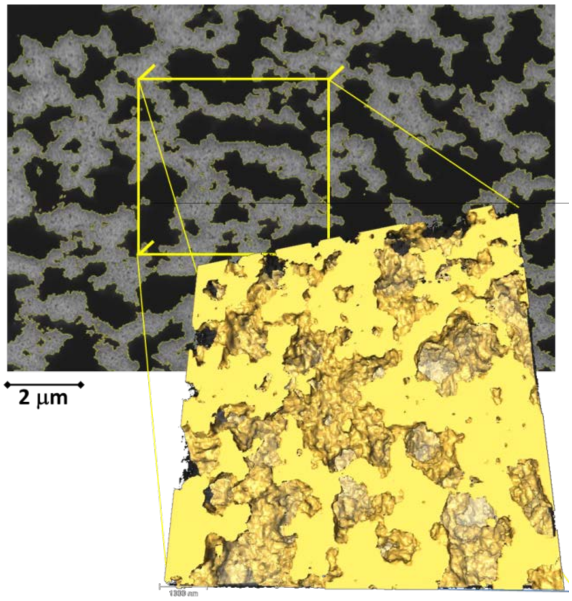

Fig. 5: 3D nanoscale morphology analysis of micro- and macroporous silica for application in HPLC: segmented digital slices through the 3D volume and volume rendering of a small area.
In collaboration with D. Stöckel, B. Smarsley, Univ. Gießen and C. Kübel, R. Prang, KIT.

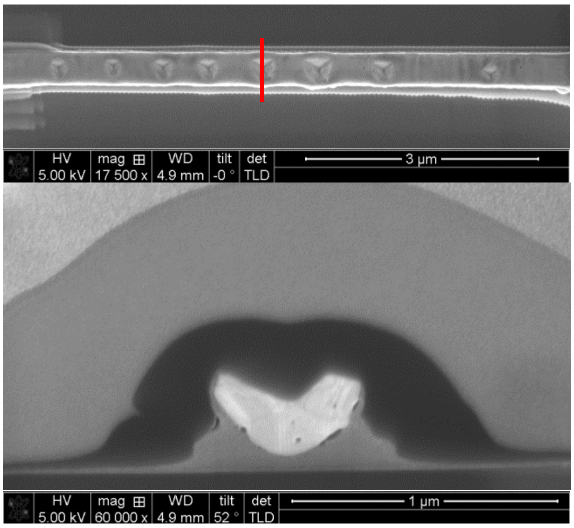

Fig. 6: TEM cross-section target preparation of a nanoindent in an Ag nanowire.
In collaboration with A. Kobler, T. Beuth and R. Prang, KIT.

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Hot Embossing

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

See KNMF website or contact the KNMF User Office.

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

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Injection Moulding

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 multi-functional devices.

Contact

See KNMF website or contact the KNMF User Office.

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/Al_2O_3 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 ZrO_2 and Al_2O_3, Si3N4, mixture ceramics like TiN-Al_2O_3
- with defined material properties, e.g. electrical conductivity
- Subsequent densification and reduction of porosity by HIP

Typical structures and designs

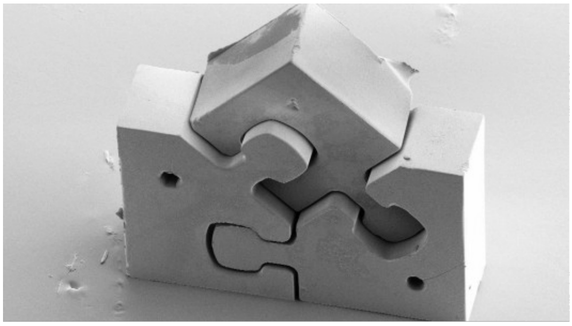

Fig. 1: Smallest puzzle of the world whose pieces have been made of PMMA using singular LIGA mould inserts.

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Injection Moulding

Typical structures and designs
(continued)

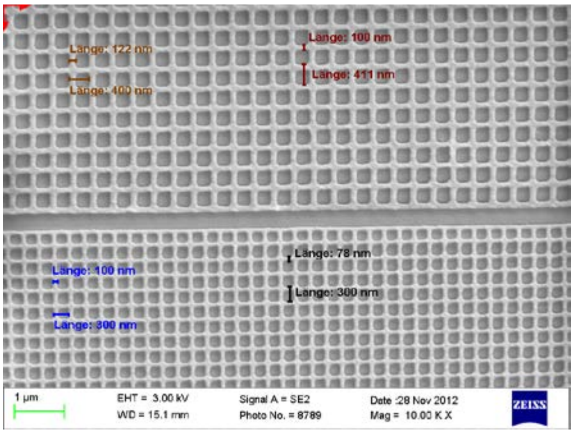

Fig. 2: SEM figure of polymer part (PMMA) with nano-sized structures made by injection moulding.
(Länge ≙ length).

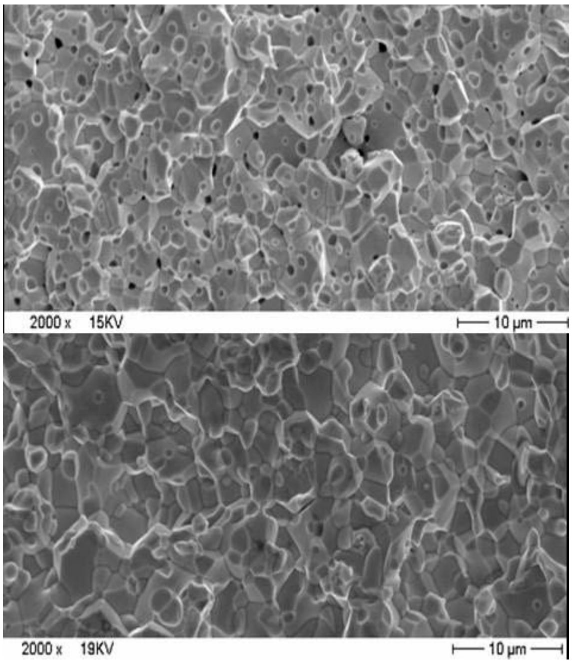

Fig. 3: SEM-picture of pure tungsten after sintering (above), the same material after additional HIP densification showing significantly reduced porosity (below).

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).

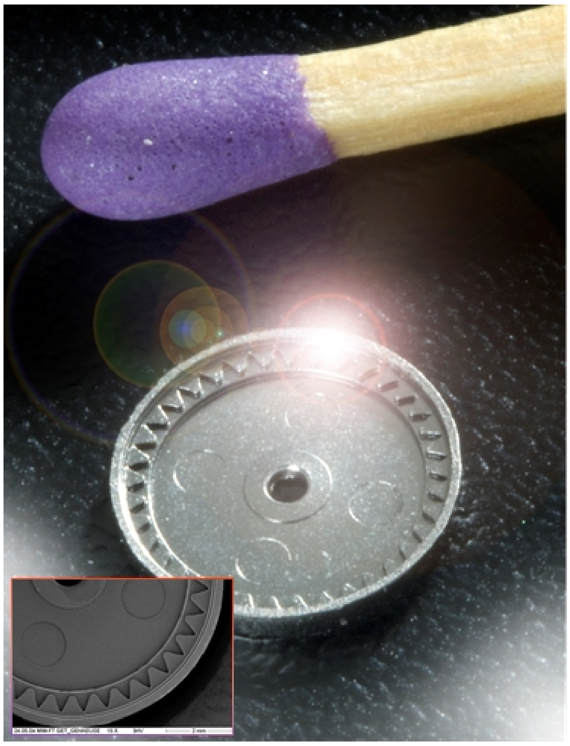

Fig. 5: Ring gear of planetary gear set, 1.4542.

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Laser Materials Processing

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

See KNMF website or contact the KNMF User Office.

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 R_a =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, SnO_2, $LiCoO_2$,...), standard battery materials ($LiCoO_2$, $LiMn_2O_4$, NMC, $LiFePO_4$), steel, nickel, brass, WC, Al_2O_3, ZrO_2, SiC
- Cutting:
  steel, Ti, NiTi, quartz, Al_2O_3, PMMA, PI, PS
- Laser-LIGA: nickel
- Surface modification:
  PS, PC, PMMA, amorphous carbon thin films

Typical structures and designs

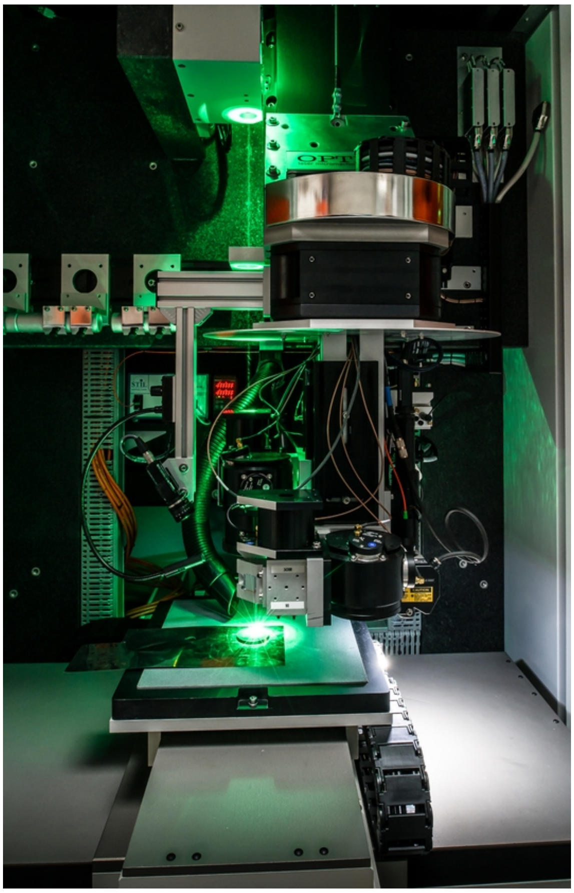

Fig. 1: Micro-machining workstation (PS450-TO, Optec) equipped with a tunable ultrafast laser (Tangerine, Amplitude SYSTEMES) and a tunable short pulse fiber laser (IPG photonics)

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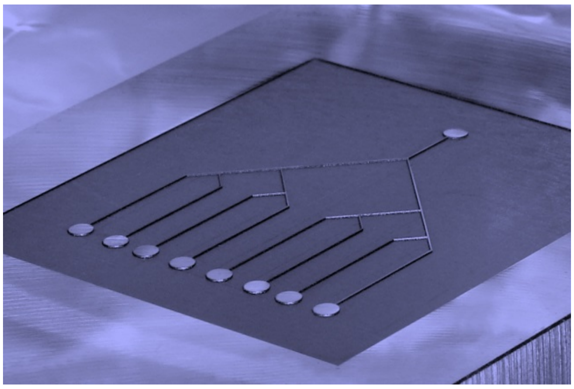

Fig. 2: Laser structured mould insert made of steel for replication of microfluidic chips (channel structure width 50 µm)

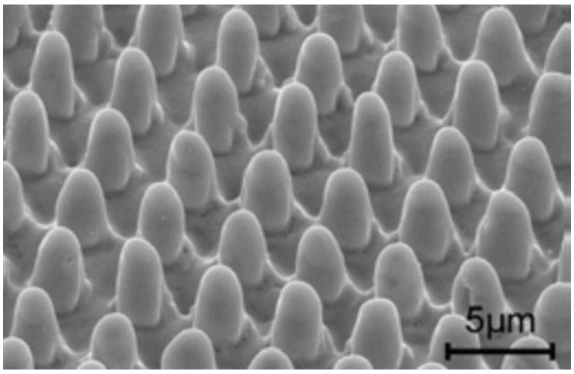

Fig. 3: Laser microstructured SnO_2 anode material

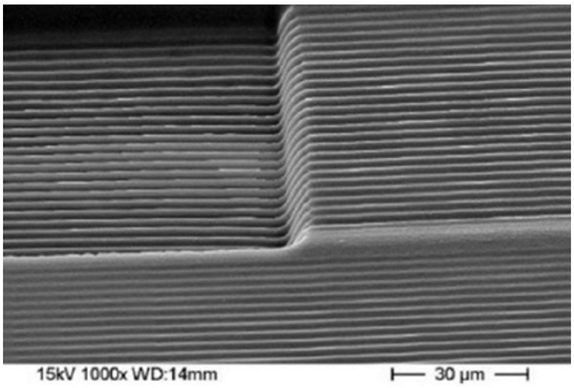

Fig. 4: Laser generated microgrooves in polystyrene

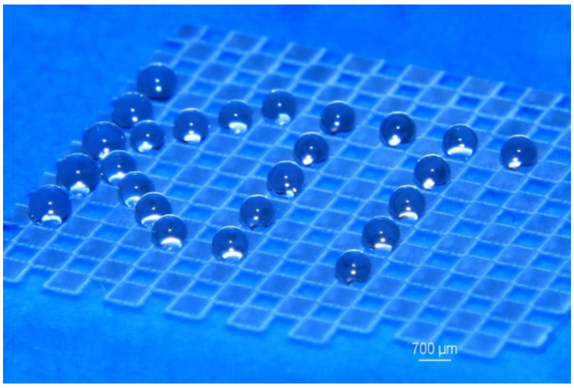

Fig. 5: Local control of wetting behaviour due to laser-assisted surface modification of PDMS

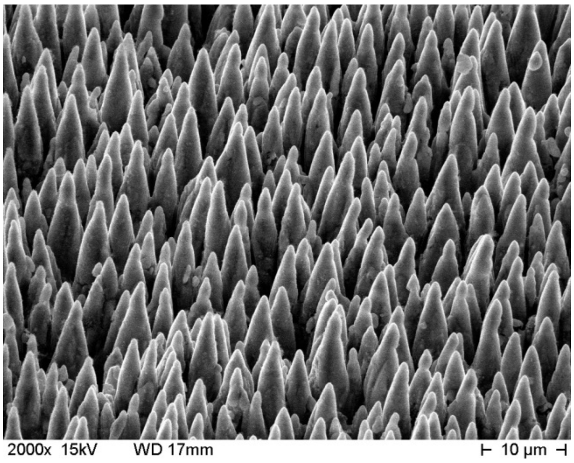

Fig. 6: SEM image of conical microstructures in NMC cathode material formed by excimer laser radiation

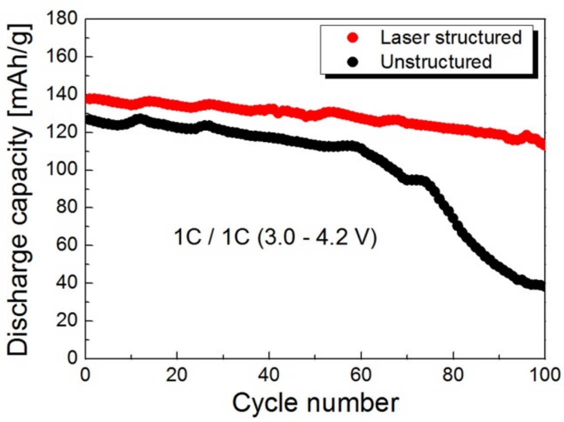

Fig. 7: Specific discharge capacities of an unstructured and laser structured cathode material for lithium-ion batteries (Swagelok® design)

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Surface-Anchored Metal-Organic Frameworks (SURMOFs)

SURMOFs represent a new class of highly porous and highly crystalline materials that may be used as host structures for molecules or nanoparticles with high application potential in gas storage systems, optical sensors, or catalytically active materials, to name a few examples. Metal-Organic Frameworks (MOFs) - in general - consist of two main components: metallic nodes and organic linker molecules. An automated layer-by-layer (LBL) deposition procedure [1] allows to deposit the metal compound and the linker molecules in an alternating fashion on chemically functionalized substrates (oxides, gold-coated substrates). The thickness of the layers is determined by the number of synthesis cycles while the size and chemical properties of the SURMOFs are defined by the used linker molecules. Different methods, e.g. spray or dip coating, are available for the SURMOF preparation. The application of additional treatments (e.g. ultrasonication), results in SURMOFs with high optical quality and high transparency. [2]

In contrast to MOF thin films prepared from powders deposited on substrates by painting or doctor-blade techniques, the SURMOFs are monolithic, highly oriented and exhibit a low density of defects. Due to these outstanding properties SURMOFs can be used not only as model system for studying crucial intrinsic properties of MOF materials, including diffusion of guest species and the formation of surface barriers (see [3] as a review) but also as model host substrates for nanoparticles [4] or molecules to investigate e.g. diffusion processes [5] or charge transport behavior [6]. After fabrication, SURMOFs are characterized by X-Ray diffraction (XRD, to verify crystallinity and growth orientation) and by IR reflection absorption spectroscopy (IRRAS, for chemical characterization)..

Contact

See KNMF website or contact the KNMF User Office.

Features

The following three different **SURMOF systems** are available:

- **HKUST-1**
  [J. Am. Chem. Soc., 2007, 129 (49), pp 15118–15119]
- **Cu(BDC)**
  [Appl. Phys. Lett. 103, 091903 (2013)]
  [Scientific Reports 2, Article number: 921 (2012)]
- **$Cu_2(BDC)_2(dabco)$**

The following types of **substrates** can be coated:

- SiO_2 wafers
- (Quartz) glass
- Porous oxides
- Au coated substrates (Si-wafer, glass, mica)

Limitations/constraints

- Substrate size should not exceed 20 mm x 20 mm
- Porous oxides substrates have to be delivered by the user

References

[1] O. Shekhah, H. Wang, S. Kowarik, F. Schreiber, M. Paulus, M. Tolan, C. Sternemann, F. Evers, D. Zacher, R. A. Fischer, C. Woll: Step-by-step route for the synthesis of metal-organic frameworks. *J. Am. Chem. Soc.* **129** (2007), 15118-15119.
[2] Z.-G. Gu, A. Pfriem, S. Hamsch, H. Breitwieser, J. Wohlgemuth, L. Heinke, H. Gliemann, C. Wöll: Transparent films of metal-organic frameworks for optical applications. *Microporous and Mesoporous Materials* **211** (2015), 82-87.
[3] J.L. Zhuang, A. Terfort, C. Wöll: Formation of oriented and patterned films of metal-organic frameworks by liquid phase epitaxy: A review. *Coordination Chemistry Reviews* **307**(2016), 391-424.
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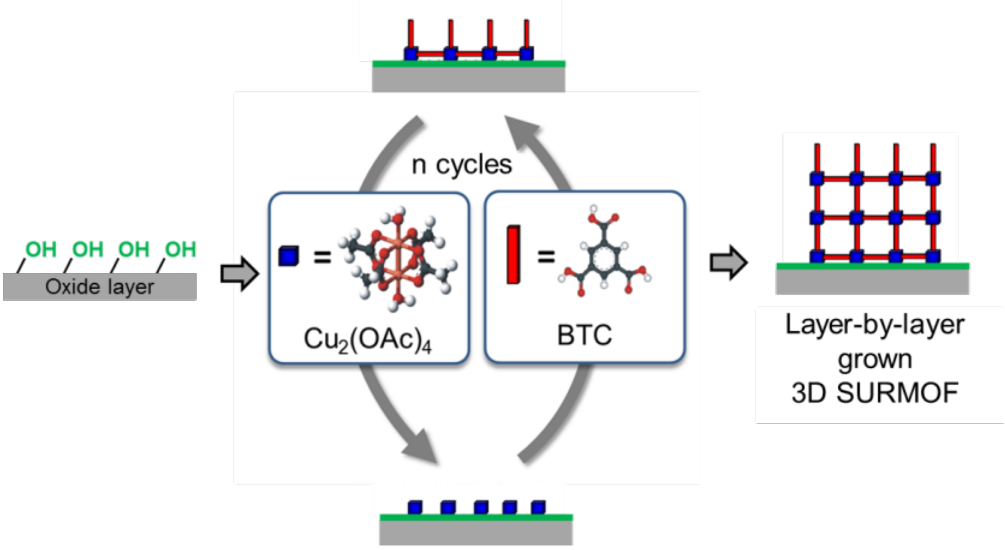
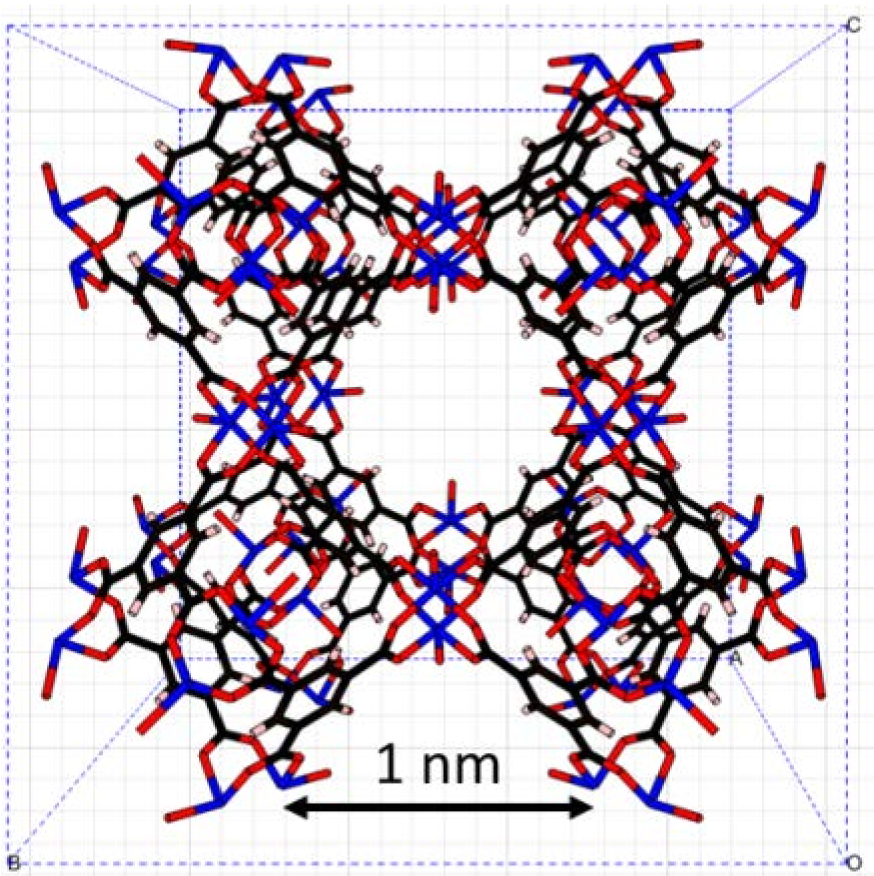
Surface-Anchored Metal-Organic Frameworks (SURMOFs)

Layer-by-layer growth and typical properties of HKUST-1

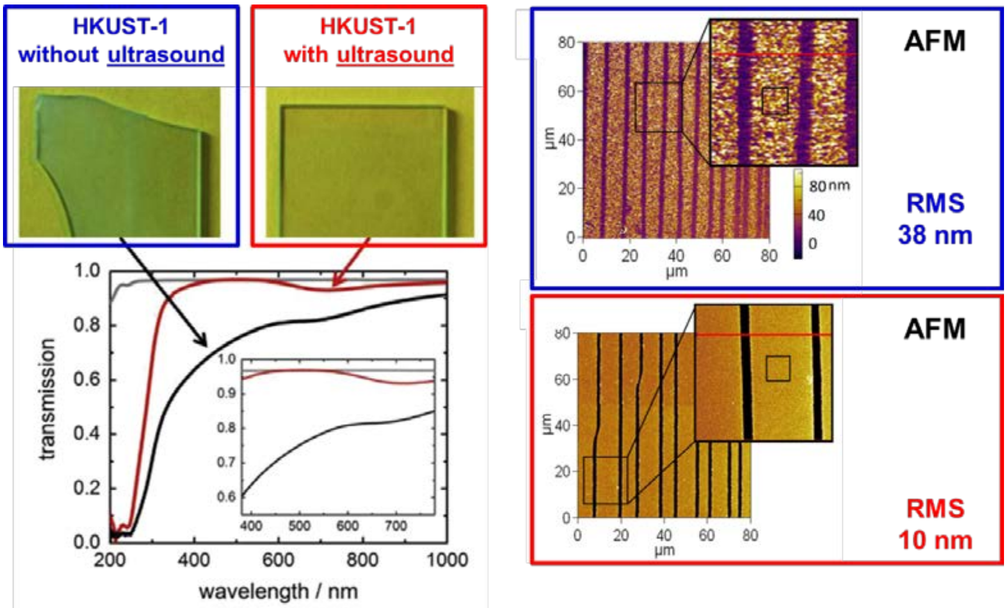


Schematic layer-by-layer deposition process

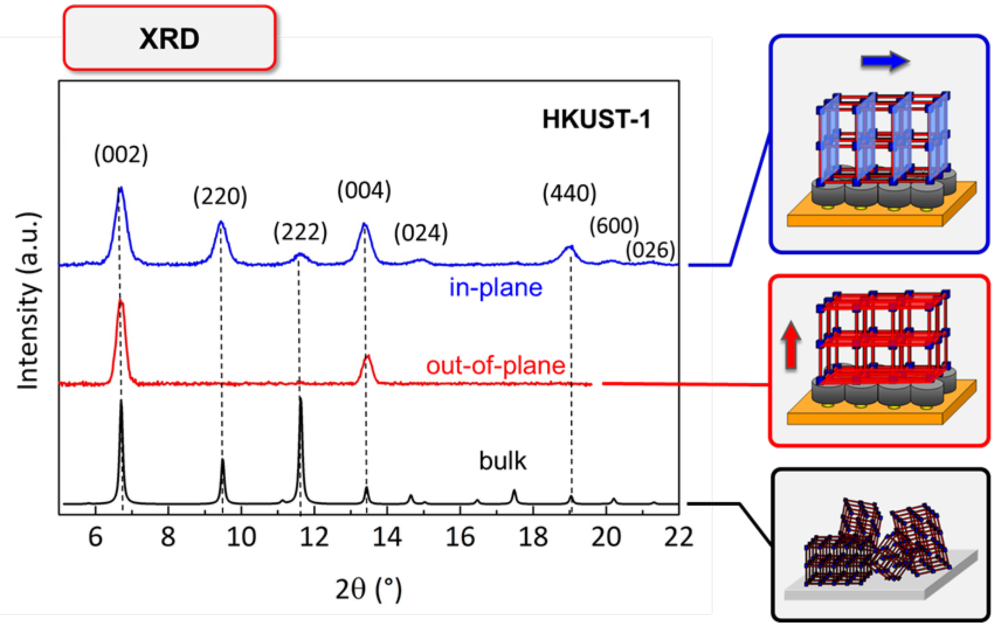


Crystal structure for the example of HKUST-1 SURMOF

Surface-Anchored Metal-Organic Frameworks (SURMOFs)



Typical optical and interfacial properties of an HKUST-1 SURMOF grown on a glass substrate w/o (blue) and with (red) ultrasound [2].



Typical XRD pattern of HKUST-1 SURMOF investigated in the in-plane and out-of-plane mode. The out-of-plane data proofs oriented growth of the SURMOF along the (100) direction.

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3D Atom Probe Tomography (APT)

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

See KNMF website or contact the KNMF User Office.

Features

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

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

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Atomic Force Microscopy

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

See KNMF website or contact the KNMF User Office.

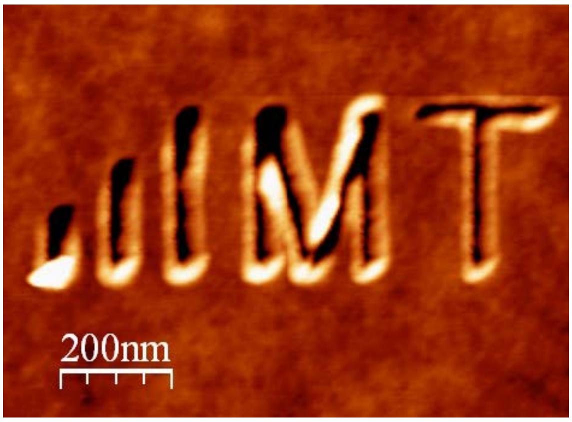
Features

- 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)

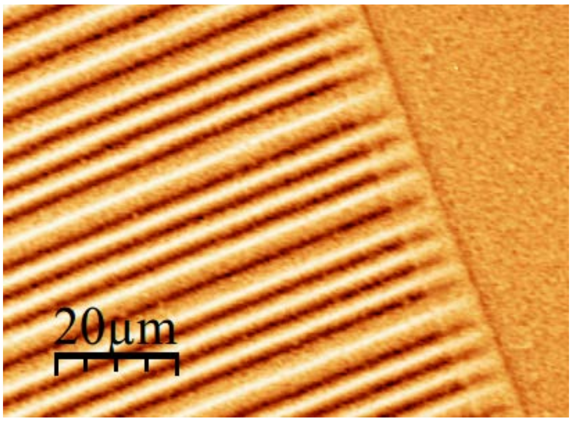
Limitations/constraints

- 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

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Auger Electron Spectroscopy

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

See KNMF website or contact the KNMF User Office.

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 N_2 cooling for in situ fractures (grain boundary analysis)

Limitations/constraints

- Maximum sample size Ø 60 mm
- Sample has to be solid at RT and stable under vacuum conditions (10^{-9}Torr)
- Depending on the chemical composition samples might be sensitive to the electron beam

Equipment

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

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Typical measurements

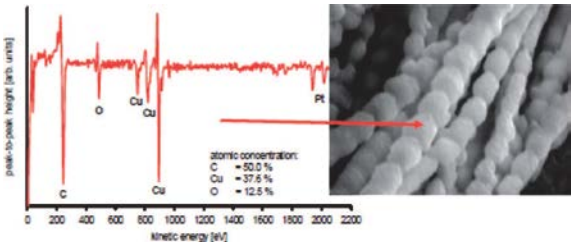

Fig. 2: Auger survey and SE image of Cu nano wires

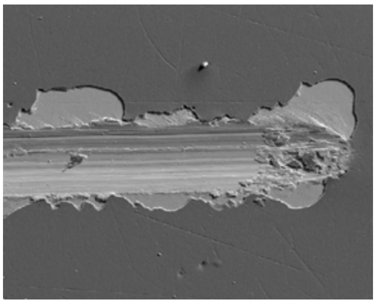

Fig. 3: SE image

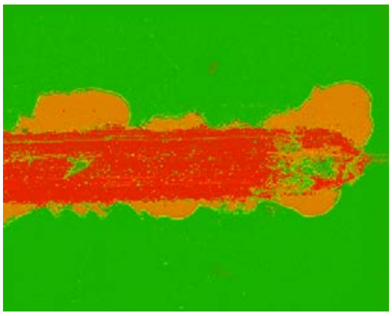

Fig. 4:AES element mapping

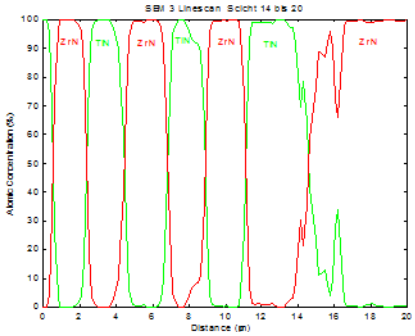

Fig. 5: AES line scan

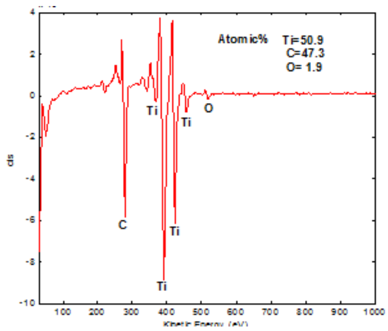

Fig. 6: AES spectrum

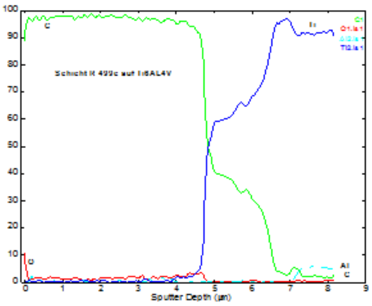

Fig. 7: AES depth profile

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KNMF Laboratory for Microscopy and Spectroscopy

Bulk and Trace Analysis of Nanomaterials

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

See KNMF website or contact the KNMF User Office.

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 SiO_2, TiO_2, Al_2O_3, glass, other nano oxides/carbides/nitrides

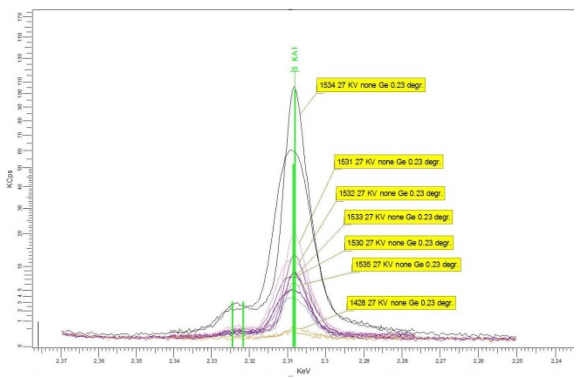

Fig. 1:
S KA1. Analysis from a filter cake on a paper filter. Concentration range 0.01 – 3.0 mass %.

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Bulk and Trace Analysis of Nanomaterials

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

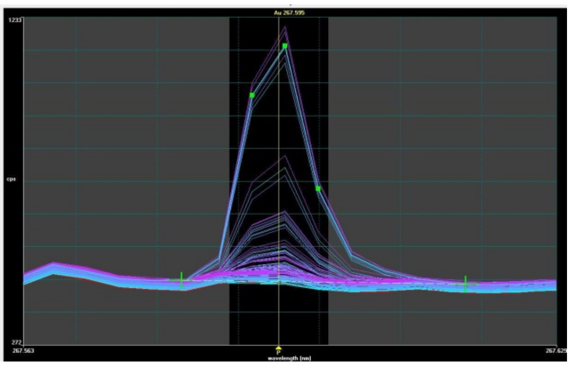

Fig. 2:
Au with the ICP-OES by 242.795 nm. Chemical digestion from a mixture of Au and TiO_2 NM with aqua regia. Concentration range 0.005 – 0.20 mg/l

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

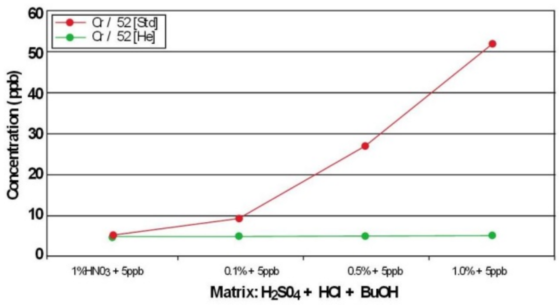

Fig. 3:
Consistent interference reduction in a variable, complex matrix using He mode. Comparison plots showing Std mode (no cell gas - red) and He mode (green) spike recovery data for 5 ppb Cr in a variable matrix (up to 1% each of HCl, H_2, SO_4 and Butanol). Potential interferences on 52 Cr include ArC, ClOH and SO.

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Bulk and Trace Analysis of Nanomaterials

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.

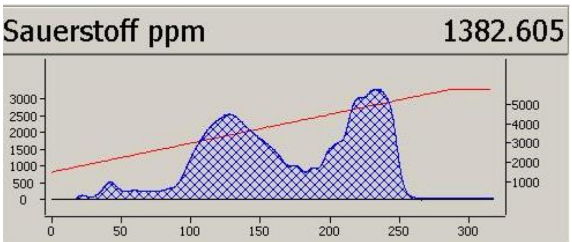

Fig. 4:
Different kind of oxygen bonds in steel

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 CO_2 and SO_2
- Analysis range: < 0.0005 to 100%

Typical samples
Solids: metals, inorganic and organic materials

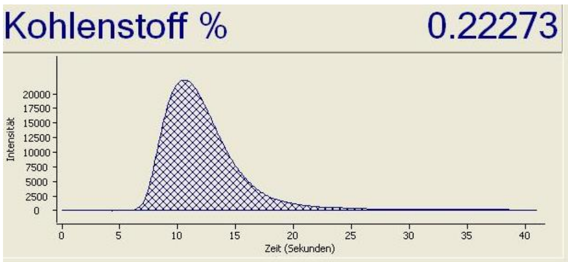

Fig. 5:
C in WC

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Helium Ion Microscope (HIM)

The Zeiss Orion NanoFab 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 and neon ion beams can be used for machining at the nanoscale with feature sizes 10-20 times smaller than achievable using a gallium 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 SiO_2 from precursor gases using the helium and neon ion beams.

Contact

See KNMF website or contact the KNMF User Office.

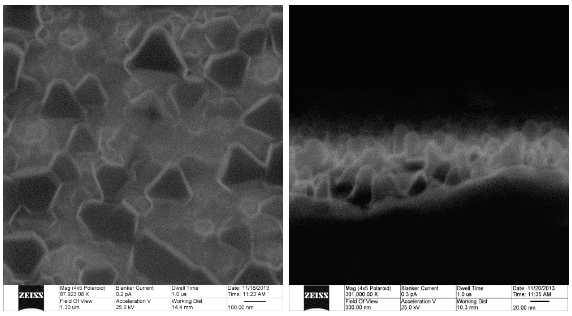
Features

- Zeiss Orion NanoFab with He and Ne ion beams
- Resolution (He/Ne)
  ≤ 0.5 nm / ≤ 1.9 nm
- Acceleration voltage (He/Ne)
  10 kV – 35 kV / 10 kV – 30 kV
- Beam current (He/Ne)
  0.1 pA – 100 pA / 0.1 pA – 50 pA
- Field of view
  900 µm – 100 nm
- Detectors
  Everhart-Thornley Secondary Electron Detector (ETD)
- Charge compensation
  Electron flood gun
- Chamber base pressure
  2 x 10^{-7} Torr
- Stage
  Tilt 0-56°
  Maximum travel in x and y ±25 mm from center
- Gas injection system for Pt, W, insulator (Siloxane)
- NanoPatterning and Visualization Engine (NPVE) for advanced patterning

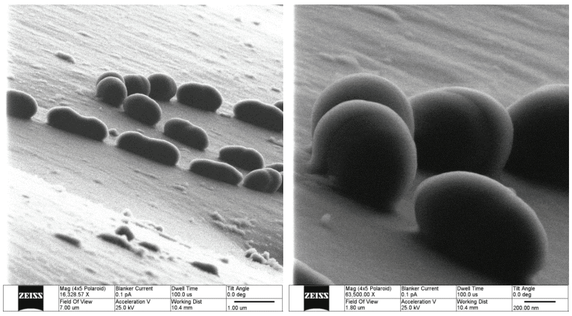
Limitations/constraints

- Sample has to be a solid at room temperature, dry and stable under high vacuum conditions
- Maximum sample height 10 mm
- Samples should be very clean prior to imaging to minimize carbon contamination

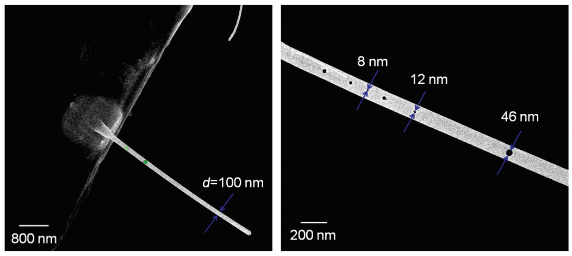
Application examples



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)

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Single Crystal X-ray Diffraction

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

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Soft X-Ray Spectroscopy, Microscopy, and Spectromicroscopy

IFP's soft x-ray analytics facility WERA at ANKA provides a coherent combination of electron spectroscopies and microscopies for studying in detail the chemical (electronic) and magnetic structure of bulk materials, thin films, and micro- and nanostructured objects.

Contact

See KNMF website or contact the KNMF User Office.

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 ΔE/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.

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

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

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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 SXMCD. 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 SXMCD 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.

Soft X-Ray Spectroscopy, Microscopy, and Spectromicroscopy

Typical samples

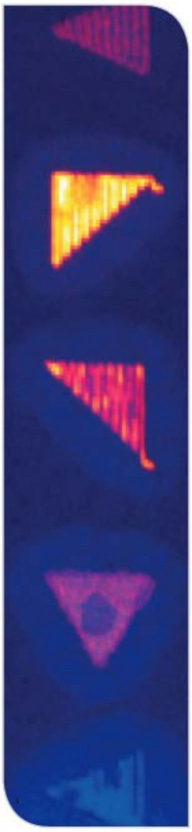

Fig. 2:
Nanolithography of phospholipids by the dip-pen method (see KNMF Laboratory for Micro- and Nanostructuring). The written triangles are 10 µm wide. The increasing admixture of nickel-chelating lipids (with a single Ni atom per lipid macromolecule) to the carrier appears in this Ni-sensitive, spectromicroscopic 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).

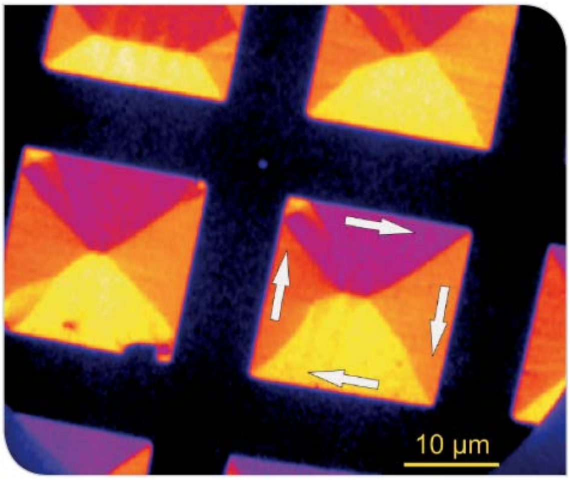

Fig. 3:
Magnetic domains and possibly defect-related subdomains with various orientation are visible in this magnetic dichroism image of thin film permalloy squares.

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Time-of-Flight Secondary Ion Mass Spectrometry

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

See KNMF website or contact the KNMF User Office.

Equipment

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

Features

- Bi/Mn Source (Bi^+, Bi_3^+, Bi_3^{++}, Mn^+)
- Mass resolution: up to 11000 m/Δm @ 29 amu (bunched mode)
- Spatial resolution < 150 nm (collimated mode)
- Surface sensitivity < 1nm
- Cs thermion source and O_2 EI source for sputter depth profiling, Zalar-rotation possible
- C_{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.

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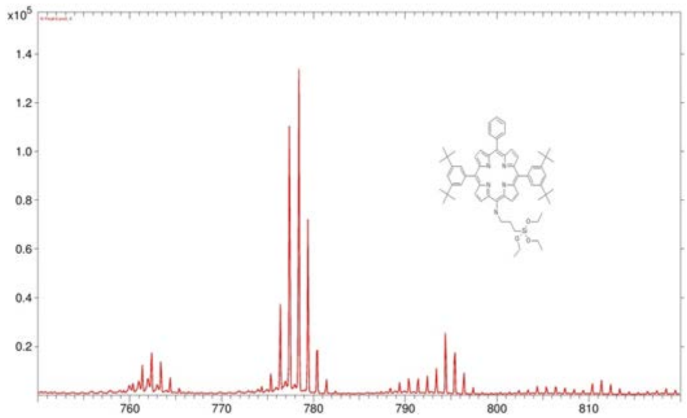
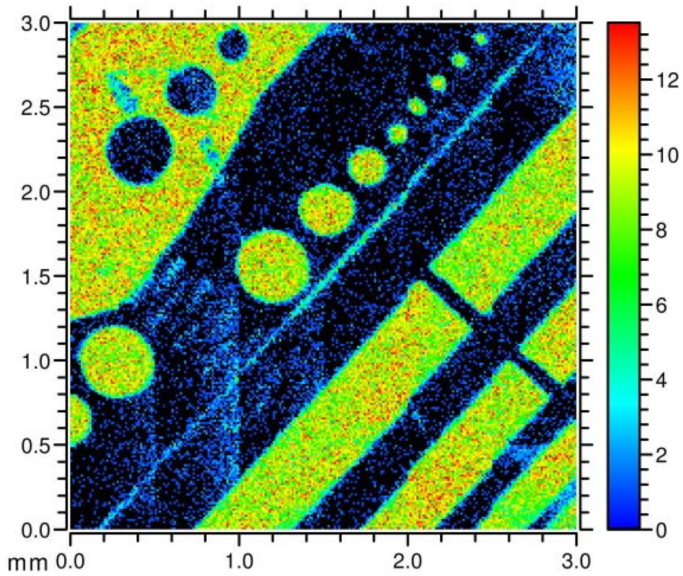
Time-of-Flight Secondary Ion Mass Spectrometry

Typical results

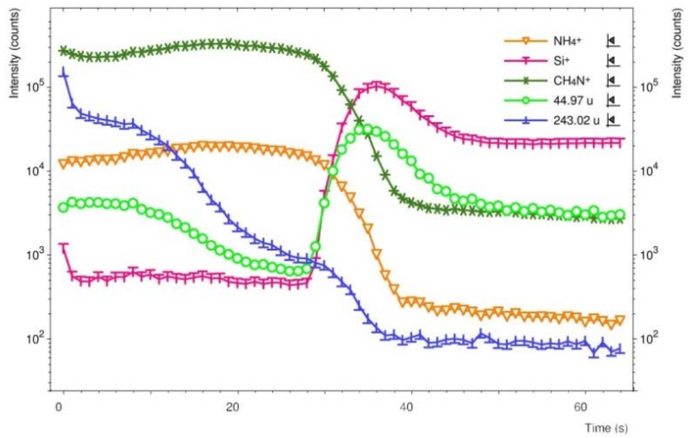


High mass range of a positive polarity secondary ion spectrum of a porphyrin derivative immobilized via silanol groups onto a silicon wafer (Bi_3^+).

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 $C_2H_3O_2^-$ 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_3^+) of a polymer sample produced according to merrifield peptide synthesis. The depth distribution of triphenylmethyl proteting groups is shown by the 243 m/z signal (blue).

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Transmission Electron Microscopy

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

See KNMF website or contact the KNMF User Office.

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 ºC; Gatan 652: RT-800 ºC)
  - Cooling (Gatan 915: LN2-80 ºC)
  - 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 LN_2 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

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Transmission Electron Microscopy

Typical results

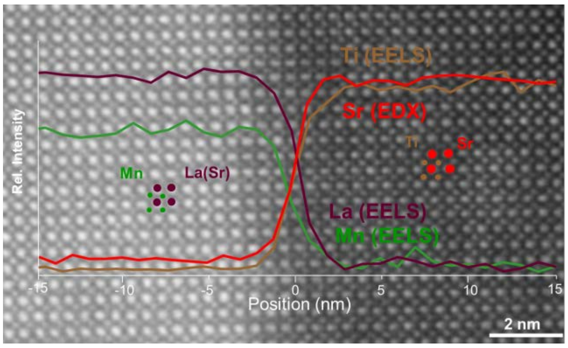

Fig. 1: HAADF-STEM image (filtered by NAD) of a $La_{1-x}Sr_xMnO_3/SrTiO_3$ interface with the individual atomic columns well resolved across the interface. Overlaid is an EELS/EDX intensity profile across this interface. P.M. Leufke and D. Wang et al., Thin solid films, 2012, 520, 5521-5527.

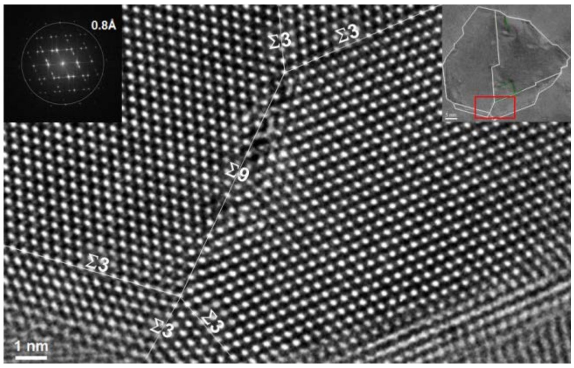

Fig. 2: Atomic resolution TEM image of a triple and a quadruple line at the interface between Σ3 boundaries and a Σ9 boundary in nanocrystalline palladium. H. Rösner and C. Kübel et al., Acta Mat., 2011, 59, 7380-7387.

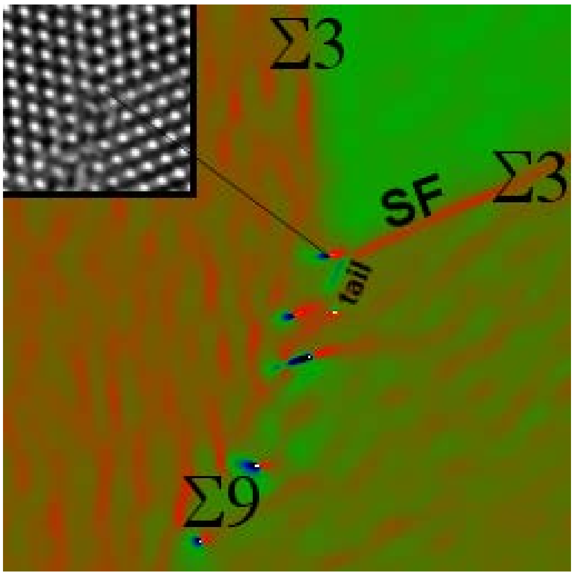

Fig. 3: Geometric phase analysis reveals the local strain distribution around the triple line in the image above. H. Rösner and C. Kübel et al., Acta Mat., 2011, 59, 7380-7387.

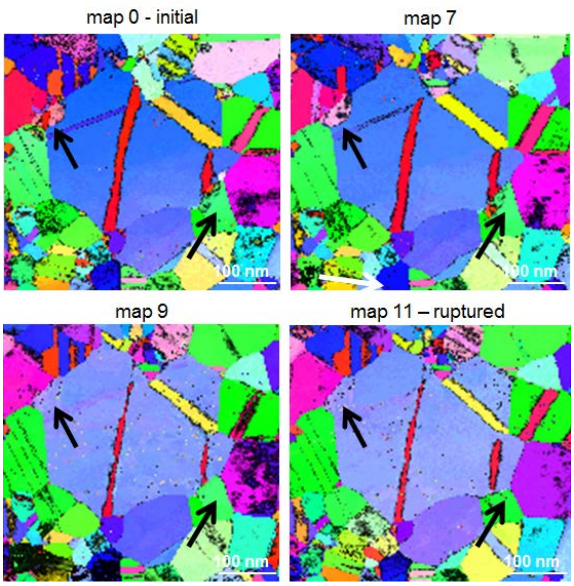

Fig. 4: In-situ orientation mapping (different color correspond to different crystal orientations) of the grain structure changes in nanocrystalline gold during straining – selected images of the straining series showing anomalous grain growth. A. Kobler and C. Kübel et al., Ultramicroscopy, 2013, 128,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

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Typical results *(continued)*

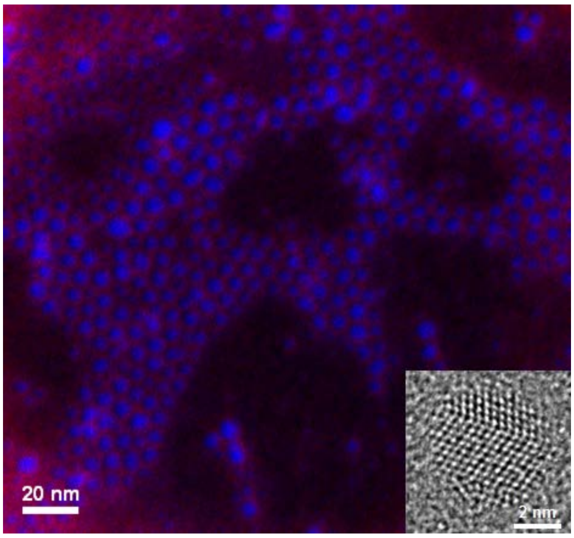

Fig. 5: EFTEM mapping (Si-blue, C-red) and HRTEM image of nanocrystalline silicon particles with a covalently bound C18 shell. The EFTEM maps reveal the ~1.2 nm wide carbon shell around the silicon core. Sample provided by G. Ozin, University of Toronto.

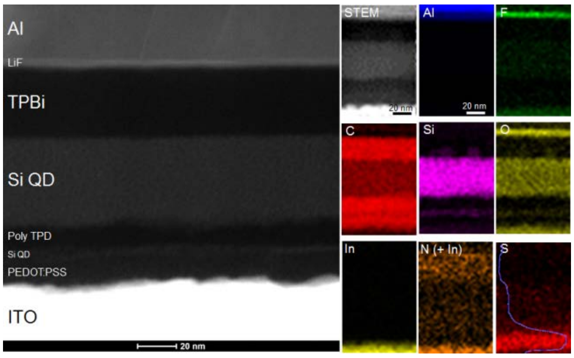

Fig. 6: HAADF-STEM image with EDX compositional mapping of the different layers in a silicon quantum dot based organic LED (SiLED). F. Maier-Flaig and C. Kübel et al., Nano Letters, 2013, online; DOI: 10.1021/nl400975u.

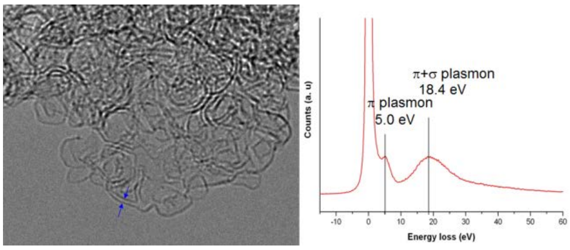

Fig. 7: HRTEM image of nano graphene with the corresponding low-loss EELS spectrum showing the characteristic π and π+σ plasmon losses. J. Biener and D. Wang et al., Adv. Mater. 2012, 24, 5083–5087.

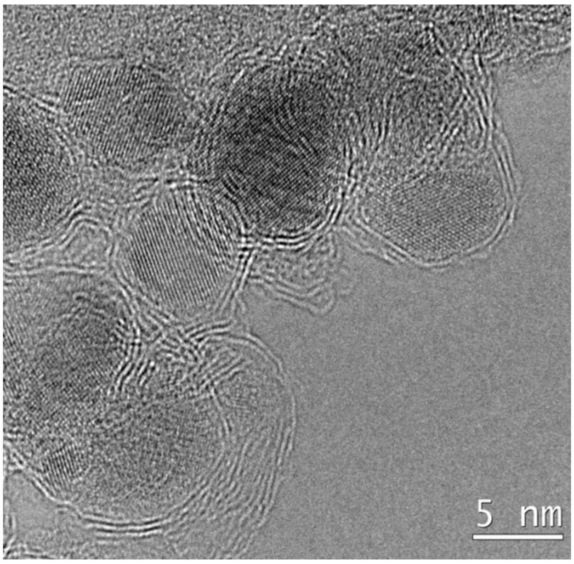

Fig. 8: HRTEM image of a Fe/LiF/C anode for lithium ion batteries revealing α-iron nanoparticles each surrounded by a few graphene layers. R. Prakash and C. Kübel et al., J. Power Sources, 2011, 196, 5936-5944.

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Typical results *(continued)*

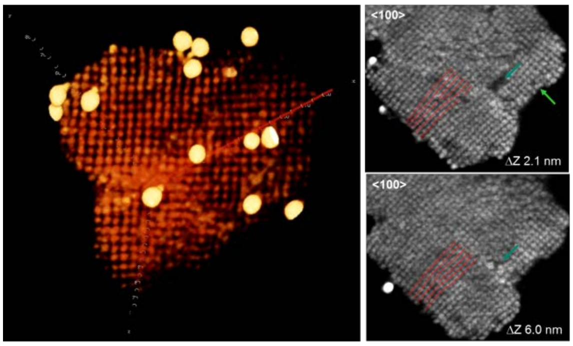


Fig. 9: Electron tomographic reconstruction of a self-assembled CdS nano cluster superlattice (with additional 5 nm gold particles in yellow). The two digital slices, one unit cell apart, show a single vacancy, an extended vacancy and dislocations in 3D. T. Levchenko and C. Kübel et al., Chem. Eur. J.,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

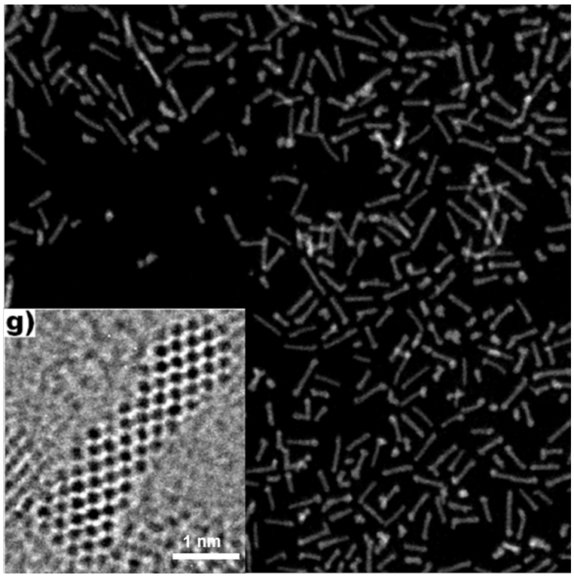


Fig. 10: HAADF-STEM and HRTEM imaging of uniform ThO_2 nanorods. D. Hudry and E. Courtois et al., Chem. Eur. J, 2013, 19(17), 5297–5305.

Travelling Wave Ion-Mobility Time-of-Flight Mass Spectrometry

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

See KNMF website or contact the KNMF User Office.

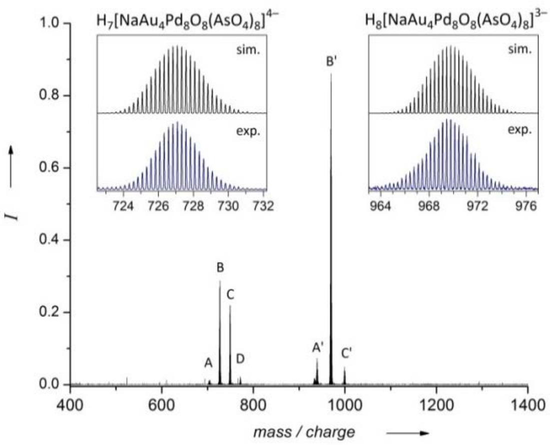
Features

- 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-Au_4Pd_8 in H_2O/DMSO. The two insets highlight peaks B and B’ and compare their isotope distribution to the simulated ones for the molecular ions as labelled.

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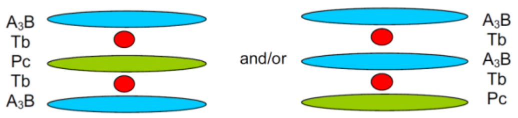
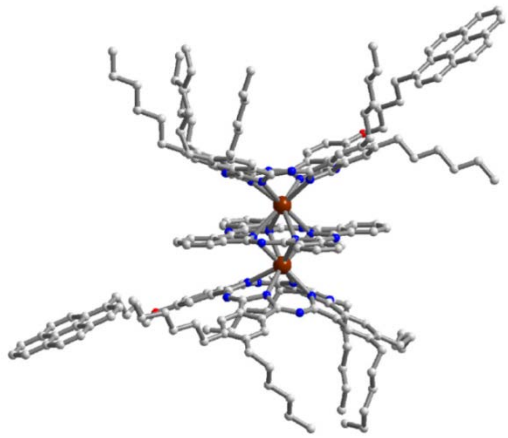
Typical results (continued)

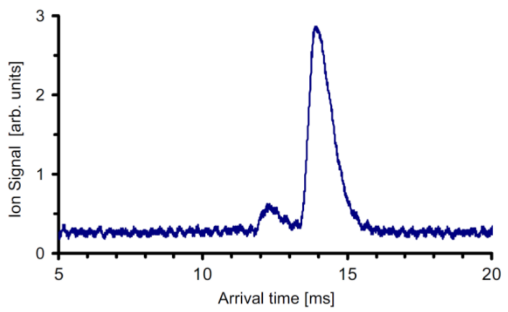
(a)



(b)



(c)



(a) Schematic structures of the two possible stacking configurations for $Tb_2(A_3B)_2Pc$ complex: a symmetric one (on the left) and an asymmetric one (on the right).
(b) Calculated structure of $(A_3B)_2PcTb_2$ (symmetric stacking)
(c) IMS arrival time distribution of the anion of $Tb_2(A_3B)_2Pc$ indicating the presence of two different isomers.

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X-Ray Photoelectron Spectroscopy

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. The gas cluster ion source additionally enables sputter depth profiling of organic materials while preserving the chemical information.

Contact

See KNMF website or contact the KNMF User Office.

Features

K-Alpha & K-Alpha+ XPS Instruments

- Mono AlKα X-ray source, spot size 30–400 µm (spatial resolution)
- Energy resolution < 0.5 eV FWHM Ag $3d_{5/2}$
- Rapid snap map chemical imaging (K-Alpha+)
- Ion gun for sputter depth profiling, 100–3000 eV Ar^+ ion energy (K-Alpha)
- Mono Atom and Gas Cluster Ion Source (MAGCIS), Mono Ar^+: 500 eV to 4 keV, Cluster Ar^+: 2 keV to 8 keV, cluster size: 75 up to 2000 atoms (K-Alpha+)
- Charge neutralisation system
- 50 x 60 mm^2 sample stage, sample height max. 15 mm
- Glove-box for atmosphere-contact free sample transfer: O_2 < 1ppm, H_2O < 1ppm (K-Alpha)

ESCA 5 / Alpha 110 analyser

- MgKα/AlKα dual anode X-ray source
- Energy resolution < 0.85 eV FWHM Ag $3d_{5/2}$ (MgKα)
- Angle resolved XPS
- Ion gun for sputter depth profiling, 300–3500 eV Ar^+ ion energy
- Max. sample dimensions 15 x 15 x 2 mm^2
- In-situ sample cooling and heating (-190–500 °C)
- 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

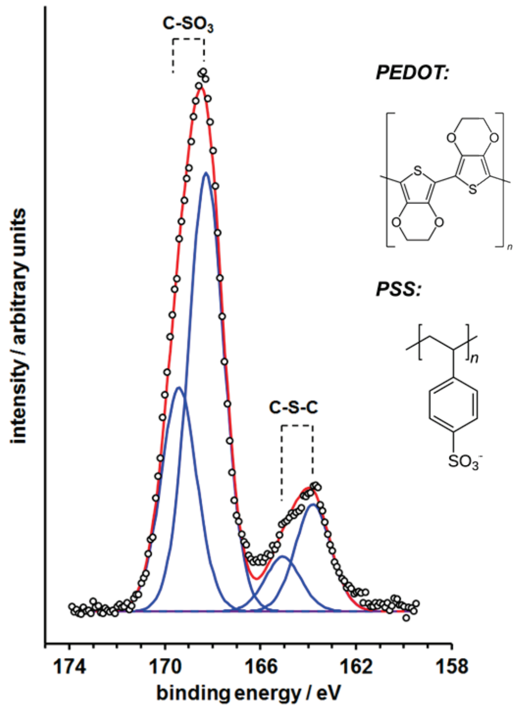

Fig. 1: Non-degraded S 2p XPS spectrum of a PEDOT:PSS thin layer after 1000 s Ar_{1000}^+ cluster ion sputtering using the K-Alpha+ MAGCIS source at 8 keV energy.

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Typical results (continued)

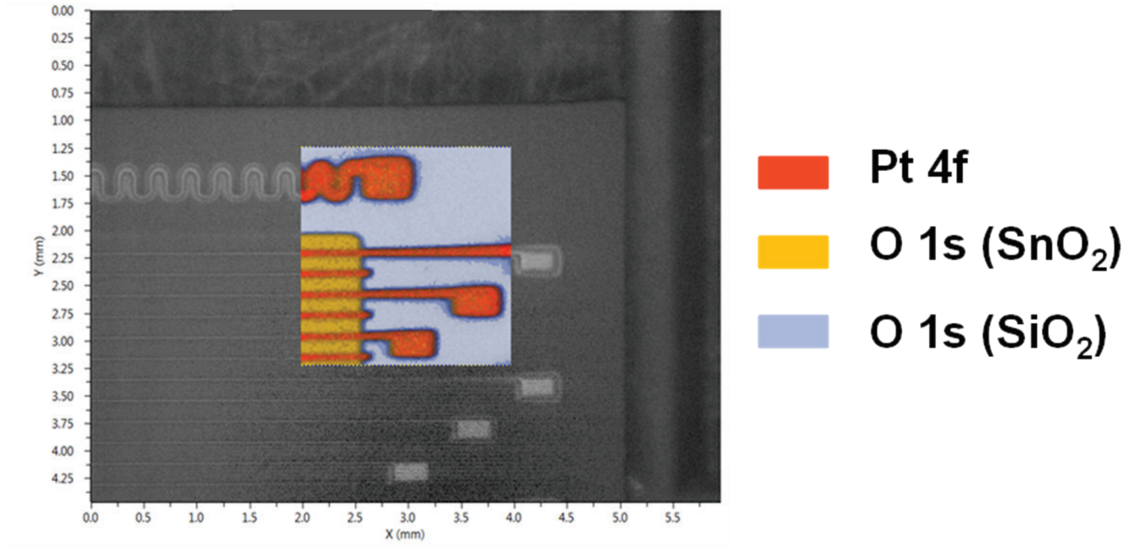

Fig. 2: Rapid snap map chemical image and respective overlaid video image of a gas sensor micro array (SiO_2 chip substrate, SnO_2 detector field subdivided by 50 µm Pt electrodes).

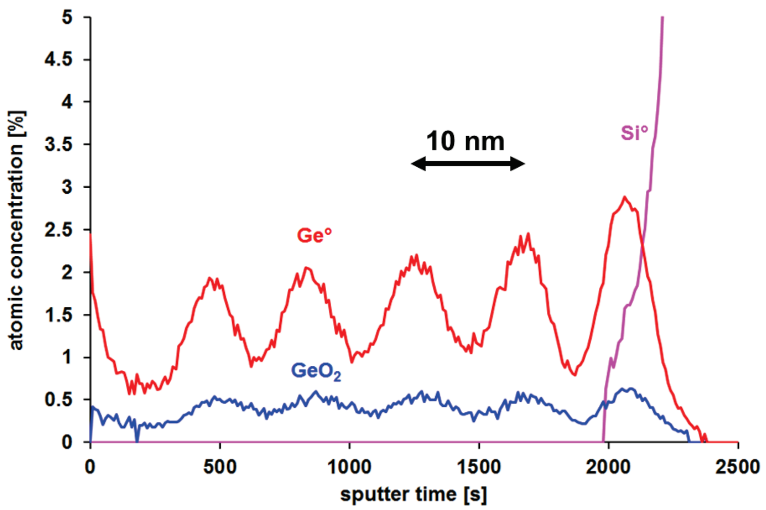

Fig. 3: Mono Ar^+ ion sputter depth profile of a sputter deposited Ge-SiO_2/SiO_2 multilayer system.

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