



Academia

FAHL Academia am 26.-27. 9.2005

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Tuesday, 27.9.2005

9:00

A comparison of different growth approaches for the preparation of ZnO-based nanostructures

Dr. Marc Kreye

Technische Universität Braunschweig

9:30

ZnO nanowire arrays - controlled growth and spatial resolved characterization

Priv.-Doz. Dr. Margit Zacharias

MPI für Mikrostrukturphysik, Halle/S

10:00

Optical modes in ZnO micro- and nanoresonators

Dipl.-Phys. Thomas Nobis, Universität Leipzig

10:30-11:00 **Coffee break**

11:00

Electrical Transport in Nanowires

Dr. Andreas Fuhrer, Lund University

11:30

Synthesis of compound-semiconductor nanowires and nanotubes

Prof. Dr. Sabine Schlecht

Philipps-Universität Universität Marburg

12:00-13:00 **Lunch**

13:00-14:30 **Visit of vulcano**

14:30-15:30 **Poster Session**

15:30

Sculptured thin films fabricated with glancing angle deposition (GLAD)

Dr. Eva Schubert, Leibniz-Institut für Oberflächenmodifizierung, Leipzig

16:00

Multifunctional nanowires, nanotubes and membranes for thermoelectric coolers, magnetic data recording and sensors

Dr. Kornelius Nielsch

MPI für Mikrostrukturphysik, Halle/S

16:30

Rolled-up nanotubes: Fabrication, properties and perspectives

Dr. Oliver Schmidt

MPI für Festkörperforschung, Stuttgart

17:00-17:15 **Refreshments**

17:15

Possibilities of alternative lithographic techniques for the fabrication of nanostructures

Priv.-Doz. Dr. Hartmut Leipner Interdisziplinäres Zentrum für Materialwissenschaften,

Universität Halle-Wittenberg

17:45

Nanofabrication with a mass-separated focused ion beam (FIB)

Dr. Lothar Bischoff

FZ Rossendorf

A comparison of different growth approaches for the preparation of ZnO-based nanostructures

M. Kreye

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ZnO materials have received broad attention in the last years due to its wide direct bandgap of 3.37 eV at room temperature, large exciton binding energy (60 meV in bulk ZnO), piezoelectric properties and excellent chemical and thermal stability. Due to its unique properties ZnO is a very promising material for short wavelength optoelectronic and electric applications. Moreover ZnO also shows a pronounced ability to build a variety of nanostructures. Nanosized materials are of special interest due to novel electrical, mechanical, chemical and optical properties that are introduced by surface and quantum confinement effects. Various chemical, electrochemical and physical growth techniques have been reported in the literature offering the possibility to produce ZnO nanostructures. Gas-phase approaches that are based on evaporation and condensation steps are frequently used due to their simplicity and high-quality products. However, the economic potential of gas-phase grown structures is restricted because these approaches require rather expensive and / or insulating substrate materials.

TEM and XRD methods clearly reveal that MOVPE grown ZnO nanopillars show an excellent structural quality, no visible defects and are virtually unstrained. High vertical alignment is combined with a high homogeneity of in plane orientation. However, the vertical growth mode was only observed using a-plane sapphire substrate materials. In photoluminescence experiments very sharp lines in the near band edge region are observed corresponding to donor bound excitons. The narrow half widths of only 0.5 meV indicate excellent optical quality of these unstrained gas-phase grown nanopillars. Typical growth parameters in MOVPE are $400 \leq T / ^\circ\text{C} \leq 700$ and slightly reduced pressures of about 400 mbar. We were also able to produce ternary nanopillar systems ZnMgO (Mg 9%) and ZnVO (V 1%). In case of the magnesium containing nanopillars we find a ZnMgO related and blue shifted PL signal that surprisingly appears only after postgrowth annealing at 850°C. The magnetic properties of the ZnVO system have been additionally characterised using the magnetic force microscopy MFM and SQUID experiments. Whereas the SQUID method shows only indications of ferromagnetism due to clear contributions even from the substrate material MFM clearly reveals the presence of ferromagnetic domains in ZnVO nanopillars even at room temperature.

In recent years aqueous chemical growth (ACG) approaches gain in importance because ACG is a cost-efficient, little substrate dependent, large scale and low temperature growth technique ($T \leq 90^\circ\text{C}$). We were able to generate highly homogeneous and vertical aligned, densely packed ($\sim 10^{10} \text{ cm}^{-2}$) and wafer scale arrays of ZnO nanopillars on various substrate materials Si (100, 111), sapphire, ITO coated glass and even on polymer substrates (PEN foil, silicones). By changing reactant concentrations the average nanopillar dimensions can be controlled. XRD measurements show zincite diffraction peaks, the absence of additional phases and confirm very high vertical alignment of the nanopillar arrays. Even from as-grown samples we find band-edge luminescence that is strong in compare with other ACG approaches in the literature. The broad visible luminescence may be attributed to the presence of interstitial oxygen ions rendering ACG an oxygen rich method. Using a postgrowth lithographic technique we were able to generate micrometer-scale patterned arrays of ZnO nanopillars on various substrate materials. ACG using conducting and flexible substrate materials shows a big economical impact and with further improvements this technology should find applications in solar cells, light emission, gas sensors and other devices.

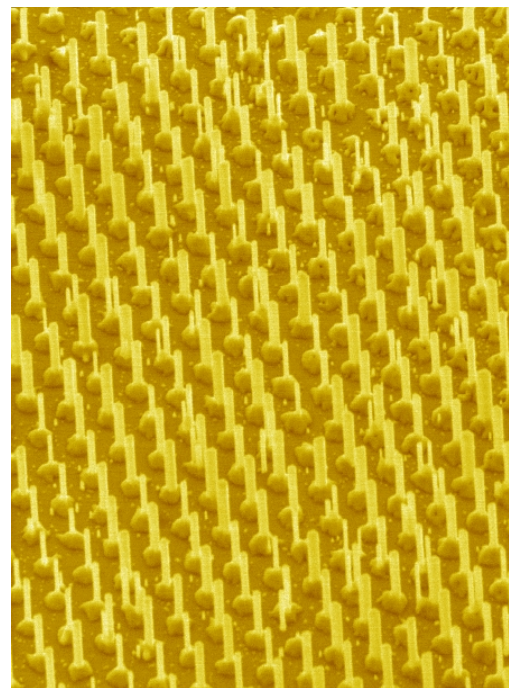
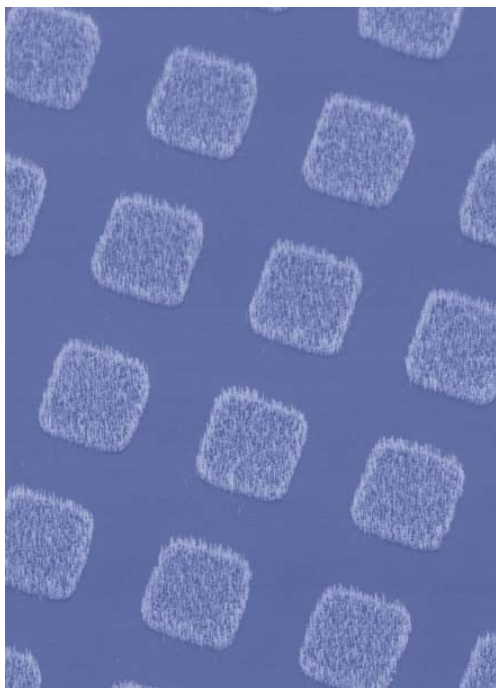
ZnO Nanowires and Nanowire Arrays: controlled growth and spatial resolved characterization

Hongjin Fan and Margit Zacharias

Max Planck Institute of Microstructure Physics, Halle, Germany

During the last years, semiconductor nanowires have received increasing attention as potential building blocks for nanoscale electronic and optical devices. A variety of methods has been employed for the manufacturing of nanowires. These methods include chemical vapor deposition, pulsed laser deposition, chemical beam epitaxy, metalorganic vapor phase epitaxy, and thermal evaporation methods. Frequently, the preparation requires complicated experimental setups or do not permit the desired degree of growth control with respect to position and size of the wires. The grown nanowires were often curved and tangled together, without any control over positions, size (etc.).

For vapor-liquid-solid growth the deposition of metal dots at specific positions can establish selective starting points for the growth. In the talk the growth of ZnO nanowires will be discussed. The successful fabrication of periodically arranged single-crystalline ZnO nanowire and nanopillar arrays are demonstrated by combining substrate patterning and the catalyst-directed epitaxial growth. The pillars show strong excitonic emissions up to room temperature. The growth process and the detailed investigation will be reported.



H.J. Fan, F. Fleischer, W. Lee, K. Nielsch, R. Scholz, M. Zacharias, U. Gösele; A. Dadgar, A. Krost, Patterned growth of aligned ZnO nanowire arrays on sapphire and GaN layers. *Superlattice and Microstructure* 36 (2004) 95.

H.J. Fan, W. Lee, R. Scholz, A. Dadgar, A. Krost, K. Nielsch, M. Zacharias, Arrays of vertically-aligned and hexagonal-arranged ZnO nanowires: a new template-directed approach. *Nanotechnology*, 16 (2005) 913.

Optical Modes in Zinc Oxide Micro- and Nanoresonators

Thomas Nobis*, Andreas Rahm, Michael Lorenz and Marius Grundmann

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Zinc oxide (ZnO) micro- and nanostructures are a unique system to study low order optical whispering gallery modes (WGMs). Their lateral dimensions are in the order of a few wavelengths of the resonant light wave and the refractive index of ZnO is comparably large ($n_r \sim 2$). Such features lead to total internal reflection and photon confinement within the nanostructures, similar to sound in a macroscopic acoustic whispering gallery. Using cathodoluminescence spectroscopy and polarization-resolved micro-photoluminescence spectroscopy we observed whispering gallery effects for hexagonal zinc oxide nanopillars down to the lowest order of mode numbers $m = 1$ [1]. To utilize the whispering gallery effect, e.g. to probe optical constants of individual nanostructures, a satisfying theoretical model for low order hexagonal WGMs is required.

Our talk presents such numerical analysis of low order hexagonal WGMs. Modes are theoretically investigated with respect to mode energies, line widths, mode degeneracy, mode patterns and polarization. Series of radially higher order modes have been found, and a system of mode numbering is given. The dependence on the refractive index is analyzed additionally.

Our results enable us to theoretically simulate experimentally detected resonance spectra. Line shapes and relative intensities of the resonant modes are well-reproduced without free parameter using literature values of the refractive index of ZnO bulk material. Therefore, we give an experimentally and theoretically consistent description of a real physical resonator system on nanoscopic scale.

[1] Th. Nobis, E. M. Kaidashev, A. Rahm, M. Lorenz, and M. Grundmann, Phys. Rev. Lett. **93**, 103903 (2004)

Transport studies on nanowire quantum dots

A. Fuhrer, C.I. Fath, A.E. Hansen, L.E. Jensen, M.T. Björk, M.W. Larsson, L. Samuelson

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Semiconducting nanowires are a hot topic due to their potential in biological, optical and electronic device applications. They have also spurred interest in more fundamental physical properties related to their intrinsic low dimensional nature.

In this talk we present our recent results on transport through quantum dots defined in InAs nanowires by inserting multiple InP barriers of variable thickness during Au-catalyst assisted CBE - growth. Both in single and double quantum dots we find a clear shell structure which can be linked to the confinement given by the hexagonal cross-section of our nanowires grown in (111)-direction. By varying the length and the coupling of such quantum dots we are able to tune the effective g-factor of the electrons as measured in the excited state spectrum and from shifts in the Coulomb peak positions. In these heterostructure quantum dots we use a common backgate to tune the number of electrons down to the last one. However, for more complex quantum electronic circuits a key ingredient is individual tunability of spin and charge on each dot and the coupling between them. In the second part of the talk we present a flexible approach on how to realize such tunability in InAs nanowires. We report on low-temperature transport measurements on single and double quantum dots defined using local gates to electrostatically deplete homogeneous InAs nanowires. This technique allows us to define multiple quantum dots along a semiconducting nanowire and tune the coupling between them.

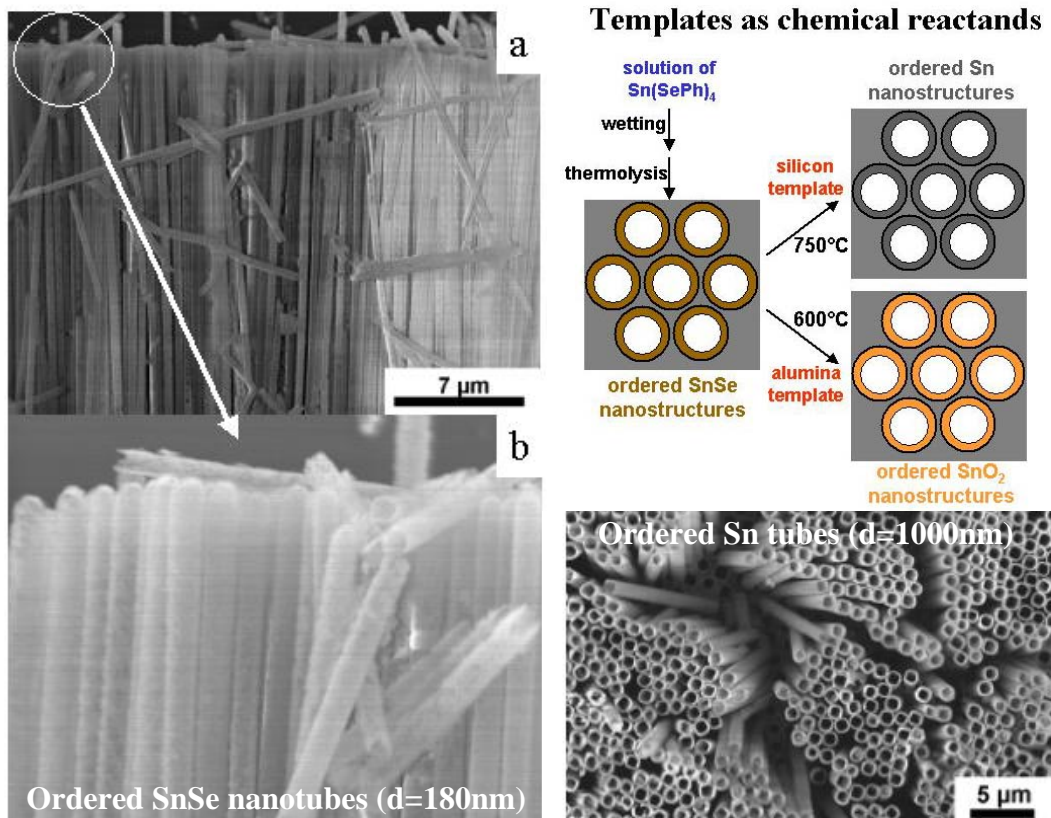
Synthesis of compound-semiconductor nanowires and nanotubes

S. Schlecht

Fachbereich Chemie, Philipps-Universität Marburg, Germany

Size and shape selective syntheses of semiconductor nanostructures have been playing an important and fundamental role in materials chemistry in recent years. Apart from preparative pathways involving surface capping additives for size selection and control, template assisted syntheses. Depending on the desired pore size, different classes of ordered porous supports such as mesoporous silica (SBA-15) with pore sizes of 3-9 nm, porous alumina membranes with pore diameters of 25-200 nm or macroporous silicon with pore sizes of 400-1000 nm were used.

To ensure a broad applicability of the synthetic concept, a purely physical infiltration of the pores with the precursor was carried out which had not to rely on a specific chemical binding of the precursor to the template walls. The single-source precursors were incorporated into respective the template either in the form of dilute solutions in a low boiling solvent or as a precursor melt and were subsequently thermolysed in the pores at 350°C. As single-source precursors we used aryl chalcogenolates of the general compositions $M(\text{QPh})_4$ ($M=\text{Ge}, \text{Sn}; \text{Q}=\text{S}, \text{Se}, \text{Te}$) and $M'(\text{QPh})_2\cdot\text{TMEDA}$ ($M'=\text{Zn}, \text{Cd}$) for the classes of IV/VI semiconductors and II/VI semiconductors. Hence, one family of precursor molecules could be used to produce different classes of semiconductor nanostructures of pre-defined size and shape. Additional types of compounds became accessible through the use of the templates as chemical reactands as shown for the conversion of SnSe nanostructures into single-crystalline SnO_2 nanowires (oxidising pore wall) and into single-crystalline nanotubes of elemental tin (reducing pore walls).



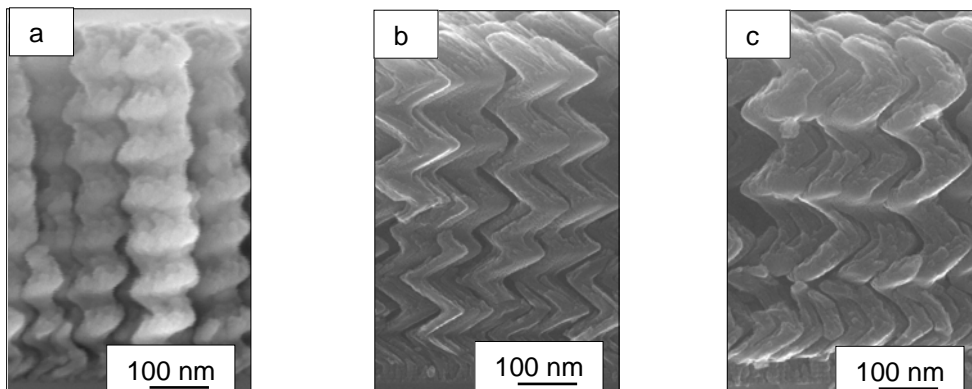
Sculptured thin films fabricated by glancing angle ion beam assisted deposition (GLAD)

E. Schubert, J. Fahlteich, F. Frost, T. Höche, and B. Rauschenbach

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Nanostructures with a complex geometry promise a high application potential for instance as optical and photonic materials, magnetic storage devices or sensors. Glancing angle deposition in combination with a computer controlled substrate rotation is a sophisticated method to customize manifold nanostructure varieties.

Ion beam sputtering of a silicon target is performed to generate a particle flow, which arrives at the [001] silicon substrate with an extremely oblique angle of incidence (typically 85 deg respective to the normal). By using this deposition configuration, highly porous thin films are created, which consist of slanted amorphous silicon needles with a diameter from 20 nm to 50 nm, and competitive growth mechanism due to geometric shadowing determine the nucleation and growth processes. By applying an appropriate substrate rotation during growth, the nanostructure geometry can be tailored. Chevrons and square spirals (Abb. 1b and Abb. 1c) are created with a symmetric stepwise substrate rotation of 180 deg and 90 deg, respectively. The fabrication of circular spirals, screws (Abb. 1a) and vertical posts is realised by a constant substrate rotation, and the nanostructure geometry depends on the ratio



from deposition rate to substrate rotation speed.

Abb. 1: Chiral Nanostructures from silicon grown by GLAD: screws (a), chevrons (b), and square spirals (c).

Regular nanostructure arrays are demonstrated by using prepatterned substrates, and a self-ordering phenomenon depending on the substrate rotation symmetry is found for the nanostructure growth on unseeded substrates.

Chiral thin films are investigated with emphasis on their structural and optical properties. For example, the self-ordering phenomenon yields to a periodic arrangements of the nanostructures, which act like nanogratings during interaction with visible light. Chiral nanostructures exhibit a fibre-like fine structure, whereas the fibres have a diameter of approximately 20 nm and the amount of fibres within one bundle determines the final diameter of the nanoobjects. Annealing experiments up to $T = 1000^{\circ}\text{C}$ have been performed to investigate recrystallisation effects and the resistance of silicon nanospirals against high temperature treatment.

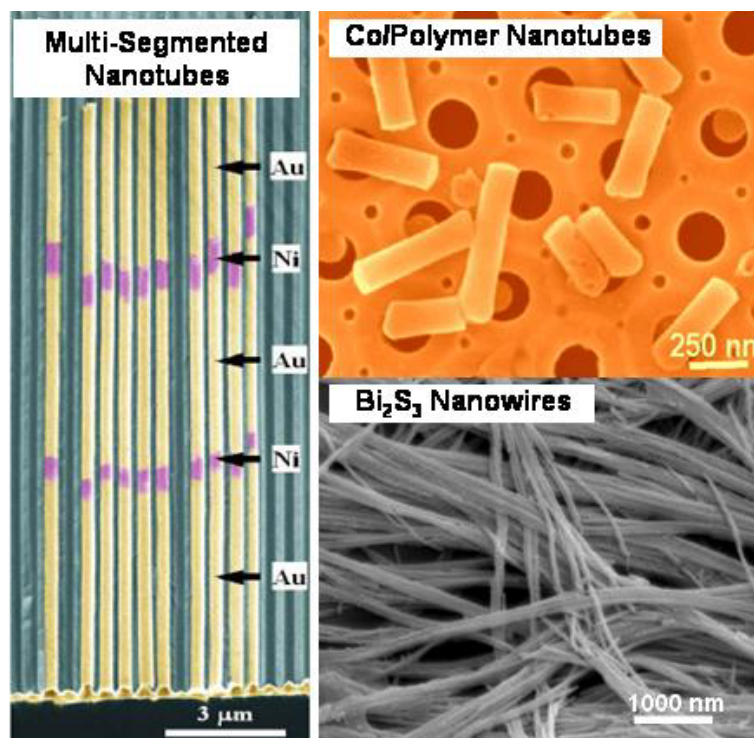
MULTIFUNCTIONAL NANOWIRES, NANOTUBES AND MEMBRANES FOR THERMOELECTRIC COOLERS, MAGNETIC DATA RECORDING AND SENSORS

Kornelius Nielsch, Woo Lee, Jana Sommerlatte, Ran Ji and Mato Knez

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Monodisperse nanorods are highly desirable for many applications in magnetic storage, sensing, nanoelectronics and for biomedical applications ranging from diagnostics of cancer cells to drug delivery. Self-ordered alumina membranes, based on an approach of Masuda et al. [Science 1995, 268, 1466], have attracted a lot of interest as a template material for the synthesis of 1D nanoobjects with a defined diameter (~10 %). In analogy to polycrystallites, the pore channels are hexagonally self-arranged in domains. By introducing imprint lithography in the fabrication process of our templates, a perfect pore arrangement on a cm^2 -scale is obtained. In the first part of this presentation an overview about the recent developments of porous alumina as a template system will be given.

In the second part we will show several examples on the synthesis of nanowires and nanotubes based on polymers, noble metals, ferromagnetic and thermoelectric materials. The nanowires and nanotubes are produced by using a pulsed electrodeposition process with conducting materials in alumina pore arrays. Ferromagnetic Co/polymer multilayer nanotubes have been synthesized based on a polymer wetting technique. Finally, a perspective outlook on conformal coating of the alumina membranes by atomic layer deposition (ALD) will be given.



Rolled-up nanotubes: Fabrication, properties and perspectives

Oliver G. Schmidt, R. Songmuang, R. Costescu, S. Mendach, and Ch. Deneke

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In this talk, we will shed light on the formation mechanisms and structural properties of rolled up nanotubes. We investigate the formation process by optical and electron microscopy as a function of number of layers and relevant layer thicknesses. We show that multiple stacks of strained bilayers form into multiple nanotubes, which can roll-up on the substrate surface with different velocities and diameters.

We also reveal the surprising result that thin films composed of a single material can roll-up into nanotubes, thus lifting the fundamental bilayer material restriction which is currently imposed on all roll-up processes. We present detailed results on single inversely rolled-up Si nanotubes, and we show that the effect is of general validity. Our structural characterisations include transmission electron microscopy and micro-Raman spectroscopy, which allow us to develop a first model for the formation process.

The talk will be concluded by recent preliminary developments in our group and future prospects in this exciting field of nanotechnology.

Possibilities of alternative lithographic techniques for the fabrication of nanostructures

Hartmut S. Leipner

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The fabrication of structures and templates in the nanometer scale for applications in the field of nanotechnology research and development is often a bottleneck in time and costs. Commonly used lithographic techniques, e. g. electron beam lithography, are expensive, complicated, and not always available. Among alternative lithographic techniques, different variants of nanoimprinting using basically a prestructured mold have already reached a high level of maturity with commercially available instruments. A short overview is given about the possibilities of nanoimprint lithography.

Nanosphere lithography (NSL) is another alternative lithographic technique which is suitable for the optimization of processes in nanotechnology. Especially in research, flexible and cost-effective nanostructuring procedures are required. There is a high demand for a fast technique using small wafers and low quantities, which allows the quick variation of parameters for process optimization. Especially in cases, where “only” simple periodic structures with varying parameters are needed, nanosphere lithography may be the method of choice.

The basic principle is the deposition of a monolayer or bilayer of monodisperse particles. A broad range of substrates can be used. The deposited particle layer (e. g. monodisperse polystyrene spheres) act as a mask for further lithographic steps, for instance the deposition of metal. By the variation of deposition parameters, different structures like dots, rings, pyramids etc. can be produced. We have adopted the NSL technique to fabricate highly ordered metal dots, which are used in a further step for the growth of semiconductor nanowires on different substrates. The uniformity of the wires and their arrangement are defined by the NSL pre-patterning of the substrate.

Nanofabrication with a mass-separated FIB

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In the last two decades focused ion beams (FIB) have become a very useful tool for many tasks in micro- as well as in nano-technology. Probe sizes of less than 10 nm and current densities of more than 10 Acm^{-2} are now available and allow to use these beams for many applications. Integrated circuit repair and modification, failure analysis, TEM specimen preparation, lithographic mask repair or FIB lithography as well as the writing maskless implantation are the main application in microelectronic research and industry. Especially, in R&D the FIB is very advantageous because of its high spatial resolution and its flexibility varying dose, energy and pattern design on one chip, or even in one structure detail. Therefore also in the field of basic and applied research the FIB became more important, including plasmonics, photonics and nano-optics.

Most of the FIB systems employ a Ga liquid metal ion source (LMIS). Due of the broad spectrum of applications with Ga beams many cases suffer from the impurity incorporation. For special purposes like writing ion implantation or ion mixing in the μm - or nm range different ion species are needed. Therefore alloy liquid metal ion sources (LMIS) are used and the FIB column must be equipped with a mass separation, namely an ExB mass filter system.

The assembly of a modern FIB system is presented and the development and investigation of different alloy LMIS`s working with other materials than Ga as well as their corresponding application in FIB nano-fabrication will be discussed.

The energy distribution of the ions from an alloy LMIS is one of the determining factors for the performance of an FIB column. Different source materials like $\text{Au}_{73}\text{Ge}_{27}$, $\text{Au}_{82}\text{Si}_{18}$, $\text{Au}_{77}\text{Ge}_{14}\text{Si}_9$, $\text{Co}_{36}\text{Nd}_{64}$, $\text{Er}_{69}\text{Ni}_{31}$, and $\text{Er}_{70}\text{Fe}_{22}\text{Ni}_5\text{Cr}_3$ were investigated with respect to the energy spread of the different ion species as a function of emission current, ion mass and emitter temperature. The alloy LMIS`s discussed above have been used in the Rossendorf FIB system IMSA-100 and later in the improved IMSA-OrsayPhysics machine especially for writing implantation to fabricate nm pattern without any lithographic steps. A Co-FIB was applied for the ion beam synthesis of CoSi_2 nano-structures. Also the Co-FIB was applied to modify thin magnetic multi-layers in the nm-scale. Additionally, the possibility of varying the current density of the FIB by changing the pixel dwell-time was used to investigate the radiation damage and the dynamic annealing in Si and SiC at elevated implantation temperatures. Furthermore, a broad spectrum of ions was employed to study the sputtering process depending on temperature, angle of incidence and ion mass on a couple of target materials using the volume loss method. Especially this technique was used for the fabrication of various kinds of micro-tools and other 3D devices. A nano-hole was made into an AFM tip acting as an aperture in a single ion implantation project.

The presented examples underline useful application possibilities to different tasks of nanotechnology (or nanofabrication) applying FIB tools equipped with a mass separation system to chose from a broad spectrum of ion species the desired one.