

## DS 15: Focus Session: Direct-Write Nanofabrication and Applications I (Electron Beam Induced Processing) (joint session DS/TT)

Part I: Advances in Focused Particle Beam Processing & New Approaches

Focused electron beam induced deposition (FEBID) and focused electron beam induced etching (FEBIE) are direct-write approaches for the fabrication of 2D- and 3D-nanostructures made from different materials, such as superconductors, magnetic materials, alloys and intermetallic compounds, as well as meta-materials in which suitable materials combinations result in a desired functionality for various application fields (strain / magnetic / dielectric sensing, multi-functional scanning probe sensors, 3D plasmonic structures, 3D magnetic structures etc).

The Focus Session aims at providing a concentrated presentation of various new developments of the most versatile direct-write techniques for functional nanostructures to a broader audience within the condensed matter community.

### Organizers:

- Michael Huth, Physikalisches Institut, Goethe-Universität, Frankfurt, Germany
- Harald Plank, FELMI-ZFE, TU Graz, Austria

Time: Wednesday 9:30–12:30

Location: H32

### Invited Talk

DS 15.1 Wed 9:30 H32  
**3D-Nanoprinting with Focused Electron Beams. Advances and Applications** — •ROBERT WINKLER<sup>1</sup>, JASON D FOWLKES<sup>2,3,4</sup>, JÜRGEN SATTELKOW<sup>1</sup>, PHILIP D RACK<sup>2,3,4</sup>, and HARALD PLANK<sup>1,5</sup>  
 — <sup>1</sup>Christian Doppler Laboratory - DEFINE, Institute of Electron Microscopy, Graz University of Technology, 8010 Graz, Austria — <sup>2</sup>Bredesen Center for Interdisciplinary Research, The University of Tennessee, Knoxville, 37996, USA — <sup>3</sup>Nanofabrication Research Laboratory, Center for Nanophase Materials Sciences, Oak Ridge National Laboratory, Oak Ridge, 37831, USA — <sup>4</sup>Materials Science and Engineering Department, The University of Tennessee, Knoxville, 37996, USA — <sup>5</sup>Graz Centre for Electron Microscopy, 8010 Graz, Austria

While 3D printing of objects down to the micrometer scale is well established, techniques for controlled additive manufacturing at the nanoscale are only few. Based on the progress in recent years, Focused Electron Beam Induced Deposition (FEBID) has evolved into a 3D nano-printing technology, allowing mask-less direct-write fabrication of complex 3D nano-architectures on almost any substrate. The growing availability of different precursor types expand the functionalities of those FEBID structures from electrically over magnetically towards optically active purposes, enabling applications, which have been very challenging in the past. Here, we introduce the technology and sketch possibilities and limitations for a comprehensive FEBID portfolio picture. We focus on recent advances in accuracy and predictability based on local heating effects and finally present selected applications of such 3D-nanoprinted structures in research and industry.

DS 15.2 Wed 10:00 H32

**Modeling FEBID frequency maps: Lateral deposit resolution and surface diffusion** — JAKUB JURCZYK<sup>1,2</sup>, CZESLAW KAPUSTA<sup>2</sup>, and IVO UTKE<sup>1</sup> — <sup>1</sup>Empa, Swiss Federal Laboratories for Materials Science and Technology, Feuerwerkstrasse 39, CH-3602 Thun, Switzerland — <sup>2</sup>AGH University of Science and Technology Krakow, Al. Mickiewicza 30, 30-059 Kraków, Poland

Focused electron beam induced deposition (FEBID) is governed by four main processes: adsorption, desorption, surface diffusion and dissociation of precursor molecules on the sample surface [1],[2]. All of them influence growth rate, lateral resolution and shape of deposited structures; hence we can distinguish four different deposition regimes: reaction-rate limited, mass transport limited, diffusion enhanced and the combination of the two latter [3]. In their recent work, Sanz-Hernandez et al. [4] visualized these regimes based on characteristic frequencies for every process, creating frequency maps for the growth rates and coverage [4]. In this contribution we expand this approach to include the lateral deposit resolution and the influence of surface diffusion into the frequency maps. Results of simulations will be discussed in the context of their physical basis and future designed experiments. [1] I. Utke et al., J. Vac. Sci. Technol. B, 26, (2008), 1197-1276; [2] M. Toth et al. Beilstein J. Nanotech., 6, (2015), 1518\*1540[3]; A.

Szkudlarek et al., Appl. Phys. A, 117, (2014), 1715\*1726; [4] D. Sanz-Hernandez et al., Beilstein J. Nanotechnol., 8, (2017), 2151\*2161

DS 15.3 Wed 10:15 H32

**On the reduction of proximity effects by exploring (metal-) organic materials as substrates/resists for gas-assisted electron beam lithography** — CHRISATIAN PREISCHL, ELIF BILGILISOY, FLORIAN VOLLNHALS, and •HUBERTUS MARBACH — Physikalisches Chemie II, FAU Erlangen-Nürnberg

We investigated organic and metal-organic materials as substrates/resists in different Focused Electron Beam Induced Processing (FEBIP) techniques. Here, FEBIP methods rely on the local decomposition of the volatile precursors Fe(CO)<sub>5</sub> and Co(CO)<sub>3</sub>NO, by the direct impact of the focused electron beam (Electron Beam Induced Deposition, EBID) or through the interaction of the precursor with pre-irradiated/activated surface areas (Electron Beam Induced Surface Activation, EBISA).<sup>1-3</sup> The investigated materials range from porphyrin layers<sup>1,2</sup> over surface anchored metal-organic frameworks (SURMOFs)<sup>3</sup> to self-assembled monolayers (SAMs).<sup>4</sup> Application of our surface science approach, i.e. working in an ultra-high vacuum environment, allows to obtain chemically well-defined deposits. A major advantage of the used materials are reduced electron proximity effects, i.e. reduced electron scattering and quenching of secondary electrons within the latter materials.<sup>1-3</sup> An illustrative example is the fabrication of test structures with Fe(CO)<sub>5</sub> on a SURMOF with an average line width value below 10 nm.<sup>3</sup>

[1] Marbach, H., Appl. Phys. A, 117 (2014) 987-995. [2] Drost, M., et al., Small methods, 1(2017)1700095. [3] Drost, M., et al., ACS Nano. 12 (2018) 3825 . [4] Turchanin, A., et al., Adv. Mater. 28 (2016) 6075.

DS 15.4 Wed 10:30 H32

**FEBIP on Self-Assembled-Monolayers and Carbon Nanomembranes** — •CHRISTIAN PREISCHL<sup>1</sup>, ELIF BILGILISOY<sup>1</sup>, FLORIAN VOLLNHALS<sup>1</sup>, LE HOANG LINH<sup>2</sup>, SASCHA KOCH<sup>2</sup>, ARMIN GÖLZHÄUSER<sup>2</sup>, and HUBERTUS MARBACH<sup>1</sup> — <sup>1</sup>Physik. Chemie II, FAU Erlangen-Nürnberg, GER — <sup>2</sup>PSS, Bielefeld University, GER

In our approach, we investigate two different FEBIP methods in UHV on Self-Assembled-Monolayers (SAM) and on nanometer thick Carbon Nanomembranes (CNM). These thin CNM sheets can be produced out of SAMs by electron-induced crosslinking.<sup>[1]</sup> The two FEBIP methods of choice are EBID<sup>[2]</sup> and Electron-Beam-Induced-Surface-Activation (EBISA). In EBISA, the surface is locally activated by an electron beam and the subsequently dosed precursor is catalytically decomposed at the activated sites and forms a deposit.<sup>[3]</sup> These two approaches were explored on 1,1',4',1''-terphenyl-4-thiol (TPT) and the corresponding cross-linked CNM with Fe(CO)<sub>5</sub> and Co(CO)<sub>3</sub>NO. Whereas EBID works with both precursors on both substrates, EBISA can only be driven successfully on the non cross-linked TPT with

Fe(CO)<sub>5</sub>. Regarding this result, we observe a chemical selectivity in EBISA between the two precursors, which was already reported in previous studies on different substrates.<sup>[4]</sup> Furthermore upon crosslinking, TPT loses its catalytic activity towards the EBISA process.

<sup>[1]</sup> A. Turchanin, A. Gözlhäuser, *Adv. Mater.* 28 (2016), 6075 <sup>[2]</sup> W. van Dorp, C.W. Hagen, *J. Appl. Phys.* 104 (2008), 081301 <sup>[3]</sup> H. Marbach, *Appl. Phys. A* 117 (2014), 987 <sup>[4]</sup> Drost et al., *Small Methods* 1 (2017), 1700095; M. Drost et al., *ACS Nano.* 12 (2018), 3825

DS 15.5 Wed 10:45 H32

**Fabrication of Photonic and Optomechanics Devices in hBN by Electron Beam Induced Etching** — ●JOHANNES FROECH<sup>1</sup>, SEJEONG KIM<sup>1</sup>, PRASOON SHANDILYA<sup>2</sup>, BISHNUPADA BEHERA<sup>2</sup>, CHRIS HEALY<sup>2</sup>, JAMES BISHOP<sup>1</sup>, MATTHEW MITCHELL<sup>2</sup>, DAVID LAKE<sup>2</sup>, PAUL BARCLAY<sup>2</sup>, IGOR AHARONOVICH<sup>1</sup>, and MILOS TOT<sup>1</sup> — <sup>1</sup>University of Technology Sydney, Ultimo, NSW, 2007, Australia — <sup>2</sup>University of Calgary, Calgary, AB, T2N 1N4, Canada

Exceptional efforts have been undertaken in recent years to identify suitable platforms for solid state quantum photonic technologies. Several schemes exploit materials that typically host on-demand single photon emitters and can be easily processed in a robust and reliable manner to yield functional nanostructures. A potential material for applications in this field is hexagonal Boron Nitride (hBN), based on the discovery of room-temperature, stable, ultra-bright quantum emitters. However, until recently, fabrication of complex hBN geometries was not viable. Here, we demonstrate new processing approaches for the fabrication of complex photonic and optomechanics nanostructures in suspended hBN and hBN/ Si hybrid systems using the technique of Electron Beam Induced Etching (EBIE). It is minimally invasive and allows for post fabrication editing to tune optical properties. In combined systems, the etching technique is highly selective and allows for precise and maskless fabrication. Overall, our methodology and results set the foundation for cavity quantum electrodynamics experiments and further work in integrated optomechanics systems to be performed utilizing hBN quantum emitters.

DS 15.6 Wed 11:00 H32

**Energy from Green House Gas Stored in Nanogranular Material** — ●HANS KOOPS — HaWilKo GmbH, Ober-Ramstadt, Germany

Light energy from the sky having Ultra-Violet to 10  $\mu\text{m}$  wavelengths is stored as Bosons in nano-granular material. The upper atmosphere of the earth contains layers of green-house material and this emits infrared radiation with 380 W/ m<sup>2</sup>, as measured by NASA in 2009. Nanogranular compound material, Platinum nanocrystals in Fullerene crystals layers, can absorb the IR-radiation and stores it in electron-hole-Bosons with parallel spin. There the electrons can tunnel to the close by conductor, which carries a potential higher than the Boson-layers. Experiments at KNMF in KIT delivered from a Pt/C ribbon of 150 nm thickness and 1  $\mu\text{m}$  width a 4 V a current of 0,6 A without melting the nano-granular Pt/C ribbon. The stored charge can be moved using a field gradient, which shifts the Bosons to the end of the material. There the electrons can tunnel to the close by conductor, which carries a potential higher than the Boson-layers. Experiments at KNMF in KIT delivered from a Pt/C ribbon of 150 nm thickness and 1  $\mu\text{m}$  width a 4 V a current of 0,6 A without melting the nano-granular Pt/C ribbon.

15 min. break.

Invited Talk

DS 15.7 Wed 11:30 H32

**Resist-free fabrication of graphene devices using focused ion beam patterning and direct-write ALD** — ●AGEETH BOL — Eindhoven University of Technology, Eindhoven, the Netherlands

Graphene has long been proposed as ideal candidates to replace silicon in future nanoelectronic devices and has therefore attracted considerable attention from the scientific community. Regardless, graphene struggles to leave the lab as many challenges for large-scale integration still exist. This presentation addresses one of these challenges:

the resist-free fabrication of graphene devices. For the fabrication of graphene devices, graphene sheets must be patterned into individual devices, and then contacted to form electrical connections. The conventional approach involves lithography using resist films. To avoid contamination by resist residues as much as possible, a direct patterning and contacting approach was developed. We showed that a focused ion beam (FIB) is able to directly etch graphene from a substrate. By optimizing the pressure as well as reducing the amount of ions used, ion scattering could be minimized, making FIB patterning a feasible alternative to conventional lithography. Next, the FIB-patterned graphene was used to fabricate and characterize electrical devices. For the first time, Pt contacts were deposited by using a combination of electron-beam induced deposition and area-selective Pt which further avoids the use of resist films. The ALD-contacted devices show remarkable improvements compared to conventionally deposited Pt contacts.

DS 15.8 Wed 12:00 H32

**Atomic layer deposition on electron beam written nanostructures** — CASPAR HAVERKAMP<sup>1</sup>, HANNO KRÖNCKE<sup>1</sup>, PATRYK KUSCH<sup>2</sup>, FELIX OERTEL<sup>1</sup>, CATHERINE DUBOURDIEU<sup>1,3</sup>, STEPHANIE REICH<sup>2</sup>, and ●KATJA HÖFLICH<sup>1</sup> — <sup>1</sup>Helmholtz-Zentrum Berlin, Hahn-Meitner-Platz 1, 14109 Berlin — <sup>2</sup>Freie Universität Berlin, Fachbereich Physik, Arnimallee 14, 14195 Berlin — <sup>3</sup>Freie Universität Berlin, Institut für Chemie und Biochemie, Takustr. 3, 14195 Berlin

The uniform coverage of complex three-dimensional structures is highly beneficial for the process of direct electron beam writing. Thereby, crucial issues in thermal and chemical stability of electron beam written structures can be successfully addressed, while in other cases the desired material response may be realized. Atomic layer deposition (ALD) is a self-limited deposition technique, that allows to deposit conformal ultrathin films on surfaces. The deposition consists of an iterative growth sequence of four steps. First, precursor molecules bind to the surface forming a monolayer. Then, excess precursor molecules and volatile byproducts are purged from the reactor. In a third step, the co-reactant is introduced to react with the chemisorbed precursor molecules. Finally, all volatile products are purged again. While mostly known for the deposition of oxides, other materials and especially metals are available for ALD as well. Therewith, atomic layer deposition constitutes an ideal counterpart to trigger various applications of direct electron beam writing. Examples are demonstrated for plasmonic antennas of different types, optimized for chiroptical interaction, tip-enhanced Raman scattering or nonlinear interactions.

DS 15.9 Wed 12:15 H32

**Towards all-metallic nano-structures using FEBID and ALD** — ●PETER GRUSZKA and MICHAEL HUTH — Goethe Universität, Frankfurt am Main, Deutschland

In recent years, conventional methods of nano-structuring are slowly reaching their lower limits. A novel bottom-up and maskless approach emerged<sup>[1]</sup>, which combines focused electron beam induced deposition (FEBID) and area-selective atomic layer deposition (AS-ALD). FEBID is a serial, bottom-up and direct-write technique yielding structures with superior lateral resolution (< 10 nm), but with poor material quality. In contrast, ALD and especially AS-ALD are parallel bottom-up approaches with exceptional thickness control in the sub-nm regime resulting in high purity films.

We successfully performed the AS-ALD process in our custom ALD micro-reactor on ultra-thin platinum seed layers prepared in a Nova 600 Dual Beam scanning electron microscope by FEBID. The seed layers were purified with a technique developed by Sachser et al.<sup>[2]</sup>. Additionally, we monitored the AS-ALD process via in-situ conductance measurements which enabled us to tune the resistance to a desired value. Low-temperature measurements on standard four-probe structure show metallic behaviour with an RRR of about 2.6 and a Debye temperature of about 230K. First results on high-resolution nanostructure fabrication by FEBID/AS-ALD and their low-temperature transport characteristics are presented.

[1] Mackus, et al., *J. Appl. Phys* 107 (2010), 116102

[2] Sachser, et al., *ACS Appl. Mater. Interfaces* 6 (2014), 15868