

## KFM 16: Lithography I: Focused Electron Beam Induced Processing: 3D Nano-Printing for Material Science (Focused Session): Morning Session (joint session DS/KFM)

Considering 3D printing using fused-deposition modeling or higher-resolution variants with lasers applicable to polymers and metals, an analogous approach exists on the nanometer scale. With the aid of focused electron beam-induced deposition (FEBID) it is possible to create solid-state structures on the nanoscale. However, in contradistinction to large-scale 3D printing of simple plastic or metallic structures, FEBID is able to directly provide nano-materials with a wealth of interesting electronic, optical and magnetic properties. Due to this, focused electron beam-induced deposition has experienced a rapid expansion in the breadth of its application fields over the last 10 years. FEBID uses precursor gases which, being adsorbed on a surface, are dissociated in the focussed electron beam to form the deposit. Intensive research has pushed the capabilities of FEBID in two important areas. It is now possible to obtain fully metallic nanostructures of Fe, Co and FeCo-alloys and also of Au and Pt. In addition, very recently the simulation-guided nano-manufacturing of 3D structures has matured to such a degree that even complex 3D objects can now be fabricated under controlled conditions. The focused session will address these new developments spanning the range from the fundamentals of electron-precursor interaction, covering aspects of the rational design of optimized precursors, and showing recent work on superconducting, magnetic and plasmonically active materials, both in 2D and 3D.

Organized by

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Time: Wednesday 9:30–13:00

Location: H 2032

### Invited Talk KFM 16.1 Wed 9:30 H 2032

**3D direct-write nanofabrication using an electron beam** — ●JASON FOWLKES<sup>1</sup>, ROBERT WINKLER<sup>2,3</sup>, EVA MUTUNGA<sup>4</sup>, BRETT LEWIS<sup>5</sup>, HARALD PLANK<sup>2,3</sup>, and PHILIP RACK<sup>5</sup> — <sup>1</sup>Center for Nanophase Materials Sciences, Oak Ridge National Laboratory, Oak Ridge, Tennessee 37831, USA — <sup>2</sup>Institute for Electron Microscopy and Nanoanalysis, Graz University of Technology, 8010 Graz, Austria — <sup>3</sup>Graz Centre for Electron Microscopy, 8010 Graz, Austria — <sup>4</sup>Bredesen Center for Interdisciplinary Research, The University of Tennessee, Knoxville, 37996, USA — <sup>5</sup>Department of Materials Science and Engineering, The University of Tennessee, Knoxville, 37996, USA

The deposition of 3D nanomaterials with precise geometry and function constitutes a major goal of nanoscience. Currently, the preeminent method for nanoprinting is focused electron beam-induced deposition (FEBID). During FEBID, the electron beam is scanned along a surface inducing the fragmentation and deposition of adsorbed precursor molecules. Until recently, the suite of 3D objects that could be deposited was limited by the trial and-error nature of experiments and poor material quality. Our team has taken significant steps toward overcoming both roadblocks, the former being the focus of the current presentation. A FEBID CAD program will be presented that makes it possible to deposit complex, 3D nanoscale mesh style objects spanning micrometer length scales. A FEBID simulation will also be discussed. The simulation to CAD to experiment process flow will be demonstrated for the case of tailoring mesh object nanowire cross-sections.

### Invited Talk KFM 16.2 Wed 10:00 H 2032

**Nanosuperconductivity with Focused Particle Beam Induced Deposition structures** — ●ROSA CÓRDOBA<sup>1,2</sup>, JAVIER SESÉ<sup>2,3</sup>, and JOSÉ MARÍA DE TERESA<sup>1,2,3</sup> — <sup>1</sup>Instituto de Ciencia de Materiales de Aragón (ICMA), CSIC - Universidad de Zaragoza, Spain — <sup>2</sup>Departament de Física de la Materia Condensada, Universidad de Zaragoza, 50009 Zaragoza, Spain — <sup>3</sup>Laboratorio de Microscopías Avanzadas (LMA), Instituto de Nanociencia de Aragón (INA), Universidad de Zaragoza, Spain

In this contribution, we will show an inclusive scenario of Focused Particle Beam Induced Deposition (FEBID/FIBID) to grow nanosuperconductors and nanomagnets by using as a primary beam, heavy ions

(Ga<sup>+</sup> FIBID), light ions (He<sup>+</sup> FIBID) and electrons (FEBID). First, Ga<sup>+</sup> FIBID nanosuperconductors will be introduced, which include the mechanical properties of three-dimensional (3D) nanowires [1], the non-local vortex transport in sub-50 nm nanowires [2] and the magnetotransport properties of sub-10 nm superconducting nanowires [3]. Second, He<sup>+</sup> FIBID nanotubes will be presented, highlighting their specific growth method, superconducting properties and microstructure [4]. Finally, FEBID 3D magnets will be shown, which are integrated in a hybrid (superconductor/ferromagnet) system [5,6]. [1]Córdoba et al., Nanotechnology 28-44 445301 (2017). [2]Córdoba et al., manuscript submitted to Applied Physics Letters. [3]Córdoba et al., manuscript in preparation. [4]Córdoba et al., manuscript submitted to Nano Letters. [5]Córdoba et al., Nanotechnology 27 355301 (2016). [6]Rouco et al., Sci. Rep. 7 5663 (2017).

### Invited Talk KFM 16.3 Wed 10:30 H 2032

**Chemistry for Electron-Induced Nanofabrication** — ●PETRA SWIDEREK — University of Bremen, Institute for Applied and Physical Chemistry, Bremen, Germany

The European COST Action CELINA (Chemistry for Electron-Induced Nanofabrication [1]) has, from 2013 to 2017, created a research network that aims at advancing focused electron beam induced deposition (FEBID) processes. It has done so by stimulating collaborative research that unravels the chemical reactions that are fundamental to FEBID, develops novel and improved precursor molecules, and tests their performance in the actual FEBID process. CELINA has thus assembled under its roof groups with expertise in electron-driven chemistry, precursor synthesis, and experts in FEBID from both academia and industry. This multidisciplinary effort is needed because of the many different physical and chemical aspects involved in the formation and processing of FEBID deposits as well as in their applications.

This contribution gives an overview of CELINA's research program and highlights some of its results. Furthermore, it will discuss the different types of chemical processes inherent in FEBID and how they can be investigated using a combination of gas phase mass spectrometry and surface science experiments. Understanding and controlling each of these different chemistries poses significant challenges but is the key to ultimate deposit purity, spatial resolution, and deposition speed.

References: [1] <http://celina.uni-bremen.de/celina>

**Invited Talk** KFM 16.4 Wed 11:00 H 2032

**The direct electron beam writing of plasmonic nanostructures** — ●KATJA HÖFLICH — Helmholtz Zentrum Berlin für Materialien und Energie, Hahn-Meitner-Platz 1, D – 14109 Berlin, Germany

Future IT systems will rely on photons instead of electrons. The potentially huge bandwidth of photons and their extremely small switching times render light indispensable for telecommunication and for information processing. The use of plasmonic nanostructures constitutes a promising approach for nanoscale optical devices since their minimum geometric features are not restricted by the diffraction limit.

The technique of electron beam induced deposition (EBID) has the potential to evolve as a novel route for the fabrication of complex plasmonic devices. EBID nanostructures are grown by the local dissociation of precursor molecules in the focus of an electron beam. While the focused electrons account for minimum structural features, the direct writing provides access to three dimensions in a single step.

A major challenge lies at depositing pure materials. It can be addressed by testing novel precursor compounds, oxidation-based purification or by using EBID nanostructures as a scaffold to be coated with the metal of choice. Recent examples of EBID-based plasmonic nanostructures include silver and gold helices with a strong dichroism in the visible range as well as silver nanostructures based on a novel carboxylate precursor.

**15 min. break.**

KFM 16.5 Wed 11:45 H 2032

**Direct printing of 3D nano-structures via focused electron beam induced deposition: Pattern generation** — ●LUKAS KELLER and MICHAEL HUTH — Institute of Physics, Goethe University, Max-von-Laue-Str. 1, 60438 Frankfurt am Main, Germany

Fabrication of three-dimensional (3D) nano-architectures by focused electron beam induced deposition (FEBID) has matured to a level that highly complex and functional deposits are becoming available for nanomagnetics and plasmonics. The main issue of generating a desired 3D nano-structure is the control of the electron beam in the x-y-plane. However, the generation of suitable pattern files that define the electron beam deflection at any time during the deposition and reliably map the desired target 3D structure from a purely geometrical description to a shape-conforming 3D deposit is non trivial. Here we present our implementation of a pattern file generator that handles proximity effects, corrects for height-dependent precursor coverage and avoids shadowing effects regarding the directed component of the precursor flux. Several examples of successful 3D nano-fabrication using different precursors are presented that attest the effectiveness of the implementation.

KFM 16.6 Wed 12:00 H 2032

**Direct-Write Fabrication of Electric and Thermal High-Resolution Nano-Probes on Self-Sensing AFM Cantilever** — ●JUERGEN SATTELKOW<sup>1,2</sup>, JOHANNES FROECH<sup>1,2</sup>, ROBERT WINKLER<sup>1,2</sup>, CHRISTIAN SCHWALB<sup>3</sup>, ERNEST FANTNER<sup>3</sup>, VLADO STAVROV<sup>4</sup>, and HARALD PLANK<sup>1,2</sup> — <sup>1</sup>Institute of Electron Microscopy and Nanoanalysis, Graz University of Technology, Graz, Austria — <sup>2</sup>Graz Centre for Electron Microscopy, Graz, Austria — <sup>3</sup>GETec Microscopy Inc. & SCL Sensor.Tech. Fabrication Inc., Vienna, Austria — <sup>4</sup>AMGT, Botevgrad, Bulgaria

Atomic Force Microscopy (AFM) has evolved into an essential part in research and development due to its quantitative 3D surface characterization capabilities on the spatial nanoscale together with the possibility to access laterally resolved magnetic, chemical, mechanical, optical, electric or thermal properties of the sample surface. In this contribution, we demonstrate the direct-write fabrication of 3D nano-probes via focused electron beam induced deposition (FEBID) together with its respective AFM application. For conductive-AFM, Pt-C nano-pillars are initially fabricated by FEBID and then chemically transferred into pure Pt via gas assisted post-growth purification. For thermal nano-probes we use platinum temperature dependent, electric properties as transducing element together with FEBIDs 3D fabrication capabilities to realize free-standing nano-bridges. We demonstrate the reversible, quantitative temperature response together with fast response times of less than 30 ms/K. Finally, we show scanning thermal microscopy measurements, revealing thermal surface gradients on the nanoscale.

KFM 16.7 Wed 12:15 H 2032

**High-Fidelity 3D-Nanoprinting via Focused Electron Beams:**

**Growth Fundamentals** — ●ROBERT WINKLER<sup>1,2</sup>, BRETT LEWIS<sup>3,4</sup>, JASON FOWLKES<sup>3,4</sup>, PHILIP RACK<sup>3,4</sup>, and HARALD PLANK<sup>1,2</sup> — <sup>1</sup>Institute for Electron Microscopy and Nanoanalysis Graz University of Technology, 8010 Graz, Austria — <sup>2</sup>Graz Centre for Electron Microscopy, 8010 Graz, Austria — <sup>3</sup>Center for Nanophase Materials Sciences, Oak Ridge National Laboratory, Oak Ridge, Tennessee 37831, USA — <sup>4</sup>Department of Materials Science and Engineering, University of Tennessee, Knoxville, Tennessee 37996, USA

3D-printing of functional structures has emerged to an important technology in research and development, although it becomes very challenging when aiming on nano-sized geometries. Among the very few direct-write techniques on that scale, Focused Electron Beam Induced Deposition (FEBID) has been demonstrated to be a promising candidate as it allows the fabrication of functional, freestanding 3D nano-architectures on almost any substrate, enabling novel applications. Predictable, reliable and reproducible fabrication, however, often suffered due to the numerous process parameter and their mutual relationships. In this contribution, we comprehensively discuss the complex interplay between most process parameters and successfully trace back their implications to the precursor working regime. Beside the fundamental aspect of our findings, we separate dominant parameters from those with minor implications. By that, we are able to explain unwanted deviations during 3D growth and derive certain rules for precise, predictable and reproducible 3D-nanofabrication via FEBID.

KFM 16.8 Wed 12:30 H 2032

**Mechanical Properties of 3D Nano-Architectures Fabricated via Focused Electron Beam Induced Deposition** — JOHANNES FROECH<sup>1,2</sup>, JUERGEN SATTELKOW<sup>1,2</sup>, ROBERT WINKLER<sup>1,2</sup>, CHRISTIAN SCHWALB<sup>3</sup>, ERNEST FANTNER<sup>3</sup>, and ●HARALD PLANK<sup>1,2</sup> — <sup>1</sup>Institute for Electron Microscopy and Nanoanalysis Graz University of Technology, 8010 Graz, Austria — <sup>2</sup>Graz Centre for Electron Microscopy, 8010 Graz, Austria — <sup>3</sup>GETec Microscopy Inc. & SCL Sensor.Tech. Fabrication Inc., 1220 Vienna, Austria

With the recent introduction of controlled 3D nano-printing via focused electron beam induced deposition, an entirely new range of applications such as nano-optics, -mechanics, or -electronics comes within reach whose fabrication is extremely challenging or even impossible with alternative techniques. In this contribution, we focus on mechanical properties of freestanding, Pt based 3D nano-architectures for atomic force microscopy (AFM) based application as high-resolution thermal nano-probes. A combined approach of finite element simulation, AFM based force spectroscopy and real-time imaging via scanning electron microscopy is used to identify and compensate highly unwanted peculiarities. In more detail, we discuss an unexpectedly strong influence of non-straight side branches as well as the consequences of fabrication mismatches on the lowest nanoscale, leading to non-linear mechanical behaviour and morphological twisting, respectively. The combined outcome of our findings demonstrate the high demands on nanoscale accuracy during 3D nano-printing to exploit the full potential in terms of predictable mechanical properties.

KFM 16.9 Wed 12:45 H 2032

**Correlative in-situ characterization of 3D nanostructures by combining SEM and AFM** — ●CHRISTIAN SCHWALB<sup>1</sup>, MARCEL WINHOLD<sup>1</sup>, PINAR FRANK<sup>1</sup>, STEFAN HUMMEL<sup>1</sup>, ROLAND SACHSER<sup>2</sup>, MICHAEL HUTH<sup>2</sup>, JUERGEN SATTELKOW<sup>3</sup>, and HARALD PLANK<sup>3</sup> — <sup>1</sup>GETec Microscopy GmbH, Vienna, Austria — <sup>2</sup>Physikalisches Institut, Goethe University Frankfurt, Germany — <sup>3</sup>FELMI, Graz, Austria

Focused electron-beam-induced processing represents one of the most flexible approaches for functional nanostructure fabrication. During and after the growth process, e.g., electrical in-situ measurements as well as energy-dispersive X-ray spectroscopy are commonly employed to characterize electrical and chemical properties of fabricated structures. However, one major drawback is the lack of further in-situ analysis tools which grants access to real 3D topographic information, laterally resolved conductance maps, local magnetic or mechanical properties. We present a novel AFM that allows correlative in-situ analysis by combining the full SEM and AFM capability. The AFM measurement takes place in the field of view of the electron beam and thus allows for non-destructive and non-contaminating analyses of FEBID structures directly after fabrication. We make use of novel self-sensing cantilevers that are equipped with different specialized tips fabricated by 3D nano-printing of sharp purified metallic or magnetic tips. We present correlative in-situ conductive, magnetic and mechanical analysis of 3D nanostructures using these novel cantilever tips and discuss future applications.