DS 23: Direct-Write Nanofabrication and Applications III (Electron Beam Induced Processing) (joint session DS/TT)

Part III: Material Properties & Applications

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

(Synopsis provided with part I of this session)

Time: Thursday 15:00–17:45 Location: H32

Invited Talk DS 23.1 Thu 15:00 H32

Artificial nano-granular heterostructures: fundamentals and applications — •Oleg Udalov[1,2] and Igor Beloborodov[1] — [1]California State University, Northridge, USA — [2]Institute for Physics of Microstructures RAS, Nizhny Novgorod, Russia

Granular composites are a large class of artificial materials in which nanograins are embedded into a hosting matrix. Combining different types of grains and matrix one can obtain composites with desirable functionalities. Fabrication of granular materials is scalable and is much cheaper than, for example, the thin film preparation techniques. Such an advantage makes these materials very attractive for various applications, including medical, photonic, radiofrequency, mechanical, etc. Properties of granular materials are defined by the interplay of disorder, dimensionality, surface and size effects, superconductivity, magnetic interactions, strain, the Coulomb blockade, etc. Therefore, studying of these materials is a complicated challenge promising unexpected novel properties. Nanograins ensembles are often used as a prototype of systems with competitive interactions and strong many-body effects. This makes granular materials of a great fundamental interest. In this talk we will focus on recent developments in solid granular materials. First, we discuss different types of granular media and their applications. In particular, we will consider granular metals, ferromagnets, superconductors and semiconductors. Next, we will discuss granular multiferroics where two order parameters are combined: 1) magnetization and 2) polarization. We will consider theoretical and experimental works on magneto-electric coupling in these materials.

DS 23.2 Thu 15:30 H32

Ac conductivity of nano-granular metals prepared via FEBID — •Marc Hanefeld[1], Michael Huth[1], Joshua Gies[2], and Martin Kind[2] — [1]Physikalisches Institut, Goethe Universität, Max-von-Laue-Str. 1 — [2]Institut für Anorg. und Analyt. Chemie, Goethe Universität, Max-von-Laue-Str. 7, 60438 Frankfurt a. M., Germany

Focused Electron Beam Induced Deposition (FEBID) is a versatile technique to create nano-granular metals with tunable electronic transport properties [1]. In granular metals metallic nanoparticles are surrounded by a dielectric matrix which leads to a transport mechanism based on thermally assisted tunnelling. This opens up promising possibilities for sensing applications [1] and has triggered ongoing research concerning their response to a time-dependant ac stimulus [2].

Current research focuses mainly on two different material properties, namely an apparent universal power law and a temperature-independent scaling behaviour in the real part of the complex ac conductivity, both present in many disordered solids and recently reported in granular metals of palladium in zirconia [2].

We present recent results on the ac conductance response of nano-granular Pt(C)-FEBID deposits and show the capabilities of FEBID to create an ideal model environment for an in depth analysis of the ac conduction characteristics of granular metals. Possible applications of these results in dielectric sensors combining nano-granular Pt(C) with metal-organic frameworks will be discussed.

[1] Huth, et al., Microelect. Eng. 2017. doi:10.1016/j.mee.2017.10.012.
[2] Bakkali, et al., Sci. Rep. 2016;6:29676. doi:10.1038/srep29676.

DS 23.3 Thu 15:45 H32

Post-growth electron beam irradiation of co-deposited PtC-FeCo FEBID nanostructures — •Roland Sachser and Michael Huth — Physikalisches Institut, Goethe-University, Frankfurt am Main, Germany

FEBID using the precursor $(CH_3)_3CH_3C_5H_4Pt$ leads to low conductive, nanogranular samples. The resistivity is determined by the coupling of Pt nanocrystals by the carbonaceous matrix. This matrix, and thus the conductivity, can be strongly influenced by post-growth electron beam irradiation. On the other hand, deposits prepared employing the precursor $HFeCo_3(CO)_{12}$ feature high metal contents and metallic resistivity behavior. These samples are ferromagnetic but the tunability of their transport properties by post-growth treatments is limited. By co-deposition using both precursors under well-controlled mixing ratios it is possible to obtain a highly tunable granular ferromagnet. Granular ferromagnetic systems are known to show an enhanced anomalous Hall effect, which can also be observed in our deposits that are treated by post-growth electron beam irradiation under in situ conductance monitoring. The conductance can be tuned over a wide range, which is also prominently reflected in the magneto-transport properties that can be directly monitored in situ in the low field regime at room temperature. Complementary low temperature magneto-transport measurements reveal the optimal post-growth irradiation dose to obtain the largest anomalous Hall effect.

DS 23.4 Thu 16:00 H32

Micro-Hall Magnetometry of FEBID-fabricated 3D magnetic nano-architectures — •Jens Müller[1], Mohanad Al Mamoori[1], Lukas Keller[1], Michael Huth[1], and Christian Schröder[2] — [1]Institute of Physics, Goethe University Frankfurt — [2]Institute for Applied Materials Research, Bielefeld University of Applied Sciences

Whereas most of the previous studies of nanoscale magnetic structures – owing to the fabrication by means of lithographic processes – have focused on one- or two-dimensional (1D/2D) nanomagnets, thanks to advances in fabrication and sensing techniques it is now possible to investigate free-standing magnetic 3D structures individually, in small arrays, or in complex connected lattice architectures. Here we report on the combination of focused electron beam induced deposition (FEBID) and micro-Hall magnetometry, as fabrication and high-resolution sensing tools, respectively. Free-form structures with minimal feature size of a few tens of nm of the metallic ferromagnets Fe, Co and Fe-Co are deposited directly on a semiconductor Hall sensor which serves as the substrate and allows to detect the magnetic stray field as a function of the external magnetic field and temperature. In the talk, we will discuss the measured and simulated magnetization reversal of small arrays of Fe-Co nanotrees and -cubes [1,2]. Furthermore, we will give an outlook on future possibilities of fabricating magnetic structures with geometrical frustration (towards 3D artificial spin ice) and studying dynamical processes by means of magnetic flux noise spectroscopy.

[1] L. Keller et al., *Sci. Rep.* **8**, 6160 (2018).
[2] M. Al Mamoori et al., *Materials* **11**, 289 (2018).

15 min. break

Invited Talk DS 23.5 Thu 16:30 H32

3D nanomagnetism and superconductivity: Current status and potential for future work — •Oleksandr Dobrovolskiy and Michael Huth — Goethe University, Frankfurt am Main

Extending 2D structures into the third dimension has become a general trend in various areas, including photonics, plasmonics and magnetics. This approach provides a means to modify conventional and to launch novel functionalities by tailoring vector potentials inducing anisotropic and chiral effects. Recently, there has been significant progress in the fabrication of free-standing ferromagnetic and superconducting nanostructures by focused particle direct-write techniques which is in part reviewed in [1]. In this respect, 3D shell structures such as framed tubes, spheres, Swiss rolls and helices are especially interesting as they offer unprecedented prospects for nanomagnetism and superconductivity because of topology and geometry-controlled effects. Namely, in magnetism, curvilinear geometry brings about two exchange driven interactions - effective anisotropy and antisymmetric vector exchange, i.e. an effective Dzyaloshinskii-Moriya interaction. In addition, another magneto-chiral contribution emerges due to the dipole-dipole interaction. In the case of superconducting nanostructures, the combination of low-dimensionality with a curvilinear geometry allows in principle for the observation of topology-driven effects, such as unconventional phase slips, reversible and irreversible switching, fractional flux-flow instabilities, and the Berezinskii-Kosterlitz-Thouless transition. [1] M. Huth, F. Porrati, O. V. Dobrovolskiy, FEBID meets materials science, Microelectron. Engineering, 185-186, 9-28 (2018).

DS 23.6 Thu 17:00 H32

3D Nano-Printing via FEBID: Complex 3D Nano-Structures — •Lukas Keller and Michael Huth — Institute of Physics, Goethe University, Max-von-Laue-Str. 1, 60438 Frankfurt am Main, Germany

[3]: https://youtu.be/v8s24WvGj9E
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 [1] and plasmonics. The main issue of generating a desired 3D nano-structure is to navigate the electron beam in the x-y-plane.
Here we present our pattern file generator program [2]. It considers several issues caused by limited precursor replenishment dynamics, which is important for the successful fabrication of 3D nano-structures. The target geometry can be defined by hand or using powerful 3D software tools like "blender" [3]. Several examples of 3D nano- and micro-structures using different precursors are presented.

[1]: Scientific Reports 8, 6160 (2018)
[2]: Beilstein J. Nanotechnol., 9, 2581-2598 (2018)
[3]: https://youtu.be/v8s24WvGj9E

DS 23.7 Thu 17:15 H32

Crystalline NbC superconducting nanowires by direct-writing — •Fabrizio Porrati[1], Sven Barth[2], Roland Sachser[1], Oleksandr Dobrovolskiy[1], Anja Seybert[3], Achilleas Frangakis[3], and Michael Huth[1] — [1]Goethe-University, Institut of Physics, Frankfurt a. M. — [2]TU Wien, Institute of Materials Chemistry — [3]Goethe-University, Buchmann Institute

We present a comparative study of planar nanowires and 3D free-standing nanowires grown by focused electron- / ion (Ga+)-beam induced deposition (FEBID/FIBID) using the novel precursor $Nb(NMe_2)_3$(N-*t*-Bu). FEBID planar nanowires contain 67at%C, 22at%N and 11at%Nb; FIBID planar nanowires 42.9at%C, 12.9at%N, 15.5at%Ga and 28.7at%Nb. TEM analysis shows that FEBID samples are amorphous, while FIBID samples are made of fcc NbC polycrystals, with grains of 15-20nm diameter and lattice constant 4.47Å. The RT electrical resistivity is $\approx 10^4$ $\mu\Omega$cm and 550 $\mu\Omega$cm for FEBID and FIBID samples. Conductivity vs. temperature measurements show that the FEBID nanowires are insulating, following a variable-range-hopping behavior. FIBID nanowires, with RRR=0.87, become superconducting at T_C=5.0 K. T_C can be slightly tuned either by an electron irradiation treatment (T_C=5.4, for 20nC/*m^2) or by changing the width of the nanowires (T_C=4.3-6.4, for w=50-1000 nm). 3D free-standing nanowires were grown by FIBID. TEM experiments confirm a fcc NbC microstructure. The electrical measurements of these nanowires, with RRR=1.02, show a superconducting transition with T_C 11K, a value close to the value of bulk NbC.

DS 23.8 Thu 17:30 H32

Effective protection of few-layer black phosphorus from reactive species generated by in-situ focused electron beam irradiation. — •Lobo Charlene and Elbadawi Christopher — School of Mathematical and Physical Sciences, University of Technology Sydney, Ultimo NSW 2007

Recent studies have shown that exposure of few-layer black phosphorus (FLBP) to reactive oxygen species (ROS) via photoactivated oxidation induces an immediate and permanent reduction in the electron and hole mobility of FLBP. Electron-beam irradiation in H2O vapour is known to generate ROS (such as *O2-, *OH and OH-) that are responsible for the degradation of FLBP in ambient environment, but FLBP stability has not been assessed during prolonged exposure to other gaseous environments.

Here, we study the stability of FLBP in H2O, O2, NF3 and NH3 environments using environmental scanning electron microscopy (ESEM) and in situ electrical conductance measurements. The electron beam is used both for ESEM imaging, and also to generate reactive species such as *O, *OH, *F and *H that can drive spatially-localized chemical reactions at the sample surface. Using this approach, we demonstrate two promising methods of protecting FLBP and other moisture-sensitive two-dimensional materials from degradation by ROS. Encapsulation of FLBP with ionic liquids dramatically slows the rate of degradation in ROS environments. FLBP degradation can also be prevented by maintaining the temperature in the range ~125-300 *C during ROS exposure, without requiring any protective coating.