CELINA

EUROPEAN COOPERATION
IN SCIENCE AND TECHNOLOGY

COST Action CM1301 - CELINA

Workshop on

Perspectives of C-containing FEBID precursors

March 1, 2016, Bremen, Germany

Book of Abstracts

Local Organizer and Action Chair: **Petra Swiderek**
(University of Bremen, Germany)

Universität Bremen

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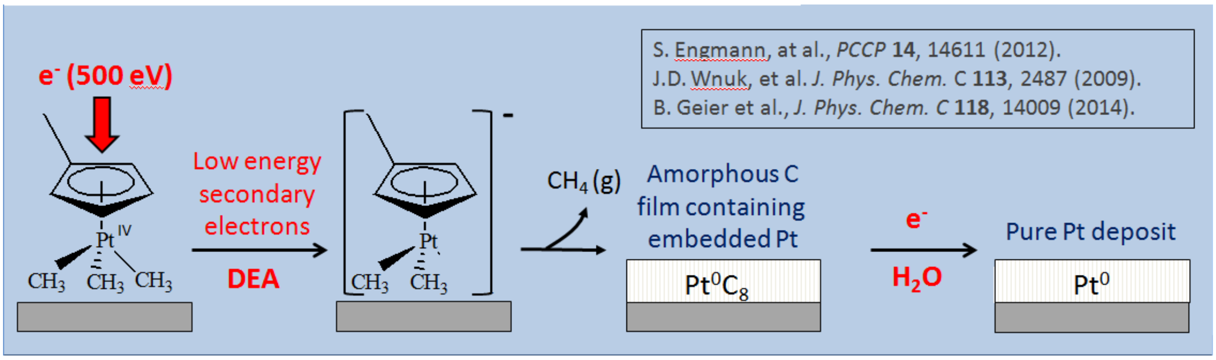
Aim of the meeting

Carbon-containing precursors have, for a long time, been considered as unsuitable for FEBID. Many such compounds have been developed for CVD with favourable properties regarding stability and volatility. However, when applied in FEBID, these precursors typically yield deposits containing significant amounts of carbon and thus poor properties. The recent finding that electron irradiation of the deposit in the presence of added process gases such as water significantly reduces the carbon content is thus highly promising. If such purification processes can be applied to a wider range of precursors, this would enable a targeted approach for the development of better FEBID precursors by using or modifying readily available CVD precursors. The aim of this meeting is to discuss this approach and develop it into a joint research strategy.



S. Engmann, at al., *PCCP* **14**, 14611 (2012).
J.D. Wnuk, et al. *J. Phys. Chem.* C **113**, 2487 (2009).
B. Geier et al., *J. Phys. Chem. C* **118**, 14009 (2014).

Programme		
09:00-09:15	Petra Swiderek	Welcome and scope of the workshop
09:15-10:00	Harald Plank	Towards Pure Metal Deposits via E-beam Assisted Purification using H_2O Vapor at Room Temperature
10:00-10:15	Petra Swiderek	Surface science studies on purification processes
Coffee break / Discussion / Posters		
10:45-11:15	Hassan Abdoul-Carime	Iron methylene ion production from hot ferrocene selected by 0 eV electrons
11:15-11:45	Juraj Fedor	Cluster-beam experiments and the insight they bring into FEBID
11:45-12:15	Petr Carsky	Theory in Service of FEBID
Lunch break / Opportunity for lab visit /Posters		
14:00-14:30	Sangeetha Hari Kees Hagen	Electron beam induced etching of carbon with water
14:30-14:50	Volkmar Zielasek	XPS study of thermal and electron-induced decomposition of Ni and Co acetylacetonate thin films for metal deposition
14:50-15:20	Maciej Gutowski	Electron-Driven Proton Transfer in Hydrogen-Bonded Systems
Coffee break		
15:30-16:00	Concluding discussion with coffee	
Lab visit		
Further discussion during Bremen city walk and dinner		

Posters	
Jaroslav Kočišek	Electron induced chemistry in complex systems studied on CLUB
Markus Rohdenburg	Novel insights into the water-assisted purification of FEBID deposits produced from $MeCpPtMe_3$
Kai Rückriem	Electron-Induced Formation of Cu Nanoparticles from Sequentially Deposited Copper(II)oxalate

Towards Pure Metal Deposits via E-beam Assisted Purification using H2O Vapor at Room Temperature Abstract Title

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In recent years, focused electron beam induced deposition (FEBID) has made significant progress with respect to potential applications ranging from passive devices, such as plasmonic gratings, towards active concepts like mechanical, magnetic or chemical sensing. Beside the undoubted advantages of FEBID as direct-write tool with spatial nanometer resolution the limitations due to low material purity still remain. In the past, several in-situ and ex-situ purification approaches have been presented with the intention to suppress carbon incorporation during or remove unwanted impurities after the deposition process, respectively. Although some concepts were excellent in terms of chemistry, deposit morphologies often suffered from cracks, small pores and / or shrinking effects which can be a problem for structures on the lower nanoscale. In 2014, Geier et-al successfully introduced an approach using H2O vapor at room temperatures in combination with a scanning e-beam to entirely remove the carbon from Pt-C FEBID structures [1]. The main advantage of this approach is the crack and pore free, highly compact morphology of final structures with a minimal lateral shrink of less than 5 % and carbon contents below the detection limits in transmission electron microscopy based electron energy loss spectroscopy. Further beneficial aspects are fast purification rates below 5 min.µm-2.nA-1, the widely uncritical process window during purification concerning beam current, pixel dwell time and point pitch but also the fact that we water at room temperature is used which is widely harmless for surrounding materials.

In this presentation the proposed approach will be briefly re-visited with focus on the ease-of-use character in combination with achievable results. In the second part, we present according simulations which give a first indication on the underlying processes and also reveal that the H2O partial pressure can be used to switch the purification direction within the deposit from top-down to bottom-up [2]. In the third part, we demonstrate the applicability of this concept to Au based FEBID precursor (Me2Au(acac)) and focus on downscaling capabilities. As will be shown, the approach allows the fabrication of pure gold double-ring structures with ring diameters and FWHM line widths of 75 nm and 20 nm, respectively. In the final part of this presentation we focus on FEBIDs unique 3D structuring capabilities. In detail we apply the H2O approach on free-standing 3D Au-C architectures and demonstrate the full carbon removal while the overall shape is widely maintained. This is of particular importance in the field of 3D plasmonics where the presented approach is one of the very few, direct-write technologies which enables real 3D fabrication of pure metals with spatial nanometer resolution. By that, this presentation spans the bow from fundamental considerations over practical aspects towards novel capabilities for FEBID based nano-structures.

[1] B. Geier, C. Gspan, R. Winkler, R. Schmied, J. D. Fowlkes, H. Fitzek, S. Rauch, J. Rattenberger, P. D. Rack, H. Plank, *Journal of Physical Chemistry C* **118**, (2014) 14009.
[2] J.D. Fowlkes, B. Geier, B. B. Lewis, P. D. Rack, M. G. Stanford, R. Winkler, H. Plank, *PCCP* **17**, (2015) 18294.

Surface science studies on purification processes

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Carbon-containing precursors have, for a long time, been considered as unsuitable for FEBID. Many such compounds have been developed for CVD with favourable properties regarding stability and volatility. However, when applied in FEBID, these precursors typically yield deposits containing significant amounts of carbon and thus poor properties. The recent finding that electron irradiation of deposits in the presence of added process gases such as water can significantly reduce the carbon content is thus highly promising [1]. If such purification processes can be applied to a wider range of precursors, this would enable a targeted approach for the development of better FEBID precursors by using or modifying readily available CVD precursors.

The chemical reactions that are fundamental to such deposit purification are typically not monitored during FEBID processes. Therefore, complementary experiments employing a surface science approach are important to gain a deeper understanding of the reactions [2]. The present contribution summarizes briefly the experimental capabilities of our group and, in particular, the combination of electron-stimulated desorption (ESD) experiments and thermal desorption spectrometry (TDS) [3]. This methodology is presently applied to study the electron-induced and water-assisted reactions in thin condensed layers of MeCpPtMe3 and similar precursors [4].

[1] B. Geier, C. Gspan, R. Winkler, R. Schmied, J.D. Fowlkes, H. Fitzek, S. Rauch, J. Rattenberger, P.D. Rack, H. Plank, *J. Phys. Chem. C* **118**, (2014) 14009.
[2] J.D. Wnuk, S.G. Rosenberg, J.M. Gorham, W.F. van Dorp, C.W. Hagen, D.H. Fairbrother, *Surf. Sci.* **605**, (2011) 257.
[3] J. Warneke, P. Swiderek, *J. Phys. Chem. C* **119**, (2015) 8725.
[4] Z. Wang, M. Rohdenburg, J. Warneke, P. Swiderek, to be published.

Iron methylene ion production from hot ferrocene selected by 0eV electrons

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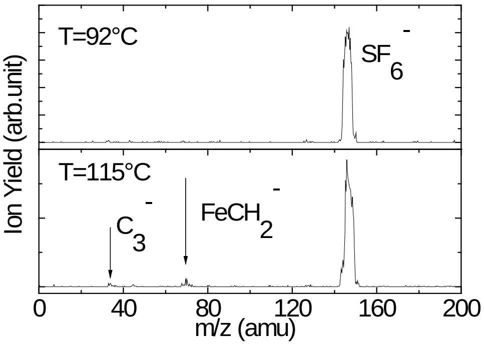
Metal carbene is implicated in a large variety of catalytic transformation. For instance the reaction of iron methylene complex with hydrogen molecules produces iron and the methane counterpart [1,2] that can be a process for the elaboration of specific nanosurface. The iron carbene molecules are in general synthesized from photolysis and/or reactions induced by the co-condensation of iron vapor with n-alkane at low temperatures (e.g., 170K) [1].

In this work, we show that iron methylene ions can be produced from hot ferrocene molecules. The ferrocene molecules are firstly thermally decomposed onto a molybdenum surface. The sub-products after interacting with ~0eV electrons lead to the formation of $FeCH_2^-$ ions. These ions can further be used to implant surfaces at the nanoscale for potential applications.



Mass spectra (with 0eV electrons)

[1] C. Almansa, M.L. Garcia, C. Jaime, A. Moyano, M.A. Pericas, F. Serratosa, *in Strain and its implication in organic chemistry: Organic stress and reactivity*, Edits A. de Meijere and S. Blechert, Kluwer Academic Press (1989).
[2] Y.G. Abashkin, S.K. Burt, N. Rosso, *J.Phys.Chem. A* **101**, (1997) 8085.

Cluster-beam experiments and the insight they bring into FEBID

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We report the first experiments on electron collisions with clusters containing FEBID precursors. We have two ways of preparing such clusters: either co-expanding the precursor molecules with buffer gas and thus creating aggregates of pure precursor, or picking up multiple precursors on a surface of large argon clusters (hundreds of Ar atoms) and thus creating precursor aggregates on a 'nanosupport'. I will present a case study of the $Fe(CO)_5$ precursor, where our experiments showed that the self-scavenging of electrons plays a central role in the synthesis of complex iron-containing anions. Furthermore, I will discuss experimental problems that we have encountered with MePtCpMe3 precursor.

Theory in Service of CELINA

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In our contribution we would like to show the progress in the development of our program for calculations of electron scattering by polyatomic molecules. We show the results for adamantane, C10H16, the biggest molecule we have treated so far. However, the primary objective of our presentation is to get some feedback from the audience to learn how our research should oriented to meet the needs of CELINA. In particular we would appreciate hearing comments on the following issues:

1. Are the results of gas-phase scattering calculations of any use to FEBID?
2. As we know, the presence of free "gas-phase" molecules of precursors deteriorate focusing the electron beam. We can calculate scattering of these primary electrons. Is that of any use?
3. Instead of geometry averaging for gas-phase experiments, we can also do calculations for a fixed geometry of the target and a pair of selected directions of the beams of incoming and outgoing electrons. Such a model ignores (so far) interaction target-surface. Is it still of any use?
4. Calculations of dissociative attachment is a hard job. But, we can calculate vibrational excitation of polyatomic molecules by electron impact, and we have arguments for claiming that the found resonances for vibrational excitations is an indication for a bond fission. Any comment?
6. We are now working on AuClCO and the next precursor should be Pt(PF3)4. Suggestions for other (preferably small) would be appreciated.

Electron beam induced etching of carbon with water

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Electron Beam Induced Deposition (EBID) has great potential for lithography. However, while single nanometer lines and spaces have been demonstrated, the other advantages of EBID are yet to be properly exploited. The carbon content in the deposits remains a concern as does the broadening of lines. Since EBID lines typically have a Gaussian profile, the tails contribute to deposition in between the lines, building up in thickness as the lines become denser, resulting in interconnects and cross talk. Further, the Gaussian profile hinders the accurate determination of line width and height which are critical for lithography. We aim to control the shape of EBID lines to realise the potential of the technique for high resolution lithography. Since the composition of small EBID structures is often carbonaceous, for this study we pattern lines using a carbon precursor. The blue plot in Fig. 1 shows the integrated AFM profile of the as-deposited lines where the presence of material in between them is clearly visible. To remove the interconnects we carried out Electron Beam Induced Etching (EBIE) in between the lines using water as a precursor, a technique previously shown to slim carbonaceous nanowires. The red plot in Fig. 1 shows that the lines have successfully been separated and the line profile itself somewhat modified. As a next step, we applied this technique to modify the 3D shape of the carbon EBID lines themselves to achieve vertical sidewalls that are desirable for lithography. Fig. 2 shows the as-deposited EBID line profile and the profile after etching of the sidewalls by EBIE. The advantages of combined EBID and EBIE for shape control are therefore clear, together with the need for an understanding of the mechanism and governing parameters.

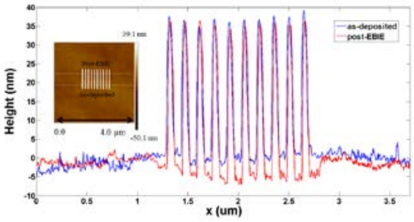

Fig. 1 Integrated AFM profile of an array of carbon EBID lines: as deposited (blue) and post-etching using water (red), demonstrating the removal of connecting

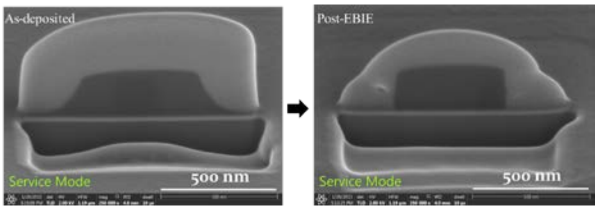

Fig. 2 3-dimensional profile of as-deposited EBID line and modified profile after sidewall etching, as seen in FIB cross sections

XPS study of thermal and electron-induced decomposition of Ni and Co acetylacetonate thin films for metal deposition

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Optimizing thin metal film deposition techniques from metal-organic precursors such as ALD, CVD, or EBID with the help of surface science analysis tools in ultrahigh vacuum requires a contamination-free precursor delivery technique, especially in the case of the less volatile precursors. For this purpose the preparation of layers of undecomposed Ni(acac)$_2$ and Co(acac)$_2$ was tried via pulsed spray evaporation of a liquid solution of the precursors in ethanol into a flow of nitrogen in a CVD reactor. Solvent-free layers of intact precursor molecules were obtained when the substrate was held at a temperature of 115 °C. A qualitative comparison of thermally initiated and electron-induced precursor decomposition and metal center reduction was carried out. All deposited films could be analyzed with respect to chemical composition *quasi in situ* by XPS.[1] Thermally initiated decomposition yielded higher metal-to-metal oxide ratios in the deposit than the electron-induced process for which ratios of 60:40 and 20:80 were achieved for Ni and Co, respectively. Compared to continuous EBID processes, all deposits showed low levels of carbon impurities of ~10 at.%. Therefore, post-deposition irradiation of metal acetylacetonate layers by a focused electron beam and subsequent removal of intact precursor by dissolution in ethanol or by heating is proposed as electron beam lithography technique on the laboratory scale for the production of metal nanostructures.

[1] Th. Weiss, M. Nowak, U. Mundloch, V. Zielasek, K. Kohse-Höinghaus and M. Bäumer, *Rev. Sci. Instrum*. **85**, (2014) 104104.

Electron-Driven Proton Transfer in Hydrogen-Bonded Systems

Maciej Gutowski[1,*], Zibo G. Keolopile[1,2], Angela Buonaugurio[3], Evan Collins[3], Xinxing Zhang[3], Jeremy Erb[3], Thomas Lectka[3], Kit H. Bowen[3] and Michael Allan[4]

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Intermolecular proton transfer in hydrogen bonded complexes has been studied computationally and experimentally using anion photoelectron spectroscopy (PES) technique. I will present results for model hydrogen bonded systems and for complexes involving nucleic acid bases. Anion photoelectron spectroscopy and electron energy-loss spectroscopy (EELS) probe different regions of the anionic potential energy surface. These complementary techniques provided information about anionic states of acetoacetic acid (AA). Electronic structure calculations facilitated the identfication of the most stable tautomers and conformers for both neutral and anionic AA, and determined their relative stabilities and excess electron binding energies. The valence anion displays a distinct minimum which results from proton transfer from the carboxylic to the keto group, thus we name it an ol structure. The minimum is characterized by a short intramolecular hydrogen bond, a significant electron vertical detachment energy of 2.38 eV, but a modest adiabatic electron affinity of 0.33 eV. The valence anion was identified in the anion PES experiments and the measured electron vertical detachment energy of 2.30 eV is in good agreement with our computational prediction. The results of EELS experiments do not provide evidence for an ultrarapid proton transfer in the lowest π* resonance of AA, which would be capable of competing with electron autodetachment. This observation is consistent with our computational results indicating that major gas phase conformers and tautomers of neutral AA do not support the intramolecular hydrogen bond that would facilitate ultrarapid proton transfer and formation of the ol valence anion. This is confirmed by our vibrational EELS spectrum.

[1] Z.G. Keolopile, M. Gutowski, A. Buonaugurio, E. Collins, X. Zhang, J. Erb, T. Lectka, K.H. Bowen, M. Allan, *J. Am. Chem. Soc.* **137**, (2015) 14329.

Electron induced chemistry in complex systems studied on CLUB

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CLUB (Fig. 1) is a complex cluster-beam apparatus to study various environmental effects on reaction dynamics. The presentation overviews our recent experiments on the electron-induced chemistry. We will focus on the methods, techniques to prepare nanoparticles and typical changes in electron-induced chemistry caused by the environment. Examples will include experiments on clusters of simple molecules – water and acetylene. Acetylene acts as a model specie for cluster-ion polymerization process. In such process, nanostructures are produced in isolated form in vacuum, thus without the influence of substrate. Water is now intensively studied by FEBID community, not only as the omnipresent impurity but also as an important additive to the FEBID process which increases the purity of metal deposits.

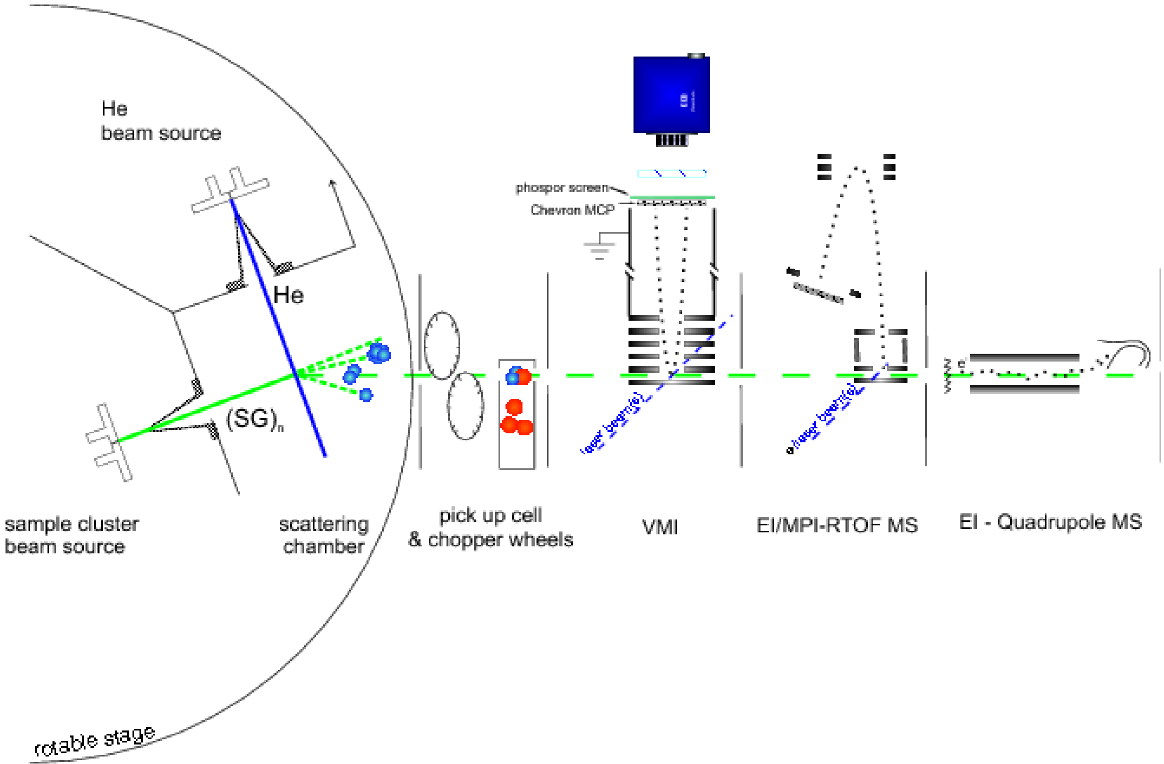

Fig. 1: Scheme of the CLUB experimental setup.

Novel insights into the water-assisted purificationof FEBID deposits produced from $MeCpPtMe_3$

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The widely applied FEBID precursor trimethyl(methylcyclopentadienyl)-platinum(IV) ($MeCpPtMe_3$) is renowned as a ‘notoriously bad’ precursor typically yielding deposits with a Pt content of only about 20% [1,2]. Fundamental studies have identified the reason for the low Pt content of FEBID deposits produced from $MeCpPtMe_3$. It was found that DEA predominantly leads to the loss of only one CH_3 group from the precursor [3]. This exactly matches the typical deposit composition of Pt:C = 1:8 obtained under UHV conditions, i.e., without contributions of residual vapours to deposit formation [2]. The ligand material remaining after loss of the first CH_3 thus becomes embedded in the deposit, a result that underlines, at the same time, the importance of low-energy electron-induced chemistry in FEBID processes.

Surprisingly, it was shown very recently and in contrast to earlier attempts [4] that deposits produced from $MeCpPtMe_3$ can be efficiently purified by a post-deposition electron beam treatment in the presence of water vapour thus yielding densely packed carbon- and oxygen-free Pt material [5]. The authors suggested that the initial carbon content diffuses to the surface of the deposit and desorbs as small volatile products (CO, CO_2, CH_x). Herein, a study applying a combination of electron-stimulated desorption (ESD) experiments and thermal desorption spectrometry (TDS) of electron-induced and water-assisted reactions in thin condensed layers of $MeCpPtMe_3$ is presented. In addition, deposit composition was monitored by Auger electron spectroscopy (AES). The results give insight into the electron-induced reactions of $MeCpPtMe_3$ in the presence of water. The lack of precursor hydrolysis in the absence of an electron beam underlines that the purification relies on the synergy between water and electron irradiation.

[1] I. Utke, P. Hoffmann, J. Melngailis, *J.Vac.Sci.Technol. B* **26** (2008) 1197.
[2] J.D. Wnuk, J.M. Gorham, S. Rosenberg, W.F. van Dorp, T.E. Madey, C.W. Hagen, D.H. Fairbrother, *J.Phys.Chem. C* **113** (2009) 2487.
[3] S. Engmann, M. Stano, Š. Matejčík, O. Ingólfsson, *Phys. Chem. Chem. Phys.* **14** (2012) 14611.
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Electron-Induced Formation of Cu Nanoparticles from Sequentially Deposited Copper(II)oxalate

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Nanoscale materials display particular physical and chemical properties as compared to the bulk of same composition. When grown or deposited on a surface they can thus provide their support with a wide range of useful functions. Although not as prominent as their Au and Ag analogues, nanoparticles (NPs) of the more affordable metal Cu exhibit similarly useful properties [1-4]. A number of applications has consequently been proposed. This includes, among others, the application as antibacterial and antifungal agents, the use of supported Cu NPs as catalysts, as substrate for surface-enhanced Raman scattering (SERS), and potentially a range of further plasmonic applications.

We have recently established a layer-by-layer deposition procedure to produce well-defined surface layers of copper(II)oxalate [5]. On a carboxy-terminated self-assembled monolayers these layers were grown using a step-by-step approach by dipping the surfaces alternately in ethanolic solutions of copper(II)acetate and oxalic acid with intermediate thorough rinsing steps. The deposition was monitored by reflection absorption infrared spectroscopy (RAIRS), a quartz microbalance with dissipation measurement (QCM-D), scanning electron microscopy (SEM), and Helium ion microscopy (HIM). Here, we show that such layers can be efficiently transformed to pure Cu NPs by low-energy electron irradiation at room temperature. This process also offers the perspective of adding capping layers in-situ via an electron-induced deposition process from the gas phase thus addressing the potential problem of Cu oxidation.

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