COST
EUROPEAN COOPERATION IN SCIENCE & TECHNOLOGY

Report on the outcomes of a Short-Term Scientific Mission[1]

Action number: CA20129 - Multiscale Irradiation and Chemistry Driven Processes and Related Technologies (MultIChem)

Grantee name: Krzysztof Mackosz

Details of the STSM

Title: Focused Electron Beam Induced Deposition of copper nanostructures using $Cu(hfac)_2$ precursor

Start and end date: 31/08/2025 to 13/09/2025

Description of the work carried out during the STSM

Description of the activities carried out during the STSM. Any deviations from the initial working plan shall also be described in this section.

(max. 500 words)

This work was focused on a systematic investigation of Focused Electron Beam Induced Deposition (FEBID) of copper nanostructures employing the $Cu(hfac)_2{\cdot}xH_2O$ precursorAll experiments were performed with a commercial Orsay Physics OptiGIS gas injection system (GIS) integrated into a Tescan S9000 FIB-SEM microscope.

A dedicated preparation protocol was established to ensure precursor stability and reproducibility. One gram of $Cu(hfac)_2{\cdot}xH_2O$ (Sigma-Aldrich) was purified and degassed in an Edwards T-Station 75 vacuum workstation at $7{\times}10^{-5}$ Pa for one hour. The precursor was then loaded into the GIS reservoir in a 1:1 weight ratio with deionized water. During deposition, the precursor reservoir was maintained at 35 °C, ensuring a chamber pressure of approximately $1{\times}10^{-3}$ Pa. After three days of continuous operation, the reservoir temperature was increased to 40 °C to preserve a constant injection rate. Silicon substrates with a native oxide layer were kept at room temperature.

The experimental program addressed several groups of parameters critical to FEBID performance. Systematic series were carried out to explore the effect of acceleration voltage, dwell, scanning strategy, and beam current. Each condition was evaluated by SEM imaging to assess deposition rate, lateral resolution, geometric accuracy of the patterns, and morphological uniformity. Among the tested conditions, the scanning path with flyback, a dwell time of 1 μs, and a beam current of 5 nA were identified as the most favorable parameters

[1] This report is submitted by the grantee to the Action MC for approval and for claiming payment of the awarded grant. The Grant Awarding Coordinator coordinates the evaluation of this report on behalf of the Action MC and instructs the GH for payment of the Grant.

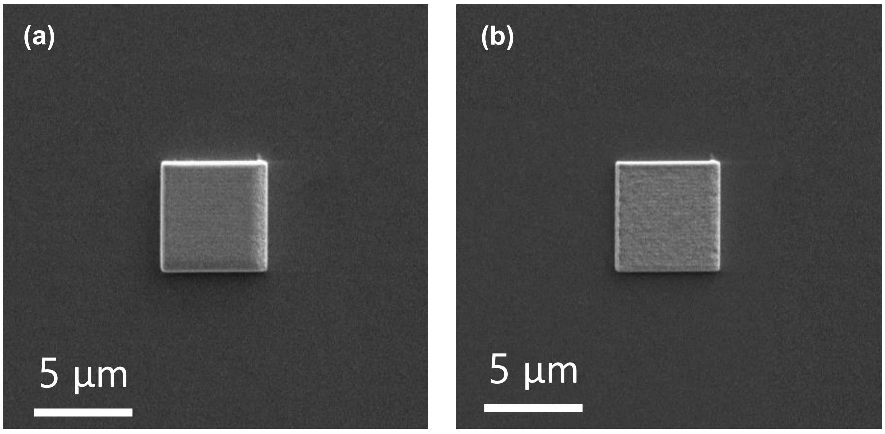


Figure 1. Representative SEM image of a Cu-FEBID deposit obtained at 5 kV, 5 nA, (a) with flyback, and (b) zig-zag scan strategy.

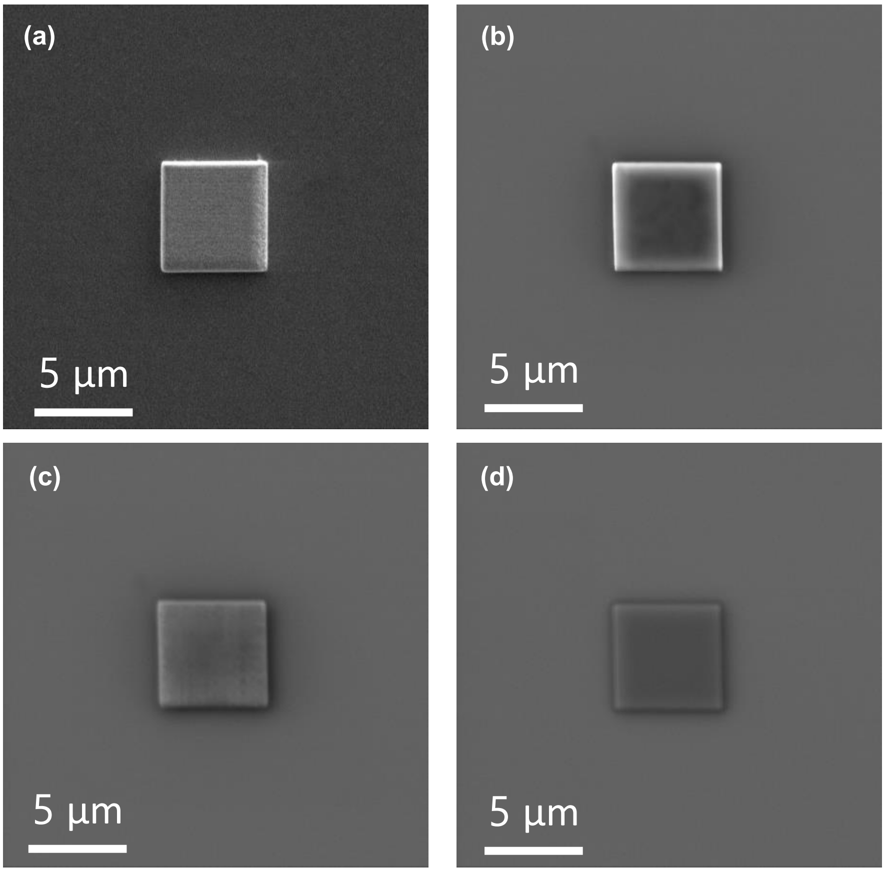


Figure 2. Influence of beam current variation (a) 5 nA, (b) 1 nA, (c) 100 pA, (d), 50 pA at 5 kV

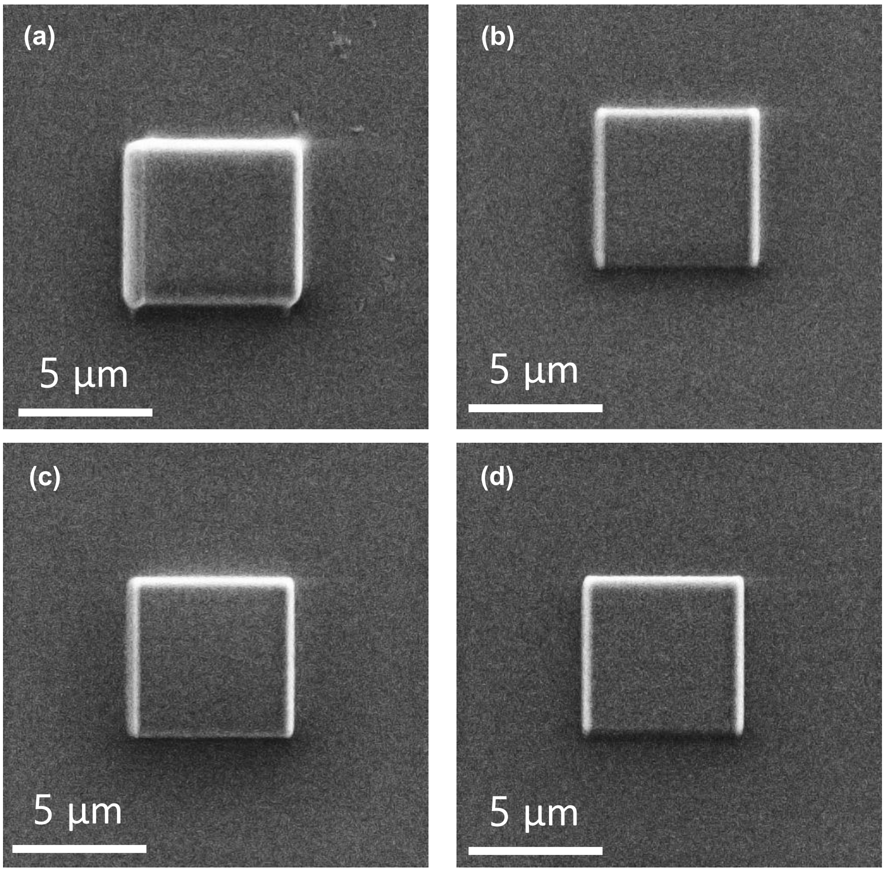
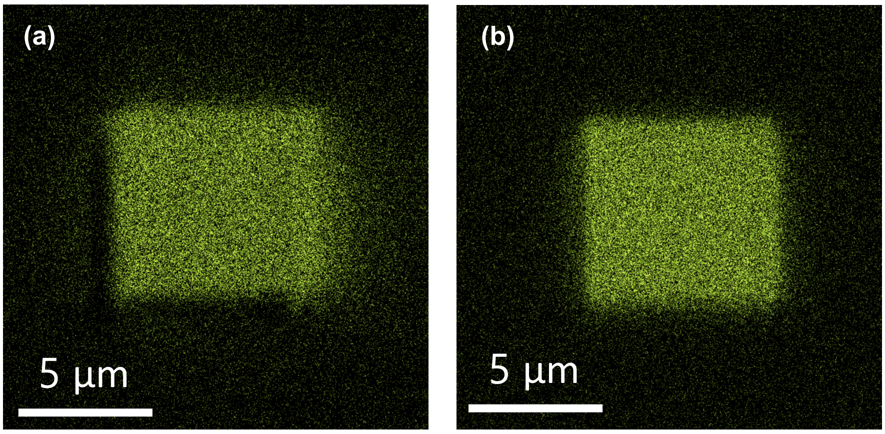


Figure 3. SEM comparison of deposits obtained at different acceleration voltages (a) 5 kV, (b) 10 kV, (c) 20 kV, (d) 30 kV.

Complementary chemical analysis was performed using an EDAX Octane Elite EDS/EDX detector integrated into a Tescan AMBER X platform. Elemental quantification of Cu, C, O, and F allowed direct assessment of precursor decomposition efficiency and identification of optimal deposition conditions. For this purpose, analyses were carried out under the most favorable set of parameters, a dwell time of 1 µs, a beam current of 5 nA, a scanning path with flyback. Based on compositional analyses, all tested conditions yielded a comparable copper content of approximately 16–17 at.%. The primary effect of increasing the acceleration voltage was a reduction in the fluorine fraction, while the relative concentrations of carbon and oxygen remained largely unchanged



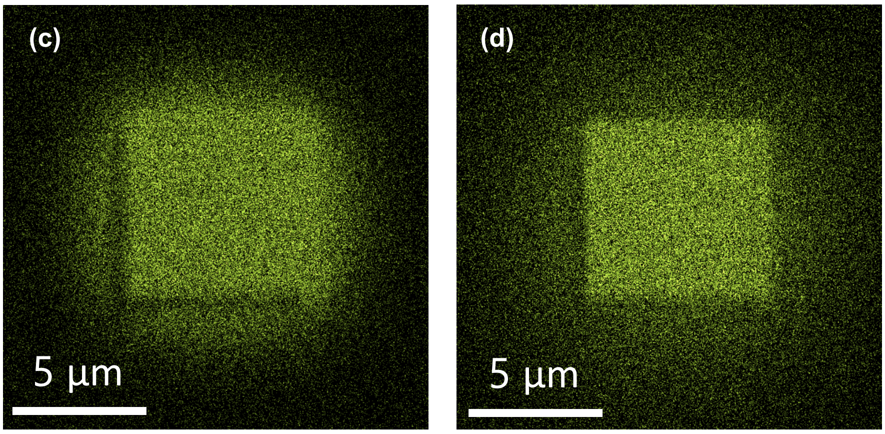


Figure 4. Elemental maps at Cu L line and corresponding atomic concentrations of representative Cu-FEBID deposits obtained at different acceleration voltages (Cu/C/O/F at.%): (a) 5 kV – 16.8/38.5/28.9/15.8; (b) 10 kV –17.1/36.2/28.2/18.6; (c) 20 kV – 16.3/39.8/30.1/13.8; (d) 30 kV – 16.0/43.5/31.8/8.8.

Description of the STSM main achievements and planned follow-up activities

Description and assessment of whether the STSM achieved its planned goals and expected outcomes, including specific contribution to Action objective and deliverables, or publications resulting from the STSM. Agreed plans for future follow-up collaborations shall also be described in this section.

(max. 500 words)

The STSM successfully fulfilled its aims by systematically optimizing focused electron beam induced deposition of copper structures from the $Cu(hfac)_2$ precursor. The central objective of the mission was to identify deposition conditions that yield high copper content while minimizing contamination from carbon, oxygen, and fluorine. This goal was pursued by carefully varying and evaluating critical process parameters, namely the electron beam acceleration voltage, beam current, dwell time, and scanning strategy. Through this systematic exploration, it was possible to determine processing windows that consistently produced deposits of improved purity and stable morphology.

Another important objective was the preservation of the geometric fidelity of deposited patterns, together with control over the vertical growth rate and surface morphology. These aspects were verified by SEM imaging across the full parameter matrix, confirming that optimized deposition conditions allow for reproducible and well-defined copper-containing structures. Complementary compositional analysis by energy-dispersive X-ray spectroscopy (EDS) further validated that the optimized settings indeed enhanced copper incorporation relative to other elements present in the precursor.

The campaign provided valuable experience in operating commercial gas injection systems integrated into state-of-the-art electron microscopes, thus offering practical insight into both the opportunities and the inherent limitations of commercially available FEBID platforms. This knowledge is directly relevant for the broader application of FEBID in nanotechnology and supports the development of standardized methodologies within the research community.

The outcomes of this STSM form the basis for the preparation of a scientific publication focusing on copper FEBID with $Cu(hfac)_2$. The planned manuscript will combine these results with previously acquired data on gallium and xenon focused ion beam induced deposition (FIBID), thereby enabling a comprehensive comparison of electron- and ion-driven growth processes. In addition, complementary transmission electron microscopy (TEM) studies are planned at the home institution, EMPA Thun, to provide further insight into the nanoscale structure and the electron irradiation mechanisms.

Finally, the mission strengthened the collaboration between the applicant and the host institution, creating a foundation for future joint work. Follow-up activities will include TEM-based microstructural analysis, comparative FEBID–FIBID studies and coordinated contributions to broader benchmarking of precursors and process mechanisms. These efforts will culminate in joint publications, thereby ensuring that the outcomes of the STSM directly contribute to the advancement of irradiation-driven nanofabrication processes.