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D10.6

Test experiment of transfer and positioning system to a soft X-ray microscopy setup

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Executive Summary

This JRA5 deliverable report D10.6 “Test experiment of transfer and positioning system to a soft X-ray microscopy setup” describes the status of the implementation of the transfer and positioning system to the soft X-ray microscopy setups at SOLEIL, France. All technical prerequisites have been developed, are readily available for use and are in particular as part already offered to NFFA transnational access users.

In particular, both, the nano-transfer tool developed within JRA5, including the three columns i) the Matlab and Python implementation of the software script, ii) the “hierarchical marker design on demand” as well as iii) the electron- (EBID) and ion-(IBID) induced deposition, as well as the capabilities for a nano-transfer at the SOLEIL beamlines are all implemented and integrated.

The goals of all three columns have been achieved, as separately documented in several JRA5 deliverable reports. The software-setup and the marker technology has been tested and approved, and are ready to use. At the soft X-ray beamlines at SOLEIL dedicated for the “Advanced Nano-Object Transfer and Positioning” the equipment and technical infrastructure ensures compatibility and easy implementation.

In a test experiment to be scheduled at the time of the deadline, the full integration of the nano-transfer tool on one hand and the positioning devices at the SOLEIL beamline at the other hand will be shown. At the same time, the array markers created by direct-write non-linear lithography at FORTH will be utilised to permit a nano-transfer based on the re-location of selected nano-objects at a nano-science centre to a soft-X-ray beamline setup at an Analytical Large Scale Facility (ALSF).

We in particular stress here that the compatibility between the modular transfer and positioning system and the hardware at the two dedicated SOLEIL parallel imaging X-ray photoemission electron microscope (XPEEM) and the scanning microscope (STXM) beamlines at SOLEIL has been achieved and the technical feasibility of D10.6 is assured.

1. Concept

For this deliverable report D10.6 we rely on previous reports documenting in greater detail the achievements of the subtasks developed so far within the Joint Research Action 5 “Advanced Nano-Object Transfer and Positioning”. In particular, these comprise

I THE HIERARCHICAL AND PARALLEL MARKER TECHNOLOGY INCLUDING

II THE “MARKER DESIGN ON DEMAND”,

III THE ELECTRON- (EBID) AND ION- (IBID) INDUCED DEPOSITION FROM A PT PRECURSOR MATERIAL BY GAS INJECTION INSIDE AN SEM TO WRITE HIERARCHICAL MARKERS, AND IN PARTICULAR THE

IV PARALLEL ARRAY MARKER TECHNOLOGY THAT

V HAS BEEN DEVELOPED AND TRANSFERRED FROM A BULK SUBSTRATE ONTO THIN SIN MEMBRANES TO FACILITATE THE TRANSMISSION MEASUREMENTS AT A SOFT X-RAY BEAMLINE, SEE, E.G., SECTION 2.1 AND 2.2., AS WELL AS

VI THE SOFTWARE TOOLS, SEE, E.G., SECTION 2.3., AND, FINALLY,

VII THE TECHNICAL CAPABILITIES AT THE SOFT-X-RAY BEAMLINES AT SOLEIL PERMITTING A NANO-TRANSFER, AS DESCRIBED IN SECTION 2.4.

2. Design specification

2.1 Hierarchical Marker Technology

A versatile tool to create well-defined hierarchical markers on dedicated positions is the electron- (EBID) or ion- (IBID) induced deposition of a precursor gas in an SEM or FIB-SEM. EBID or IBID permits to mark regions of interest after the pre-selection process based on the microscopic view onto distinct nano-objects [1]. As such, the most useful, promising and meaningful nano-objects can be selected well in advance of the X-ray beamtime. The marker optimization in the framework of the JRA5 and the development of this workflow is described in the deliverable report D10.4 “Methods for deposition of hierarchical markers”. In cooperation between JRA2 and JRA5 a series of thickness calibrations were performed in order to create markers with a desired thickness, and the results are described in detail within the deliverable report D7.14 “Test of re-alignment of nanostructures in synchrotron experiment by in-situ marker deposition”, in the framework of the cross-JRA cooperation between JRA2 - Research on High Precision Manufacturing and JRA5 and serve as bases to document acceptance of the design and protocols for the nano-transfer at nanocenters and ALSFs within the JRA5 milestone report MS13 “Design and protocols accepted at nanocenters and ALSF”. Figure 1 shows AFM height images of a series of markers written in the SEM that were used to calibrate the two EBID process parameters i) acceleration voltage of the electrons and ii) the spot-size, i.e. the aperture defining beam-size of the electron beam. This calibration procedure shows how an optimal marker thickness can be obtained by calibrating EBID/ IBID processing parameters. The bar on the left hand side of Figure 1 shows the colour code indicating that the thickness of all tested EBID markers deposited with all applied settings is lying in the range of 2-200 nm.

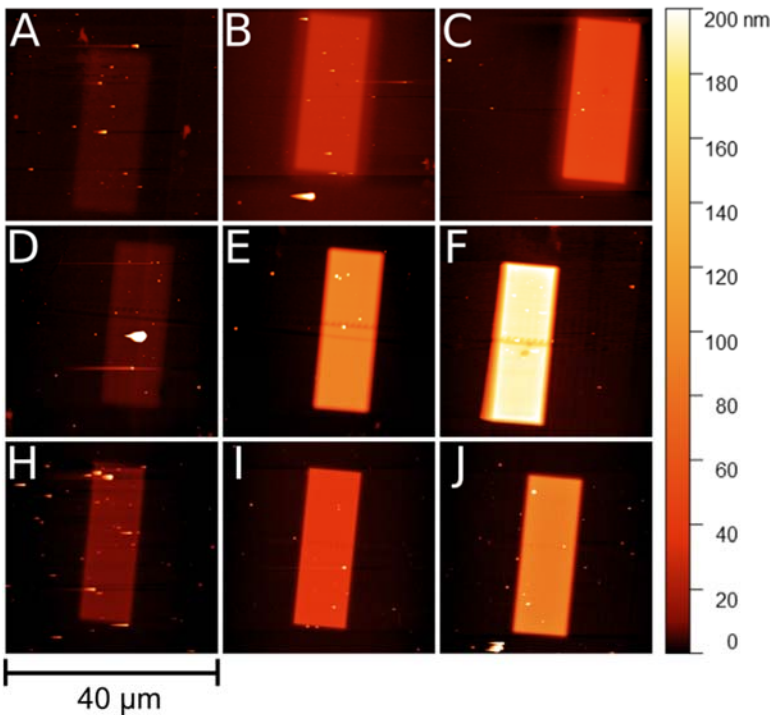

Figure 1: AFM height measurements of markers created by EBID in an SEM with varying the processing parameters spot size and acceleration voltage. a-c) deposited at 20 kV, d-f) at 5 kV, h-j) written at 10 kV. Spot-sizes in the left, centre and right column are 7, 6 and 5, respectively (C. Seitz, T. F. Keller, DESY, taken from the JRA5 deliverable report D7.14 and the JRA5 milestone report MS13).

2.2 Parallel Marker Technology

Optimized direct-write non-linear lithography and EDX imaging parameters have been obtained to achieve a resolution of line width of 50 nm in the secondary electron imaging mode of the SEM. Furthermore, EDX line profile for e.g., the constituent polymer resist element zirconium results in a lateral resolution down to 30 nm (full width at half maximum, FWHM), indicating a concentration and enrichment of the element into the center of the lines of the photoresist material. Fig. 2 show corresponding SEM images, see, e.g. for further details, the JRA5 milestone report MS8.

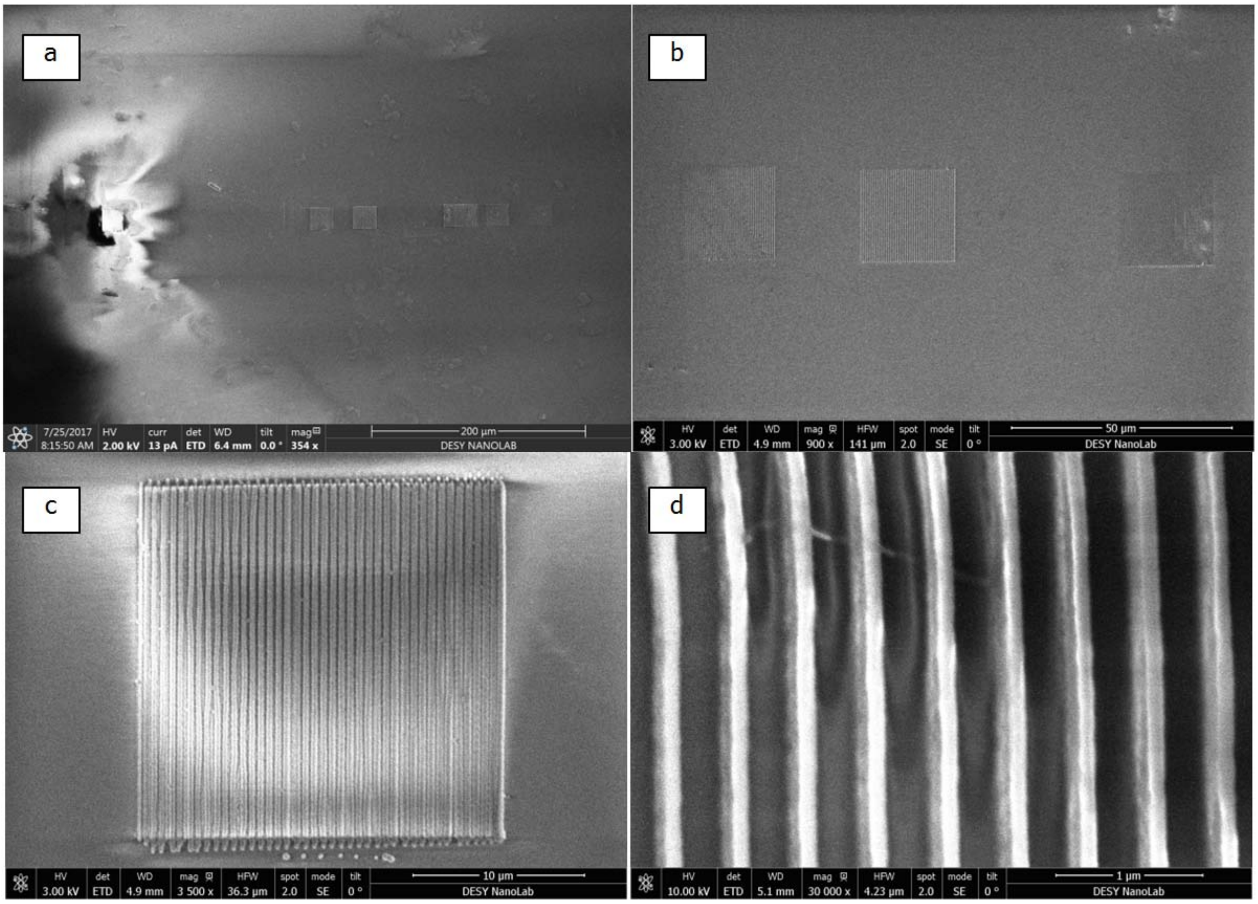

Figure 2: Representative SEM overview images of initial marker arrays fabricated via laser-based direct-write non-linear lithography (E. Stratakis, FORTH, taken from JRA5 mile stone report MS8).

From Figure 2 and more details described in MS8, it is clear that fluorescence markers with a lateral line resolution of ~50 nm have been successfully fabricated using a two-step process. Initially, sub-100 nm array markers are produced via laser-based direct-write non-linear lithography. Subsequently, isotropic plasma etching was applied to thin down lines of the marker arrays, realizing a lateral line resolution of ~ 50 nm.

In a next step, as will be described below, at FORTH, the array markers were written by laser-based direct-write non-linear lithography on Si_3N_4 carrier membranes. Transferring the technology to membranes enables the application of the array marker strategy to transmission experiments like, e.g., transmission electron microscopy, TEM, scanning transmission electron microscopy, STEM, and, as described here, nano-focused X-ray beam experiments in transmission at analytical large scale facilities, ALFSs, such as the soft X-ray beamlines at SOLEIL. This will allow developing a general array marker scheme for re-localization of top-down and self-assembled bottom-up nano-objects.

2.3 Software Tools

Software tools are needed to enable users from a broad range of scientific fields to conduct novel nano-science based on one-to-one structure-property-relationships of single nano-objects or nano-assemblies, combining high-end nano-instrumentation and synchrotron-based X-ray analysis.

Usually, the time dedicated to do an experiment is limited, for example in the framework of a granted beamtime, usually lasting several days. With the availability of the stage coordinates of all markers and nano-objects, the use of the developed software tool facilitates a fast, simple and easy sample transfer and re-location of the region of interests. The software scripts developed within JRA5 considers all coordinates of hierarchical markers as first input. This software basically changes the coordinates system and gets the absolute and relative distances of all the important objects in the experiment in the new system of reference. The Matlab implementation of the Advanced Nano-Object Transfer and Positioning protocol developed within JRA5 as shown in Figure 3 offers a graphical user interface (GUI) with 3 regions of user interaction, as described within the JRA5 deliverable reports D10.1 “Software routine for coordinate definition and relocation of single nano-objects”, D10.5 “Demonstration of relocating nanoobjects based on in-situ deposited fluorescence” and the JRA5 milestone report MS13 “Standardization of sample transfer and nanopositioning”:

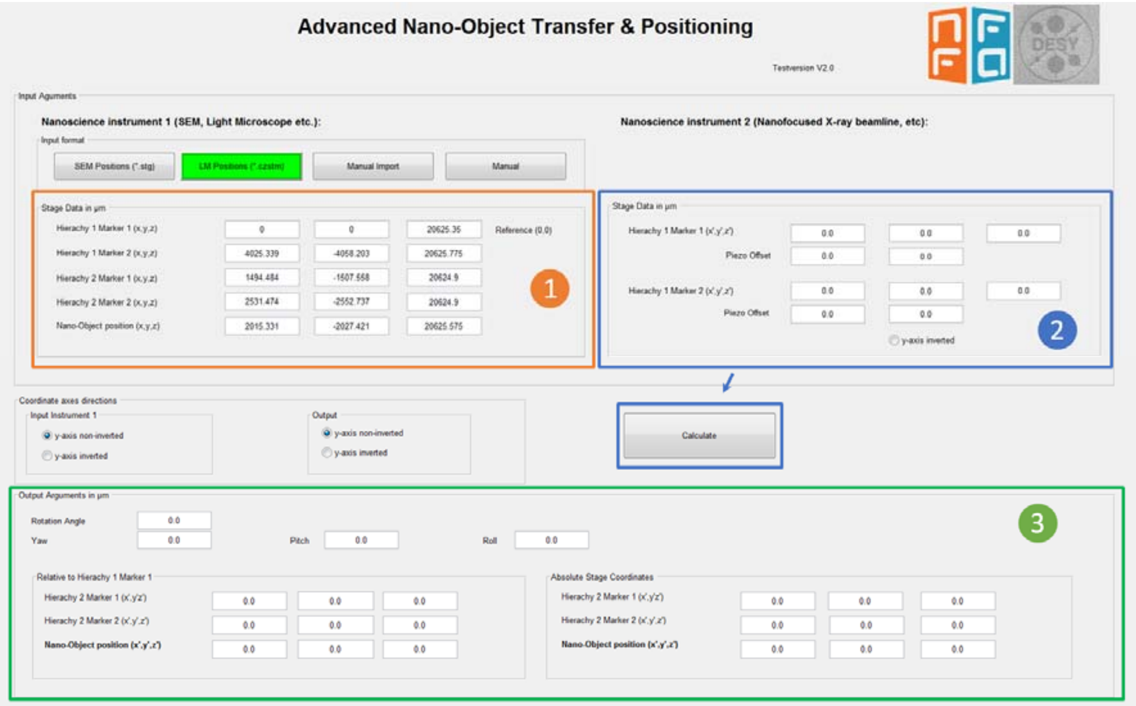

Figure 3: Screenshot of the MATLAB scripts in 3 steps that users have to follow in this version of the software (A. Böhme, M. Abuin, T.F. Keller, A. Stierle, DESY, taken from JRA5 deliverable report D10.1).

“1” indicates the area for the input coordinates obtained from the nano-instrument at the nano-science centre, by e.g., using an SEM, optical light microscope or AFM, “2” marks the input section for the new motor position coordinates on the instrument or beamline, where the region of interest shall be re-located, and “3” indicates the area containing the outcome of the coordinate transform calculation with the resulting coordinates of the region of interest. The output furthermore comprises coordinate-transformed mutual relative distances and absolute values for all saved positions, including all markers and all nano-objects of interest, which can also be exported in a tabulated ASCII format file.

2.4 Transfer capabilities at Soft X-ray beamlines at SOLEIL

Both dedicated two soft X-ray beamlines, the parallel imaging X-ray photoemission electron microscope (XPEEM), and the scanning microscope (STXM) at SOLEIL are equipped with an optical light microscope capable to localise fields of parallel marker arrays or 0. order hierarchical Pt based markers that can lead to the nano-region of interest to be analysed by the respective soft x-ray technique using the software tool described in section 2.3. Figure 4 shows such optical light microscopic images of guiding marker structures already in use.

Figure 4: Optical light microscopic images of dedicated markers with close-by nano regions of interest obtained from a soft X-ray beamline at SOLEIL (R. Belkhou, CNRS / SOLEIL).

Here, as e.g., for the samples shown in Figure 4, markers are produced by lithography masks. The X-ray stage beamline motors have an overall positioning accuracy < 100 nm.

As such, the setup is overall fully compatible with the developments within JRA5, i.e., the hierarchical markers written by the IBID/EBID process (DESY) and their sample specific design, the parallel array markers created by the direct-write non-linear lithograph (FORTH) as well as the software scripts. Due to their stand-alone nature of the software implementations they are largely independent from any hardware implementation provided all necessary sample (stage) coordinates (x,y,(z)) can be electronically or even manually read out and fed into the Matlab or Python "Advanced Nano-Object Transfer and Positioning" script described in section 2.3.

3. Results

Based on the achievements of the high resolution arrays and lines on bulk samples created by the direct-write non-linear lithograph at FORTH, the creation of array markers was subsequently transferred to thin SiN membrane samples, which are suitable for soft X-ray transmission experiments. Figure 5 documents that marker arrays can be successfully written by laser-based direct-write non-linear lithography onto Si_3N_4 carrier membranes. For this upcoming experiment at SOLEIL, a 10 µm wide period of parallel lines was chosen, and optical light microscopy proved the line width to be around 350 nm.

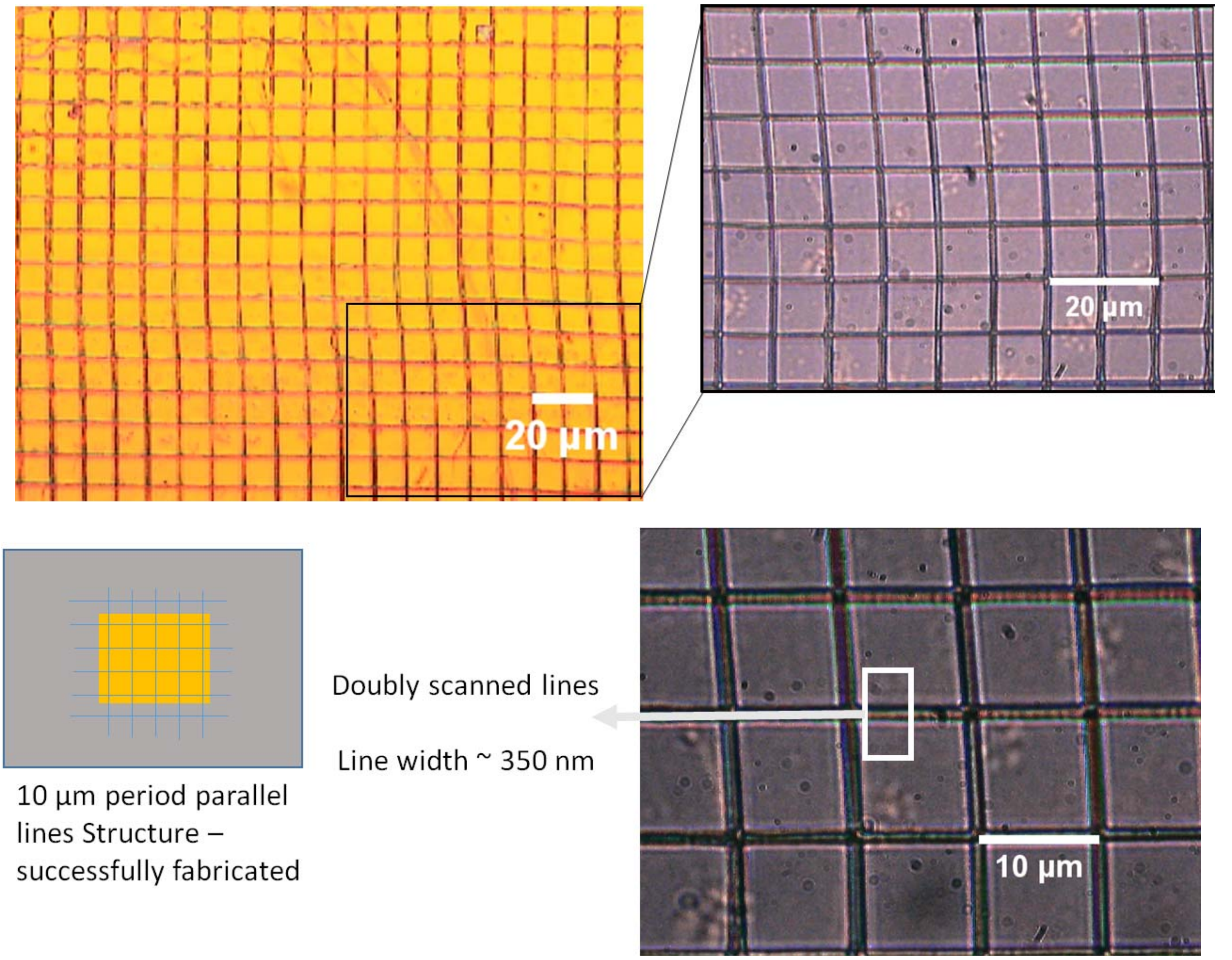

Figure 5: SiN membrane samples equipped with array patterns written by laser-based direct-write non-linear lithography onto (E. Stratakis, FORTH).

Due to the limited availability of the soft X-ray beamlines the "Advanced Nano-Object Transfer and Positioning" protocol has to be tested in the framework of a research project with scientific relevance. This overall aim to combine the test experiment on a scientifically useful outcome led us to postpone the test experiment such that it is still pending at the date of submission of this deliverable report D10.6. However, it has to be stressed, that all subtasks for itself are achieved, and the technical feasibility is given based on all the outcome and developments of JR5 and the infrastructure available at the two dedicated soft X-ray beamlines at SOLEIL. The arrays on the membranes will be used to identify single nanoparticles pre-selected in an SEM after attachment via solvent evaporation of a nanoparticle suspension (according to standard sample preparation of nanoparticles onto TEM membranes).

4. Conclusions and perspectives

Overall, all major steps to fully reach the deliverable D10.6 has been achieved, and are available, or technical feasible. Contrary to the expectation and schedule, we were not able to achieve the experimental test at the beamline at the time of the deadline. This remains pending due to the limited scheduling possibilities at either of the two dedicated soft X-ray beamlines at SOLEIL, and the development of the involved process technology transfer, as, e.g., the implementation of the direct write lithography on the SiN membrane surface, which has now been achieved and is here documented.

Most importantly, we stress that the compatibility of all soft and hardware components required for a successful test experiment of an "Advanced Nano-Object Transfer and Positioning" at a soft X-ray beamline at SOLEIL have been achieved and the software-setup is ready to use.

References

[1] Stierle, A., et al. "DESY NanoLab." Journal of large-scale research facilities JLSRF 2 (2016): 76.