LITESCOPE™ AFM-IN-SEM: ADVANCED TOOL FOR CORRELATIVE IMAGING AND SURFACE CHARACTERIZATION

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Abstract

Atomic force microscope (AFM) LiteScope™ produced by NenoVision is carefully designed for direct integration into many different types of scanning electron microscopes (SEM). It is equipped with unique technology for true correlative imaging - Correlative Probe and Electron Microscopy™ (CPEM). It allows simultaneous measurement of various signals of SEM, AFM and other related techniques like Electron Beam Induced Current (EBIC) or Catodoluminiscence (CL). LiteScope also enables using other methods such as Focused Ion Beam (FIB), Gas Injection System (GIS) or Electron Dispersive X-ray (EDX) to modify and right away analyze the sample surface. Among the main applications belong e.g. 3D surface characterization, height/depth profiling, surface roughness calculation, precise tip navigation, nanoindentation and nanomanipulation, variety of spectroscopic regimes, measurement of electrical and mechanical sample properties, etc.

Keywords: Correlative Probe and Electron Microscopy (CPEM), Atomic Force Microscopy (AFM), Scanning Electron Microscopy (SEM), Focused Ion Beam (FIB)

1. INTRODUCTION

A correlative microscopy is an approach which benefits from imaging of the same object by different techniques. Correlation between the data, measured by two or more independent methods, can provide a better understanding of the sample features, which could be too complicated to analyze by those methods separately. Correlative imaging by AFM and SEM is challenging due to different coordinate systems, spatial resolution, scanning nonlinearities, etc. A novel technique CPEM offers installation of AFM microscope LiteScope into the SEM chamber which overcomes these problems and provides true correlative imaging.

2. PRINCIPLE OF CPEM

Correlative Probe and Electron Microscopy technique is based on simultaneous measurement of AFM topography and SEM signal. In CPEM imaging, the electron beam is kept still (spot mode) in a constant distance close to the stationary AFM tip while scanning the sample by piezo scanner underneath, see **Figure 1 (a)**. Neither the e-beam nor the AFM probe are moving during CPEM image acquisition. All data are recorded in the same software NenoView, where the constant offset between AFM and SEM image can be compensated, see **Figure 1 (b)**. Shift of the AFM and SEM images with the identical pixel size by a constant offset provides the perfect overlap of both images. Another advantage of the simultaneous sampling from an identical spot is the same kind of image distortion for both measurements. Thus, the correlative CPEM imaging really performs the analysis on the same surface, at the same time and under the same environmental conditions. This way, **Figure 1 (c)** shows the possibility of multiple signal correlation of several techniques included in the SEM and AFM microscope which enables complex sample characterization.

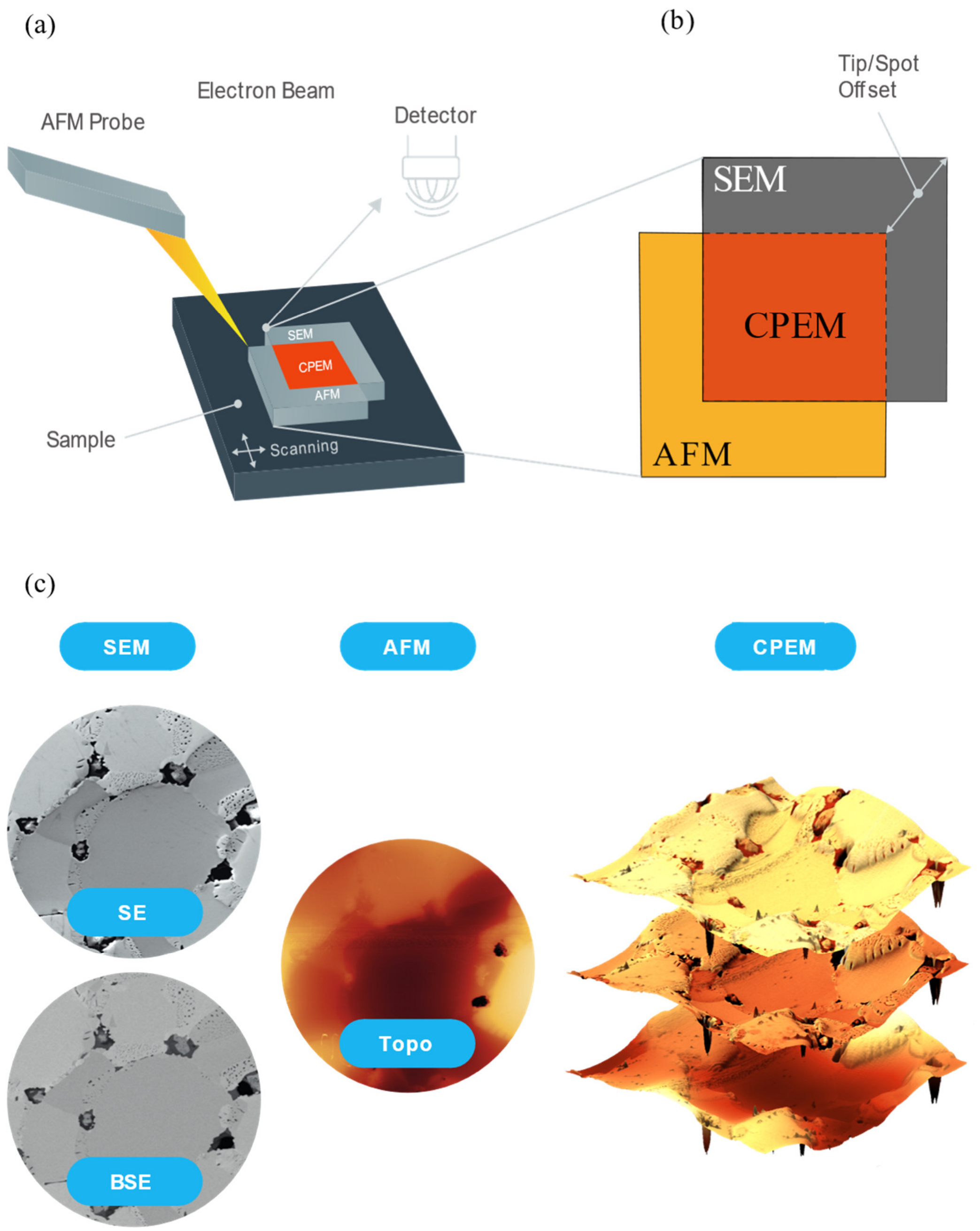

Figure 1 The principle of correlative imaging by CPEM. **(a)** Scheme of CPEM simultaneous measurement of the same area in the same coordinate system. **(b)** Scheme of perfect overlap of SEM and AFM images. **(c)** Example of CPEM measurement - tungsten-chromium alloy with HfO_2 particles. 3D view shows the topography alone (bottom), topography + BSE signal (middle) and topography + SE signal.

3. APPLICATION

LiteScope with CPEM is a powerful tool for investigation of 1D, 2D, and 3D nanostructures in several application fields such as nanotechnology, nanoelectronics, material science, life science, etc. CPEM imaging is a noticeably time-saving technique ensuring the same measurement conditions. Further methods such as FIB can be used to modify the sample surface enabling easier interpretation of the sample features.

In this section, we will describe the advantage of in-situ LiteScope measurements on a few unique applications like silicon nanopillars covered by WSe_2 flakes or metastable iron thin film whose magnetic properties depends on FIB irradiation.

3.1. Silicon nanopillars covered by WSe_2 flake

Tungsten diselenide (WSe_2) thin layer covering silicon nanopillars is an example of nanoelectronic application. WSe_2 is an optically active semiconductor material belonging to the group of transition metal dichalcogenides. Thanks to its flexibility, the exfoliated WSe_2 flakes can be formed to create the mechanical tension in the material and influence the photoluminescence properties. For instance, the certain shape of the layer on the nanopillar creates a single-photon emitter [1]. A localized deformation of WSe_2 shown in **Figure 2** was prepared by placing the WSe_2 flake on silicon nanopillars by hot dry transfer. Approximately 100 nm high Si nanopillars were fabricated by Electron Beam Lithography (EBL) and etched by Reactive Ion Etching (RIE).

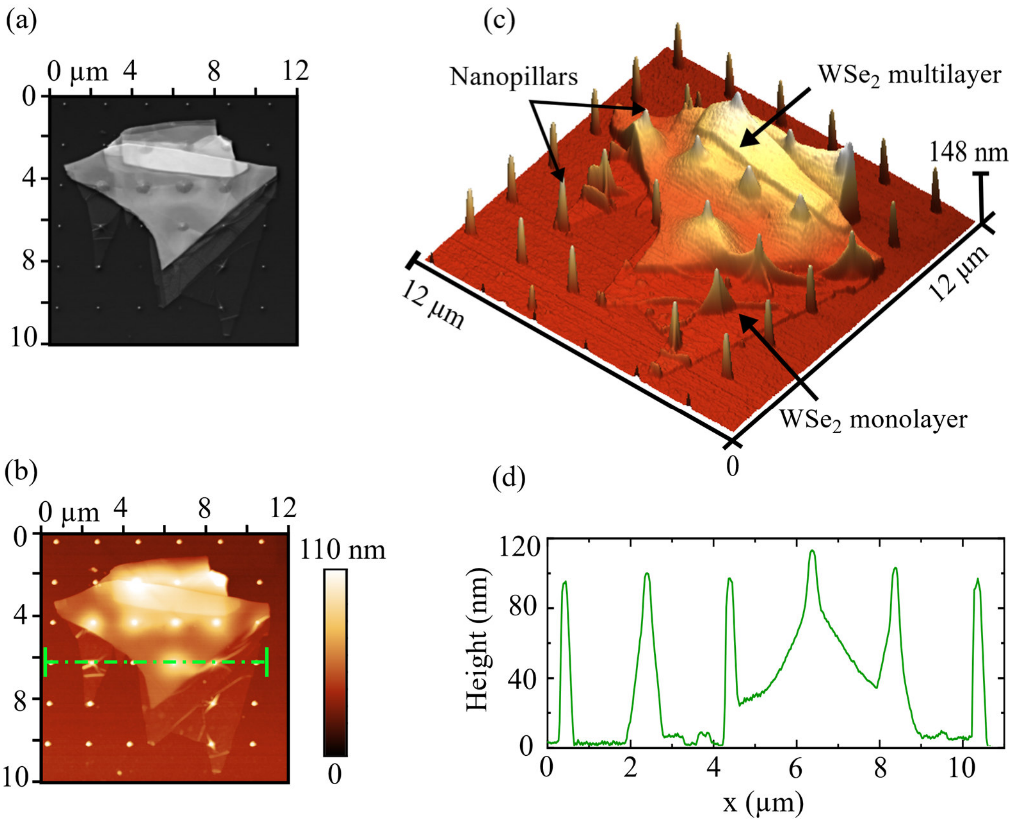

Figure 2 CPEM measurement of WSe_2 flake deformed by Si nanopillars. Simultaneous measurement of **(a)** secondary electrons (SEM signal) and **(b)** AFM topography of the sample surface. **(c)** 3D CPEM view of deformed WSe_2 flake where the monolayer is distinguished from multiple layers of WSe_2. **(d)** True height profile along the green line in (b) shows how the WSe_2 flake copy the surface of Si nanopillars.

The advantage of in-situ AFM in SEM is an easy localization of the WSe_2 flake and the fast navigation of the AFM probe to the area of interest, thanks to the large view field, high resolution and the visible material contrast of the different number of layers in SEM (**Figure 2 (a)**). On the other hand, AFM topography provides information about the nanopillars height and WSe_2 flake thickness (**Figure 2 (b)**). Thus, CPEM view reveals the exact shape of an intact WSe_2 flake, which fully covers the nanopillars. It is possible to distinguish the monolayer and multiple layers of the nanomaterial (**Figure 2 (c)**) and observe the true height profile of WSe_2

flake copying the Si nanopillars (**Figure 2 (d)**). The monolayer seems to be almost transparent while multiple layers get brighter on SEM. The presence of monolayer was also confirmed by Raman spectroscopy.

3.2. Metastable iron thin film grown on cooper monocrystal

LiteScope is indispensable for FIB applications and allows investigation right after the surface modification without breaking the vacuum or moving the sample. It is an advantage especially for sensitive samples whose surface properties change when exposed to the atmospheric conditions. **Figure 3** shows a metastable iron thin film grown on Cu(100) substrate, whose magnetic properties can be selectively changed when irradiated by an ion beam.

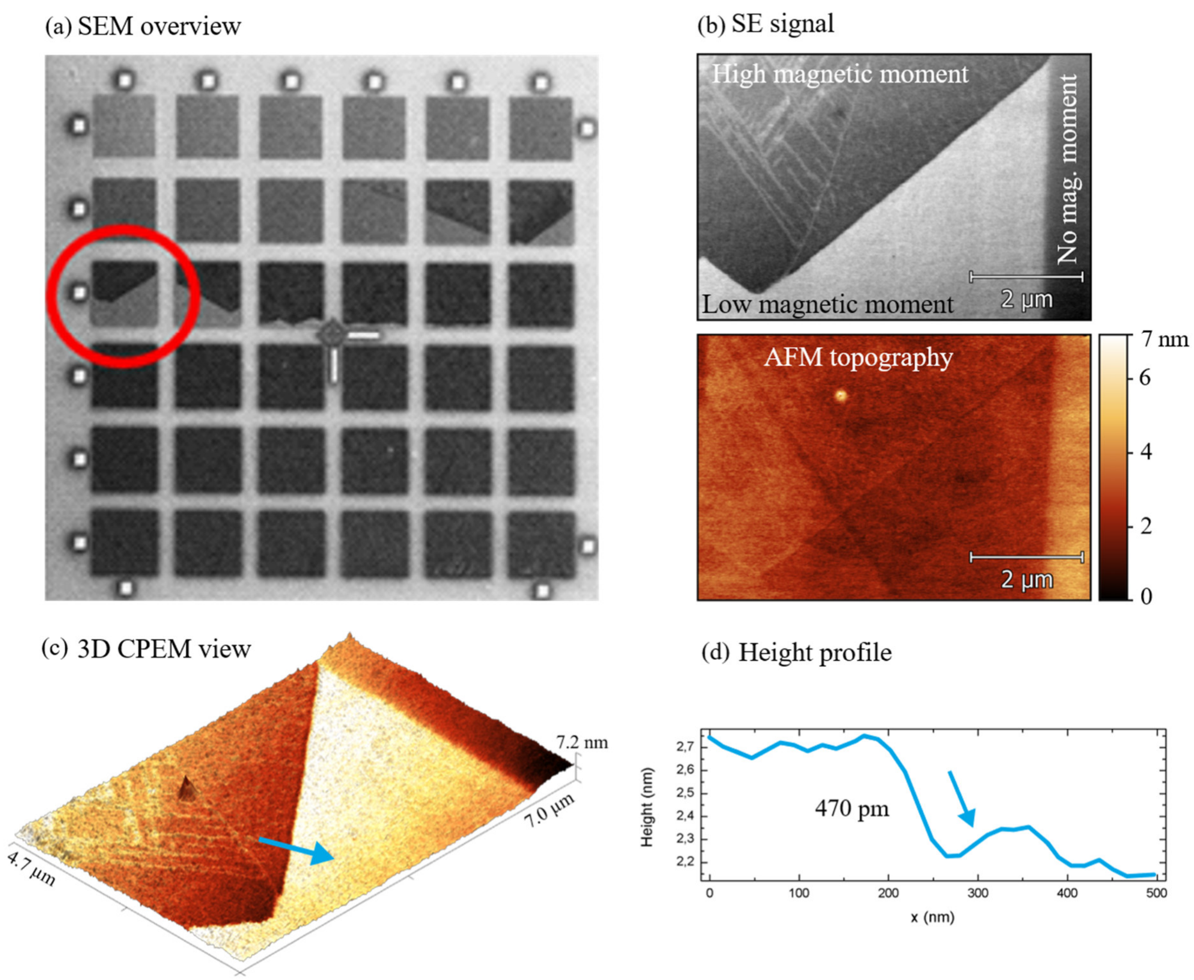

Figure 3 Transformation of metastable Fe/Cu(100) thin film by FIB. **(a)** An exposure test (overview) of selectively irradiated areas in SEM. **(b)** Simultaneous measurement of the square of the interest by secondary electrons in SEM (top) and AFM topography (bottom) after FIB irradiation. **(c)** 3D CPEM view and **(d)** height profile of the step of transformed material along the blue line.

First, the sample was irradiated by different gallium ion doses. In the SEM image (**Figure 3 (a)**), diverse material contrast was observed due to the material transformation from paramagnetic (fcc) to magnetic (bcc). Such a system of in-situ AFM in SEM enables simultaneous measurement of the modified surface (**Figure 3 (b)**). Thus, CPEM measurement allows precise inspection of magnetic transformations, see 3D CPEM view in **Figure 3 (c)**. By selecting the proper ion dose, the number of scans over the inspected area and the FIB scanning direction with respect to the domains, it is possible to optimize the magnetic anisotropy of the modified patterns [2]. Height profile in **Figure 3 (d)** proves that the transformation has not only the crystallographic contrast, but it is also visible in AFM topography. The step height between (fcc) and (bcc) domains was estimated to be 470 pm.

4. CONCLUSION

Atomic force microscope LiteScope is a powerful instrument for in-situ AFM measurements in SEM and it is used for surface examination in material science, nanotechnology, life science and so on. It is not only easy target localization and fast AFM probe navigation which makes the in-situ LiteScope special. CPEM technology enables simultaneous measurement of multiple SEM and AFM signals while the coordinate system and image nonlinearities like distortion are the same and therefore negligible. CPEM highlights the diversity of AFM and SEM image and provides complex information about the sample. Further, the CPEM can be combined with other techniques included in SEM or AFM. For instance, it is possible to modify the surface by FIB and right away continue with CPEM measurement which provides exact information about the sample surface without exposing the sample to the atmospheric conditions.

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