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Two Microscopes are better than One – *In-situ* Correlative Analysis by combination of AFM, SEM, and FIB

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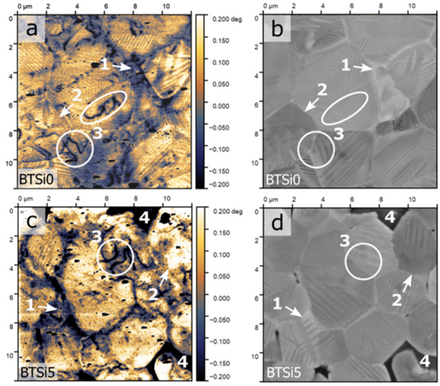
Combining different analytical methods in one instrument is of great importance for the simultaneous acquisition of complementary information. Especially the *in-situ* combination of scanning electron microscopy (SEM) and atomic force microscopy (AFM) enables completely new insights into the micro and nano-world. In this work, we present the unique *in-situ* combination of scanning electron / ion microscopy (SEM/FIB) and atomic force microscopy (AFM) for nanoscale characterization [1-2].

A particularly important aspect for advanced AFM measurements are the cantilevers itself. Especially the cantilever tips have to provide special properties to measure not only the topography, but also magnetic and electrical characteristics of materials. In this context, the FEBID process is a very promising approach for the design and fabrication of sophisticated tips with unique properties. We will present some case studies to highlight the advantages of interactive correlative *in-situ* nanoscale characterization for different materials and nanostructures, using FEBID-constructed tips. We show results for the *in-situ* electrical characterization by conductive AFM for 2D materials as well as electrostatic force microscopy (EFM) measurements of piezoceramic films that enables the precise analysis of grain boundary potential barriers in semiconducting $BaTiO_3$-based ceramics [3]. The grain boundaries were located via BSE-SEM and measured afterwards using the in-situ EFM method. The barriers were shown to be significantly thinner and more pronounced as the amount of SiO_2 was increased from 0 to 5 mol% (see Fig. 1). These results can be directly correlated with electron backscatter diffraction (EBSD) measurements in order to link the AFM and SEM data to the crystallographic microstructure.

In addition, we will present results for the *in-situ* characterization of magnetic nanostructures by combination of SEM and high-vacuum magnetic force microscopy (MFM). For the *in-situ* MFM measurements, special high aspect ratio magnetic cantilever probes fabricated by electron beam induced deposition (FEBID) were used, which surpass conventional cantilevers in terms of lateral and magnetic resolution. The SEM enables to identify the grain boundaries on multilayer thin-film samples or stainless steel in order to measure the magnetic properties directly via MFM with nanometer resolution (see Fig. 2).



	C–V [µm]	EFM [µm]
BTSi0	0.89 ± 0.04	0.92 ± 0.13
BTSi2	0.47 ± 0.02	0.50 ± 0.08
BTSi5	0.53 ± 0.03	0.52 ± 0.08

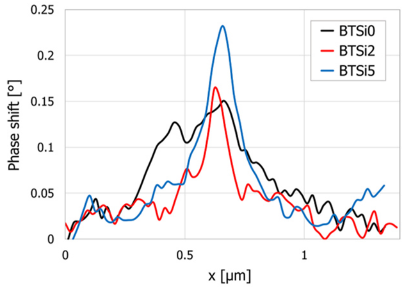


Fig 1: (Left) EFM signal of (a) BTSI0 and (c) BTSi5 with corresponding BSE-SEM images (b) and (d) of the exact same sample area. The EFM images were recorded with an external voltage of -3 V. (Right) Grain boundary potential barriers of $BaTiO_3$ based ceramics with varying SiO_2 content. The Images were extracted from [3].

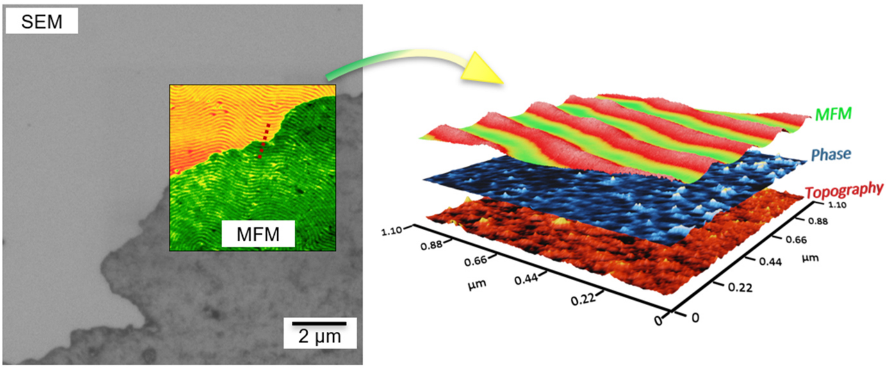


Fig 2: (Left) SEM image of a multilayer magnetic sample with correlative MFM image at a grain boundary. (Right) AFM data overlay of topography, phase, and MFM signals.

[1] D. Yablon, et al.; *Cross-platform integration of AFM with SEM: Offering the best of both worlds*; Microscopy and Analysis 31 (2) (**2017**), 14.

[2] S.H. Andany, et al.; *An atomic force microscope integrated with a helium ion microscope for correlative nanoscale characterization*; Beilstein Journal of Nanotechnology 11 (**2020**), 1272.

[3] J.M. Prohinig, J. Hütner, K. Reichmann, S. Bigl; *Complementary evaluation of potential barriers in semiconducting barium titanate by electrostatic force microscopy and capacitance-voltage measurements*; Scripta Materialia 214 (**2022**), 114646.