doi:10.1017/S1431927613007150
Microsc. Microanal. 19 (Suppl 2), 2013

Methods for Micro- to Nanometer Correlative Tomography

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Tomographic methods in material and life science have experienced an increasing interest, since a third spatial dimension provides significant insight into their "special" arrangement not readily observed with surface of projection imaging techniques. Even better than correlating morphological features, is the ability to combine characterization methods, which deliver complementary information on compositions, physical properties and features. Even though interrogating surfaces or viewing 2-D projections of materials is invaluable, tomographic data from different modalities on one and the same sample, will reveal new insights not realized before. Achieving this correlative ultimate tomographic analysis of a material is non-trivial however, and requires sample handling and correlation of data between different imaging techniques.

In this contribution we discuss a variety of methods we are investigating not only to correlate 3-D images between microscopes, but extend combinatorial techniques to including the correlation between analytical information of chemistry and crystallography. Micron scale structures of bone, first identified via CLSM and harvested with the FIB to achieve 3-D density measurements with cSAXS, were finally sequentially FIB milled and imaged to reveal ultrafine structures (Fig. 1).[1]

Similar techniques can be used in correlations with atom probe tomography. Basic yet fruitful correlations between HRSEM images and Atom Probe Tomography (APT) can reveal geometric inputs for reconstructions. More direct chemical correlations are feasible between STEM tomography and APT. Methods we will discuss to achieve the spatial and chemical correlations involve modified TEM specimens and well known pillar geometries (Fig.2). In the interest of maximizing the field-of-view within the S/TEM of the microstructural regions of interest (ROI), and localizing these particular ROIs at the end for an atom probe specimen, a combination of sample preparation techniques are needed. We will discuss methods such as of tripod [2] or broad ion beam polished in combination with a final FIB pillar formation on selected ROI.. Examples from both nanostructured metal and semi-conductor materials will be presented, as well as first attempt on bioorganic material to show the potential in closing the gap from the micro- to the nanometer scale in combination with additional analytical characterization such as the atomic compositional level by atom probe investigation.

References:

[1] M. Dierolf *et al*, Nature **467** (2010), p.436-439.
[2] D. Saxey *et al*, Ultramicroscopy **107** (2007), p.756.
[3] The authors acknowledge the *Swiss National Science Foundation for support via R'Equip and the support from Oliver Bunk at the SLS Beam line, PSI, Switzerland as well as the cooperation with Philip Schneider from the Biophysic Department of ETH, which provided the mouse bone samples and his interest in correlative microscopy.*

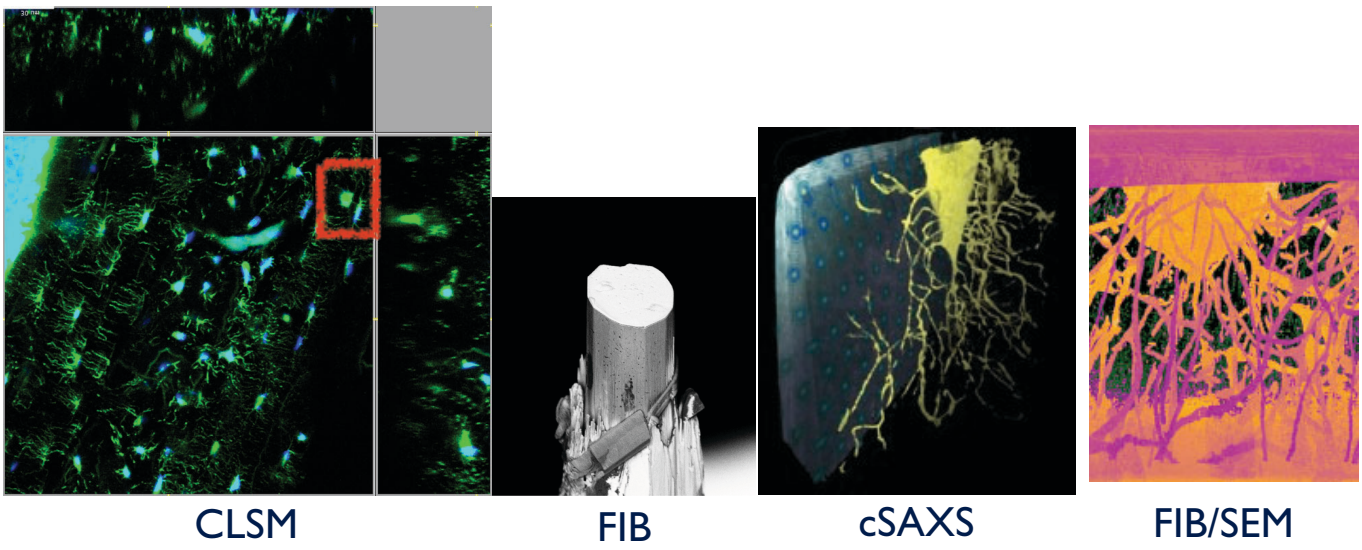

Figure 1. The workflow, of correlating micron sized 3D features in light microscopy to density variations from cSAXS Tomography to the final ultrafine structure determination by FIB/SEM slice and view tomography, is shown from left to right with representative images at the same specimen area in the different imaging modalities. .

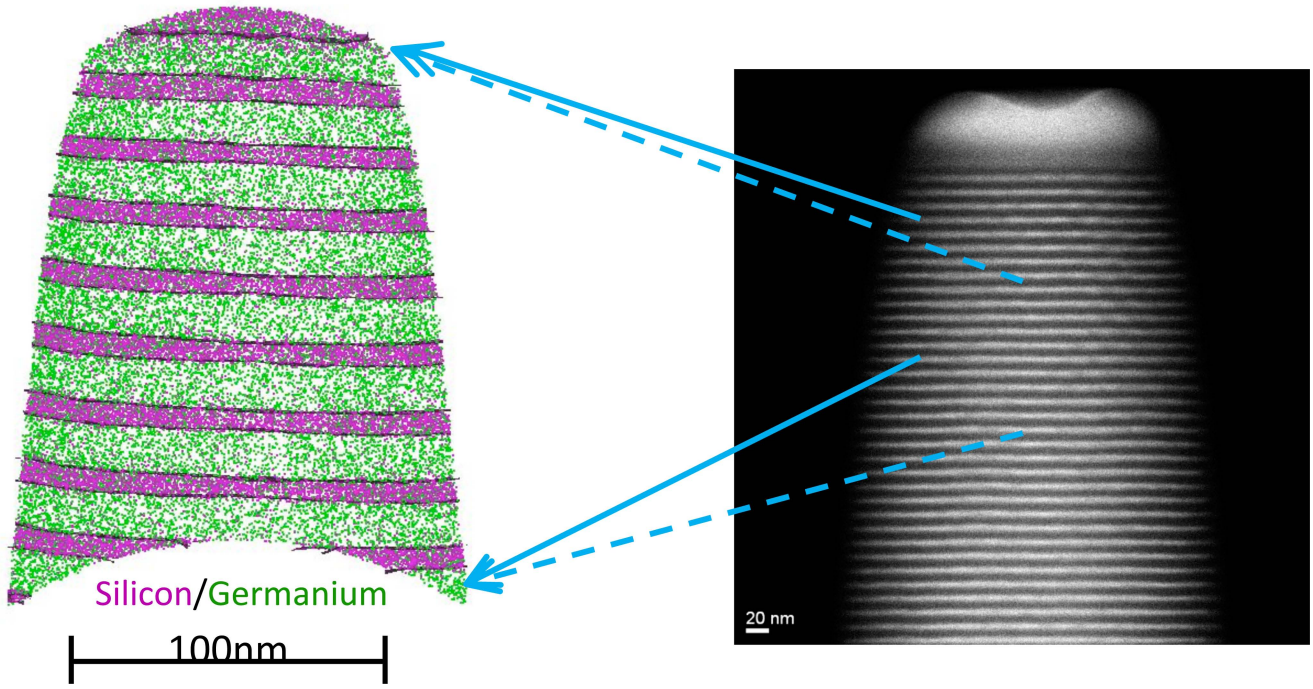

Figure 2. Correlations between APT (left) and STEM tomography (right) are essential for determining precisely which part of the larger structure was resolved in APT and to correct for the position of the single atoms in the 3D model.

https://doi.org/10.1017/S1431927613007150 Published online by Cambridge University Press