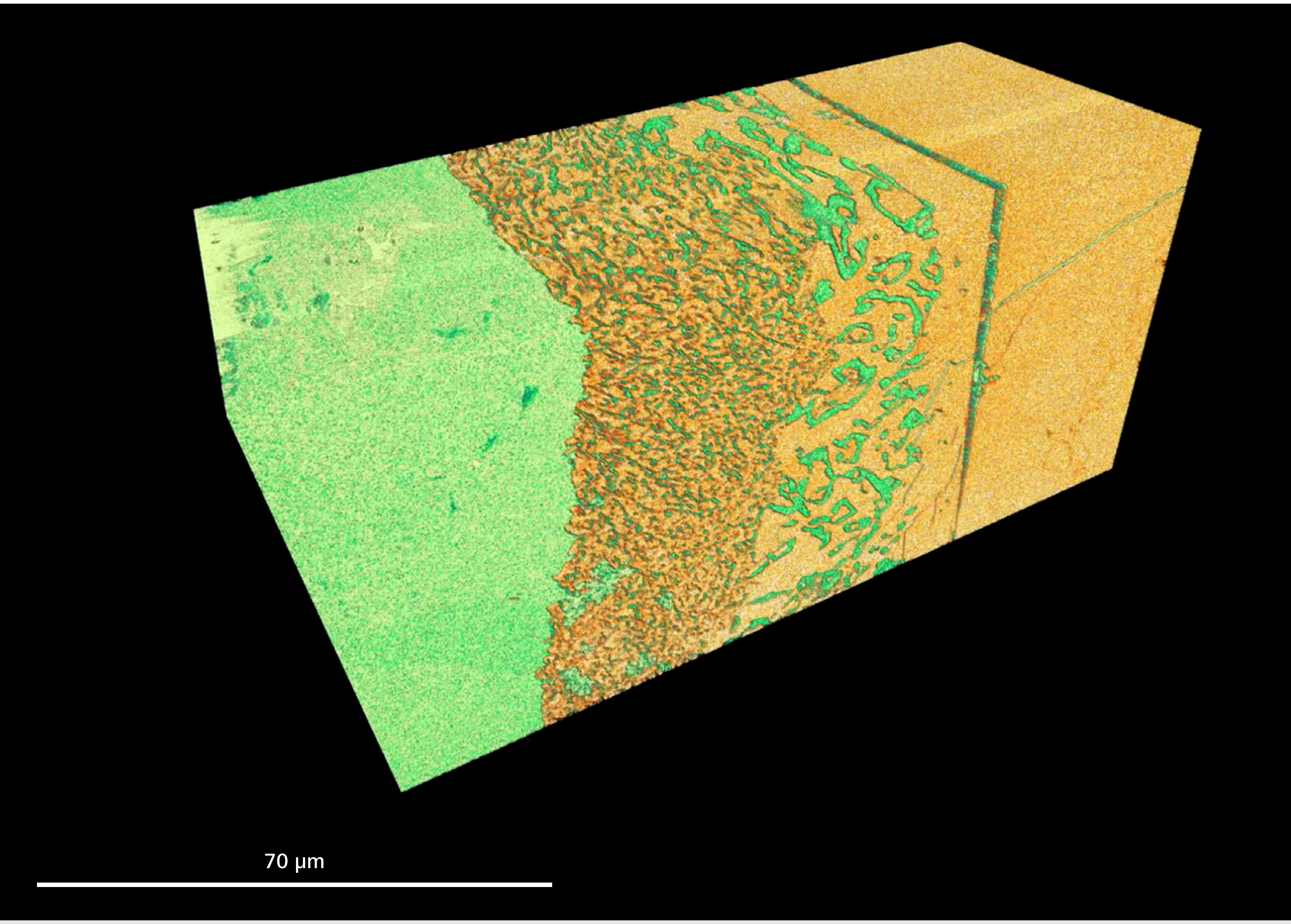
Application Note



Correlative XRM-FIB/SEM Study of Thermoelectric Materials

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Correlative XRM-FIB/SEM Study of Thermoelectric Materials

Sample courtesy: Dr. Ali Chirazi , Dr. Stéphanne Gorsse,
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ICMCB-CNRS

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Increasingly materials research requires multi-scale investigation, or need to combining or correlate information from different experimental modalities at the same region of interest. This can be a challenge since the region of interest can be below the sample surface. One of several investigations utilizing laboratory X-ray Microscopy (XRM) at the Bordeaux Institute of Condensed Matter Chemistry (ICMCB-CNRS) involves a closer sub-surface look at a special class of thermoelectric materials for automotive applications. As much as 40% of an internal combustion engine's energy use goes to producing exhaust gas, and thermoelectric materials are utilized to recoup wasted energy in the form of heat generated by engines by converting this heat to electricity, boosting overall automotive efficiency.

Several prototypes have been explored in the past, but now more recent classes of materials are being studied in order to significantly boost the thermoelectric efficiency, via a 'materials by design' computationally-assisted approach. Designing a microstructure and composition requires an understanding of how to optimize electrical and thermal conductivity, heat capacity and phonon scattering, many of which are directly affected by 3D micron-scale and nanoscale structure. Thermal treatments can alter this microstructure, and are of interest to be studied, how this impacts thermoelectric performance.

A thermoelectric Mg_2Sn-Mg_2Si system is studied in closer detail. After certain heat treatments, a transition zone which defines the boundaries of these two phases forms, often with a very complex microstructure. First, it is of interest to image and quantify the volume of each phase (Mg_2Sn, Mg_2Si, and transition zone) via X-ray tomography. This step has been performed using the ZEISS Xradia 510 Versa X-ray Microscope, to image the entire sample with multiple resolutions (down to sub-micron) non-destructively. Figures 1 and 2 illustrate the ability to identify each phase (and defects) of this material system by utilizing sub-micron XRM. This provides a volumetric measure of the amount of each constituent phase and also the amount of defects (voids) present. It also can provide a starting point for identifying the region of interest for further more detailed analysis (e.g. FIB-SEM).

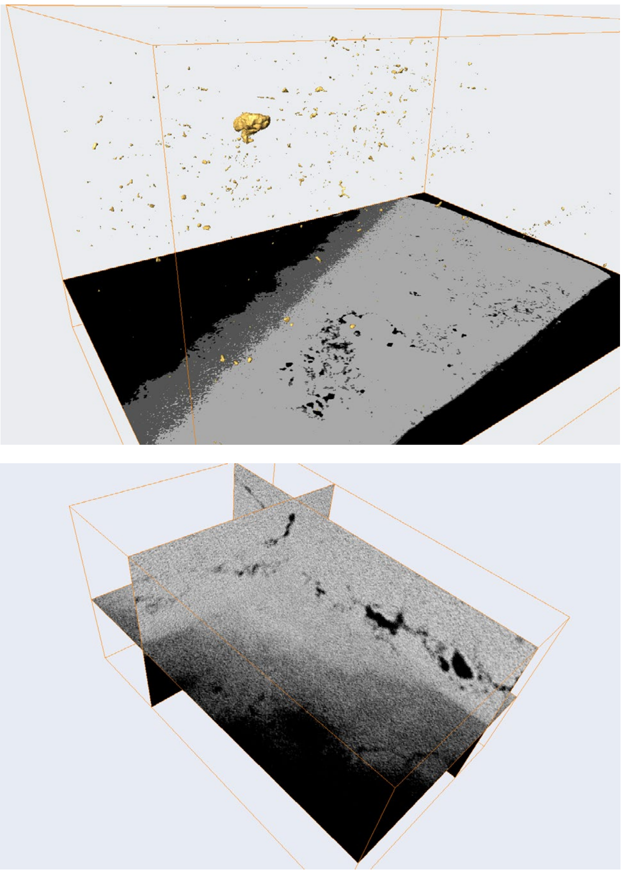

Figure 1 *Top: 790 nm voxel XRM acquisition (ZEISS Xradia Versa) of ~1 mm region of thermoelectric sample, including volume renderings of pore/defect structure (yellow) Bottom: 200 nm voxel XRM acquisition of thermoelectric material showing 3 phases (black=*Mg_2Sn*, medium grey = transition zone, light gray =* Mg_2Si*).*

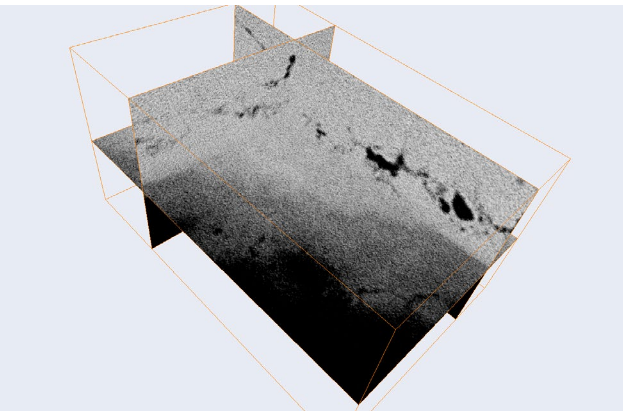

Figure 2 *Volume rendering (yellow) of XRM dataset (~0.5 mm FOV) showing transition zone defects and coarse diffusion pathways in this thermoelectric system.*

Figures 3 and 4 highlight the 3D ZEISS FIB-SEM acquisitions and results. The XRM dataset was used to inform the acquisition of the transition region. The fine structure of the transition zone (~25 µm in width) is able to be located with XRM, but then fully characterized by FIB-SEM analysis. The ZEISS EsB detector has clearly shown the ability to separate both primary phases by grey level, as well as the defects. This leads to a more straightforward path toward quantification and segmentation.

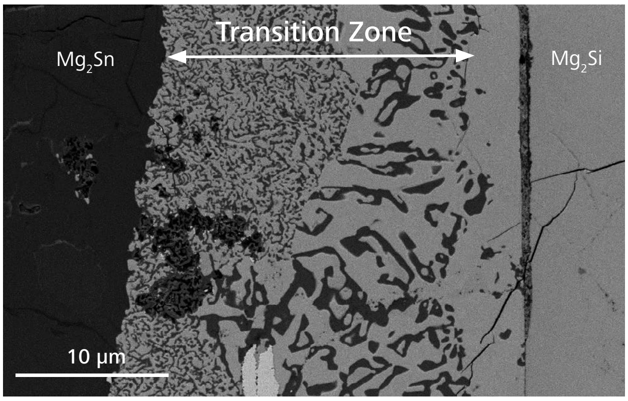

Figure 3 *Single slice from ZEISS FIB-SEM acquisition, using EsB detector. 5k × 2.5k pixels, corresponding to ~40×20×5 µm volume, acquired with isotropic 8 nm voxels.*

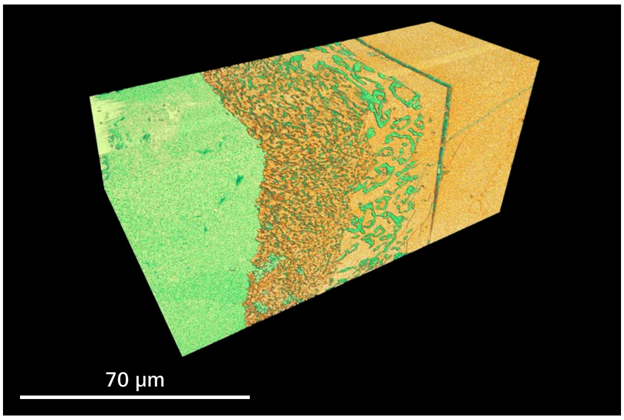

Figure 4 *Volume rendering of ZEISS FIB-SEM acquisition, showing a larger volume than in Figure 3 Si-rich phase is depicted in orange, and the Sn-rich phase in green, with clear interdiffusion in the transition zone ~25-30 µm wide in the center.*

This brief study has illustrated the complementary ability for both XRM and 3D FIB-SEM to study a functional material with hierarchical structures of interest, thereby aiding 'materials by design' challenges. Future work on these materials systems will point to performing time dependent (4D) XRM analysis on the same sample volume with the ZEISS Xradia Versa as a function of heat treatment, followed by navigation to regions of interest with the ZEISS Crossbeam 540. Such microstructure evolution studies via XRM prior to high-resolution FIB-SEM analysis are representative of a large and growing field of materials research activities, including mechanical loading [Patterson, et al], corrosion [Burnett, et al.], electrochemical device performance [Eastwood, et al] *(in operando)* and many more.

References:

Eastwood, D. S., et al (2014). Batteries: Lithiation-Induced Dilation Mapping in a Lithium-Ion Battery Electrode by 3D X-Ray Microscopy and Digital Volume Correlation. Advanced Energy Materials, 4(4), doi:10.1002/aenm.201470016

Burnett, T. L., et al. (2014). Correlative Tomography. Scientific Reports, 4. doi:10.1038/srep04711

Patterson, B. M., Henderson, K., & Smith, Z. (2013). Measure of morphological and performance properties in polymeric silicone foams by X-ray tomography. Journal of Materials Science, 48(5), 1986–1996. doi:10.1007/s10853-012-6965-2

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