

Three-dimensional Analysis of Materials at Multiple Length Scales

Joshua Sugar¹, Norm Bartelt¹, David Robinson¹, Bonnie Antoun¹, Chris San Marchi¹, Thale Smith¹, Coleman Alleman¹, Hanping Ding², Dong Ding² and Suzy Vitale³

¹Sandia National Laboratories, Livermore, California, United States, ²Idaho National Laboratory, Idaho Falls, Idaho, United States, ³Carnegie Institution for Science, Washington DC, District of Columbia, United States

One challenge of any microscopic technique is that the data generated is generally a two-dimensional slice or projection of a three-dimensional object. Typically, that two-dimensional image still provides a wealth of information about a materials' or organism's microstructure, but there are always limitations when trying to understand a specimen's true three-dimensional nature. This motivates the need for many of the 3D materials characterization techniques and tools available today. A number of techniques are available that span decades in both voxel size/resolution and analyzed material volume [1]. I will discuss the use of electron microscopes for analyzing materials in three dimensions. In particular, I will show how electron tomography in the transmission electron microscope (TEM) is useful for analyzing the size and distribution of nanoscale He bubbles (~2 - 4 nm in diameter) in Pd that formed by tritium decay [2]. Figure 1 shows an example of 2D and 3D data collected on these nanoscale bubbles. In addition, I will show examples of serial sectioning in the FIB/SEM instrument and its use for simultaneously obtaining three-dimensional morphological, compositional, and crystallographic orientation information with imaging, EDS, and EBSD. Examples in fuel cell device stacks [3] and laser-deposited stainless steel [4] will be presented that demonstrate the use of three-dimensional analysis.

As with any technique that utilizes a FIB for sample preparation and/or analysis, it is necessary to understand artifacts that could appear in the sample as a result of the ion species-sample interaction. It is known that the FIB can implant the ion into the specimen, but this can also cause changes in phase and crystallographic orientation of a sample [5-7]. Therefore, the use of the FIB for analyzing large volumes requires consideration of preparation-induced artifacts in the analysis. Figure 2 shows an example of a Ga FIB causing a phase change in austenitic stainless steels. The challenges associated with these types of artifacts as they apply to nanoscale He bubbles, fuel cell stacks, and stainless steels will also be discussed in the context of 3D materials analysis. With proper application of the FIB for either serial sectioning or TEM sample preparation, artifact-free 3D materials analysis at a range of length scales is accessible to answer questions when two-dimensional images are insufficient. [8]

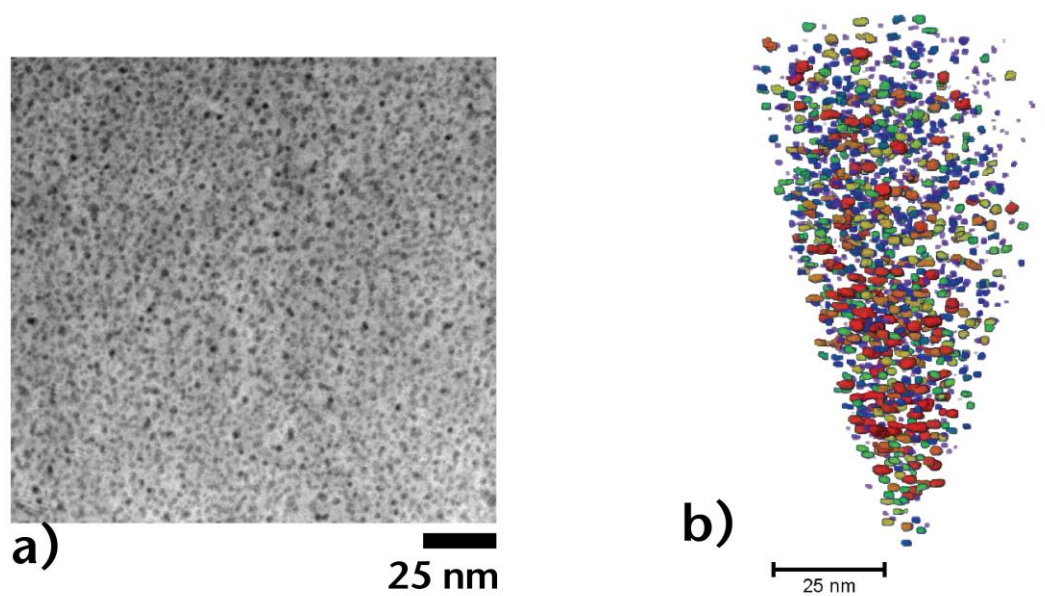


Figure 1. a) HAADF STEM image of He bubbles in a PdNi alloy that result from tritium decay after 3.7 years. The bubbles are approximately 2 – 4 nm in diameter. It is difficult to determine their distribution in space with a two-dimensional image, but electron tomography can be used to generate a 3D volume reconstruction of the bubble distribution as shown in b). Quantification of their size and nearest neighbor distance is only possible with volumetric data.

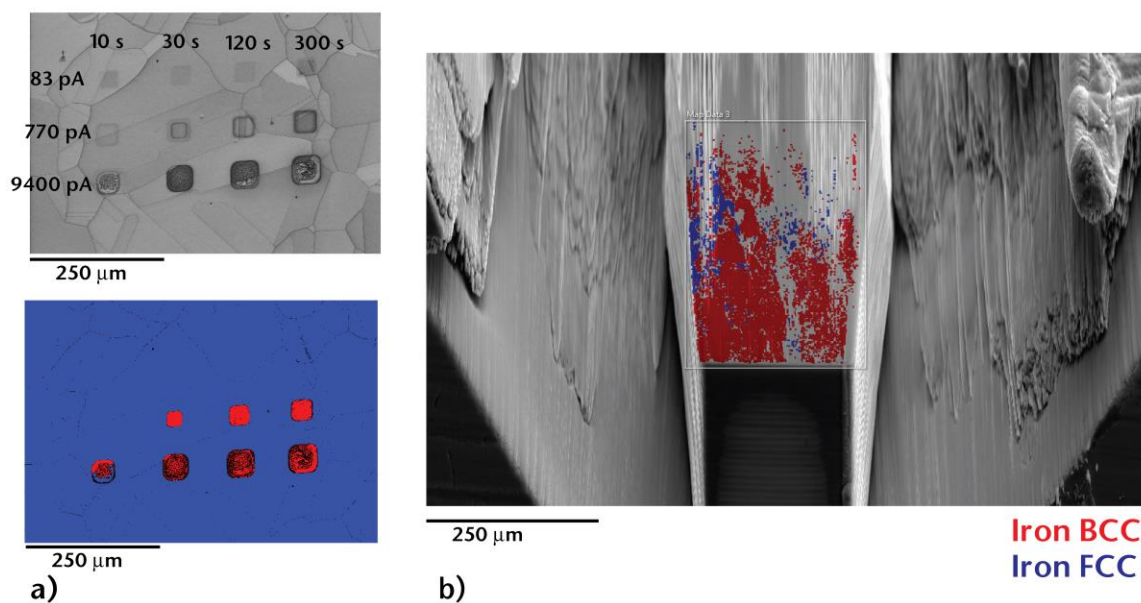


Figure 2. (a) The top image is a band contrast image where Ga implantation parameters are shown in the image for small squares patterned with a FIB/SEM. The bottom image shows how at some Ga doses, stainless steel can transform from the austenitic fcc phase, to the ferritic bcc phase. The image in b) is a single slice from a 3D EBSD serial sectioning experiment on this stainless steel. The slice is almost completely transformed to the bcc phase as a result of the ion-beam polishing. These artifacts must be minimized to obtain 3D data that truly represents the materials microstructure.

References

- [1] T. Burnett, B. Winiarski, R. Kelly, X. Zhong, I. Boona, D. McComb, *Microscopy Today*, 24 (2016) 32-39.
- [2] N.R. Catarineu, N.C. Bartelt, J.D. Sugar, S.M. Vitale, K.L. Shanahan, D.B. Robinson, *The Journal of Physical Chemistry C*, 123 (2019) 19142-19152.
- [3] Y. Gao, L. Neal, D. Ding, W. Wu, C. Baroi, A.M. Gaffney, F. Li, *ACS Catalysis*, 9 (2019) 8592-8621.
- [4] T.R. Smith, J.D. Sugar, C. San Marchi, J.M. Schoenung, *Acta Materialia*, 164 (2019) 728-740.
- [5] R.P. Babu, S. Irukuvarghula, A. Harte, M. Preuss, *Acta Mater*, 120 (2016) 391-402.
- [6] A. Basa, C. Thaulow, A. Barnoush, *Metall Mater Trans A*, 45a (2014) 1189-1198.
- [7] J.R. Michael, *Microsc Microanal*, 17 (2011) 386-397.
- [8] Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525. This paper describes objective technical results and analysis. Any subjective views or opinions that might be expressed in the paper do not necessarily represent the views of the U.S. Department of Energy or the United States Government.