

SEM-Based Electron Tomography of Turfs Comprised of Lineal Structures

O.M. Fakron, D.P. Field

School of Mech. and Matls. Eng, Washington State University, Pullman, Washington 99163-2920

Recent developments in nanostructures have brought to light exceptional electromagnetic, thermal and optical properties of a class of nanostructures formed of disordered, intertwined, one-dimensional, structural units (nanowires, nanobelts, nanotubes) of carbon nanotubes, and GaN and SiO₂ nano-wires and springs, among others. Such disordered assemblies are named *turfs* [1]. Applications include thermal switches, flat panel displays, hard discs drives, solar collectors, and chemical and biological sensors. Complete topological characterization of such structures requires either stochastic and stereological approaches [2] or three-dimensional imaging. Dual beam, SEM/FIB instruments have made remarkable progress in recent years and have enabled three-dimensional imaging of a multitude of structures. Because of the small scale of the nano-structured turfs and their relatively fragile nature, it is not possible to image these features using standard three-dimensional, parallel serial sectioning techniques. Even if the structures were somehow impregnated first with a polymer based space-filler, the morphology of the structures would be altered by the impregnation and subsequent ion beam milling required for imaging. Finally, simple imaging of stereo pairs, as is often done in the SEM to recreate the surface topography of a bulk specimen, cannot be performed with reasonable results on such specimens because the technique necessarily assumes a fully connected surface structure. A typical secondary electron image of a carbon nanotube turf obtained using a field emission SEM is shown in Figure 1.

In recent years, electron tomography has enabled the three-dimensional characterization of features observed in TEM foils by tilting the specimens about an axis and reconstructing the 3-D images from a reduced tilt series. In electron tomography there are three steps in obtaining three-dimensional images. Firstly, data acquisition in obtaining a tilt series of images is required. The tilt series might consist of single axis tilt, dual axis tilt, or conical tilt. The second step is alignment and reconstruction of the images to construct the tomogram. Most alignment methods are cross-correlation using fiducial markers. There are also many methods for constructing the projected images such as the weighted back projection (WBP) method, the algebraic reconstruction technique (ART), and the simultaneous iterative reconstruction technique (SIRT). The final requirement is image enhancement that consists of two steps; de-noising and image filtering. After each of these is performed the three dimensional model can be visualized [2].

In the present research SE images of turf-type structures obtained in the SEM have been used for tomographic reconstruction. Because of the high tilt precision required for nano-turf structures, SE images were taken at different tilting angles from fine steel wool specimens. (The steel wool shows images remarkably similar to those obtained from CNT turfs, for example.) To obtain three dimensional images for steel wool by using SEM, a series of tilt images was taken every 1 degree over a range of 70 degrees total tilt angle. This essentially obtains a tilt series of projected images, similar in nature to what is obtained in electron tomography or X-ray computed tomography. Three dimensional images are reconstructed from the tilt series created from these two dimensional images

using Matlab-based techniques. It is shown that images similar in nature to those obtained by TEM-based electron tomography can be obtained using this technique.

References

- [1] McCarter C M, Richards R F, Mesarovic S Dj, Richards C D, Bahr D F, McClain D, Jiao J, J. *Mat. Sci.* 41 (2006) 7872-7878
- [2] H Malik, KJ Stephenson, DF Bahr, and DP Field, *J. Mat. Sci.* 46 (2011) (*doi: 10.1007/s10853-010-5192-y*)
- [3] M. Barcena, M. Radermacher, J. Bar, G. Kopperschlager, and T. Ruiz, *Microsc Microanal* 10, Suppl 2 (2004) 1506-1507
- [4] Paul A. Midgley and Rafal E. Dunin-Borkowski *nature materials* 8 (2009) 271-280.
- [5] The authors acknowledge financial support of the CMMI division of the US National Science Foundation under grant number 0856436.

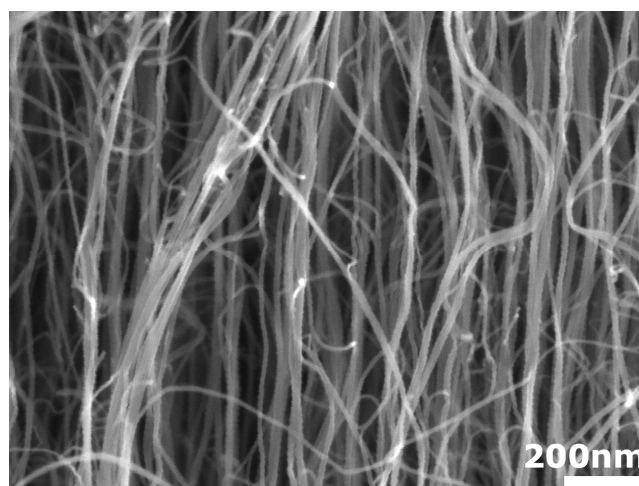


Figure 1 – Typical SE image of carbon nanotube turf structure.

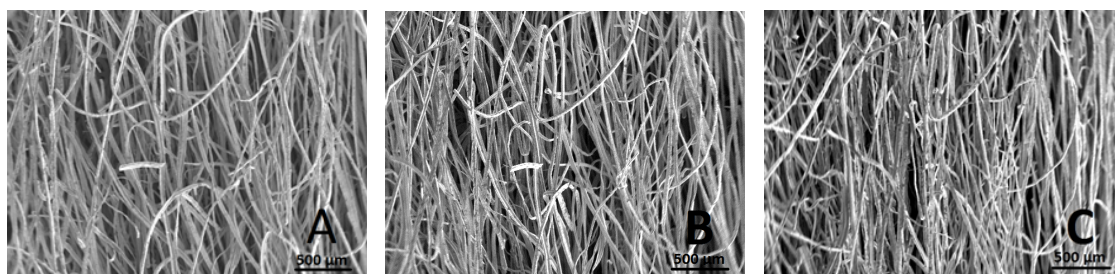


Figure 2 –Steel wool sample at (A) minus10° tilt angle, (B) minus20° tilt angle and (C) 50° tilt angle.