doi:10.1017/S1431927614011301
Microsc. Microanal. 20 (Suppl 3), 2014

The Role of Microscopy in Understanding Ceramic Processing

C. Barry Carter[1-3]
[1] Dept of Chemical & Biomolecular Engng, U. of Connecticut, 191 Auditorium Road, Storrs, CT 06269
[2] Dept of Materials Science & Engng, U. of Connecticut, 97 North Eagleville Road, Storrs, CT 06269
[3] Institute of Materials Science, U. of Connecticut, 97 North Eagleville Road, Storrs, CT 06269

Although it goes without saying, we'll say it: microscopy is essential if ceramic processing is to be understood and processes designed for reproducibility. It is essential that the materials involved, from the precursors to the completed product, should be well understood. In this talk, I will illustrate this truism with illustrations from our work on nanoparticles and fibers and the processing of the same. I will also suggest directions of research that will become more important in the future and that take advantage of the new developments in microscopes and especially in specimen holders for in-situ studies.

The image shown in Figure 1 serves as a cautionary note for all electron microscopists—the electron beam may change the specimen. In this case, changes in the shapes of the crystallites are seen even when the images are formed on successive scans. (The TEM used here was a Titan ChemiSTEM operating at 200kV.) Actually, the situation is much worse than that—the oxide was actually amorphous before the first scan. So, just scanning the beam across the sample once changed the structure. Even in ceramic materials, it may be necessary to use low-dose techniques but how does the microscopists know when this will be the case?

Ceramic fibers have long been used for a wide range of technological applications [1] ranging from thermal insulation (fiber glass and the space-shuttle tiles), to optical transmission (fiber optics) to strengthening composites (fiber reinforcement). The electro-spinning process has long been used for producing polymers but also has enormous potential for ceramics and organic-inorganic composites. The use of the FIB combined with SEM and TEM provides clear insights into how the processing affects the materials and suggests how these novel structures might be manipulated for novel, affordable applications. SEM images and a FIB image are shown in Figure 2.

The in-situ study of reactions between oxides, which are ubiquitous in ceramics processing, has long been difficult because of the environment in the TEM especially; this problem was somewhat overcome for the SEM with the development of commercial instruments using novel electron detectors. Now new specimen holders that allow some control of the environment around the specimen are becoming widely available for the TEM. The type of study that is becoming possible is illustrated by Figure 3, which shows nanoparticles of MgO reacting with a thin film of Al_2O_3. In this case, the reaction was carried out *ex situ,* but this approach is now being extended to in-situ studies involving oxides that would reduce in the conventional TEM environment.

References:

[1]. Carter, C.B. and Norton, M.G., 2013,Ceramic Materials; A textbook for Materials Science, 2nd Ed. Springer; NY.
[2] The author thanks Neal Shinn and Sandia National Laboratory where CBC is a CINT Distinguished Affiliate Scientist, and S.K. Sundaram and James E. Martinez for the invitation to present this work. Personal thanks to my former group members (who are listed on the web site www.CBarryCarter.com) and his current collaborators, especially Paul Kotula, Joe Michael, Yang Liu, Katie Jungjohann, Aravind Suresh, Chris Cornelius, Joysurya Basu, Jonathan Winterstein,

Lichun Zhang, Radenka Maric, Justin Roller and Matt Janish. Special thanks go to Grant Norton, co-author of the textbook, and Paul Hlava, our minerals guru.

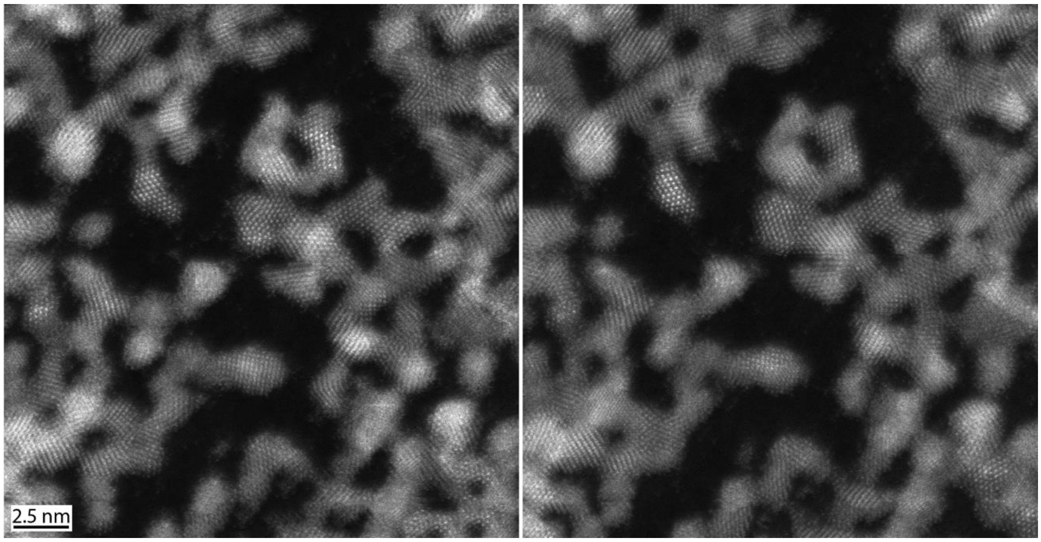
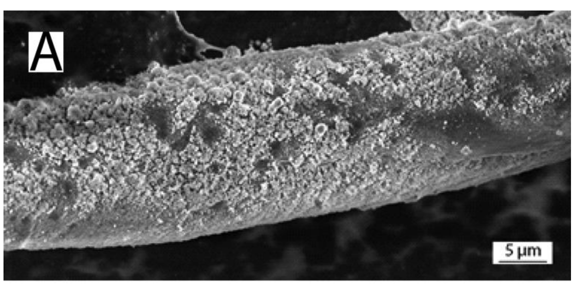


Figure 1. TEM images of particles of IrO_2 on successive passes of the scanning electron beam under HAADF imaging conditions.



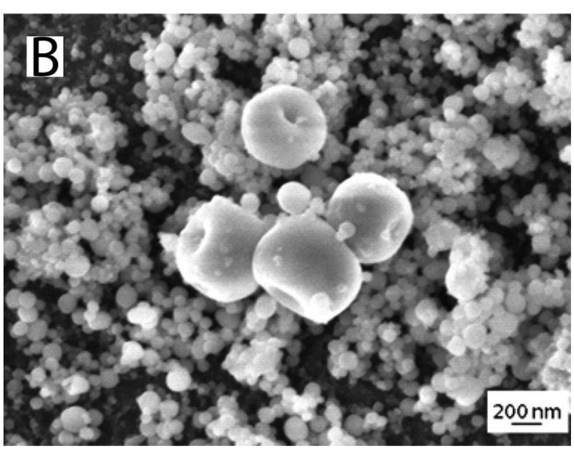


Figure 2 (A,B and above). Images of an electrospun TiO2 fiber. A and B show the fiber and doughnuts/cheerios deposited on it; Above is a partially FIBbed fiber showing the porosity.

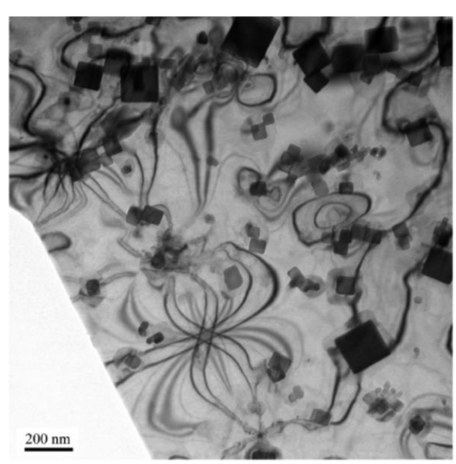


Figure 3 (right). Observing solid-state reactions between MgO nanoparticles and a thin Al_2O_3 substrate.