

## Transmission Microscopy: Beginning Automation

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The TEM [1] has been used for studying changes that occur in materials since it was first developed. Heating and straining holders were quickly developed and radiation damage (sometimes a misnomer) could be studied using any holder, of course! Today's TEMs allow us to study these processes with atomic resolution; we can actually determine locations with sub-Å lateral resolution. These advances are aided by new cameras that allow us to record processes in milliseconds (or innovative 'illumination' systems that allow us to record changes that may occur in nanoseconds—the DTEM). In an ETEM, or using an 'environmental' holder in a CTEM or STEM, we can now modify and control the environment around the specimen so that it is not necessarily UHV but could be an oxidizing gas or even a liquid [2]. Not only has resolution improved due to the achievement of Cs correction but CEOS and Nion have joined JEOL and the late FEI with acronyms for company names! Future TEMs may have much more space around the specimen when Cs correction becomes the norm, as it has long been for VLM.

Diffraction was the poor relative of HRTEM, being either SAD or CBED, but now we can use TKD to map out both orientations and phases and then use a MEMS or NEMS holder to control mechanical deformation of the thin film, measuring the actual applied stress, while mapping orientations and changing them—causing grain growth, cracking and potentially doing this in a controlled environment. TKD is an excellent example of why you should know what the letters represent so you'll never use TEBS! TKD essentially uses SAD but happily is not limited by the need to have a SAD aperture. CBED from sub-unit cell volumes, was explored early on by Cowley. One challenge that is now being addressed is that spatial resolution has been limited in the direction of the beam (so 2D materials are very popular); with FEGs providing the electron version of the laser (a laser is ... without capitalization!), the confocal approach used in VLM is now being applied to TEM. Cryo-TEM was recognized as the method of the year in 2015. In 2017 the Nobel Prize was awarded to key contributors for the development of cryo-TEM. Today, automation is playing a growing part in this analysis with large areas of microtomed specimens being imaged automatically—all images being in focus—and the results shipped to external researchers to analyze. A direct-electron-detector camera can produce a 15-minute video that requires 3 terabytes of memory, which no human will have the time to look at (since several such videos may be recorded in one session)—so it must be analyzed by other intelligent means. The question might be asked then: will we still need trained TEM users? The answer is, of course, a resounding 'yes' or 'no'. An example of teaching in progress is shown in Figure 2 of the companion paper.

Figure 1 shows an image of a specimen of GST that has been examined in a 300kV CTEM while heating the specimen at 800°C [3]. Two very different grain sizes can be observed; both regions were heated for the same time at the same temperature. The difference is that the region with the smaller grain size was observed during heating while the region with larger grains was only imaged after heating. This in-situ experiment thus emphasizes the importance of carrying out in-situ experiments while not looking at the specimen. The temptation to do otherwise is quite strong! Figure 2 illustrates two pairs of

sequential images: namely, conventional CTEM images from quartz [4] and STEM images from 2D the layer material, WS<sub>2</sub> [5]. In both examples, the changes we see are due to the electron beam [6].

#### References:

[1] Williams DB, Carter CB (2009) *Transmission Electron Microscopy: A Textbook for Materials Science*, 2<sup>nd</sup> Ed. Springer, NY.

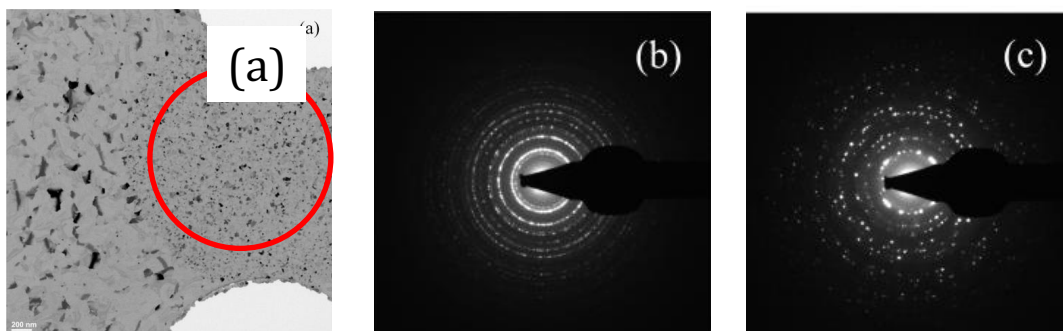
[2] Carter CB, Williams DB (2016) *Transmission Electron Microscopy: Diffraction, Imaging, and Spectrometry*, Chapter 2, 2<sup>nd</sup> Ed. Springer, NY.

[3] Tripathi S, Janish MT, Noor N, Jungjohann KL, Pete D, Kotula PG, Silva H, Carter CB (2018) presented at Fall MRS.

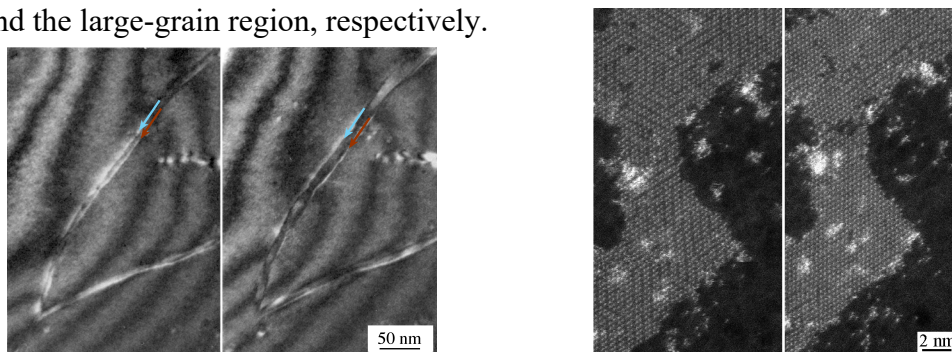
[4] Carter CB, Kohlstedt DL, *Phys Chem Minerals* 7 (1981), p. 110.

[5] Tripathi S, Janish MT, Mook WM, Jungjohann KL, Dongare AM, Doble A, Carter CB (2018) presented at Fall MRS.

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**Figure 1.** a) CTEM image of a PCM that has been heated to 800°C; b,c) SAD patterns from the small-grain region and the large-grain region, respectively.



**Figure 2.** Left) 2 sequential CTEM images of quartz; Right) 2 sequential STEM images of WS<sub>2</sub>.