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Studying Materials in-situ with the Dynamic Transmission Electron Microscope (DTEM)

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In response to a need to be able to observe dynamic phenomena in materials systems with both high spatial (~1nm or better) and high temporal (~1μs or faster) resolution, a dynamic transmission electron microscope (DTEM) has been developed at Lawrence Livermore National Laboratory (LLNL). The high temporal resolution is achieved in the DTEM by using a short pulse laser (~1μs or faster) to create the pulse of electrons through photo-emission (here the duration of the electron pulse is approximately the same as the duration of the laser pulse). This pulse of electrons is propagated down the microscope column in the same way as in a conventional high-resolution TEM. The only difference is that the spatial resolution is limited by the electron-electron interactions in the pulse (a typical 10ns pulse contains ~10^8 electrons) – the shortest pulses suffer the greatest amount of spatial resolution degradation as the electron density is highest [1]. To synchronize this pulse of electrons with a particular dynamic event, a second laser is used to "drive" the sample a defined time interval prior to the arrival of the electron pulse. The important aspect of this dynamic DTEM modification is that one pulse of electrons is used to form the whole image, allowing irreversible transitions and cumulative phenomena such as nucleation and growth, to be studied directly in the microscope (Figure 1).

As the DTEM is based on a standard 200kV JEOL microscope column, advanced designs for in-situ stages can be easily incorporated into the microscope to obtain dynamic images under both ambient gas and fluid environments. To investigate the effect of surface chemistry on phase transformations and to study catalytic reactions, an in-situ gas stage has been developed in collaboration with Fischione Instruments (Figure 2). By using the DTEM laser to heat the sample, rapid heating can be obtained to high temperatures in localized areas allowing multiple regions of the same specimen to be studied and reacted independently. In the case of the fluid stage, an added benefit of the DTEM is that the effect of Brownian motion is negated by the fast pulse of electrons and high resolution images can be obtained from samples free to move in the fluid. In a development with Hummingbird, both static and flow fluid stages are being developed for use in the DTEM (Figure 3). These stages are being used to study biomineralization processes and also to study live hydrated biological samples (this work is being accompanied by the purchase and installation of an aberration corrected DTEM at UC-Davis).

In this presentation, a summary of the development of in-situ gas and fluid stages for both the existing DTEM at LLNL and the new DTEM at UC-Davis will be described. Particular attention

will be paid to the application of the fluid stage to study biological specimens. In addition, the potential improvements in the spatial and temporal resolution for in-situ studies that will be afforded by the new DTEM will be discussed, along with the correlation of the DTEM results with similar studies in conventional and aberration corrected high resolution TEM/STEM [2].

[1] B. W. Reed, M. R. Armstrong, N. D. Browning, G. H. Campbell, J. E. Evans, T. B. LaGrange, D. J. Masiel, *Microscopy and Microanalysis* **15**, 272-281 (2009)

[2] Development of the DTEM, the specification/incorporation of the in-situ gas stage, and the biomineralization study were performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory and supported by the Office of Science, Office of Basic Energy Sciences, Division of Materials Science and Engineering under contract DE-AC52-07NA27344. The development of the in-situ fluid holder at UC-Davis was supported by NIH grant number RR025032-01.

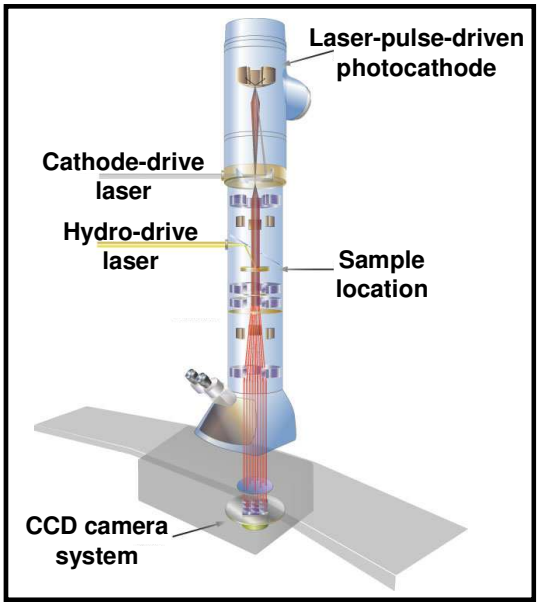

Figure 1: The DTEM produces a short pulse of electrons by illuminating a Ta cathode with a UV laser of pulse duration ~10ns. The transition/reaction in the sample is initiated by the specimen drive laser and the time resolution is obtained by controlling the delay between the initial drive pulse and the arrival of the electron pulse.

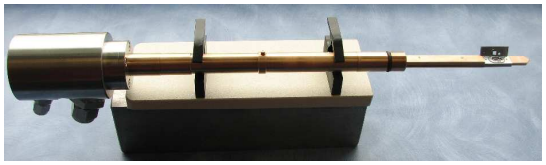

Figure 2: In-situ gas stage developed for the DTEM.

Figure 3: In-situ continuous flow fluid stage.

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