#127 Nanosecond Imaging in the Dynamic TEM Reveals Unquenchable Transient Microstructure

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Until recently, materials science characterization techniques have lacked the ability to directly observe sub-micron, dynamic materials processes that occur faster than the microsecond scale. Due to electron current density limitations and slow CCD readout times, Transmission Electron Microscopy (TEM) techniques that reveal nanoscale phase and morphology through real-space imaging and structure through diffraction have been limited to video-rate ($\sim10^{-3}$ s) time-resolution observation. The use of pump-probe methods to investigate the dynamics of chemical reactions and phase transitions in materials has typically utilized laser induced reactions/transitions coupled with either laser or X-ray interrogation methods. However, electron probe methods have the potential to provide higher sensitivity and resolution by utilizing bright electron sources, a controllable the electron beam, and the stronger interaction of electrons with matter [1]. High temporal resolution is currently attainable by ultrafast electron diffraction (UED) [2] and the potential for direct high spatial resolution imaging has recently been demonstrated by the Dynamic Transmission Electron Microscope (DTEM) [3, 4].

Based on a novel design from TU Berlin[5], and developing it for increased functionality and spatial resolution, we have constructed the modern Dynamic TEM (DTEM) to explore this realm of short times in nano- space. Figure 1 illustrates the DTEM concept employed at Lawrence Livermore National Laboratory (LLNL) on a JEOL 2000FX TEM. Using laser induced photoemitted electrons in a single, 10ns long cluster of $1x10^7$ electrons, we are able to achieve nanosecond *in situ* electron microscopy with resolution better than 20nm (Figure 2). Characterization of fast materials science processes such as Martensitic transformations, solid-to-liquid phase transitions, nanowire growth, and grain boundary and dislocation motion that cannot be directly observed by other modern techniques is now possible.

In this study of Reactive Multilayer Foils (RMLF) of Al and Ni polycrystalline thin film bilayers we have established that the DTEM is proficient for nanosecond science in a TEM with both diffraction and imaging capabilities. The foils undergo an exothermic self-propagating reaction as the bilayers mix to form intermetallics. This reaction front travels at a velocity of ~10m/s and is observed directly in the DTEM for the complete progression of the material transition, which is partially illustrated in Figure 3 [6]. By studying the transient states of this dynamic material to understand the mechanisms that govern the rate of heat generation and transport, we can understand more about atomic diffusion between thin films and phase boundary motion for optimized engineering applications. A comparative study of quenched foils and post-mortem TEM showed that the transient morphology observed in the DTEM images could not be found, making the DTEM is essential for fast high-resolution materials characterization. The latest results from the DTEM will be presented highlighting the current capabilities for both temporal and spatial resolution. Additionally, a future model column for high temporal and spatial resolution studies in the DTEM will be described [7].

References

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[7] This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory supported by Office of Science, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering of the U.S. Department of Energy under Contract DE-AC52-07NA27344. UCRL-ABST-235696

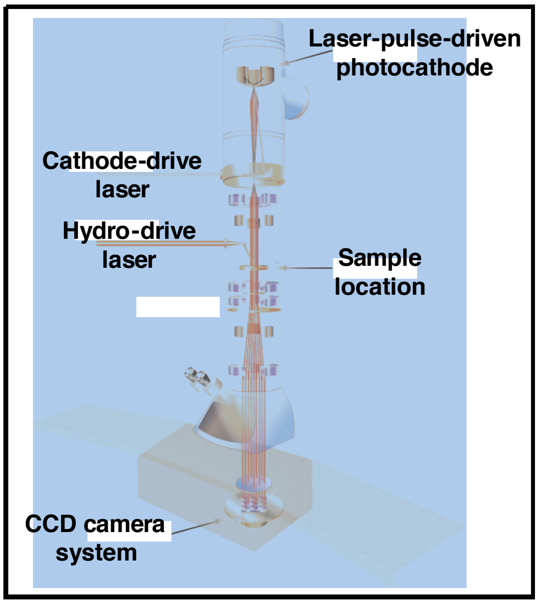
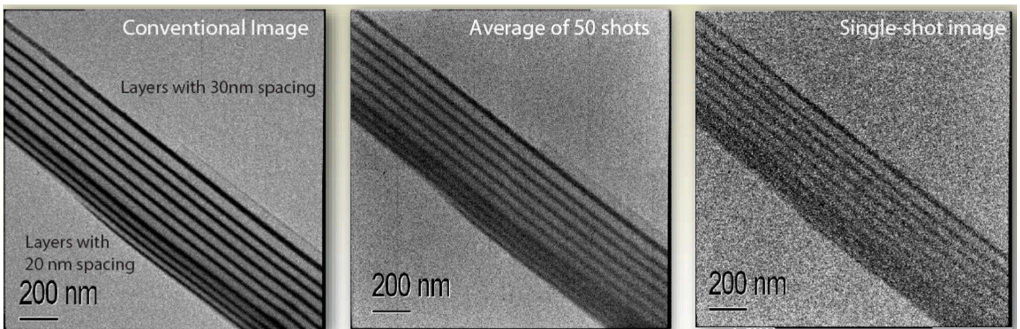


Figure 1: The LLNL DTEM produces a short pulse of electrons by illuminating a Ta cathode with a UV laser of pulse duration ~30ns. The transition/reaction in the sample is initiated by the specimen drive laser and the time resolution is obtained by controlling the delay between the initial drive pulse and the arrival of the electron pulse.

Figure 2(below)**:** The left image is a conventional TEM image of a Au/C multilayer foil. The center image is an average of 50 DTEM pulses and the right image is a single 30ns shot image of the same area showing spacings of ~20nm can be resolved.



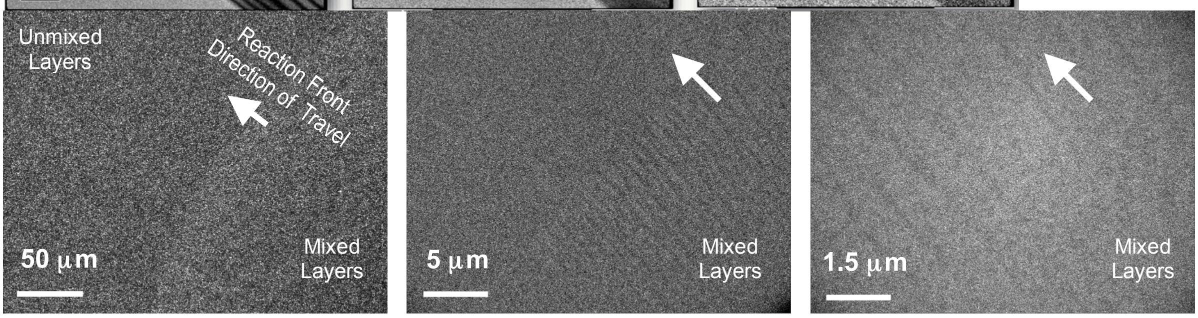


Figure 3: The exothermic phase formation zone is imaged in a plan-view Al/Ni multilayer foil with a single electron pulse. These "snap-shot" micrographs show the reaction front detail at increasing magnifications. They reveal a transient dendritic-type structures that cannot be found in post-mortem TEM studies of quenched foils.