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Quarterly progress report for Q1 FY06 for Complex Transient Events in Materials Studied Using Ultrafast Electron Probes and Terascale Simulation (FWP SCW0289)

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Disclaimer

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Complex Transient Events in Materials Studied Using Ultrafast Electron Probes and Terascale Simulation (FWP SCW0289)

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Executive Summary

This quarter (Q1 FY06) marked the first time that the LLNL dynamic transmission electron microscope (DTEM) configuration had advanced to the point whereby it was possible to conduct *in-situ* experiments on specimens. DTEM improvements continue to progress at a rapid pace. We summarize important achievements in the following list:

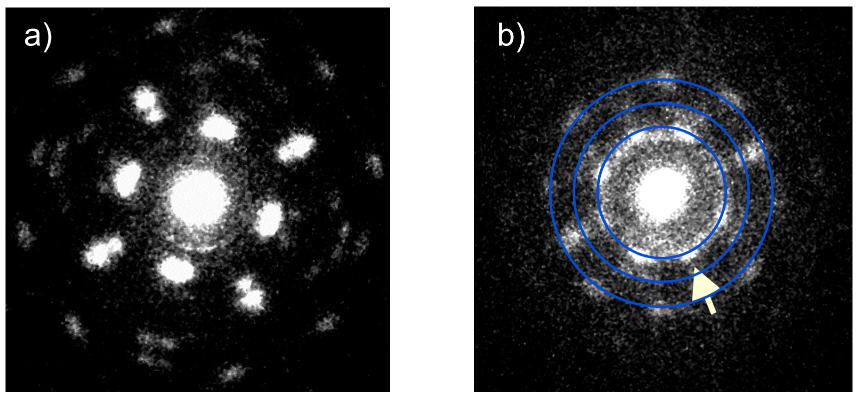
1. Instrument performance and design improvements:
   - Reproducibly achieving >$1x10^7$ e^- per pulse. Adjustments in the cathode laser system design led to an improved quantum efficiency and electron yield per pulse. The current number of electrons in the pulse is sufficient for acquiring high quality, single-shot electron diffraction patterns.
   - Implementation of computer interface and Labview® programs for cathode and specimen drive alignment and cathode and pump laser trigger and delay settings. These controls provide a user friendly interface and ease in the experimental setup and implementation.
   - Cathode test chamber (offline test apparatus to asses photocathode design and laser induced photoemission) construction has been completed.

2. Notable instrument features brought into service:
   - Drive laser system was enhanced to improve beam shape and uniformity and to include continuous laser energy monitoring. The drive laser spot size on the specimen was also reduced from 70 µm x 110 µm to 50 µm x 75 µm.
   - New phosphor coated face plate manufactured by TVIPS was installed. The sensitivity and signal noise ratio improved by factor 2 (sensitivity ~110 CCD counts/e^- and signal to noise ratio ~5).

3. Experimental Progress:
   - **First time-resolved experiment**: observation of the α (hcp) to β (bcc) phase transition in pure Ti films via single shot electron diffraction. Results of this experiment were published in the MRS Fall 2005 proceedings and are under review for article in the FEMMS proceedings, which will to be published in Journal of Material Science. See Experimental Results section for more on this experiment.
   - **First single-shot image captured on the LLNL DTEM** (image of diffraction grating replica with dispersed latex spheres): It is believed that this image holds the world's record for the highest combined spatial and temporal resolution in single shot image. The image was generated using a 1.5 ns-long electron pulse and has better than 20 nm spatial resolution.

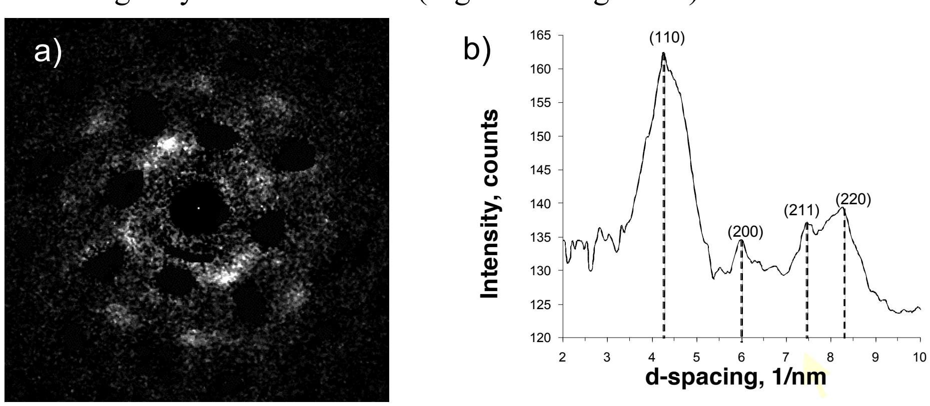
Experimental Results

The transient events of the α-β martensitic transformation in nanocrystalline Ti films were explored via single-shot electron diffraction patterns with 1.5 ns temporal resolution. The transient phenomena of the martensitic transformations in nanocrystalline Ti is ideally suited for study in the DTEM, with their rapid nucleation, characteristic interface velocities (~1 km/s), and significant irreversible microstructural changes. Free-standing 40-nm-thick Ti films were laser-heated at a rate of ~10^{10} K/s to a temperature above the 1155 K transition point, then probed at various time intervals with a 1.5-ns-long, intense electron pulses. Diffraction patterns show an almost complete transition to the β phase within 500 ns (see Figure 1).



<u>Figure 1.</u> *Single-shot diffraction patterns recorded with 1.5 ns time resolution, a) HCP structure acquired at room temperature before laser treatment, b) The superimposed blue rings index the pattern as the BCC structure. This diffraction pattern was acquired 500 ns after the 15 μJ pump laser pulse. Arrow indicates the appearance of new spots that did not exist in the HCP pattern.*

A clearer indication of the change in phase was revealed by taking the difference pattern (Figure 2a) of the two diffraction patterns in Figure 1 and plotting the radial averaged intensity of the difference pattern with reciprocal lattice units (d-spacing) in Fig. 2b. The peaks present in Figure 2b show that it is indeed BCC. There was approximately 75 ± 5% BCC, β-phase present in Figure 1b, which was calculated first by fitting the peaks in Figure 2b with a Lorentzian profile function and determining their integrated intensities that were then compared with the integrated intensities of difference pattern containing only HCP reflections (Figure 1b–Figure 2a).



<u>Figure 2.</u> *a) Difference pattern between Figures 1a and b, showing residual BCC reflections, b) Radial intensity vs. d-spacing for the rotationally averaged pattern in Figure 2a.*

Post-mortem analysis (after the sample is allowed to cool) shows a reversion to the α phase coupled with substantial grain growth, lath formation, and texture modification (see Figure 3). The cooled material also shows a complete lack of apparent dislocations, suggesting the possible importance of a "massive" short-range diffusion transformation mechanism. Observing the interfaces through single-shot imaging during the transformation would certainly help to divulge the mechanism, which is planned for future investigations. In general, the near-term research plans are focused on the measurement of the transformation rate as a function of temperature and laser heating rate and the development of novel isothermal transformation and continuous cooling curves for nanocrystalline Ti with nanosecond temporal resolution. These curves will not only serve to increase fundamental scientific understanding of this transition in pure Ti but will also clearly demonstrate the unprecedented capabilities of the instrument.

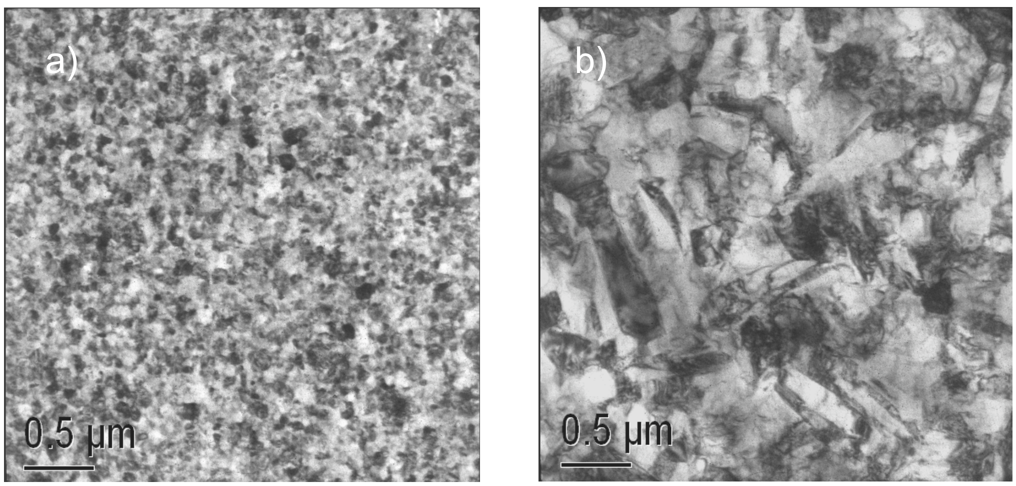


Figure 3. *a) As-deposited Ti film microstructure, b) After laser treatment.*