

**P.19.01.1***Acta Cryst.* (2005). A61, C457**Generation and Characterization of Ultrashort Electron Pulses**Christoph T. Hebeisen, Jason R. Dwyer, Robert E. Jordan, Maher Harb, Ralph Ernstorfer, R.J. Dwayne Miller, *Departments of Physics and Chemistry, University of Toronto.* E-mail: heby@lphys.chem.utoronto.ca

Femtosecond Electron Diffraction (FED) has been developed to directly observe structural transitions on the atomic scale in real time. The duration of the electron pulse dominates the time resolution of any current FED setup. Velocity mismatch between the electron probe and the laser pump pulse requires the use of transmission mode electron diffraction to achieve femtosecond temporal resolution. Hence, thin film samples with a thickness on the order of tens of nanometers have to be used. Intense heating of the sample due to the pump laser combined with the extreme surface-to-volume ratio makes most transitions non-reversible. A high number of electrons per pulse is thus required to achieve high signal-to-noise in one or few shots.

We have developed an electron gun that provides high flux electron pulses at pulse durations down to 250 fs. This has been achieved by minimizing the space-charge induced broadening of the electron pulses. Traditional methods for characterization of high flux electron pulses like streak cameras fail below the picosecond range. We have proposed the use of the ponderomotive potential [1] of an intense laser field to sample the temporal profile of the electron pulse by selectively scattering parts of the electron beam. This method and other approaches providing a direct cross-correlation between light and electron pulses hold promise of measuring pulses as short as 100 fs.

[1] Siwick B.J., Green A.A., Hebeisen C.T., Miller R.J.D., *Characterization of Ultrashort Electron Pulses by Electron-Laser Pulse Cross-Correlation*, in press.

**Keywords:** ultrafast electron diffraction, electron pulse propagation dynamics, electron pulse characterization

**P.19.01.2***Acta Cryst.* (2005). A61, C457**Ultrafast Melting in Metals Probed with Femtosecond Electron Diffraction**Jason R. Dwyer, Robert E. Jordan, Christoph T. Hebeisen, Maher Harb, Ralph Ernstorfer, R.J. Dwayne Miller, *Departments of Chemistry and Physics, University of Toronto, Toronto, Canada.* E-mail: jdwyer@LPhys.chem.utoronto.ca

Femtosecond (fs) lasers are an ideal tool to excite materials on timescales even shorter than vibrational periods, typically  $\sim 100$  fs. The ability to resolve all of the resulting structural dynamics depends on having a technique with fs temporal resolution and capable of providing high structural resolution. Femtosecond electron diffraction satisfies both criteria and offers an unprecedented view of the fastest possible structural dynamics [1].

By using a fs laser, one is able to very quickly deposit energy into a material—in these experiments gold and nickel. This leads to superheating of the metal and thereby permits the study of the familiar phenomenon of melting, only in this case the process is strongly driven. Even under these conditions, however, the material properties mediate the material response. In our first work [1], aluminum melted in 3.5 picoseconds (ps); under equivalent conditions, gold melts in 12 ps and nickel more quickly than gold. The difference in timescales is a consequence of a material parameter—the electron-phonon coupling constant—that differs by an order of magnitude between the gold and nickel and determines how quickly the laser energy absorbed by the electrons is transferred to nuclear motion. The observed structural changes, however, are the same for the two metals and thus allow for a generalized description of the melting mechanism.

[1] Siwick B.J., et al., *Science*, 2003, **302**, 1382.

**Keywords:** ultrafast electron diffraction, ultrafast melting, time-resolved structure

**P.19.02.1***Acta Cryst.* (2005). A61, C457**3-D Structure Determination of CaMoO<sub>4</sub> using HVEM**Jin-Gyu Kim<sup>a</sup>, Youn-Joong Kim<sup>a</sup>, Jong-Pil Kim<sup>b</sup>, <sup>a</sup>*Electron Microscopy Team, Korea Basic Science Institute, Yuseong-Ku, Daejeon.* <sup>b</sup>*Busan Branch, Korea Basic Science Institute, Kumjung-ku, Busan, Korea.* E-mail: jjinta@kbsi.re.kr

The three-dimensional (3-D) structure of an inorganic crystal, CaMoO<sub>4</sub>, was solved by electron crystallography utilizing the HVEM installed at the Korea Basic Science Institute (KBSI) in 2003. It has capability of high tilting ( $\pm 60^\circ$ ) and atomic resolution (0.12 nm). HRTEM images from 4 different major zone axes and selected-area electron diffraction patterns from 18 different zone axes were obtained. The 3-D electron diffraction data resulted in the basic crystallographic information of CaMoO<sub>4</sub>, space group  $I4_1/a$ ,  $a = 5.29\text{\AA}$  and  $c = 11.54\text{\AA}$ . A crystallographic image processing (CIP) of HRTEM images was used to determine the atomic coordination of CaMoO<sub>4</sub>.

The initial values of average phase errors ( $\phi_{res}$ ) for [001], [100], [110] and [111] HRTEM images were  $14.1^\circ$ ,  $3.3^\circ$ ,  $7.7^\circ$  and  $22.1^\circ$ , respectively. Further refinement of the structures was carried out using additional HRTEM images and electron diffraction patterns in different microscopic conditions. Effects of image quality, diffraction quality, specimen tilting, irradiation damage and energy filtering on the structure refinement have been evaluated.

[1] Crichton W.A., Grzechnik A., *Z. Kristallogr.*, 2004, **28**, 1. [2] Zhang Y., Holzwarth N.A.W., Williams R.T., *Physical Review B*, 1998, **57**(20), 12738.

**Keywords:** electron crystallography, inorganic materials, HRTEM

**P.19.02.2***Acta Cryst.* (2005). A61, C457**Increasing the Precision of Quantitative CBED Structure Factor Measurements**Philip Nakashima, *School of Physics and Materials Engineering, Monash University, Victoria 3800, Australia.* E-mail: Philip.Nakashima@spme.monash.edu.au

A new method of eliminating residual geometric distortions in convergent beam electron diffraction (CBED) patterns has been developed. It is integrated into quantitative CBED (QCBED) pattern-matching refinements of structure factors and significantly reduces uncertainties in experimental charge density measurements.

At present, a three- to fourfold improvement in structure factor measurement precision is reported in the case of corundum, over more conventional geometric distortion corrections employed in QCBED. This has resulted in a spread of multiple measurements of the same structure factor that is much smaller than the difference between different theoretical charge density models (density functional theory (DFT) and periodic Hartree-Fock (PHF) calculations) [1].

This new QCBED form shows heightened sensitivity to uncertainties in atomic coordinates, unit cell and atomic displacement parameters (ADPs). This is shown by introducing changes to these parameters and comparing resultant distributions of structure factor measurements using the new technique and parallel cycles of the more conventional form of QCBED (acting as a control).

Significant improvements to the precision of structure factor measurements at the higher order end of the QCBED range implies that merging QCBED and X-ray structure factor measurements will result in more reliable scale factor determinations for the latter and therefore, more accurate experimental charge density maps.

[1] Nakashima P.N.H., *J. Appl. Cryst.*, 2005, in press.

**Keywords:** QCBED, geometric distortions, structure factors