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Atomic Resolution at 50 – 300 kV Obtained Using Low Dose Rate HRTEM

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State-of-the-art high resolution TEM (HRTEM) using aberration correction allows for imaging of suitable samples with sub-Å lateral resolution [1]. The object exit wave can be recovered from through focal series [2]. In addition, the use of efficient, ultra-fast direct electron detectors [3] can enable the tracking of sample dynamics, such as atomic diffusion paths [4] and catalytic reactions [5]. Obtaining sufficient image signal-to-noise ratios (SNRs) for sub-atomic resolution generally depends on system stability and high-intensity illumination conditions [1].

However, it is proposed that sub-Å structural resolution in HRTEM is not only limited by instrumental parameters, but also by beam-induced atom movements such as the excitation of 3D lattice phonons [6]. Preliminary experiments with STO strongly suggest that exposure dose rates <10 $e^-/Å^2s$ will significantly reduce such movements and allow for a more precise evaluation of exit wave phases.

In addition, a growing number of "soft" materials of interest [7], composed predominantly of light atoms (H,C,N,O…) do not permit HRTEM analysis with atomic resolution at present. Those samples are often beam-sensitive and subject to fast degradation under usual HRTEM conditions with dose rates of > 1,000 $e^-/Å^2s$ (HDR conditions). In order to obtain atomically resolved structural information from soft materials, it is desirable to find imaging conditions with low dose rates of ~10 $e^-/Å^2s$ (LDR conditions). Furthermore, displacement damage can be reduced by lowering the electron energy towards 50-20 keV. Therefore, combining LDR and low voltage conditions appears to be a promising approach to HRTEM of soft and hard materials.

We show that using the aberration-corrected TEAM 0.5 instrument equipped with a coherent high brightness electron source (XFEG) and a monochromator permits imaging of Si crystals at atomic resolution using 300 to 50 keV. Si(110) dumbbells can be resolved in both conventional HDR and LDR conditions down to a dose rate of 22 $e^-/Å^2s$. Aligned averages of 5 consecutive LDR images show sufficient SNR for exit wave reconstruction using the Gerchberg-Saxton algorithm. Comparison of exit wave phases from HDR and LDR datasets point towards a sharper phase signal for dumbbell atoms under LDR conditions. In the future, low-voltage LDR HRTEM, combined with ultra-fast direct detection and a physical phase plate, will be used to image hard-soft matter interfaces in materials relevant to chemical catalysis and harvesting of solar energy [8].

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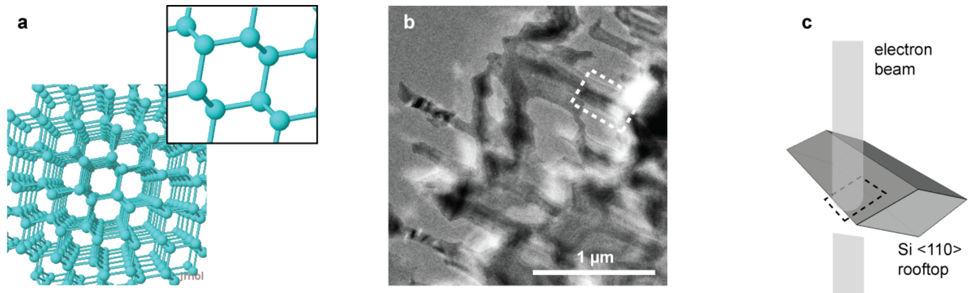

FIG. 1. : (a) Model of a Si crystal viewed along <110> direction. The characteristic dumbbells have a projected distance of 136 pm. (b) TEM image of the edge of a Si[110] plan view specimen obtained from a wafer by dimpling and subsequent etching with hydrofluoric acid (HF) and potassium hydroxide (KOH). (c) The etching process produces a "rooftop" topography favorable for HRTEM imaging.

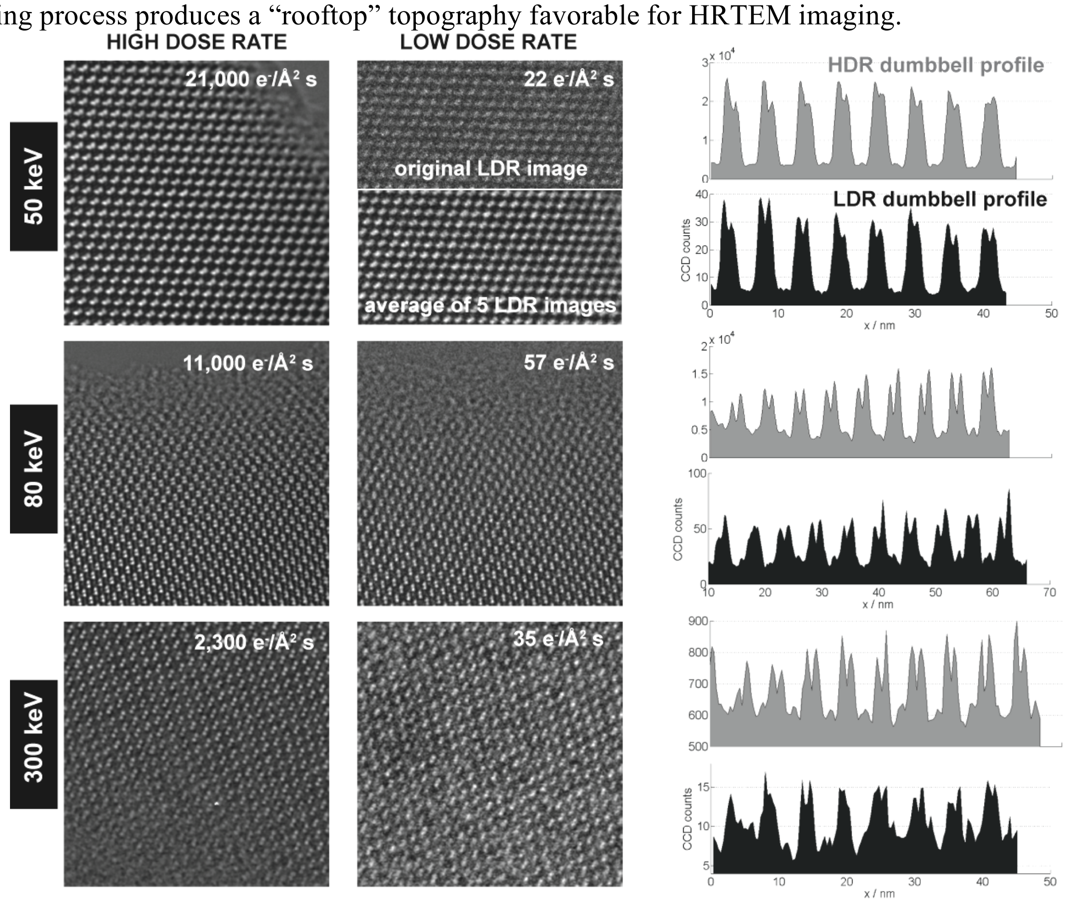

FIG. 2. Si(110) "rooftop" sample imaged using the aberration-corrected, monochromated TEAM 0.5 microscope under conventional HDR and LDR conditions at 50, 80 and 300 keV electron energy. While individual LDR images show a low SNR, sufficient signal can be obtained by aligned averaging of 5 LDR images. 136 pm dumbbells can be resolved in HDR and LDR between 50 and 300 keV electron energy, even with a dose rate as low as 22 $e^{-}/Å^{2}s$.

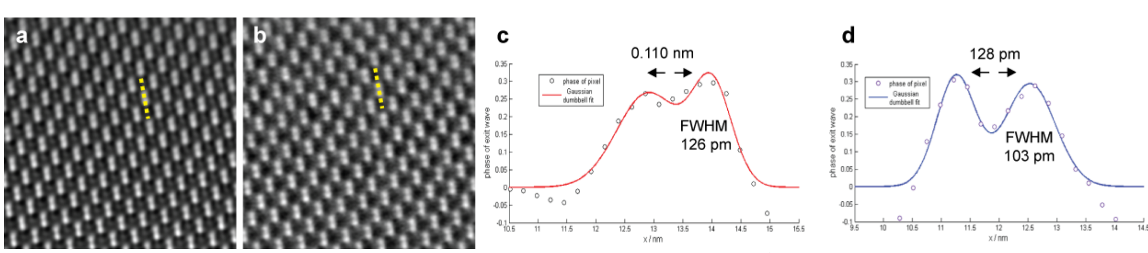

FIG. 3. Phase of exit wave reconstruction from HDR (a; 11,000 $e^{-}/Å^{2}s$) and LDR focal series (b; 57 $e^{-}/Å^{2}s$). (c,d) LDR conditions may yield sharper atomic phase images and better dumbbell peak separation.

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