

Low Dose STEM for Applications to Materials Science

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Achieving atomic resolution in scanning transmission electron microscopy (STEM) requires the beam to be focused to a small electron probe, which results in a substantial reduction in the total beam current. Thus, increasing the beam current has always been a major issue in advances in STEM. This is compounded by the fact that the two most popular techniques of STEM -- high angle angular dark-field (HAADF) imaging and for electron energy loss spectroscopy (EELS) -- inherently only collect a small amount of electrons available in the electron probe. In general, most researchers aim to increase the beam current in order to achieve high signal-to-noise data. Now, with C_S -corrected STEM, the beam current can be increased by orders of magnitude [1].

On the other hand, the massive increase in the probe current density can lead to a very high specimen radiation dose. This can cause devastating consequences for specimens such as zeolites, catalysts, and bio-materials. Catalysts and zeolites are believed to damage at doses on the order of $100 \text{ e}/\text{\AA}^2$ [2], and non-stained biological samples can only be imaged at high-resolution at doses below $5 \text{ e}/\text{\AA}^2$ when kept at liquid nitrogen temperature [3]. It is therefore advantageous to develop low dose STEM techniques to investigate these materials.

Therefore, we have investigated ways to reduce the specimen electron dose in STEM while still retaining atomic resolution. This can be approached by either increasing the scan rate and/or by decreasing the gun current. Experimental images were obtained using a C_S -corrected JEOL 2100F. Figure 1a shows an image of SrTiO₃ taken with a dwell time of $0.5 \text{ }\mu\text{s}/\text{pixel}$. The total dosage is estimated to be $300 \text{ e}/\text{\AA}^2$ at the specimen level. The power spectrum and Fourier-filtered image are shown in figures 1b and 1c, respectively. At such high scan-speeds, artifacts occur due to the response time of the STEM detectors, which causes streaking in the image. Consequently, a slower dwell time of $2.0 \text{ }\mu\text{s}/\text{pixel}$ was found to be ideal. In order to decrease the dosage further, it was necessary to decrease the gun current. Figure 2a shows an image of the same specimen with the gun current lowered by 50 times. The scan rate was decreased to $2 \text{ }\mu\text{s}/\text{pixel}$, to reduce the scan artifacts. The dosage in this second image is estimated to be about $25 \text{ e}/\text{\AA}^2$. Atomic resolution can still be seen, as demonstrated by power spectrum and Fourier-filtered image shown in figures 2b and 2c.

The resulting images have a poor signal to noise ratio (SNR), due to the low electron count statistic. Computer image averaging and unbending techniques can be used to increase the amount of high resolution extractable from the images [4].

We will illustrate ways to combine the low dose method described above with appropriate statistical methods, which may be a way to utilize the full advantage of STEM techniques for characterizing beam sensitive materials.

References

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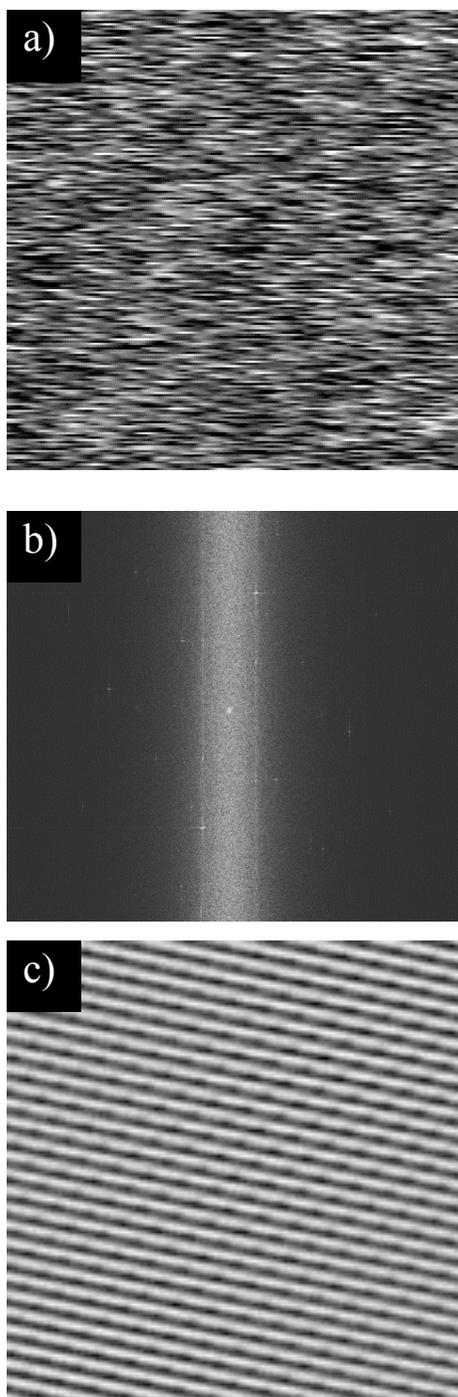


Fig. 1. a) Fast scanned image of SrTiO₃ at a scan speed of 0.5 ms/pixel with b) the power spectrum showing second order reflections. The vertical stripe of noise comes from artifacts induced from the fast scan rate resulting in smearing of the atomic columns in c) the Fourier-filtered image.

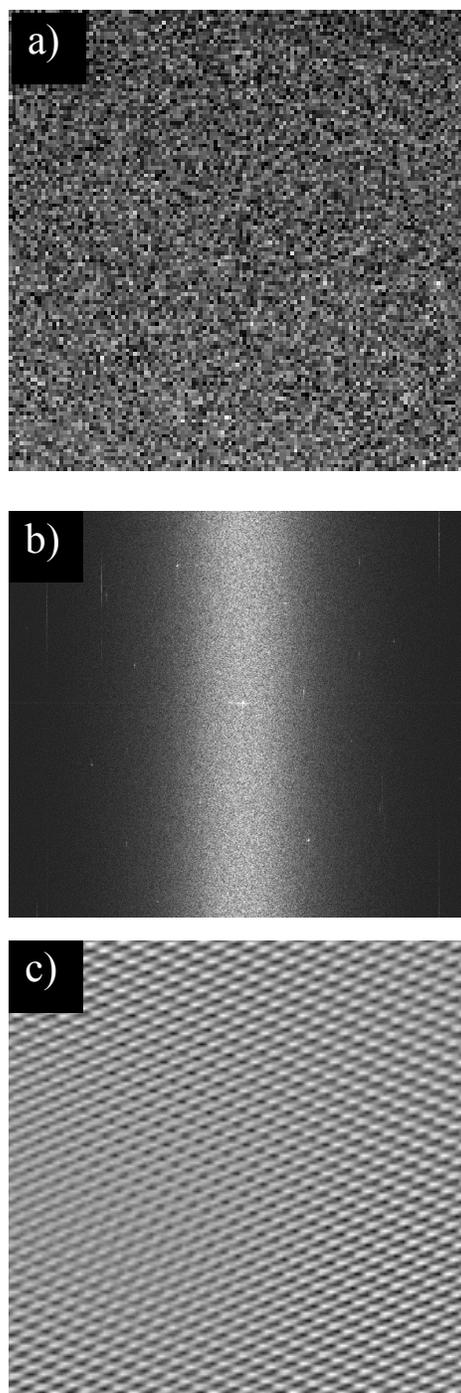


Fig. 2 a) Image of SrTiO₃ with a slower scan rate (2ms/pixel) and reduced gun current. Artifacts in b) the power spectrum are noticeably decreased, and c) the Fourier-filtered image shows atomic columns clearly in both directions.