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Accurate Objective Lens Defocus Calibration for Focal-Series Aberration-Corrected HRTEM at Sub-Ångström Resolution

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Aberration correction via software or hardware [1] allows an HRTEM to reach much higher values of resolution. For aberration correction using focal-series reconstruction of the electron wave at the specimen exit surface [2,3] to be able to extend resolution to the microscope information limit [4], we need accurate values for all known microscope parameters, including Cs and defocus. For best possible resolution in a reconstruction of the exit-surface electron wave from focal-series of images, we need images containing the highest possible spatial frequencies, preferably to the microscope information limit, $d_\Delta = \sqrt{\pi\lambda\Delta/2}$. Unlike the "fixed" parameters such as Cs and higher-order aberrations (such as three-fold astigmatism), defocus varies from image to image. Since defocus must be accurate for each image (at least to within the correction limits of the reconstruction code), both the focal series step size and starting defocus must be known accurately.

A common set-point for measurement of microscope defocus is the minimum contrast defocus. We have found that the position of the minimum contrast defocus (usually given as $0.44\sqrt{Cs\lambda}$ – i.e., in terms of just Cs and wavelength) is dependant on coherence parameters. Figure 1 shows how determination of minimum contrast is easy for strong damping (A) where small changes in defocus slide the positive-going peak up or down the damping curve thus changing image contrast [4], but not when the damping curve is almost flat (B). Optimum defocus [5] is a preferable set-point because small changes in defocus produce large changes in the main passband split (C).

Defocus step size can be calibrated using a thin amorphous specimen. Figure 2 shows results for the CM300-OÅM [6] revealing that defocus step size varies by 24% over the operating range. Step size is greater when the lens is weaker (underfocus) and decreases as the lens grows stronger [7].

Acquisition of a focal series of images can be carried out using the script supplied in Gatan's high-resolution software package, running within Digital Micrograph®, to step through defocus and acquire an image at each step. The script can be modified to accept optimum defocus as the set-point, and then to use the calibrated step size to move to alpha-null defocus [8] and commence focal-series acquisition. Figure 3 shows how correct use of defocus can produce resolution out to the information limit [9,10]. Incorrect values produce artifacts such as extra peaks [11].

Accurate measurement and calibration of defocus, essential for accurate reconstruction of the exit-surface wave, can be achieved to extend microscope resolution to microscope information limit [12].

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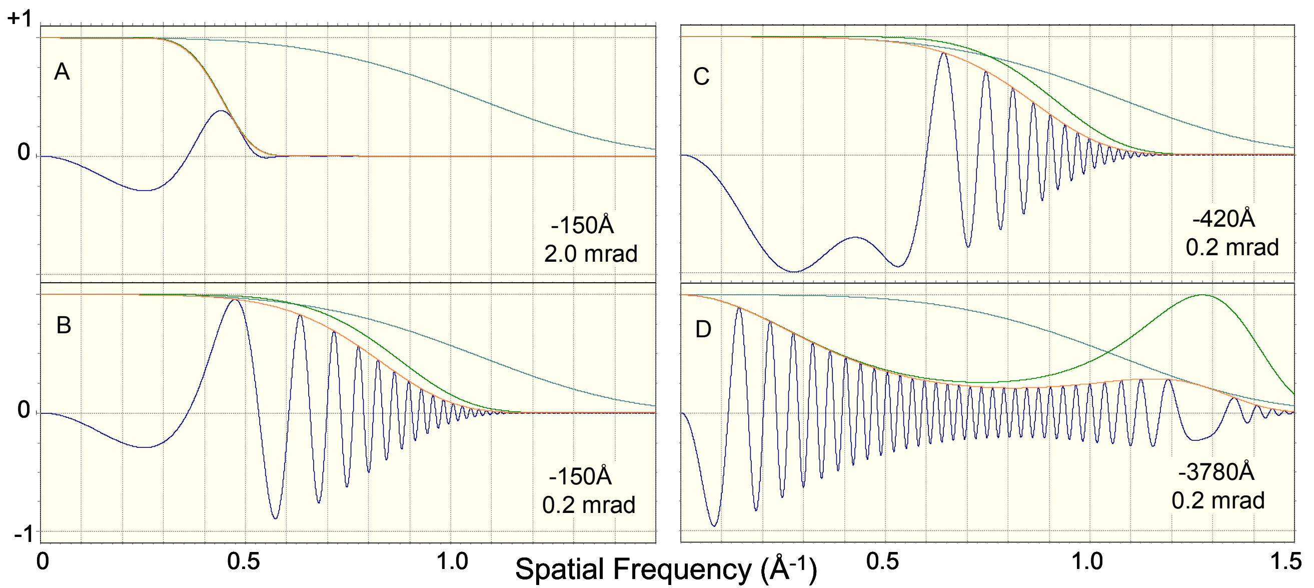
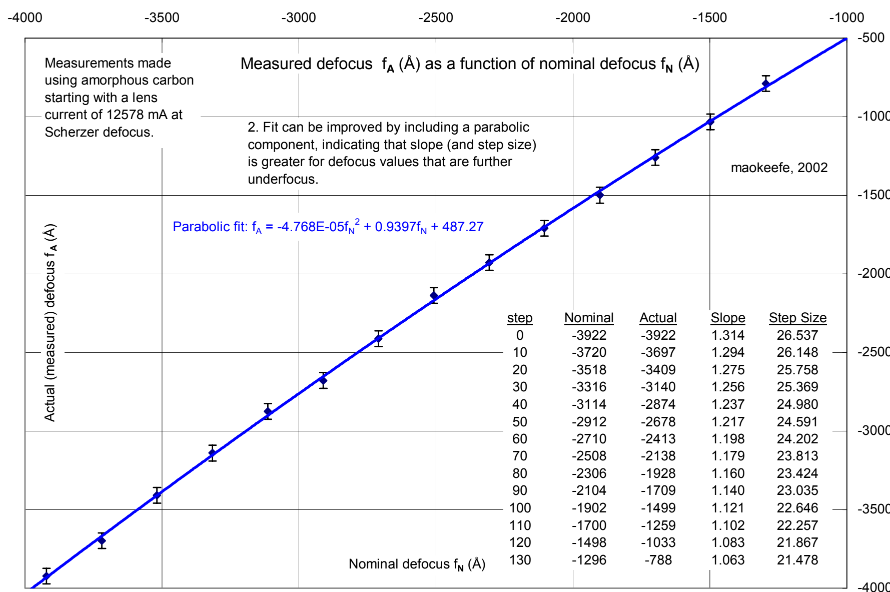


FIG.1. Contrast transfer functions (CTFs) drawn for 300keV and 0.6mm Cs. Minimum contrast defocus (-150Å) is obvious at an incident beam convergence of 2 milliradian (A), but not at 0.2 mrad (B). Transfer at optimum defocus (-420Å) is limited by the convergence of 0.2 mrad. Transfer at the 0.784Å silicon 444 frequency (1.275Å^{-1}) is considered optimized at alpha-null defocus (-3780Å) because the limiting effect is due to spread of focus instead of convergence (D).



step	Nominal	Actual	Slope	Step Size
0	-3922	-3922	1.314	26.537
10	-3720	-3697	1.294	26.148
20	-3518	-3409	1.275	25.758
30	-3316	-3140	1.256	25.369
40	-3114	-2874	1.237	24.980
50	-2912	-2678	1.217	24.591
60	-2710	-2413	1.198	24.202
70	-2508	-2138	1.179	23.813
80	-2306	-1928	1.160	23.424
90	-2104	-1709	1.140	23.035
100	-1902	-1499	1.121	22.646
110	-1700	-1259	1.102	22.257
120	-1498	-1033	1.083	21.867
130	-1296	-788	1.063	21.478

FIG.2 Plot of defocus (from diffractograms of images of amorphous carbon) as a function of nominal defocus for the CM300-OÅM [7]. Mean slope is 1.188, so the mean actual defocus change per click of focus step 2 is 2.377Å in comparison with the nominal 2.02Å. Detailed analysis [7] shows local slope is greater farther underfocus. Slope varies from 1.314 to 1.063 over the defocus range from –3900Å to –890Å and step size per #2 click varies from 2.654Å to 2.148Å over this range [7].

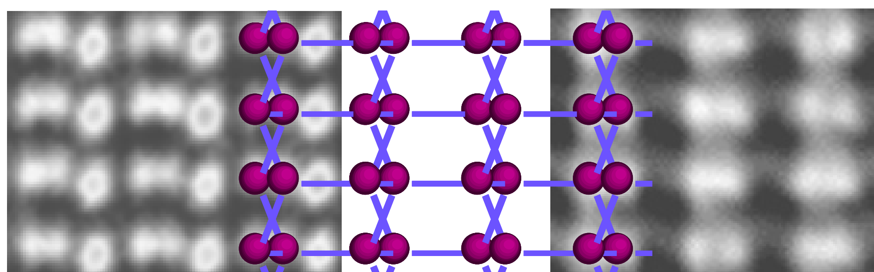


FIG 3. Si [112] exit-surface wave phase is reconstructed from incorrect defocus (left) and correct defocus (right). The incorrect reconstruction produces an extra peak (compare with model), and is not an accurate depiction of the exit-surface wave [11].

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