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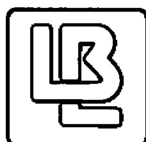

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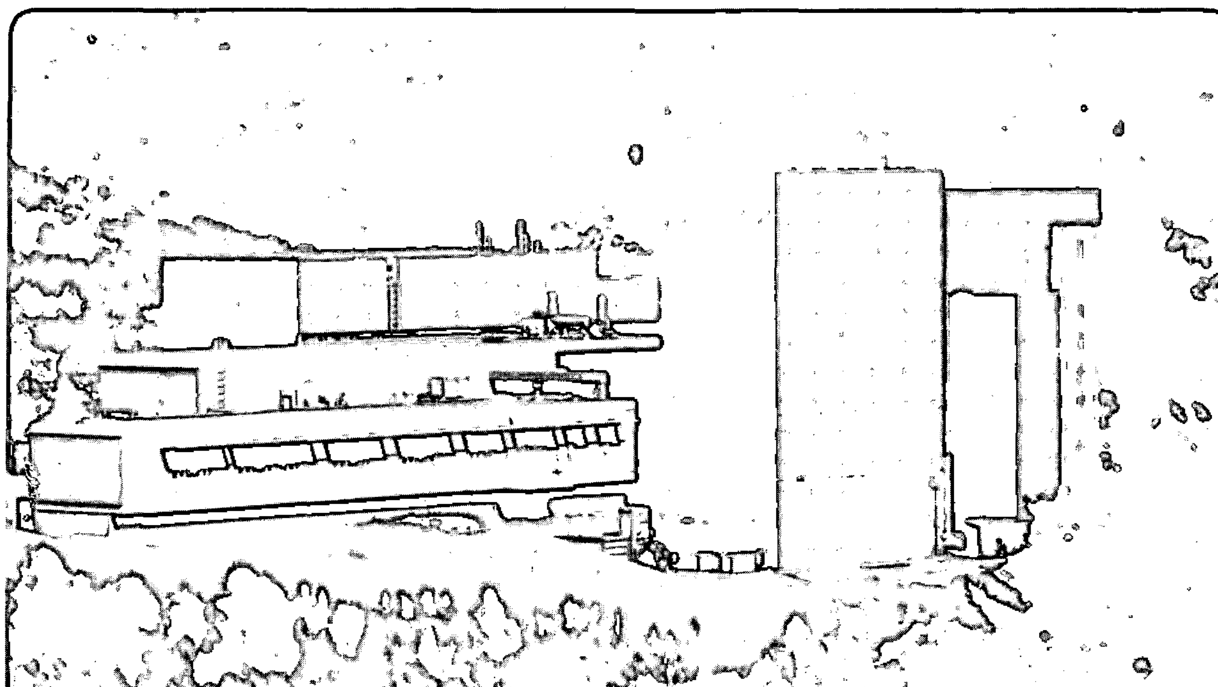
R. Gronsky

March 1991



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Advancing Conventional High-Resolution Electron Microscopy

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ADVANCING CONVENTIONAL HIGH-RESOLUTION ELECTRON MICROSCOPY

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Due to the exceptional performance of most modern commercial transmission electron microscopes, the achievement of phase-contrast imaging resolution in the sub-2Å range is today a routine exercise, provided the samples are compliant. Nonetheless, there remains room for improvement, and the purpose of this manuscript is to highlight procedures that might be employed by the practicing microscopist for advancing conventional high resolution electron microscopy.

RETRIEVING RESOLUTION

In the simple and inaccurate weak phase object approximation, the resolution performance of a TEM is most commonly defined by the first crossover of the phase contrast transfer function

$$T(u) = 2\sin\left(\frac{2\pi}{\lambda}\left(C_s\frac{\lambda^4 u^4}{4} + \Delta f\frac{\lambda^2 u^2}{2}\right)\right) \qquad \text{Eq. 1.}$$

at Scherzer defocus, shown schematically in Fig. 1. Provided the transfer characteristics of the lens are not limited by mechanical instabilities and/or chromatic errors, it is easy to buy some resolution by furthering defocusing the lens and opening up a higher-frequency pass band at the expense of multiple low-frequency zeroes in the transfer function (Fig. 2). Rarely is the severe filtration imposed by these low-order zeroes acceptable however, and this visualization of the problem has led to the idea[1] that if such images could be combined (imagine "adding" Fig. 1 and Fig. 2 to "fill in" the zeroes of the transfer function), the resolution performance of the microscope could be substantially improved. This procedure of reconstructing the object from a through-focus series of images amounts to on-axis holography, and requires special care in the reconstruction protocol. Statistical requirements for combining images are assessed in the literature[2], and the required computational power for handling large digitized image sets is now routinely available. As of this writing, newer methodologies for enhancing resolution performance by on-axis holography are also under development[3].

EXTENDING THE ENVELOPE

Of course, these reconstruction methods are possible only when the damping envelope function that accounts for all mechanical instabilities, electronic instabilities, and source coherence is well-behaved. In their mildest forms, such instabilities may be alleviated by time averaging, but in their severest forms, they completely damp the transfer function to the point where no pass bands can be opened beyond the Scherzer cutoff limit. The extent to which the operator can control vibration (particularly microphonics), source coherence, and electronic stability (filament, accelerating voltage, and lens currents) determines whether or not the above techniques for resolution retrieval can be applied.

SELECTING SPECIMENS

Still the most critical activity associated with transmission electron microscopy, the proper selection and preparation of specimens remains the key to achieving the best return of time invested in improving high resolution imaging. Specimens must be clean (no contamination, no reaction product overlayers), flat (no discontinuities in orientation), and thin (less than one

extinction distance for the primary diffraction vector) to yield useful phase contrast images, and there must of course be no artifacts associated with the specimen preparation procedure. New methodologies include precision crystallographic cleaving of specimens, sequential reactive and mechanical methods for thinning and surface cleaning, direct synthesis of thin films on grids, surgical machining, and combined *in-situ* thinning and deposition procedures. Significant advancements in conventional high resolution electron microscopy will continue to track any and all such improvements in specimen preparation, as they have in the past.

OPERATOR OPTIONS

The formation of a high resolution image of the conventional variety requires the establishment of phase contrast by a multi-beam, large-objective-aperture condition under operator control. Exercising patience in the orientation of the sample is essential, since a misoriented sample yields an image that is not only inferior but also impossible to interpret. The operator can make use of convergent beam electron diffraction for localized assessments of orientation that are as accurate as the highest order HOLZ lines will allow. Selecting an objective aperture of a diameter that just cuts off the most severely aberrated beams at high scattering angle enhances image contrast at no expense to resolution. This aperture diameter can be calculated using Eq. 1 to match the value of the reciprocal space parameter u at which T(u) crosses the zero axis beyond its largest pass band, before it begins its most rapid oscillations (c.f. Figs. 1 and 2). For specific materials that are repeatedly imaged, custom-designed apertures are in vogue.

COMPUTER CONTROL

As on-board CPU grows in size for modern instruments, so does the extension of its influence. Several operational codes exist for auto-alignment, auto-focus, and auto-compensation for astigmatism, and fewer for automatic manipulation of specimen orientation and position, all of which have influence over the formation of high resolution images. It is anticipated that resolution enhancement codes will be available in the near future for on-line on-axis holographic reconstruction.[4].

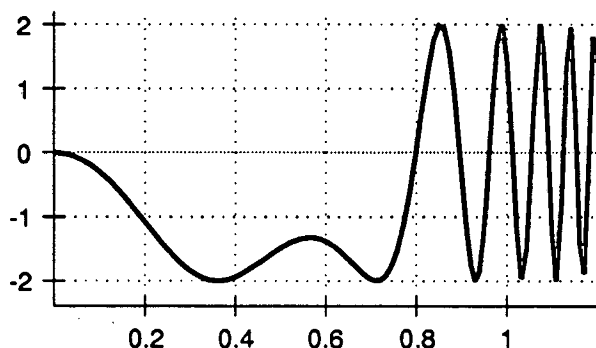

Fig.1 Undamped phase contrast transfer function at Scherzer defocus. Instrumental resolution limit defined at first crossover of zero axis.

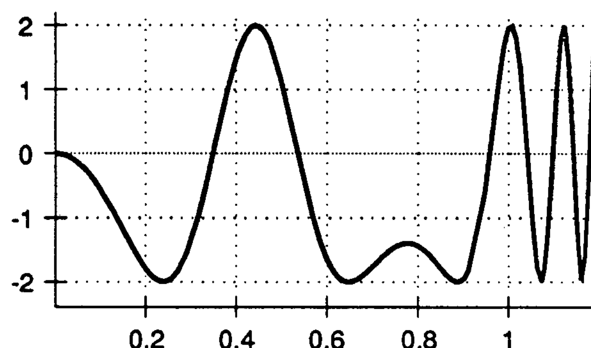

Fig. 2 Undamped phase contrast transfer function at first pass band defocus. A 10% increase in high resolution detail is retrievable.

[1]E.J. Kirkland, *Ultramicroscopy* **15**, 151 (1984).
[2]D.R. Brillinger, K.H. Downing, R.M. Glaeser, and G. Perkins, *J. Appl. Statistics* **16**, 165 (1989).
[3]M.A. O'Keefe, (to be published).
[4]This work is supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Division of Materials Sciences, U.S. Department of Energy, under Contract No. DE•AC03•76SF00098.

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