

Toward Quantitative Defect Analysis Using HREM

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The electron microscope has evolved to the level where it is now straightforward to record high-resolution images from thin samples (1-10 to 20 nm) that are directly interpretable in terms of atomic arrangements. Whilst recorded images necessarily represent two-dimensional projections of the structure, many defects such as dislocations and interfaces may be linear or planar in nature and this might be expected to be amenable to detailed characterization. In this review, we briefly consider the recent significant progress that has been made in quantitative defect analysis using the high-resolution electron microscope and then discuss some drawbacks to the technique as well as potential scope for further improvements. Surveys of defect modeling for some small-unit-cell materials¹ and interfaces² have been recently published, and reference should be made to other papers in this symposium for further examples.

The technique of structure imaging originated in the early '70s with observations of large-unit-cell block oxides^{3,4}. Image interpretation was initially validated by prior knowledge of the crystal structure, whereas it became commonplace in later studies to verify postulated structural models on the basis of agreement between experimental micrographs and calculated images, mostly simulated using the multislice algorithm⁵. The comparisons were usually qualitative and somewhat subjective, but the models were generally considered as more acceptable if an image "match" was achieved for more than one micrograph from a through-focal series⁶. It has, however, been established that the apparent locations of atomic columns at aperiodic features such as interfaces may vary by as much as 0.03 nm depending on the defocus value⁷. If structure refinement to this level of accuracy or better is desired, then improved methods for establishing the actual defocus values used for image recording must therefore be developed. In this regard, the use of automatic cross-correlation tech-

niques to compare the Fourier coefficients of diffracted beams in reciprocal space appears to be rapid, reliable and thus highly practical for evaluating periodic image features in the vicinity of the defect of interest⁸. Some studies have been published wherein the residual differences between experimental and calculated images have been minimized significantly using least-squared refinement methods, despite the need for correction of photographic nonlinearities (see, for example, Ref. 9). Nevertheless, such successes are by no means universal - as exemplified in recent work on Ga_xAl_{1-x} . As where the serious mismatch between image intensity, contrast and pattern appearance defied an exhaustive search to establish a conclusive cause for the differences¹⁰.

There are several factors that clearly could markedly affect the reliability of the refinement process, especially in achieving quantitative agreement between experimental and simulated micrographs. For example, image simulation programs utilize atomic scattering factors that may not be sufficiently accurate and, because of limited sampling in reciprocal space, approximations are invariably made for electron scattering in the vicinity of defects. On the practical side, multiple and inelastic scattering, amorphous surface overlayers, and electron irradiation damage may all influence the integrity of the recorded image to a greater or lesser (unknown?) extent. For example, structural modification, especially for atoms in the vicinity of lattice defects and surfaces, is inevitable in the face of the high beam current densities required for image recording at very high magnification. Total beam exposure should therefore be restricted whenever possible. Surface oxide or contamination layers, oftentimes caused by sample preparation or even exposure to air, must degrade the appearance of the final image which originates from electron transmission through the entire projected sample. In the absence of signal averaging, which is obviously inapplicable to aperiodic defects, image quantification can be substantially improved through careful attention to minimizing surface generated noise¹¹. Inelastic scattering cannot be avoided nor can its effects be easily incorporated into image simulations because of the lack of reliable information about the energy (and angular) spread associated with scattering of electrons from

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defects. The use of energy imaging filters could, however, facilitate image recording with only those electrons that have not lost energy in traversing the specimen - loss of contrast from the out-of-focus energy-loss electrons could thereby be alleviated or possibly removed¹². In some special cases, where the defect of interest is close to the edge of the sample, it might instead be possible to utilize the off-axis electron hologram which is energy-filtered¹³. Finally, whilst multiple scattering is difficult to incorporate reliably into dynamic calculations, its effect can at least be minimized by restricting imaging to very thin regions.

Several possibilities are available for improving the speed, reliability and/or accuracy of the defect analysis. It is obvious, for example, that digital recording with a slow-scan CCD camera provides a high DQE with wide dynamic range, and hence better counting statistics (signal-to-noise ratio) whilst also avoiding the nonlinearities associated with photographic recording¹⁴. However, for some applications the effective pixel size referred to the sample needs to be of the order of 0.01 nm or smaller but then the typical 1024x1024 pixel field of view could be considered as being restrictive (although interpolation techniques to locate the exact positions of contrast maxima and minima could be used to alleviate this limitation). None of the defect analyses so far published appear to have been carried out under computer-controlled operation of the microscope. Nevertheless, it is abundantly clear that location of the coma-free axis for beam alignment, adjustment of objective lens astigmatism and selection of defocus can be routinely accomplished using computer control with greater accuracy than that attainable by even the experienced microscope operator¹⁵. Subsequent defect refinement should thus be significantly simplified since it would not be necessary to take extraneous factors into account¹⁰. Improved image resolution is not likely to have a great impact on defect analysis at this stage of development since it is really signal-to-noise rather than resolution *per se* that determines the precision with which atomic columns can be located. However, the extra flexibility of imaging in additional projections should certainly facilitate the development of more

accurate three-dimensional defect models. Finally, it appears that more attention needs to be given to the structure refinement process itself, in particular the problems of optimizing the recursive algorithm (what parameters/what accuracy?) and minimizing the residual discrepancy between experimental and simulated images¹⁶. The possible influence on the refinement of factors unrelated either to the structure or the model has been pointed out¹⁶, and some of the issues relating to establishing uniqueness or "goodness of fit" have also been discussed¹.

In conclusion, defect analysis with the high-resolution electron microscope has now reached the stage where truly quantitative comparisons with structural models has become a reality. Applications to an increasing variety of materials over the next several years can be safely predicted and evaluation of different theoretical approaches to modeling might even be feasible¹⁷. ■

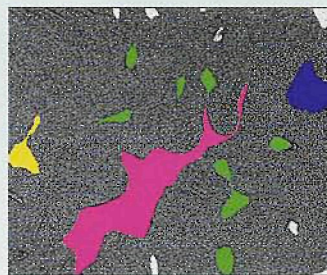
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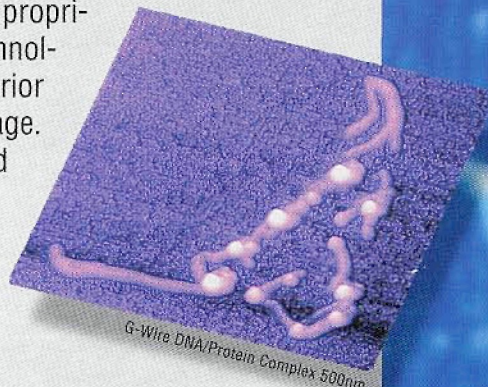
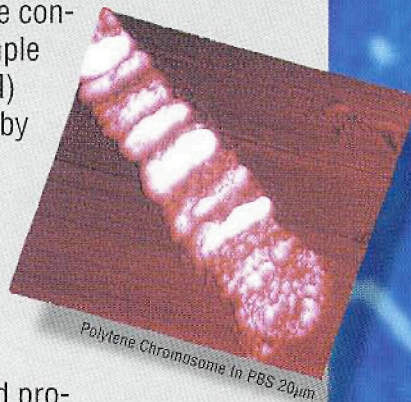
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