# DETERMINATION OF ATOMIC POSITIONS USING ELECTRON NANODIFFRACTION PATTERNS FROM OVERLAPPING REGIONS: Si[110]

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A procedure for determining atomic positions is described that utilizes electron nanodiffraction patterns from overlapping regions. The technique is applied using experimental data collected for Si[110] with a beam 4 Å in diameter. Autocorrelation functions are calculated for each beam position and correlated with theoretical functions. The positions of the Si atoms are established with an accuracy of  $\pm 0.2$  Å, and images with 1 Å resolution are computed for individual beam positions. It is also demonstrated how the technique may be used to determine, with similar resolution, the structure of noncrystalline regions adjacent to crystalline regions.

### **1. Introduction**

Electron nanodiffraction patterns may be obtained using beams as small as 3 Å in diameter with the HB5 STEM at ASU and stored on video tape at the rate of 30 patterns/s [1]. These diffraction patterns, which change quite drastically when the beam is translated a small fraction of its diameter, contain information on the structure of the atoms within the beam to a much higher resolution than the beam dimension. The resolution limit for the data analyzed in this paper is approximately 1 Å; however, in principle, it should be possible to collect data with a resolution of 0.5 to 0.7 Å.

In a previous paper [2], a method is described whereby structural information may be derived from sets of nanodiffraction patterns from overlapping regions of any thin specimen leading to calculation of an image having considerably better resolution than can be obtained from normal STEM imaging. From each nanodiffraction pattern an autocorrelation function (AC) may be

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calculated to show the interatomic vectors corresponding to pairs of atoms in the illuminated area of the specimen. By noting the beam positions for which the AC peaks have maximum value, it is possible to identify the mid-points on the interatomic vectors and hence to determine the atomic positions. For this procedure to be effective, the nanodiffraction patterns must be accurately collected to a sufficiently large scattering angle so that the dominant features in the calculated AC functions are resolved interatomic vector peaks. This approach is not effective for deriving the information on the scale of 1 Å contained in the experimental data analyzed in this paper. The reason is that AC functions calculated with 1 Å resolution data obtained with a 4 Å beam contain prominent features that are not directly interpretable in terms of interatomic vectors. The limited data introduces details due to series termination into the ACs. Damping the intensity data before Fourier transforming to obtain the ACs would reduce series termination effects, but would also reduce the resolution of the ACs. Additionally,

details in the ACs are a function of sample thickness, instrument focus and astigmatism. Any technique that fully utilizes the nanodiffraction data must incorporate and determine these variables.

For these reasons an alternative approach has now been developed in which the information contained in the nanodiffraction patterns and in the corresponding AC functions is used more explicitly. For crystalline regions the AC functions may be analyzed by comparison with corresponding functions calculated from model structures. This comparison may be used to determine the experimental parameters with the high accuracy necessary for determination of atom positions. The data from a crystalline region can then be used as a basis for extending the analysis to adjacent non-crystalline regions or to crystal defects.

This paper reports on the analysis of experimental data for Si[110]. An area 17 Å  $\times$  13 Å was scanned with a beam 4 Å in diameter, and nanodiffraction patterns were recorded at approximately 0.5 Å intervals. Each diffraction pattern contains information to a resolution of about 1 Å. Analysis of the patterns yields the positions of the Si atoms with an accuracy of  $\pm$  0.2 Å. The pairs of Si atoms separated by 1.36 Å are clearly resolved.

Theoretical calculations will be briefly described and used to illustrated the structural information contained in each diffraction pattern. The experimental data will then be compared with the theoretical predictions, and the positions of the atoms will be established. Finally, the potential effectiveness of the technique for determining the positions of atoms in regions of unknown structure will be examined.

## 2. Theory

For a point electron source, the beam wave function at the sample, g(r), for a beam centered at  $r_0$ , is taken to be the Fourier transform of the objective lens contrast transfer function,  $\exp[-i\chi(u)$  [3]:

$$g(r) = \mathscr{F} \{ \operatorname{Ap}(u) \exp[-i\chi(u)] \exp(-2\pi i u r_0) \}$$
$$= \mathscr{F} \{ G(u) \}, \qquad (1)$$

where

$$\chi(u) = \pi \Delta \lambda |u|^2 + \frac{1}{2} \pi C_{\rm s} \lambda^3 |u|^4$$

 $\mathscr{F}$  indicates a Fourier transform,  $|u| = 2 \sin (\Theta)/\lambda$ ,  $\Theta$  is the angle between the center of the main beam and the scattered electrons,  $\lambda$  is the electron wavelength,  $C_s$  is the objective lens spherical aberration coefficient,  $\Delta$  is the defocus and Ap(u) is the aperture limiting the angle of beam convergence. The radius of Ap was 0.32 Å<sup>-1</sup> for the data collecting,  $C_s = 0.85$  mm, and  $\Delta$  was approximately -445 Å. The real (Re) and imaginary (Im) parts of the beam wave function are displayed in figs. 1a and 1b.

The incident beam interacts with the scattering potential of the sample. Modified versions of the ASU multislice programs [4] were used to evaluate the electron wave as it passed through the sample. The slice thickness was taken to be 3.84 Å, the periodicity of Si parallel to the beam direction. The atoms were placed in a cell 27.15  $\text{\AA} \times 27.15$  $Å \times 3.84$  Å and the scattering potential was calculated at (27.15 Å/128 = 0.212 Å) intervals. The diffraction phase grating for this slice of atoms, which is the Fourier transform of the scattering potential, consists of a square array of Bragg reflections with a spacing of (1/(27.15 Å) =0.037  $Å^{-1}$ ). The 5959 beams with scattering vectors of magnitude less than 1.6  $Å^{-1}$  were used to propagate the wave through the sample. The real and imaginary parts of the scattering potential for a small region of a single slice are illustrated in figs. 1c and 1d.

The electron wave at the exit surface of the sample,  $\psi$ , may be expressed as the sum of scattered,  $\psi_{\rm S}$ , and unscattered,  $\psi_{\rm B}$ , components:

$$\psi(r) = \psi_{\rm S}(r) + \psi_{\rm B}(r)$$

The diffraction pattern is  $\Psi(u)\Psi^*(u)$ , where  $\Psi(u)$  is the Fourier transform of  $\psi(r)$ :

$$\Psi(u) \Psi^{*}(u) = \left[ \Psi_{S}(u) + \Psi_{B}(u) \right] \left[ \Psi_{S}(u) + \Psi_{B}(u) \right]^{*} = \Psi_{S}(u) \Psi_{S}^{*}(u) + \Psi_{S}(u) \Psi_{B}^{*}(u) + \Psi_{S}(u) \Psi_{B}^{*}(u) + \Psi_{S}^{*}(u) \Psi_{B}(u) + \Psi_{B}(u) \Psi_{B}^{*}(u), \qquad (2)$$

where the asterisk indicates a complex conjugate.



Fig. 1. (a) Real part of beam wave function for 4.0 Å beam. The parameters are defined in the text. (b) Imaginary part of the beam wave function. (c) Real part of scattering potential (minus constant term) for silicon viewed in [110] direction. Nanodiffraction data produced with beams centered at positions labeled A, B and C are discussed in detail in the text. (d) Imaginary part of the scattering potential for silicon.

Fig. 2a shows the diffraction pattern calculated for a 46 Å thick sample (12 slices) with the beam centered between Si atoms separated by 1.36 Å (position A in fig. 1c). The sample has been tilted  $0.3^{\circ}$  about the horizontal axis for this calculation to approximately duplicate the misalignment of the experimental sample. The disc of zeroed intensity at the center of the pattern is the main beam region within which all of the components of eq. (2) containing  $\Psi_{\rm B}$  are located.

The Fourier transform of the diffraction pattern is

$$\mathcal{F} \{ \Psi(u) \Psi^{*}(u) \} \\ = \psi_{S}(r) * \psi_{S}^{*}(-r) + \psi_{S}(r) * \psi_{B}^{*}(-r) \\ + \psi_{S}^{*}(-r) * \psi_{B}(r) + \psi_{B}(r) * \psi_{B}^{*}(-r), \quad (3)$$



Fig. 2. (a) Diffraction pattern calculated for beam centered at position A. The parameters are defined in the text. (b) Positive portion of the difference intensity formed by subtracting the average intensity for the unit cell from (a). Marker represents 1 Å<sup>-1</sup>.

where \* between terms signifies a convolution.

The features in the first term on the right side of eq. (3) (which we call the autocorrelation function, AC) contain the interatomic distance information modified by the beam and multiple scattering. The AC is a complex function. The real part of it, which we concentrate on in this paper, is the real part of the exit surface scattered wave function convoluted with itself plus the imaginary part of the exit wave convoluted with itself [2].

The desired features in the autocorrelation function (AC) may be isolated from the other components in eq. (3) by omitting the main beam region of the intensity when taking the Fourier transform. However, in doing so, the scattered portion of the electron beam under the unscattered main beam is also lost. The distortions this loss introduces into the AC may be evaluated by examining the components of the real space exit surface wave function.

Figs. 3a and 3b show the full real space exit surface wave function corresponding to the diffraction pattern in fig. 2a (position A, 46 Å thick). This function was phase-shifted by  $58^{\circ}$  for illustration purposes. Such a phase shift does not affect the calculated intensities or ACs, but results in the central atom positions having maximum positive values in the real part of the exit wave. Figs. 3c and 3d illustrate that portion of the exit surface wave function obtained by Fourier transformation of the portion of the reciprocal space wave function with  $0.35 < |u| < 1.0 \text{ Å}^{-1}$ , the region of the experimental diffraction patterns that has been transformed in the analyses. Figs. 3e and 3f display the portion of the exit surface wave function omitted in figs. 3b and 3c. Omission of the low-angle scattering removes the broad feature in the exit surface wave function centered between the atoms and actually results in somewhat increased resolution. Structural details of the resolution shown in figs. 3c and 3d are the objective of the experimental data analyses.

#### 3. Experimental data analyses

A region 17 Å  $\times$  13 Å was scanned with a beam approximately 4 Å in diameter in 24 s. The 714 diffraction patterns corresponding to beam positions separated by 0.5 Å horizontally along the sweep and 0.6 Å vertically were stored on video tape.

The data were transferred from the video tape to a Panasonic WORM optical disc that stores the video signal in a manner that may be randomly accessed. Each diffraction pattern was digitized with a PCVISION frame grabber. The data were averaged in regions 0.04  $\text{\AA}^{-1} \times 0.04$   $\text{\AA}^{-1}$ , and a



Fig. 3. (a) Real part of theoretical exit surface wave function for 48 Å thick Si calculated using diffraction data; (b) imaginary part corresponding to (a); (c) real part of theoretical exit surface wave function calculated using data 0.35 < |u| < 1.0 Å<sup>-1</sup>, the limits used with the experimental data; (d) imaginary part corresponding to (c); (e) shows (a) minus (c); (f) shows (b) minus (d).



Fig. 4. (a) Average theoretical intensity for a unit cell. (b) Experimental intensity for position A. Marker represents  $1 \text{ \AA}^{-1}$ .

56 × 56 array of intensities for each beam position was transferred via Ethernet to the NRL Cray XMPII for analyses. To illustrate the high resolution of the results, an 8.5 Å × 9.0 Å section of the area scanned (17 × 14 beam positions) is displayed in the illustrations rather than the full 17 Å × 13.4 Å area.

Figs. 4a and 4b show a theoretical intensity averaged over a unit cell and a single experimental intensity. When these are compared with the diffraction patterns in fig. 2, it is apparent that a substantial background ( $\approx 75\%$  of the intensities) is present that does not change with beam position. This background intensity may arise in part from inelastic scattering leading to the excitation of plasmons or intra-band transitions. Because these inelastic scattering processes are not sufficiently localized, their excitations may not be sensitive to incident beam position on a 1-4 Å scale. Another source of the background may be contamination of the objective aperture. Small regions on the edge of the aperture hole are commonly seen to charge-up, giving strongly localized variations of phase of the electron wave and hence broad background features in the diffraction plane.

In order to extract an intensity that varies with beam position, an average intensity for that scan line was subtracted from each theoretical and experimental intensity. In this manner, the extra background present in the experimental data could be removed. These experimental difference intensities were then Fourier-transformed to produce AC functions for each beam position.

Fig. 2a displays the full theoretical pattern for position A, along with the associated difference intensity, fig. 2b, that will be described later. Fig. 5b shows the associated real portion of the AC obtained by Fourier-transforming this difference intensity. The most prominent features are the  $\pm 1.36$  Å peaks corresponding to the separation of the silicon atoms.

This theoretical AC was correlated with all experimental ACs and the resulting correlation coefficients:

$$\operatorname{corr}(r_{0}) = \frac{\sum_{r} [\operatorname{AC}_{\operatorname{ex}}(r)] [\operatorname{AC}_{\operatorname{theo}}(r)]}{\left\{ \sum_{r} [\operatorname{AC}_{\operatorname{ex}}(r)]^{2} \sum_{r} [\operatorname{AC}_{\operatorname{theo}}(r)]^{2} \right\}^{1/2}},$$
(4)

are plotted in fig. 6a for all 238 beam positions  $r_0$ . Correlation coefficients, which have been used extensively in this analysis, may assume values ranging from 1.0 for perfect positive correlation through zero for no correlation to -1.0 for perfect negative correlation. Because of the short interatomic distance that dominates the scattering for position A, the main features in the theoretical AC are not extremely sensitive to the value of  $\Delta$ .



Fig. 5. (a) Real part of experimental autocorrelation for beam position A. (b) Real part of theoretical autocorrelation for beam position B. (d) Real part of theoretical autocorrelation for beam position B.

and, as a result, the map in fig. 6a is quite insensitive to parameters chosen for the theoretical calculation. In fact, a nearly identical map, divorced completely from theory, is obtained when the value of the 1.37 Å experimental AC peak is plotted versus beam position.

Fig. 5a shows the average experimental AC for the four beam positions closest to peak centers in fig. 6a. The correlation coefficient relating figs. 5a and 5b is 0.85. Figs. 7a and 7b illustrate the theoretical and the experimental difference intensities from which the ACs were computed. The correlation coefficient relating them is 0.62. The correlation between the intensities is lower than that between the ACs because the sample tilt, which affects primarily the imaginary part of the AC for this beam position, has not been refined. In addition to the correlation coefficient, an agreement R value similar to the one used in crystallography may be quoted. For position A



Fig. 6. (a) Correlation of real theoretical autocorrelation for beam position A with experimental autocorrelations for all 238  $(17 \times 14)$  beam positions indicated with thick marks. Si atom positions are marked with dots. (b) Same as (a) except theoretical autocorrelation for beam position B is used. (c) Same as (a) except theoretical autocorrelation for beam position C is used. Inset in lower right is theoretical peak shape that was fit to the

peaks in (a), (b) and (c) to determine their locations.

$$R(\text{intensity}) = \frac{\sum |I(\text{obs}) - I(\text{calc})|}{\sum I(\text{calc})} = 0.45$$

This corresponds to an R(amplitude) of approximately 0.23. For crystallographic data of 1 Å resolution or higher, this generally signifies that the essential features of the structure have been determined and that least-squares refinement of the model parameters to fit the theory to experiment will be successful. Similar refinement will be employed as this technique is applied to samples containing regions of unknown structure.

Fig. 5d displays the real portion of the theoretical AC for beam position B. Fig. 6b plots the correlation coefficient versus beam position. This map, involving a longer interatomic distance (2.36 Å versus 1.36 Å), is sensitive to the theoretical defocus value. Values for  $\Delta$  ranging from -100 to -700 Å were tested, and -450 Å produced the best resolved peaks. Fig. 5c displays the average of the four experimental ACs nearest the peak centers; the correlation coefficient relating figs. 5c and 5d is 0.91. Figs. 8a and 8b are the theoretical and experimental difference intensities for position B and are related by a correlation coefficient of 0.48 and an R(amplitude) = 0.27.

#### 4. Estimation of Si atom positions

In order to locate the Si atom positions, the theoretical shapes for the peaks in fig. 6 were calculated. The theoretical shapes were approximated with a Gaussian function ( $\sigma = 0.7$  Å) from which a constant was subtracted. This analytic function is shown as an inset in fig. 6c. A least-squares fit was made of this function to each peak in figs. 6a-6c. The least-squares estimates for the standard deviations in peak positions were approximately 0.1 Å for position A and 0.15 Å for positions B and C. A map,  $\rho(r)$ , of the scanned area was then prepared

$$\rho(r) = \sum_{i} \frac{\operatorname{corr}(i)}{\sigma(i)} \times \exp\left(-\frac{\left[r(i) \pm \frac{1}{2}r(i, v) - r\right]^{2}}{2\sigma^{2}(i)}\right).$$
(5)



Fig. 7. (a) Theoretical difference intensity for beam position A. (b) Experimental difference intensity for beam position A. Marker represents  $1 \text{ Å}^{-1}$ .

where corr(i) is the correlation coefficient at refined peak position r(i);  $\sigma(i)$  is three times the least-squares estimate for the standard deviation in the accuracy of r(i); r(i, v) is the interatomic vector that dominates the AC for position *i*. The resulting map is shown in fig. 9. The atomic positions are well resolved.

#### 5. Images for individual beam positions

When the position of the electron beam relative to the atoms is approximately known, meaningful

images may be formed using theoretical phases and experimental amplitudes. Additionally, the computed intensity for each beam position may be fitted to the experimental intensity by refining atomic positions, sample thickness, and beam parameters. For the work reported here, a rather simple variation of defocus and thickness parameters indicated that -445 Å and 50 Å were approximately the correct values. Figs. 10e and 10f are the real and imaginary parts of the theoretical exit surface wave function for position A formed by taking the Fourier transform of the reciprocal



Fig. 8. (a) Theoretical difference intensity for beam position B. (b) Experimental difference intensity for beam position B. Marker represents  $1 \text{ Å}^{-1}$ .



Fig. 9. Equal probability contours determined by the technique described in the text for the positions of the Si atoms in the 8.5 Å×9.0 Å area for which 238 diffraction patterns were collected. The closest pairs of atoms are separated by 1.36 Å.

space wave function composed of theoretical amplitudes and theoretical phases. Figs. 10c and 10d were obtained by Fourier transforming theoretical phases and theoretical difference amplitudes,  $ADIFF_{theo}(u)$ 

$$ADIFF_{theo}(u) = \{I_{theo}(u) - IAV_{theo}(u)\}^{1/2},$$

where  $IAV_{theo}(u)$  is an average over a unit cell. For the images calculated with the difference intensities, all intensities less than zero were set to zero. Figs. 10c and 10d are very similar to figs. 10e and 10f. Figs. 10a and 10b are formed with experimental difference amplitudes and theoretical phases. The differences between the images calculated with experimental amplitudes and those calculated with theoretical amplitudes are due to errors in the experimental intensity measurement and incorrect instrumental parameters. For a well-refined model structure, the image most resembling the exit surface wave function would be obtained by using theoretical phases and amplitudes obtained by taking the square root of the sum of the experimental difference intensity and the average theoretical intensity. The addition of the average theoretical intensity to the experimental difference intensity would result in a positive total intensity.

Figs. 11a–11f illustrate the corresponding images for position B. It may be emphasized that positions A and B differ by a beam translation of only 1.7 Å. The correlation coefficient relating the experimental difference intensities at positions A and B is -0.12, indicating very little correlation. This gives a measure of the dramatic change in diffraction pattern associated with a small beam translation.

# 6. Determination of atomic positions for unknown structures

Had the structure of Si not been known, could it have been derived from the nanodiffraction data? Yes, it could have been, since so few atom columns are in the main portion of the beam at any one time. The [110] projection of Si is composed of one column of Si atoms per 5.2  $Å^2$ , or two columns on the average within a beam 4 Å in diameter (see figs. 10 and 11). The origin of an AC is the integral of the difference intensity for that beam position over all scattering angles. A plot of the origin of the ACs versus beam position is nearly identical to fig. 6a. This plot identifies the beam positions at which maximum scattering occurs. The experimental ACs associated with these positions are then examined. Fig. 5a displays this AC. The positive peaks would be considered candidates for interatomic vectors. The AC suggests that pairs of atoms are separated by about 1.3 Å. A plot of this AC peak versus beam position also is nearly identical to fig. 6a, and determines approximately the positions of the atoms. Theoretical ACs could then be calculated with these approximate atom positions and compared with the experimental ones as previously described. In this manner both the model parameters and the instrument parameters could be refined. This technique differs from the automatic procedure described in ref. [2] in that the beam positions with greater than average scattering are first identified, then only the vectors associated with peaks in these ACs are plotted versus beam position, rather than plotting all possible vectors versus beam position.



Fig. 10. (a) Real part of image for position A formed with experimental difference amplitudes and theoretical phases. (b) Same as (a) except imaginary part of image. (c) Real part of image for position A formed with theoretical difference amplitudes and theoretical phases. (d) Same as (c) except imaginary part of image. (e) Real part of theoretical exit surface wave function for position A. (f) Same as (e) except imaginary part of function.



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Fig. 11. (a) Real part of image for position B formed with experimental difference amplitudes and theoretical phases. (b) Same as (a) except imaginary part of image. (c) Real part of image for position B formed with theoretical difference amplitudes and theoretical phases. (d) Same as (c) except imaginary part of function. (e) Real part of theoretical exit surface wave function for position B. (f) Same as (e) except imaginary part of function.



Determination of the structure of a defect within a crystal or the structure of an interface adjacent to a crystal presents a more difficult problem. We propose that it may still be possible to determine the atomic structure of such regions by considering nanodiffraction data for overlapping regions that move progressively from the oriented crystalline region, within which the atom positions have been determined, into the region of unknown structure.

A 46 Å thick specimen of Si will have approximately 24 atoms within the beam. In the approximation that the beam intensity is confined to a column 4 Å in diameter, two beam positions separated by 0.5 Å will have 80% of their irradiated volume in common. Thus, translating the beam by 0.5 Å subtracts 5 atoms from the beam and adds 5 atoms on the average.

Consider a beam position for which 80% of the volume is occupied with atoms of known positions. For the Si system we are considering, this means that the positions of 19 of the 24 atoms are known; i.e. they are part of the model structure that is being refined to reproduce the experimental data. The approximate projected structure of the 5 unknown atoms may be determined by calculating the exit surface wave using experimental amplitudes and theoretical phases computed from the positions of the 19 known atoms, a technique long used in crystallography to establish the positions of unknown atoms when the majority of the atom positions are known. The degree to which the projected atoms may be resolved depends on the type of structure present in the defect or interface region and the resolution of the diffraction data. If the atoms are arranged in columns parallel to the beam, they should be easily resolved with data of the accuracy presented in this paper. If the atoms were completely random (which stereochemical considerations prohibit) then each projected unknown atom would occupy only 0.43 Å<sup>2</sup>

Fig. 12. (a) Real part of the theoretical exit surface wave function for position A calculated with an atom missing. (b) Real part of image for position A formed with experimental difference amplitudes and phases calculated with a missing atom. (c) Difference Fourier map obtained by subtraction of (a) from (b). The main features are the missing atom and its ghost. The cross marks the position of the known atom.

on the average. Thus, atoms would be separated by only about 0.7 Å on the average in projection and accurate diffraction data to a resolution of about 0.5 Å would be required to resolve most of the atoms. Completely resolved or not, atoms would be added to the model structure and their positions refined to reproduce the experimental diffraction data before moving on to the next diffraction pattern and determining several more atom positions in the same manner.

The experimental data may be used to demonstrate how a nanodiffraction pattern obtained from a sample volume within which some of the atomic positions have been established may be analyzed to determine the positions of additional atoms. Let us assume that all of the atom positions in fig. 1c to the right of the position labeled A have been established through analyses of nanodiffraction patterns from that region. The model structure with which we are computing theoretical nanodiffraction patterns for comparison with experimental ones contains no atoms to the left of position A. A beam centered at position A will contain one column of atoms for which we know the coordinates and one column of which we are feigning ignorance and have not yet included in our model. We wish to find the position of this unknown column by using the experimental diffraction pattern for the beam centered at A in conjunction with the previously established position for the column immediately to the right of A.

Fig. 12a shows the theoretical exit surface wave function for a beam centered at A with the atom (column) immediately to the left of the beam center missing. The phases from this theoretical calculation employing the incomplete model will be combined with the experimental amplitudes for the beam at position A for the purpose of determining the positions of the atoms within a beam that are missing from the incomplete model. Fig. 12b is formed with the theoretical phases calculated with the incomplete model and the experimental difference amplitudes for position A. A peak is present at the position of the missing atom, and a ghost atom is present to the right of the known central atom. Since the known atom immediately to the right of the beam center dominates the calculated phases, fig. 12b is quite similar to the real part of the AC for position A shifted to place the origin on the known atom. Fig. 12c, formed by subtracting 12a from 12b, is known in crystallography as a difference Fourier map. The dominant features are the missing atom and the ghost atom. The correlation coefficient relating the theoretical intensity calculated with the missing atom to the experimental data is 0.10. Inclusion of the missing atom in its correct position yields a correlation coefficient of 0.62, whereas adding the atom at the ghost produces a correlation coefficient of -0.16. In this way known atom positions may be used in conjunction with experimental data to determine the positions of atoms in adjacent regions. These newly acquired atom positions need not be a continuation of the crystalline region.

# 7. Conclusion

Nanodiffraction data may be collected from overlapping regions of crystalline samples with an accuracy that permits the positions of the atoms within the scanned region to be determined with an accuracy of several tenths of an ångström. It is hoped that future application of these techniques will determine with similar accuracy the projected positions of atoms in regions of unknown structure.

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#### References

- [1] J.M. Cowley, Ultramicroscopy 14 (1984) 27.
- [2] J. Konnert and P.D'Antonio, Ultramicroscopy 19 (1986) 267.
- [3] J.M. Cowley, Ultramicroscopy 1 (1976) 255.
- [4] Multislice Computer Program, High Resolution Electron Microscope Facility, Arizona State University, Tempe, AZ 85281, USA.