Electron microscopy of atoms in crystals

Now we can see atoms in crystals directly by electron microscopy, allowing us to determine structures of both ordered and disordered solids and to study the way atoms cluster around crystal defects.

John M. Cowley and Sumio Iijima

From its beginnings the field of electron microscopy has sought, as its ultimate aim, the ability to study the structure of matter by imaging the individual atoms that compose it. The resolution necessary to attain this goal is obtainable in principle, because the electron wavelengths of the beams normally used are less than 0.1 Å. What has kept this goal from realization are practical difficulties in the design and construction of electron microscopes. These difficulties have now been overcome to the extent that resolutions of 2–3 Å, close to the interatomic distances in some solids, have been achieved.

For certain favorable classes of compounds, the structures of crystals and the forms of their defects can be studied directly by imaging the positions of the heavier atoms. This has become the basis for a revolution in the methods of structure analysis, especially for solid-state chemists and mineralogists. A number of the major laboratories around the world are using the high-resolution techniques for the systematic study of defect structures, non-stoichiometry, phase transformations and other solid-state reactions.

Some results of significance to solid-state physics have already appeared as well. Others will proliferate as the improved resolution of the new generation of electron microscopes now being developed in Japan and Europe allows equivalent visualizations of atom positions in metals, semiconductors and simple inorganic compounds. The application of high-resolution electron microscopy to the investigation of the structures of thin crystals constitutes the first of many practical applications of the "atomic resolution" of modern microscopes in the field of materials science.

Individual atoms on the surfaces of crystals were seen first by use of Erwin Müller's field-ion microscopy technique. Valuable though this technique is, the variety of materials and the range of problems that can be studied with it are seriously limited. Individual heavy atoms, sitting on light-atom supporting films, were first imaged by use of the transmission electron microscope about seven years ago by Albert Crewe and his co-workers[1] in Chicago with their dark-field scanning transmission electron microscope. Others soon followed, using the conventional type of microscope in either bright field or dark field.

The production of an image with black or white spots that can be associated with the presence of individual heavy atoms, although an important technical achievement, does not by itself provide any information concerning the structure of matter. The initial aim of much of the work on single-atom imaging, still being actively pursued, was to use heavy atoms attached to particular chemical subunits as markers to indicate the sequencing of these subunits in complex macromolecules of biological significance, such as DNA or proteins. The major difficulty with these projects has been the radiation damage caused by the incident electron beam. This not only destroys the biological material so that the macromolecules themselves may not be imaged, but it also induces motions in the heavy atoms so that the information implied by their positioning is lost. Only in the case of the radiation-resistant phthalocyanines could the shapes of organic molecules, periodically arrayed in crystals, be resolved with anything approaching atomic resolution.[2] As may be expected, experience with single-atom imaging has suggested possibilities for new approaches and new types of applications. In the meantime the virtual absence of a radiation-damage problem has allowed the high-resolution techniques to be applied consistently and routinely to the study of the structures of thin crystals of complex oxides as well as other systems.

Images of niobium oxides

The structures of the niobium oxides and the mixed oxides of niobium with titanium, tungsten and zirconium have long been of interest to solid-state chemists for the wide variety of ordered and disordered superlattice structures that they exhibit. Sometimes a range of compositions can be accommodated within the one crystal structure. Often small compositional variations cover a multitude of different superlattice structures, all having large unit cells with dimensions of 15–40 Å. The frustrations of trying to sort out this complexity by x-ray diffraction methods led David Wadsley of Melbourne, Australia to suggest the use of high-resolution microscopy.

In the late 1960's John Allpress and John Sanders in the CSIRO Division of Tribophysics (Melbourne) showed that the two-dimensional periodicities and some suggestions of characteristic atom groupings could be distinguished in images of thin crystals.[3] The idea that images of crystals could be interpreted directly in terms of structural features was met at first with considerable skepticism because of the well known complications of the dynamical scattering theory for electrons and crystals. However, confidence grew with experience and with confirmation from the careful, systematic exploration of the theoretical basis by

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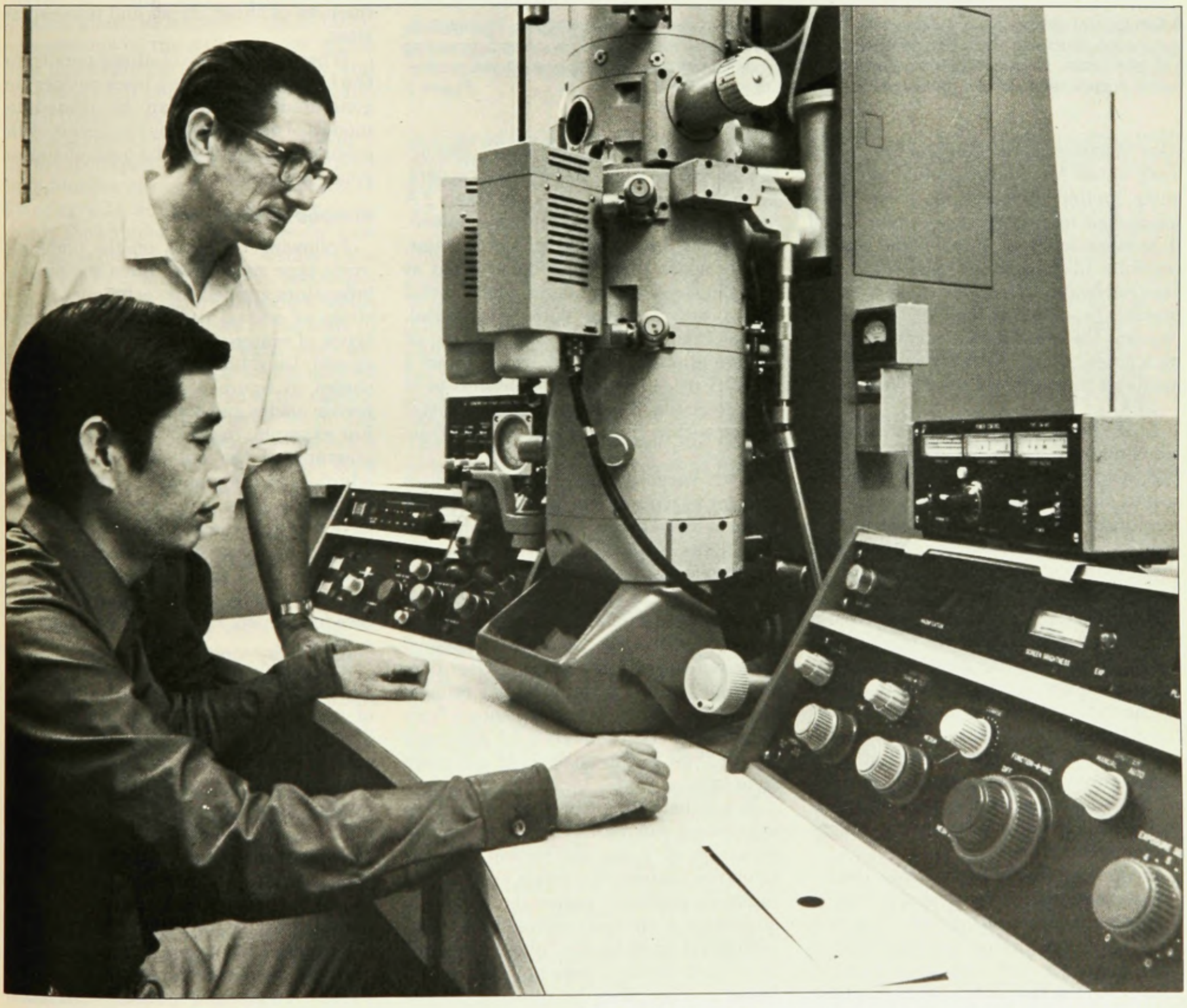
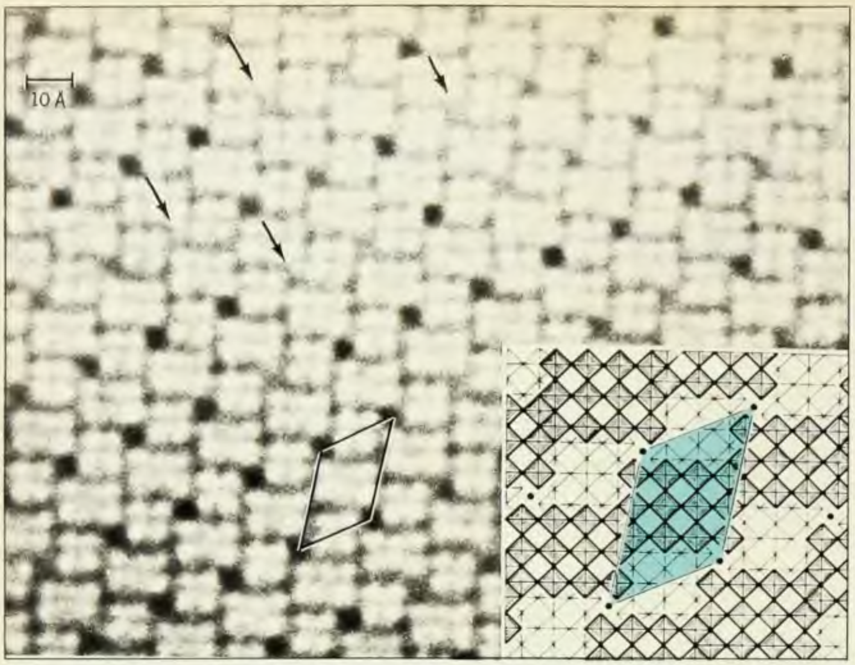


With their electron microscope modified for high-resolution imaging, the authors, John Cowley and Sumio Iijima, have been able to make atoms and structure defects visible in some crystals. The instrument is a standard one equipped with a goniometer stage; this is inside and not visible, but the drives (rectangular boxes mounted on the microscope tube) and the control box (left, above panel) can be seen. Figure 1



A thin crystal of $Nb_{22}O_{54}$ is imaged with the high-resolution electron microscope. The inset diagrams the structure: Each shaded square represents an octahedron of oxygen atoms surrounding a niobium atom; black dots at the corners of the outline unit cell represent niobium atoms in tetrahedral coordination. Arrows show tetrahedral sites of low contrast. Figure 2

Alex Moodie and his collaborators.[3] The work of the Melbourne groups was seriously limited, however, by inadequate microscope resolution. To study a crystal it is essential that its principal axis is carefully aligned to be parallel to the electron beam, so as to give a clear interpretable projection of the structure. This requires the use of a doubly tilting specimen stage, which, with the microscopes available then, limited the resolutions to 7–10 Å.

In 1971 one of the authors of this article, Sumio Iijima, used a specially modified version of one of the newer microscopes, the JEM-100B, to achieve 3.5–4 Å resolution consistently in images of thin crystals. Figure 1 shows the authors with their modified electron microscope at Arizona State University.

A tilting stage permitted Iijima to obtain clear indications of the positions of the metal atoms in the niobium and titanium–niobium oxides, as shown in figure 2. As indicated in the inset diagram of this figure, the structures of these oxides are based on the cubic packing of octahedra of oxygen atoms sharing corners, with one metal atom in each octahedron and a distance of 3.8 Å between neighboring metal atoms.

In the direction of projection—the incident-beam direction—the stacking of these octahedra is well ordered. For each metal-atom position in the projection of the structure a dark spot appears in the image. In the two directions at right angles to the beam direction there are large periodicities created by the periodic occurrence of crystallographic "shear planes," the planes along which the octahedra share edges rather than corners. Here the metal-atom rows are about 2 Å apart in projection and are not resolved, so that the images show lines of broader, darker spots. Big black spots occur at steps in these shear planes where metal atoms are included with tetrahedral, rather than octahedral, coordination of oxygen atoms. Thus the image shows a *directly interpretable representation* of the projection of the distribution of metal atoms within the unit cell of the structure.

With varying metal-to-oxygen ratios and for varying proportions of different types of metal atoms, the shear planes and tetrahedral sites of such structures may occur with various periodicities, giving a wide variety of superlattice structures. Iijima imaged a number of these as well as crystals with the related tetragonal tungsten-bronze structures. After confirming that it is possible to give reliable results by imaging structures known from x-ray diffraction analyses, he extended the method to the analysis of unknown structures.

It will be a long time before electron microscopy can match the resolution and accuracy of x-ray diffraction for the structure analysis of crystals, even for the radiation-resistant materials. One big advantage of electron microscopy is that, with direct imaging, the "phase problem" of x-ray diffraction does not exist. As a result it may often be possible for the initial difficult stage of deducing a rough structure to be done by direct imaging, after which the structure analysis may be refined by x-ray diffraction. Another advantage is that only a few unit cells are needed to provide an interpretable image of a structure: Relative to x-ray diffraction the volume of crystal needed for a structure analysis may be reduced by a factor of 10^{14}.

The most important aspect of this imaging method is that the resolution does not depend on the periodicity of the object. Atoms in crystal defects are seen just as clearly as those in perfectly crystalline regions. For the niobium oxides and related materials the atomic arrangements in crystal faults and localized defects are particularly easy to interpret because the deviations from the perfectly periodic structure are mostly two-dimensional, with perfect order retained in the one dimension parallel to the b axis, which is usually taken as the incident-beam direction. In figure 3, for example, the defects that do not disturb the regularity of the chains of atoms parallel to the beam are readily interpretable in terms of irregularities such as those in the sequences of shear planes and tetrahedral sites.

Where the chains of atoms parallel to the beam are perturbed, black patches are generated. These can be related to models of defects only by comparison with calculations made on the basis of appropriate assumptions.

Bronzes and minerals

Following the work on the niobium-oxide-type structures, Iijima and his collaborators applied his techniques to the study of crystal defects in many other types of materials. These include hexagonal tungsten bronzes such as that shown in figure 4, other non-stoichiometric oxides and a number of minerals.[6] For example, figure 5 is an electron micrograph of the mineral enstatite. In addition to the people at Arizona State University, other groups around the world have taken up the method in recent years and are applying it to a great variety of systems. J. S. Anderson's group at Oxford were early starters and groups in Sweden, Japan, France and Australia are now active.

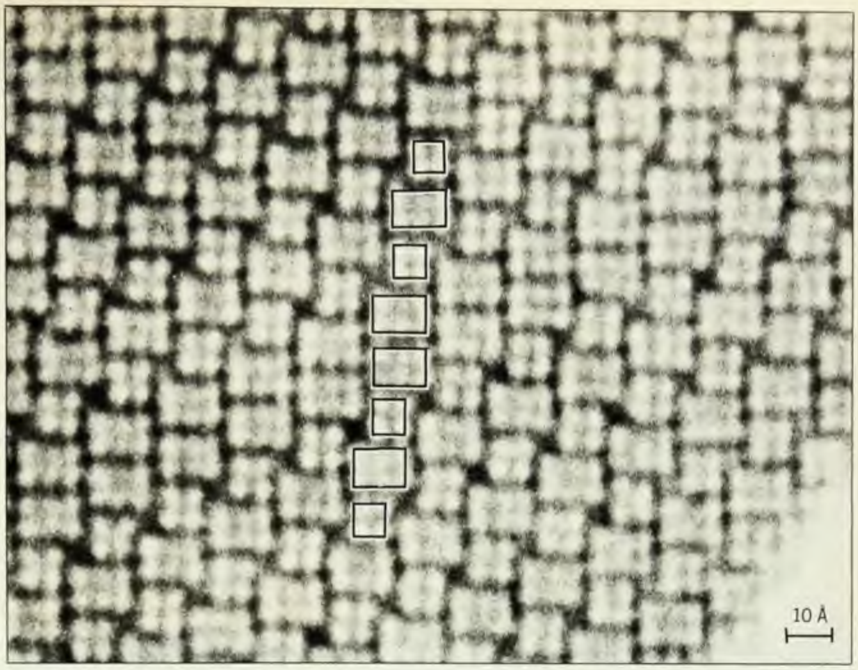
The problems solved with these methods include those on the accommodation of non-stoichiometry in oxide systems. Changes in the metal–oxygen ratio, resulting from changes in the average valency of metal ions or from changes in the relative proportions of two metal ions, may be accommodated by varying the spacing of the shear planes in structures such as those of figures 2 and 3. Because the oxygen octahedra along the shear planes share edges rather than corners, the number of oxygen atoms per metal atom is decreased along these planes. Recent x-ray- and neutron-diffraction work by the Anderson group suggests that the occupancy by metal atoms of the tetrahedral sites in structures such as those of figure 2 may be variable. This is verified by direct observation in the high resolution images. Figure 2, for example, shows large variations of the image intensity at these sites. Although localized "atomic defects" were previously thought not to exist in these oxide systems, Iijima observed isolated spots in images of off-stoichiometric $Nb_{12}O_{29}$ that appear to be localized defects with two excess oxygen atoms each, presumably in interstitial positions.

The atomic rearrangements involved in phase transitions were studied in several cases. Because high-resolution electron microscopy requires the specimens to be in stable configurations, the observations are normally made on specimens quenched during various stages of a phase transformation occurring at high temperatures. Often, however, the strong electron-beam irradiation of the sample by the microscope itself can be used to enhance the diffusional processes, so that the stages of a transformation can be observed at room temperature. This method allows us to deduce the detailed mechanisms of the processes by which new phases are nucleated and grow, and by which shear planes are generated and order themselves.

Metal atoms or defects in a crystal may form superlattice structures, as observed in a number of cases. The methods of imaging with atomic resolution has already shown itself to be a powerful new technique for investigating crystals in the early stages of ordering, such as the potassium tungsten bronze in figure 4, which reveals directly the nature of the state of short-range order. The local atom arrangements in this state, which may be vital for the determination of the electrical and mechanical properties of the crystal, are only partially accessible to the available diffraction techniques. New information on short-range order has been obtained for several oxide systems[5] as well as for a number of minerals.[6]

The fundamental problems of mineral structure being resolved by the new techniques parallel those of the oxides to some extent, but have their own peculiarities as a consequence of the complexity and variety of the compositions and structural types.[6] Questions apart from the problems of non-stoichiometry, the ordering of atoms and the form of defects include

- complicated disorders of stacking sequences such as that illustrated in figure 5,
- microtwinning and
- exsolution (the precipitation of one solid crystalline phase from another, with or without continuity of structure across the phase boundaries).



A stacking fault is evident in this high-resolution electron micrograph of the same niobium oxide as in figure 2. In the fault, 4 × 3 blocks of octahedra are adjacent instead of being separated by 3 × 3 blocks. Note the variable contrast at tetrahedral sites. Figure 3

Theoretical basis of imaging

Only under very specific and well defined conditions can the images of crystals be directly interpreted in terms of projections of the atomic arrangements, and even then reservations are necessary because the representation may be nonlinear and distorted.

The crystals must be thin: For the 100-keV electrons commonly used and resolutions of about 3.5 Å, the thickest crystals that can be used are 50–100 Å. Although the electron scattering is mostly through angles of 0.05 radians or less, the spread of the electron wave gives rise to complicated interference effects between neighboring image points for greater thicknesses. Hence images are usually obtained from the thin edges of minute crystal fragments.

To obtain a recognizable projection of the structure, the crystal axes must be aligned with respect to the incident beam with an accuracy of 2–3 milliradians. This implies that a tilting specimen stage, preferably a goniometer stage, must be used in the microscope. The provision of such a stage, sufficiently stable to give atomic resolution, and its use constitute the principal instrumental difficulties of the technique.

Paradoxically, the images must be recorded out of focus. Because the action of a thin crystal on an electron wave is mainly to change its phase rather than its amplitude, the formation of the image must involve one of the phase-contrast processes, and the easiest and most reliable of these ways of obtaining phase contrast with coherent illumination is to go out of focus. For electron microscopy of very thin, weakly scattering specimens there is an optimum defocus where the phase changes due to spherical aberration and defocus of the objective lens are balanced to give an optimum representation of the projected electrostatic potential distribution of the object.

For current 100-keV microscopes this optimum is an underfocus of about 900 Å theoretically. Because of the finite thickness of the crystals, it is actually somewhat less. In the operation of the electron microscope, this implies that once a specimen region is chosen and properly aligned, a reference point for defocus must be established, such as the minimum-contrast defocus value for a thin amorphous material (about 200–300 Å underfocus); the optimum defocus then must be obtained by adjusting the current in the objective-lens electromagnet. The image is then recorded, usually at a magnification of greater than 5×10^5 on the photographic plate, with an exposure time of 2–3 seconds.

The basis for our confidence in the interpretation of the images is the excellent agreement obtained with theoretical calculations for known structures. For any given crystal structure, image intensities can be calculated with an accuracy far beyond that of present experimental observations, for any values of the experimental parameters of crystal thickness and orientation and microscope aberrations and defocus.

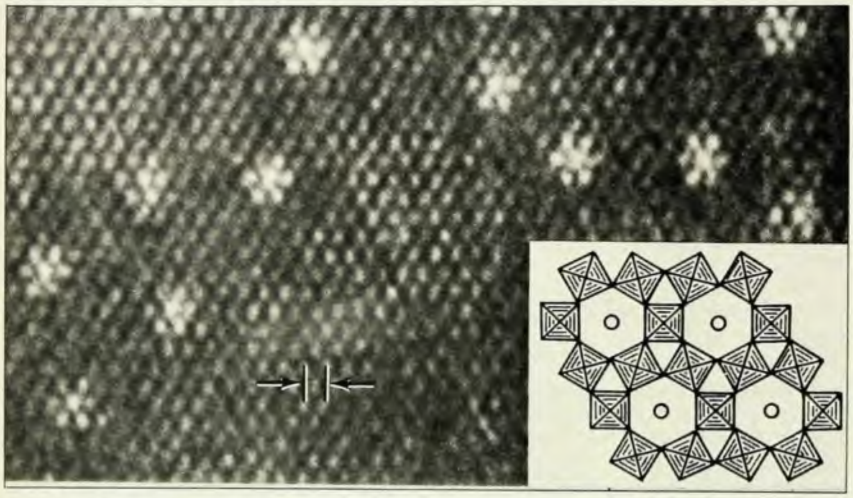
The interaction of the electron beam with the crystal is calculated by use of dynamical diffraction theory for many waves. This is far from the two-beam dynamical theory familiar from x-ray diffraction and often used for electron microscopy of metals. The number of beams coherently interacting in the crystal, n, is normally taken to be 500 to

1000 for a reasonably large unit cell. Of these beams some 50 to 200 pass through the objective aperture of the electron microscope and contribute to the image. The image contrast is therefore obtained by summing a Fourier series in which the coefficients are the amplitudes of these beams, modified in phase by the lens aberrations and defocus.

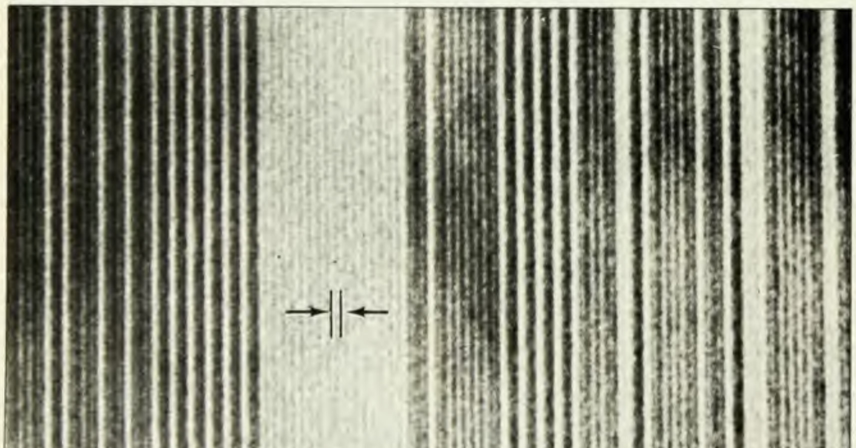
An outline of the computational method, based on the multislice formulation of the dynamical theory[7] is given in the Box on page 38. Although the computations refer to periodic structures, they can be extended to crystal defects by assuming the defects to recur periodically to form a regular superlattice structure. Refinements of the calculations are now made to include the effects of the convergence of the electron beam incident on the specimen and the chromatic aberration of the electron-microscope lenses.

An indication of the level of agreement now common between observed and computed intensity distributions is given by the series of micrographs at varying defocus for $GeNb_9O_{25}$ in figure 6. This comparison was the end point of a refinement of the structure model for this oxide, which involved the determination of the partial occupancies of sites having tetrahedral and octahedral coordination around the tetrahedral positions (dark spots in the image).

In general, the image can be assumed to be a representation of the projected potential distribution in the object—but it is a nonlinear representation. There is a nonlinear, but usually monotonic, variation of image intensity with the number of atoms superimposed in the beam direction. Additional complications of interference between neighboring sites, which occur strongly in the case of $GeNb_9O_{25}$, serve to emphasize that the only way to be confident of image interpretations is by detailed comparison of observed and calculated images.



Clusters of open channels, exhibiting short-range order, are made visible here. The sample is a thin crystal of hexagonal potassium tungsten bronze, $K_{0.2}WO_3$; atomic spacing indicated is 7.4 Å. Such local atom arrangements can only be determined partially by diffraction. Figure 4



An electron micrograph of the mineral enstatite, showing the complicated disorder in the stacking of the monoclinic form, which is multiply twinned. When the twin planes are 9 Å (the distance indicated) apart, the two-layer orthorhombic structure of enstatite is formed. Figure 5

Boosting the resolution

For the images used to illustrate this article the effective resolution is about 3.8 Å. If a field-emission gun had been available to allow more parallel incident electron beams to be used and to provide a smaller energy spread of the electrons, the resolution would have been about 3.5 Å; this would have retained the one-to-one correspondence between the maxima or minima in the image intensity and the atom positions. If the objective aperture were opened wider to allow more diffracted beams to contribute to the image, still finer detail would be seen in the image. This detail, however, would not be a direct representation of object structure because the relative phases of these additional beams would be strongly and erratically affected by the spherical aberration of the objective lens. This is discussed and illustrated in the Box on page 40. For image detail that can be directly interpreted in terms of structure, the resolution limit Δx is given approximately, in terms of the spherical-aberration coefficient C_s and the electron wavelength λ, by the simple formula

$$\Delta x \simeq 0.6\, C_s^{1/4} \lambda^{3/4}$$

which is similar to the familiar formula for incoherent optics but is derived on a different basis.

One way of improving the useful resolution is therefore to decrease the spherical-aberration coefficient. In the last few years the value of C_s for objective lenses in 100-keV microscopes has been reduced from about 3 mm to 0.7 mm, but the ¼ power in the above equation implies that the improvement in resolution is not large.

Alternatively, we could decrease the electron wavelength by increasing the accelerating voltage. Electron microscopes with accelerating voltages of 1 MeV are now common and 3-MeV microscopes are operating in Japan and France, but until recently the difficulties of stabilizing these high voltages have prevented an improvement in resolution. Now high-voltage high-resolution microscopes are being built (with a 500-keV machine in Japan already in operation) and it appears likely that the resolution limit for interpretable images of crystals will soon be reduced to about 1.5 Å.

Powerful new tools

The future improvement of resolution of interpretable images from the present 3.5 Å to about 1.5 Å would broaden the possibilities for application of high-resolution imaging immensely. At the moment it is possible to distinguish the dis-

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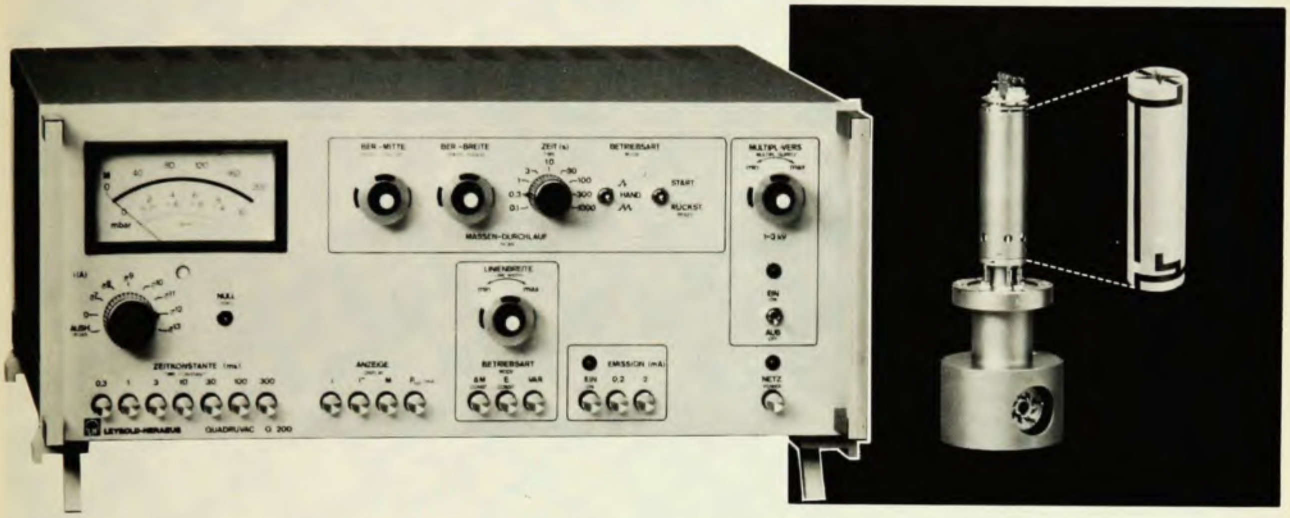

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The calculation of crystal-structure images

The computer programs now available for the calculation of the intensities in high resolution images of thin crystals are based on the multislice formulation of the dynamical theory of electron diffraction.[7] Because electron scattering is very largely forward scattering, the electron wave entering a crystal can be considered as modified by successive slices of the crystal, each a few angstroms thick, extending indefinitely in directions perpendicular to the beam.

Each slice acts as a phase object, modifying the phase of the electron wave by multiplying the incident wave function by a transmission function

$$q(x,y) = \exp[-i\sigma\phi(x,y)]$$

where σ is the interaction constant ($=\pi/\lambda E$, where E is the accelerating potential) and $\phi(x,y)$ is the projection of the potential distribution in the direction of the incident beam, the z direction. Between slices the electron wave is propagated by Fresnel diffraction, which has the effect of convolution of the wave function with a propagation function

$$p(x,y) \simeq (i/c\lambda)\exp[-2\pi i(x^2 + y^2)/c\lambda]$$

where c is the interslice distance.

Then a recursion relation relates the wave function after the $n+1$ slice to that after the n slice:

$$\psi_{n+1}(x,y) = [\psi_n(x,y) * p(x,y)] \cdot q_{n+1}(x,y)$$

The equivalent relationship of diffraction amplitudes is given by Fourier transform

$$\begin{aligned}\Psi_{n+1}(u,v)& \\ &= [\Psi_n(u,v) \cdot P(u,v)] * Q_{n+1}(u,v)\end{aligned}$$

where $P(u,v)$ and $Q(u,v)$ are the Fourier transforms of $p(x,y)$ and $q(x,y)$. For periodic objects the convolution integral becomes a summation over indices h,k of the diffracted beams:

$$\begin{aligned}&\Psi_{n+1}(h,k) = \\ &\sum_{h'}\sum_{k'} \Psi_n(h',k')P(h',k')Q(h-h', k-k')\end{aligned}$$

These summations are readily carried out with a computer and are repeated for each slice to find the electron wave at the exit face of the crystal.[8]

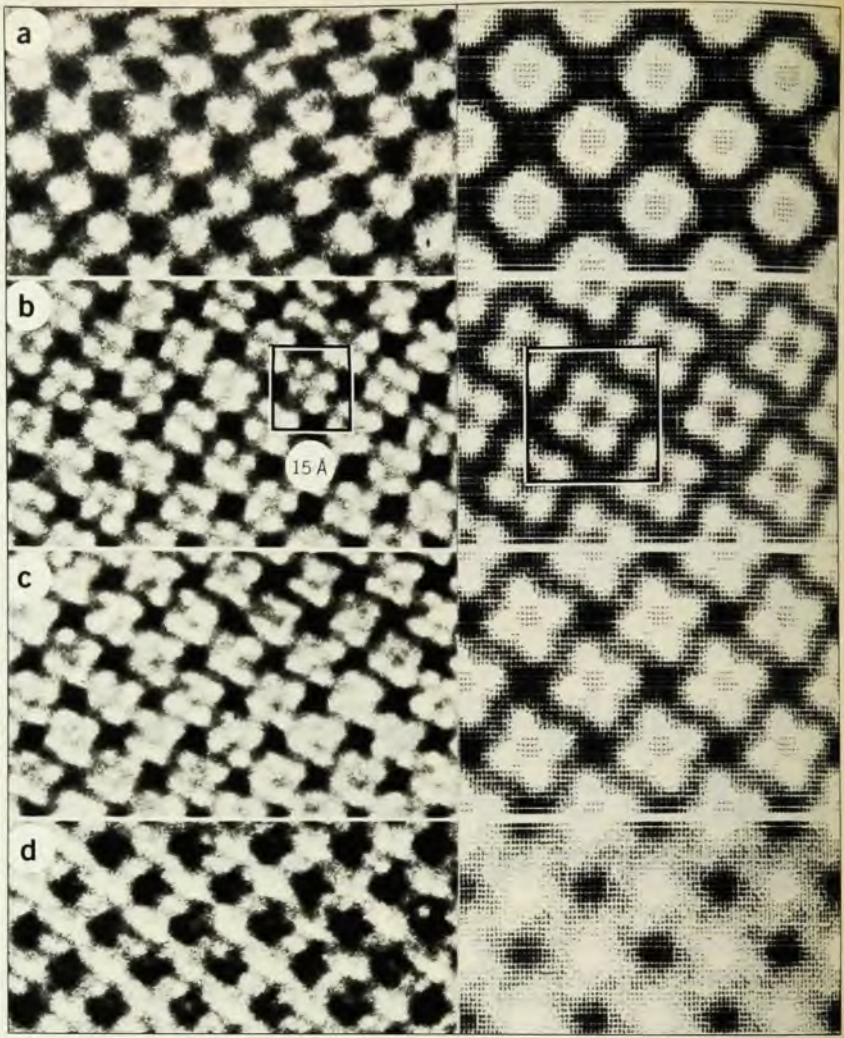
The amplitude distribution of the image is found by modifying these diffracted-beam amplitudes by the phase factors corresponding to the objective lens defocus and lens aberrations and summing the Fourier series

$$\begin{aligned}\psi(x,y) = \sum_h\sum_k \Psi_n(h,k)\exp[i\chi(h,k)]& \\ \times \exp[2\pi i(hx + ky)]&\end{aligned}$$

where

$$\begin{aligned}\chi(x,y) = \pi\Delta f \cdot \lambda(u^2 + v^2)& \\ + \tfrac{1}{2}\pi C_s\lambda^3(u^2 + v^2)^2&\end{aligned}$$

Here Δf is the amount of defocus and $u = h/a$, $v = k/b$; a,b are the lattice periodicities. The image intensity distribution is given by $I(x,y) = |\psi(x,y)|^2$.



Two sets of images at varying defocus values of a "$GeNb_9O_{25}$" crystal (approximate composition; see reference 9). They compare the observed views (left) with their calculated counterparts (right). Electron micrograph **b** shows the disordered occupancy of the tetrahedral sites (dark spots). The defocus values for the pairs are: **a,** −440 Å; **b,** −700 Å; **c,** −950 Å, and **d,** −1200 Å, where the negative sign denotes an underfocus of the objective lens. Figure 6

tributions of heavier atoms only in those favorable cases in which they are separated in projection by more than 3.5 Å. However, in most materials, including metals, alloys and semiconductors, the atom separations in the most favorable projections come within the range 1.5–2.5 Å. Within a few years we can expect to be able to see directly the arrangements of atoms in ordered crystals and around crystal defects for all such materials. This is one of the objectives of the proposed National Center for High Resolution Electron Microscopy, which, it is hoped, will be established at the Lawrence Berkeley Laboratory.

The use of high voltages offers advantages beyond those of resolution. In going from 100 keV to 1 MeV, for example, with the anticipated values for lens parameters, the thickness of crystals that can be used to give interpretable images will increase by a factor of four or five for the same resolution, making it easier to prepare suitable specimens. Furthermore, if the expected resolution gain is achieved, the contrast of bright-field images will be improved. For single heavy atoms the contrast of 10–15% now obtainable will be increased to almost 50%. A comparable improvement will apply to the imaging of localized crystal defects such as isolated vacancies or interstitials.

The difficulties, of course, will not be trivial. The radiation damage caused by the incident electron beam, which now prohibits the atomic-level imaging of biological and organic materials, will also become increasingly serious for inorganic materials as the resolution is improved. However, progress is being made with techniques for maximizing the information gained from the elastic-scattering processes in relation to the damage caused by inelastic scattering.

With improved resolution, the higher contrast produced by small structural

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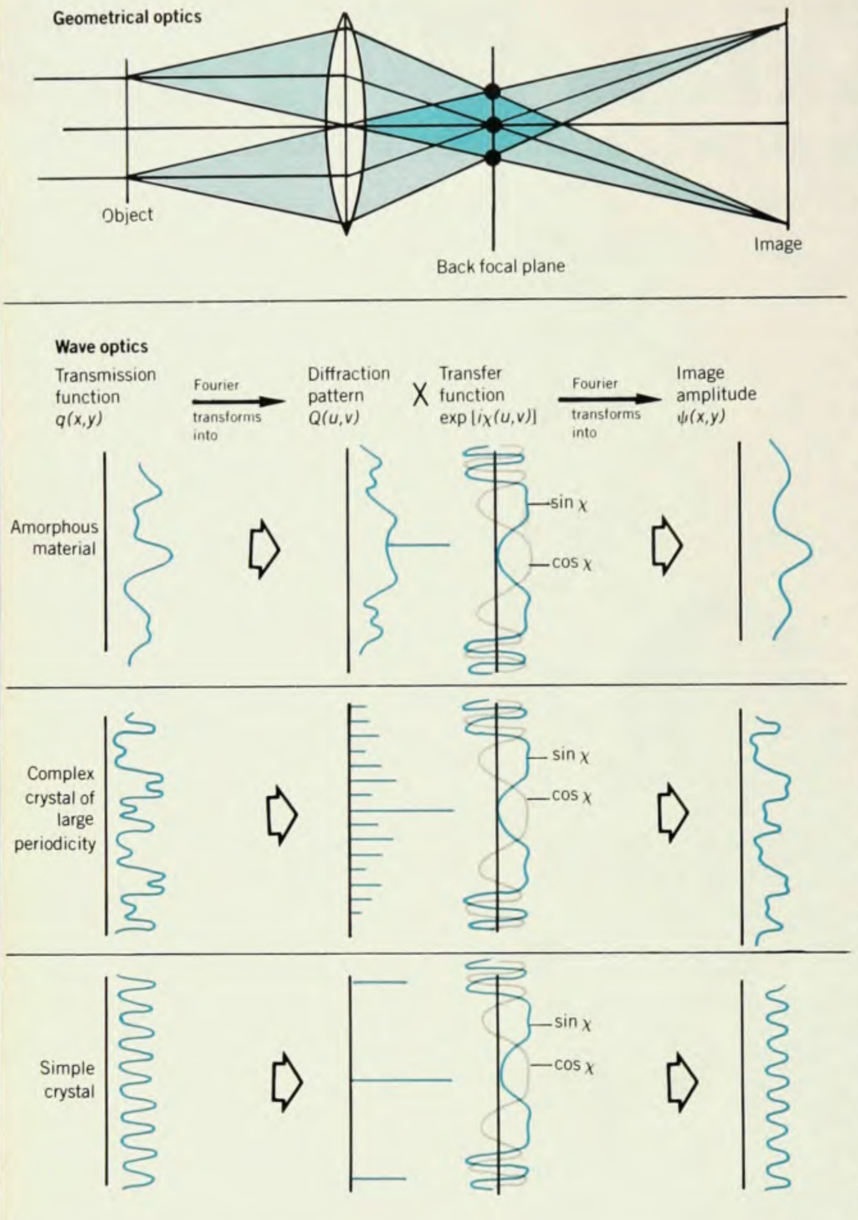

Imaging and resolution

The simple representation of imaging processes by the ray diagram of geometrical optics is familiar. Rays from the same point of the object reach the same point of the image. Parallel rays from different parts of the object are superimposed and interfere (for coherent illumination) in the back-focal plane of the lens, which therefore shows the Fraunhofer diffraction pattern of the transmission function of the object.

In the wave-optical formulation, the transmission function $q(x,y)$ of the object is Fourier transformed to give the Fraunhofer diffraction pattern amplitude $Q(u,v)$ in the back-focal plane. This is multiplied by the phase factor $\exp[i\chi(u,v)]$ due to lens aberrations and defocus. The simplest form for the function $\chi(u,v)$ is

$$\chi(u,v) = \pi\Delta f \cdot \lambda(u^2 + v^2)] + \tfrac{1}{2}\pi C_s\lambda^3(u^2 + v^2)^2$$

where C_s is the spherical-aberration coefficient and Δf the defocus. A further Fourier transform applied to this product then gives the image amplitude. For small radii in the back-focal plane, the function $\chi(u,v)$ varies slowly and the diffracted beams can be recombined to give meaningful image intensities. However, for large radii the real and imaginary parts of the phase factor, $\cos\chi$ and $\sin\chi$, oscillate rapidly. Thus the phases for the higher-order Fourier coefficients (high spatial frequencies) are strongly and erratically modified so that the finer detail of the image due to these beams has little relation to object structure.

The outer parts of the diffraction pattern are usually masked out by an aperture. Then the resolution in the image is limited to a minimum resolvable distance of

$$\Delta x \simeq 0.6 C_s^{1/4}\lambda^{3/4}$$

In favorable cases the image may be directly interpretable in terms of object structure. If strong outer diffracted beams such as those from simple crystals are allowed to contribute to the image, very fine detail (such as lattice fringes with spacings of 1 Å or less) may be visible in the image, but the image intensity has no clear relationship to atom positions in the specimen.

defects implies also that the irregularities of specimen surfaces may have increased influence on the images. While this may be an unwanted complication in efforts to study defects within crystals, it will on the other hand enhance the power of transmission electron microscopy enormously as a tool for the study of surface structure.

Already at 100 keV, single heavy atoms sitting on crystal surfaces and surface steps one atom high have been observed with a lateral resolution of 3–4 Å, although so far this has been achieved only with low contrast and under specially favorable circumstances.[10]

The development of high-resolution microscopes specifically for the observation of surface structure and reactions could provide powerful new tools for surface physics and chemistry.

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The authors won the Bertram E. Warren Award of the American Crystallographic Association; this article is an adaptation of their addresses to the Association in response to the Award, given 9 August 1976 at Northwestern University, Chicago.

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