X-RAY MICROSCOPY

A small step to higher resolution

A new kind of X-ray microscopy can visualize single-unit-cell steps on a crystal surface. This is an order of magnitude better depth-resolution than current X-ray microscopes can achieve.

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The history of X-ray microscopy goes back almost as far as the discovery of X-rays, but its development has been hampered by many technical challenges. The greatest of these arises from the fact that the refractive index of most materials to X-rays is close to unity, making it virtually impossible to create even the most rudimentary refractive X-ray lens. With advances in microfabrication techniques over the past four decades, though, it is now possible to construct diffractive optical elements, such as Fresnel zone plates (FZP)[1], which act as X-ray lenses. Such advances along with the arrival of high-intensity synchrotron X-ray sources have driven considerable recent progress in this field[2,3]. On page 700 of this issue[4], Fenter *et al.* report the next step towards the development of atomic-scale X-ray microscopy by demonstrating a technique that can resolve height variations as small as a single unit cell (0.65 nm) in an otherwise flat crystal surface. Although the lateral resolution of this approach is much lower — hundreds of nanometres, compared with the 15–30-nm resolution achieved by other approaches[5] — the ability to monitor molecular-scale height variations could have important implications for the study of surfaces under harsh physical or chemical conditions.

Although resolution is the principal attribute usually considered when assessing the performance of any imaging technique, it is certainly not the only limiting factor. Image contrast — the ability to clearly distinguish features within an object or from its surroundings — must also be considered. It is no use having excellent spatial resolution if the resolved features are invisible! In this regard the history of X-ray imaging has followed a similar path to that of light microscopy. In both cases, contrast is most simply generated by the absorption of radiation by a sample, and so the first way to improve it is to increase this absorption — in the case of light by staining the specimen with an absorbing chemical that is preferentially concentrated in certain features of an object, and in the case of X-rays by the presence of heavy elements that do the same. But for many transparent specimens, staining may not be practical

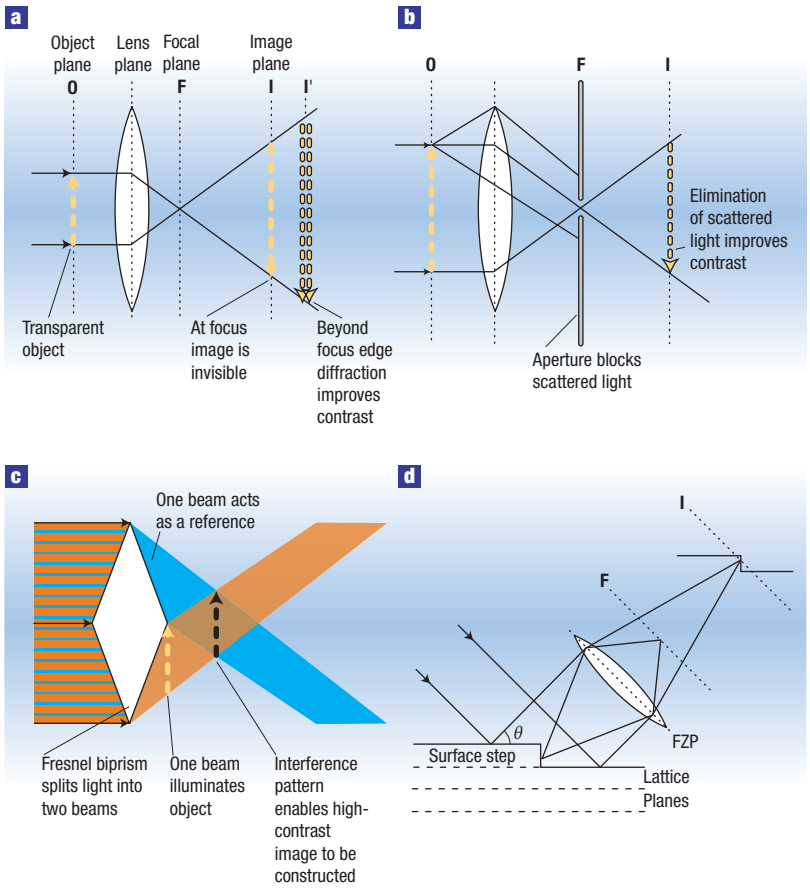

Figure 1 Techniques for improving contrast in a microscope image. **a**, Bringing the image of a transparent object into perfect focus can make it barely visible. Pushing it just beyond focus creates Fresnel diffraction fringes at its edges (and other discontinuities) that generate contrast. **b**, Blocking the passage of diffuse scattered light from an object by placing an opaque aperture at the focal point of the system reduces the amount of light contributing to its image, improving its contrast relative to the surrounding bright field. Similar improvements can be realized by replacing the opaque aperture with a transparent phase-shifting plate, which gives rise to Zernike phase contrast. **c**, The sensitivity of optical interference to phase shifts introduced by the passage of light through most materials provides a powerful technique for generating contrast. Spatial shifts in the interference fringes produced by splitting a beam in two (with, for example, a Fresnel biprism), passing one part through an object and recombining, can be used to reconstruct high-contrast images. **d**, The approach taken by Fenter et al. is an interferometric one. Although the exact mechanistic details are uncertain, this approach generates contrast from a subnanometre size step in an otherwise flat surface by controlling the interference of light reflected from either side of the step.

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or desirable. In such instances, the next level of sophistication involves taking advantage of the fact that the features within a sample not only absorb radiation (however poorly), they also slow its passage. Although the difference in intensity of the radiation transmitted through (or reflected from) different parts of a sample — which is what is used to generate conventional microscope images — may be small, the differences in the speed with which it passes through different parts of an object (as a consequence of either thickness or compositional variations) can be significant. Such differences are manifest in changes to the phase of the radiation used to image a sample. The trick is to find a way of converting this phase information — which cannot be measured directly — into variations in the intensity of a formed image, also known as 'phase contrast'.

In an ideal optical system, the optical field formed at its image plane will be generated by a fine balance of the spatial Fourier components of the light passing through the system. Consequently, any perturbation of this balance introduced by the optical system should result in contrast from even very subtle features of a transparent object. Three conventional means of enhancing the contrast generated by such perturbations are illustrated in Fig. 1a–c. The first two (Fig. 1a,b) involve relatively straightforward adjustments to a conventional imaging system, whereas the third (Fig. 1c) is based entirely on interferometry, in which phase shifts in the wavefront are displayed as fringe displacements.

The method used by Fenter *et al.*[4], illustrated in Fig. 1d, is somewhat different. Being a surface imaging technique, it uses a reflection rather than a transmission configuration. Also, it is specific to a crystal surface. As is well known, X-ray reflection from a perfect crystal is concentrated in specific directions for which the glancing angle θ obeys Bragg's law. These angles are such that the phase difference between waves reflected from successive lattice planes is 2π (or an integer multiple). The same will apply to the wavefronts reflected from either side of the single-unit-cell step shown in Fig.1d. Halfway between these angles — at the 'anti-Bragg conditions' — the phase differences will be π (or an odd-integral multiple), and interference between the wavefronts adjacent to the step should give a decreased intensity, or in other words a (negative) step contrast. (We note that the perfect-crystal reflection conditions are relaxed at a surface, which breaks the ideal infinite crystal periodicity, so that there is a very small but finite reflectivity at such off-Bragg angles.)

Such contrast is observed by Fenter *et al.*[3] in images of a cleaved specimen of orthoclase, and their explanation, along the above lines, is backed by quantitative measurements of contrast as a function of θ, which were consistent with the behaviour of a single-unit-cell step. Deeper consideration of the details suggests that such a straightforward explanation is not the whole story. Indeed, if the authors' setup behaved as an ideal imaging system, it should generate no contrast at all in the image plane I (see Fig. 1d), as for a pure phase object. But of course the system is not ideal, and this is undoubtedly essential for generating the contrast they observe. In particular, the width of the step image is around 200 nm. This relatively low resolution indicates a loss of high-spatial-frequency components of the image. This would have the effect of 'smearing' (convolving) the image with a point-spread function, effectively allowing for the interference of adjacent parts of the image at the image plane. As the authors acknowledge, a proper treatment will include details of illumination, surface topography, imaging optics, including aberrations, and possible defocus.

Other techniques do exist for imaging unit-cell surface steps, notably atomic force microscopy and reflection electron microscopy. But the ability of X-rays to access surfaces or interfaces in a range of environments *in situ* could make this method, which the authors have termed the X-ray reflection interface microscope (XRIM), a useful addition to available surface analysis techniques.

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