SHORT COMMUNICATIONS

Observation of the soft X-ray diffraction pattern of a single diatom.

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Abstract

Part of the diffraction pattern of a single biological cell (diatom) has been observed using 32 Å synchrotron X-rays.

Historically, obtaining diffraction patterns capable of serving as a basis for detailed X-ray structure analysis has depended on the existence of a macroscopic single crystal or ordered fiber of the material in question. More recently, with the advent of high-intensity synchrotron radiation sources, several investigators have shown that it is possible to obtain such patterns from a microscopic single crystal (Bachmann, Kohler, Schulz & Weber, 1985). In either case, the existence of a large number of identical or nearly identical copies of the structure being studied is required.

In earlier publications (Sayre, 1982; Sayre, Haelbich, Kirz & Yun, 1984) we suggested that the use of longer-wavelength X-rays from synchrotron sources might permit the recording of the diffraction pattern of a general microscopic specimen, and that this might in turn open the way to detailed imaging of structures which do not occur in nature in accurately duplicated form. Important examples of such structures are whole cells (or major cell substructures) in biology and numerous types of microparticles in the materials sciences. The longer wavelengths would preclude imaging at atomic resolution, but at the wavelengths suggested (*ca* 30 Å) the resolution would still be approximately 15 Å if the entire diffraction pattern could be obtained and used.

In this note we report the fact that using 32 Å radiation from the National Synchrotron Light Source (NSLS) at Brookhaven we have recently recorded part of the diffrac-

tion pattern of a single biological cell (diatom). The apparatus used is shown schematically in Fig. 1.

32 Å photons from the lower-energy (750 MeV) NSLS ring were formed into a 20 μm diameter beam by a focusing toroidal grating monochromator, slit and pinhole. Two further pinholes were carefully positioned to minimize the radiation reaching the detector after scattering from pinhole edges. The specimen was mounted on an electron-microscope grid and placed on the collimator axis with the aid of an optical microscope. Fig. 2 shows an optical micrograph of a specimen similar to the one used, showing the relationship between the specimen and the area illuminated by the 20 μm beam. The diffraction pattern was recorded on a Kodak type 101 low-gelatine UV spectrographic plate, the plate itself serving as the beam stop. The distance from the first pinhole to the specimen was approximately 34 mm and that from the specimen to the plate was 20·3 mm. The toroidal grating and slit were in ultra-high vacuum, and the collimator, specimen and detector were in moderate vacuum.

Fig. 3 shows the diffraction pattern obtained (Fig. 3*b*), together with evidence that the pattern rotates correctly with rotation of the specimen, and appears and disappears correctly with insertion and removal of the specimen and/or filters. In addition, the major feature of the diffraction

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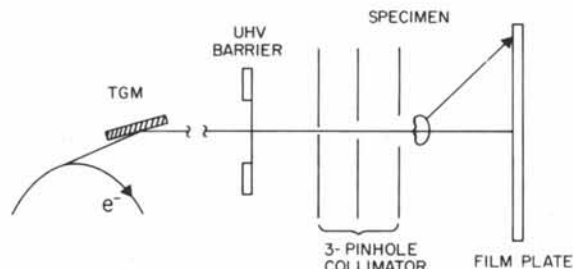

Fig. 1. Schematic drawing of the experimental arrangement. The apparatus was installed at NSLS beamline U15 at Brookhaven.
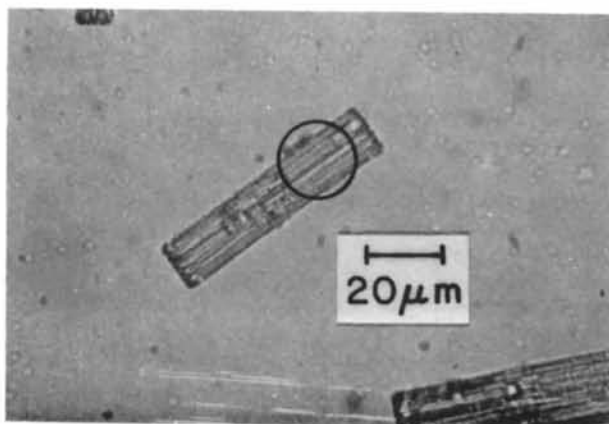

Fig. 2. Optical micrograph of a specimen similar to that used, with the area illuminated by the 20 μm X-ray beam indicated. The specimen is shown in the orientation used in recording Figs. 3(*b*) and (*d*). The longitudinal striations, just visible here, can be seen in SEM images to be lines of holes in the diatom surface, with an average inter-hole spacing of approximately 2010 Å.

pattern, namely the lines showing a strong spacing at 2030 Å normal to the diatom axis, is consistent with scanning electron microscope images of specimens similar to that which produced the pattern of Fig. 3; these show the surface of the diatom to be dotted with lines of holes, the lines being parallel to the diatom axis and the average measured inter-hole spacing being 2010 Å. (There are also suggestions in the SEM pictures that the holes are correlated with additional deeper-lying structure in the diatom.) Exposure times for Fig. 3 were approximately $\frac{1}{2}$ h with 100 mA of beam current circulating in the synchrotron ring.

A negative aspect of the findings to date is the fact that the patterns thus far recorded do not extend to spacings beyond approximately 400 Å, although on the basis of the diffraction limit they should extend to 16 Å. We have made computer simulations which suggest that the problem can be cured by increasing the spatial and temporal coherence of the X-rays used, or by decreasing the specimen size, and we hope to improve our apparatus to permit this to be tested. This problem is not due to an atomic scattering-factor effect, since with soft X-rays the atomic scattering factor does not fall off appreciably with increasing scattering angle (Henke, 1981).
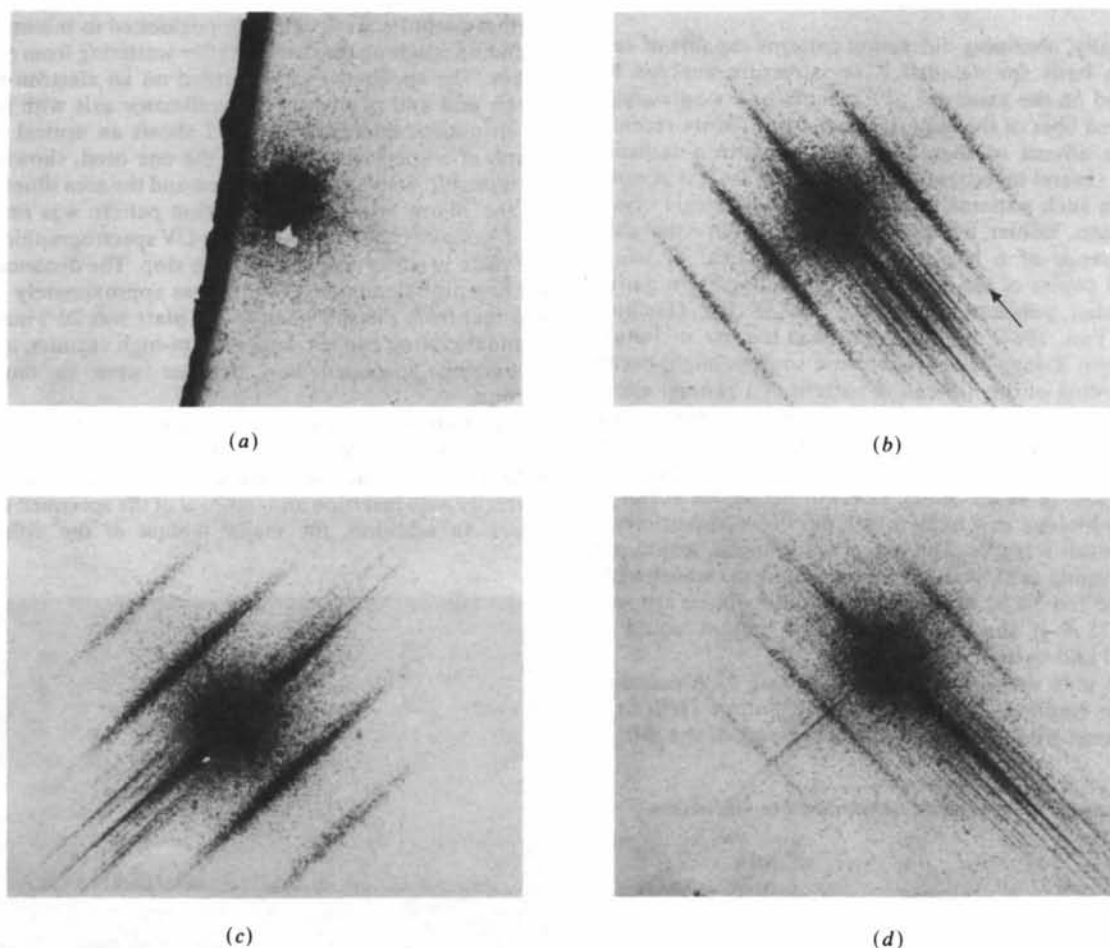
(a)

(b)

(c)

(d)

Fig. 3. (a) The pattern recorded with collimator but no specimen in place. The line to the left of the beam spot is the edge of the photographic plate. (b) The pattern with specimen added. (c) The specimen was rotated by 90° about the beam axis; the pattern has rotated similarly. (d) A 700 Å-thick aluminium foil was placed between specimen and detector. Such a foil is opaque to visible and UV light but transmits 32 Å X-rays. The pattern is essentially unchanged. Not shown is an additional photograph in which a filter consisting of a piece of clear plastic film was placed between specimen and detector. This transmits visible light but absorbs UV and soft X-rays. In this case no signal was recorded by the detector. The diffraction-pattern feature indicated by the arrow in (b) corresponds to a spacing of approximately 2030 Å, compared with an average spacing of 2010 Å for features which are visible in SEM images of similar specimens (see text and Fig. 2). The pattern extends to spacings of approximately 400 Å, or maximum scattering angle 2θ of approximately 4·5°.

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Nonperiodic tessellation with eightfold rotational symmetry. By Y. WATANABE, M. ITO and T. SOMA,

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Abstract

A tessellation with eightfold rotational symmetry was obtained by a self-similar subdivision of unit cells into rhombi and squares.

Early algebraic work to generate a two-dimensional quasi-lattice like the Penrose tessellation (Penrose, 1974) was carried out by de Bruijn (1981) and Mackay (1982). Since the discovery of a quasi-crystal of an Al–Mn alloy (Schechtman, Blech, Gratias & Cahn, 1984), several model structures of the quasi-lattice and its diffraction pattern have been studied by many authors (Hiraga, Hirabayashi, Inoue & Masumoto, 1985; Kimura, Hashimoto, Suzuki, Nagayama, Ino & Takeuchi, 1985; Levine & Steinhardt, 1984, *etc.*). There are two methods of obtaining a quasi-lattice; one is a projection of cubic cells in a higher-dimensional space to a lower one (Kramer & Neri, 1984; Duneau & Katz, 1985), and the other is a self-similar subdivision of unit cells constituting the tessellation (Mackay, 1982; Ogawa, 1985)

We obtained a tessellation with eightfold rotational symmetry by a self-similar subdividing operation of two kinds of cells, a rhombus and a square. Each unit cell is derived by dividing a regular octagon, which is uniquely divided into 16 rhombi and eight squares with eightfold symmetry at the center. In this subdividing operation the rhombus is divided into four squares and six rhombi, and the square into six squares and eight rhombi respectively (Fig. 1). The first-generation pattern of the square has a rhombic
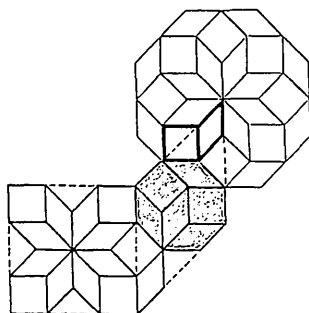

Fig. 1. First-generation pattern derived from division of a regular octagon.

assembly with eightfold symmetry in its center and that of the acute rhombus includes a regular sub-octagon which consists of two squares and four rhombi, as shown by the shaded area of Fig. 1, having mirror symmetry. Therefore eight different tessellations could be derived using the two kinds of subdivided unit cells obtained by rotating the sub-octagon about the center. The tessellation after n self-similar subdividing operations is called the nth-generation pattern.

The ratio of the number of squares to rhombi for the nth generation with n tending to infinity is obtained by solving a recurrence formula. Let S_n be the number of squares and R_n be the number of rhombi in the nth generation. Then the recurrence formula written in matrix form is

$$\mathbf{X}_n = A\mathbf{X}_{n-1} = A^n\mathbf{X}_0. \tag{1}$$

Here $\mathbf{X}_n$ is a column vector given by

$$\mathbf{X}_n = \begin{bmatrix} S_n \\ R_n \end{bmatrix} \tag{2}$$

with $S_0 = 1$ and $R_0 = 1$, and A is a matrix

$$A = \begin{bmatrix} a_{11} & a_{12} \\ a_{21} & a_{22} \end{bmatrix} \tag{3}$$

with $a_{11} = 6$, $a_{12} = 8$, $a_{21} = 4$, $a_{22} = 6$. After a simple calculation we obtain A^n:

$$A^n =$$

$$\begin{bmatrix} \frac{1}{2}\{(6+4\sqrt{2})^n + (6-4\sqrt{2})^n\} & \frac{\sqrt{2}}{2}\{(6+4\sqrt{2})^n - (6-4\sqrt{2})^n\} \\ \frac{\sqrt{2}}{4}\{(6+4\sqrt{2})^n - (6-4\sqrt{2})^n\} & \frac{1}{2}\{(6+4\sqrt{2})^n + (6-4\sqrt{2})^n\} \end{bmatrix}.$$

$$\tag{4}$$

The ratios of the number of nth-generation squares to rhombi are s_n in a square, r_n in a rhombus, and t_n in a pattern of both added together, where

$$s_n = a_{11}^{(n)}/a_{12}^{(n)}, \qquad r_n = a_{21}^{(n)}/a_{22}^{(n)},$$

$$t_n = (a_{11}^{(n)} + a_{21}^{(n)})/(a_{12}^{(n)} + a_{22}^{(n)}). \tag{5}$$

If one substitutes the relevant matrix elements of (4) into (5), and lets n approach infinity, then the ratios s_n, r_n and t_n converge to $\sqrt{2}/2$. This irrational number proves that the