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EXPERIMENTAL OBSERVATION OF DIFFRACTION PATTERNS FROM MICRO-SPECIMENS*

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Experimental Observation of Diffraction Patterns from Micro-Specimens

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1. INTRODUCTION

In an earlier paper [1] we calculated the photon scattering cross-section of a small specimen, averaged over the elementary Shannon volumes in diffraction space, as $r_0^2 \lambda^3 m Z^2$ for the Shannon volumes in the total Ewald sphere (case of multiple orientations of the specimen in the beam, and full 3-dimensional imaging), and a/λ times that for the Shannon volumes on a single Ewald sphere (case of a single orientation in the beam, and partial 3-dimensional imaging). Here r_0 is the classical electron radius, λ is the photon wavelength, m is the mean number of atoms per unit volume of the specimen, Z is the rms atomic number of the atoms of the specimen, and a is the specimen diameter. By a small specimen is here meant one for which a is less than an absorption length for the photons. The elementary Shannon volumes pick out the informationally distinct scattering directions for the general (i.e. non-periodic) small specimen considered here. In the soft x-ray region nearly all scattered photons are scattered without energy loss, and the scattered photon pattern is conveniently spoken of as a diffraction pattern.

These formulae, assuming they are correct, are basic to x-ray imaging microscopy and holography as well as diffraction imaging, as all three methods are scattered-photon imaging methods. Part of the purpose of this paper is to cite evidence that the formulae are at least generally correct.

One piece of evidence was supplied by YUN [2]. The diffraction pattern from a uniform sphere is known analytically [3]; Yun constructed (in the computer) a model specimen consisting of uniform protein spheres of radius 1.25nm placed at random in a water sphere of radius 40nm, and for a given orientation of the specimen numerically calculated its diffraction pattern, on an absolute scale, over a fairly large range of diffraction space (Fig. 1). Yun showed that the average number of photons per Shannon volume for this diffraction pattern is in good agreement with the second of the formulae above.

Based on these formulae, one may calculate expected exposure times for the observation of diffraction patterns. For a mean 5% error in observing diffraction amplitudes (mean of 100 photon counts per Shannon volume), one obtains an estimate of 2 minutes for a single orientation of a 1 μ m biological specimen on a soft x-ray undulator source like that being installed on the

Fig. 1. Part of the calculated diffraction pattern of a model protein-water specimen

X1 beamline at NSLS, and 1/2 day for collecting the total Ewald sphere. Note that the latter figure is independent of specimen size; as the specimen size decreases, the exposure per orientation increases, but the number of orientations required decreases. These estimates strike us as reasonably favorable for the future of scattered-photon imaging.

2. OBSERVATION OF PATTERNS USING THE U15 BEAMLINE

In the present paper, the experimental results reported are from a lower-brightness source, the bending-magnet source on the U15 beamline at NSLS. For this source the exposure-time estimate for a single orientation for a $1\mu\text{m}$ biological specimen was 10 days, which was deemed impractical. For this reason the specimens used in this paper were larger ($a = 10$ to $20\mu\text{m}$). Assuming the formula for the single orientation case to be correct, we expected to be able to observe diffraction from these specimens with 1/2- to 1-day exposures.

This proved to be correct. An apparatus was built [4] in which 3.2nm photons from the U15 monochromator were passed through a carefully aligned 3-pinhole collimator to form a $20\mu\text{m}$ beam with minimal stray radiation outside the beam. The specimen was placed in this beam and the scattered photons allowed to fall on a Kodak type 101 soft x-ray silver halide plate placed several centimeters downstream from the specimen. Fig. 2 shows the pattern observed after a few hours exposure with a diatom with a diameter of $20\mu\text{m}$ as specimen. Evidence that the pattern is a diffraction pattern is that it (a) rotates with the specimen, (b) disappears with removal of the specimen, (c) disappears with the insertion of a soft x-ray opaque filter ($1\mu\text{m}$ mylar), (d) is unchanged by the insertion of a soft x-ray transparent filter ($0.1\mu\text{m}$ aluminum), and (e) contains prominent maxima which correspond to a spacing of 203nm, corresponding to prominent features in SEM images of the specimen with average spacings of 201nm. Patterns were also recorded with similar exposure times with muscle and bone as specimens. These patterns are believed to be the first such patterns recorded from microscopic, non-crystalline specimens.

Fig. 2. Diffraction pattern of a diatom in 3.2nm x-rays

3. COHERENCY CONSIDERATIONS

A second feature of the U15 patterns also supports the belief that our understanding of the diffraction signal intensity is essentially correct. For the diffraction pattern to have good visibility at diffraction angle θ , the uncertainty in position in diffraction space at angle θ caused by small variations $\Delta\lambda$ in the wavelength and $\Delta\theta_0$ in the direction of arriving photons must be less than the size of a Shannon volume. Quantitatively [2]:

$$2(1 - \cos \theta)(\Delta\lambda/\lambda)^2 + (\Delta\theta_0/\lambda)^2 \leq (1/a)^2, \quad (1)$$

or for small θ :

$$\theta^2 \leq (\lambda/\Delta\lambda)^2 [(\lambda/a)^2 - (\Delta\theta_0)^2].$$

Inserting values of $\Delta\lambda$, $\Delta\theta_0$, λ , a for the U15 experiment, we conclude that the diffraction pattern should not be visible in that experiment at diffraction angles θ greater than about 5° . The results of the U15 experiment confirm this conclusion.

This point, confirmatory as it is of the theory, raises the question whether, in an X1 experiment, we may expect diffraction to be observable to the full diffraction angle of 180° . For $\lambda = 3.2\text{nm}$, here are two sets of parameter values satisfying (1):

	(A)	(B)
Beam divergence $\Delta\theta_0$ (mr)	.1	1
Monochromaticity $\lambda/\Delta\lambda$	2000	600
Specimen size a (μm)	3.2	1
Maximum θ	180°	180°

Set (B) is well within the design range of the X1 beamline, and set (A) is about at the limit of the design range. Taken with the earlier definition of a small specimen and estimates of exposure time, X1 should thus permit

observation of soft x-ray diffraction patterns from small specimens to $\theta = 180^\circ$.

4. SUMMARY

The findings of this paper, partly experimental and partly theory, are that even small non-periodic specimens scatter soft x-rays strongly enough to permit scattered-photon imaging. For the type of imaging principally studied here (diffraction-pattern imaging), the requirements on divergence and monochromaticity of the illumination are also moderately well understood, and appear to be capable of being sufficiently well met by undulator beamlines to permit imaging to the diffraction-limited resolution of $\lambda/2$, provided of course that a method of phasing the diffraction pattern is available. It may be remarked that for holography the illumination requirements, while varying with the form of holography, are more severe than for diffraction-pattern imaging, while for imaging microscopy they may be considerably less severe, depending on the angular and frequency bandwidths of the imaging element. It would appear to be for this reason that imaging microscopes are currently achieving exposure times of a few seconds even on bending-magnet beamlines [5].

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