Implementation of diffraction-enhanced imaging experiments: at the NSLS and APS

Z. Zhong\textsuperscript{a,*}, W. Thomlinson\textsuperscript{b}, D. Chapman\textsuperscript{c}, D. Sayers\textsuperscript{d}

\textsuperscript{a}National Synchrotron Light Source, Brookhaven National Laboratory, Upton, NY 11973-50000, USA
\textsuperscript{b}European Synchrotron Radiation Facility, B.P. 220, 38043 Grenoble, France
\textsuperscript{c}CSRI, Illinois Institute of Technology, 3301 South Dearborn, Chicago, IL 60616, USA
\textsuperscript{d}Physics Department, North Carolina State University, Raleigh, NC 27695, USA

Received 4 January 2000; accepted 14 February 2000

Abstract

Diffraction-enhanced imaging is a recently developed X-ray imaging technique that has demonstrated enhanced contrast for dense, highly absorbing materials of interest in materials science and medicine. The implementation of this technique in experiments at the National Synchrotron Light Source and at the Advanced Photon Source is described in detail. © 2000 Elsevier Science B.V. All rights reserved.

PACS: 87.59.-e; 87.62.+n; 07.85.Qe

Keywords: Radiography; Diffraction-enhanced imaging; Rocking curve

1. Introduction

Diffraction-enhanced Imaging (DEI) is a recently developed imaging technique for radiography of dense, highly absorbing materials that uses monochromatic X-rays to probe the internal structure of a thick object [1,2]. In addition to the absorption of X-rays (either monochromatic [3–5] or poly-chromatic) as a contrast mechanism, DEI is sensitive to the refraction contrast [6–9] and extinction contrast [1] (contrast due to the rejection of small-angle scattering from the sample). This technique has demonstrated enhanced image contrast for selected materials of interest in materials science and medicine [10–14]. Experiments have been carried out at the National Synchrotron Light Source (NSLS) at low energies (less than 30 keV), and at the Advanced Photon Source (APS) at higher energies (up to 60 keV).

Successfully implementing of DEI involves considering the available flux and crystal optics, and eliminating vibration [15]. During the last three years, through trial and error, a stable, practical DEI system was developed at the NSLS and experiments were conducted at both the NSLS and the APS. This paper describes in detail the establishment of DEI at both synchrotron facilities. We hope, hereby, to facilitate its use by other researchers, free from the pit falls that we experienced.
2. General considerations

In a DEI experiment, a fan beam of monochromatic X-rays is generated by a double-bounce crystal monochromator. This beam then traverses the sample and is diffracted by an analyzer crystal. The analyzer is non-dispersive with respect to the monochromator’s crystal and can diffract either in a Laue- or Bragg-mode. Discussion will be limited to the latter since this is the configuration used in most of our experiments.

The refraction of X-rays by the sample causes them to deviate from the original direction by a small amount, $\Delta \theta_z$ (on the order of $10^{-7}$ rad). When the analyzer is tuned to the shoulder of its rocking curve, this refraction changes the intensity ($\Delta I$) of the X-rays diffracted by the analyzer crystal, giving rise to the refraction contrast (Fig. 1). The refraction contrast depends on the angular width of the analyzer rocking curve and on the analyzer’s position on the rocking curve.

Extinction contrast is another type of contrast revealed by DEI. The range of angles which can be accepted by the analyzer is of the order of micro-radians. Therefore, the analyzer rejects small-angle scattering at the micro-radian level, which is below the capabilities of conventional anti-scatter grids. This scattering intensity, normally appearing in the image, is missing and appears as absorption in the image. Specifically, the scattering from the sample is effectively rejected when the analyzer is on the peak of the rocking curve, but is picked up when the analyzer is off the peak position. Thus, DEI can be optimized to either reject or pick up the scattered X-rays for a specific type of small-angle scattering, thus producing contrast between samples which are similar in absorption but have different small-angle-scattering properties.

Both the refraction and extinction contrast originate from the extremely small angular width of the analyzer’s rocking curve which is characterized by the Darwin width of perfect crystal diffraction. The Darwin width corresponds to a spreading of the reciprocal lattice points in the direction normal to the diffracting planes due to the limited depth of X-ray penetration, and thus, to the limited number of lattice planes participating in diffraction. The Darwin width for a symmetric diffraction is calculated from [16]

$$\omega = \frac{2\lambda}{\pi A_s \sin 2\theta_B}$$  \hspace{1cm} (1)

where $\theta_B$ is the Bragg angle, $\lambda$ is the X-ray wavelength, $A_s$ is the symmetric extinction length given by

$$A_s = \frac{V_e}{r_e^2 K_{HF}}$$  \hspace{1cm} (2)

where $V_e$ is the volume of the unit cell, $r_e = e^2/m_e c^2 = 2.818 \times 10^{-15}$ m is the classical electron radius, $K$ is the polarization factor, and $F_{HF}$ the structure factor. For hard X-rays which can be transmitted through thick objects, the Darwin width of crystalline silicon is around $1\text{–}10 \mu$rad depending on the energy and the order of crystal diffraction.

3. Experimental details

3.1. Monochromator

The ideal monochromator for DEI would be a channel-cut device, but due to the extreme width (130 mm) required for the images, it is difficult to fabricate. Instead, an alternative to a channel-cut system is used. The monochromator consists of two parallel Bragg crystals. The crystals are perfect float-zone silicon with the surface parallel to the [1 1 1] planes. Thus, each crystal can be used in the symmetric Bragg mode with [$nnn$] diffraction ($n$ can be 1, 3, 4, 5, etc.) depending on the angular resolution desired.
The monochromator is a box-type design with a fixed offset of 1 cm between the two crystals. The first and second crystals are mounted on the bottom and top plates of the box, respectively. Each crystal is fixed to a kinematic mount allowing the Bragg angle and azimuthal angle to be adjusted. The box is mounted on a cradle so that the middle of the first crystal’s surface is at the center of rotation to accommodate changes in the beam’s energy. The resolution of the cradle is $2.5 \times 10^{-4}$. The top plate of the box can slide along the direction of the incident beam in 12.7 mm steps to change the distance between the two crystals. This simple box-type design is particularly resistant to vibration. Due to this mechanical stability, with the crystals diffracting in the [3 3 3] mode at 18 keV, the intensity modulation of the monochromatic beam is less than 1% peak-to-peak.

The monochromator assembly is placed inside a stainless-steel tank into which helium at atmospheric pressure flows at 50 cm$^3$ per minute to prevent corrosion caused by ozone. Both the incident and exit windows of the tank are made of 100-μm thick Kapton. The tank has a flange to accommodate supply lines for electricity, water and helium. Each crystal is 10 mm thick and 150 mm wide with strain-relief cuts (2 mm wide and 8 mm deep) on the top at 10 mm from its ends. Thus, the useful width (perpendicular to the beam) of the crystal is 128 mm. The first crystal is 60 mm long and the second is 90 mm long. The length of the second crystal allows for a long range of change in energy without the need to adjust the distance between the two crystals. For example, in the [3 3 3] mode, if the second crystal is centered to diffract 40 keV (the distance between the center of the two crystals being 66.5 mm), the monochromator’s energy can be changed from 15 to 65 keV by adjusting the angle of the box with the cradle without sliding the second crystal.

Each crystal is supported on its back side by three balls placed under the strain-relief region and secured on the other side by clamps. The first crystal is thermally coupled to a gravity-water-cooled copper block through a gallium–indium eutectic (Ga:In = 75.5:24.5) which fills the 1 mm space between the crystal and the copper block.

The second crystal’s Bragg angle and azimuthal angle can be adjusted by three piezo-driven screws (New Focus Picomotor 8303). The resolution of the Picomotor is less than 0.03 μm, so the second crystal can be finely adjusted to lie parallel to the first crystal.

3.2. Analyzer

The analyzer crystal is the same as the second crystal of the monochromator (150 mm wide × 90 mm long with similar strain-relief cuts). The crystal’s Bragg angle is controlled by a 1-m-long tangent arm. The tangent arm is driven by a linear translator with 0.1 μm resolution, providing an angular resolution of 0.1 μrad. This resolution is sufficiently accurate to position the analyzer at any location on the analyzer’s rocking curve which has a full-width at half-maximum (FWHM) on the order of 1 μrad. The tangent arm is mounted on the same optical table or granite block as the monochromator. The analyzer is attached to the tangent arm by a motorized kinematic mount which adjusts the azimuthal angle of the analyzer crystal.

3.3. Detector

Images are acquired by scanning the sample vertically through the horizontal fan beam. Although a line detector would be optimum for the present line-scan system, it is currently not commercially available. Most of our experiments were carried out with image-plate readers (Fuji Medical Systems, model BAS2000 or AC3). Since the Bragg analyzer inverts the beam like a mirror, the image plate is scanned in the direction opposite to the sample’s scan direction to avoid blurring the images. The image-plate scanner is also tilted to an angle of $20\theta_S$ from the vertical direction, so that the image plate is perpendicular to the beam diffracted by the analyzer. This configuration avoided blurring of the image due to the beam’s height and the tilting of the diffracted beam (Fig. 2).

The pixel size of the images is 100 μm × 100 μm. Images are typically read at a latitude ($L$) of 4, and a sensitivity ($S$) of 400 with 1024 grey levels ($G$).
Fig. 2. Scanning of the image plate.

Intensities are linearized on a pixel-by-pixel basis using

\[ I_{\text{lin}} = \frac{4000}{S} I_{\text{raw}}(G - 0.5) \]  

(3)

where \( I_{\text{raw}} \) and \( I_{\text{lin}} \) are the values of the raw data and linearized intensity, respectively.

3.4. Implementation at NSLS

DEI was initiated at beamline X27 of the NSLS. Since January 1998, a half-time dedicated DEI facility was established at the NSLS X15A beamline. X15A is a standard NSLS bending-magnet beamline. At the entrance of the experimental hutch, which is 16.3 m from the source, the flux (at 200 mA ring current) of the \( \sigma \) polarized white beam is \( 1.4 \times 10^{12} \) \( \text{ph/s/mm}^2/\text{keV} \) at 18 keV. The width of the fan beam in the hutch is 130 mm, and a height of up to 3 mm is available.

Fig. 3 shows the setup of DEI at X15A. The monochromator and analyzer are both positioned on a granite block which is 0.375 m wide, 0.3 m tall, and 2.1 m long. The granite block is isolated from the floor’s vibration by three stacked layers of rubber (each 10 mm thick) placed under the table holding the block.

The monochromator tank is mechanically isolated from the beamline pipe by a 2 cm air-gap. The ozone generated by X-rays passing through this air gap is reduced to an insignificant level by a 1-mm-thick aluminum filter placed downstream of the beamline’s Be window. The section between the Be window and aluminum filter is also protected from ozone by a helium flow.

The sample’s scanning stage and shutter are placed on a platform that is mechanically isolated from the granite block by supporting the platform directly from the floor.

Since the incident beam is polychromatic, non-dispersive crystals for both the monochromator and analyzer make use of the full beam height. The flux of the monochromatic beam on the sample is determined by the integrated reflectivity of the two-crystal monochromator,

\[ P = P_0 E \cot(\theta_b) I \]  

(4)

where \( P_0 \) is the beam’s intensity incident on the monochromator at energy \( E \) in terms of \( \text{ph/s/mm}^2/\text{keV} \), and \( I \) is the integrated reflectivity of the monochromator system,

\[ I = \int_{-\infty}^{\infty} r(\beta) r(\beta - \varepsilon) \, d\beta \]  

(5)

where \( r(\beta) \) is the reflectivity curve of the perfect crystal, and \( \varepsilon \) is the relative detuning of the two monochromator crystals. \( r(\beta) \) can be calculated from first principles [16].

Fig. 4 shows the calculated monochromatic beam flux \( P \) at X15A using Eq. (4) for silicon \([1 1 1] \), \([3 3 3] \), \([4 4 4] \), and \([5 5 5] \) diffractions, assuming \( \varepsilon = 0 \) and a 200 mA ring current.
When the [333] diffraction is used at energy $E$, beams with energy $E/3$, $4E/3$, and $5E/3$ are also diffracted by [111], [444] and [555] diffractions, respectively. Table 1 shows the ratio of these harmonic intensities to the [333] diffraction intensity at various energies. The 1-mm-thick aluminum filter essentially reduced all $E/3$ intensities to zero for an $E_{333}$ less than 30 keV. [555] harmonics are relatively small for $E_{333}$ greater than 22 keV. [444] harmonics are not small and should be further reduced by appropriate K-edge filtering.

### 3.5. Implementation at APS

At the APS bending-magnet beamline (Sector 1, SRI CAT), the 1BMA hutch is 31 m from the source. The beam is 80 mm wide at the entrance of the hutch. With a critical energy of 20 keV, the harmonics and scattering in the hutch could be a problem if a white beam was used, as in the case of the NSLS. Thus, at the APS, the beam was pre-monochromated before reaching the experimental hutch using a silicon [220] double-Bragg monochromator (27.2 m from the source) at 60 keV. The monochromatic beam was post-monochromated in the hutch by the [333] monochromator. Fig. 5 shows the setup in the hutch.

The order of diffraction of the pre-monochromator and the post-monochromator was mismatched. Fig. 6 shows the Dumond diagram of the [220] pre-monochromator and the [333] post-monochromator for a beam of 2 mm height. The [220] parallelogram represents the angular and wavelength distributions of the X-rays created by the pre-monochromator. Only a small vertical divergence of the beam prepared by the pre-monochromator is picked up by the

### Table 1

<table>
<thead>
<tr>
<th>$E_{333}$ (keV)</th>
<th>$P_{333}$ ($10^7$ ph/s/mm$^2$)</th>
<th>$P_{111}/P_{333}$ (%)</th>
<th>$P_{444}/P_{333}$ (%)</th>
<th>$P_{555}/P_{333}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>14</td>
<td>2.4</td>
<td>0</td>
<td>144</td>
<td>38</td>
</tr>
<tr>
<td>18</td>
<td>5.8</td>
<td>0</td>
<td>50</td>
<td>8.0</td>
</tr>
<tr>
<td>22</td>
<td>5.8</td>
<td>0</td>
<td>28</td>
<td>3.2</td>
</tr>
<tr>
<td>26</td>
<td>4.3</td>
<td>0.80</td>
<td>19</td>
<td>1.6</td>
</tr>
<tr>
<td>30</td>
<td>2.7</td>
<td>38</td>
<td>13</td>
<td>0.78</td>
</tr>
<tr>
<td>34</td>
<td>1.5</td>
<td>426</td>
<td>9.9</td>
<td>0.46</td>
</tr>
<tr>
<td>38</td>
<td>0.85</td>
<td>2360</td>
<td>7.6</td>
<td>0.28</td>
</tr>
<tr>
<td>42</td>
<td>0.45</td>
<td>9280</td>
<td>5.8</td>
<td>0.17</td>
</tr>
</tbody>
</table>
post-monochromator. The FWHM of the post-monochromator tuning curve is approximately

$$W_{\text{post}} \approx W_{\text{v}} \left( \frac{(d\lambda/d\theta)_{220}}{(d\lambda/d\theta)_{333}} - 1 \right)$$

(6)

where $W_{\text{v}}$ is the vertical divergence of the incident white beam, $(d\lambda/d\theta)_{220}$ and $(d\lambda/d\theta)_{333}$ are the slopes of the [2 2 0] and [3 3 3] diffraction in the Dumond diagram, respectively, calculated by

$$d\lambda/d\theta = 2d_{\text{hkl}} \cos(\theta_B).$$

At 60 keV, $W_{\text{post}} = 0.84W_{\text{v}}$. With a vertical beam of 2 mm in the hutch, the beam's vertical divergence ($W_{\text{v}}$) is 64 µrad. Thus, the rocking curve of the in-hutch post-monochromator against the beam coming off the pre-monochromator has a flat plateau 54 µrad wide ($W_{\text{post}}$). Since the post-monochromator is tuned to the center of this plateau, the relative vibration between the pre- and post-monochromators will not result in an oscillation of the beam's intensity delivered to the sample, as the [3 3 3] crystal diffracts only a small vertical slice of the beam provided by the pre-monochromator.

Scanning of the sample and the analyzer was configured in the same way as in the NSLS system; the analyzer was put on the same optical table as the post-monochromator.

### 3.6. Crystal alignment

Alignment of the crystals involves lining up the monochromator's crystals and tuning the analyzer. For each crystal, the azimuthal angle and Bragg angle must be aligned.

To align the monochromator using [3 3 3] diffraction at X15A, there is a great likelihood that other reflections will be diffracted and detected by the ion chamber, since the first crystal is in the wide white beam and the second crystal is a large one. Thus, when the second crystal is scanned in the Bragg angle, there are a few diffraction peaks (“spurious reflections” [17]) that are close to each other, making it hard to distinguish which one is the [3 3 3] diffracted beam. It was found much easier to first align the monochromator crystal using the [1 1 1] diffraction, and then change its angle to identify the [3 3 3] diffraction at the desired energy. The monochromator is then finely aligned using the [3 3 3] diffraction.

When the azimuthal angle is not aligned, the diffracted fan beam’s intensity has a hot spot moving from side to side as the crystal is tuned in the Bragg angle ($\theta$). In other words, the peak position of the rocking curve differs from one end of the crystal to the other. This observation was used in tuning the azimuthal angle. For this purpose, a sectioned ion chamber with a pitch of 12.7 mm was constructed to monitor the diffracted beam. Nitrogen or argon flowed through the ion chamber, depending on the sensitivity needed. Rocking curves of different sections of the fan beam are measured simultaneously by the ion chamber as the crystal is tuned. The azimuthal angle is adjusted until the peak positions of the rocking curves coincide, measured by different sections of the ion chamber.

For both the second crystal of the monochromator and the analyzer crystal, the adjustment
of the azimuthal angle is sensitive to 0.1 mrad, corresponding to a motion of 15 μm on one end of the 150-mm-wide crystal.

3.7. Types of measurement

Typically, several types of measurements were taken to investigate various aspects of the sample and DEI effects.

For a normal radiograph, the image plate is placed behind the sample without the analyzer crystal and the sample and image plate scanned vertically together at the same speed. This is comparable to conventional digital X-ray radiograph and was taken for comparison with the DEI images.

For a DEI scan, the analyzer is fixed at a specific angle and the sample and the image plate scanned at the same speed in opposite directions. A multi-scan is a series of DEI scans, each differing in the angle of the analyzer at which the image is acquired. Multi-scan images are spatially separated on the image plate by translating the image plate appropriately in-between scans. In this way, images of the same sample at different analyzer positions can be acquired using the same plate. Since most images are less than 100 mm × 100 mm, the multi-scan mode efficiently uses the image-plate area (200 mm × 250 mm) and reduces the time spent in reading the plates.

Another technique to study the response of a feature in a sample is a rocking-curve scan. In this type of scan, the region of interest on the sample is moved into the beam and a series of fixed-time exposures are taken at incremental analyzer angles. The image-plate position is regularly increased vertically so that different exposures are separated on the image plate. As a result, a vertical profile on the image plate represents a rocking curve of the analyzer through a point on the sample. Rocking-curve scans can reveal extinction contrast easily, and represent a spatially resolved measurement of the small-angle scattering by the sample.

All scans are controlled by a PC running a Windows-based C program. The program was specifically written to control the DEI experiment. The program obtains from the user the type of scan, scan range and analyzer positions, and performs the scan by controlling the image-plate motion (both horizontal and vertical), the sample’s motion and the analyzer’s position through stepper motors. The program also monitors the ion-chamber response throughout the scan to obtain dose information and controls the shutter.

The flux measured by the ion chamber ranges from 10⁶ to 10⁸ ph/s/mm², depending on the ring current, X-ray energy, and the reflection used. The scan speed is of the order of 1 mm/s, and is adjusted according to the flux to give a surface exposure of 200–1000 mR on the sample. The exposure on the image plate ranged from 10³ to 10⁴ ph/pixel depending on the analyzer’s position.

4. Results

We developed techniques to characterize and diagnose the DEI system. This section discusses these tests and presents some recent experimental results.

4.1. Intensity stability

Since the DEI contrast changes dramatically with the analyzer’s position on the narrow rocking curve, vibrations of the analyzer with respect to the monochromator reduce the information content of a DEI image. After the crystals are aligned and adjusted, the stability of the intensity was measured, both at the exit of the monochromator and at the analyzer, by looking at the ion-chamber signals on an oscilloscope. If the intensity oscillation of the beam diffracted by the analyzer is not in synchrony with the intensity of the monochromator’s exit beam, DEI contrast is reduced. Oscillation of the monochromator exit-beam also causes horizontal banding in the images.

A major technical challenge for DEI imaging is to stabilize the angle of the crystal analyzer relative to the monochromator, and control it to within a tenth of the Darwin width. An audio amplifier was used to listen to the noise of the beam to identify the source of mechanical oscillation. The problem usually can be solved by turning off the source of vibration, or putting rubber pads under the table supporting the monochromator and analyzer.
The intensity oscillation on the monochromator is typically less than 20% (peak-to-peak) at the APS (60 keV), and less than 1% at the NSLS (18 keV). The intensity oscillation of the beam diffracted by the analyzer is less than 30% at the APS and less than 5% at the NSLS. The higher modulation at APS is partially due to the narrower rocking-curve width (hence, increased sensitivity to vibrations) at higher energy, and partially due to experimental conditions which can be improved.

4.2. Analyzer rocking curve

The rocking curve of the analyzer crystal characterizes the angular sensitivity of a DEI system. The rocking curve is the convolution of the analyzer’s reflectivity curve and the angular distribution of the beam generated by the monochromator, and can be calculated by

\[
R(\theta) = \frac{I(\theta)}{I_0} = \frac{\int_{-\infty}^{\infty} r(\theta - \beta) r(\beta) r(\beta - \varepsilon) d\beta}{\int_{-\infty}^{\infty} r(\beta) r(\beta - \varepsilon) d\beta} \tag{7}
\]

where \( r(\theta) \) is the reflectivity curve of a crystal, and \( \varepsilon \) is the angular detuning of the monochromator’s second crystal from the first crystal.

Fig. 7 shows the rocking curves measured at 18, 30, and 60 keV for a tuned monochromator (\( \varepsilon = 0 \)). The solid lines show the theoretical rocking curves calculated using Eq. (7). The measured analyzer rocking curves agree well with the theoretical ones at all energies. Table 2 shows the measured and theoretical FWHM of the rocking curves at these energies. The measured widths are only slightly higher than the theoretical widths, indicating that the broadening of the rocking curve caused by the crystal’s imperfections, thermal loading, and stress is well below the Darwin widths of the crystal at these energies.

The monochromator detuning \( \varepsilon \) can be non-zero. This reduces the intensity of the beam from the monochromator, but when properly adjusted (\( \varepsilon \) between 0.5\( \omega_0 \) and \( \omega_0 \), \( \omega_0 \) being the Darwin width), the analyzer’s rocking curve has a constant-reflectivity region similar to that of the intrinsic Bragg reflectivity curve \( r(\theta) \), as opposed to a triangular shape measured with a tuned monochromator. DEI on top of this flat rocking curve is expected to have extinction contrast which is free from refraction contrast. Also, a detuned monochromator offers a slight gain in refraction sensitivity (increase in \( dR/d\theta \)) on the sides of the analyzer rocking curve.

4.3. Characterization of the refraction contrast

The refraction contrast is defined as the angle \( \Delta \theta_z \) in the plane of diffraction by which the X-rays are deflected from their original direction by the sample. In the absence of small-angle scattering, \( \Delta \theta_z \) can be obtained by measuring the intensity of the X-rays transmitted through the sample and then diffracted by the analyzer, with the analyzer at the high-angle side (\( \theta_H \)) and the low-angle side (\( \theta_L \)) of the rocking curve. \( \Delta \theta_z \) is given by [1]

\[
\Delta \theta_z = \frac{I_H R(\theta_L) - I_L R(\theta_H)}{I_L(dR/d\theta)_{\theta_H} - I_H(dR/d\theta)_{\theta_L}} \tag{8}
\]
where \( I_H \) and \( I_L \) are the measured intensities with the analyzer at \( \theta_H \) and \( \theta_L \), respectively. \( R(\theta) \) is the rocking curve of the analyzer. \((dR/d\theta)_{\theta_H} \) and \((dR/d\theta)_{\theta_L} \) are the slopes of the rocking curves at \( \theta_H \) and \( \theta_L \), respectively.

Measurements were taken at \( \theta_H \) and \( \theta_L \) which are symmetrical points on both sides of the rocking curve, thus \( (dR/d\theta)_{\theta_H} = -(dR/d\theta)_{\theta_L} \). To measure the refraction contrast, data pairs were taken at \( \theta_H = W/2 \) and \( \theta_L = -W/2 \) where \( W \) is the FWHM of the analyzer’s rocking curve. Thus, \( R(\theta_L) = R(\theta_H) = 1/2 R_p \), where \( R_p \) is the analyzer’s peak reflectivity. Under these conditions, Eq. (8) is reduced to

\[
\Delta\theta_z = \frac{-R_p}{2(dR/d\theta)(-W/2)} \frac{I_H - I_L}{I_H + I_L}.
\]

(9)

A lucite phantom consisting of wedges of different slopes was constructed to characterize the refraction contrast. The phantom, 42.8 mm wide \( \times \) 15 mm high, consists of nine wedges with \( a \) ranging from \(-0.8 \) to \( 0.8 \) in steps of \( 0.2 \), where \( z \) is the angle between the two surfaces of the wedge.

Fig. 8 shows the image of the phantom refraction angle \( \Delta \theta_z \) obtained at 18 keV at the NSLS. Each image was reconstructed on a pixel-by-pixel basis using Eq. (9) from two images taken at \( W/2 \) above and below the rocking-curve peak position. The theoretical values of \( dR/d\theta \) were used to calculate the refraction contrast. As shown in the image, the measured refraction angle is independent of variation of thickness from the top to the bottom of each wedge, and depends only on the wedge angles, indicating that the algorithm is correct and the raw images used were linear.

To compare the measured refraction angle with the theoretical predictions based on the known wedge angle, the X-ray refraction angle for each wedge can be calculated by

\[
\Delta\theta_z = (1 - n)\tan z = \frac{1}{2\pi} r_s N \lambda^2 \tan z
\]

(10)

where \( n \) (usually slightly less than unity) and \( N \) are the refractive index and number of electrons per unit volume, respectively, of the wedge material, \( r_s \) is the classical radius of the electron, and \( \lambda \) is the X-ray wavelength.

Eq. (10) can be approximated by [18]

\[
\Delta\theta_z \approx 1.3 \times 10^{-6} \rho \lambda^2 \tan z
\]

(11)

where \( \rho \) is the density of the material in \( \text{g/cm}^3 \) and \( \lambda \) is in \( \text{Å} \).

Fig. 9 plots the refraction contrast \( \Delta \theta_z \) (average of the refraction contrast on the same wedge) at 18 and 60 keV versus the wedge slope, \( \tan z \). To calculate \( \Delta \theta_z \) at 60 keV, we used \( dR/d\theta \) of \( 1.08 \times 10^6 \) at \( W/2 = 0.53 \mu\text{rad} \). The error bars reflect the statistical noise in the image. The solid curves are the least-squares fitting of a line to the data. Within the experimental error, the refraction contrast is proportional to the slope of the wedge. At 18 keV, the slope of the fitted line in Fig. 9 is \( 7.5 \times 10^{-7} \), agreeing with the theoretical value of \( 7.3 \times 10^{-7} \) calculated using Eq. (11), and \( \rho = 1.19 \) for lucite. At 60 keV, the measured slope and theoretical values are \( 6.7 \times 10^{-8} \) and \( 6.6 \times 10^{-8} \), respectively.

![5 mm](image)

**Fig. 8.** Image of the wedge phantom refraction angle at 18 keV. From left to right, the wedges have slopes of \(-0.8, -0.6, -0.4, -0.2, 0, 0.2, 0.4, 0.6 \) and \( 0.8 \).

![image](image)

**Fig. 9.** Refraction contrast versus slope of the wedge phantom.
4.4. Demonstration of extinction contrast

An in vitro mouse was studied at NSLS X15A using the silicon [3 33] monochromator and analyzer at 18 keV. Fig. 10a is a normal radiograph of the thoracic region (around 20 mm thick) of the mouse taken at 18 keV. Figs. 10b and c are DEI images of the same region with the analyzer at $-10$ and 0 $\mu$rad, respectively. Although the same exposure was used, Fig. 10b has more statistical noise than Fig. 10c, due to the reduced flux on the image plate when the analyzer is off the peak position.

A rocking-curve scan was performed with the beam across the lung of the mouse, indicated by the horizontal dashed lines in Figs. 10b and c. Fig. 10d shows the rocking curve scan; vertical direction corresponds to changing angle of the analyzer (from $-50$ to 50 $\mu$rad), and the horizontal direction corresponds to the position across the animal’s body.

Fig. 11 plots the rocking curves of the analyzer with the beam through specific regions of interest A, F, M, and L. The position of A, F, M, and L in the images of the mouse and rocking-curve scan are indicated in Figs. 10b–d. Curve A is the analyzer’s rocking curve with the beam passing outside the mouse, through the air. F is the rocking curve through the fur on the mouse’s side (only fur is present). M corresponds to a typical region of the body, including the animal’s two thin layers of fur in front and back, and other body tissues, such as muscles and fat. The rocking curve through the lung region is represented by L. To account for the absorption of X-rays, the rocking curves were normalized so that the area under each rocking curve is equal to the integrated reflectivity of the analyzer (area under the theoretical analyzer reflectivity curve $r(\theta)$).

The peak values of the normalized rocking curves in Fig. 11 are the amount of extra contrast due to DEI’s rejection of scatter in images taken

![Fig. 10. (a) Normal radiograph of a mouse at 18 keV. Darker color represents greater X-ray intensity. (b,c) DEI images with the analyzer at $-10$ and 0 $\mu$rad, respectively. Although the same exposure was used, Fig. 10b has more statistical noise than Fig. 10c, due to the reduced flux on the image plate when the analyzer is off the peak position.](image)

![Fig. 11. Normalized analyzer rocking curves through the air (A), fur (F), lung (L), and muscle (M) regions.](image)
with the analyzer on top of the rocking curve. The values are 0.44, 0.18, and 0.15 for M, L, and F, respectively. And the corresponding normal absorptions (e^{-\mu t} obtained from Fig. 10a) are 0.61, 0.48, and 0.99. Due to the extinction contrast, the fur and lung appear very absorbing (more so than the bones) in a DEI image with the analyzer on the peak of the rocking curve (Fig. 10d). The extinction contrast is sensitive to the sub-microscopic structure of the object. For example, the layer of fur (F) has negligible absorption in the normal radiograph (Fig. 10a), yet has a factor of 7 in apparent attenuation in the analyzer peak image. This is due to the fibers of the fur generating small-angle scattering. Extinction contrast also is observed for lung (L), which has negligible absorption in normal radiograph, but is distinctly different from the other tissues of the body in extinction contrast. These features can be seen by comparing Fig. 10a and c, and are clearly demonstrated in Fig. 12, which is a plot of intensity through the dashed lines in Figs. 10a–c.

It is interesting to note the reversal of contrast between the fur and the body as the analyzer moves away from the peak position. Fig. 11 shows that this reversal occurs when the analyzer’s angle is greater than 3 \( \mu \text{rad} \). In the DEI peak image, the fur and lung appear denser than other tissues, while in the off-peak image (Fig. 10b) the X-rays passing through the fur and lung appear more intense than other tissues and the background. The enormous contrast gain of the lung and fur demonstrates DEI’s sensitivity and tissue specificity not normally observed in radiography.

5. Discussion

For imaging the lung, in conventional radiography, absorption contrast is directly related to the dose received by the subject. More contrast means more absorbed X-rays which is proportional to the subject’s dose. In DEI, the extinction contrast does not come from the X-rays being absorbed, but from the rejection of scattered X-rays. Since they are absorbed in the same way as in the conventional radiography, the DEI image dose is comparable to that in conventional radiography at the same X-ray energy. Thus, the additional contrast comes without an additional dose to the subject. The DEI extinction contrast of lung is so great that X-rays with much higher energies than in conventional radiography can give satisfactory contrast. A higher energy means there is less absorption and hence, a lower dose to the subject.

From the experiments performed so far, we find that DEI using a [1 1 1] crystal monochromator and analyzer has little extinction or refraction contrast for most samples, except for refraction at the edges of features, and the extinction contrast of lung. [3 3 3] reflection is far superior to [1 1 1] in terms of sensitivity to these two types of contrast. Experiments are being carried out to further increase the extinction and refraction sensitivity of the analyzer crystal by going to higher-order diffraction, such as [4 4 4] or [5 5 5].

Acknowledgements

We would like to thank A. Dilmanian, E. Johnston, E. Pisano and D. Washburn for providing the samples, D.P. Siddons, C. C. Kao and J. Hastings at the NSLS for helpful discussions, D. Mills and G. Srajer for support and beam-time at
the APS SRICAT 1-BM beamline, and I. Pipkin and L. Sawyer of Fuji Medical System for loan of the AC3 image-plate reader. This work was supported in part by US DOE contract DE-AC02-CH10886, ARPA contract AOB227, US ARMY grant DAMD17-96-1-6143, and by the State of Illinois Higher Education Cooperative Agreement.

References