A classroom demonstration of reciprocal space
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I. INTRODUCTION

Scientists use scattering experiments to analyze materials, with the most common sources being electron, x-ray, and neutron beams. All of these techniques take advantage of the wave properties of the particles.

Typically one selects the wavelength of the incoming waves such that it matches the structure of interest. In this experiment, we do the reverse—we use a laser as the monochromatic wave source and customize an array of nanowires to fulfill the diffraction conditions. By proper design of the nanowire array, the ratio of the nanowire pitch to the laser wavelength (∼3 μm/0.532 μm ∼ 6) is directly comparable to the ratio of a typical crystal lattice spacing to synchrotron-generated x-rays (∼3 Å/0.5 Å ∼ 6).

The concept of reciprocal space is often difficult for students to comprehend. It can be compared to learning to ride a bicycle—once you can do it, it is hard to say what is so difficult about it. The demonstration experiment described here has been used in the course “Introduction to X-Ray Physics” at the Niels Bohr Institute, and helps students better understand the subject.

Inexpensive diffraction gratings, or even a compact disk, can be used to demonstrate diffraction by a one-dimensional structure. However, gratings do not show that the points in an array can be regarded as planes, and therefore also lack the possibility of changing the diffraction pattern by rotation.

Only a few experiments for visible light interacting with nanowires have so far been reported. None, to our knowledge, use a two-dimensional (2D) grid. In this paper, we calculate and measure the positions of the diffraction spots from a 2D array of nanowires.

II. DEMONSTRATION EXPERIMENT

A laser pointer with a wavelength in the visible band and a periodic array of nanowires are the only equipment needed for the diffraction experiment. The laser beam is pointed towards an area with precisely positioned nanowires, and the diffraction pattern is displayed directly on a white screen. The experiment can be performed without any preparation and works every time. The only thing to be aware of is stray laser light, as this may cause eye damage. With proper handling and a low power laser (1 mW), classroom experiments are safe.

The sample can be held in one hand and the laser pointer in the other, and by rotating the sample, one can watch the diffraction spots appear and disappear. In Fig. 1(a), a simple mount is used for the laser and the sample. Different arrays of positioned nanowires give easily distinguishable diffraction patterns. Likewise, different colored laser pointers (e.g., red, green, and blue) can be used to illustrate the wavelength dependence of the diffraction conditions.

A. Entering reciprocal space

Diffraction arises from interference of electromagnetic waves (e.g., visible light or x-rays) when scattered on planes. Scattering on an array of points (e.g., nanowire arrays or atoms in a crystal lattice) can be regarded as scattering on planes, as shown in Fig. 1(e), and is described by Bragg’s law,

\[ 2d \sin \theta = n \lambda, \]

where \( \lambda \) is the wavelength of the incoming wave, \( n \) is an integer, \( d \) is the lattice spacing, and \( \theta \) is the angle between the incoming wave and the scattering planes. From Eq. (1), it is seen that Bragg’s law is satisfied only for \( \lambda \leq 2d \).

One can also use the Laue condition,

\[ \Delta \vec{k} = \vec{G}, \]

\[ \Delta \vec{k} = h\vec{b}_1 + k\vec{b}_2 + l\vec{b}_3, \]

where \( \vec{b}_1, \vec{b}_2, \) and \( \vec{b}_3 \) are unit vectors of the reciprocal space, \( h, k, \) and \( l \) are integers known as Miller indices. For an interpretation of the Laue condition, one can look at an algebraic statement of the scattering wave vector, as in Ref. 6. For elastic scattering, where no energy is transferred to the crystal, the magnitude of the scattering wave vector can be written as...
The exact positions of the diffraction spots can be calculated using the Laue condition, Eq. (2), and the geometric interpretations of the total system. A sketch of the diffraction setup for the experiments is shown in Fig. 3, where all quantities are geometrically defined. Monochromatic light is coming in from the left, represented by the incoming wave vector \( \vec{k} \). In our case it is a laser source, but it could in principle be
any monochromatic waves, such as synchrotron-generated x-rays when the lattice spacing is on the atomic scale. The incoming light scatters on the nanowires, which are positioned in a 2D array spanned by the real plane lattice vectors $\vec{a}_1$ and $\vec{a}_2$. For the derivation presented here, we will assume that the lattice vectors are orthogonal (i.e. $\omega = 90^\circ$). The diffracted beams that fulfill the Bragg conditions will give rise to diffraction spots observed on a screen a distance $L$ from the crystal. The positions of diffraction spots are calculated without any reflection or scattering, as indicated in Fig. 3. The ratio of $S_y$ to the screen distance $L$ is equal to the ratio of the $y$-component of the diffracted wave vector to its $x$-component; that is,

$$\frac{S_y}{L} = \frac{k_y'}{k_x'}.$$  

By substituting Eqs. (5) and (11) into the Laue condition (2), an expression for $k_y'$ and $k_x'$ is found, which, when combined with Eq. (12), gives

$$S_y = L \cdot \frac{(h/d_1)\sin \phi + (k/d_2)\cos \phi}{(1/\lambda)\cos \phi + (h/d_1)\cos \phi - (k/d_2)\sin \phi}.$$  

In a similar way, the vertical deflection can be calculated, although $k_x'$ is slightly more cumbersome to calculate. Here, we use the fact that the length of the diffracted wave vector can be written as

$$|\vec{k}'|^2 = k_x'^2 + k_y'^2 + k_z'^2.$$  

Using the Laue condition (2), this is equivalent to

$$k_z'^2 = |\vec{k}'|^2 - (k_x' + G_x)^2 - (k_y' + G_y)^2.$$  

As we assume elastic scattering, the length of the scattered wave vector is equal to the incoming one, that is, $|\vec{k}|^2 = |\vec{k}'|^2 = (2\pi/\lambda)^2$. Substituting Eqs. (5) and (11) into Eq. (15) then gives

$$k_z'^2 = 4\pi^2 \left[ \frac{1}{\lambda^2} (1 - \cos \phi) \frac{h^2}{d_1^2} - \frac{k^2}{d_2^2} + \frac{2\cos \phi}{\lambda} \left( \frac{k}{d_2} \sin \phi - \frac{h}{d_1} \cos \phi \right) \right].$$  

Finally, the vertical deflection on the screen is found to be

$$S_z = L \cdot \frac{k_x'}{(1/\lambda)\cos \phi + (h/d_1)\cos \phi - (k/d_2)\sin \phi}.$$  

The above calculations have assumed a rectangular grid for the crystal structure. For $\omega \neq 90^\circ$, the basis lattice vectors must be modified accordingly. In the case of $\omega > 90^\circ$, Eqs. (6) and (7) are modified to
\[ \vec{d}_1 = d_1 \begin{pmatrix} \cos \phi \\ \sin \phi \\ 0 \end{pmatrix} \tag{18} \]

and

\[ \vec{d}_2 = d_2 \begin{pmatrix} -\sin \phi \sin \omega + \cos \omega \cos \phi \\ \cos \phi \cos \omega + \cos \phi \sin \omega \\ 0 \end{pmatrix} \tag{19} \]

By repeating the calculations above, one can derive the modified versions of Eqs. (13) and (17) to account for a non-rectangular lattice.

B. Experimental setup

Though a simplistic setup is sufficient for understanding the basic principles, a more advanced setup is needed to take full advantage of the derived theory (see Fig. 4). The sample is mounted on a two-axis goniometer on top of a 360° turntable [Fig. 4(b)]. The two axes of freedom from the goniometer can be used to center the sample, such that it rotates around the same point, when the turntable is rotated. This mimics the setup of a standard x-ray diffraction experiment with 3S + 2D degrees of freedom, meaning that three circle motions can control the sample, and two circle motions the detector. The setup is such that the angle of incidence is equal to the detector angle. The whole setup is mounted on a bread board for optimal stabilization.

A HeNe laser with a wavelength of \( \lambda = 632.8 \text{ nm} \) is used as the coherent light source. This type of laser has the advantage of a low output intensity, and some models are equipped with a range of neutral density filters, making it possible to have an output intensity of less than 1 mW; HeNe lasers are therefore very suitable for student experiments. Three lenses, with focal lengths of 100 cm, 12.5 cm, and 5 cm are used in the setup as illustrated in Fig. 4(c). The first lens is used to obtain a collimated beam and the two others are set in a telescope configuration to decrease the beam size by a factor of 0.4.

Mirrors with micrometer screws are used for guiding the beam. The two mirrors closest to the sample are used to raise the beam and point it down at the center of the sample. The angle of incidence can be varied by moving these mirrors.

The amount of unwanted scattering can be reduced greatly by taking advantage of Brewster’s angle. At an angle determined by the indices of refraction for the media, only light polarized perpendicular to the plane of incidence is reflected. By using a half-wave plate (\( \lambda/2 \)) to linearly polarize the beam parallel to the plane of incidence, most of these reflections can be eliminated. For InAs, Brewster’s angle is \( \theta_B = 73.8^\circ \) with respect to the plane of incidence, and therefore an incidence angle close to \( \alpha = 90^\circ - \theta_B = 16.2^\circ \) is used in the setup. We found this step of major importance, as the background was reduced by a factor of five. Reducing the room light gives additional signal-to-noise improvement.

A white sheet of paper was used for the screen, as it had a good thickness and texture. Various other types of materials were tested for the screen, but none could beat the white paper. Furthermore, the paper had the advantage of being straightforward to print a grid on for scaling.

For data acquisition, a Nikon D40 digital single-lens reflex camera is used; it has a charged-coupled-device (CCD) sensor with a 6.1 megapixel resolution. Data are saved in RAW format ensuring no loss of quality due to compression. The camera was first checked for dead pixels by taking a picture with the camera lens covered, loading it into MATLAB, and looking for pixels with a zero intensity value. Two dead pixels were found and these were omitted in later data analysis. All images are acquired in full manual mode with an aperture of 1/4.0 and a shutter time of 1/3 s.

C. Analysis of a single image

For a given setup, an image is acquired with the digital camera and transferred to a computer. MATLAB is used for the manipulation and measurements of the images.

A 3D plot of the data before background subtraction is shown in Fig. 5(a), where the intensity is plotted along the \( z \)-axis. Because a lot of noise is present, even for an image obtained in a dark room at Brewster’s angle, the data are filtered. First, a background image is made using morphological opening of the image by removing all disk-shaped spots in the image with a radius less than 24 pixels. This background image is then subtracted from the original image. Second, a threshold value is defined, high enough to remove random noise pixels but low enough to include the peaks. A binary image is now formed and used as a mask on the original image, thereby creating an image with only nonzero values at the peak positions. The final result is shown in Fig. 5(b). A more intuitive way of subtracting the background is to use ImageJ, where a threshold value can be chosen graphically, and the “analyze particle” function set to count areas only in a certain size range.

Based on the theoretical framework derived in Sec. IIIA, the diffraction spots on the obtained images can be categorized; an example is shown in Fig. 5(c). A script written in MATLAB calculates the theoretical position of the peaks using the modified version of Eqs. (13) and (17), and looks for the experimental intensity peak in the vicinity of this spot. The
1. Decide on the basic parameters, including wavelength and experiment is:
to estimate described below, they detected this deception and were able
told that it was a rectangular lattice. Using the method
d structure by fitting peaks for different angles. For instance,
around
This is due to a secondary periodicity with larger lattice distance,
can be labeled using this algorithm.

The basic protocol to align a sample for a scattering
configuration.

2. Find the center of the sample, or the point of interest.
3. Adjust the sample to rotate uniformly around the point
where the incoming beam hits the sample.
4. Align the system parameters to the reciprocal lattice vec-
tors of the crystal.

The basic parameters are determined by the laser system
and the requirement for fulfilling the Brewster’s angle condi-
tion. The second step is relatively straightforward as the
HeNe laser emits light in the visible range. The rough align-
ment is made by hand and subsequently fine-tuned using the
micrometer screws on the mounts of mirrors 3 and 4.

The next step is more challenging and required the use of
the two-axis goniometer. The turntable is rotated (angle \( \phi \))
while monitoring the specular intensity peak. The goniometers
are now adjusted until the specular intensity peak remains sta-
tionary during a 360° rotation. Measurements of the aligned
sample are shown in Fig. 6(a); the position deviates by less
than 0.5 mm throughout a full rotation of the sample.

The last step is performed by rotating the sample until the
intensity peaks for (0, 0), (–1, 0), and (–2, 0) are vertically
aligned, and (0, –1) and (0, 1) horizontally aligned, defined
as \( \phi = 0^\circ \). For a rectangular lattice, a rotation of \( \phi = 90^\circ \)
should bring the system into another configuration where the
spanned planes are perpendicular. The lack of this symmetry
led the students to discover the skewed lattice.

When identifying the diffraction points a systematic devi-
ation of the experimental position was found. The deviation
was found to increase with the distance to the specular spot,
which can be attributed to the screen being slightly tilted, an
imperfect lattice, and/or lens distortions. The camera lens
was investigated for distortion effects (e.g., spherical aberra-
tion and coma) by imaging a piece of paper with a printed
millimeter scale, and found to be negligible for the given
setup (<1 mm). Given that the screen has been properly
aligned (i.e., not tilted), the deviation from this effect should
be small as well. The main deviation therefore originates
from an imperfect lattice. To correct for this, we gathered
calculated and experimental intensity peaks are plotted in
Fig. 5(d). It is clear that the diffraction pattern is a mapping
of the reciprocal lattice. All expected spots are visible and
can be labeled using this algorithm.

Some of the lower-order diffraction spots are degenerate.
This is due to a secondary periodicity with larger lattice distance,
introduced by the stitching during the e-beam lithography. For
this study we look only at the brightest spot, which is expected
to be the 0th-order diffraction from the secondary lattice.

In Fig. 5(d), both the theoretically derived and experimen-
tally found positions of the diffraction spots are plotted. The
uncertainties in the theoretical values come from the meas-
ured angles and distances in the nanowire arrays. The values
used in this study are summarized in Table I along with their
estimated uncertainties.

### D. Aligning the sample for rotation

The diffraction pattern changes when the sample is rotated
around \( \phi \). This change can be used to calculate the lattice
structure by fitting peaks for different angles. For instance,
\( d_1, d_2, \) and \( \omega \) can be estimated in this way. To test the setup,
a group of students was given a sample with \( \omega = 95^\circ \) but
told that it was a rectangular lattice. Using the method
described below, they detected this deception and were able
to estimate \( \omega \) within 0.2°.

The basic protocol to align a sample for a scattering
experiment is:

1. Decide on the basic parameters, including wavelength and
scattering configuration.

<table>
<thead>
<tr>
<th>Quantity</th>
<th>Value</th>
<th>Std. dev.</th>
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<td>( d_1 )</td>
<td>2.90 ( \mu )m</td>
<td>±0.05 ( \mu )m</td>
</tr>
<tr>
<td>( d_2 )</td>
<td>3.90 ( \mu )m</td>
<td>±0.05 ( \mu )m</td>
</tr>
<tr>
<td>( L )</td>
<td>9.4 cm</td>
<td>±0.1 cm</td>
</tr>
<tr>
<td>( \phi )</td>
<td>Varies</td>
<td>±0.1°</td>
</tr>
<tr>
<td>( x )</td>
<td>15.6</td>
<td>±0.1°</td>
</tr>
</tbody>
</table>

Fig. 6. Deviations of the diffraction points. (a) The movement of the specu-
lar spot when the sample is rotated. It can be seen that the sample is very
well aligned in both the horizontal and vertical directions. (b) Vertical and
(c) horizontal deviations of the experimentally observed diffraction spots
from the theoretically derived values.
FIG. 7. Images of the diffraction patterns for different rotation angles \( \phi \) of the sample. The alignment of the sample is optimized such that the position of the specular spot \((0, 0)\) is static under rotation. See the online article for a video made from a collection of images showing a full rotation at 5° intervals. The video is also available as supplementary material (see Ref. 12) enhanced online [URL: http://dx.doi.org/10.1119/1.4773979.1].

the deviation from the theoretically calculated position of every diffraction spot for all the data points. Plots of the vertical and horizontal deviations are shown in Figs. 6(b) and 6(c), respectively. To adjust for all of the above described effects, a linear fit was made to all the data points and this was subtracted from the measurements.

E. Advanced experiments

The sample is rotated around \( \phi \) and images acquired with an interval of 5° for a full rotation of 360°. A few of the images are shown in Fig. 7 and all the images have been collected into a movie. It is noteworthy that the position of the specular peak \((0, 0)\) is almost stationary during the rotation, showing that the sample has properly aligned. The images are analyzed using the software described in Sec. III C, and the values \( d_1, d_2, \) and \( \omega \) are derived. The calculated values have a deviation of less than one percent from the data obtained using a scanning electron microscope (SEM). However, SEM length measurements are not exact because they depend on how well the beam is focused on the sample. The SEM images were analyzed using the software ImageJ.17

The setup can be further improved to obtain additional results. For instance, one could use a 2D detector, instead of the white paper, to improve the sensitivity. In this case, it might be possible to measure the intensity of the individual points precisely enough to calculate the form factor, and thereby obtain the height and width of the nanowires.

A more advanced experiment would be to calculate the Young’s modulus of the nanowires. This can, for instance, be done by monitoring the intensity of a single diffraction spot and noting how this changes when the nanowires are set to vibrate.

In addition, using a small laser beam and a system for scanning the surface of the crystal, e.g., by mounting piezos on the backside of the mirrors, one could potentially check an array of nanowires for defects. Such a procedure would be valuable in large-scale production of vertical transistors, as one defect could render the device useless.

IV. CONCLUSION

With a periodic array of nanowires and a laser, we have mimicked the diffraction conditions for a crystal in a synchrotron beam. This experiment can be used as a simple classroom demonstration of reciprocal space and as an individual undergraduate experiment. For teaching, this experiment has already proven to be a valuable tool for easier understanding of reciprocal space, and to see the connection to x-ray-, neutron-, and electron-scattering experiments.

As an experiment for students this exercise fulfills several important requirements. Most importantly, it is relatively easy to obtain the initial results; it is also possible to improve the setup and the range of experiments is limited only by your imagination. By comparing the theoretically derived positions of the diffraction spots to the experimental results for a variety of different angles, the structure factor of the artificial crystal has been determined with an accuracy of less than 1%.

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See supplementary material at http://dx.doi.org/10.1119/1.4773979 for a guide on fabrication of ordered arrays and a video of diffraction images showing a full rotation of an array with nanowires.


For help with morphological filtering using MATLAB, see <http://www.mathworks.com/help/images/morphological-filtering.html>.

The ImageJ homepage can be found at <http://rsweb.nih.gov/ij/>.