Introducing Mineralogy Students to X-Ray Diffraction through Optical Diffraction Experiments using Lasers

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ABSTRACT

Optical diffraction experiments using lasers provide an excellent introduction to x-ray diffraction. We have used them with great success both as lecture demonstrations and as hands-on student exercises during class or lab periods. Although the optical diffraction experiments involve one- or two-dimensional gratings, the nature and geometry of the resulting diffraction patterns bear a close resemblance to the patterns obtained from x-ray diffraction by three-dimensional mineral "gratings." In particular, the optical diffraction patterns formed by sieves or other two-dimensional gratings are very similar to single crystal, x-ray precession photos. The optical diffraction experiments, with their instantaneous visible results, provide a tangible, stimulating bridge to the initially more mysterious diffraction experiments involving invisible x-rays.

Keywords: Apparatus; education – undergraduate; geology – teaching and curriculum; mineralogy and crystallography.

Introduction

The development of modern, computer-controlled, powder x-ray diffractometers is engendering a renaissance in the use of x-ray diffraction in the undergraduate geology curriculum. No longer is x-ray diffraction merely a demonstration experiment in mineralogy classes. It is reasonable and routine for students to gather high-quality x-ray diffraction data for minerals, rocks, and sediments as part of their coursework and research projects (Brady and others, 1995). Of course a good understanding of diffraction is a prerequisite for the design and interpretation of x-ray experiments. In this paper we describe some classroom exercises involving optical diffraction of laser beams that we have used successfully to teach x-ray diffraction fundamentals to our students.

Atoms and x-rays are both invisible. Understanding the diffraction of the invisible by the invisible, in three dimensions, challenges even the best students. Several years ago, it occurred to us to try an optical diffraction experiment as a way to "illuminate" the principles of x-ray diffraction experiments. We used an inexpensive (<$100) red laser pointer and various familiar diffraction gratings. The results were immediate, dramatic, and exciting! Using a laser in class really captures the imagination and attention of the students. With the help of several mineralogy classes, conversations with physics colleagues, and a wonderful publication by Lisensky and others (1991), we have refined this approach sufficiently to share it. We believe that the experiments are of great pedagogic value, regardless of whether x-ray equipment is available.

A New Use for Sieves

Even if they have not had a sedimentology course, most geology students have a working knowledge of sieves. That a sieve might cast a patterned shadow is a reasonable idea. Therefore, students are not particularly surprised to see the diffraction pattern that is produced when a fine sieve (for example, 45 µm, #325) is placed in a laser beam (Figure 1a). And many are willing to make a prediction about how the pattern will change if the experiment is repeated with a sieve of a larger mesh size (for example, 90 µm, #170, Figure 1b). However, using "shadow logic" most will predict incorrectly that the 90 µm sieve will produce a pattern with a wider spacing than the 45 µm sieve.

When the coarser sieve produces a finer diffraction pattern, the fun really begins. Students will want to confirm the result and perhaps try still another sieve. Unless the lecture room is very large (making possible a large distance from the sieve to the projection surface), sieves of about 150 µm (#100) are the largest that will be useful. You will want to explore with your class the effects of sieve and laser location. Be sure to pass around the sieves and have the class look through them at the laser spot on the projection screen, ceiling, or blackboard. **At all times take precautions to be sure that the laser does not shine or reflect off the sieve directly into anyone's eyes.**

At this point you may wish to introduce other diffraction gratings. Some of our favorites include: grating glasses ("Rainbow Glasses," *Edmund Scientific*, Catalog No. M42-319), audio or computer compact disks (be careful about the reflections), ruled gratings (*Edmund Scientific*, Carolina Biological Supply), and 35mm slide gratings made by photographing laser-printed patterns (Lisensky and others, 1991). Use the grating glasses to look at the laser spot on a projection screen (never look directly at the laser source). Set up the compact disk (CD) so that the diffraction pattern produced by reflection appears on the surface of a table. Use the ruled gratings and laser-printed pattern gratings like the sieves, possibly with the aid of clamps to hold and position the laser and grating. Transparent line gratings can be superimposed at various angles to create plane lattice nets with different symmetries.

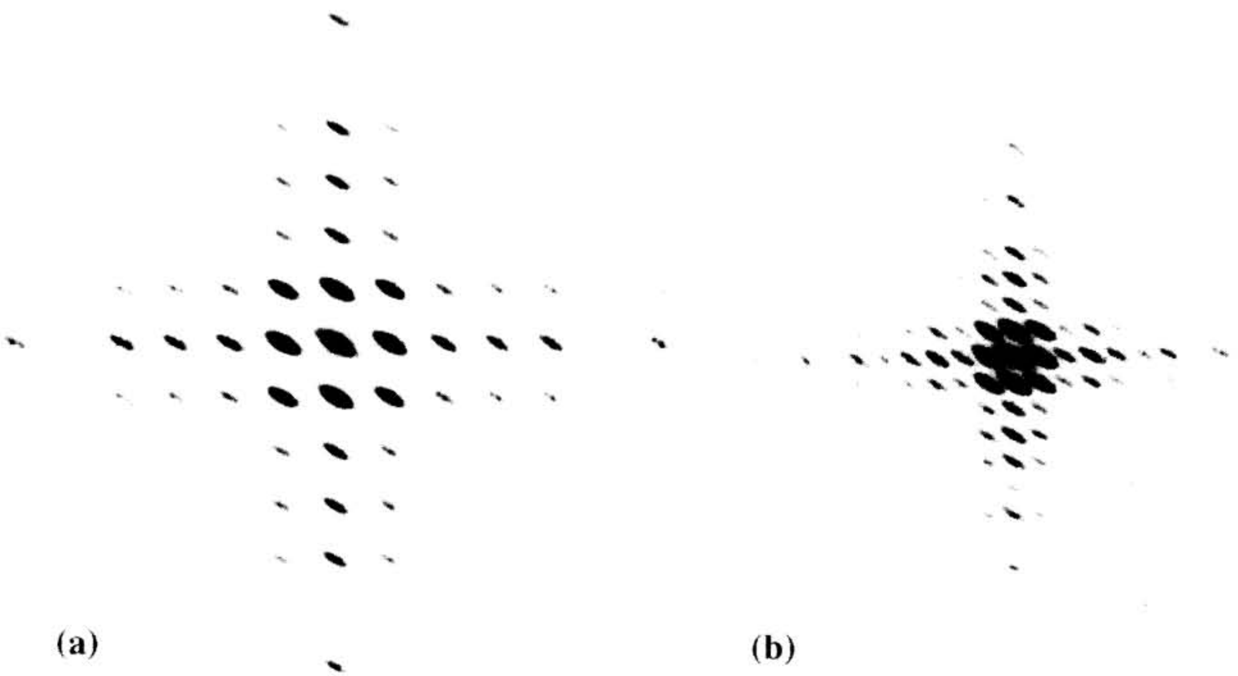

Figure 1. Visible patterns created by the diffraction of a red (670±10 nm) diode laser pointer by (a) 45 μm and (b) 90 μm sieves. The diffraction spots are oval because the laser pointer emits an elongated beam of light. The horizontal and vertical rows of spots are not exactly perpendicular, so one can conclude that neither sieve has exactly perpendicular mesh wires. The fine structure of the spots is due to non-uniform mesh spacing. These images were produced by exposing photographic paper to the laser pattern with a sieve to paper distance of 765 (±5) mm.

A good diffraction pattern can also be observed with a petrographic microscope (Richard A. Yund, personal communication, 1990) as follows: (1) place a grating on the microscope stage; (2) focus on the grating using a 10x objective; (3) place an aluminum foil mask with a tiny pin hole centered over the light source; (4) insert the Bertrand lens to see the pattern. Because the microscope has an objective lens and an eyepiece, the Bertrand lens is necessary to image the back focal plane and view the diffraction pattern (see Figure 2). Yund uses several sizes of transmission electron microscope Cu sample grids permanently sandwiched between glass slides, but other gratings will work. See also Power and Pincus (1974).

Some of the gratings listed above will surely elicit questions regarding the colors produced when white light is diffracted. For example, all of the students will have seen colors on CD surfaces. Build on these questions and propose additional experiments. (1) Create a point source of white light by masking a lamp with aluminum foil with a small hole in it and look at the point source of light through sieves, grating glasses, or an inexpensive "Star Spectroscope" (available from Project Star, Learning Technologies Inc., 59 Walde Street, Cambridge, MA 02140). (2) After dark, look at distant sodium- or mercury-vapor street lamps through any of the gratings. (3) Borrow an additional laser with a different wavelength from your physics department and compare diffraction patterns from the same sieve for two different wavelengths. Green (λ=543.5 nm) He-Ne lasers can be purchased for about $350 (Edmund Scientific). (4) Place an interference filter with a narrow transmission bandwidth over the light source of a petrographic microscope when viewing a diffraction pattern as described in the previous paragraph.

The Fraunhofer Equation

Eventually, you will want to collect and summarize the observations of the class into a predictive equation. We find this easier to do while examining a one-dimensional line of diffraction spots from a ruled grating "replica." One conceptual model that works for us is to have the students imagine a tiny light bulb at each hole in the diffraction grating with each light bulb glowing **in phase** with the others. Explore the question, "At what angles will parallel rays from adjacent bulbs be **in phase**?" The answer, which requires a knowledge of simple trigonometry and an understanding of the concept of two rays being in phase, can be expressed by:

$$\mathbf{d}\ \sin(\theta) = \mathbf{n}\lambda\ , \qquad (1)$$

(Feynman and others, 1963, p.30-3) where **d** is the spacing of the grating, θ is the angle of diffraction, **n** is an integer, and λ is the wavelength of the light (Figure 3).

Once the Fraunhofer diffraction equation is in hand, more quantitative experiments can be undertaken. These include (1) predicting the number of observed diffraction spots based on the fact that $\mathbf{n} \leq \mathbf{d}/\lambda$

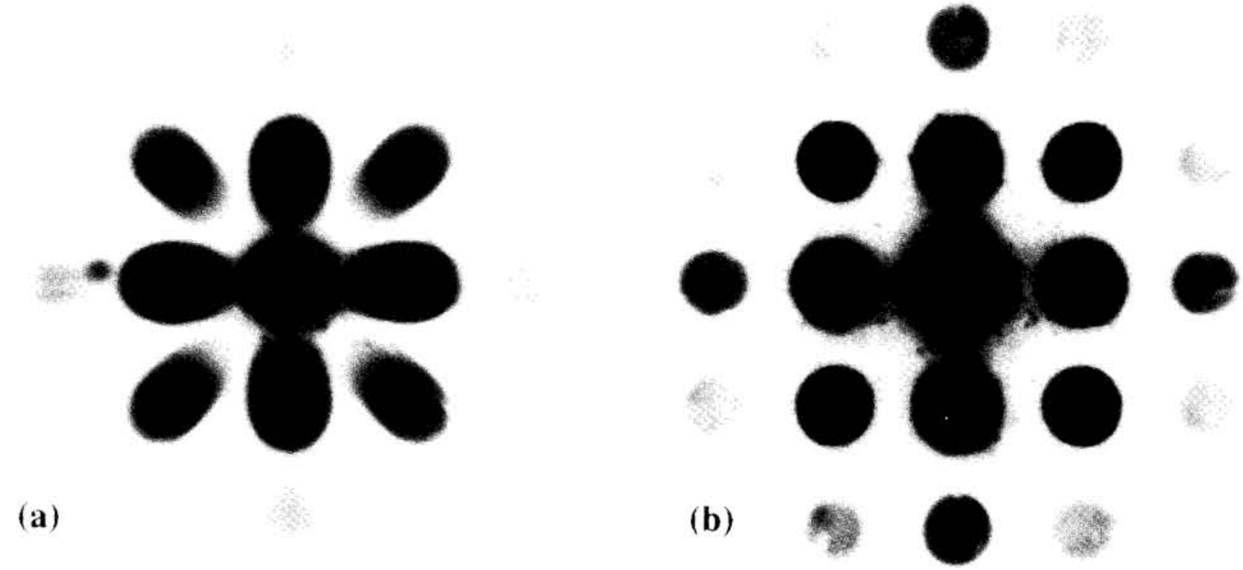

Figure 2. Black and white image of the diffraction pattern of a square grid diffraction grating (2.5 μm spacing) photographed through the 10x objective and Bertrand lens of a petrographic microscope illuminated by (a) white light and (b) monochromatic light. The light source has been masked with a microscope "peep sight" and aluminum foil. An interference filter (589±10 nm) was placed over the pin hole to produce the monochromatic image. The spots are stretched along radial lines in (a) because the white light source yields many solutions for θ in equation (1) for the many wavelengths λ in white light. A photographic print was scanned and inverted (black to white) by computer to produce this figure.

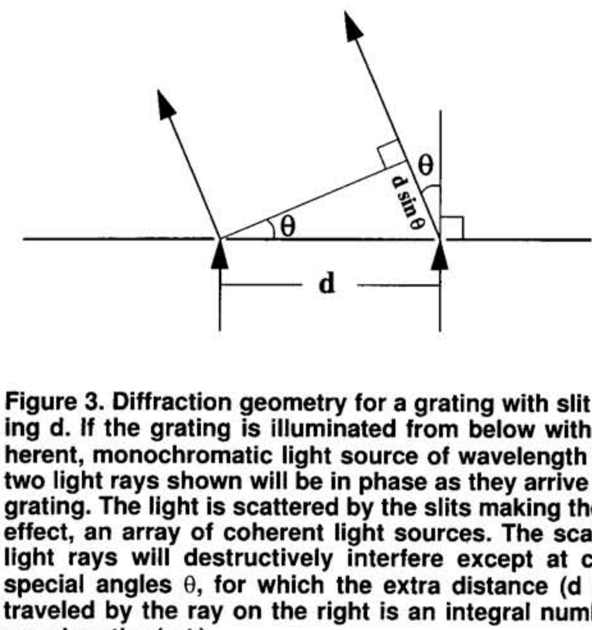

Figure 3. Diffraction geometry for a grating with slit spacing d. If the grating is illuminated from below with a coherent, monochromatic light source of wavelength λ, the two light rays shown will be in phase as they arrive at the grating. The light is scattered by the slits making them, in effect, an array of coherent light sources. The scattered light rays will destructively interfere except at certain special angles θ, for which the extra distance (d sin θ) traveled by the ray on the right is an integral number of wavelengths (n λ).

(because $\sin(\theta) \le 1$), (2) determining the wavelength of a laser using a grating of a known spacing (red diode λ=670(±10) nm, red HeNe λ=632.8 nm, green HeNe λ=543.5 nm), (3) determining the spacing of a grating using a laser of known wavelength, (4) determining the thickness of the wires in a sieve based on the difference between the diffraction spacing and the sieve openings. For example, the photos in Figure 1 can be measured and, using equation (1), sieve mesh spacings of (a) 73 (±1) μm and (b) 148 (±1) μm are obtained. The differences between the nominal sieve openings (45 μm and 90 μm) and the calculated mesh spacings are the sieve wire thicknesses (28±1 and 58±1 μm). Smith College physics classes estimate the spin rate of CD's (~900 rpm) based on their measurements of reflected (onto a table top) CD diffraction patterns.

X-Ray Diffraction

Making the transition from optical-diffraction experiments to x-ray diffraction by minerals is straightforward but challenging. We suggest using a geometric explanation of Bragg's Law, as is done in many mineralogy textbooks and which is similar to our development of the Fraunhofer equation (see Lisensky and others, 1991). Bragg's equation,

$$2\mathbf{d}_{(hkl)} \sin(\theta) = \mathbf{n}\,\lambda\ , \qquad (2)$$

is mathematically very similar to the Fraunhofer equation, where $\mathbf{d}_{(hkl)}$ is the interplanar spacing of the crystal planes (hkl), θ is the angle of incidence and of diffraction, **n** is an integer, and λ is the wavelength of the x-rays. Having first-hand experience with optical diffraction and the Fraunhofer equation, students are better able to make sense of Bragg's Law. For example, equations (1) and (2) both place limits, through the sine function, on the useful values of λ/d for diffraction. In both cases the spacing of the grating and the wavelength of the illumination must be similar.

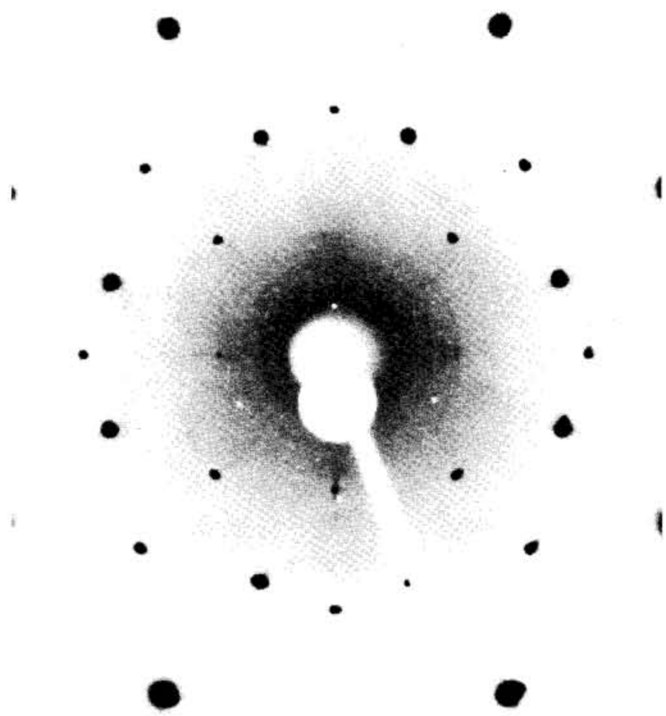

Figure 4. Transmission Laue diffraction pattern of a crystal of periclase (MgO). The photo was taken on a stationary precession camera, with the x-rays parallel to [100] of the periclase crystal, using a Polaroid film cassette and a crystal to film distance of 44 mm. The image was scanned and inverted (black to white) by computer for printing.

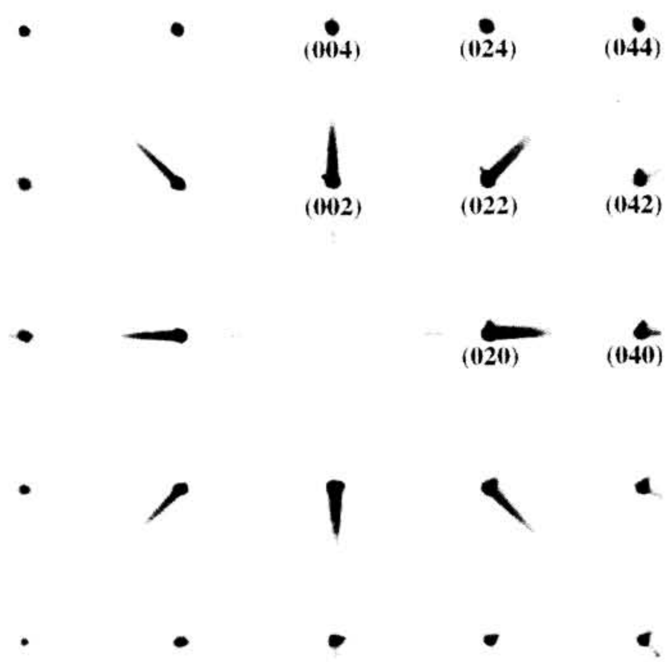

Figure 5. Precession photo (a-axis, zero level) of a synthetic periclase crystal. Note the 4mm symmetry. Some of the diffraction spots have been labeled (by inspection) with Miller indices according to the rules for space group Fm3m, namely that k and l must be even for all (0kl) reflections. The b*-axis is horizontal and the c*-axis is vertical. The "tails" on the spots are due to the fact that the x-rays (Mo tube, Zr filter) are not monochromatic. The photo was taken using a Polaroid film cassette and a crystal to film distance of 60.0 mm. The image was scanned and inverted (black to white) by computer for printing.

Once we have presented Bragg's Law, we like to ask students how they might design an x-ray-diffraction experiment for a crystal. What should the result be for a stationary mineral and a fixed x-ray source? We may take or project a Laue photograph of periclase (Figure 4) as an aid in that discussion. Laue photos are difficult to interpret, so we do not dwell on this point. More useful as a bridge to powder diffraction are x-ray diffraction patterns obtained from single crystals using an x-ray precession camera (Buerger, 1964). The precession camera moves both the film and the crystal in front of a fixed x-ray source to yield an image that preserves both the two-dimensional symmetry and lattice spacing of a zone of the crystal. Two measurable precession photos are included here (Figures 5 and 6) as examples for readers who do not have access to a precession camera.

The similarity of precession camera x-ray-diffraction patterns to the laser-diffraction patterns from two-dimensional gratings (Figure 1) is striking and helps make believers out of students. Because of the design of the precession camera, there are some differences between the precession photos and the laser diffraction patterns. For example, the spacing between the diffraction spots on a precession photo is constant – not a function of the value of **n** as in the laser experiments. Also, the diffraction spots on precession photos have "tails" because, unlike lasers, x-ray sources are not monochromatic. However, optical diffraction from

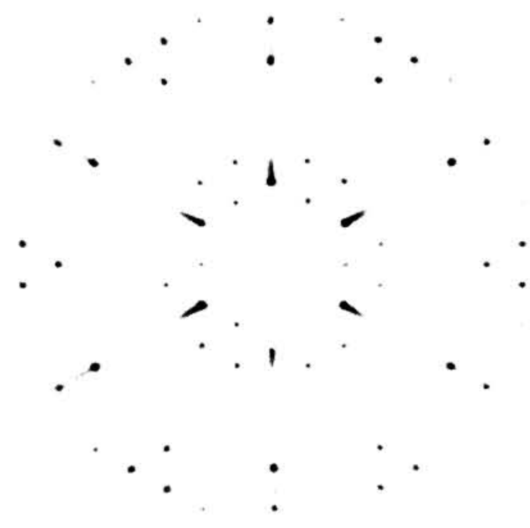

Figure 6. Precession photo (c-axis, zero level) of a tourmaline crystal. Note the 6mm symmetry. The photo was taken using a Mo tube, Zr filter, a Polaroid film cassette, and a crystal to film distance of 60.0 mm. The image was scanned and inverted (black to white) by computer for printing.

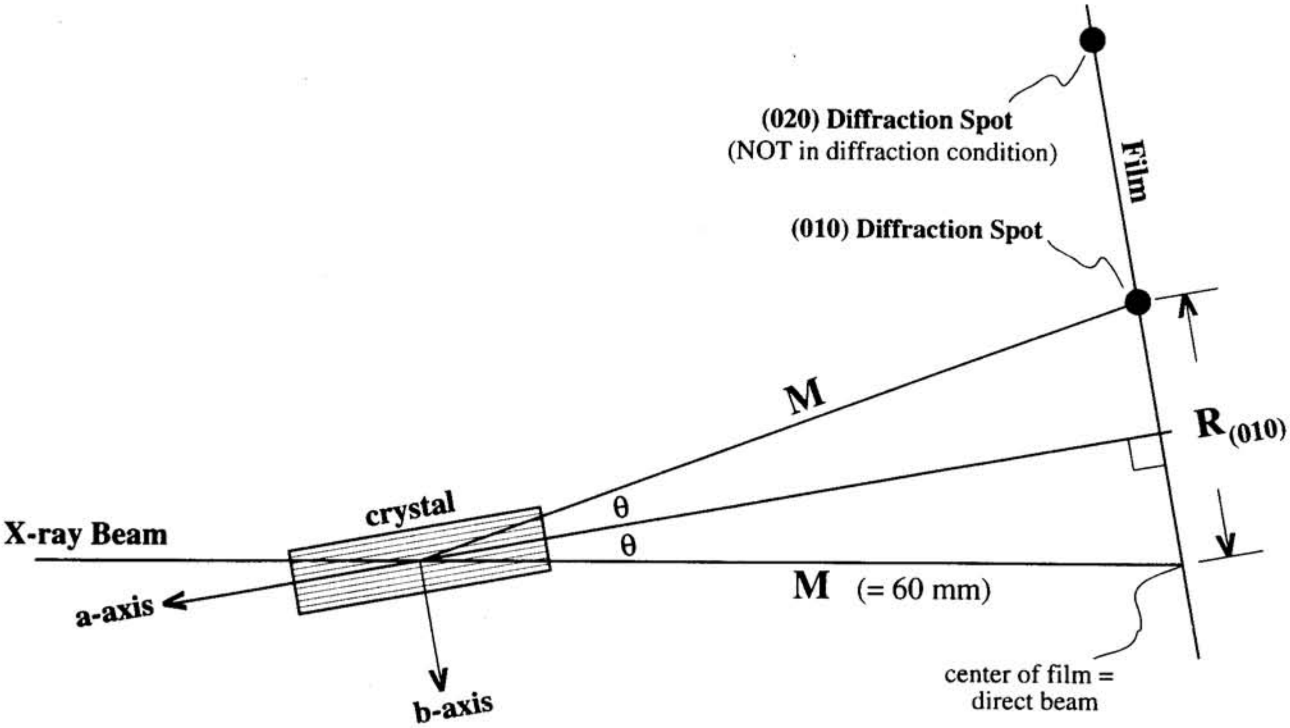

Figure 7. Schematic precession camera geometry for the (010) planes of a crystal in the diffraction condition. The precession camera moves both the crystal and the film to keep the film perpendicular to the axis of precession. M is the distance from the center of the crystal to the center of the film. $R_{(010)}$ is the distance on the film from the center (direct beam location) to a (010) diffraction spot. θ is the Bragg Law angle.

polychromatic sources (the petrographic-microscope experiment or the grating-glasses experiment) do yield diffraction spots with colored "tails" and can be studied for comparison. Most students will accept precession photos as evidence that x-rays are diffracted by crystals in a manner analogous to the way visible light is diffracted by optical diffraction gratings. This reinforces the concept of the crystal lattice as a three-dimensional array of atoms. Electron diffraction patterns of minerals photographed by a transmission electron microscope (TEM) are also very similar to optical diffraction patterns.

Precession photos are great for bringing Miller indices into the discussion of x-ray diffraction. The spots along a single line radiating from the center of the photo are all due to diffraction by a single set of atomic planes with a single Miller index (hkl). However, because "**n**" in the Bragg equation is different for each spot, the diffraction spots along a single radial line are indexed as (nh nk nl). For example, the spots along the horizontal line passing through the center of Figure 5 are all due to diffraction by the (0k0) planes of periclase. We expect to index these spots by inspection as (010), (020), (030), and so on, where these indices are simply (010) multiplied by the "**n**" in Bragg's law, (for **n** = 1, 2, 3, and so on). There is a small complication in indexing precession photos, however, due to the possible presence of "systematic extinctions," diffraction spots "missing" because of the symmetry of the crystal and our choice of unit cell. For example, the spacing of (010) planes of atoms for a crystal like periclase with a face-centered-cubic unit cell is one-half the length of the b-axis. Therefore, the spots on the precession photos due to diffraction by (010) planes are twice as far apart as they would be if the same unit cell had no face centering. Hence these spots are indexed (020), (040), etc. rather than (010), (020), etc. The (0k0) spots for odd values of k are said to be missing or extinct (according to the conditions – 0kl: k,l=2n – limiting possible reflections for space group Fm3m).

Once the Miller indices (nh nk nl) for the spots are identified on a precession photo, students can proceed, using Bragg's law, to determine $\mathbf{d}_{(hkl)}$ for any set of planes from the appropriate spot spacing in a manner analogous to the determination of grating spacing in the optical diffraction experiments. Students should measure the radial distance $\mathbf{R}_{(nh\ nk\ nl)}$ from the center of the precession photo to the spot (nh nk nl) in mm and use Bragg's Law (2) and the precession camera constants (Figure 7) to solve for $\mathbf{d}_{(hkl)}$ as follows:

$$\mathbf{d}_{(hkl)} = [\mathbf{n}\ \lambda] / [2\ \sin(\theta)]\ , \quad (3)$$

$$\mathbf{d}_{(hkl)} = [\mathbf{n}\ \lambda] / [2\ (\mathbf{R}_{(nh\ nk\ nl)}/2\mathbf{M})], \quad (4)$$

$$\mathbf{d}_{(hkl)} = [\mathbf{n}\ \lambda\ \mathbf{M}] / \mathbf{R}_{(nh\ nk\ nl)}\ . \quad (5)$$

where **M** is the distance from the crystal to the film and λ is the x-ray wavelength. In Figure 5 (which is to scale and can be measured) the value of **R** for (020) is 20.25 mm, **M** is 60.0 mm, λ is 7.0926 x 10^{-8} mm ($MoK\alpha_1$). And **n** is 2. Substituting these values into equation (4) yields $d_{(010)}$ = 0.420 nm, which is the unit cell edge for periclase. In solving for $\mathbf{d}_{(hkl)}$ on a powder diffractometer pattern, sin(θ) for equation (3) is obtained from the measured value of (2θ) rather than film measurements.

Moving from single-crystal to powder x-ray diffraction means that the information on all the radiating lines of a precession photo is condensed into one line, like closing a collapsible fan, with the brightness of the diffraction spots becoming the intensity of diffraction peaks. Some of the geometric aspects of powder-diffraction experiments can be demonstrated with a simplified, but very effective fishbowl analog of a Debye-Sherrer powder camera. A line grating is suspended in the center of an inverted fishbowl, which has been lined with paper ("x-ray film") along the curved sides. The laser is directed through a hole in the paper, strikes the grating, and produces several orders of diffraction spots including "back-reflections." By introducing smoke into the bowl (we use an incense stick) the path of the diffracted beams becomes visible within the "camera." Diffraction angles can be measured easily with a protractor.

Discussion

Optical diffraction is a common phenomenon with many interesting aspects and uses. Some mineralogy students will have had experience with optical diffraction in their high-school or college physics classes. Many will also have used diffraction gratings in an optical spectrometer in a chemistry lab. All will have seen "toys" brightly colored by diffraction from a reflective "grating foil" on their surfaces. This is fertile ground for stimulating experiments and exciting learning that can make connections between experience, mineralogy, physics, and x-ray diffraction. Physics **is** fun!

In reviewing this paper, Mickey Gunter pointed out two additional mineralogical phenomena that are the result of optical diffraction. Chatoyancy, the bright band of light seen in fibrous mineral aggregates such as tiger eye, is due to optical diffraction by the parallel arrangement of similarly-sized mineral fibers (effectively a one-dimensional grating). Asterism, the symmetrically arranged, radiating bands of light seen in minerals such as star sapphires and star garnets, is due to optical diffraction by symmetrically arranged inclusions (effectively a two-dimensional grating).

A nice section on optical diffraction and its relationship to x-ray diffraction can be found in the recent textbook by Andrew Putnis (1992, p. 41-50). If you are interested in more information about optical diffraction, you may wish to examine the books by C.A. Taylor (Taylor and Lipson, 1964; Harburn and others, 1975). Not only will these books show you diffraction patterns from a wide variety of gratings or "masks," but they will also explain how optical transforms can be used to help solve crystal structures. Diffraction by strands of a spider web is described in an article by Greenler and Hable (1989). And Power and Pincus (1974) discuss petrofabric analysis of thin sections using optical diffraction.

When we presented these ideas at an MSA-NAGT Theme Session on Teaching Mineralogy (Brady and Boardman, 1993), it was pointed out that some British mineralogists have been using optical diffraction in mineralogy courses for years. However, it was also confirmed that many American mineralogists are unaware of the approach. We hope that this article will spread the news about this great teaching tool.

Acknowledgments

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References

Brady, J.B., and Boardman, S.J., 1993, Optical diffraction experiments as a hands-on aid in teaching x-ray diffraction by minerals: Geological Society of America Abstracts with Programs, v. 25, p. A-347.

Brady, J.B., Newton, R.M., and Boardman, S.J., 1995, New uses for powder x-ray diffraction experiments in the undergraduate curriculum: Journal of Geologic Education, v. 43 p. 466-470.

Buerger, M.J., 1964, The Precession Method: New York, John Wiley and Sons, 276 p.

Feynman, R.P., Leighton, R.B., and Sands, M., 1963, The Feynman Lectures on Physics: Reading, MA, Addison-Wesley, v. 1.

Greenler, R.G., and Hable, J.W., 1989, Colors in spider webs: American Scientist, v. 77, p. 368-373.

Harburn, G., Taylor, C.A., and Welberry, T.R., 1975, Atlas of Optical Transforms: Ithaca, NY, Cornell University Press.

Lisensky, G.C., Kelly, T.F., Neu, D.R., and Ellis, A.B., 1991, The optical transform, simulating diffraction experiments in introductory courses: Journal of Chemical Education, v. 68, p. 91-96.

Power, P.C., and Pincus, H.J., 1974, Optical diffraction analysis of petrographic thin sections: Science, v. 186, p. 234-239.

Putnis, A., 1992, Introduction to Mineral Sciences: Cambridge, Cambridge University Press, 457 p.

Taylor, C.A., and Lipson, H., 1964, Optical transforms, their preparation and application to x-ray diffraction problems: Ithaca, NY, Cornell University Press, 182 p.