

Supplementary material to accompany the Notes and News item of J. Appl. Cryst. Volume 42, Part 2 (April 2009) from J. R. Helliwell

A Public Lecture on X-ray Crystal Structure Analysis: from W L Bragg to the Present Day including various Lecture Demonstrations

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Synopsis

A Public Lecture was delivered at the University of Manchester as part of its 150th Anniversary Celebration focussing on X-ray crystal structure analysis and W L Bragg (1890 – 1971) and included a variety of lecture demonstrations illustrating the principles of diffraction and describing crystal structures and properties. The aim of this note is to describe those lecture demonstrations in sufficient detail to either allow other lecturers to make their own version or be of interest to students.

Keywords: Lecture Demonstrations; Crystallographic Teaching;

Abstract

A Public Lecture was delivered at the University of Manchester as part of its 150th Anniversary Celebrations focussing on X-ray crystal structure analysis and W L Bragg and included a variety of lecture demonstrations illustrating the principles of diffraction, crystal structure and properties. This article briefly describes the contents of the Lecture, and which has been described in detail by the author as a contribution to the 125th Anniversary celebration of Z. Kristallogr. 217 (2002) 1–5. The main purpose of this short article is to link to the electronic version of the Lecture and assist the viewer to navigate in and select portions of the Lecture. This is so as to access, and have a commentary from myself on, the demonstrations. The demonstrations include a combination of microwave and laser analogues of single crystal and powder diffraction, describing the phases of reflections in crystallography via a ‘viewgraphs Fourier analysis simulator’ and of the double refraction and polarisation optical effect in calcite and relation to its crystal structure. Rather than dissect out the lecture demonstrations the whole lecture is also provided for those who wish to see it in its entirety. The author wishes to make clear that this lecture is not in any way a replacement for any of the excellent biographies available.

1. Introduction and background

Since its origins 150 years ago the University of Manchester has had many famous staff and students in the history of science including a variety of Nobel Laureates.

The University had a significant involvement in the development of science at the global level and included Earnest Rutherford's 'splitting of the atom'. W L Bragg (1890 – 1971) succeeded Rutherford ((1871 – 1937) both in Manchester and in Cambridge and in his 18 years in Manchester contributed a major part of that science history. Bragg's scientific interests embraced crystallographic methods, strongly based on physical optics, and their applications in chemistry and biology, the latter during his Cambridge ie final part of his career.

A Public Lecture was given by the author in 2001 as the 'W L Bragg Lecture', delivered at the University of Manchester's Physics Department ('Schuster Laboratory'), on 5th February 2001 as part of the University's 150th Anniversary Celebration. A detailed article was prepared for the Manchester Literary and Philosophical Society Memoirs and subsequently was reproduced in Z. Krist., with permission of the Society, as a contribution to Z. Krist's 125th Anniversary (Helliwell 2002). With the advancement of movie media being more easily made available on the www it is possible to make the Lecture and especially the Lecture demonstrations available on the internet.

The Lecture included a variety of lecture demonstrations:- a microwave analogue of diffraction from a 'sodium chloride crystal', a laser diffraction powder pattern simulation from a rotating optical diffraction grating, describing the phases of reflections in crystallography via a 'viewgraphs Fourier analysis simulator and a demonstration of double refraction in calcite and the associated polarisation effects and its explanation based on calcite's X-ray crystal structure. These lecture demonstrations all had personal links to W L Bragg including Bragg's Law of diffraction, the first crystal structure (by Bragg) of sodium chloride, the optical diffractometer, the Manchester 'powder school' and the relationship of the calcite optical properties to its crystal structure, worked on by Bragg.

The Lecture has been converted from video format into digital format and edited to improve the visual quality of the viewgraphs via 'splicing in' retyped text displays. The Lecture is split into 'chapters' to facilitate an easier selection by the viewer eg of particular Lecture demonstrations. The Lecture can of course be viewed as a whole lasting in total 50 minutes. The Lecture and the individual lecture extracts of each demonstration are deposited as supplementary material and are available at [url:- http://www.iucr.org/education/teaching-resources/bragg-lecture-2001](http://www.iucr.org/education/teaching-resources/bragg-lecture-2001) The website makes it clear at which time of the lecture's progress each demonstration took place and can therefore be taken in full context of the lecture if so desired.

2. Details of the Lecture Demonstrations (including detailed credits)

2.1 A microwave analogue of diffraction from a 'sodium chloride crystal'

This was a commercially available piece of apparatus sold by Griffin and George. It comprised a source of microwaves of wavelength 2.8cm and a microwave receiver. A simple rotary turntable was connected via metal rods to the source and the receiver ('detector') thus making a simple diffractometer where the crystal sample was a metal framework model of sodium chloride with inter-ion spacing being 5.6cm. [The spacing in the Face Centred Cubic lattice of ions of NaCl being 5.6Å.] The overall size of the apparatus was approximately 1.5 metres at longest dimension and obviously readily fitted on the front bench of the lecture theatre, and which is quite typical, in the author's experience. The University of Manchester School of Chemistry Electronics workshop linked the output of the reading-dial of the microwave receiver to a loudspeaker; thus a reflection was a loud sound and absence of reflection was

'quiet'. By rotation of the sample on the rotary stage the (100) and (110) microwave reflections could easily be found by adjustment of the receiver angle; thus Bragg's Law of diffraction via reflection at specific angles for (100) and (110) planes could be demonstrated. The limitations of this apparatus were (i) that the divergence angle of the emitted microwave beam was significant and at 'low angles' the receiver would pick up the edge of the incident beam (ii) the set up was not an ideal 'Fraunhofer far field' geometry but more of a Fresnel near-field geometry. Thus the distance of the receiver to the sample, which was adjustable to be closer to or further from the sample also led to maxima and minima of the received microwaves. In the Public Lecture the viewing angle of the videoing camera from the demonstration did not allow a view from above ie the Lecturer's view, or from the audience for that matter. Thus this demonstration would work best in a tutorial or small classroom setting. Nevertheless the Public Lecture gave a reasonable view of the components of the 'diffractometer' and of the ideas behind Bragg's Law.

2.2 A laser diffraction powder pattern simulation from a rotating optical diffraction grating

This was a basically home made apparatus inspired from my time at the University of Keele Physics Department when teaching the Physical Optics Course; an electron microscope (EM) 2-D grid/mesh in a holder allows transmission of a laser beam thus giving a red-laser 2D array of spots; a motorised fan belt rotated the holder thus giving a powder pattern of rings. The adjustment of the speed of the motor allows the transition between spots and rings to be readily seen by the audience. At the University of Manchester School of Chemistry Mechanical and Electronics Workshops a replica of this set up was easily made. The EM grid was replaced by a crossed-pair of 35mm projection slides each holding an optical diffraction grating purchased from a physics teaching supplier. The crossed pair of gratings, being heavier than an EM mesh, meant that the motor and driving belt were a little bit more robust. In this Public Lecture the usual dimming of lights was needed and a large distance between the rotating gratings and the projection onto the Lecture theatre side wall. The video cameraman does capture the rings to 2D pattern of spots nicely. The sharpness of the 'powder' rings is not as good as the live showing presumably due to coarse pixel size of the video. A further variant of using laser diffraction is by reflecting off a metal ruler with different rulings on it eg 1 mm, 0.5mm, fractions of an inch (1/16ths, 1/32nds, 1/64ths etc). Thus by reflecting off different portions of the ruler one can readily demonstrate the doubling of interspot distances derived from a halving of the rulings' distances (the 'unit cell'). The optimal set up I have found is with an optical bench but can be done, as I first saw it by Prof Alan Leadbetter CBE, with a steady pair of hands holding the laser and the ruler and again pointing onto the side of the Lecture Theatre wall (ideally white).

2.3 Describing the phases of reflections in crystallography via a 'viewgraphs Fourier analysis simulator

I am grateful to Prof Henk Schenk for this. The Fourier summation is of the diffraction waves added via one viewgraph at a time whereby the projection view of hexachlorobenzene gradually builds up. The projection allows the hexachlorobenzene to be viewed 'in plane' and the whole unit cell shows several molecules. The Lecture Theatre has to be with lights dimmed but not off as the audience needs to see the viewgraphs being added one by one. A limitation of viewgraphs is that they are not completely transparent and so one has to adapt to that by using one viewgraph to add three reflection waves. Overall a total of approx 30 reflections gives an atomic resolution image. The video cameraman in the final shot of this zooms away from the screen and the longer distance shot of the several molecules in view I found particularly pleasing. A little later on in the Lecture I return to this collection of stacked viewgraphs and emphasised then the point as to how each viewgraph had to

be carefully aligned to get the image of the molecules in the unit cell. Thus by shuffling the viewgraphs out of alignment one shows the loss of the phases relative to one another in losing the clarity of the molecules in the image. Computer versions of this Fourier simulator can be found eg on the CD in the book by C Giacovazzo et al (2002) and using the interactive tutorial at the website set up by K Cowtan <http://www.ytbl.york.ac.uk/~cowtan/sfapplet/sfintro.html>

2.4 A demonstration of double refraction in calcite and the associated polarisation effects and its explanation based on calcite's X-ray crystal structure

The optical effect of calcite, known originally as Iceland Spar, was first written about by Bartholinus in the late 17th century. I readily was able to purchase a large single crystal of calcite from my local Crystal and Gems shop measuring 11.5cmx4cmx3.5cm. The School of Chemistry Mechanical Workshop drilled a linear array of ten holes in a metal plate; each hole was diameter 2mm and spaced 1cm apart so that the total length of the array was less than the long length of this calcite crystal. Overall the plate was of sufficient size to cover completely the illuminated area of the overhead projector ie 31.5x31.5cm. There is not a special requirement to follow this specific approach but Thus a single array of lit up spots is projected onto the Lecture Theatre screen facing the audience. By placing the calcite crystal onto the array of holes a double array of holes is now seen. There is not a special requirement to follow this specific approach with the metal plate (see below) but I do commend it as one option for showing off the precision of the crystal itself in producing two lines of equally geometrically perfect lines of, now polarised, light spots. The connection to the atomic layout of the calcium carbonate in calcite is shown via a combination of viewgraphs of figures in W L Bragg's book 'The Development of X-ray Analysis', the key feature being the planar trigonal arrangement of each of the carbonates and the polarisation selection of the light coming up through the calcite crystal. I also show a neat diagram from WLB sketching out the ray paths of the ordinary and the extraordinary ray. The climax of the demonstration is to show that each of the two lines of ten lit-up spots now involves differently polarised states of light. Thus by rotating a piece of Polaroid directly above the crystal one can show one row and then the other appearing and disappearing. [Incidentally, this demonstration I have found as the first ie best one to use at University Public Open Days on our 'Crystallography' stand; the opening line to the passing prospective student and parents of "Have you ever seen the double refraction optical effect in calcite?" leads them into getting them interested in the crystal structures we have on display and a variety therefore of structural chemistry including optical and electrical properties of crystals and as well, finally, of structural biology.] When I gave a Friday Evening Discourse at the Royal Institution in 2003, splendidly held in the Faraday Lecture Theatre, I showed this demonstration there. The RI had completely perfect, home-grown, calcite crystals. I wore white gloves during the demonstration, at the RI Technician's instruction. I was afraid the crystal would slip out of my gloved hands but fortunately I held on fine! Instead of the lit up array of spots, as above, the word calcite in large letters was written on a viewgraph, again provided by the RI technician. Thus placing the crystal over it gave the two images of the 'calcite' word. This was also a neat way of showing the effect.

Dedication of these Lecture Demonstrations to Sir Lawrence Bragg

I wish to dedicate these Lecture demonstrations to the memory of Sir Lawrence Bragg, and who himself pioneered ways of popularising science in general and crystallography in particular at The Royal Institution, and provided an inspiration to me to use Lecture Demonstrations in explaining crystals and crystallography.

Further Acknowledgements

JRH is very grateful to Brian McMahon at IUCr Chester for learning the necessary technical means for the digital conversion of the video of my Lecture and providing the careful editing. JRH is grateful to Brian as well as to Peter Strickland at IUCr Chester and to Prof Paola Spadon, Chair of the IUCr Commission on Crystallographic Teaching, for all their encouragement to make the Lecture more widely available for the benefit of Lecturers, students and even members of the public interested in crystallography, its principles and its applications across the sciences, as well as its historical roots. Permission to reproduce the Lecture on the web in this way, in its shortened and segmented form, has been granted by the University of Manchester and to whom JRH is also very grateful. I also wish to thank the JAC Education Coeditor, Dr Katherine Kantardjeff, and the three referees for their thorough and yet prompt handling of all the material in this 'article' submission.

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