

THE LAUE METHOD

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Preface

The Laue method was the vehicle for the discovery of the diffraction of x rays by crystals sixty years ago. It marked the beginning of a tremendous advance in science which led to our present detailed knowledge of the structure of crystalline matter. We enjoyed the advantages of a modern technology of solid materials in no small measure as a direct consequence of this discovery.

The Laue method was the forerunner of other ways of using x-ray diffraction for the study of matter. To each of these methods there are characteristic advantages and disadvantages. Because it has its own advantages, the Laue method is still utilized, at least to some extent, in substantially every x-ray diffraction laboratory. Unfortunately, the treatment of the Laue method is now confined to, at most, a chapter in books which deal with the entire field of x-ray diffraction, and, indeed, the only book devoted to this venerable method, Schiebold's "Die Lauemethode," is now forty years old and no longer available. To discuss the Laue method properly and to make known some of its advantages, this book was written.

The fantastic pace at which the science of x-ray crystallography has advanced has left the Laue method in a half-forgotten corner, basically because it is not well suited to the popular field of crystal-structure analysis. Its commonest use is to check and adjust the orientation of a crystal which is destined to be used in another method, probably leading to the determination of its crystal structure. The Laue method is also used to assess the perfection of a crystal, although this application is not used by all who might well benefit from it. One of its more recent and important applications is in the identification and study of disorder in crystals by the analysis of diffuse scattering. For this purpose, a relatively rapid survey of all reciprocal space is required, for which the Laue method offers special advantages. Many who might well study the disorder in crystals do not do so because the application of the Laue method in this connection is not generally recognized and understood.

The use of the Laue method in studying the cell and symmetry of a crystal is practiced by relatively few scientists, and then chiefly in the study of the simplest crystals. We have taken the trouble to discuss just what can be ascertained in this field, and how the investigation can best be done. It is a curious fact that, before the Laue method was discovered, an elegant body of crystallographic calculus had been developed. This was applied to getting crystallographically useful data from the angular measurements made on the surface planes of crystals with the aid of the optical goniometer. While the calculus applied to such data did yield some useful results, the data were too limited to lead to the results really desired. For example, because the original data were so limited, the attempt to identify crystals by using the calculus to rework the data was so close to a failure that only a few devotees attempted to identify crystals from such data. The situation actually changed drastically when the Laue method was discovered, for this supplied an overabundance of data. It took some sixty years to realize this, however, for the attention of crystallographers had turned almost completely to the solution of easy crystal structures, and to finding a general solution for all crystal structures. Accordingly, we have taken the trouble to demonstrate why and how the Laue method provides an easy vehicle for identification of crystalline species.

The Laue method should have an appeal to various kinds of scientists who study crystals. It is obvious from what has just been said that the method supplies a natural research vehicle for those interested in the calculus and projection methods of classical crystallography. Accordingly, the more important aspects of classical crystal theory and projection methods (such as the stereographic, gnomonic and stereognomonic projections) are carefully developed in this book. It is our hope that those interested in classical crystallography will discover that the older crystallography acquires a new significance in terms of the interpretation of crystal data available in the Laue method, which furnishes a speedy and simultaneous goniometry of all crystal planes of interest. We also hope that this easy goniometry and the abundant information that the photographs contain may encourage many metallurgists, who ordinarily make only a limited use of the information available, to take advantage of the additional information which these photographs could still provide.

On another level, the dual nature of the information contained in the Laue photograph should always be kept in mind. The sharp spots of the photograph provide information about the reciprocal-lattice points contained in the volume of the Ewald sphere defined by the minimum wavelength, while the background provides information, through the monochromatic component, concerning the part of the reciprocal space on the surface of the Ewald sphere corresponding to the characteristic radiation.

The subject matter of this book falls naturally into two parts. The first part, consisting of Chapters 2 through 8, provides, at an elementary level, a simple and compact treatment of the Laue method and the background needed to make use of it. The second part, Chapters 9 through 13, treat the Laue method on a higher level; this is written for the research scientist who would like to exploit all the unique advantages inherent in the Laue method. In addition to these two ranges of chapters which constitute the main subject matter of the book, the book begins with a Prologue, in which the originating ideas, first experiment, and initial interpretation are analyzed in historical retrospect; the book ends with an Epilogue which is concerned with a very simple new interpretation of the Laue method.

This book is accordingly written for both student and specialist. Both will find the Laue method a useful technique for the study of crystals. The references have been selected so that the basic points of each chapter are supported by significant literature so that the reader can deepen his knowledge and extend his study into related fields.

We wish especially to express our appreciation to Dr. L. V. Azároff, Director of the Institute of Materials Science at the University of Connecticut, for providing appropriate facilities (including a temporary residence) so that the coauthors could bring their respective contributions to the chapters into mutually acceptable form.

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Chapter 1

Prologue

Over 50 years have elapsed since Laue made his historic discovery of x-ray diffraction by crystals. Although the story of this turning point in science has often been told, it is especially appropriate to use it as an introduction to a book on the Laue method.

Historical background

About 1912, an exceptional group of scientists was in residence in Munich; among others were Professor Paul von Groth (the dean of crystallographers), Professor Wilhelm Konrad Röntgen (the discoverer of x rays), and Professor A. Sommerfeld (the well-known theoretical physicist). Associated with Röntgen were a number of people working in experimental research on the nature and properties of x rays: Von Angerer was measuring the energy of such radiation; Bassler, its polarization; and Friedrich, the azimuthal distribution of its intensity. In the field of theoretical physics, the numerous papers on the theory of x rays by Sommerfeld, a follower of Boltzmann, were well known. Max von Laue joined Sommerfeld's group in the fall of 1909. He was a pupil of Planck and had obtained his degree in Berlin. At that time, he was working on the theory of interference and wave optics.

Of no less importance was the presence of the old master of crystallography, Paul von Groth, whose views on the molecular structure of

crystals were well known. For many years crystallographers had realized that crystals were discontinuous solids with three-dimensional repetitive order, and it was believed that the motifs of their patterns were molecules. This idea, however, was little more than a good working hypothesis. It was thought that the crystal faces were expressions of the lattice aspect of the pattern while cleavage was an experimental evidence of the discontinuous aspect which would be expected because of the lattice translations. The close connection of crystallographers and physicists therefore provided an appropriate setting for the discovery which was to follow.

The starting spark for the discovery was the doctoral thesis of P. P. Ewald, in which he attempted to account for the optical properties of crystals as an interaction of atomic dipoles with the electromagnetic waves of visible light. Ewald showed his thesis to Laue a few days before submitting it to the Philosophical Faculty on 16 February 1912. Ewald explained to Laue that in his study of the dispersion he had assumed the resonators to be situated at lattice points, because crystals were thought by crystallographers to have such internal regularity. Laue then asked what was the distance between resonators and what would happen if very much shorter waves would travel through the crystal. Ewald pointed out that the derivation of one of the equations of his manuscript thesis was valid also for short wavelengths.

From a consideration of specific gravity, molecular weight, and the mass of the hydrogen atom, the lattice translations of crystals were known to be of the order of 10^{-8} cm. Moreover, Wien and Sommerfeld had shown that the wavelength of the x rays should be of the order of 10^{-9} cm. Diffraction by a three-dimensional grating had never been considered, but, as Laue pointed out later on[†], his optical intuition told him at that time that if the wavelength is of the same magnitude as the atomic distances in the regular arrangement in the crystal, this must lead to some kind of diffraction effect with these shorter wavelengths.

Laue discussed his feelings with Sommerfeld, Wien, and others. It was argued that the temperature motions of the atoms would disrupt the regularity of the grating to such an extent that no pronounced maxima could be expected.

Laue further discussed the matter from a theoretical point of view in a seminar; the opinion then prevailed that experiment was safer than theory. As a consequence of his enthusiasm and clarity of ideas, Friedrich, at that time Sommerfeld's assistant, became interested in the problem, as did Paul Knipping, a research student who had just finished his thesis work in

[†] Max von Laue, On the discovery of x-ray interference. Nobel Prize Lecture, Stockholm (June 3, 1920).

Röntgen's institute. Both volunteered to assist in an experimental test of this theory. Friedrich and Knipping then developed the provisional experimental arrangement schematically shown in Fig. 1, which is a reproduction of their original drawing. An x-ray beam of 1-mm cross-section was isolated by means of four lead screens B_1 to B_4 from the hemisphere of radiation produced by the anticathode A of a Muller x-ray bulb. The x-ray beam so selected passed through a crystal Kr mounted on a goniometer head G . The x-ray bulb had been provided by Röntgen and the crystal by von Groth. A crystal of copper sulfate was used as the diffraction grating, and in different directions and at different distances, there were placed photographic plates on which the diffracted beams were to be recorded. In order to prevent the undesired direct radiation from reaching the photographic plate, a larger planar lead screen S and lead case K were placed between the x-ray bulb and other parts of the apparatus. The tube was operated with a current of 2–10 mA and the exposure time varied between 1 and 12 hours. Initial pitfalls and the subsequent improvement of the technique can be deduced from Fig. 2, which contains copies of the

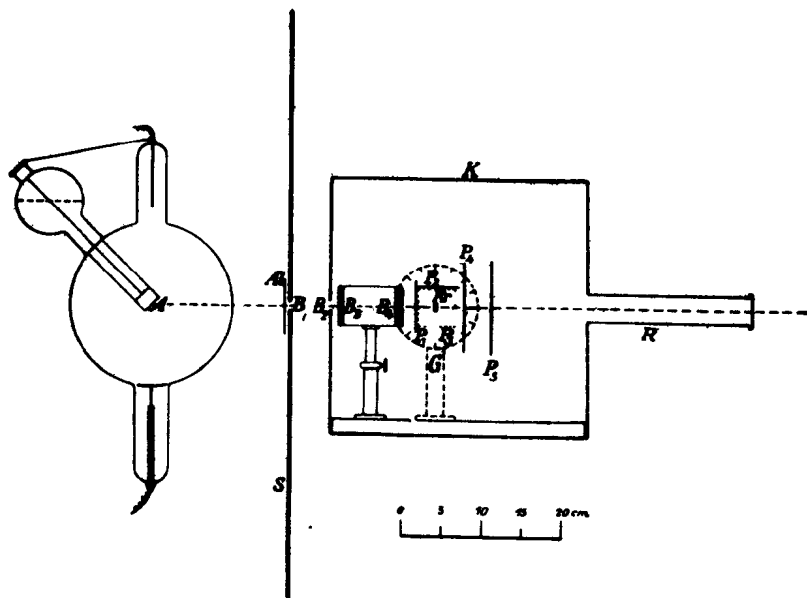


Fig. 1. Scheme of the original setup of Friedrich and Knipping's experiment. **A:** anticathode; **Al**, aluminum foil; **S**, lead screen; **B₁, B₂, B₃, B₄**, openings of the collimator system; **Kr**, crystal; **G**, pedestal; **P₁, P₂, P₃, P₄, P₅**, photographic plates; **K**, lead case; **R**, beam stop. [From Friedrich *et al.*¹, p. 313]

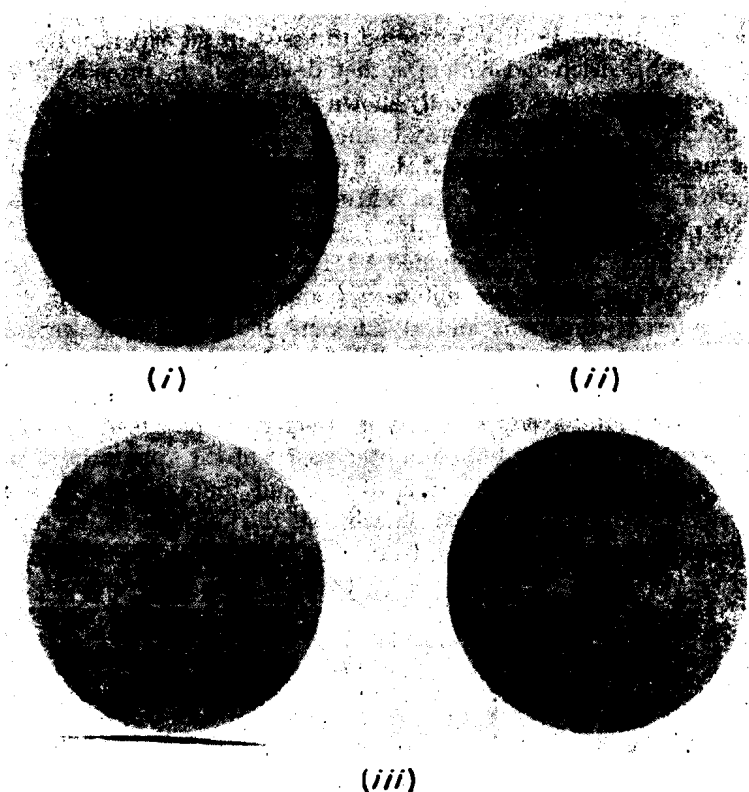


Fig. 2. Reproduction of the original Laue photographs of Friedrich and Knipping. (i) The first picture; (ii) picture obtained from a powder sample; (iii) other attempts. [From Friedrich *et al.*¹, Tafeln I and II.]

original photographs from the research by Friedrich, Knipping, and Laue that was communicated to the Bavarian Academy of Sciences at the meetings of 8 June and 6 July 1912 by A. Sommerfeld, a fellow of the academy.

Friedrich and Knipping, both true experimentalists, took every precaution to reassure themselves, as well as possible critics, that the observed phenomenon was due to the presence of crystalline material. Photographs taken with the beam irradiating different portions of the same crystal gave constant results. Friedrich and Knipping showed a great deal of

naivety, even taking a photograph of the pulverized crystal to show that no diffraction effect was observed from a noncrystalline material (Fig. 2ii).

The analysis of the photographs obtained from the irradiated single crystal showed the presence of spots other than the one produced by the primary x-ray beam. This, together with fact that by moving the photographic plate backward or forward it could be seen that the spots were formed by rectilinear pencils spreading in all directions from the crystal, showed the experimenters that diffraction really did occur, so that Laue's intuition was confirmed. Thus, there was experimental proof both of the periodic nature of the crystals and of the wave nature of x rays. At the same time the *Laue method* was born.

Early interpretation of Laue photographs

The initial interpretation of the experimental results was due to Laue. This was one of those rare occasions on which one knows the exact place and moment of the discovery. Laue himself gives a vivid narration of the event.⁴

It was not the first, but the second [picture] that gave a result. The [x-ray] transmission photograph of a piece of copper sulfate showed near the primary x-ray beam a crown of diffracted lattice spectra. Deep in thought, I was heading home through Leopoldstrasse after Friedrich had showed to me the photograph. And very near to my home, Bismarkstrasse 22, by the house Sigfriedstrasse 10, occurred to me the idea for the mathematical theory of the result.

Shortly before that day, in fact, Laue had written an article for the *Enzyklopaedie der mathematischen Wissenschaften* in which he had given a new foundation to the old theory of diffraction by an optical grating: by applying the equation of the theory twice over, the theory of diffraction by a cross grating could be obtained. Laue realized then that in order to interpret the new discovery, he had to write the equation three times, one for each periodicity of the space lattice. In only a week's work, he was able to have a quantitative theory of the diffraction of x rays by crystals. Thus, the famous Laue equations were formulated.

The theory was based on the application of monochromatic x rays, as required by interference theory. At that time Laue, Friedrich, and Knipping were convinced that the diffracted rays should consist of characteristic radiation emitted by the crystal under the influence of the incident ray. The choice of the copper sulfate crystal was made with this in mind. Thus Laue tried to associate the diffraction spots of the diagram with five

different wavelengths. Because he was unable to get exact results, he thought that his equations held only approximately.

The correct interpretation came a few months later, not from his group, but from another researcher, William Lawrence Bragg, a young student of physics at Cambridge and the son of William Henry Bragg, Cavendish Professor of Physics at the University of Leeds. It was not by chance that the Braggs became interested at once in Laue's experiment and theory. The father was already experienced with x rays, having set up the first x-ray tube in Adelaide (Australia) shortly after Röntgen's discovery. At the time of Laue's discovery, W. H. Bragg held the theory that x rays were a type of corpuscular radiation. In fact, in October 1912 he proposed an alternative explanation of the fourfold symmetry of the Laue photograph of sphalerite; he proposed that all the directions of the secondary pencils in this position of the crystal are "avenues" between the atoms of the crystal. His son was actually making some unsuccessful experiments to get evidence of his father's views. They soon, however, accepted the wave theory of x rays as explaining the diffraction experiments. Nevertheless, W. L. Bragg was convinced that Laue's analysis of the x-ray photograph was not correct. Instead of the small number of wavelengths assumed by Laue, Bragg proposed the existence of a continuous spectrum in the incident radiation. At the same time, he considered the crystal as a diffraction grating from a point of view different from that of Laue, and this led him to important simplifications. Bragg showed that the spots in Laue's photographs could be explained as partial reflection of the incident beam in sets of parallel planes on which the atoms were arranged in the crystal. On trying the Laue experiment with a sheet of mica, Bragg proved that the laws of reflection were obeyed, and he was able to formulate the condition for diffraction in a very simple and compact form, the famous Bragg equation. Bragg also showed that Laue's equations were satisfied not approximately, but rather rigorously. He showed that exposures of only a few minutes, instead of long hours, were sufficient to take a Laue photograph.

At Cambridge, interaction between physicists and crystallographers proved to be successful. By studying Pope's and Barlow's papers, W. L. Bragg became familiar with their views on crystal structure, and he was able to demonstrate that the Laue photograph of sphalerite was characteristic of a face-centered cubic crystal. Pope further encouraged W. L. Bragg to try Laue photographs of NaCl and KCl, for which Barlow had long since proposed what is now known as the sodium chloride structure. These analyses established the structure of the sodium chloride group of crystals and crystal-structure determination was initiated.

Laue's theory of diffraction by crystals

A monochromatic wave of wavelength λ and unit intensity can be represented by

$$z_1 = \exp[ik(ct - \alpha_0 x - \beta_0 y - \gamma_0 z)] \quad (1)$$

where c is the velocity of light; k a constant equal to $2\pi/\lambda$; t the time; $\alpha_0, \beta_0, \gamma_0$ the direction cosines of the wave normal in the orthogonal system x, y, z . The planes

$$x\alpha_0 + y\beta_0 + z\gamma_0 = \text{constant} \quad (2)$$

are the planes of equal phase of that wave. If the time term is disregarded, (1) can be written as

$$z_1 = \exp[-ik(\alpha_0 x + \beta_0 y + \gamma_0 z)]. \quad (3)$$

Let us assume now that this monochromatic wave impinges on a mono-atomic crystal in which the positions of the atoms are given by

$$\mathbf{r}_1 = l_1 \mathbf{a}_1 + l_2 \mathbf{a}_2 + l_3 \mathbf{a}_3 \quad (4)$$

where \mathbf{a}_i are the crystallographic translation vectors. Under the influence of the incident wave, an atom P_1 becomes the origin of a secondary spherical wave that, at a distance R_1 from the atom, can be expressed as a periodic function, such as

$$z_2 = (\psi/R_1) \exp(-ikR_1) \quad (5)$$

where ψ is the amplitude diffracted by the atom. These new waves have the same wavelength as the incident wave because the diffraction is coherent. Each atom in the crystal thus becomes a secondary center of emission. The resulting amplitude from the whole crystal is just the product of (1) and (5), so that

$$A = \sum_{l_1} \sum_{l_2} \sum_{l_3} (\psi/R_1) \exp[-ik(R_1 + \alpha_0 x_1 + \beta_0 y_1 + \gamma_0 z_1)] \quad (6)$$

where x_1, y_1, z_1 are the orthogonal coordinates of the atom at the lattice point $P(l_1 l_2 l_3)$.

The interference-effect of the wavelets generated by the various atoms is observed at a distance R_0 from the center of the crystal. Because this distance is very large in relation to the dimensions of the crystal, it can be assumed that $R_0 = R_1$, in which case the amplitude at this point is

given by

$$A'_{R_0} = \psi' \frac{\exp(-ikR_0)}{R_0} \times \sum_{l_1} \sum_{l_2} \sum_{l_3} \exp\{ik[(\alpha - \alpha_0)x_1 + (\beta - \beta_0)y_1 + (\gamma - \gamma_0)z_1]\} \quad (7)$$

where α, β, γ are the direction cosines of the direction R_0 ; at the same time we know that

$$\begin{aligned} x_1 &= l_1 a_{1x} + l_2 a_{2x} + l_3 a_{3x}, \\ y_1 &= l_1 a_{1y} + l_2 a_{2y} + l_3 a_{3y}, \\ z_1 &= l_1 a_{1z} + l_2 a_{2z} + l_3 a_{3z} \end{aligned} \quad (8)$$

where the a_{ix}, a_{iy}, a_{iz} ($i = 1, 2, 3$) are the components of the base vectors $\mathbf{a}_1, \mathbf{a}_2, \mathbf{a}_3$ on the orthogonal reference system. Laue introduced the reduction

$$\begin{aligned} A_1 &= k[(\alpha - \alpha_0)a_{1x} + (\beta - \beta_0)a_{1y} + (\gamma - \gamma_0)a_{1z}], \\ A_2 &= k[(\alpha - \alpha_0)a_{2x} + (\beta - \beta_0)a_{2y} + (\gamma - \gamma_0)a_{2z}], \\ A_3 &= k[(\alpha - \alpha_0)a_{3x} + (\beta - \beta_0)a_{3y} + (\gamma - \gamma_0)a_{3z}], \end{aligned} \quad (9)$$

which allowed him to write (7) in the form

$$A_{R_0} = \psi \frac{\exp(-ikR_0)}{R_0} \times \sum_{l_1=0}^{2M_1-1} \exp(iA_1 l_1) \sum_{l_2=0}^{2M_2-1} \exp(iA_2 l_2) \sum_{l_3=0}^{2M_3-1} \exp(iA_3 l_3). \quad (10)$$

Taking into account that

$$\sum_{l=0}^{2M-1} \exp(iA l) = \frac{\sin M_1 A_1}{\sin \frac{1}{2} A_1} \exp\left[\frac{i(2M_1 - 1)A_1}{2}\right], \quad (11)$$

Eq. (10) can be written in a more condensed form,

$$\begin{aligned} A_{R_0} &= \psi \frac{\exp(-ikR_0)}{R_0} \exp\{i[M_1 A_1 + M_2 A_2 + M_3 A_3 - \frac{1}{2}(A_1 + A_2 + A_3)]\} \\ &\times \frac{\sin M_1 A_1}{\sin \frac{1}{2} A_1} \frac{\sin M_2 A_2}{\sin \frac{1}{2} A_2} \frac{\sin M_3 A_3}{\sin \frac{1}{2} A_3}. \end{aligned} \quad (12)$$

The intensity at the point of observation is the product of A_{R_0} by its