It also proves that the asymmetry effect around a multibeam point is intrinsically due to interference among different Bragg waves and its existence does not depend on absorption, refraction, diffraction geometry, crystal shape and boundary conditions. The good agreement between the present theory and the Si 442 experimental data, as well as the exact n-beam dynamical calculations, indicates the validity of the perturbational approach, at least in the weak-scattering regime. It is hoped that more experimental and theoretical investigations may be generated in the future in the area of multibeam diffraction effects and the phase problem in X-ray crystallography.

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The Current Status of Phase Determination by Means of Multiple Bragg Diffraction

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Abstract

The feasibility of multibeam diffraction for determining phases of structure factors is assessed on the basis of recent contributions and experiments in progress. It is shown that the method works well in situations in which the global interaction between X-ray photons and the crystal is weak, in which case diffraction takes place by single scattering events, and crystal perfection does not play a role in interpreting the experimental results. Two successful examples of phase determinations using the notion of virtual Bragg scattering are presented. One case is of particular interest, because the crystal (V_3Si) is mosaic, and the phases were *a priori* unknown. Some problems and limitations of the method are encountered when trying to extend this technique to organic crystals of relatively large cell size, with spherical or irregular shape. Some data and calculations are presented for benzil, $C_{14}H_{10}O_2$, a crystal isomorphous with quartz. It is concluded that the resolution presently available from standard laboratory set-ups is not adequate for application of this method to a crystal like benzil, but that use of synchrotron radiation beams will probably remove these obstacles.

1. Introduction

It is indeed a pleasure to write a paper dealing with multiple-beam diffraction for this issue of *Acta Crystallographica*, because this was in fact a topic in which Paul Ewald had a special interest. The last two of Ewald's papers in *Acta* are precisely on this subject (Ewald & Héno, 1968; Héno & Ewald, 1968) and it is fitting to make reference here to these two papers and appreciate the progress made during the last eighteen years.

There has been, recently, a renewed interest in the idea of using multiple Bragg scattering for extracting

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phases in diffraction experiments (Colella, 1974; Post, 1977; Gong & Post, 1983; Chapman, Yoder & Colella, 1981; Chang, 1982). The idea had been around for some time (Lipscomb, 1949; Fankuchen & Eckstein, 1949; Hart & Lang, 1961), but progress was hindered by computational complexities that made it imperative to use large digital computers for numerical solutions. The two papers by Ewald and Héno mentioned earlier show how analytical closed-form solutions are hard to come by, and then only in special situations.

A general solution applicable to the interpretation of Renninger plots,* with consideration of the Bragg case of diffraction, was presented by one of us (Colella, 1974) and compared with experimental results obtained from a perfect crystal (germanium). While the comparison between the calculated and measured integrated intensities was satisfactory, one basic problem became immediately apparent. It was not clear how dynamical theory, the only one that preserves phase information, can be applied to 'real' (imperfect) crystals, the only ones for which phase problems are encountered.

Further progress was made in 1981, when the concept of 'virtual Bragg scattering' was discovered (Chapman, Yoder & Colella, 1981). It was shown then that the asymmetries on the wings of the Renninger peaks carry phase information and it was also surmised that they should be independent of crystal perfection. Further work (Tischler, Shen & Colella, 1985; Schmidt & Colella, 1985) demonstrated the correctness of these ideas. It seems at this point that the way is paved for the ultimate, not yet achieved, goal of this research project: namely, the solution of an unknown structure.

However, the situation is not that simple. Most cases in which real phase problems do exist involve organic macromolecular crystals. The application of *n*-beam diffraction to such crystals is not obvious. The large size of the crystal cell results in a densely populated reciprocal lattice, which means that the Renninger peaks are numerous and *very close* to one another. Most macromolecular crystals are composed by light atoms, with weak scattering factors. More importantly, the theory is normally developed for crystals in the shape of plane-parallel slabs, which makes it easy to write down the boundary conditions for the electric and magnetic fields. Normally, the crystals used by crystallographers for structural determinations are tiny fragments with undefined shapes or, in many cases, with spherical shapes. It is not clear what are the effects of not using plane-parallel slabs in Renninger plots.

The purpose of this paper is to assess the present situation and to analyze and evaluate the problems to be solved in order to make further progress. The work done so far will be summarized, and some recent results will be reported concerning the effect of shape and the extension to organic crystals with large unit cells.

2. Method of attack

The basic idea is to realize a situation in which the global interaction between X-ray photons and a crystal is weak. Multiple scattering is then minimized, and the distinction between dynamical and kinematic theory disappears. It is advantageous in such a case to use dynamical theory, because phases are preserved there and hopefully can be sorted out from intensity data. A convenient mode of operation is to select a weak reflection, corresponding to planes (*HKL*) parallel to the surface, and to adjust conveniently the azimuthal angle φ around the scattering vector in such a way that another node (*hkl*) is *close* to, but not coincident with, the Ewald sphere. The *hkl* reflection is, in general, strong, but weakly excited. The perturbation induced on the *HKL* two-beam intensity carries phase information, and is independent of crystal perfection. Since such perturbations can be felt a few degrees away, on the φ scale, from the center of the Renninger peaks (Chapman, Yoder & Colella, 1981), owing to the effect of virtual Bragg scattering, and the intensity varies smoothly with φ in those regions, the mosaic structure of the crystal does not play any role. Phase information can then be sorted out by comparing computed Renninger plots, obtained on the basis of pre-assigned phases, with the experimental plots. Various structural models, corresponding to different phase assignments, can be tried out, and the best fit will provide the answer.

While the *NBEAM* program (Colella, 1974) is able to execute the computations needed for this kind of comparison, provided a sizable digital computer with a relatively large memory and 12–13 significant digits is available,* it is possible to exploit the perturbational nature of our approach and develop an analytical formalism that is valid only on the wings of the Renninger peaks. Such a formalism has, in fact, been developed by one of us (QS) and is described in the preceeding article (Shen, 1986). Using this formalism, the computations needed for interpreting *n*-beam Renninger plots can be executed using a desk calculator.

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* By Renninger plot is meant, in this paper, a plot of the intensity I_{HKL} of a given *HKL* reflection, integrated with respect to θ, the angle of incidence on the (*HKL*) lattice planes, as a function of φ, the azimuthal angle of the crystal around the scattering vector *HKL*. A Renninger peak is a peak in such a plot.

* All the computations referred to in this paper have been executed with a CDC 6600 computer. A typical rocking curve (101 points) for a three-beam case requires about 23 s, and an eight-beam case requires 342 s. The memory size, for a maximum of ten beams, is about 33 000 words (60 bits each).

3. Successful determinations

An ideal situation for testing the feasibility of using virtual Bragg scattering for phasing structure factors is one in which a given phase can be varied in a known way by turning a knob on the apparatus. Such a wonderful opportunity is offered by the 442 reflection in silicon, which can be made positive or negative depending on temperature. The 442 is a forbidden reflection, which owes its existence to the bonding charges along the tetrahedral bonds in diamond structures, and to anharmonic motion of the thermal atomic vibrations.

The coexistence of these two effects, and their relative importance at different temperatures, was demonstrated in a famous and beautiful experiment (Trucano & Batterman, 1972), recently repeated in the context of a more extensive investigation on forbidden reflections in silicon and germanium using synchrotron radiation (Tischler & Batterman, 1984).

The experiment shows that the intensity of the 442, starting from room temperature, *decreases* with temperature, vanishes at about 523 K, and then increases again. This result was interpreted, following a previous suggestion put forward by Dawson & Willis (1967), as due to competition between two opposing effects. The atomic site symmetry in silicon is not centrosymmetric. For example, along a [111] direction, on one side of any atomic site there is a bonding charge at $\frac{1}{8}$, $\frac{1}{8}$, $\frac{1}{8}$ and a nearest neighbor at $\frac{1}{4}$, $\frac{1}{4}$, $\frac{1}{4}$, whereas the opposite side is an empty cavity. Presumably the thermal vibrations will be anisotropic, with a charge build up, in the time average, on the side opposite to that of a nearest neighbor. Such an effect is temperature dependent, whereas the bonding charge is presumably not affected by temperature. From Fig. 4 of the paper by Trucano & Batterman (1972), it appears that F_{442} has the same magnitude at room temperature and 700 K, but (presumably) opposite sign. In fact, a phase determination of the 442 was inferred by Tischler & Batterman (1984) in their recent experiments at the Cornell synchrotron (CHESS).

We felt that this experiment should be repeated with particular attention being paid to the wings of the Renninger peaks. The results are shown in Figs. 1 and 2, and the interpretation is unmistakable. The details on the wings of the Renninger peaks predicted by our theory are faithfully reproduced in the experiment, and the sign identification is fully confirmed.

The next step is to show that this method can also be used for a 'real' crystal, to which dynamical theory would not ordinarily be applicable. A case in point is V_3Si, a cubic centrosymmetric *A*15 structure, for which a number of thermally excited forbidden reflections were predicted to exist (Borie, 1974). Two such reflections have been found and carefully monitored as functions of temperature between 5·5 and 500 K: the 140 and the 153 (Schmidt & Colella, 1986).* The 140 essentially reflects the lack of cubic symmetry of each V site, and is found to decrease with decreasing temperature. It is shown by Borie (1974) that $F_{140} = 32 f_V \exp(-M_V)\alpha$, where f_V is the atomic scattering factor for V atoms, $\exp(-M_V)$ is their Debye–Waller factor and $\alpha = 2\pi^2(\langle x^2\rangle - \langle y^2\rangle)$, where the time-averaged quantities in parentheses are the mean-square vibrational amplitudes of V atoms, projected along two different {100} directions, arbitrarily called *x* and *y*. In order to determine the direction along which the thermal displacement is greater, we need to know the *sign* of F_{140}. This has been done using multibeam diffraction, and the results are shown in Fig. 3. The unmistakable conclusion is that F_{140} is *negative*, which can be interpreted (Borie, 1974) as meaning that the displacements of V atoms perpendicular to the chains are *greater* than those parallel to the chains.

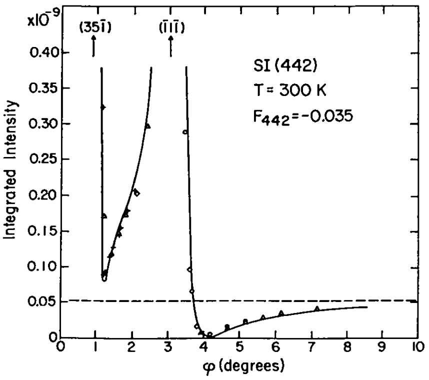

Fig. 1. Integrated intensity (with respect to the angle of incidence θ) of the 442 reflection of Si *vs* φ, azimuthal angle, at 300 K. The horizontal line corresponds to the two-beam intensity which has been used for standardizing all points on an absolute basis. The continuous solid line is calculated from theory, using ten beams.

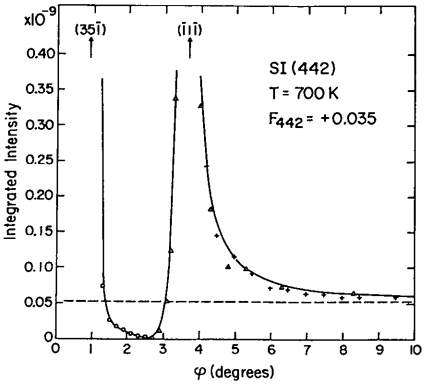

Fig. 2. Same as Fig. 1, except for temperature and sign of F_{442}.

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* The 140 had previously been seen by Borie (1981).

Another interesting reflection for which a successful phase determination has been accomplished is the 153 in V_3Si. It is shown by Borie (1974) that $F_{153} = -256\beta\, f_V \exp(-M_V)$, where $\beta = 4\pi^3\langle xy^2\rangle$. β would normally be zero for a harmonic crystal. A non-zero F_{153} is a *direct* indication of anharmonicity. The experimental results are shown in Fig. 4 and the conclusion is that F_{153} is negative. The results of this work can be used for critical tests of theoretical models of anharmonicity in V_3Si.

The work on V_3Si is particularly important because it shows the feasibility of multibeam diffraction and virtual Bragg scattering with mosaic crystals.

4. Present difficulties and problems looming ahead

It seems at this stage that the way is paved for a complete solution of the phase problem *via n*-beam diffraction. This is true in principle, but there are still practical problems along the way whose solution is far from being at hand.

One obvious limitation, evident in the examples given above, is the fact that all other structure factors, the strong ones involved in generating the Renninger peaks, were known. This may not always be the case, and it is not clear how such a problem can be tackled in general.

Another limitation is that the orientation of the crystal must be completely known, in order to index properly all observed reflections. This may not be possible, for example, when enantiomorphism is present. We have considered the case of benzil, a crystal isomorphous with quartz, a good example of the kind of crystals for which phase problems are actually encountered in crystallographic laboratories. Since benzil can be found in two different space-group forms, it would seem that n-beam diffraction should be able to distinguish between the two forms. In fact it does, but the problem is that the attribution of Miller indices to Bragg spots in orientation photographs (Laue, precession, Weissenberg *etc.*) depends on which of the two possible space groups the crystal belongs to (D_3^6 or D_3^4). The interpretation of any conceivable n-beam diffraction experiment *depends on this initial choice*, and there is no way to remove the ambiguity between D_3^6 and D_3^4. This problem does not exist, to be sure, with anomalous dispersion, which has, in fact, been used, in the case of quartz and other enantiomorphous crystals, to identify the space group unequivocally.

Another important problem, which is being investigated at the present time in our laboratory, in collaboration with Professor M. Colapietro and his collaborators at the CNR Crystallographic Laboratory, Monterotondo Stazione, Italy, is the effect of sample *shape* on Renninger peaks. The theory used to calculate Renninger peaks is based on the hypothesis that the crystal is shaped as a plane-parallel plate, which is used in establishing the boundary conditions. Actually, most crystals used for structural determinations have spherical, ellipsoidal or totally irregular shape. It is not clear to what extent the results of the computations depend on crystal shape. The perturbational treatment of n-beam diffraction presented in a separate article of this issue (Shen, 1986) does not make use of boundary conditions of any sort. It would appear, then, in view of the good agreement of that treatment with experiment, that when the wings of the Renninger peaks are concerned the actual shape of the crystal is not important.

To resolve the issue, a small (0·2-0·4 mm) quartz crystal of approximately ellipsoidal shape was mounted on a standard four-circle diffractometer, and the weak $4\bar{2}2$ reflection was carefully monitored over an azimuthal angle of 360°. Selected portions of the Renninger plot obtained in this experiment were then compared with the same plots obtained from a large parallel-sided quartz crystal. While many groups of Renninger peaks show more or less the same intensity ratios in the two different plots, a gross violation of this rule has been found in at least one case. This

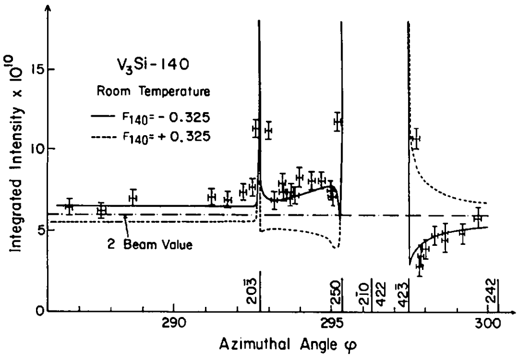

Fig. 3. Integrated intensity of the 140 reflection of V_3Si at room temperature *vs* φ, the azimuthal angle. The solid and dotted lines are calculated from theory using eight beams, with different signs for F_{140}. The horizontal dashed line corresponds to the two-beam value.

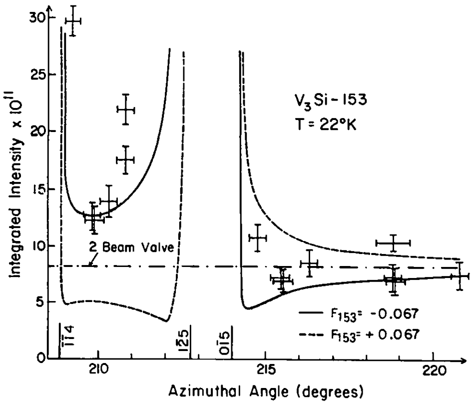

Fig. 4. Similar to Fig. 3, except that the intensity values are for the 153 reflection.

work will be described in more detail in a forthcoming article (Cappuccio, Ferrari, Colapietro, Pifferi, Shen & Colella, 1986).

Even though *n*-beam diffraction does not help in solving the ambiguity resulting from enantiomorphism, we have taken some data on benzil, nevertheless, in order to test the feasibility of the method. The crystal had a very small mosaic spread* and was carefully prepared in the form of a large thick plane-parallel plate, with both surfaces parallel to (101). It was grown from the melt using the Bridgman technique, and then annealed for about two weeks and slowly cooled to room temperature. It was cut with a thin diamond wire, and then polished and etched with acetone. So far we have not been able to see the asymmetry effect on the wings of the Renninger peaks. The reason for this becomes obvious when considering Fig. 5, a typical sample of the many cases we have analyzed theoretically and experimentally. It is clear from Fig. 5, a theoretical profile computed using the *NBEAM* program,† that: (i) the asymmetry effect is small and (ii) the resolution needed on the φ scale ($\simeq 1'$) is normally not available in standard laboratory experiments. The only way to achieve this kind of resolution is to use a beam from a synchrotron light source, which will be done in the near future. The reason for both (i) and (ii) points above is, we believe, that in an organic crystal like benzil the structure factors are usually quite small, because only low-*Z* atoms are present.

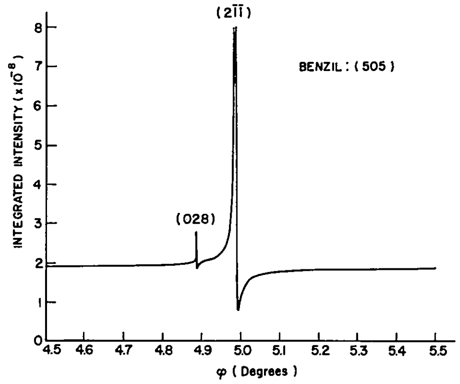

Fig. 5. Azimuthal plot of the benzil 505 reflection, calculated with four beams. Wavelength $\lambda = 2{\cdot}2909$ Å. The zero on the φ scale corresponds to the normal ($\bar{1}20$) lying on the diffraction plane, mostly antiparallel to the incident beam. The two-beam value is smaller than the experimental value shown in Fig. 6 because the structure factor F_{505} calculated from the data of Brown & Sadanaga (1965) is too small. See footnote † on p. 537. The points on the $21\bar{1}$ peak ($\simeq 60\times10^{-8}$) are not plotted here, in order to show more detail on the wings of the peak.

Since we were not able to see asymmetry effects in the Renninger plots of benzil, we tried to compare integrated intensities. In Fig. 6 we show a Renninger peak of 505.* The monochromator was a Si(111), for which the polarization ratio could be calculated exactly using dynamical theory. Cr $K\alpha_1$ radiation ($\lambda = 2{\cdot}2896$ Å) was used for this experiment because the lattice constants of benzil are quite large and the Renninger peaks are normally too close on the φ scale with standard wavelengths ($\lambda = 1{\cdot}4$–$1{\cdot}6$Å). This reflection was chosen because it is relatively weak. The two-beam calculations in fact show that the dynamical and kinematic integrated intensities only differ by 8%. The peak shown in Fig. 6, being only 50% more intense than the two-beam value, was taken to be a good candidate for comparison with theory. We consider, for this comparison, the quantity $P = (A-B)/B$, where A is the integrated intensity R^{θ}_{505}, integrated a second time over φ between two arbitrary limits φ_1 and φ_2, and B is the two-beam value of 505 multiplied by $\Delta\varphi$ ($=\varphi_2-\varphi_1$). In this way P is independent of the structure of the incident beam and P^{calc} can be compared with P^{exp} provided $\Delta\varphi$ is the same for both. For the peak shown in Fig. 6, P^{exp} is $0{\cdot}228$ (with $\Delta\varphi = 1°$) and P^{calc} is $0{\cdot}079$, about three times smaller. We believe that this discrepancy is due to kinematic effects. The Renninger peak of Fig. 6 looks weak, but in fact is contributed by one strong structure factor.† It looks weak only because the experimental width $\Delta\varphi^{\text{exp}}$ (essentially the divergence of the incident beam perpendicular to the diffraction plane) is much greater than the intrinsic width $\Delta\varphi^{\text{theor}}$. A real weak Renninger peak, contributed by weak structure factors, would probably not be visible with the present experimental apparatus.

At this stage, it does not look likely that integrated intensities of Renninger peaks could be used for phase identification in crystals like benzil. It is, however, possible that the substantial improvement in resolution afforded by synchrotron beams may change the situation radically. The tunability and the availability of large intensities at long wavelengths will help in optimizing the conditions for the Renninger plots.

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* The rocking curves were about 3′ wide, most of which was due to intrinsic spectral width of $K\alpha_1$, combined with mismatch between first and second crystals.

† The incident beam is supposed to be a plane wave.

* In order to obtain agreement between the experimental and the calculated azimuthal angles in the Renninger plot of benzil, the following lattice parameters were used: $a = 8{\cdot}417$, $c = 13{\cdot}680$ Å instead of the values $a = 8{\cdot}376$, $c = 13{\cdot}700$ Å given by Brown & Sadanaga (1965).

† $F_{505} = 1{\cdot}547$; $F_{2\bar{1}\bar{1}} = 39{\cdot}16$; $F_{028} = 6{\cdot}171$. These values were obtained and readjusted from data published by Brown & Sadanaga (1965). The experimental absolute two-beam value of the 505 reflection was much too great, about four times the calculated value. Since the 505 is a very weak reflection, we felt that a readjustment of one or two thermal factors was needed. By trial and error, it was found that it was enough to increase $B_{12}(\text{O})$ from $4{\cdot}97$ to $6{\cdot}97$ Å^2 in order to obtain the right value for F_{505}, without affecting appreciably the other structure factors, especially the strong ones.

Experiments in this direction are planned for the near future.

5. Concluding remarks

The method of multibeam diffraction for phasing structure factors has been successfully used in several instances, and in one case it has been used to determine unknown phases in a mosaic crystal. There are several problems, however, to be solved before the method can be applied to attack the phase problem for solving an unknown structure. It is not clear, for example, to what extent dynamical theory developed for a parallel-sided crystal slab can be applied to a small spherical or spheroidal crystal. There are also problems stemming from the poor divergence of the incident beam, perpendicular to the diffraction plane. These problems have to do with the large cell size of molecular crystals for which phase problems are normally encountered, requiring high resolution for the incident beam and tunability in large-wavelength regions, in order to optimize the structure of the Renninger peaks. With the resolution available in standard laboratory set-ups, the asymmetric features on the wings of the Renninger peaks are not visible in an organic crystal like benzil, and the weak Renninger peaks, the only ones that can be treated using dynamical theory, are not measurable. We hope that the beams available at synchrotron radiation sources will provide the resolution needed to overcome these problems.

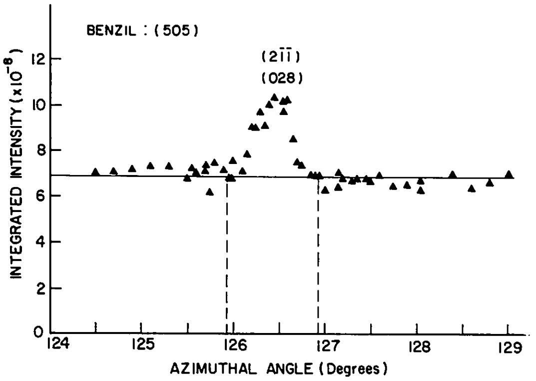

Fig. 6. Experimental values of benzil 505 as a function of the azimuthal angle. The φ values indicated here are direct readings from the experimental apparatus, and correspond to the same convention indicated in the caption to Fig. 5. The time needed to collect the data for this plot was 200 h. The two vertical dotted lines indicate the range $\Delta\varphi$ used for comparing calculated and experimental integrated intensities.

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