Optics

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Source, optical and detector requirements for X-ray diffraction and scattering

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The standard curves used to describe the properties of synchrotron radiation sources usually consist of a plot of the flux or brightness from the source as a function of wavelength. These curves are useful for the case where a high flux or brightness is required. Many experiments do not fall into this category. An alternative description of the source requirements is to provide the maximum flux into the phase space volume defined by the specimen. A diagrammatic way of illustrating how this can be achieved is derived. This illustrates how the source, optics and detectors can be matched to the requirements of a particular experiment. This approach is illustrated using, as examples, a beamline on the SRS and two beamlines planned for DIAMOND, the proposed new UK third-generation source.

Keywords: beamlines; detectors; X-ray diffraction; X-ray scattering.

1. Introduction

A method is presented for specifying the optimum X-ray source, optics and detectors for diffraction and scattering. Simply asking for the highest flux or highest brightness is misleading. A better definition of the requirements would be to have the maximum flux in the phase space volume required by the specimen. The optics can then be used to transform the shape of the phase space volume to match that required by the specimen, so that the size, divergence and wavelength spread of the synchrotron beam are optimized for the specimen. The evaluation of the requirements should, therefore, start from the characteristics of the range of specimens which are to be studied. For X-ray diffraction and scattering experiments, the specimen itself can be considered to be an optical element in the experimental set-up. Fig. 1 illustrates this approach, which is similar to that given by Rosenbaum & Holmes (1980). In an ideal set-up the source would have a dimension and divergence which can be transformed by the optics to match the acceptance of the specimen, and the detector should have a resolution to adequately sample the features in a scattered beam. A diagram is derived in this paper to illustrate how this can be achieved.

2. The sample

The main characteristics of a sample are its physical size and some characteristic repeat or dimension within the sample. The size of the sample determines the dimension of the X-ray beam required and the unit-cell dimensions (or some other dimension for non-periodic samples) determines the angular spread between diffraction features at a particular wavelength and, therefore, the divergence of the X-ray beam required. One can, therefore, define the acceptance of a sample at a particular point in position–angle space using these criteria.

This simple description is adequate for many purposes. However, extra considerations can apply. It is often advantageous to ensure that intrinsically sharp diffraction features are preserved during data collection. This can give an advantage when recording weak diffraction features in the presence of a high background. Knowledge of the sample perfection and the consequent angular broadening of the diffracted beam is, therefore, a relevant parameter to consider. An analysis of this for protein crystals is given by Nave (1998).

3. A diagrammatic way of matching the requirements

Having defined the acceptance of the sample, it is possible to plot its position on a size–angular divergence diagram (Fig. 2). The approximate position for a variety of samples or experiments is shown on the diagram. It must be emphasized that this is merely illustrative. Each type of experiment could cover a significant area on the diagram. As an example of this, the position of a standard lysozyme protein crystal is shown. If the requirement is merely to resolve the diffraction features (with a periodicity of less than 100 Å), the angular acceptance is fairly modest. However, if one wants to exploit the high degree of perfection of such crystals (see Colapietro *et al.*, 1992; Fourme *et al.*, 1995; Snell *et al.*, 1995; Stojanoff *et al.*, 1996, 1997) by collecting data in very fine angular (phi) increments, then a much narrower angular range is required for the incident beam. Similarly, the position for high-pressure powder diffraction experiments is shown for typical sizes of diamond anvil cells. For the consequent small volumes, the individual crystals have to be small if one wants to avoid a spotty powder pattern. The small crystals themselves could then lead to diffraction broadening of the powder rings. In addition, the angular range of the diffracted beam is often degraded at high pressure.

The interpretation of the axes in Fig. 2 in terms of specimen size and beam divergence applies to the case (shown in Fig. 1)

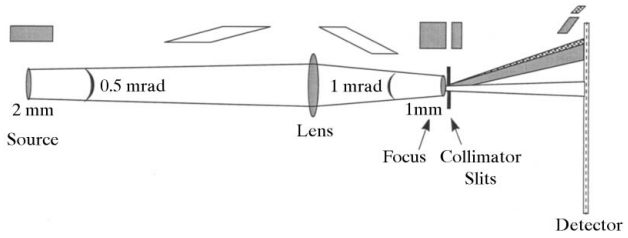

Figure 1
Schematic diagram showing a simple focusing optical arrangement for X-ray scattering in which the 2 mm size source is demagnified to produce a 1 mm size focus with a consequent increase in angular divergence. The incident beam is then collimated down to match the size of the specimen. The specimen intercepts and scatters the incident beam. The scattered beam is broadened (hatched area) with respect to the incident beam due to sample effects. The scattered beam is intercepted by a detector which has a resolution shown by the vertical dashed line along the detector. Position–angle phase-space diagrams are shown for the beam at various positions along the optical path with the horizontal axis representing the beam size and the vertical axis the beam divergence. Similar diagrams can be drawn for the case where one is focusing on the detector instead of the sample.

where one focuses on the specimen. However, the same specimen acceptance arguments, in terms of the size and divergence of the X-ray beam at the focus, apply for the case where one focuses on the detector.

Fig. 2 also shows various sources of radiation and the acceptance of detectors. These are discussed in subsequent sections. Diagrams such as these can also be extended to a third dimension by specifying the wavelength spread accepted by the specimen. In many cases, the wavelength spread can be defined from the angular acceptance using the relation $\delta\lambda/\lambda = \delta\theta\cot\theta$. This illustrates that the wavelength acceptance should be determined from the maximum value of $\cot\theta$, *i.e.* the maximum Bragg angle to be measured. This relation ensures that the wavelength bandpass is sufficiently small to resolve the diffraction features. Many X-ray monochromator systems give a much narrower bandpass than is required for this purpose and there is a resulting loss of incident flux. In some cases (*e.g.* some anomalous-scattering experiments) a narrow bandpass is required. The bandpass required in these cases is determined from the width of the spectral features to be probed.

4. The X-ray source

The small size of many samples means that the incident X-rays have to be concentrated into a small area. In order to resolve the diffraction orders or scattering features, the incident beam has to have good angular collimation. As the scattering power is weak, a large number of photons is required. This implies that a source with a large number of photons s^{-1} mm^{-2} $mrad^{-2}$ is necessary, *i.e.* a high-brightness source. This requirement of high brightness has driven the design both of microfocus X-ray tubes and of synchrotron sources over the past decades.

The emittance of a source is given by the size and angular divergence of the radiation and can be defined separately in the horizontal and vertical directions. Many X-ray sources have little intrinsic angular collimation. This applies to X-ray tubes and, in the horizontal direction, to bending magnet sources of synchrotron radiation. In these cases an aperture is placed some distance from the X-ray source to limit the radiation to within a defined angular range. In general, there is little advantage or disadvantage in using a source with lower emittance than that required by the specimen. Provided the emittance is low enough, the requirement is then for maximum flux. The source requirements can, therefore, be described concisely as that giving the maximum flux in the phase space volume required by the specimen. As a simple example, for conventional X-ray sources, it is common to use larger focal spot sizes on the X-ray target for small-molecule crystallography than for protein crystallography. The resulting larger X-ray flux can be usefully exploited for those cases where the emittance requirements are modest.

The horizontal emittance of three sources is plotted in Fig. 2 together with the flux obtained from these sources within the defined emittance. For the proposed DIAMOND source (a 3 GeV machine) this shows that, for some samples, the multipole wiggler is a better source than the undulator. This is because it has more flux and a sufficiently low emittance for a large number of samples. Multipole wiggler sources have quite complex characteristics as they have a significant depth of source effect. This means that the apparent source size increases as the angular aperture used is increased. It is possible to analyse such sources using a phase-space representation (Dorrsen *et al.*, 1993). Such calculations are useful in achieving the best compromise between a large number of poles and having a sufficiently high magnetic field strength to give the required critical wavelength in the device to produce X-rays of sufficient energy. The emittances of the multipole wigglers shown here have been defined using this procedure.

5. Optics

Fig. 1 shows a simple example of how the optics transform the properties of the beam to match the requirements of a particular experiment. The optics in this case consist of a lens to demagnify the source and a slit to provide spatial collimation. This is used to match the beam size to the specimen size (or in some cases the detector resolution).

Simple focusing optics essentially move the position of the beam along the lines of constant emittance shown in Fig. 2. More

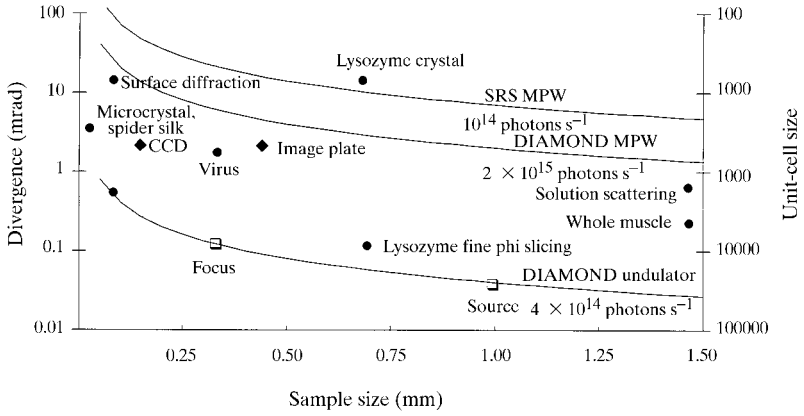

Figure 2
Size–divergence diagram for X-ray scattering. The horizontal axis represents the size of the specimens to be studied and the required divergence of the beam is shown on the left-hand vertical axis. The right-hand axis gives a corresponding unit-cell dimension. This would be on the limit of being resolved with an X-ray beam of this divergence using 1 Å radiation. The same argument applies when focusing on the detector. In this case, the angular resolution of the diffracted beam is determined by the size of the X-ray beam focus at the detector and the specimen size determined by the divergence of the X-ray beam from detector to sample. Three sources are shown as curved lines having emittances of 7, 2 and 0.04 mm mrad. These are full width at half-maximum (2.35σ) values. The flux from these sources in a 0.1% bandpass is shown. This diagram is, therefore, a slice at a bandpass of 0.1% through the 3D size–divergence–bandpass diagram. ● The requirements for some representative samples or experiments. ■ Source and focus for a proposed undulator beamline. ◆ Positions of two detectors for collecting data at 3 Å resolution.

complex behaviour occurs with some types of crystal optics. These can select a particular volume in position–angle–wavelength space and transform the shape of this volume. It is not the intention here to cover this aspect other than to point out that a 3D (three-dimensional) version of Fig. 2 can be derived to illustrate this. The beam can be transformed so that it no longer remains along the line of constant emittance in the size–angular divergence section shown in Fig. 2. An illuminating graphical representation of the way optical systems transform the properties of the beam is given for position–angle space by Matsushita & Kaminaga (1980*a*) and for position–angle–wavelength space by Matsushita & Kaminaga (1980*b*).

6. Detectors

Two detectors are also shown in Fig. 2 in order to illustrate how these could be matched to the experimental requirements. The assumption is that one wishes to measure data to a Bragg spacing ($d_{\min}$) of 3 Å. Both detectors have a spatial resolution which allows measurement of 150 orders of diffraction from the centre of the pattern to the perimeter. They can, therefore, resolve a cell of 450 Å spacing to a Bragg spacing of 3 Å. A smaller detector with the same spatial resolution would not resolve the 450 Å cell and would, therefore, be placed higher up the (vertical) angle axis. The position of the detectors on the size axis is given by its spatial resolution, which ideally should be matched to the specimen size if both the specimen and detector are near the focus.

The two detector systems shown here are of different size and spatial resolution. They can both measure the same number of diffraction orders and, therefore, have, in principle, the same angular resolution for a particular diffraction experiment. The smaller detector requires smaller diffraction features in order to resolve the diffraction orders. It is, therefore, better matched to the diffraction requirements from small specimens. Using this type of detector for studying larger specimens would require the beam to be collimated. This would lead to a loss in flux, if the consequence was that a smaller volume of the sample was illuminated. As shown in Fig. 1, the size of the diffraction features on the detector is a combination of the size of the beam at the specimen and the effect of beam divergence due to the source and specimen properties. The effect of divergence increases with distance from the specimen in a way which matches the difference in spatial resolution for the larger detector placed at a greater distance.

A similar analysis, using the phase-space representation shown in Fig. 1, can be carried out for the case where one focuses on the detector. This arrangement is frequently used, particularly for small-angle diffraction and scattering studies (see also the caption to Fig. 2). It can allow efficient use of detectors with good spatial resolution when studying larger specimens. The constraints are matching the size and spatial resolution of the detector to the beam and specimen properties, with the additional constraint determined by the Bragg resolution ($d_{\min}$).

7. Conclusions

The emphasis in this paper is to discuss a graphical way of matching the source, optics and detectors in position–angle space to the requirements of the experiment. Familiarity with this approach gives an intuitive feel for the requirements of X-ray diffraction and scattering. This can be considered as the first stage in the design process for experimental facilities. It does not replace detailed ray-tracing analyses, which are necessary to ensure the various components are properly specified (and not over-specified). The approach adopted here is also applicable to experiments other than X-ray diffraction and scattering. The commonly shown plots of flux or brightness from synchrotron radiation sources only give a good indication of the capabilities of a source for certain cases. The emphasis here is on a phase-space description of the requirements of the experiments and matching all the facilities to these requirements. This gives a useful indication of how to optimize the performance of any facility for particular experiments.

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