tube (typically a melting-point tube) to a depth of 3 mm and held in place with a thin 'secotine cement' plug. The length of the tube was such that when inserted in the beam-stop tube and the fluorescent end attached, the standard samples were ~2 mm from the open end of the beam stop (see Fig. 1). Usually, the aim was to produce two reflections from the standard material in order to give a more precise calibration. In the original procedure the position of the tube in the beam stop was adjusted by interposing washers between the threaded ends of the beam-stop tube. A more sophisticated method involved the use of an adjustable threaded post mounted in the beam-stop tube carrying a glass capillary of 0·03 mm diameter. This system required precise machining of components but had the advantage of using a smaller diameter powder sample which reduced the width of the diffraction lines giving more precise measurements. One disadvantage was that exposure time was increased.

Both the glass tube and the threaded post were chosen for their close fit with the inside of the beam-stop tube. Hence, if the original camera-manufacturing process was precise and no damage had occurred in use, the external standard was correctly aligned without any adjustment by the operator. If the beam-stop tube has been bent out of alignment then the rotation of the tube will compensate and produce a pattern corresponding to a larger diameter tube, *viz* a broader line profile in the analagous manner to an incorrectly centred sample in the normal sample position providing the damage to the tube is insufficient to remove it from the X-ray beam altogether.

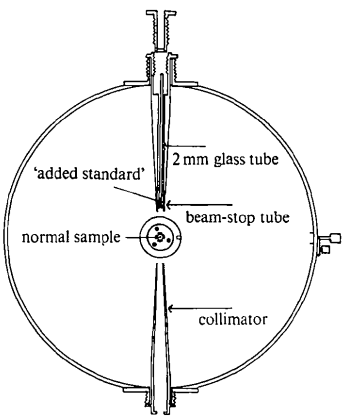

Fig. 1. Plan view of Philips type PW1024 camera showing 'added standard' carried in 2 mm glass tube mounted in beam-stop tube.

The use of standards mounted in the beam-stop tube produced diffraction patterns in the back-scatter region of the film, but did not produce any detactable scatterings at low angles due to the absorption of that part of the pattern by the beam stop, resulting in a very sharp cut-off to the additional standard pattern. The position of the beam-stop cut-off was adjusted by varying the depth of the standard surface below the end of the beam-stop tube as described.

The choice of standard was determined by the duration of the exposure. If the exposure was for a short period (~2 h) then the heavier elements (W, Sb) were used to give reasonable intensities to the calibration lines. Antimony was not preferred because of the large number of lines appearing in the back-scatter region. If the exposure were of longer duration (~6 h) then the lighter elements were used. The standards may be of any crystalline solid which produces a diffraction pattern in the required region. The examples chosen here were air-stable materials spanning the range of scattering power required.

Verification of the procedure was obtained for standards mounted in the normal sample position in the presence of these external standards. When potassium chloride was mounted in the normal sample position the correction factors for film shrinkage based on the potassium chloride and either molybdenum or tungsten powders agreed to 1 part in 10 000 although the '*d*' spacings of the metal powders were in error by 0·008. When allowance for the displacement of the external standard from the axis of the camera was made and the measured arc dimensions corrected using the formula developed by Wilson (1970) the '*d*' spacings for the 310 and 321 reflections of molybdenum and tungsten were within 0·0002 of the accepted values.

Secondary patterns arising from scattering by the normal sample of the beam scattered by the external standard were not observed for any of the materials tested with any of the $K\alpha$ X-rays from iron, copper, molybdenum and tungsten tubes.

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References

Ekstein, H. & Siegel, S. (1949). *Acta Cryst.* **2**, 99–104.

levinš, A. & Straumanis, M. E. (1936). *Z. Phys. Chem. Alot. B*, **33**, 265–274.

Nelson, J. B. & Riley, D. P. (1945). *Proc. Phys. Soc. London*, **57**, 160–177.

Wilson, A. J. C. (1940). *Proc. Camb. Philos. Soc.* **36**, 485–489.

Wilson, A. J. C. (1970). *Elements of X-ray Crystallography*, p. 23. London: Addison Wesley.

Wilson, A. J. C. & Lipson, H. (1941). *Proc. Phys. Soc. London*, **53**, 245–250.

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Symposium on Crystallographic Studies Using Energy-Dispersive Diffraction, American Crystallographic Association, Boston University, Boston, Massachusetts, USA, 12–17 August 1979

The development of intense sources of synchrotron radiation has led to renewed interest in X-ray energy-dispersive diffraction (XRED) as a tool for crystallographic studies. Previous efforts in this area with conventional X-ray sources have been hampered by the complicated energy dependence of the incident intensity. However, in the past two or three years, Buras at DESY and Glazer at NINA and their colleagues have exploited many of the special features of synchrotron radiation in a variety of crystallographic studies with promising results. The energy-dispersive technique was therefore adopted as the theme of a special symposium organized by C. T. Prewitt (SUNY, Stony Brook), D. E. Cox (Brookhaven National Laboratory), B. Buras (University of Copenhagen and Riso National Laboratory) and W. Parrish (IBM, San Jose), and held August 13 and 14 at the American Crystallographic Association Summer Meeting in Boston. Because a number of synchrotrons are presently under construction throughout the world, including the NSLS at Brookhaven, special emphasis was placed on the role of synchrotron radiation in XRED. Travel support provided by the National Science Foundation made it possible to invite some of the principal European practitioners in this field.

The opening talk by L. Gerward (Technical University of Denmark), and

co-author Buras, dealt with special features of synchrotron radiation as applied to XRED. Among these, Gerward emphasized the high incident intensity and consequently rapid data collection, the smooth continuous spectrum and the ability to calculate the spectral distribution from a storage ring from the machine parameters, the high degree of collimation, and the fixed-angle geometry which is particularly well suited to experiments involving samples in special environments. He also discussed optimization of experimental parameters from resolution considerations, and the limitations of current solid-state detectors at high counting rates, and suggested possible exploitation of the time structure of the synchrotron pulses for studies involving periodic processes with appropriately short relaxation times. As examples of crystallographic studies carried out at DESY he quoted high-pressure studies on Bi, and structural refinement of powder data from $BaTiO_3$, urea and naphthalene by means of the Rietveld profile-refinement method.

Some of the characteristics of Si and Ge solid-state detectors were discussed in more detail by A. C. Thompson (Lawrence Berkeley Laboratory). The energy resolution and maximum counting rate are limited by both the device parameters and the processing electronics, with Ge offering potentially better resolution, but at the expense of escape peaks which tend to complicate the spectrum. Fabrication of arrays of detectors offers the prospect of overcoming some of the counting-rate limitations. Some further consideration of these detectors and their application to X-ray scattering measurements was provided by C. T. S. Sparks Jr (Oak Ridge National Laboratory), who paid particular attention to how they could be used to discriminate between the elastically scattered radiation and the various types of background radiation such as fluorescence, Compton, Raman resonance and *Bremsstrahlung*. Some other factors are transparency effects, which can cause apparent shifts in line positions, substrate effects and air scattering. He emphasized the advantages of rapid data collection, reduction in background, separation of higher-order wavelength components, and the ability to monitor scattering and fluorescence simultaneously. Against this must be offset the limited counting rates and energy resolution, typically 1–2%.

Some recent applications of XRED were described in subsequent talks. T. Egami (University of Pennsylvania) described the determination of the pair distribution function in amorphous alloys using a conventional tungsten target. Better statistical accuracy is obtained than with conventional angular dispersive methods. The fixed-angle geometry is an important feature of these studies, whereas the limited energy resolution is relatively unimportant. Careful attention must be paid to the data analysis, however, which is complicated by a lack of knowledge about the energy dependence of the incident intensity, the absorption and various geometrical factors, and corrections for Compton and multiple scattering. By Fourier transformation of the corrected data, valuable information about the atomic structure of and structural relaxation in amorphous alloys can be obtained.

Another aspect of the XRED techniques was covered by J. B. Hastings (Brookhaven National Laboratory), namely the accurate measurement of very weak Bragg peaks in single-crystal experiments. Spurious effects such as multiple diffraction can be minimized by appropriate selection of wavelength and geometry, and the signal-to-noise ratio can be improved with appropriate mirrors or filters. As examples he presented measurements on the forbidden 222 reflection in diamond-type structures. The technique also offers the possibility of rapid single-crystal data collection with a multi-detector array.

The advantages of the fixed-angle geometry and rapid counting rate for crystal-structure studies at high pressures was demonstrated by G. Will (University of Bonn), who presented powder data for various mineralogically important systems obtained with a conventional tungsten source and two types of pressure cell, a miniature piston-cylinder press for up to 4 GPa, and a diamond-anvil squeezer for up to 25 GPa. Analysis of the data is accomplished by profile refinement, and the technique allows a reduction in counting time by a factor of seven or so. Preliminary experiments at DESY showed further improvements. High-pressure studies in a diamond-anvil cell were also described by F. A. Mauer, with co-authors S. Block and G. J. Piermarini (National Bureau of Standards, Washington), who emphasized the importance of proper collimation and particle size, and presented results obtained for CdS, CuCl, and ice VII. Fixed-angle geometry was also exploited by M. Mantler (Technical University of Vienna) in some high-temperature experiments on the crystallization of amorphous SiO_2 and the α–γ transition in iron. In the latter, the progress of the critical temperature through the specimen can be observed with a time resolution of less than a second.

The analogous neutron time-of-flight technique was covered by J. D. Jorgensen (Argonne National Laboratory), who discussed data collection and analysis using this method, with particular reference to the forthcoming pulsed neutron source, which has the advantage that no chopper is required. He described a structural study of polycrystalline NiF_2 carried out at high pressures, in which the Rietveld profile refinement clearly demonstrated a lowering of symmetry. The time-of-flight technique was further discussed by A. H. Reis Jr with co-authors S. W. Peterson, J. M. Overhage, A. J. Schults, J. Kolenko and P. Day (Argonne National Laboratory) in a talk about the current status of their single-crystal diffraction system planned for IPNS. This is based upon a multi-wire position-sensitive detector to enable data to be collected in the Laue mode over a wavelength range 0·6 to 3·5 Å. Encouraging preliminary results have been obtained on a small crystal of NaCl.

The symposium was well attended and generated some lively discussion, and provided an opportune forum to demonstrate the advantages and limitations of the energy-dispersive techniques for crystallographic studies. It appears that the initial impact of intense synchrotron sources will be in investigations of polycrystalline or amorphous (perhaps liquid) systems in highly restrictive environments such as pressure cells, furnaces, or cryostats, but there is certainly considerable potential for single-crystal data collection as well. The current limitations of solid-state detectors were clearly defined, but some cautious optimism about improvements in performance was expressed.

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