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A comment on the method of determination of the polarization ratio for crystal-monochromated X-rays by Vincent & Flack.

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Abstract

By reference to previously unpublished tests, the validity of the experimental procedure proposed by Vincent & Flack [*Acta Cryst.* (1980), A**36**, 614–620] for the measurement of the polarization factor is questioned.

Introduction

In a recent group of papers (Vincent & Flack, 1980*a*,*b*; Flack & Vincent, 1980), aspects of the polarization ratio for crystal-monochromated X-rays have been treated. A method of determining this ratio was proposed (Vincent & Flack, 1980*b*) which involved two series of measurements, one with a β filter and the other with a monochromator crystal. The numerical results reported appear to differ significantly from others recorded in the literature.

Comments arising from experimental work of a similar type, carried out in 1972 but not previously reported, are offered.

Procedure

The procedure used was based on use of a number of low-intensity reflexions of α-glycine ($C_2H_5NO_2$), with 2θ values selected to span the effective range of the diffractometer (Picker). Choice of the low-intensity reflexions was to avoid or at least minimize the effects of extinction [a naive viewpoint held at the time of the experiment but subsequently modified (Mathieson, 1979)]. Measurements were carried out using Cu $K\alpha$ radiation (*a*) with a β filter and (*b*) with a pyrolytic graphite crystal mounted on a device (Mathieson, 1968) which allowed ready change from one configuration to the other. The change involved lowering the scintillation counter, removing the monochromator crystal and inserting the β filter. Being a post-monochromator arrangement (see Mathieson, 1968), the basic components of the experiment, the glycine crystal and the diffractometer, were presumed not affected in any way.

To test the effectiveness of the procedure, a graphical presentation was preferred. This was based on the following; for the monochromator in the perpendicular position, Fig. 1, with counter azimuth, $\varphi = 90°$ (see Mathieson, 1968), the diffracted intensity is proportional to $k + \cos^2 2\theta$ where k is the polarization ratio of the monochromator crystal, while with the β filter it is proportional to $1 + \cos^2 2\theta$. The ratio of intensity with the monochromator to that with the β filter, $R_{m/\beta}$, is therefore given by

$$R_{m/\beta} \propto (k + \cos^2 2\theta)/(1 + \cos^2 2\theta). \quad (1)$$

This can be rearranged:

$$R_{m/\beta} \propto (k - 1)(1 + \cos^2 2\theta)^{-1} + 1, \quad (2)$$

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so that a plot of $R_{m/\beta}$ against $(1 + \cos^2 2\theta)^{-1}$ should yield a straight line, the value of k being derived from the slope. Note that (2) is symmetrical about $2\theta = 90°$.

The tests which I carried out established to my own satisfaction that the procedure was not generally suited to its particular purpose. For the 0004 reflexion of pyrolytic graphite, a typical run, Fig. 2(*a*), gave what appeared to be an acceptable result. The slope gave an estimate of the polarization factor, $k = 0{\cdot}389$, which might be interpreted as 78% $\cos^2 2\theta$ plus 22% $\cos 2\theta$. However, for the more practically important 0002 reflexion, the readings were rather scattered, Fig. 2(*b*), and clearly were unlikely to yield an acceptable numerical value. Attempts to improve the situation and determine the cause or causes of the wide divergence of values were unsuccessful and this investigation was, at the time, laid aside.*

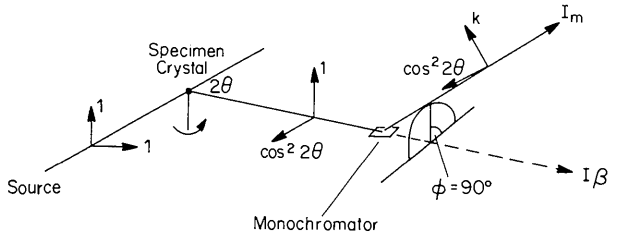

Fig. 1. Diagram of the experimental arrangement, with various polarization components indicated. I_m is the intensity measured with the monochromator crystal in the diffracted beam path while I_β is the intensity measured with the β filter in the beam path. $R_{m/\beta}$ is the ratio of I_m to I_β, adjusted in scale if required.

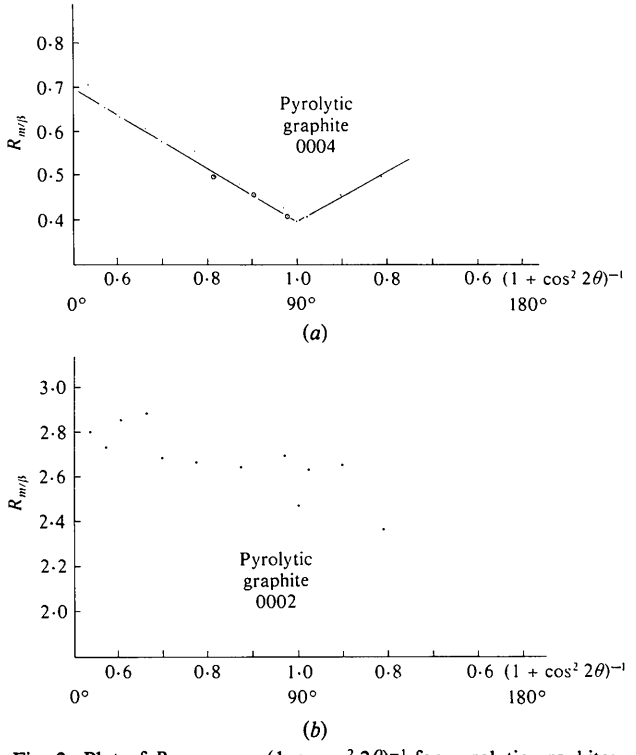

Fig. 2. Plot of $R_{m/\beta}$ *versus* $(1 + \cos^2 2\theta)^{-1}$ for pyrolytic graphite: (*a*) for the 0004 reflexion; (*b*) for the 0002 reflexion. In (*a*), circled points are from the regions beyond $2\theta = 90°$, being 'reflected' into the region below $2\theta = 90°$.

Conclusion

The evidence of our experience, taken in conjunction with the anomalously low values that Vincent & Flack (1980*b*) report, suggests that this procedure is inappropriate for the determination of the polarization factor. This conclusion need not reflect upon the theoretical content of the other papers in this series. The subject of polarization is a difficult one with much remaining to be done; see the recent critique by Jennings (1981).†

The more direct procedure referred to by Le Page, Gabe & Calvert (1979), which also uses a graphical procedure, is preferred. With a slightly more elaborate device (Mathieson, 1968), measurement of the polarization ratio can be made, not only for the integral value, but also across the reflexion profile of a pyrolytic graphite crystal (Calvert, Killean & Mathieson, 1974*a*,*b*).

There is, of course, an experimental arrangement, involving a monochromator crystal, for which experimental determination of the polarization factor is unnecessary (Mathieson, 1978).

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* Evans, Hine, Richards & Tichy (1980) have also noted a difference in the behaviour of the 0004 and 0002 of pyrolytic graphite as a post-monochromator. These authors suggested that the difference is associated with the very large area but my own view is that it is more likely to arise from the large difference in 'level of interaction' (Mathieson, 1979).

† Note in Fig. 2 of Jenning's paper that he recognizes the existence only of values of k above the kinematical value.

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