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An ambiguity in indexing Renninger diagrams and the distinction between some opposite directions in cubic crystals.*

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In the course of a discussion of the nature of an ambiguity in the indexing of Renninger peaks the possibility was discovered of distinguishing some opposite directions in a cubic crystal without recourse to anomalous scattering. A multiple-diffraction experiment performed on a (111) Ge plate rotated about [222] enabled distinction between the $[1\bar{1}0]$ and $[0\bar{1}1]$ directions to be made. The azimuthal angles counted from alternative zero meridians containing $[1\bar{1}0]$ or $[0\bar{1}1]$ are the same, but give rise to peaks indexed *hkl* and $2-l,2-h,2-k$ respectively. However, dynamically equivalent situations 222, $hkl/(2-h,2-k,2-l)$ and 222, $(2-l,2-h,2-k)/lhk$ are geometrically different, being distinguished as Bragg–Laue and Bragg–Bragg, respectively, or *vice versa*.

A multiple-reflection, Renninger diagram (Renninger, 1937) is indexed by calculating the azimuthal angle that the crystal must rotate from a preselected origin until a given reciprocal-lattice point (RELP) reaches the Ewald sphere. For instance, in the Renninger diagram for germanium when the primary reflection is 222 the origin was taken in that position when the vector $[1\bar{1}0]$ coincides with the plane of incidence pointing towards the X-ray source. $[0\bar{1}1]$, displaced by 60°, is an equivalent choice. Identical azimuths and intensities are calculated for both origins, but the corresponding peaks are assigned different indices (see Table 1), thus giving rise to an ambiguity which, however, can be solved as shown below.

The origin of the azimuths is indeterminate and one does not know which set of indices is the correct one for Renninger peaks observed in a particular experiment. It is to be noticed that the only relevant indeterminacy is the one provided by two origins of the azimuths 60° apart, since those which are 120° apart can be identified simply by renaming the axes a_H and b_H in a hexagonal system of reference. The question might, at first sight, seem trivial or irrelevant. In fact it is not so.

Representing a three-beam case by

$$(A)\ (000)\ (222)\ (hkl)/(2-h,2-k,2-l)$$

where (*hkl*) is the secondary reflection and $(2-h,2-k,2-l)$ is the coupling reflection, it is easy to verify that the situation

$$(B)\ (000)\ (222)\ (2-l,2-h,2-k)/(lhk)$$

appears 60° away. The relation between situations *A* and *B* is quite interesting since, if for a given *hkl* set of indices *A* is a Bragg–Laue case, then *B* is a Bragg–Bragg case and *vice versa*. If one lets the indices *hkl* take the values of the secondary reflections in column 2 of Table 1, then the corresponding *B* cases are in column 3.

Moreover, situations *A* and *B* differ in that the roles of the secondary and of the coupling reflections are interchanged (see Fig. 1), but as has been shown by Ewald & Héno (1968) the dynamical interaction is the same for both cases. Then, instead of considering the diffracted intensities, it will be much more convenient to identify cases *A* and *B* on the basis of the geometry in reciprocal space. The pair (*A*) (000) (222) $(\bar{1}\bar{1}1)/(331)$ and (*B*) (000) (222) $(133)/(1\bar{1}\bar{1})$, where strong reflections are involved, was chosen in order to facilitate the measurements. It can be seen that the geometrical difference between the two cases lies simply in the position of the secondary RELP relative to the equator of the Ewald sphere. The former is a Bragg–Laue and the latter a Bragg–Bragg case.

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Table 1. *Indexing of the Renninger diagram of* Ge(222) *taken with* Cu $K\alpha_1$ *radiation referred to origins* $[1\bar{1}0]$ *and* $[0\bar{1}1]$ 60° *apart*

Azimuthal angle (°)	Origin on $[1\bar{1}0]$ Secondary/Coupling	Origin on $[0\bar{1}1]$ Secondary/Coupling
2·087	$3\bar{5}1/\bar{1}71$	$1\bar{1}7/13\bar{5}$
3·484	$51\bar{1}/\bar{3}13$	$3\bar{3}1/\bar{1}51$
5·424	$113/11\bar{1}$	$\bar{1}11/311$
7·735	$3\bar{1}5/\bar{1}3\bar{3}$	$\bar{3}13/53\bar{1}$
13·820	$33\bar{5}/\bar{1}5\bar{3}$	$\bar{3}\bar{1}5/53\bar{3}$
13·976	$53\bar{1}/\bar{3}53$	$33\bar{5}/1\bar{5}\bar{3}$
14·161	$31\bar{1}/\bar{1}13$	$3\bar{1}1/\bar{1}31$
15·720	$3\bar{5}3/\bar{1}7\bar{1}$	$\bar{1}\bar{1}7/33\bar{5}$
18·302	$1\bar{1}5/13\bar{3}$	$\bar{3}13/51\bar{1}$
18·561	$5\bar{1}\bar{1}/\bar{3}33$	$3\bar{3}3/\bar{1}5\bar{1}$
21·869	$\begin{cases}13\bar{1}/153\\513/3\bar{1}\bar{1}\end{cases}$	$\begin{cases}31\bar{5}/\bar{1}1\bar{3}\\\bar{1}\bar{3}1/351\end{cases}$
22·081	$511/3\bar{1}1$	$1\bar{3}1/151$
22·326	$1\bar{3}5/15\bar{3}$	$\bar{3}15/51\bar{3}$
25·180	$\bar{1}\bar{1}3/33\bar{1}$	$\bar{1}33/3\bar{1}\bar{1}$
28·212	$\bar{1}\bar{1}1/331$	$133/1\bar{1}\bar{1}$
29·522	$\bar{1}\bar{1}3/35\bar{1}$	$\bar{1}35/3\bar{1}\bar{3}$
29·883	$1\bar{5}3/17\bar{1}$	$\bar{1}17/315$

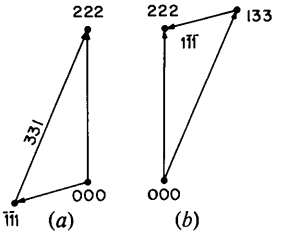

Fig. 1. Geometries of (*a*) case *A* (000,222,$\bar{1}\bar{1}1$/331) and (*b*) case *B* (000,222,133/$1\bar{1}\bar{1}$). It is to be noted that both cases include the same vectors.

To distinguish between cases *A* and *B* the following experiment was performed. The crystal was aligned to measure the primary reflection with the counter set at the proper angle to monitor the primary intensity. The idea is then that if the situation conforms to case *B* (Bragg–Bragg) the secondary reflection could be measured on the same side of the crystal as the incident beam, while if it is in case *A* (Bragg–Laue) the secondary reflection would be eventually detected on the other side of the crystal.

In a given experiment, the intensity of the primary reflection around the azimuth 28·212°, corresponding to the secondary reflection $\bar{1}\bar{1}1$ or 133 depending on whether case *A* or *B* was being produced, was measured. In order to distinguish between the two cases the detector was moved to that position on the same side of the crystal where reflection 133 would appear in the Bragg–Bragg case (*B*). That this was actually the case is demonstrated by the intensities recorded in both measurements, shown in Fig. 2.

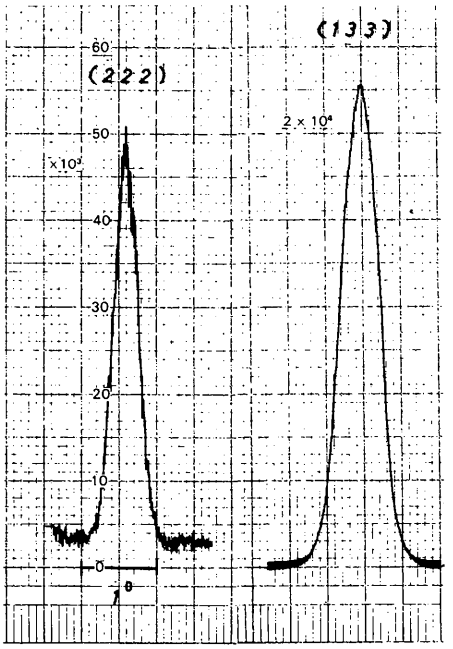

Fig. 2. The recorded intensities of the primary (222) reflection and of the secondary (133) reflection in the case of (000,222,133/1$\bar{1}\bar{1}$) (Cu $K\alpha_1$).

Once the Bragg–Bragg case has been identified the corresponding *umweg* peak in the Renninger diagram is attributed the indices 133, then the other multiple diffraction peaks can be indexed without ambiguity.

The method can be applied whenever there exist a couple of corresponding dynamical situations, involving two three-beam cases, one of the Bragg–Bragg and the other of Bragg–Laue type, where the secondary and the coupling reflections are interchanged. In the case of cubic structures we have actually proved that any pair of reflections 60° away in the Renninger diagram satisfy this exigence. In conclusion, we have thus been able to distinguish between two situations which are dynamically equivalent, thus producing the same intensity measurement, on the basis of diffraction geometry.

It is observed that the method makes a clear distinction between the two directions [1$\bar{1}$0] and [0$\bar{1}$1] which served to mark the origins of the Renninger diagram. Since any of them is transformed by the threefold axis into the opposite of the other, this implies that by using this method one is able to distinguish between the direction [1$\bar{1}$0], and its opposite [$\bar{1}$10] without recourse to anomalous dispersion.

Finally, it is worth while to point out that the method just discussed does not provide a general way to distinguish among equivalent reflections. In fact, since germanium belongs to the centrosymmetric space group *Fd3m*, it is impossible to distinguish between the direction [222] and its opposite [$\bar{2}\bar{2}\bar{2}$]; moreover, the distinction would not make any sense from a physical point of view since the structure looks exactly alike from both directions and the X-ray diffracted intensities are exactly the same, as they would be for any centrosymmetric crystal.

However, after choosing the indices of the primary reflection, and because of the peculiarities of the diffraction geometry, some particular opposite directions in the structure can be distinguished, as has been shown above in the case of [1$\bar{1}$0] and [$\bar{1}$10].

References

EWALD, P. P. & HÉNO, Y. (1968). *Acta Cryst.* A**24**, 5–15.
RENNINGER, M. (1937). *Z. Phys.* **106**, 141–176.

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Seminvariants in space groups $P312$, $P3_112$, $P3_212$, $P\bar{6}$, $P\bar{6}m2$ and $P\bar{6}c2$.

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Errors in the tables given by Hauptman & Karle [*Acta Cryst.* (1956), **9**, 45–55] are corrected.

The number of reflections needed to fix the origin in the space groups $P312$, $P3_112$, $P3_212$, $P\bar{6}$, $P\bar{6}m2$ and $P\bar{6}c2$ is 1, not 2 as stated by Hauptman & Karle (1956), Giacovazzo (1974) and by Karle (1974) in *International Tables for X-ray Crystallography*. The seminvariant vector in these space groups is $(2h + 4k + 3l)$ and the seminvariant modulus is 6. This is equivalent to the pair of congruences $(h - k) \equiv 0 \pmod 3$ and $(l) \equiv 0 \pmod 2$. Since in all other space groups the number of elements in the seminvariant vector is equal to the number of reflections needed to fix the origin, it is