MODIFIED REFLECTION OF X-RAYS: NAPHTHALENE

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1. *Introduction*

THE new type of X-ray reflections, recently discovered by Raman and Nilakantan, have attracted the attention of several workers in this field and the subject is now being pursued in various laboratories. We are, at present, engaged in making a detailed study of this phenomenon and some results obtained with calcite have been reported by us earlier in these *Proceedings.*[1] In the course of an attempt to extend the investigations to the region of organic crystals, we have noticed some interesting results in naphthalene and an account of these is given in the present paper.

2. *Experimental Technique*

The source of X-rays is a Shearer tube with a copper anticathode. The incident beam of X-rays, which is carefully collimated by a system of slits, thus contains CuK_α and CuK_β rays along with a certain amount of white radiation. The technique adopted by us for recording the new reflections is similar to the one already described by Raman and Nilakantan[2] in connection with the (111) planes of diamond and by the authors (*loc. cit.*) in connection with the (211) planes of calcite. This technique and the results to be expected therefrom are described here in some detail as it is believed that such a description will clearly bring out the distinction between the normal and the modified X-ray reflections.

A particular set of planes in the crystal is chosen for investigation and by knowing the appropriate spacing, the crystal is set such that these planes are inclined to the incident pencil of X-rays approximately at the correct Bragg angle.* If the reflected spectrum is then recorded, it is clear that one would normally expect to find only a single Laue spot. Such a spot may be attributed to a Bragg reflection from the set of planes under investigation. The reflected beam will consist of CuK_α radiation itself, if the crystal

[1] *Proc. Ind. Acad. Sci.*, 1940, **12**, 337. References to other investigators in this field are contained in our earlier paper.

[2] *Ibid.*, 1940, **11**, 389.

* Correct Bragg angle referred to here relates to CuK_α radiation with $\lambda = 1 \cdot 537$ A.U.

setting is quite accurate. Otherwise, it will consist of either the CuK_β radiation or white radiation of appropriate wave-length lying in the close neighbourhood of the characteristic wave-lengths. The position of this Laue spot will depend very much upon the crystal setting and it will gradually move away from the centre as the crystal is rotated so as to slowly increase the angle of incidence. In addition to this normal type of Bragg reflection, two new spots, which have to be regarded as modified reflections, are usually recorded in this mode of investigation. These two spots come at places where the ordinary Bragg reflections due to CuK_α and CuK_β would appear when the crystal is set suitably for such reflections to take place and they continue to appear at those places or shift only slightly, even when the crystal is tilted considerably away from the critical settings. This behaviour is in marked contradiction to the behaviour of the Bragg reflections. The intensity of the modified spot due to a certain wave-length, say CuK_α, reaches a maximum when the crystal is so oriented that the planes under investigation are also causing CuK_α to be reflected in accordance with the usual Bragg law. It falls off gradually and sometimes rapidly as the crystal is rotated away from this setting. This technique is particularly suited for picking out specific planes, when the crystal structure is known, and studying the modified reflections from them.

3. *Results*

An organic crystal, namely naphthalene, has been studied in the present investigation. It belongs to the monoclinic prismatic class and crystallises as plates parallel to the (001) plane. This is also a marked cleavage plane and shows an intense Bragg reflection. The spacing is 7·251 A.U. and the corresponding angle of reflection for CuK_α is 6° 5′. The second order reflection by this set of planes (002) occurs at an angle of 12°7′ and is somewhat less intense. Two pictures (Figs. 1 and 2) which have been obtained with this crystal are reproduced in the Plate. The crystal flake is so mounted that the *b*-axis is vertical and that the (001) plane makes about 6° (Fig. 1) and 12° (Fig. 2) with the incident pencil of X-rays. In Fig. 1, the setting is therefore 6° away from the critical setting appropriate to the (002) planes and in Fig. 2, it is similarly 6° away from the critical setting appropriate to the (001) planes. No trace of any modified spot is to be found in the expected place in either case, even though the normal Bragg reflections are recorded very intensely on the axial line. This result is very interesting as it has been the experience of the authors that the modified spots are quite easily recorded at moderately large angles, especially in the inorganic crystals.

With a view to decide whether such reflections are altogether absent in this crystal or are very weak, pictures have been taken in which the exposures and the resolution are carefully controlled. Figs. 4, 5 and 6 correspond to inclinations of 5°, 6° and 7° respectively of the (001) planes. An aperture of 1·5 mm. diameter has been used in the path of the incident beam with a view to increase the resolution and the exposure time has been cut down with a view to prevent spreading and overlapping. In Figs. 4 and 6, the setting is just one degree different from the proper setting of the (001) planes in respect of the CuK_α radiation and to the left of the intense spot in the former case and to its right in the latter case, may be seen the faint Bragg reflections of appropriate wave-lengths. The intense spot in both these cases is to be regarded as the modified reflection arising from the (001) planes. At larger angles, the modified spot does not appear. If longer exposures are given at smaller angles, the spots are not seen separately because of overlapping due to spreading. Modified reflections from the (002) planes also behave similarly.

Other orientations of the crystal have also been tried but modified spots could not be recorded with appreciable intensity for any of the settings. In the case of the ($20\bar{1}$) plane, which has a large structure factor, a weak modified spot has, however, been recorded at very close angles. This may be seen as a weak spot immediately to the right of the intense Laue spot due to ($\bar{2}01$), seen on the axial line in Fig. 3.

We have accordingly to conclude that the modified reflections in naphthalene crystals possess appreciable intensity only in the close neighbourhood of the correct Bragg settings. Their intensity falls off very rapidly as the deviation from the Bragg setting increases.

4. *Summary*

Modified X-ray reflections due to (001), (002) and ($20\bar{1}$) planes of naphthalene have been recorded only when the crystal setting is very near that of the critical setting in each case. For orientations which differ appreciably from the above settings, the intensity of the modified spots appears to be very low.