X-RAY STUDIES ON POLYCRYSTALLINE GYPSUM

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1. Introduction

Gypsum is one of the most important of the commoner minerals and the different forms in which it is found and their structural characters are therefore matters of considerable interest. Brauns' *Das Mineralreich*[1] and Hintze's *Handbuch der Mineralogie*[2] contain illustrated accounts of the subject. We are concerned in this paper with the polycrystalline forms of gypsum. Of these the best known is the white and semi-opaque material known as alabaster. Mineralogists recognise another form which they designate as "fibrous gypsum". As the name indicates, this form is an aggregate of parallel fibres or rods. Hintze mentions that when the fibres in such gypsum are very fine, their direction is parallel to the mineralogical *c*-axis, while if the material consists of coarser fibres, their direction may depart to a greater or less extent therefrom.

In a recent paper in these *Proceedings*,[3] it has been shown that we must recognize *three* polycrystalline forms of gypsum with specific characters which are readily distinguishable from each other. The first is *alabaster* which is an aggregate of small crystals more or less randomly orientated: the second is *satin-spar* which is a truly fibrous variety of gypsum in which the fibres lie along the mineralogical *c*-axis, while the third form has been designated as *fascicular gypsum* and is an aggregate of rods more or less exactly parallel to the *b*-axis which is also the symmetry axis of the crystal-structure. This third form is readily distinguished from the other two by its external appearance. It exhibits optical phenomena of great beauty and interest which have been fully described and explained in the paper referred to above. In the present communication, we report the results of X-ray studies undertaken to confirm and reinforce the conclusions already reached in that paper from the optical evidence. We have thought it desirable to carry out such an investigation for the reason that the third form of gypsum is of common occurrence and it would seem that those who have noticed it have mistaken it for the second or fibrous species. Our X-ray studies show clearly that the direction of the rods in fascicular gypsum is along the *b*-axis, while in the truly fibrous forms it is along the *c*-axis. Further, while the X-ray patterns show that satin-spar gives a typical fibre diagram, fascicular gypsum does not do so, showing thereby that it is not fibrous in the proper sense of the word.

2. ALABASTER

This polycrystalline form of gypsum varies greatly in its appearance in different specimens, some being chalky-white and opaque even in thin layers, while other specimens appear translucent. These differences are evidently connected with the extent to which light can penetrate through the material and this again appears to depend on the size of the crystallites of which it is composed. Selecting a translucent specimen of alabaster and thinning it down to a plate about a millimetre thick, the X-ray diffraction diagram reproduced as Fig. 1 in Plate III was recorded. A collimated beam of MOK_α radiation was employed. The slit width was one millimetre and a flat film camera was used with a specimen to film distance of three centimetres. It will be seen that the diagram exhibits spotty rings. Though the pattern is not altogether symmetric, it is sufficiently nearly so to justify our regarding the material as consisting of crystallites randomly orientated in space.

3. SATIN-SPAR

The specimens employed were detached from the surface of a beautiful museum piece which externally manifests a fibrous and brilliant silky lustre. Figure 2 in Plate III is an X-ray diagram taken with a flat camera and unfiltered MO radiation: the incident beam travelled along the fibre direction. The specimen employed was about a millimetre in length and of about the same cross-section. It will be seen that the pattern exhibits numerous and nearly complete concentric circular rings.

Figure 4 in Plate IV was recorded with a cylindrical camera and CuK_α radiation. The specimen in this case was a rod about one centimetre long and about half a millimetre in thickness. It was held inside the camera with its length parallel to the axis of the cylinder and the X-ray beam traversed the specimen in a direction perpendicular to its length. The specimen was kept stationary during the exposure. Nevertheless it will be seen that the pattern is a typical rotation diagram. It was verified by measurement of the separation of the layer lines that the length of the fibre was parallel to the mineralogical *c*-axis of gypsum. The conclusion thus indicated that satin-spar is a truly fibrous material orientated along the morphological *c*-axis is, however, only valid for the material used which was detached from the external surface of the museum specimen. When a thin plate cut from the latter with its faces transverse to the fibre direction was normally irradiated by an X-ray beam in a flat-film camera, the record appeared both spotty and irregular, thus indicating a departure from the regularity of orientation shown by the material detached from its outer surface.

4. Fascicular Gypsum

As already stated, this material exhibits highly characteristic external features which enable it to be readily identified. It may be readily split into slabs or sheets of any thickness, but the surfaces thus exposed do not exhibit the smoothness and optical perfection characteristic of the cleavage of selenite and are more appropriately described as planes of parting. The most distinctive feature of the mineral is that the edges of the blocks or slabs are not smooth but exhibit parallel ridges. The inference suggested by the external appearance of the material is that it is an aggregate of rods in parallel setting, their direction coinciding approximately with the *b* or the symmetry axis of gypsum.

Figure 5 in Plate IV is an X-ray pattern recorded with cylindrical camera and CuK_{α} radiation. The specimen examined was a rod detached from the material and set with its length parallel to the axis of the camera. It was kept stationary during the exposure. The pattern exhibits irregularly disposed spots. The configuration of the latter indicated that the length of the rod was not quite parallel to the *b*-axis, but was slightly inclined to the latter. The rod was turned through $3\frac{1}{2}°$ from the previous position, but nevertheless the pattern then obtained did not exhibit the characteristic features of a rotation diagram. However, when the specimen was oscillated through 30° each way, a record was obtained which is reproduced in Fig. 6 in Plate IV and clearly exhibits layer lines. These are much closer to each other than, those appearing in Fig. 4 in the same Plate. Measurements indicated that the oscillation axis coincided with the *b* or symmetry axis of gypsum.

Figure 3 in Plate III shows an X-ray diagram obtained with a flat film camera and the MO radiation, the beam being parallel to the length of the rods. It will be seen from the pattern that the X-ray reflections recorded appear as patches located roughly on short arcs of circles. The picture indicates that while the rods consist of individual crystals having their *b*-axes parallel, the variations of orientation of the other two crystallographic axes are insufficient in magnitude to give a complete pattern of circular rings. The inferences regarding the nature of the material indicated both by the longitudinal and transverse X-ray photographs thus support each other. The X-ray results are also in agreement with the conclusions already drawn from the optical investigations and described in the earlier paper.

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