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X-RAY CRYSTALLOGRAPHY OF RINKITE

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Introduction

The name rinkite was proposed by Lorenzen (1884) for a mineral found at Kangerdluarssuk, Greenland. From the beginning, the symmetry of rinkite was very controversial. Lorenzen (1884) described it as monoclinic with

$$a:b:c: = 1.569:1:0.292 \qquad \beta = 88°47'$$

Optical axial plane normal to (010), $c\wedge\gamma = 7.5°$. Frequent polysynthetic twin lamellae (100). Zambonini (1921) did not find these twin lamellae, and stated that $c\wedge\gamma = 0°$ or, at the most, 0.5°. Gordon (1924) obtained

better morphological measurements but no evidence on symmetry. He did not find twin lamellae either. It is worth noting that according to Gordon's figures rinkite is clearly pseudo-orthorhombic, as the angle $(001)\wedge(101)$ is 11°26′ and the angle $(001)\wedge(\bar{1}01)$ is 11°21′; the two values are within the experimental error range. Gossner and Kraus (1933) could not find any deviation from orthorhombic symmetry by x-ray; they report the following lattice constants:

$$a = 18.47 \qquad b = 5.67 \qquad c = 7.46 \text{ (kX)}$$

Later on, Gossner and Kraus (1934) admitted monoclinic symmetry with the same lattice dimensions and $\beta = 91°13'$, which is the old morphological value of Lorenzen (1884). Bøggild (1957) summarized previous papers on rinkite, but some of his statements represent an original contribution to the rinkite problem:

"Zambonini cannot find the twin lamellae recorded by Lorenzen, which must be said to be very strange, as according to my experience they occur in great quantities in every section. In opposition to the obliquity of extinction of $7\frac{1}{2}°$, recorded by Lorenzen in sections

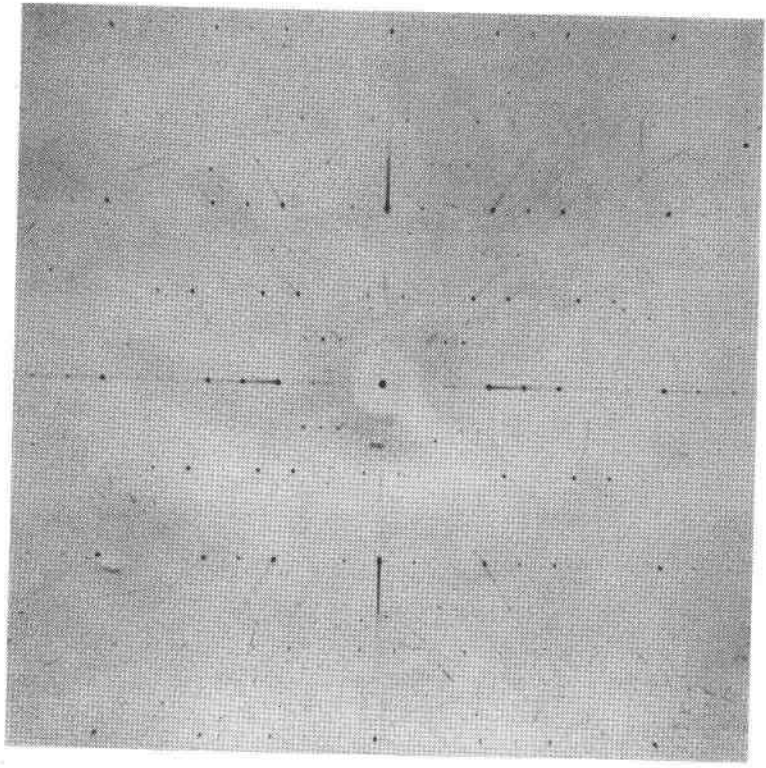

FIG. 1. Precession b_a (zero layer) of rinkite. Zr-filtered, Mo radiation.

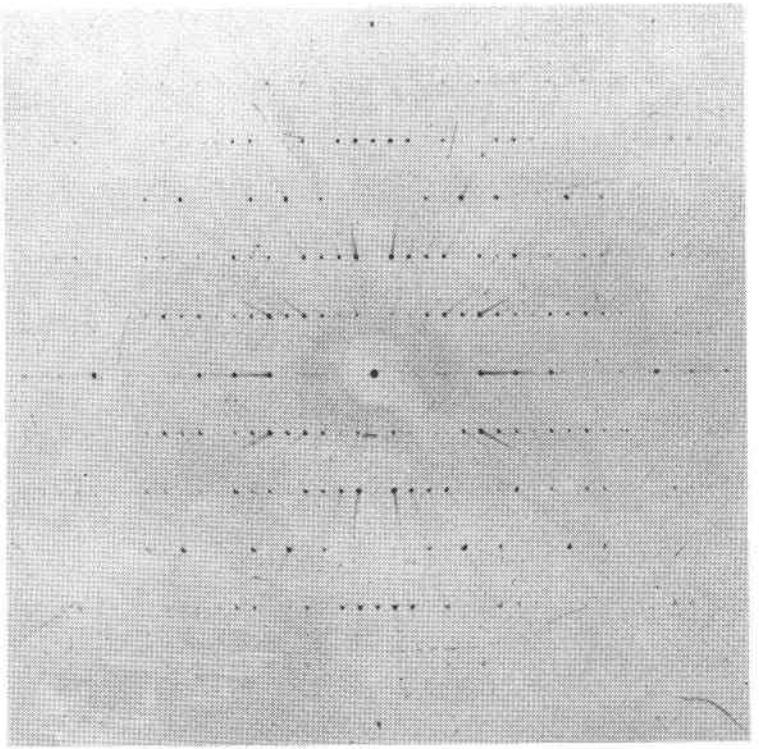

FIG. 2. Precession c_d (zero layer) of rinkite. Zr-filtered, Mo radiation.

parallel with (010), Zambonini only finds a very small obliquity c.0.5°, which more or less corresponds with what I have observed; the obliquity of the extinction it would in reality be very difficult to demonstrate without the twin lamellae, which make them extremely distinct".

So, it is clear that rinkite is pseudo-orthorhombic also from an optical point of view.

Slepnev (1957) claims rinkite to be monoclinic with

$$a = 18.52 \quad b = 5.71 \quad c = 7.46\ (\text{Å}) \quad \beta = 91°30' \text{ or } 92°$$

Sahama and Hytönen (1957) shift rinkite to triclinic symmetry with

$$a = 18.51 \quad b = 5.64 \quad c = 7.45\ (\text{Å})$$
$$\alpha = 90° \quad \beta = 101° \quad \gamma = 91°$$

(Here b and c of Sahama and Hytönen are interchanged).

The lattice constants and the symmetry of rinkite seem to be rather variable in space and time. I wanted to resolve its structure and planned intensity measurements but was unable to index my Weissenberg pic-

tures either with Slepnev's or with Sahama and Hytönen's constants, so it was necessary to start anew.

Experimental

Rinkite from Kangerdluarssuk, kindly lent by Naturhistoriska Rijksmuseet, Stockholm, (Specimen n.94:0075, the same used by Sahama and Hytönen) has been *x*-rayed with Zr-filtered Mo-radiation on a Buerger precession retigraph, with Corni and Gottardi (1964) modifications. The following photographs have been taken with a tiny (100) lamella (150×150×30 microns):

Precession a_d, zero and second layer
Precession b_d, zero and first layer
Precession c_d, zero and first layer

The exact meanings of a_d b_d c_d will be explained later on.

It is best to describe the rinkite units cell in two steps:

1) If we consider only strong reflections, or normal exposure films, rinkite appears to be orthorhombic with:

$$a_0 = 18.46 \pm 0.04 \qquad b_0 = 5.66 \pm 0.01 \qquad c_0 = 3.72 \pm 0.01 \text{ (Å)}$$

(o for orthorhombic)

This is the first unit cell of Gossner and Kraus (1933) with c halved. There is no deviation from orthogonality. Figure 1, with precession b_d (zero layer), and Fig. 2, with precession c_d (zero layer), and Fig. 2, are sufficiently clear to ascertain this point. The precession a_d (zero layer) film is not shown here, as Sahama and Hytönen also admit that $\alpha = 90°$. Plane symmetry mm is evident in all three photographs.

2) If weak reflections also are taken into consideration, or if long exposure films are examined, rinkite is monoclinic with b as unique axis.

Figure 3 shows a schematic drawing of the (h0l) plane of the reciprocal lattice. Weak reflections fall at the center of each a^* c^* parallelogram. These weak reflections show only plane symmetry 2, in both (h0l) and (h1l) photographs, in the (hk0) photograph they indicate plane symmetry mm and in the (hk1) photograph plane symmetry m. So rinkite is definitely monoclinic. Space group $P\,2_1/m$ or $P\,2_1$.

These results exclude the possibility of a (100) polysynthetic twin having been *x*-rayed: such a twin would always exhibit orthorhombic symmetry in *x*-ray films.

Figure 3 suggests three possibilities of choosing new axes suitable to index all reflections:

1) To double both a_o and c_o; the new constants would be then:

$$a_d^* = \tfrac{1}{2}a_0^* \qquad b_d^* = b_0^* \qquad c_d^* = \tfrac{1}{2}c_0^* \qquad \beta_d^* = 90°$$

$$a_\alpha = 2a_0 = 36.92 \qquad b_\alpha = b_0 = 5.66 \qquad c_\alpha = 2c_0 = 7.44 \text{ (Å)} \qquad \beta_\alpha = 90°$$

(d for doubly primitive monoclinic cell)

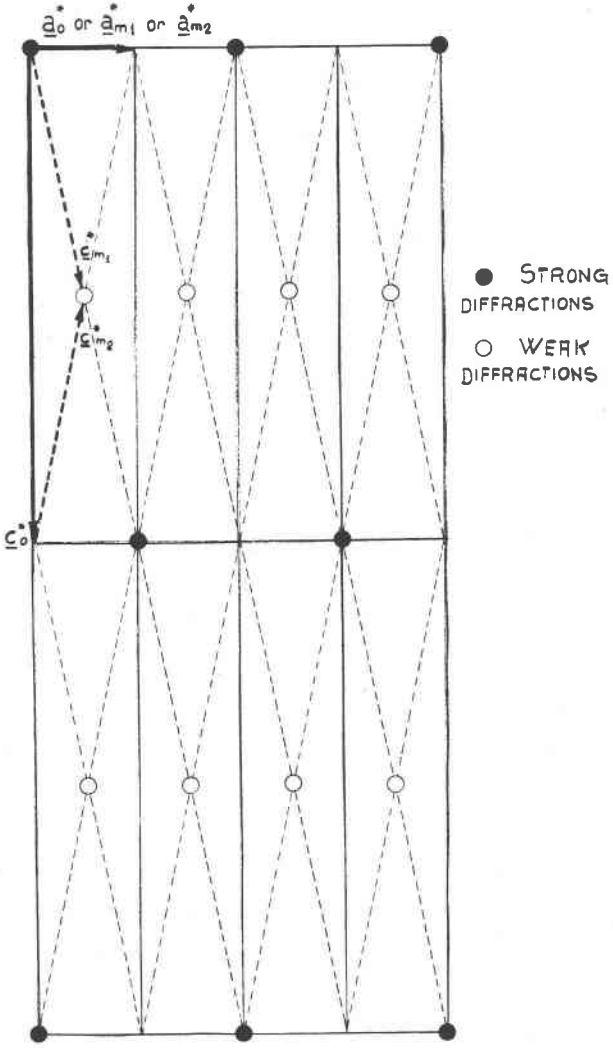

FIG. 3. Schematic drawing of the (h0l) layer of the reciprocal lattice of rinkite, showing the positions of strong and weak reflections, and the different ways of choosing a^* and c^* axes.

Space group: $B2_1/m$ or $B2_1$

2) To choose a monoclinic cell with

$$a_{m1}^* = a_0^* \quad b_{m1}^* = b_0^* \quad c_{m1}^* = \tfrac{1}{2}a_0^* + \tfrac{1}{2}c_0^* \quad \beta_{m1}^* = 90° - \text{arctg}\,(c_0/a_0)$$

$$a_{m1} = a_0 + c_0 \quad b_{m1} = b_0 \quad c_{m1} = 2c_0 \quad \beta_{m1} = 90° + \text{arctg}\,(c_0/a_0)$$

(m1 for primitive monoclinic cell of type 1)

$$a_{m1} = 18.83 \quad b_{m1} = 5.66 \quad c_{m1} = 7.44\ (\text{Å}) \quad \beta_{m1} = 101°22'$$

Space group $P\ 2_1/m$ or $P\ 2_1$

3) To choose a monoclinic cell with

$$a_{m2}^* = a_0 \qquad b_{m2}^* = -b_0^* \qquad c_{m2}^* = \tfrac{1}{2}a_0^* - \tfrac{1}{2}c_0^* \qquad \beta_{m2}^* = \beta_{m1}^*$$
$$a_{m2} = a_0 - c_0 \qquad b_{m2} = -b_0 \qquad c_{m2} = -2c_0 \qquad \beta_{m2} = \beta_{m1}$$

($m2$ for primitive monoclinic cell or type 2)

Unit cell dimensions and space group: as above.

The two monoclinic cells of type 1 and 2 can be distinguished only with intensity considerations. Reflections (505) and ($\bar{5}05$) (doubly primitive reference) are suitable for this purpose.

I propose that with reference to the doubly primitive monoclinic cell, the stronger has the index (505), the weaker ($\bar{5}05$); in unitary monoclinic reference of type 1 the stronger has the index (005), the weaker ($\bar{5}05$)—

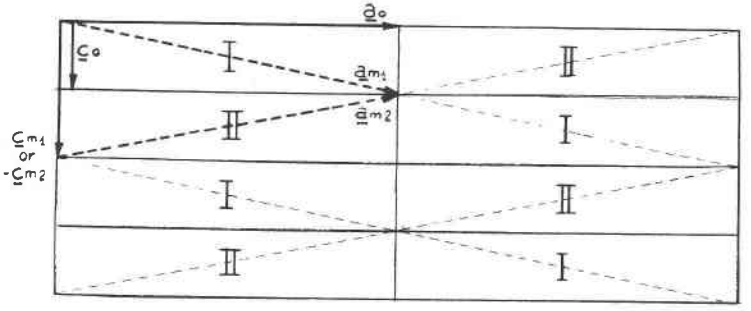

FIG. 4. Schematic drawing of the (010) direct lattice plane, showing the different ways of choosing the a and c axes.

the latter indices being reversed in unitary monoclinic reference of type 2. Fig. 4 is a drawing of the different direct lattices. The smallest unit cells (the orthorhombic ones), outlined by a continuous line, are coded I or II alternately. The cells with different code numbers must be very similar, but they cannot be equal. So the identity periods must be changed so as to have a cell with at least a double volume, as in the unitary monoclinic cells of type 1 or 2, or a fourfold volume, as in the doubly primitive monoclinic cell.

A complete structure determination is in progress in our Institute. This research was supported financially by Centro Nazionale di Cristallografia del C.N.R., Roma.

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MACKINAWITE FROM THE WITWATERSRAND CONGLOMERATES

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INTRODUCTION

Investigations by several workers (Evans *et al.*, 1962; Kuovo *et al.*, 1963; Chamberlain and Delabio, 1965) have shown that mackinawite, tetragonal iron sulfide containing varying amounts of Ni and Co, is a common though minor constituent of a great variety of rocks and economic mineral deposits. Until recently known as "valleriite" (Blomstrand, 1870), it has been lately separated from the latter, since the "valleriite"-type has proved to consist of two distinct mineral phases, namely valleriite proper, having a chemical composition close to $CuFeS_2$, and tetragonal FeS, mackinawite (Evans *et al.*, 1964). Although differing both in crystal structure and chemical composition, these two minerals are very similar in their appearance under the microscope and in their paragenetic associations which rendered a distinction between them rather difficult. Once familiar with the subtleties of their optical properties, however, it is in most cases possible to separate them without difficulty in polished section. Chamberlain and Delabio (1965) have tabulated certain optical and physical properties which help to serve as distinguishing features.

MICROSCOPIC PHYSIOGRAPHY AND CHEMICAL COMPOSITION

In the course of an ore-microscopic investigation of Witwatersrand gold conglomerates from the Orange Free State goldfield (South Africa)