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Lattice parameters of talc as measured by a back-reflection pseudo-Kossel technique

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Abstract

A back-reflection divergent beam method has been developed which enables lattice parameters to be measured with high precision in large single crystals. The method consists of a double exposure back-reflection technique employing a screen of tungsten wires to obtain direction cosine values of incident and diffracted beams. The lattice parameters of talc have been measured before and after heating to 700°C.

Introduction

According to Daw *et al.* (1972), the thermal decomposition of talc takes place in two well-developed stages. Below 650°C, approximately 0.6 percent of the total weight is lost. This is believed to be mechanically-held water, and the activation energy for this reaction is much lower than that of the second stage, dehydroxylation, which takes place at 800°C and above. As part of a study of the kinetics of decomposition, the results of which have been reported by Varela *et al.* (1977), lattice-parameter measurements have been made on single-crystal or lamellar talc from Ouro Preto, Brazil. While the X-ray powder diffractometer can give accurate d spacings for the planes lying parallel to the surface, in this case $c \sin \beta$, other reflections are too weak or there is too much overlapping to yield accurate lattice parameters. The crystals, however, are too large to use single-crystal techniques and deform too easily on cutting.

The pseudo-Kossel technique using a source of divergent X-rays outside the crystal can overcome the need to cut the crystal and can accommodate large single crystals. Difficulties due to inaccurate measurements of crystal–film and crystal–source distances can be overcome by using a direction cosine method involving a double exposure.

Experimental procedure

Pseudo-Kossel back-reflection photographs were taken with a Rigaku microflex generator employing CuK radiation. A screen of tungsten wires between the crystal and the film enabled direction cosines to be determined by the method of Shrier *et al.* (1966). Each film was exposed twice with a displacement of the cassette by an amount Δz measured with a micrometer to 0.01 mm. The precision was estimated to be ±0.02 mm for 90 percent confidence. Coordinates x and y on the film were measured with an Enraf-Nonius microdensitometer fitted with a 3× telescope. Statistical trials showed the highest precision possible was ±0.005 mm for 90 percent confidence, so that overall precision is determined by the micrometer reading. The precision of the method is also determined by film shrinkage, which can be allowed for, buckling of the film in the cassette, and non-parallel movement of the cassette.

Bragg angles, interplanar angles, and d spacings were calculated from the experimentally-determined direction cosines of the normals to the planes by using the equations of Shrier *et al.* (1966).

The talc was in the form of green, foliated crystals from Ouro Preto, Minas Gerais, Brazil. The analysis was as follows: MgO = 30.04 percent, SiO_2 = 61.13 percent, FeO + Fe_2O_3 = 3.26 percent, CaO = 0.04 percent, and Al_2O_3 = 0.04 percent. The loss of weight on heating to 1200°C was 4.95 percent. The samples used were carefully cleaved and were approximately 20 × 10 × 2 mm. They were mounted vertically in a

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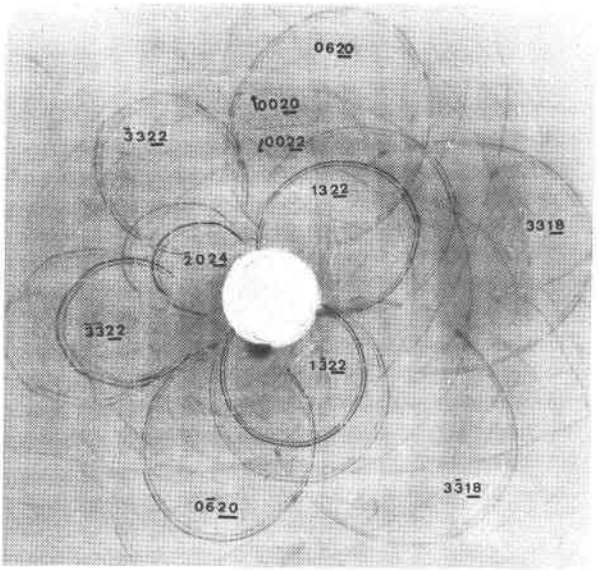

Fig. 1. Double exposure pseudo-Kossel photograph of a talc crystal (Cu*K* radiation).

goniometer and set parallel to the film cassette. The specimen-to-target distance was approximately 4 mm and the specimen-to-film distances were approximately 60 and 80 for the two exposures.

Heat treatments were in air, the same specimen being heated at 500°, 600°, 700°, and 800°C for 1 hour.

Experimental results

A typical photograph is shown in Figure 1. The lines were indexed by a comparison of d spacing and hkl-001 interplanar angles with data calculated from the monoclinic lattice of talc reported by JCPDS (1969). In addition, a comparison of intensities was made with those calculated from the atomic positions of the talc lattice from the data of Wyckoff (1968). The indexing was also confirmed by a computer-produced plot of the pseudo cones by using estimated crystal–film and crystal–source distances. A representative diagram is shown in Figure 2. The equation of Newman (1970) was used.

In Figure 1 the discontinuities caused by the tungsten wires can be seen, and it should also be noted that there is a pseudo-mirror plane from 10 o'clock to 4 o'clock. The Kossel results showed, however, that a triclinic lattice is correct, confirming the results of Raynor and Brown (1973).

Data for the 10 planes are shown in Table 1. It can be seen that there is a large imprecision in the values of θ. There are several reasons for this. The values chosen here are a combination of several (up to 20) sets of readings of 3 discontinuities. In the case of talc, it is highly likely that the crystal will be bent, and as there is a spread of position from which the diffracted beam originates, the irregularity in the crystal surface will be reflected in the d-spacing values. The data here are calculated for 90 percent confidence using the procedure of Harris (1964). In addition, certain reflections such as 0 0 22 are of low intensity and the precision of the readings is consequently lower.

These 10 readings are reduced to 6 by forming a system of normal equations, taking into account and weighting those data with higher precision. The 6 equations are then solved for reciprocal and real lattice parameters by the method of least-squares of Whittaker *et al.* (1932).

Table 2 shows the final results obtained for the talc crystals, together with the root mean square residual values for the 6 lattice parameters. The three untreated crystals T6, T3, and T8 show a certain variation in lattice parameter, the most striking being the change of the γ angle from being acute to obtuse. This parameter, however, is the most imprecise because $\cos\gamma^*$ depends on the values of the h and k indices. Reference to Table 1 shows that the peaks used in the analysis all had values of h and k that were low in comparison to l, thus lowering the precision.

A further interesting observation is that the a and b parameters are lower than those found by all other workers, while the c value is similar. The α value is

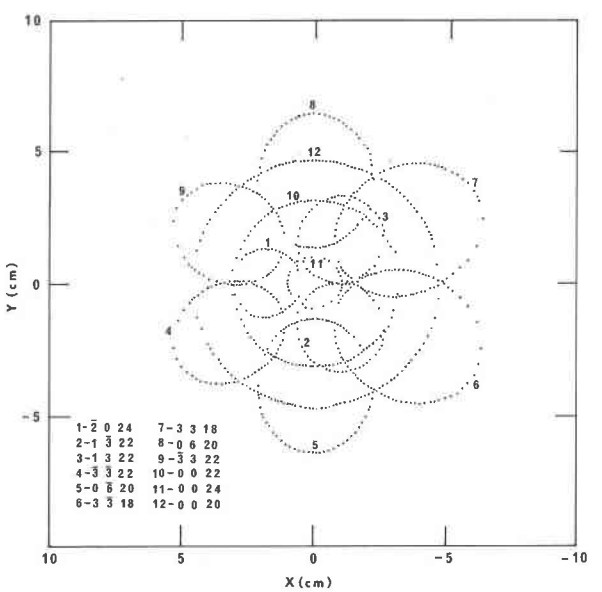

Fig. 2. Calculated pseudo-Kossel curves for talc. Crystal–film = 63 mm. Crystal–target = 5 mm. Radiation = Cu*K*.

Table 1. Measured d spacings in a talc crystal heated to 700°C (Cu$K\alpha$ radiation)

Plane	hkℓ	Intensity	θ	$d_{hk\ell}$ Å	Interplanar Angle (hkℓ) (00 22)
1	$\bar{2}024$	very strong	81.4±0.2	0.7789±0.0005	17.0±0.4
2	$1\bar{3}22$	strong	76.3±0.1	0.7927±0.0004	17.0±0.5
3	1322	strong	76.2±0.2	0.7933±0.0008	17.3±0.7
4	$\bar{3}322$	medium	78.3±0.2	0.7866±0.0008	31.5±0.4
5	$\bar{3}322$	medium	78.9±0.2	0.7850±0.0007	30.7±0.5
6	$0\bar{6}20$	medium	75.8±0.2	0.7946±0.0008	31.5±0.5
7	$3\bar{3}18$	medium	71.4±0.2	0.8126±0.0010	31.3±0.5
8	3318	medium	71.9±0.2	0.8103±0.0010	31.8±0.4
9	0620	medium	74.0±0.2	0.8013±0.0008	31.7±0.5
10	0022	weak	64.1±0.3	0.8561±0.0014	0

consistently below 90° while the β value is closer to the value of Raynor and Brown (1973) than to that of other investigators.

Heat treating of the crystal at 500° and 600°C results in a volume increase, presumably to allow the water to escape. This volume increase is mainly the expansion of the a parameter. Further heating to 700°C results in a collapse of the cell to about its original size.

The values are surprisingly coherent, especially as allowance has to be made for the following factors. (1) Talc is a very malleable material, and any distortion or bending of the surface of the crystal will result in inaccuracies. The crystal must be set parallel to the film. Better results would be expected with harder minerals.

(2) No correction has been made for physical factors such as absorption. The Bragg angles (Table 1) are above 70° with the exception of the 0 0 22, thus reducing the error.

(3) In comparing these data with those of others, it should be noted that their talcs had different chemical compositions and hence different lattice parameters.

Conclusions

The back-reflection pseudo-Kossel X-ray technique employing a direction cosine method can be successfully used for measuring the lattice parameters of large single crystals.

The first stage of dehydration of talc, up to 600°C, results in a volume increase of about 1 percent, mainly caused by expansion in the x-direction. There is a corresponding decrease on further heating to 700°C.

Table 2. Lattice parameters of talc before and after heat treatment

	a (Å)	b (A)	c (A)	α	β	γ	c sinβ (A)	V (A³)
T6 Room Temperature	5.241±0.017	9.070±0.033	18.926±0.014	89.75±0.08	98.57±0.08	90.5±0.3	18.72±0.01	890±4
500°C	5.253±0.009	9.109±0.019	18.941±0.006	89.74±0.05	98.39±0.04	90.1±0.1	18.74±0.01	897±3
600°C	5.319±0.019	9.036±0.038	18.929±0.012	89.86±0.09	98.32±0.08	90.4±0.2	18.73±0.01	900±5
700°C	5.279±0.027	9.000±0.056	18.932±0.019	89.92±0.13	98.36±0.11	90.7±0.3	18.73±0.02	890±7
T3 Room Temperature	5.243±0.024	9.069±0.033	18.932±0.013	89.87±0.09	98.78±0.30	89.4±0.3	18.67±0.01	888±5
T8 Room Temperature	5.261±0.025	9.170±0.044	18.957±0.016	89.97±0.13	98.17±0.10	89.5±0.3	18.76±0.02	897±6
Mean Value T6/T3/T8	5.248±0.022	9.103±0.037	18.923±0.014	89.96±0.10	98.51±0.16	89.8±0.3	18.71±0.02	894±5
Gruner (1934)	5.27	9.12	18.85	90	100° 5'	90	18.56	892
Hendricks (1938)	5.28±0.02	9.12±0.03	18.92±0.05	90	100°15'±15'	90	18.62±0.05	897±5
JCPDS (1969)	5.28_7	9.15_8	18.95_6	90	99.5	90	18.69_6	905±2
Raynor & Brown (1973)	5.29_3	9.179_3	18.99_2	90.57_3	98.91_3	90.03_5	18.76_2	911.5_9
Lindemann et al (1975)	5.32_5	9.17_9	18.92_6	82.3_2	94.0_3	90.5_3	18.97_9	914

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