Non destructive observation by X-ray diffraction on a berlinite crystal

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Abstract

For the characterisation of a crystal, several non destructive observations can be made by X-Ray diffraction. The crystal may be studied either as a block as grown from the autoclave or as a cut or a finished resonator. Once the crystal is placed on a special piezogoniometer its orientation may be measured and its orientation matrix calculated. Then, the most useful lattices planes are chosen, some of them allow the determination of the lattice parameters, others the detection of defects as twins or a mosaic structure. Illustrations are made on a berlinite crystal showing some macroscopic defects.

1. INTRODUCTION

The piezogoniometer we have constructed was first used for the orientation determination of quartz cuts. We have presented several methods (1 - 5) giving the orientation of simply, doubly or triply rotated cuts. In this paper we propose a new method permitting the determination of the lattice parameters of the sample when it is placed on this piezogoniometer. This method is useful even if the shape of the sample is very unusual compared to a classical slab. Parameters determination have been made with rods, faceted stones as those used in jewellery or crystals as grown from autoclave or obtained by Verneuil methods. The crystals we have already observed are quartz, garnets, diamonds, rubies, sapphires and some others mineral species. The results presented here are those obtained with a as grown berlinite crystal.

2. EXPERIMENTATION

2.1 Introduction of a new sample holder.

When the sample is a blank, it is fixed by vacuum suction on a plane perpendicular to the Φ axis of the diffractometer. This plane is the front side of a metallic disk turning around the Φ axis. The principle of the piezogoniometer is presented below, with the two very important angles ω and 2θ . The position on the Φ axis is defined by ω , and the position of the X-rays detector by 2θ .

According to the thickness of the sample this disk may be translated in such a way that the external side of the crystal is exactly on the central axis of the piezogoniometer.

When the volume of the crystal is limited by any surfaces we need a very special fixation device. The simplest way is to use a paste called "rodico paste" used in jewellery for cleaning stones. This paste is malleable under important stress but its strain is negligible if the stress is weak. A great number of experiences have shown that the rodico paste is rigid enough during all the observations made on the piezogoniometer. The crystal with its uneven shape is glued on a glass plate.

The new sample holder :

The free side of the glass is then normally fixed by suction on the rotating disk. The position on the crystal on the rodico paste is very important. We must be to be sure that the external face of the crystal is parallel to the free side of the glass plate. A good parallelism avoids defects produced by the differential absorption of the X-rays inside the crystal and it permits a very easy translation of the sample on the rotating disk. Then the thickness of the sample on its sample holder is precisely measured. This value commands the exact translation of the rotation disk in such a way that the external face of the crystal is on the central axis of the piezogoniometer. The parallelism is optically checked before the X-rays observations.

2. 2 Principle of the accurate lattice parameter determination

An accurate lattice parameter determination is obtained if the value of the Bragg angle is very high. But generally during an orientation determination the values of Bragg angles we use are too small for a reliable result. So we must introduce new lattice planes and we must be able to observe them. That means that we must know the approximate values of the angular positions ω and 2θ on the piezogoniometer permitting the observation of these special lattice planes.

At the very beginning the orientation knowledge of the crystal is not required, we only need a rough value of the lattice parameters. The first measurements concern the determination of the crystal orientation using a trial and error method. When the orientation of the crystal is known we are able to determine the angular positions ω and 2θ permitting the observation of the required lattice planes. We may note that for the orientation determination an accuracy of about one arc degree is enough. When the Φ axis and the X-rays detector are on the expected positions ω and 2θ we obtain the maximum of the intensity of the reflected X-Rays beam using this operating process.

First, the best position of the ω angle is obtained during the fast rotation motion around the Φ axis.

Second, this rotation is stopped, and manually the best position of the ϕ angle is obtained.

Third, the best position of the X-rays detector is obtained. Some fine adjustments of these angular positions may be required because we need the highest value of the reflected beam. The position of the X-rays detector is calculated considering the half-width of the peak. From several observed lattice planes the crystal parameters are determined.

2. 3 Preliminary results

The piezgoniometer and that proposed method have been verified during the measurement of the lattice parameters of a diamond crystal. With diamond, a cubic crystal, the highest value of the Bragg angle is obtained with the plane {422} and the copper Kbeta radiation. Results are presented below :

λ K beta	hkl	measured angle	calculated value
	400	51.346 °	3.5654 Å
	422	72.963 °	3.5666 Å

expected value (JCPDS 6-675) 3.5667 Å

If we compare our average value 3.5660 Å to those 3.5667 Å given by the JCPDS records we may be confident that the method gives reliable results.

Note : the calculated value with the highest Bragg angle is very close to the actual one.

3 BERLINITE CRYSTAL OBSERVATION

The berlinite crystal we have studied is an as grown crystal. All the faces have an uneven surface. The X faces seem to be flat , but they have locally roughness presenting some hillocks produced by a disordered growth. It is not possible to observe this crystal without the use of the rodico paste. One of the X face seems to be divided by a line whose the presence is unexpected. We will try to understand its behaviour. We have studied this X face and one of the Z face.

3. 1 Observation of the Z face

The determination of the c parameter is very easy using planes of the families {00.l} . With these lattice planes each measure gives a value of the c parameter. Results are presented below:

Z face c determination	hkl	measured angle	lambda	c calculated value
	00.6	22.4 °	K beta	10.959 Å
	00.9	34.975 °	K beta	10.948 Å
	00.12	49.761 °	K beta	10.9429 Å
	00.12	75.595 °	K alpha1	10.9439 Å

The expected value is 10.94761 Å . It is important to note that with a Bragg angle of low value the result is not so good.

3. 2 Observation of the X face

The determination of the value of the two parameters a and c is made on this face. Among all the lattice planes we have tried the most convenient are those of the {32.2} and {50.4} families. These two families are interesting because the {32.2} Kalpha1 Bragg angle and the {50.4} Kalpha2 Bragg angle are quite identical. This avoids unnecessary rotations of the X-Rays detector during the observation. The parameter determination is made on two points A and B. These two points are on each side of the line we have indicated. Results are presented below :

X face a and c		hkl	measured angle	lambda	calculated values
	Point A	33.2	71.014°	K alpha1	a 4.9425 Å
		50.4	70.935°	K alpha2	c 10.939 Å
	Point B	33.2	71.040°	K alpha1	a 4.9415 Å
		50.4	70.940°	K alpha2	c 10.957 Å

The observed values are not far from the expected values for a berlinite crystal, respectively 4.94291 Å and 10.94761 Å. We conclude that the two parts of the crystal on each side of this line are similar.

We have made a great number of observations of the orientation of the crystal on both sides of this line. Observations have been made successively on four points A, B, C and D. The points C and D are also on each side of that line. The positions of the diffraction peaks remain exactly the same. That means that the line does not modify the lattice of the crystal.

Sometimes with quartz crystals there are some twins on the blanks we observe. Using the same kind of investigation we have observed lattice planes of the {32.2} and {23.3} families. These planes have the same Bragg angle but they differ in their scattering factor. Again we have seen no differences between the four positions. This means that we do not have twins similar to the twins we observe with quartz crystals. The line dividing the X face is, according to the observations we have made, of no importance.

4 CONCLUSION

The use of a special soft paste permits the observation of crystals or crystalline objects with unusual shape. It is possible to determine directly their lattice parameters. The observation of a berlinite crystal as grown from the autoclave has been made by a non destructive method.

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