Proceedings of the 2003 IEEE International Frequency Control Symposium and PDA Exhibition
Jointly with the 17th European Frequency and Time Forum

THE X-RAY ANGLE MEASUREMENT OF DOUBLY ROTATED QUARTZ BLANKS WITH ANY CUTTING ANGLE USING THE Ω-SCAN METHOD

H.-A. Bradaczek, H. Berger, H. Bradaczek, G. Hildebrandt
EFG International, Research Center, Berlin, Germany

Abstract **– The application range of the Ω-Scan Method and the existing SC machine can be essentially extended by reducing the restrictions given by the measuring arrangement and the precision demands. For the not included orientations, other reflection combinations and geometries have to be used. For the determination of an arbitrary unknown orientation, the incidence angle of the X-ray beam must be varied over a larger range, until at least three reflection pairs of preselected types of reflections have been found. In order to enable such measurements, a more or less universal apparatus has to be used, working in a partially or completely automated mode. The method and the apparatus can be applied to the orientation determination and to the adjustment for the subsequent cutting of arbitrary single crystals.**

Keywords **– Cutting-angle determination, quartz, Ω-Scan Method, extension of the application range**

I. Introduction

For many years the X-ray Ω-Scan Method has been applied to the cutting-angle determination of AT-cut [1] and of doubly rotated quartz blanks [2]. The method is used for the angle sorting of blanks of arbitrary shape as well as for quartz wafers [3]. Up to now, these machines are designed for limited cutting-angle ranges, for AT-cuts on the one hand, and for SC-cut and FC-cut samples on the other hand (Angle Sorting Machines produced by EFG International Berlin). With some limitations, the SC machine can also be applied to IT-cut quartz. The application range has been roughly estimated in an earlier contribution [4]. However, in the past there has been a number of demands to extend this range, at least in the order of some tenths of a degree. In the last years, also other piezoelectric materials became more important. Therefore, it has been studied more generally, which modifications are necessary to measure the cutting angles of quartz and, besides that, of arbitrary crystalline materials in a wide range.

Firstly, it will be discussed on which conditions the application range of a given Ω-Scan arrangement can be extended. In a further point the possibilities to determine the completely unknown orientation of a single crystal will be described. Because of the importance of quartz the examples to be discussed are presented for this material.

II. Application range of the Ω-Scan Method

In the existing sorting machines based on the Ω-Scan Method two selected reflection pairs are measured and evaluated (Fig. 1). The incidence-angle range of the X-ray beam can be changed by about 3.5°. By means of simulation calculations, it has been found out, for which cutting angles the reflection pairs can be registered and evaluated. Reducing step-wise the geometrical restrictions of the given machine, also the application range can be extended.

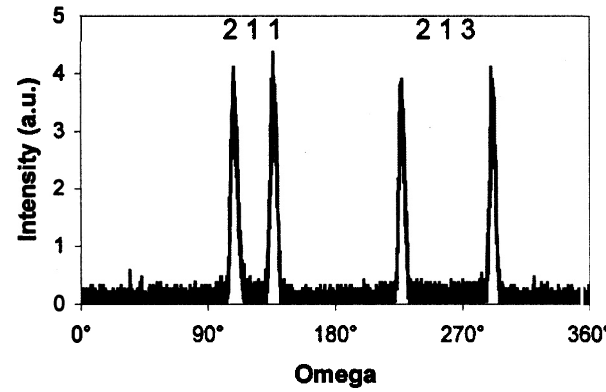

Fig. 1. Measuring diagram of SC-cut quartz. Reflections 211/213, CuKα radiation.

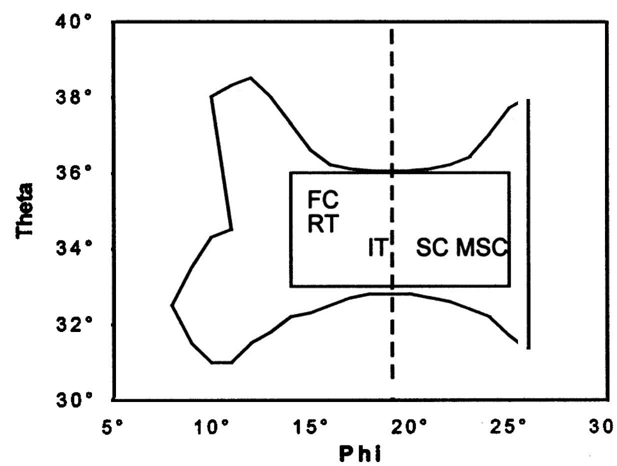

Fig. 2. Cutting-angle range of quartz measurable with the existing SC machine.
Cartesian plot of the angular coordinates Θ and Φ, reflections 2 1 1 / 2 1 3, CuKα radiation, incidence-angle range 3.5°, with some important cuts.
Inserted rectangle: cutting-angle range according to [4].
Broken line: boundary line for not distinguishable Φ values (at Φ=19.1°; explanation see text).

For the reflections 2 1 1 / 2 1 3 used in the SC machine, the corresponding application range is shown in Fig. 2 as a Cartesian plot of the angular coordinates Θ and Φ. The range estimated earlier [4] as well as the relevant quartz cuts are included. Also the RT-cut, which has a negative Θ value, is shown because it can be measured under the same geometric conditions using lattice-geometrically (not structurally) identical reflections. Such reflections have other intensities. This allows also to distinguish, e.g., SC- and RT-cuts by means of X-ray measurements. The more exactly determined application range is essentially larger as assumed up to now. Additional disturbing reflections must be avoided by suited slits in front of the detector. This is difficult for (lattice-) symmetrically equivalent reflections. Because these reflection pair tend to superimpose each other. the corresponding orientation ranges should be omitted in order to avoid complications. Therefore, a strip parallel to the line Φ=30° is excluded.

In general, the orientation measured by means of two X-ray reflections is ambiguous. This is relevant only as far as the given cut is near the corresponding boundary line in the Θ-Φ plane. For the reflections 2 1 1 / 2 1 3, this boundary is a line at Φ_0=19.1° (cf. Fig. 2). That means, Φ angles on the right and on the left side of this line cannot be distinguished.

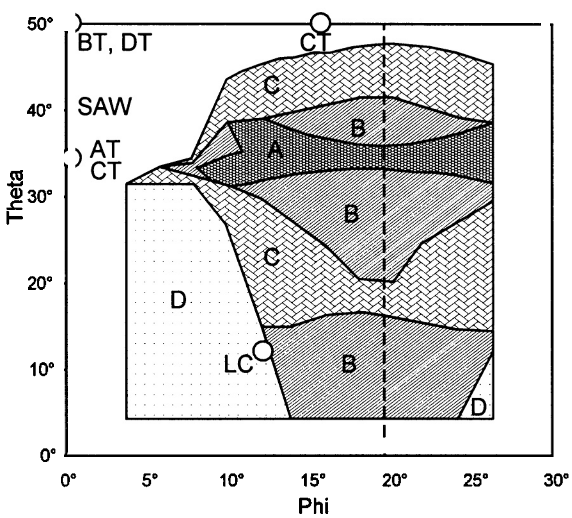

Fig. 3. Cutting-angle range of quartz measurable with reflections 2 1 1 / 2 1 3, CuKα radiation, for different restrictions. Cartesian plot of the angular coordinates Θ and Φ, with some important cuts (for area A: see Fig. 2).
Broken line: boundary line for not distinguishable Φ values (at Φ=19.1°; see Fig. 2 and text).
A: cutting-angle range corresponding to Fig. 1 (3% of all possible orientations measurable);
B: cutting-angle as A, additionally unrestricted incidence-angle range (20% of all possible orientations measurable);
C: cutting-angle as B, additionally superposition of both reflection pairs permitted (33% of all possible orientations measurable);
D: cutting-angle range without restrictions (46% of all possible orientations measurable).

This is true for IT-cut blanks. For single blanks, i.e., for semi-automatic measurements, an unambiguous determination of the Φ value is possible if the true principal surface orientation is known. However, the problem can generally be overcome by means of measuring and evaluating a *third* reflection pair, e.g., the 312 reflection pair, excited by CuKβ radiation. Then also the automatic measuring and sorting of IT-cut blanks is possible.

In order to measure cutting angles in much larger ranges, among other things the measuring apparatus must be more universal. In Fig. 3 the resulting measurable ranges are shown for various more or less extensive restrictions. The measurable part of orientations can be estimated as follows: Due to the lattice symmetry of quartz, a range of Θ from 0° to 90° and of Φ from 0° to 30° represents all possible cutting angles which can be distinguished by X-ray measurements. So the measurable areas as shown in Fig. 3 are to be related to this Θ-Φ area, both converted into their areas on a sphere surface. With the existing SC machine and using the same reflections, about 3% of all possible orientations of quartz can be measured. The measurable part yields 20% if it is allowed the X-ray tube and detector to be set in a arbitrary angular range. If also cases are included where both reflection pairs are superimposed, the useful range is enlarged to 33%. In this case, two separate detectors have to be used. The total range measurable with the same reflection combination is about 46%. Then cases are included where the standard deviation of the angular coordinates may be rather large. This situation is not optimal if utilized in a sorting machine. However, it can be used for the occasional orientation determination of a sample in a universal X-ray machine.

From the same reasons as discussed above, the Y-Z, X-Z and X-Y planes and neighboring strips of some degrees width and, hence, the corresponding singly rotated cuts like AT-cut, must be excluded from the application range of the above reflections. For these orientations, reflections on lattice planes whose normals lie in the Y-Z, X-Z or X-Y planes, respectively, are to be applied. These reflection pairs occur only once. In the AT-cut angle-sorting machines, the reflection combinations 2 0 2 / 2 0 3 and 1 0 1 / 2 0 1, respectively, are used. For SAW wafers, which are Y-cuts with, e.g., Θ=70°, the reflection combination 3 0 1 / 3 0 2 could be applied.

For cutting-angle ranges other than those discussed before, other reflection combinations have to be found. Altogether, a limited number of such combinations is sufficient to enable the precision cutting-angle determination of quartz samples of any orientation by means of the Ω-Scan Method. Eventually, the measuring conditions must be optimized using simulation calculations. In particular, it has to be checked that there are not any unwanted additional reflections. If necessary, special detector arrangements, masks and so on must be used.

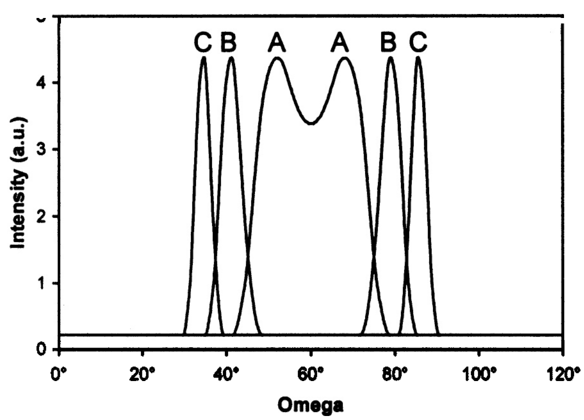

Fig. 4. Diagrams of the reflection 4 0 1 for various incidence angles. Quartz, CuKα radiation (calculated).
A: incidence angle 67.8°; B: incidence angle 67.3°;
C: incidence angle 66.8°;

III. Determination of unknown orientations

Also in the case of unknown orientations the Ω-Scan Method has special potentialities. However, the procedure has to be modified. Firstly, reflection types have to be selected so that for any orientation at least the then necessary three reflection pairs can be registered. Because of the appearance of symmetrically equivalent reflections, two types of such reflections are sufficient for quartz. Possible reflection types can be, e.g., 2 2 4 and 4 0 1. The detector (or the two detectors) must be set under the corresponding angle to the incident beam. In order to find the necessary number of reflection pairs, the angle of the incident X-ray beam and of the detector(s) have to be changed step-wise or continuously. This corresponds to the scanning of the sphere of possible lattice directions along concentric circles or a spiral line. A reflection occurs in a certain incidence-angle range. The peaks of the reflection pair appear with varying peak distances, depending on the incidence angles. An example is shown in Fig. 4. The peak distances can be evaluated at different incidence angles, what allows to select the optimum conditions and to derive additional information. If the principal orientation has been found, the precision determination using optimum reflection combinations can be applied if necessary.

In the case of a fully unknown orientation, an incidence-angle range of 50° or more may be necessary to be scanned requiring a measuring time of some minutes. Also the determination of an unknown orientation can be performed automatically.

IV. Variants of measuring arrangements

The X-ray measuring devices must be equipped according to the concrete demands. In any case, the turntable which determines the precision of the Ω-Scan measurement must be very precise and stable. A universal apparatus

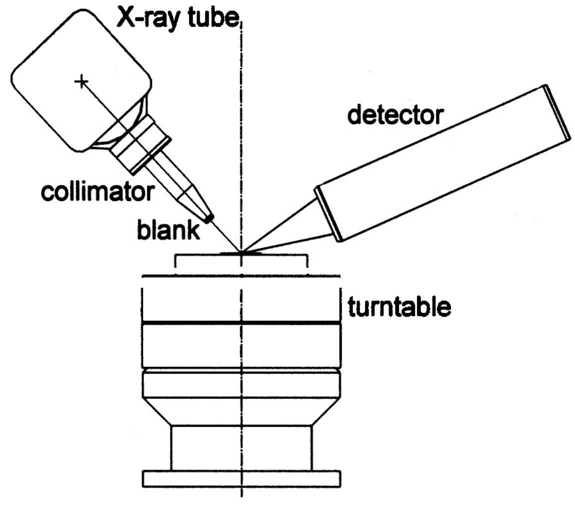

Fig. 5. Scheme of a Ω-Scan measuring arrangement, suited for small samples (blanks).

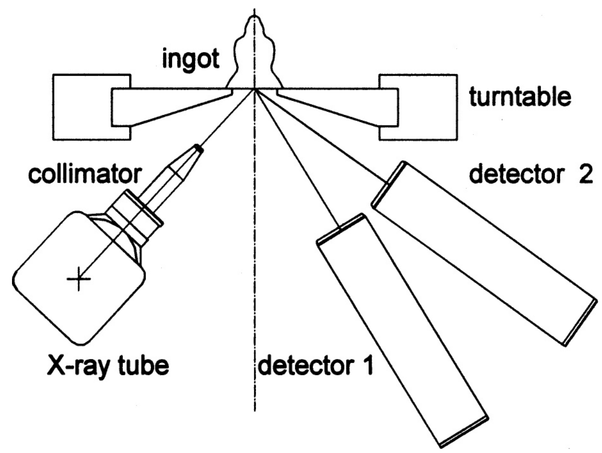

Fig. 6. Scheme of a Ω-Scan measuring arrangement equipped with two detectors, suited for big samples and their adjustment.

for arbitrary cutting-angle measurements must have adjustable circles for the incident beam (X-ray tube) as well as for the detector(s). For both, the angular range must be 90° or more. If the apparatus is provided for special applications, these ranges can be limited. The distance from the measuring point to the detector and the slits in front of the detector(s) should be adjustable, too. All these adjustments can be made manually or automatically. A scheme of the apparatus, especially suited for blanks or wafers is shown in Fig. 5. For big samples, as "stones" or ingots, an arrangement according to that shown in Fig. 6 is to be preferred. It can also be applied to the angle determination for the subsequent cutting. Accordingly, the software is to be chosen to fulfil the various demands of the

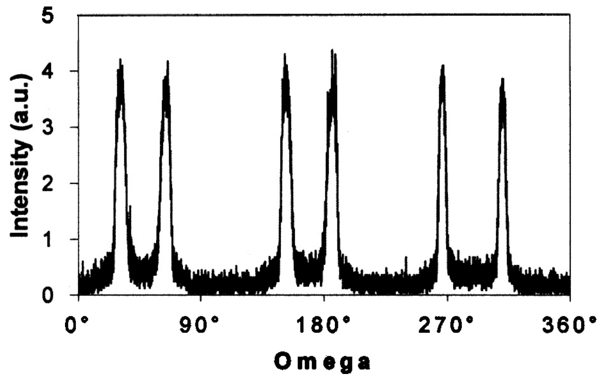

Fig. 7. Measuring diagram of Sapphire. (001) orientation, reflections of the type 1 0 10, CuKα radiation.

hardware and that of the measuring and evaluation procedures.

V. Conclusions

The methods and apparatus as described above can be principally applied to other single-crystalline materials. Because of the very similar lattice geometry of other piezoelectric materials, as crystals of the langasite group and of $GaPO_4$ to quartz, the measuring conditions can be applied immediately or with small modifications to these substances. However, the calculations and estimations are valid also to a large extent to arbitrary materials. In Fig. 7, an example of an Ω-Scan measurement of sapphire is shown, an important material applied, e.g., as substrate for semiconductor films. This is also an example for a surface orientation of high, i.e., threefold symmetry, offering three reflection pairs to be evaluated.

One can state that the X-ray Ω-Scan Method, applied up to now to a relatively small section of the total range of possible orientations of quartz can be used much more generally. According to the concrete demands, the apparatus can be chosen as a universal one or it can be more or less specified to particular purposes. In all cases, the degree of automation can be chosen.

References

[1] B. Morys, H. Bradaczek, and G. Hildebrandt, "Improved Ω-Scan for separate measurement of true AT-cutting angles and X-miscutting angles for round quartz blanks," Proc. IEEE Int. Freq. Contr. Symp., Boston, pp. 237-240, 1994.

[2] H. Berger, H. Bradaczek, H.-A. Bradaczek, and G. Hildebrandt, "Application of the Ω-Scan to the sorting of doubly rotated quartz blanks," Proc. IEEE Int. Freq. Contr. Symp., Honolulu, Hawaii, pp. 412-415, 1996.

[3] H. Berger, H.-A. Bradaczek, H. Bradaczek, and G. Hildebrandt, "X-ray angle sorting of small-size AT-cut quartz blanks using the Ω-Scan – state and perspective", Proc. IEEE Int. Freq. Contr. Symp., Tampa, Florida, this volume, 2003.

[4] H. Berger, H.-A. Bradaczek, H. Bradaczek, and G. Hildebrandt, "Further progress in the absolute orientation determination of doubly rotated quartz blanks by means of the Ω-Scan Method", Proc. IEEE Int. Freq. Contr. Symp., Seattle, Wash., pp. 393-395, 2001.