

# X-ray orientation determination of single crystals by means of the $\Omega$ -Scan Method

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**Abstract.** The X-ray diffractometric  $\Omega$ -Scan Method uses a skew reflection geometry. Only one measuring circle is necessary to measure the reflection pairs generated at two or more lattice planes. From the angular positions of the reflections, the precise orientation within a certain range can be determined. The method can be applied to the angle sorting of slices or wafers as well as to the adjusting of crystal ingots for the subsequent cutting. For the determination of the completely unknown orientation, the incidence angle of the X-ray beam must be varied over a larger range, until at least three reflection pairs have been found. The  $\Omega$ -Scan diffractometers can be constructed according to the different demands. They can work fully automatically and can be equipped to allow further diffractometric measurements.

## 1. INTRODUCTION

The orientation determination of single crystals is a basic application of the X-ray diffraction. The orientation determination is necessary in connection with the crystal growth, the oriented cutting of crystal ingots and the technological application of single crystals. Examples are the production of semiconductor materials as well as of corresponding substrate crystals, like sapphire and SiC. An important application field is the fabrication of quartz oscillators demanding a particularly high precision of the cutting angle of the oscillator crystals [1].

There exists a large number of special methods and procedures specified to the various materials, crystal sizes, orientation ranges, precision, grade of automation, and succeeding utilisation of the measuring results. The Laue method and the X-ray diffractometry are mostly applied.

The Laue method is especially suited as a simple laboratory method to give a rough overview of the principal orientation of a single crystal. However, the automatic registration and evaluation of the measured reflections and the exact orientation determination are rather difficult and demand sophisticated techniques [2-4].

The diffractometric method has the advantage that more precise results can be reached without especial efforts and that further measurements can be performed subsequently using the same device. The determination of the complete orientation needs to measure at least two different reflections each demanding special adjustments.

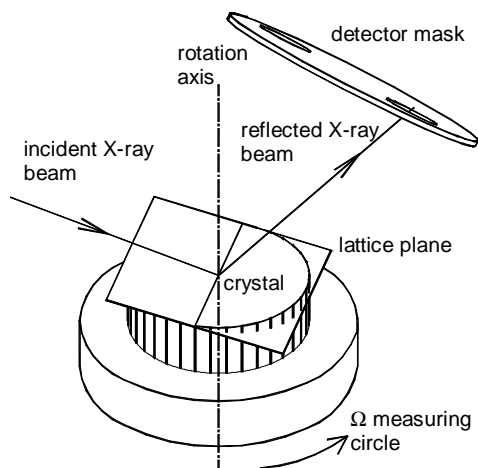
In this contribution, a special diffractometric method, the so-called  $\Omega$ -Scan, is presented. This method allows to measure all the necessary reflections during one rotation of the measuring circle. So it can be automated in a simple manner. This method has been applied for many years especially for the measurement of the cutting angles of quartz oscillators [5, 6].

In the following, the principle of the  $\Omega$ -Scan Method will be explained. The application to the precision determination of the orientation of single crystal slices and wafers and of the adjustment angles for

the subsequent oriented cutting of crystal ingots will be discussed. The procedure to determine a completely unknown orientation will be shortly described.

## 2. PRINCIPLE OF THE $\Omega$ -SCAN METHOD

During the  $\Omega$ -Scan measurement the sample is continuously rotated by means of a precise turntable (Fig. 1). The geometry of this method corresponds to some extent to that of the classical rotating-crystal method.



**Figure 1.** Principle of the  $\Omega$ -Scan measuring arrangement.

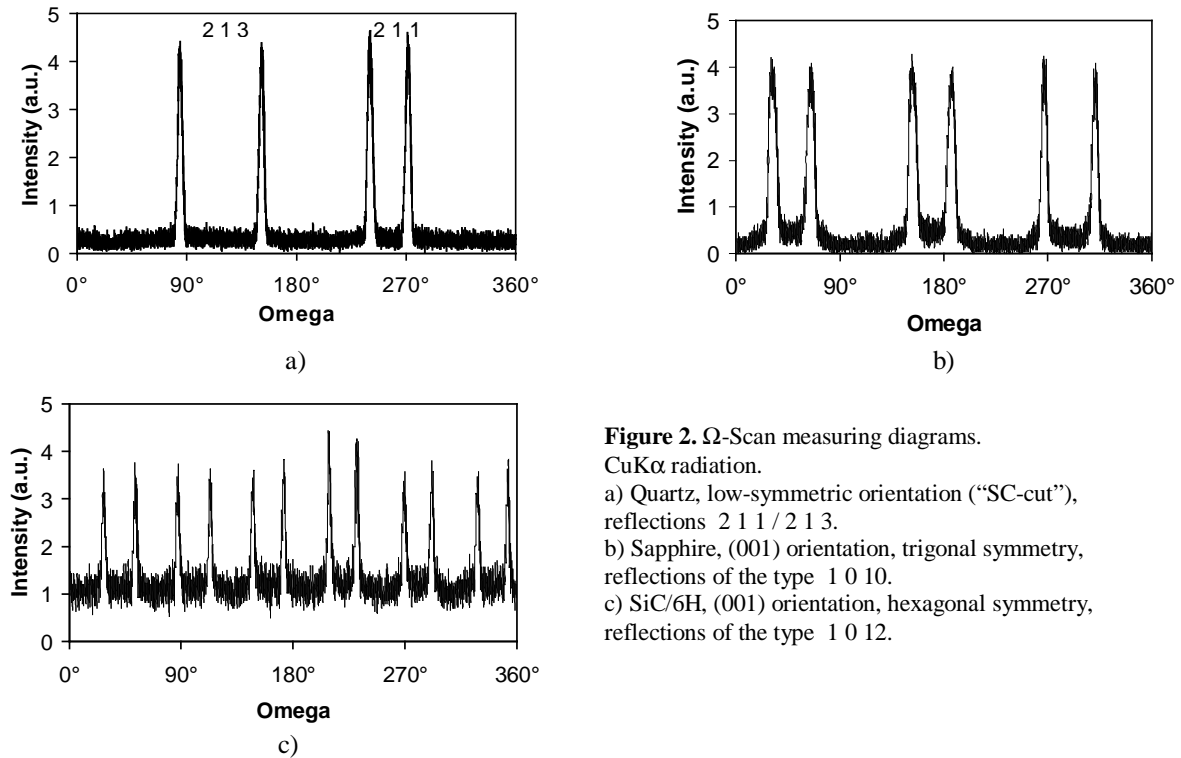
However, the rotation axis is inclined to the primary beam. The angular positions of the reflections measured in the plane perpendicular to the rotation axis, the  $\Omega$  circle, are registered, e.g., by a scintillation counter. According to the symmetry of the rotating-crystal method, the X-ray beam is successively twice reflected by each lattice plane. The geometry is not coplanar, i.e., the planes containing the incident and the diffracted beams are inclined to the rotation axis. The incidence angle of the primary beam is chosen accordingly and slits in front of the detector are positioned so that reflections at a sufficient number of lattice planes are obtained and can be evaluated unambiguously. Usually, the reflections at two lattice planes are used (Fig. 2a). Measuring crystals and orientations with high symmetry of the rotation axis, a larger number of reflections is registered (Fig. 2b and c).

From the angular positions of the reflections (at least four) the orientation of the rotation axis with respect to the crystal lattice, described in the given examples by means of two polar coordinates  $\rho$  and  $\varphi$ , can be calculated ( $\rho$ : polar distance,  $\varphi$ : azimuth; pole: c axis,  $\varphi=0$ : zone [010]). In most cases also the incidence angle can be calculated additionally. If the orientation of an even crystal surface is to be measured, the surface is set to be perpendicular to the rotation axis. Any deviation of the surface normal from this axis can be corrected by the additional evaluation of a laser signal reflected at the surface [7]. The direction of any edge or flat can also be measured by means of auxiliary optical tools.

Presumed the turntable is sufficiently precise, the reproducibility of the measured reflection positions depends mainly on the pulse statistics. Using strong reflections and a measuring time of two seconds (corresponding, e.g., to one rotation of the turntable), the angular positions of the reflections on the  $\Omega$  circle can be evaluated with a precision of about  $0.01^\circ$ . Depending on the lattice geometry, this corresponds to standard deviations in the orientation coordinates of smaller than one arcminute, in favourable cases of a few arcseconds.

The main systematic errors are due to the problem to find out the "true" peak position which would deliver the true orientation coordinates. Because the reflection curve widths may amount some degrees, these errors can reach some tenths of a degree in the reflection positions and up to one arcminute in the

orientation coordinates. The procedure to correct these errors consists in the exact simulation of the reflection curves and their evaluation in the same manner as the measured ones [8]. Besides, the not exact knowledge of the lattice parameters can also lead to a systematic error [8]. However, this error should usually be negligible.



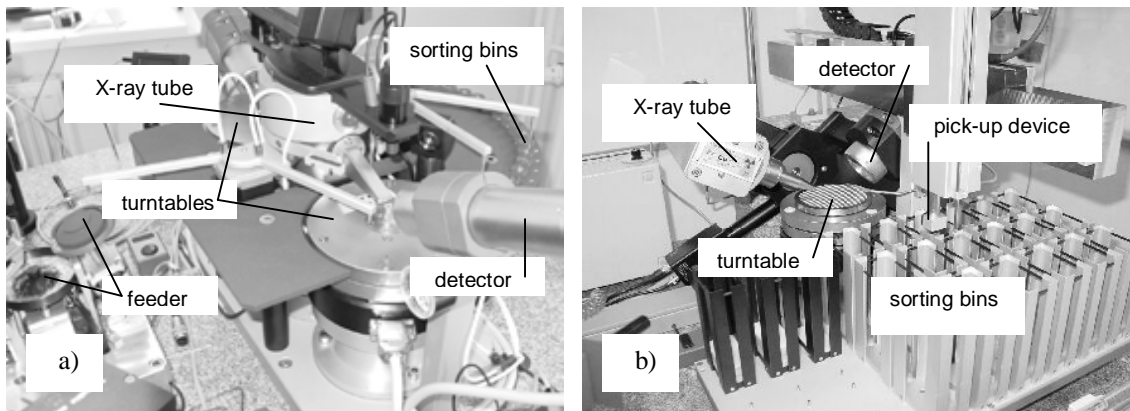
**Figure 2.**  $\Omega$ -Scan measuring diagrams.  
CuK $\alpha$  radiation.

a) Quartz, low-symmetric orientation ("SC-cut"), reflections 2 1 1 / 2 1 3.  
b) Sapphire, (001) orientation, trigonal symmetry, reflections of the type 1 0 10.  
c) SiC/6H, (001) orientation, hexagonal symmetry, reflections of the type 1 0 12.

### 3. ORIENTATION DETERMINATION IN A CERTAIN ORIENTATION RANGE

In most cases the rough orientation of the specimen is known. Then the reflection combination and the incidence angle (or angular range) can be chosen before in order to find optimal conditions concerning precision and measuring time for the  $\Omega$ -Scan. The problem is more complicated if the possible range of the orientation coordinates is widely extended. In favourable cases, samples whose orientation is unknown in a range up to a few degrees can be measured using a fixed arrangement of X-ray tube, turntable and detector. An example is an  $\Omega$ -Scan arrangement for the measuring of quartz slices with AT orientation ( $\rho \cong 55^\circ$ ,  $\varphi = 0^\circ$ ; Fig. 3a). In other measuring arrangements for AT-cut (Fig. 3b) as well as for SC-cut quartz ( $\rho \cong 56^\circ$ ,  $\varphi \cong 22^\circ$ ) the angle of X-ray tube and detector relative to the turntable must be changed about few degrees in order to secure the optimal precision for the given charge, presumed the orientation changes within the charge are sufficiently small.

One reason for the limitation of the application range of a given  $\Omega$ -Scan arrangement is that the single reflections may superimpose. The problem can be overcome using two (or more) single detectors or a position-sensitive area detector. If not symmetrically equivalent reflections are used, they may be separated by means of detectors set to the different reflection angles. Otherwise, the reflections produced in the two



**Figure 3.** Views of  $\Omega$ -Scan measuring arrangements combined with angle-sorting devices for quartz (“AT-cut”).  
 a) For slices, two measuring devices on both sides of the vertical X-ray tube, fixed tube and detector settings.  
 b) For wafers, defined shift of the wafer after each measurement on the continuously rotating turntable.

positions of one and the same lattice plane can be separated using two detectors in the corresponding positions at the same reflection angle. In this way, the six reflection pairs appearing at nearly (001) oriented hexagonal crystals like 4H or 6H SiC (Fig. 2c) can be evaluated for orientation deviations up to at least  $10^\circ$ .

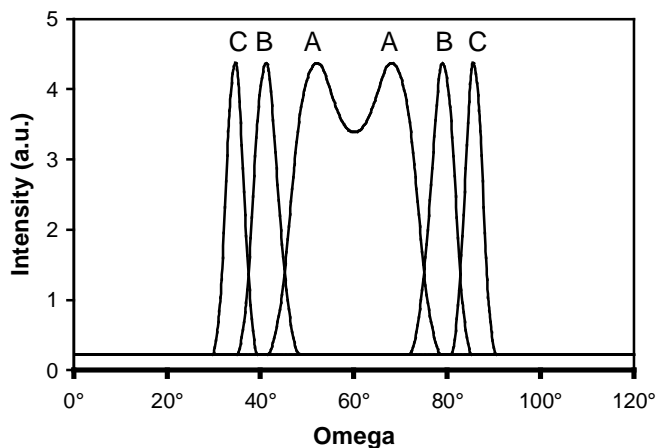
#### 4. DETERMINATION OF UNKNOWN ORIENTATIONS

Also in the case of the unknown arbitrary orientation of a crystal specimen the  $\Omega$ -Scan Method has special potentialities. However, the procedure has to be modified. Firstly, reflection types have to be selected in such a way that for any orientation at least three reflection pairs necessary for the unambiguous evaluation can be registered. Because of the appearance of symmetrically equivalent reflections, for cubic crystals one type and for trigonal, tetragonal and hexagonal crystals two types of such reflections are usually 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) coupled to it) has to be changed step-wise or continuously. This corresponds to the scanning of the sphere of possible lattice directions along concentric circles or along a spiral line. A reflection occurs in a certain incidence-angle range. The peaks of a given 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 completely unknown orientation, an incidence-angle range of  $50^\circ$  or more may be necessary to be scanned requiring a measuring time of some minutes. Also this whole procedure can be performed automatically.

#### 5. VARIANTS OF MEASURING ARRANGEMENTS

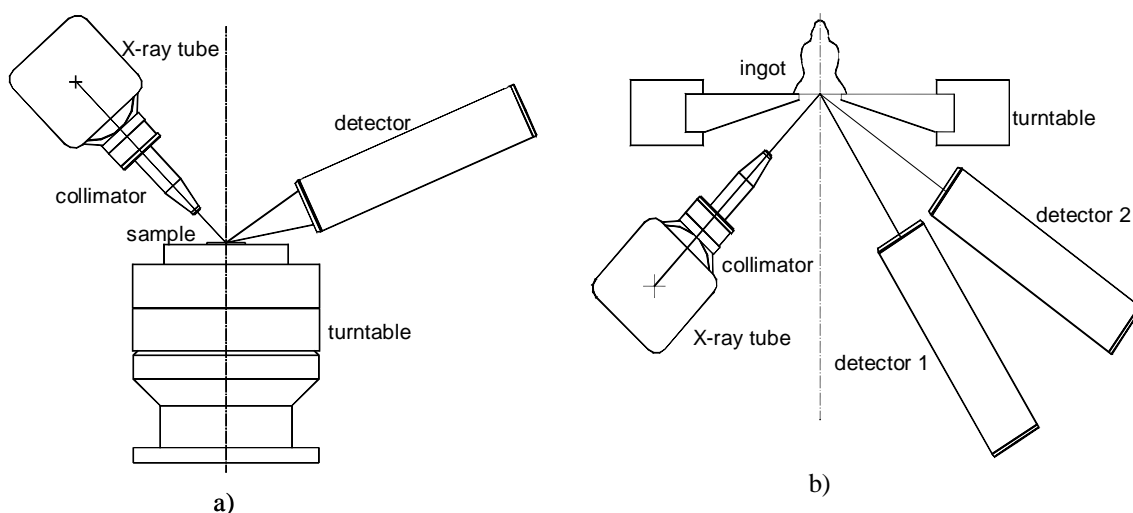
The X-ray measuring devices must be equipped according to the concrete demands. In any case, the turntable must be very precise and stable, in order not to limit the precision of the  $\Omega$ -Scan measurement. A universal apparatus for arbitrary cutting-angle measurements must have adjustable circles for the incident beam (X-ray tube) as well as for the detector(s). The angular ranges for both must be  $90^\circ$  or more. If the



**Figure 4.** Diagram of a reflection pair for various incidence angles (calculated). Quartz, CuK $\alpha$  radiation, reflection 4 0 1. A: incidence angle 67.8°; B: incidence angle 67.3°; C: incidence angle 66.8°.

apparatus is provided for a limited orientation range, these angles can be restricted. 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 an apparatus, especially suited for small slices or wafers, is shown in Fig. 5a. This arrangement can be combined with an angle-sorting device (Fig. 3). If the homogeneity of larger wafers is to be measured, it has to be equipped with corresponding tools for the parallel shift of the wafer. In the apparatus shown in Fig. 3b the sample is shifted after each single measurement using the calculated value of the azimuthal orientation and considering the constant time for lifting the wafer from the continuously rotating turntable. For high local resolution, an x-y translation table has to be installed on the turntable. For big samples, as crystal ingots, an arrangement according to that shown in Fig. 5b is to be preferred. It can



**Figure 5.** Schemes of  $\Omega$ -Scan measuring arrangements.

a) Suited for slices and wafers.

b) Suited for big samples and their adjustment (equipped with two detectors).

also be applied to the angle determination for subsequent cutting. Therefore, the rotation of the turntable must be stopped at an angular position calculated before. The ingot is adjusted in a special holder according to the measuring results, or it is immediately glued to a beam to be fixed to the cutting device. Therefore, the ingot can be adjusted on the turntable using inclination screws, or the gluing device is made adjustable. Adjusting on the turntable has the advantage that the actual orientation can be immediately checked.

## 6. CONCLUSIONS

Diffractometers based on the  $\Omega$ -Scan have been proved to be suitable for the precision orientation determination of a number of crystalline materials and concrete orientations. The diffractometers can be constructed and equipped according to the planned application. The apparatus can be chosen as a universal one or it can be specified to particular cases, so to the measurement of samples with more or less limited orientation ranges, for small slices, wafers or for bigger crystal ingots to be cut. The arrangements can be realised with different grade of automation including full automation in each case. Moreover, the diffractometers can be equipped with further X-ray optical elements like crystal collimators so that they can be additionally applied, e.g., to the precision lattice-parameter determination or to the rocking-curve measurement.

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