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Omega-Scan – an X-Ray Tool for the Characterization of Crystal Properties

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Abstract The X-ray diffractometric Ω-Scan method needs only one measuring circle to determine the lattice geometry of single crystals. From the angular positions of at least two reflection pairs the orientation in a certain range can be precisely determined. For cubic crystals the lattice parameter related to a reference crystal can be calculated additionally. The method is applied to the adjustment of crystal ingots for the succeeding processing and to the characterization of wafers. By means of an x-y table the distribution of the structural parameters over the wafer surface can be mapped with a lateral resolution of about 1 mm. In a $Si_{1-x}Ge_x$ sample, characteristic concentric distributions have been found with orientation changes up to 3 arc minutes and lattice-parameter differences up to 2.8×10^{-5} nm, corresponding to differences of the Ge content up to 0.14 at%.

1 Introduction

X-ray diffraction on single crystals (ingots or wafers) of semiconductors and substrate materials is used for the study of structural properties like lattice orientation, lattice parameters and crystal perfection. In the X-ray diffractometry, the information is averaged over the beam spot in the order of some tenths of a millimetre up to some millimetres. X-ray topography, often combined with a linear scanning mechanism, may image the whole surface of the sample. Using photographic or electronic imaging techniques, the lateral resolution can be as high as a few micrometers. Preferably, the X-ray topographs deliver qualitative or semi-quantitative information and detect dislocations and other single defects. Alternatively, an overview over the whole sample surface can be reached by X-ray diffractometry and two-dimensional scanning using an x-y table. The lateral resolution is then limited by the spot diameter, however, the full quantitative information can be obtained for each measured point.

The usual X-ray diffractometric measuring technique consists in the angular scanning of the sample relative to the incident X-ray beam and the detector. Incident and reflected beams as well as the normal of the reflecting lattice plane are nearly coplanar. In this contribution, an alternative diffractometric method, the so-called Ω-Scan, will be used [1]. It is based on non-coplanar diffraction geometry and has the advantage that all the reflections necessary for the evaluation can be measured during one rotation of the measuring circle. This allows exact measurements at short measuring times and the procedure can be automated in a simple manner. Therefore, this technique is especially suited for the mapping of surfaces. The Ω-Scan method has been applied for many years for the measurement of the cutting angles of quartz oscillators [1-3].

In the following, the principle of the Ω-Scan method will be explained. The application to the precise orientation determination of wafers and ingots, to the adjustment of ingots for the subsequent cutting and to the mapping of orientation and lattice-parameter distributions will be described.

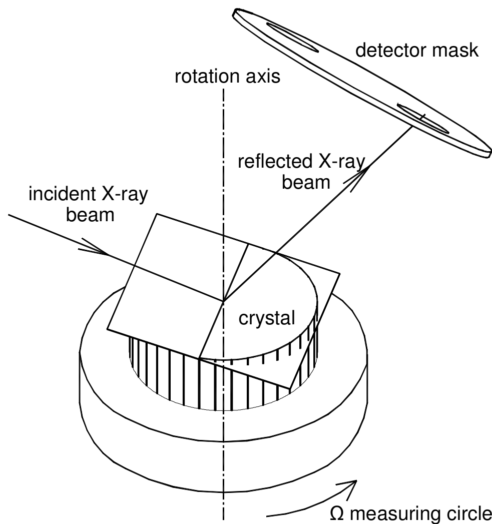


Fig. 1 Principle of the Ω-Scan measuring arrangement

2 Principle of the Ω-Scan method

During the Ω-Scan measurement the sample rotates with constant speed around an axis, the reference axis of the system. X-ray tube with beam collimator and detector with mask for the reflected beams are in fixed positions (Fig. 1). The geometry of this method corresponds to some extent to that of the classical rotating-crystal method. The X-ray beam is twice reflected from lattice planes inclined to the rotation axis. The angular positions of the reflections are measured in the plane perpendicular to the rotation axis (Ω circle). The incidence angle of the primary beam is chosen accordingly and the mask in front of the detector is positioned so that reflections from a sufficient number of lattice planes are obtained and can be evaluated unambiguously. At least the reflections from two lattice planes must be measured. Measuring crystal orientations with a symmetry axis close to the rotation axis, the corresponding number of symmetrically equivalent reflections is registered [4] (Fig. 2).

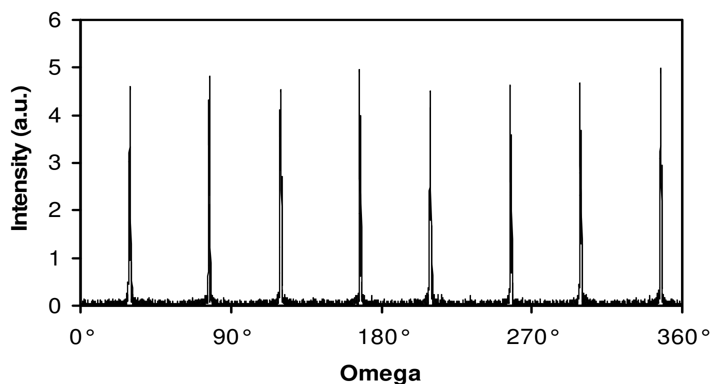


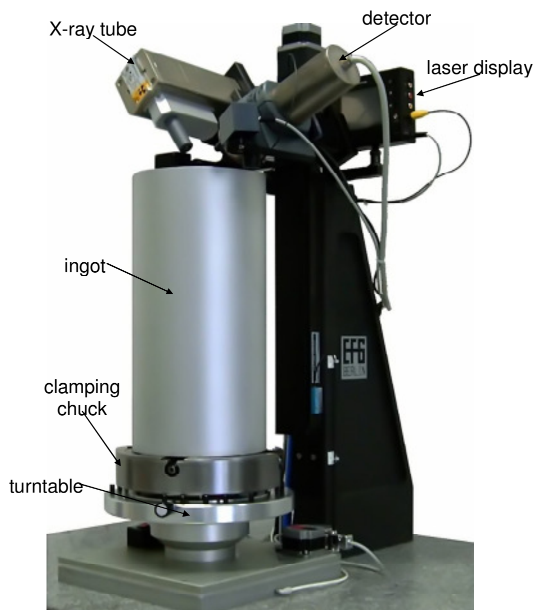
Fig. 2 Measured Ω-Scan diagram, CuKα radiation, Si, (100) orientation, reflections of the type 311

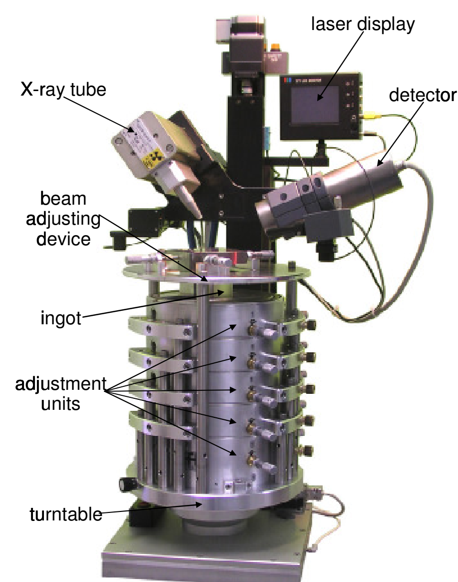
From the angular positions of the reflections, the angles between the lattice plane normals and the rotation axis as well as the incidence angle of the X-ray beam (for given lattice geometry) or one diffraction angle (for given incidence angle) can be calculated.

From the lattice-plane angles the orientation of the crystal lattice is determined, expressed, e.g., by the polar coordinates related to the crystallographic coordinate system or by the orientation deviation of the rotation axis from a low-indexed lattice plane normal. Moreover, also the azimuthal angle of the projection of any lattice direction on the Ω circle yields from the measurement. For cubic crystals, for one type of reflections and for given incidence angle, the diffraction angle and so the lattice parameter can be calculated together with the orientation. The incidence angle must be exactly determined before by means of a reference crystal with known lattice geometry. Therefore, the determined lattice parameter is related to that of the reference crystal.

Generally, to single crystals with unknown arbitrary orientation, a modified measuring variant of the Ω-Scan must be applied: The detector is set to receive one or more suited types of reflections and the incidence angle of the X-ray beam is varied step by step until reflections from at least three lattice planes have been registered [4]. The evaluation is similar to that for constant incidence angle. Utilizing the crystal symmetry, the reflection types are to be chosen in such a way that the total scanning range is as small as possible (in unfavourable cases more than 50°). For cubic crystals, one type of reflections is usually sufficient. In favourable cases (e.g., Si, Ge, GaAs, CuKα radiation), an arbitrary orientation can be measured with fixed incidence angle.

Systematic errors in the orientation coordinates and the lattice parameters are mainly due to the problem to find out the “true” peak positions of the reflections. Because the reflection curve widths may amount several degrees, these errors can reach some tenths of a degree in the reflection positions and up to one arc minute in the orientation coordinates and the diffraction angle. 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 [5, 6]. The remaining uncertainty is in the order of the usual statistic errors (see below).





a **b**

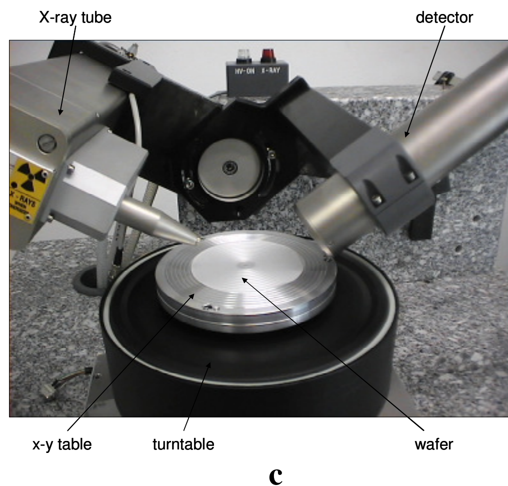


Fig. 3 Apparatus for the Ω-Scan measurement
(a) Arrangement with a Si ingot, 400 mm height, 200 mm (8 in.) diameter,
(b) with attachment for the adjusted stacking and gluing of ingots (sapphire), stacking height 300 mm, ingot diameters 50 to 100 mm (2 to 4 inches),
(c) with x-y table for mapping of wafers

3 Apparatus and Applications

Basic unit of the Ω-Scan measuring system is the turntable, which must rotate uniformly and precisely and must be sufficiently stable to bear large ingots and adjustment units (Fig. 3). In the universal version of the diffractometer, the X-ray tube with beam collimator and the detector with mask can be set to their pre-calculated angular positions. This type of machine is also to use for the determination of unknown orientations. If only one crystal type (or crystals with similar lattice geometry) in a limited orientation range (e.g., within 5° deviation from a given basic orientation) is to be measured or for special cases of arbitrary orientation (e.g., Si, Ge, GaAs, see above), an arrangement with fixed X-ray tube and detector can be used.

The height of the X-ray tube and detector bearing part is automatically adjusted to the sample height by means of a light sensor.

Presumed the turntable is sufficiently precise, the reproducibility of the measured reflection positions depends mainly on the pulse statistics. Using strong reflections, a usual sealed X-ray tube and a measuring time of two seconds (one revolution of the turntable), the angular positions of the reflections on the Ω circle and, therefore, the azimuthal angle can be evaluated with a precision in the order of 0.01°. Depending on the lattice and measuring geometries the standard deviations of the coordinates of the lattice orientation as well the diffraction angle of cubic crystals are usually in the order of a few arc seconds. For high diffraction angles (near 80°) the relative error of the lattice parameter is then in the order of 5×10^{-6}. If necessary, the reproducibility can be improved using a lower rotation speed or overlays of a few revolutions.

The relationship of the crystal lattice to the outer sample geometry is determined either mechanically (e.g., by stop edges for flats) or by photoelectric tools. The exact position of an even surface to the rotation axis can be measured by means of a reflected laser beam whose positions during the turntable rotation are additionally evaluated. Also the azimuthal orientation of a flat or notch as well as the wobbling of a cylindrical ingot can be derived from intensity changes of direct or reflected light beams. So the orientation coordinates can be converted to the surface normal or to the cylindrical axis, respectively.

The adjustment of crystal samples for further processing, e.g., of ingots for succeeding oriented cutting, can be performed manually after the orientation measurement by means of a corresponding wobble unit placed on the turntable. If the ingot is to be glued to a beam which will be fastened then to the saw, this beam can also be adjusted exactly parallel to the rotation axis as reference axis by means of the optical tools. Fig. 3b shows the machine with a stack of

adjustment units for the processing of a number of shorter single ingots (here: sapphire) and the holding and adjustment tool for a common steel beam.

For the mapping of surfaces the usual turntable is exchanged for a special one with shift mechanism of the sample support. In the example shown in Fig. 3c the two-dimensional shift is realized by means of a rotation and a translation, which halves the necessary radius of the table. The position accuracy in x and y direction is about 0.1 mm. For the mapping of sufficiently well light-reflecting surfaces, the orientation in any measuring point is related to the surface by means of the additional laser reflection. So, small irregularities in the shift mechanism do not influence the accuracy of the orientation parameters.

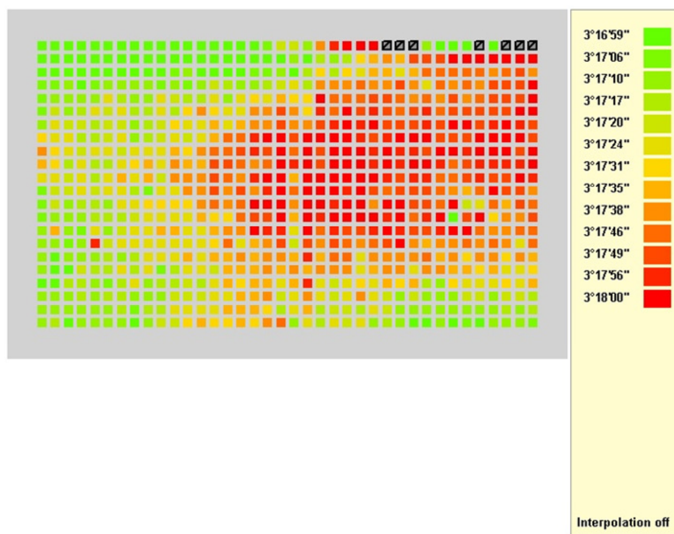


Fig. 4 Orientation map (raster 1 mm) of an AT-cut quartz wafer, 26 · 42 mm^2, reflections 2 0 2 / 2 0 3, CuKα radiation. Scale range of the main orientation coordinates: 3°16’59” to 3°18’00”

4 Examples for surface mapping

Fig 4 shows an example of the orientation map of an AT-cut quartz wafer. Whereas the reproducibility of the main orientation coordinate is about 3 arc seconds, there are large-extended orientation fluctuations up to 1 arc minute.

The comparison of orientation and lattice-parameter changes in a $Si_{1-x}Ge_x$ wafer is shown in Fig. 5. There are very well correlated concentric fluctuations in both modes. X-ray topographs [7, N.V. Abrosimov, private communication] show these structures qualitatively and with better lateral resolution, however, the mapping images allow the quantitative evaluation of the orientation as well as of the lattice parameters and, deduced from that, of the Ge concentration. The maximum orientation difference (Fig. 5a), mainly due to an additional curvature (right above to left below), is about 3 arc minutes. The lattice parameters show a similar behaviour (a small gradient in direction of the curvature), however, the radial changes are dominating. The maximum lattice-parameter difference is 2.8×10^{-5} nm. From the known non-linear connection between lattice parameters and concentration in the solid-solution system Si-Ge [8] yields that the maximum difference of the Ge content is 0.14 at% (error of concentration values about 0.01 at%).

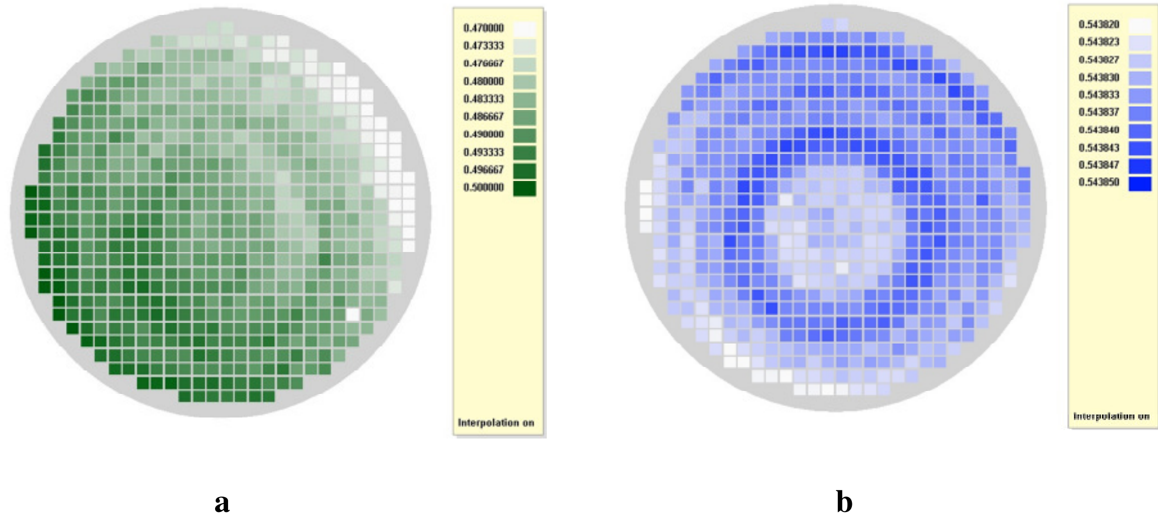


Fig. 5 Orientation and lattice-parameter maps (raster 1 mm) of a $Si_{1-x}Ge_x$ wafer, diameter 35 mm (mapping array 30 mm), reflections of the type 4 4 4, CuKα radiation
(a) Orientation map, scale range of the orientation deviations: 0.47° to 0.50°,
(b) lattice-parameter map, scale range of the lattice-parameters: 0.54382 nm to 0.54385 nm

Mappings using the Ω-Scan method as shown above can be taken in half an hour (about 600 measuring points) and so give a rapid survey over striking features of the crystallographic structure of the wafer. Higher lateral resolution down to a few tenths of a millimetre would enlarge the measuring time per point about ten times or more for comparable errors. However, such a procedure can be chosen if a pre-selected smaller area is to be characterized in more detail.

5 Conclusions

The Ω-Scan measuring process and the corresponding basic apparatus are relatively simple. Using a conventional X-ray source they deliver exact results in short measuring times. Therefore, method and set-up are especially suited for series studies and for industrial application. For this purpose, the apparatus may be equipped with automatic or semiautomatic devices for feeding and sorting of samples according to the measured results [3].

Also the potentialities of the Ω-Scan method to register simultaneously a number of X-ray reflections using one continuous rotation is not yet exhausted. So, the use of crystal collimators and/or of other X-ray-optical elements will open the possibility to measure also rocking curves for the characterization of the crystal perfection by means of the Ω-Scan geometry.

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References

1. B. MORYS, H. BRADACZEK, G. HILDEBRANDT, in *Proceedings of the 1994 IEEE International Frequency Control Symposium*, Boston (1994) 237.

2. H. BERGER, H. BRADACZEK, H.-A. BRADACZEK, G. HILDEBRANDT, in *Proceedings of the 1996 IEEE International Frequency Control Symposium*, Honolulu, Hawaii (1996) 412.

3. G. HILDEBRANDT, H. BRADACZEK, J. Optoelectr. Mater. **6** (2004) 5.

4. H. BERGER, J. Phys. IV France **118** (2004) 37.

5. H. BERGER, H. BRADACZEK, G. HILDEBRANDT, in *Proceedings of the 19th Piezoelectric Devices Conference & Exhibition*, Kansas City (1997) 12/1.

6. H. BERGER, Cryst. Res. Technol. **37** (2002) 716.

7. A. ERKO, N.V. ABROSIMOV, V. ALEX, Cryst. Res. Technol. **37** (2002) 685.

8. H. J. HERZOG, in *Properties of strained and relaxed Silicon-Germanium*, EMIS Data reviews series No. 12 (Inspec, London, 1993), p. 49.