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APPLICATIONS OF X-RAYS IN THE QUARTZ-OSCILLATOR INDUSTRY

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1. Introduction

Every year since 1946 the American IEEE (institute of Electrical and Electronics Engineers) organizes an International Frequency Control Symposium, taking place somewhere in the United States. So at last year's meeting, held in Honolulu (Hawaii), the 50th (“Golden”) Anniversary could be celebrated. The result, documented on the 1250 pages of the Proceedings [1], provides a clear impression of the importance of the still growing field of frequency control. About 180 contributions, written by authors from all over the world, cover the whole area, beginning with the already “classical” quartz crystal frequency standards, and ending with the most recent developments in atomic clocks. The latter, using excited atomic levels of Rubidium, Cesium, Hydrogen etc., provide highest precision, even enabling measurements on the relativistic effect of moving clocks.

Quartz sources are inferior in accuracy to those quantum sources by about four orders of magnitude; large-scale industrial precise oscillator application, however, is still ultimately dominated by the quartz resonator. Its performance had improved dramatically since the first AT-cut resonators had been developed in the early 1950's. A better accuracy, higher by one order of magnitude, and an improvement of other properties was later achieved using the revolutionary design of the SC-cut (= shock-compensated cut) resonator.

Attempts to replace quartz by other materials did not yield much success. So artificially grown quartz bars are still (and will probably remain in the next future) the most convenient raw material for the production of resonators on a large scale: some big companies nowadays have a weekly output of millions of oscillators.

Properly oriented thin slices of quartz, called “blanks”, with dimensions of a few millimeters and thickness of some tenths of one millimeter are the basis of the oscillator production; there is, however, a trend to smaller and thinner blanks down to dimensions of one millimeter and 20 µm thickness. Apart from the frequency precision and stability of the oscillators, the temperature and shock stability are important properties which must be considered by the producers. This requires an accuracy as high as possible of the lattice orientation relative to the surface of the blanks: the accuracy of the “cutting angle” has to be in the range of seconds of arc.

The most important cuts have already been mentioned: the “classical' AT-cut and the improved SC-cut. Their orientations within the lattice can best be presented in a projection of lattice planes. At first Fig. 1 shows (idealized) left- and right-handed quartz crystals with the main crystal faces.

A projection of these faces (or lattice planes, resp.) is given in Fig. 2 together with the indication of industrially used cut positions.

The still predominating AT-cut can easily be adjusted by one single rotation (with the rotation axis lying within the XY plane) of about three degrees from the $(01\bar{1}1)$ lattice plane (which is strongly X–ray reflecting). The adjustment of the SC-cut is much more difficult, because two independent rotations have to be considered; the result are “doubly rotated blanks”. This cut is coming more and more into use (in spite of the much higher price of these blanks) due

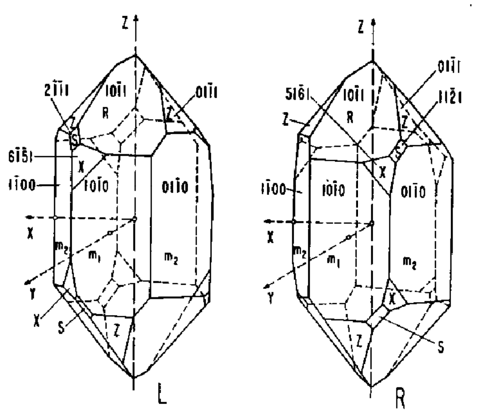

Fig. 1. Idealized left- (L) and right-handed (R) quartz crystals (from [1])

to its improved properties like increased frequency stability, higher shock resistance etc.

The desirable properties of the blanks can be guaranteed only if the calculated cutting angle is realized with an accuracy of a few seconds of arc during production, and the only means of controlling small deviations in this range are measurements of X–ray reflections.

2. Conventional Methods of Blank Measurements: The Θ–Scan

The easiest method for lattice orientation measurements is the so-called Θ–scan. It requires an apparatus with a relatively simple construction and is therefore in wide spread use. An X–ray beam originating from the line focus of an X–ray tube hits a monochromator and is reflected, after being limited by vertical narrow slits, as a small line of parallel monochromatic X–rays. This beam is directed to the sample, reflected at a suited lattice plane and then collected by a detector, cp. Figs. 3a, b. A reflection occurs only if the beam meets exactly the Bragg condition (cp. Ch. 3.2) at the plane; thus the angle between the direction of the monochromatized incident beam and the beam reflected to the detector is equal to twice the Bragg angle.

By rotating the sample around an axis lying in its surface and vertical to the plane of the beams, the

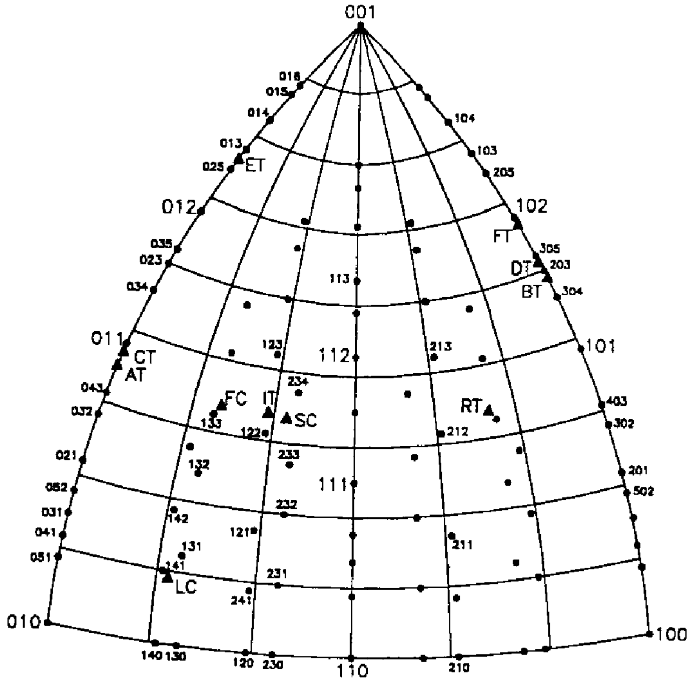

Fig. 2 Projections of the normals of 1/12 of the most important quartz lattice planes onto a sphere; projections of some cut planes are included. The projection directions 010 and 001, resp., are identical with the Y- and Z-axis, resp.

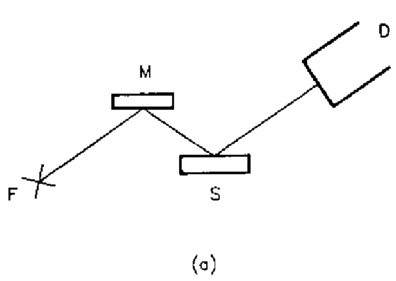

Fig. 3 Θ-scan in parallel (a) or anti-parallel mode. F focus, M monochromator, sincerely sample, D detector. The blank sincerely is rotated through the Bragg position (Θ_B Bragg angle). For some reasons mode (a) is to be preferred.

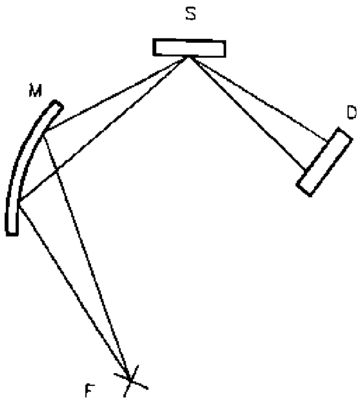

Fig. 4 Θ-scan with curved monochromator M. F focus, sincerely sample (blank), D position sensitive detector.

for further applications like sorting, cutting and so on.

sophistication has been constructed*, which uses a

detector, cp. Fig. 4.

requirement for X–ray power (a few watts are

be used either for a manual or for an automatic sorting

Both Θ–scan arrangements have a high reproducibility, because they are based on a very sensitive double crystal arrangement; but they have, at the same time, the severe disadvantage of being unable to discover special cutting errors of the sample; the same errors may be repeated with the same high precision.

Another important disadvantage is the impossibility to use these methods for measurements of round quartz blanks. These blanks are produced by rounding rectangular blanks followed by a suited lapping to bring them down to the necessary thickness for the final processing step. Therefore the measurement of the blanks after rounding and lapping guarantees for a larger reliability of the cutting angle.

3. AT-cut Measurements

3.1. Preliminary Remark

For many applications the frequency stability and precision of AT-cut oscillators are sufficient. Because the lumbered quartz bar has to be rotated for cutting in just one direction, the manufacturing process is much simpler than that for the preparation of doubly rotated blanks. For these reasons AT-cut oscillators have still by far the largest production figures.

Nevertheless there exist some sources of miscutting, namely the improper adjustment of the bar, and the possible cutting of the blade saw in a direction which is not exactly vertical to the preadjusted surface of the bar, the reference plane $(\bar{2}110)$. These miscutting errors result in nonparallelities of surfaces, XX'-tilt, and cutting errors of the edges, cp. Fig. 5.

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* Product of the EFG GmbH, Berlin (Germany).

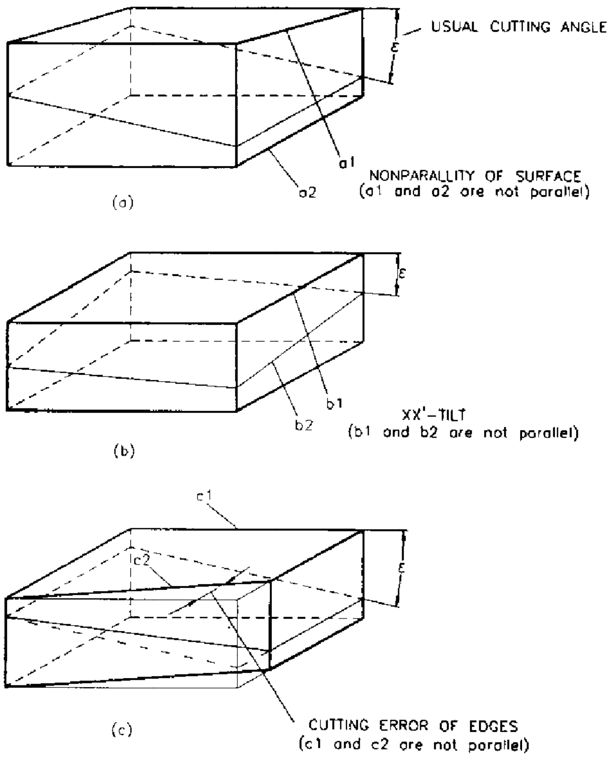


Fig. 5 Resulting cutting errors of (rectangular) blanks

3.2. The Simple Ω–Scan

If the errors in the X direction are small, the so-called Ω–Scan in its simpler form can be used for sufficiently exact measurements [2]. The Ω–Scan apparatus consists principally of an X–ray tube and detector, and a precision rotation table. The blank to be measured is set down to the table with its surface as parallel as possible to the surface of the table (or in other words, with its surface exactly perpendicular to the rotation axis). The X–ray beam with an elliptical cross-section hits the centre of the blank in a direction which does not exactly meet the Bragg condition

$$n\lambda = 2\, d_{hklm} \sin\Theta_B$$

(λ X–ray wave-length, d_{hklm} distance of the lattice planes (hklm)=$(01\bar{1}1)$, measured in second order (n=2) in our case; Θ_B Bragg angle); actually the angle of incidence Θ deviates from Θ_B by a small angle δ:

$$\Theta = \Theta_B - \delta, \text{ cp. Fig. 6.}$$

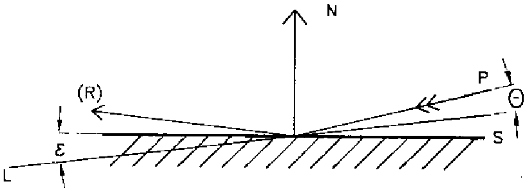


Fig. 6 Beam geometry seen in a projection parallel to the blank surface S and the $(01\bar{1}1)$ lattice plane L (ε=3° angle between S and L). The incident X-ray beam P enters L by an angle $\Theta=\Theta_B-\delta$; thus Θ is a little smaller than the Bragg angle Θ_B, a reflection (R) cannot occur within the plane of this drawing. In the Ωan the blank rotates about the lattice plane normal N and reflections occur at +Ω and -Ω.

Rotating the blank around its normal N about certain angles Ω, however, brings the lattice plane L in two symmetrical positions where a reflection R occurs, and as is shown easily the exact amount of the cutting angle ε can be calculated from the measured distance between these positions +Ω and -Ω. The accuracy of the measurement depends mainly on the accuracy of the rotation; errors due to wobbling or discontinuities have to be avoided (or considered in the computer evaluation program). The standard deviation in the measurement is in the range of 10^{-3} degrees.

The main problem, however, is not the accuracy of the instrument, but rather the low quality of the blanks: the results are influenced by dirt and fragments at the blank surfaces, by XX' miscutting, and missing flatness or parallelity. Already particles of 0.1 µm size sitting on one of the three pins which support the blank during the measurement lead to an error of 10^{-3} degrees. A proper lapping and cleaning of the blank surfaces could help to solve these problems.

3.3. The Improved Ω–Scan

The Ω–Scan is an extremely precise method to determine AT-cut errors in round or rectangular quartz blanks; accuracies down to some 3 seconds of arc, however, can be guaranteed only if there is no significant XX' miscutting contained in the blanks. As is shown in Fig. 7 larger values of XX' introduce considerable errors into the final results for the cutting angle.

Since the XX' miscutting is directed perpendicular to the AT-cut, additional independent information is needed which can be provided only from measurements of another reflection pair from a second lattice plane. Therefore the problem arises to find an additional diffraction which must meet the following two conditions: Firstly, the directions of incident and diffracted X–ray beams at the second lattice plane must be sufficiently close to those of the main reflection on (01$\bar{1}$1); and, secondly, the now excited two additional peaks must unambiguously be distinguishable from the two main peaks. In our particular case of the AT-cut measurement the lattice plane (02$\bar{2}$3) has been proved to meet these conditions [3]. An example of the four peaks resulting from diffraction at (01$\bar{1}$1) and (02$\bar{2}$3) during one complete Ω-circle is shown in Fig. 8.

The projections of (01$\bar{1}$1) and (02$\bar{2}$3), resp., are positioned together with the projection of the AT-cut position at the same meridian (cp. Fig. 2), but on opposite sides of AT. the 02$\bar{2}$3 reflection must occur just after one half turn of the blank behind the 02$\bar{2}$2 reflection (which can also be seen in Fig. 8: the centres between the two reflections at the planes (01$\bar{1}$1) and (02$\bar{2}$3), resp., are 180° away from each other); but ΔΩ equals 180° exactly only in case of absence of any XX' tilt. So any deviation of ΔΩ from 180° indicates the existence of some XX' angle which can be calculated and then introduced into the evaluation calculation to correct the final result adequately.

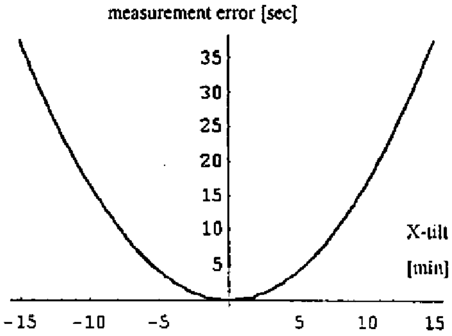


Fig. 7 Measuring error due to some tilt XX' (from [3])

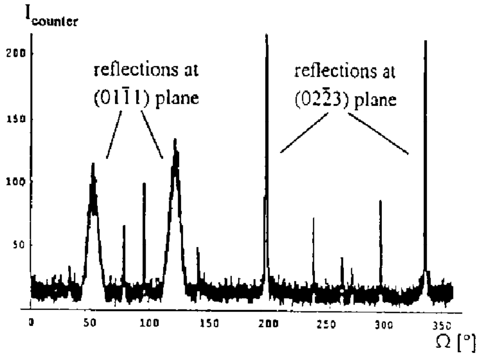


Fig. 8 Improved Ω-scan measured during one whole rotation (Ω=360°) of the sample; two pairs of peaks belong to reflections at (01$\bar{1}$1) and (02$\bar{2}$3), resp. (from [3]).

If necessary the value of XX' can be printed out separately. Using this improved Ω–Scan the influence of the XX' tilt has been reduced to the range of seconds of arc.

4. Measurements of SC-Cut (and Similar Doubly Rotated Cut) Blanks

4.1. Preliminary Remark

As has already been mentioned, the majority of quartz oscillators for industrial applications is still produced in the form of AT-cut blanks. The outstanding property of the AT-cut is its temperature coefficient close to zero, its disadvantage is its sensitivity to small temperature changes and to acceleration forces. Nowadays certain applications (e.g. in space systems like missiles, Global Positioning Systems etc.) require better performance. Since some time improvements have been achieved by using resonators with other cut positions. The best known of them is the SC-cut, and its projection, together with the projections of similar cuts like IT, FC etc., is indicated in Fig. 2. Unfortunately none of these projections has a common meridian with the projection of any important quartz lattice plane, and it is, therefore, impossible to arrive at one of these cut positions just by one single rotation from a suited reference plane. Consequently two rotations from two reference planes are needed, and this is the reason why the term 'doubly rotated cuts" came into use for these higher sophisticated cuts. Their production is much more complicated than that of the AT-cut, and the same seemed to hold for the measurement, but fortunately it turned out that the Ω–Scan could also be applied in these cases; the Improved Ω–Scan was already the first step into the direction to this new range of application.

4.2. Measurement of SC-Cut Blanks

The problem of finding two suited lattice planes for the measurement of SC-cut blanks is similar to that described above in connection with the Improved Ω–Scan, but much more complicated. The help of a detailed computer program was needed to detect two quartz lattice planes which fulfilled the geometrical condition that one and the same incident beam direction could be used and the reflected beams hit the same detector, and that the resulting peaks were clearly separated such to be identified unambiguously. The angles between the rotation axis and the normals of the two planes could be determined with high precision, and from these angles the co-ordinates of the SC cut surface were derived with sufficiently small errors [4].

The evaluation process analyses at first the angular distances between the two peaks belonging to the reflections of each of the two lattice planes. The errors in the lattice plane angles calculated from this process are then further diminished by considering the angular distance between the centres of each peak pair.

Using the reflections $12\bar{3}1$ and $12\bar{3}3$, applicable for SC- and FC-cuts, and cycle times of about ten seconds, the peak distances have been measured with standard deviations (STDV) of about 0.01°. This is sufficiently exact to calculate the lattice plane angles as well as the angular co-ordinates Θ and Φ (which are used to characterize the two rotations), with a STDV of a few arcseconds. For the measurements of other orientations, e.g. IT-cuts, similar errors result if other suited reflection pairs are applied.

5. Errors due to Surface Misorientation: A New Laser Technique

During the practical application of the Ω–Scan in the last five years the experience has been gained that problems arose if the surface of the blanks was not sufficiently flat, if dirt or fragments were deposited between surface and support, or if the wobble adjustment of the turntable was not made properly.

These difficulties could be avoided by controlling the orientation of the surface with the help of a suited equipment which should guarantee an accuracy in the range of the accuracy of the cutting angle measurement i.e. about 10^{-3} degrees. Such an equipment has now been realized using a laser, a couple of surface-costed mirrors and a high resolution CCD camera. An optical scan of the surface of the quartz blank can now be carried out to determine its particular angle towards the X–ray beam.

Very often the influence of surface non-parallelities has been discussed. Contrary to some opinions, it does not matter which one of both surfaces is taken as the reference one, unless it is marked.

The laser beam allows a check of the degree of parallelity. For a measurement without considering the laser control, the lower surface has to be taken as reference because it is in contact with the supporting rotation table; with laser correction, however, the upper surface is the reference because it is reflecting the laser beam. This opens up a possibility to check the parallelity-at least principally. If an individual check is necessary, the marking of the blanks is unavoidable. First experiences with the laser technique show that it offers several advantages. One example: the wobble correction of the turntable which usually takes about twenty minutes can be carried out within one minute with laser help. The measurement seems now to be almost uninfluenced of non-flatness or fragments and it becomes possible to handle polished, or very thin (≥20 µm) and small (≥1.5 mm diameter) blanks.

6. Technical Design of Ω–Scan Apparatus

The EFG Automated Quartz Sorting Machine for round quartz blanks has been developed with the purpose to enable the user to measure the cutting angle ε between the surface and the $(01\bar{1}1)$ lattice plane of round (and also of rectangular) AT-cut quartz blanks and to sort them, corresponding to the particular cutting angle ε, into 26 boxes.

The method of measurement has already been described in Chapters 3.2 and 3.3. The mechanics of the apparatus consists of a three-circle goniometer which has been developed especially for this task. Apart from the measuring mechanics an integrated feeding and sorting system is included. The whole system is mounted on two granite plates and protected against dust and temperature influence by a plexiglass cover which serves additionally as an X–ray shield. The motor control unit as well as the X–ray generator are mounted in the table rack.

The larger of the two granite plates, arranged in a horizontal position and covering the rack, supports the rotation axis in a vertical position. The second smaller plate, positioned vertically to the first one, supports the axis of a solid lever to which the X–ray tube and the detector are fixed in angular positions corresponding to twice the Bragg angle. This lever can be rotated around the center of the crystal surface by a few degrees to allow the adjustment of the proper angle of incidence for a specified cutting angle (for certain purposes deviating from the usual angle ε=2°58').

Although the rotation axis is arranged exactly vertically one observes deviations of the turntable from the precise horizontal position due to some wobble error. Therefore two adjustment screws are provided enabling the correction of this error (the wobble error correction is decisively simplified if the laser technique mentioned in Chapter 5 is applied).

The main part of the sorting system, the feeding arm, picks up the blanks from the vibration feeder and positions them at the support of the turntable. After one rotation, which takes about two seconds time, measurement and evaluation are finished and the sorting arm takes the blank to the proper sorting box which had been selected by the computer. One turn of the rotation table is divided into 4000 measuring

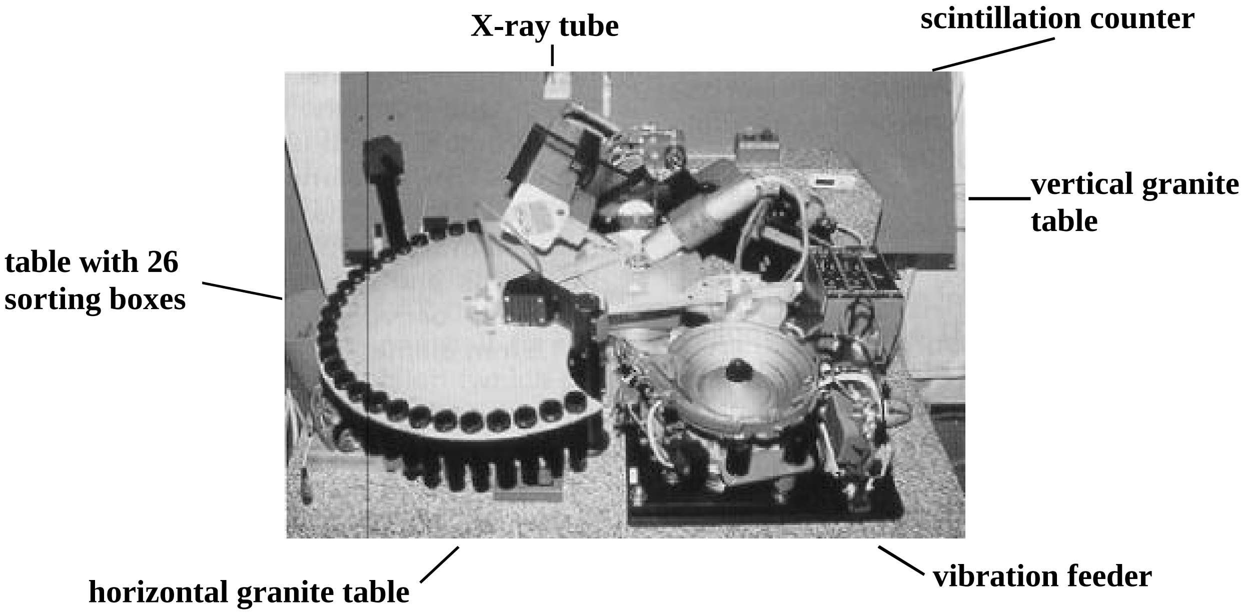
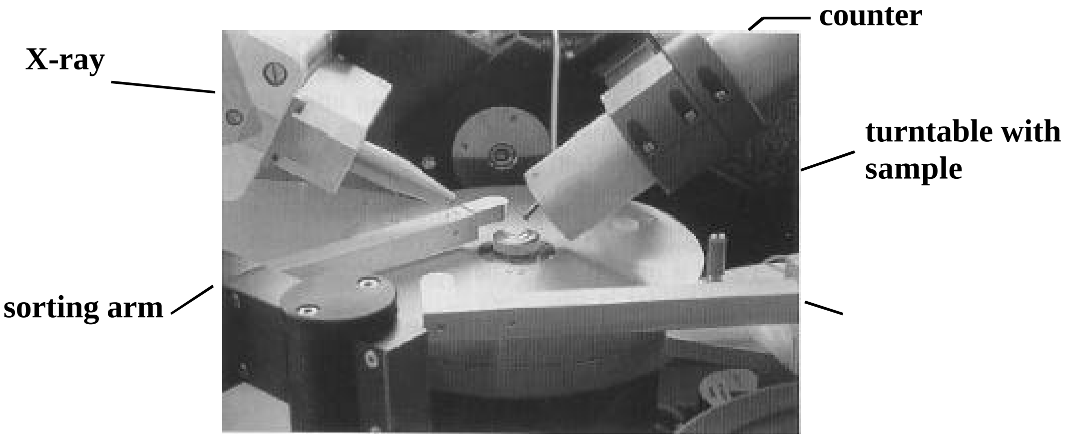


Fig. 9 View of the main parts of the EFG sorting machine.



View of the rotation table

steps. They together with the revolution velocity restrict the accuracy to a value which is usually expected by the user. If higher accuracies are wanted multiple measurements (summing over up to four turns) can be provided.

The technical design of the SC-cut sorting machine is almost the same as that of the just described AT-cut machine. However due to the fact that the production rate of SC-cut blanks is much lower than that of AT-cuts, a semiautomatic arrangement has been constructed where the blanks have to be positioned by hand onto a secondary rotation table which transports them to the measuring position. There a magnetic lift positions the blanks onto the turntable support. After the measurement each blank is transported via lift and secondary turntable out of the cabin. There it can be picked up by hand and put into the box which is indicated by the computer.

The SC-cut sorting machine is available as well as an automatic instrument.

In Figs. 9, 10 photographs of the EFG apparatus are shown.

7. Other Applications of X–Rays in the Quartz Industry

7.1. Automatical Adjustment of Bars

One of the reasons of the unsufficiently large angular distribution of the raw blanks is the inaccurate cutting process. There are various error sources: a) non proper growing of the bars, b) unsatisfying alignment of mounting the bars on the saw and c) an imperfect adjustment of the saw blades.

The only possibility to improve point b) is an X–ray controlled adjustment of the bars, because the saw defines the surface of the blanks and the X–ray measurement controls the lattice plane orientation, and both together determine the cutting angle.

While in other units as described above the Ω–Scan is in use, the bar adjustment machine is equipped with a Θ–scan X–ray system.

A glass plate is smeared with an ultraviolet hardening glue to provide the basis for the bars which are arranged in layers, each of them containing eight bars. The lowest eight bars are put in an approximately correct position. Two punches are starting to press the first bar down onto the glass plate. The X–ray beam is adjusted to hit the $(01\bar{1}1)$ lattice plane of the bar. While one punch fixes the bar, a second punch shifts the bar in a position, where the maximum value of the selected reflection is registered. If the bar is adjusted two laser beams are fixing provisionally the bar to the glass by polymerization of the glue at two points. After having orientated the first bar the system is shifted to the next one, and then successively to all eight ground bars. Now the polymerisation of the glue is enforced by a large ultraviolet light source.

The same procedure starts with the next layer of 8 bars. At least 24 bars can be adjusted and fixed automatically before the glass plate is fed to the blade

the range of 5" STDV.

7.2. X–Ray Controlled Saw for SC-Cut

The same problems as mentioned in the previous chapter occur with the cutting of SC-cut blanks, but

to be considered adjusting and cutting these "doubly rotated" blanks. These difficulties are the reason for

with SC-cut blanks.

The saw to be described here [5] contains two com-

an Ω
described in the chapter on the SC-cut sorting device.

The normal seedless (AT) bar is mounted on a rotation

and the detector remain in a fixed position. The process starts with an X–ray measurement during one

line of the provided surface. The results of the measurement are used for the correct adjustment of

computer.

The measuring procedure is followed by the cutting

cutting, while the bar holder is rotated similar to the rotation during the measuring process. There are

blade and bar rotation: better cooling of the blade and removing of the particles, and the possibility to use a

cutting process should be below 5" for both SC-cut angles.

8. Summary

The demands of the oscillator industry for improved qualities of the quartz blanks, the continuous process of miniaturization of the oscillators and the ever increasing throughputs in oscillator production require improved new methods for the measurement of the final products. The application of X–rays in the above described different types of "Ω–Scan Method" proved to be the only means which guarantees measurements with highest precision of lattice orientation in blanks.

The combination with a laser surface measurement enables to get the cutting angle with an accuracy of a few seconds of arc, allowing the sorting at a similar precision. The round blank sorting offers the advantage to carry out measurements in a later step of the fabrication process, thus avoiding errors which can occur during these steps. Several further applications are under development, and it can be foreseen that X–ray applications will still increase in the next future in quartz oscillator production.

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