

INTERNATIONAL STANDARD

NORME INTERNATIONALE

Synthetic quartz crystal – Specifications and guidelines for use

Cristal de quartz synthétique – Spécifications et lignes directrices d'utilisation



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INTERNATIONAL STANDARD

NORME INTERNATIONALE

Synthetic quartz crystal – Specifications and guidelines for use

Cristal de quartz synthétique – Spécifications et lignes directrices d'utilisation

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

SYNTHETIC QUARTZ CRYSTAL – SPECIFICATIONS AND GUIDELINES FOR USE

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International Standard IEC 60758 has been prepared by IEC technical committee 49: Piezoelectric, dielectric and electrostatic devices and associated materials for frequency control, selection and detection.

This bilingual version (2017-11) corresponds to the monolingual English version, published in 2016-05.

This fifth edition cancels and replaces the fourth edition, published in 2008. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

- order rearrangement and review of terms and definitions;
- abolition as a standard of the infrared absorbance coefficient $\alpha_{3\ 410}$;
- addition of the α value measurement explanation by FT-IR equipment in annex;
- addition of the synthetic quartz crystal standards for optical applications.

The text of this standard is based on the following documents:

FDIS	Report on voting
49/1185/FDIS	49/1190/RVD

Full information on the voting for the approval of this standard can be found in the report on voting indicated in the above table.

The French version of this standard has not been voted upon.

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

The committee has decided that the contents of this publication will remain unchanged until the stability date indicated on the IEC website under "<http://webstore.iec.ch>" in the data related to the specific publication. At this date, the publication will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

INTRODUCTION

The reason for adding synthetic quartz crystal for optical application to this International Standard is as follows.

Quartz crystal produced for optical applications is produced by many of the same suppliers manufacturing quartz for electronic applications. The equipment and methods to produce optical quartz are similar to those used in the production of electronic quartz. Also, with a few exceptions the characterization methods of electronic and optical material are similar. Therefore, IEC 60758 serves as the proper basis for including addenda related to quartz crystal for optical applications.

SYNTHETIC QUARTZ CRYSTAL – SPECIFICATIONS AND GUIDELINES FOR USE

1 Scope

This International Standard applies to synthetic quartz single crystals intended for manufacturing piezoelectric elements for frequency control, selection and optical applications.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60068-1:2013, *Environmental testing – Part 1: General and guidance*

IEC 60122-1:2002, *Quartz crystal units of assessed quality – Part 1: Generic specification*

IEC 60410, *Sampling plans and procedures for inspection by attributes*

IEC 61994 (all parts), *Piezoelectric and dielectric devices for frequency control and selection – Glossary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in IEC 61994 and the following apply.

3.1

hydrothermal crystal growth

crystal growth in the presence of water, elevated temperatures and pressures by a crystal growth process believed to proceed geologically within the earth's crust

Note 1 to entry: The industrial synthetic quartz growth processes utilize alkaline water solutions confined within autoclaves at supercritical temperatures (330 °C to 400 °C) and pressures (700 to 2 000 atmospheres).

Note 2 to entry: The autoclave is divided into two chambers: the dissolving chamber, containing raw quartz chips at the higher temperature; the growing chamber, containing cut seeds at the lower temperature (see 7.1.2).

3.2

synthetic quartz crystal

single crystal of α quartz grown by the hydrothermal method

Note 1 to entry: Cultured quartz has the same meaning as synthetic quartz crystal.

3.3

as-grown synthetic quartz crystal

state of synthetic quartz crystal prior to grinding or cutting

3.4

as-grown Y-bar

crystals which are grown by using long stick seed in the Y-direction

3.5

as-grown Z-bar

crystals which are grown by using Z-cut seed

3.6

synthetic quartz crystal batch

synthetic quartz crystals grown at the same time in one autoclave

3.7

seed

rectangular parallelepiped quartz plate or bar to be used as a nucleus for crystal growth

3.8

growth zones

regions of a synthetic quartz crystal resulting from growth along different crystallographic directions

SEE: Figure 2.

3.9

orientation of a synthetic quartz crystal

orientation of the seed of a synthetic quartz crystal with respect to the orthogonal axes specified in 3.7

3.10

orthogonal axial system of α quartz crystal

orthogonal axis system consisting of three axes with a mutually vertical X axis, Y axis and Z axis as illustrated in Figure 1

Note 1 to entry: The z-cut seed may be oriented at an angle of less than 20° to the Y-axis, in this case the axial system becomes X, Y', Z'.

3.11

AT-cut plate

rotated Y-cut crystal plate oriented at an angle of about +35° around the X-axis or about –3° from the z (minor rhombohedral)-face

SEE: Figure 3.

3.12

X-cut plate

crystal plate perpendicular to the X-axis

SEE: Figure 3b.

3.13

Y-cut plate

crystal plate perpendicular to the Y-axis

SEE: Figure 3b.

3.14

Z-cut plate

crystal plate perpendicular to the Z-axis

SEE: Figure 3b.

3.15

z (minor rhombohedral)-cut plate

crystal plate parallel to the z (minor rhombohedral)-face

SEE: Figure 3a.

3.16 dimensions

dimensions pertaining to growth on Z-cut seed rotated less than 20° from the Y-axis

3.17 effective Z-dimension

as-grown effective Z dimension defined as the minimum measure in the Z ($\theta = 0^\circ$) or Z' direction in usable Y or Y' area of an as-grown crystal and described by Z_{eff}

SEE: Figure 2.

3.18 minimum Z-dimension

minimum distance from seed surface to Z-surface described by Z_{min}

SEE: Figure 2d.

3.19 inclusions

any foreign material within a synthetic quartz crystal, visible by examination of scattered light from a bright source with the crystal immersed in a refractive index-matching liquid

Note 1 to entry: A particularly common inclusion is mainly the minerals called acmite and emeleusite.

3.20 seed veil

array of inclusions or voids at the surface of the seed upon which a crystal has been grown

3.21 etch channel

roughly cylindrical void that is present along the dislocation line after etching a quartz crystal

3.22 dopant

additive used in the growth process which may change the crystal habit, chemical composition, physical or electrical properties of the synthetic quartz batch

3.23 pre-dimensioned bar

bar whose as-grown dimensions have been altered by sawing, grinding, lapping, etc., to meet a particular dimensional requirement

3.24 impurity concentration

concentration of impurities relative to silicon atoms

3.25 dislocations

linear defects in the crystal due to misplaced planes of atoms

3.26 autoclave

vessel for the high-pressure and high-temperature condition required for growth of a synthetic quartz crystal

3.27**right-handed quartz or left-handed quartz**

handedness of quartz crystal as determined by observing the sense of handedness of the optical rotation in the polarized light

Note 1 to entry: Right-handed quartz is the crystal of dextrorotatory and left-handed quartz is the crystal of levorotatory

3.28**twins**

two or more same single crystals which are combined together by the low of symmetrical plane or axis

Note 1 to entry: The following twin types have been identified in synthetic quartz crystals:

a) Electrical twins

Quartz crystal in which regions with the common Z-axis exist showing a polarity reversal of the electrical X-axis.

b) Optical twins

Quartz crystal in which regions with the common Z-axis exhibit handedness reversal of the optical Z-axis.

3.29**infrared absorption coefficient α value**

coefficient (referred to as the α value) established by determining the relationship between absorption of two wave numbers

Note 1 to entry: One wave number is minimal absorption due to OH impurity, the other is high absorption due to presence of OH impurities in the crystal lattice. The OH impurity creates mechanical loss in resonators and its presence is correlated to the presence of other loss-inducing impurities. The α value is a measure of OH concentration and is correlated with expected mechanical losses due to material impurities.

Note 2 to entry: For the coefficient defined here, the logarithm base 10 is used. The infrared absorption coefficient value α is determined using the following equation:

$$\alpha = \frac{1}{t} \log_{10} \left(\frac{T_1}{T_2} \right)$$

where

α is the infrared absorption coefficient;

t is the thickness of Y-cut sample, in cm;

T_1 is the per cent transmission at a wave number of 3 800 cm⁻¹ or 3 979 cm⁻¹;

T_2 is the per cent transmission at a wave number of 3 500 cm⁻¹, or 3 585 cm⁻¹.

3.30**lumbered synthetic quartz crystal**

synthetic quartz crystal whose X- and Z- or Z'- surfaces in the as-grown condition have been processed flat and parallel by sawing, grinding, lapping, etc., to meet specified dimensions and orientation

3.31**reference surface**

surface of the lumbered bar prepared to specific flatness and orientation with respect to a crystallographic direction (typically the X-direction)

3.32**synthetic quartz for optical applications**

synthetic quartz which satisfies the requirements for the use of optical pickups, optical lowpass filters (OLPF) and wave plates for digital single-lens reflex camera, monitoring camera, digital video camera and optical communication module operating in the 300 nm to 1 700 nm wave length (5 882 cm⁻¹ to 33 333 cm⁻¹)

3.33**internal transmittance**

internal transmittance which does not include loss of surface refraction

Note 1 to entry: This definition applies to synthetic quartz for optical applications only. Internal transmission values require statement of sample thickness for which the value is calculated, e.g. 2 mm.

3.34**striae**

short range deviations of refractive index in quartz, growing defects in which the refractive index fluctuates with a typical period of fractions of one millimetre to several millimetres

Note 1 to entry: This definition applies to synthetic quartz for optical applications only.

3.35**growth band**

contrasting density band that can be observed in the Y-cut crystal by Schlieren and similar optical method

Note 1 to entry: The cause of this contrast is that elements such as aluminium, sodium, lithium and iron are trapped in the crystal when a crystal is growing.

Note 2 to entry: Larger amounts of trapped impurities typically cause an increase in α .

Note 3 to entry: Growth bands cannot be observed when $\alpha_{3\ 585}$ is less than 0,160 or $\alpha_{3\ 500}$ is less than 0,120.

Note 4 to entry: This definition applies to synthetic quartz for optical applications only.

4 Specification for synthetic quartz crystal**4.1 Standard values****4.1.1 Shape of synthetic quartz for optical applications**

A seed crystal is removed and the growth faces are machined to a specified uniform surface roughness and to a specified flatness with the specified crystallographic orientation.

4.1.2 Orientation of the seed

Standard orientation for the seeds are Z-cuts and rotated X-cuts, minor rhombohedral (z-minor) cut, 1°30' rotated Z-cut, 2° rotated Z-cut, 5° rotated Z-cut, and 8°30' rotated Z-cut, the Z'-axis of the latter three seeds being rotated as shown in Figure 1.

4.1.3 Inclusion density**4.1.3.1 Inclusion density of synthetic quartz for piezoelectric applications**

The inclusion density (measured as in 4.2.5.3) for each grade shall not exceed the figures in any required size range for that grade listed in Table 1.

Table 1 – Inclusion density grades for piezoelectric applications

Grade/size Range μm	Densities per cm^3			
	10-30	>30 to 70	>70 to 100	>100
I a	2	1	0	0
I b	3	2	1	1
I	6	4	2	2
II	9	5	4	3
III	12	8	6	4

Users requiring a grade in only one or more of the size ranges may designate their requirement as the grade followed by the appropriate size range.

4.1.3.2 Inclusion density of synthetic quartz for optical applications

The inclusion density grade of synthetic quartz for optical applications shall be shown at Table 2.

Table 2 – Inclusion density grades for optical applications

Grade/size Range μm	Densities per 100 cm^3
	10 to 100
OPT I	0 to 9
OPT II	10 to 20
OPT III	21 to 39

4.1.4 Striae in synthetic quartz for optical applications

A size, contrasting density and quantity should not exceed a limit sample. A limit sample should be defined between the manufacturer and the user.

4.1.5 Infrared quality indications of $\alpha_{3\,500}$ and $\alpha_{3\,585}$ for piezoelectric applications

An infrared extinction coefficient value (α value) of synthetic quartz (measured as in 4.2.6) shall be as listed under the appropriate heading for $\alpha_{3\,500}$ or $\alpha_{3\,585}$ in Table 3 for the various grades:

Table 3 – Infrared absorbance coefficient grades for piezoelectric applications

Grade	Maxima		Pre-1987 ^a Q · 10 ⁶ units
	$\alpha_{3\,500}$	$\alpha_{3\,585}$	
Aa	0,026	0,015	3,8
A	0,033	0,024	3,0
B	0,045	0,050	2,4
C	0,060	0,069	1,8
D	0,080	0,100	1,4
E	0,120	0,160	1,0

^a These Q values were obtained from α measurements and empirical correlation, and were in common usage prior to 1987. These are included here as the previous labels to maintain continuity through the change in emphasizing α labels. α is the physical measurement now used to control and specify quality in synthetic quartz.

The test limits above either correspond to or are unchanged (except in the cases of grades B and D) from the $\alpha_{3\ 500}$ limits that correspond to the Q value grades listed IEC 60758:1983. This fifth edition of IEC 60758 designated some of the same grades in terms of minimum indicated Q's in 10^6 units, as follows:

Aa = 3,8;

A = 3,0;

B = 2,2 (basis used herein), changed from 2,4 in the 1983 edition;

C = 1,8;

D = 1,4 (revised);

E = 1,0 (the same as the earlier D-grade).

4.1.6 Grade classification by α value and Schlieren method for optical applications

Grade classification is shown at Table 4.

Table 4 – Infrared absorbance coefficient grades and Schlieren method for optical applications

Grade	$\alpha_{3\ 500}$	$\alpha_{3\ 585}$	Schlieren method
OPT A	<0,033	<0,024	–
OPT B	<0,060	<0,069	–
OPT C	<0,120	<0,160	–
OPT D	$\geq 0,120$	$\geq 0,160$	growthband not observed by Schlieren method

4.1.7 Frequency-temperature characteristics of synthetic quartz for piezoelectric applications

The frequency-temperature characteristics of synthetic quartz crystal units shall be assessed by determination of the fractional frequency deviation measured at 15 °C and 35 °C with respect to the series resonance frequency at 25 °C. The fractional deviation shall satisfy the following:

- fractional frequency deviation at 15 °C: $+0,5$ to $+1,5 \times 10^{-6}$;
- fractional frequency deviation at 35 °C: $-0,5$ to $-1,5 \times 10^{-6}$.

Measurement shall be made in accordance with 4.7.3 of IEC 60122-1:2002.

4.1.8 Etch channel density ρ

4.1.8.1 Etch channel density ρ for piezoelectric applications

When required, the etch channel density, ρ , per cm^2 (measured as in 4.2.8) for each grade, shall comply with the listings in Table 3.

Table 5 – Etch channel density grades for piezoelectric applications

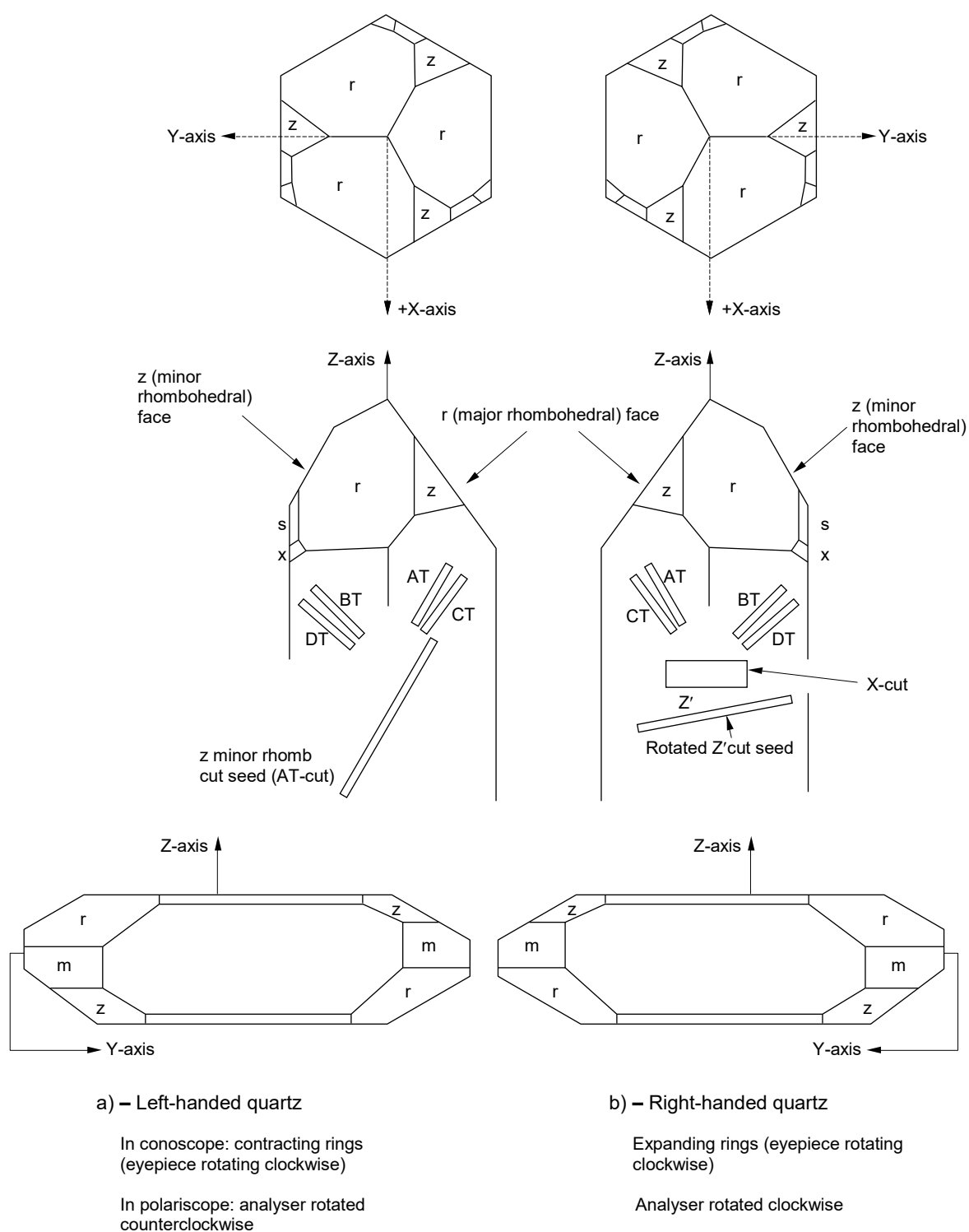
Grade	Maximum number ρ per cm^2
1aa	2
1a	5
1	10
2	30
3	100
4	300

4.1.8.2 Etch channel density ρ for optical applications

Etch channel density (measured as in 4.2.8) should be $\rho \leq 100$ per cm^2 .

4.1.9 Internal transmittance for optical applications

At the wavelengths 400 nm, 550 nm, 650 nm and 1 550 nm, internal transmittance should be 0,998 or more for a 2 mm thick sample.



IEC

Figure 1 – Quartz crystal axis and cut direction

4.2 Requirements and measuring methods

4.2.1 Orientation

The orientation of the seed shall be along specified directions, with a deviation of less than 30 min from nominal.

4.2.2 Handedness

The handedness of the seed shall be specified, either right-hand or left-hand (see Figure 1).

4.2.3 Synthetic quartz crystal dimensions

4.2.3.1 General

The dimension shall be measured by callipers or point callipers which enable the hollow point of a synthetic quartz crystal to be measured (see Annex D).

4.2.3.2 Dimension along Y or Y'-axis

The dimension shall be as specified (see Figure 2d).

4.2.3.3 Dimension along Z or Z'-axis

The dimension along the Z or Z'-axis shall be measured by a point calliper and it shall be specified as the maximum dimension along the Z or Z'-axis in the greater X zone (see Figure 2c).

4.2.3.4 Dimension Z_{eff} or Z'_{eff}

The Z_{eff} or Z'_{eff} dimension shall be specified as the minimum dimension along the Z or Z'-axis (see Figure 2c).

4.2.3.5 Dimension Z_{min} or Z'_{min}

The dimension shall be as specified (see Figures 2c and 2d).

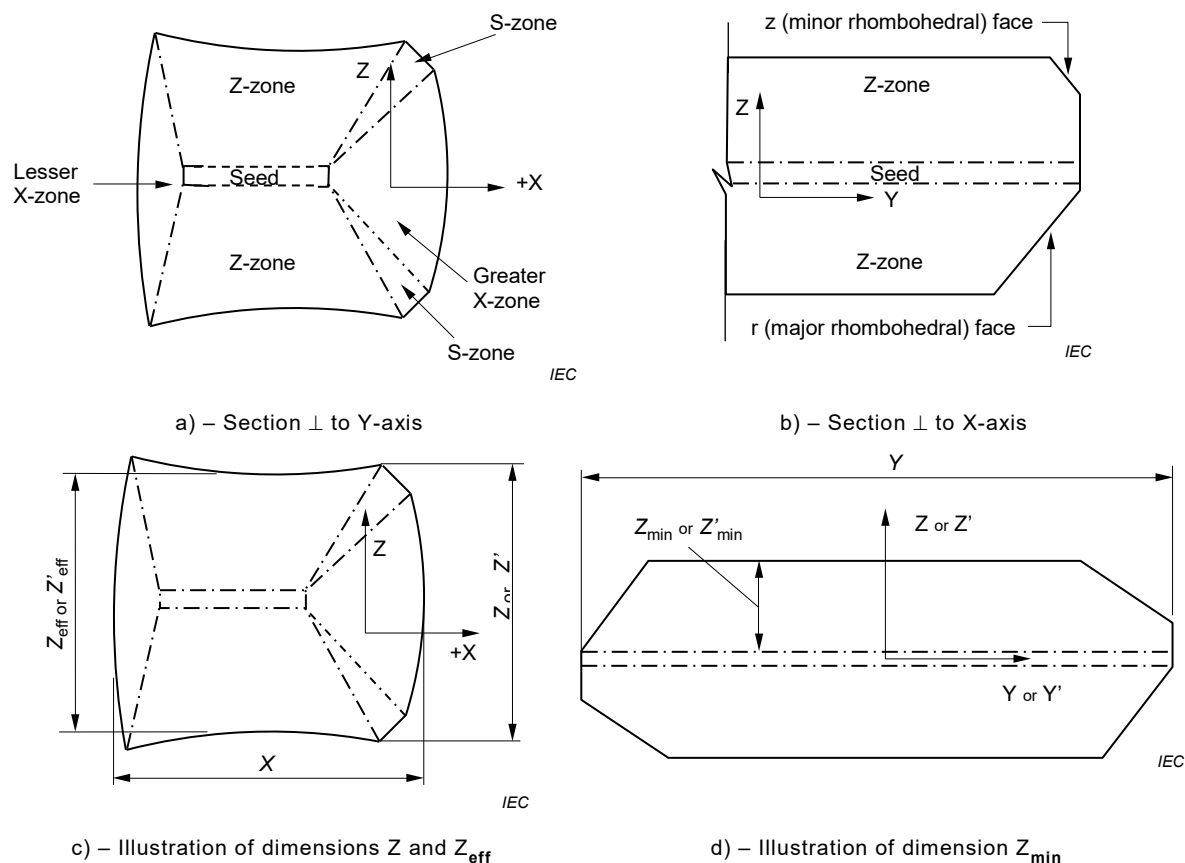


Figure 2 – Idealized sections of a synthetic quartz crystal grown on a Z-cut seed

4.2.3.6 Dimension along X-axis

The gross dimension along the X-axis shall be as specified (see Figure 2c).

4.2.4 Seed dimensions

4.2.4.1 Z or Z' dimension

The Z or Z'-dimension (i.e. thickness) of the Z-cut or rotated Z-cut seed shall be less than 3 mm, unless otherwise specified.

4.2.4.2 X-dimension

The dimension X of the seed shall be as specified.

4.2.5 Imperfections

4.2.5.1 Twinning

There shall be no electrical or optical twinning in the usable region. The existence of twinning shall be checked by visual inspection.

4.2.5.2 Cracks and fractures

There shall be no cracks or fractures in the usable region. The existence of cracks and fractures shall be checked by visual inspection.

4.2.5.3 Inclusion density

4.2.5.3.1 General

The following two measuring methods are used and either one may be chosen:

a) Method 1

Inclusions within stated ranges are counted visually per cm^3 in sample volumes within a crystal using a stereo binocular microscope operating at 30× to 40× magnification equipped for counting within either a circular or a square field and with a calibrated reticle scale for determining particle sizes, intense side illumination (such as halogen lamps) over a recessed black matt background, an index matching liquid ($n = 1,55$, approximately) for transparency, and means of measuring the dimensions of the sample volumes counted. An example for the reference sample selection procedure is given in Annex B.

b) Method 2

In case it is difficult to apply method 1, crystals are compared with reference samples appropriately representing each grade range, immersing within an index matching liquid ($n = 1,55$ approximately) for transparency, or applying such liquid to the surface. The reference samples shall be agreed upon between the supplier and the user. An example for the reference sample selection procedure is given in Annex C.

4.2.5.3.2 Sampling

Because of the considerable costs in time, labour and money, some plan for sampling both bars and regions within the bars is normally used by agreement between the supplier and the buyer when quality control of either inclusion density or etch channel density is required.

Clearly, the preferable low-cost inspection situation is the one in which the densities of inclusions or etch channels are well below the test limits, and infrequent samples can be justified. Since such situations are not always attainable, more rigorous inspection strategies will sometimes be required for appropriate density control, and shall be found, worked out, and agreed upon between the supplier and the user.

Sound statistical methods are required in order to meet appropriate agreed-upon assured quality level tests and ensure that the crystals and the volumes counted within them are sufficiently representative. Since sampling procedures and statistical confidence tests are described in the literature, their principles will not be repeated here.

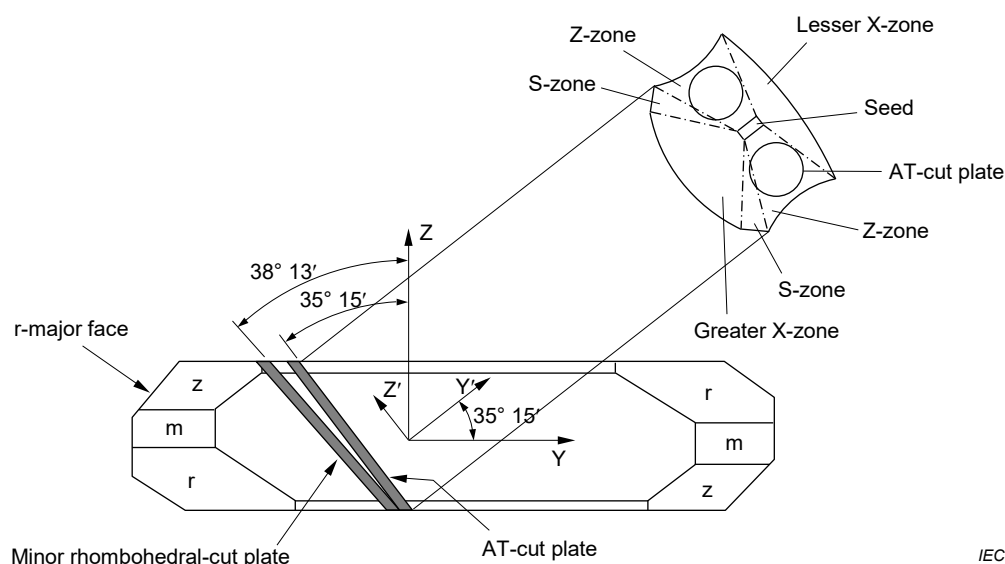
4.2.5.3.3 Batch sampling

In most batch sampling, a suitable sample bar or group of bars is chosen to represent the batch population. The number of bars shall depend on the number in the batch, the type of crystal, the intended application, the separation between the mean and the target inclusion densities and the AQL (acceptable quality level) confidence level requirement needed to provide sufficient assurance that the batch inclusion density in each size range shall be below their applicable grade test limits. The sample bar group shall reasonably represent the batch with respect to inclusion densities. Deviations, if any, are allowed and shall be towards higher, not lower, inclusion densities for safe assurance.

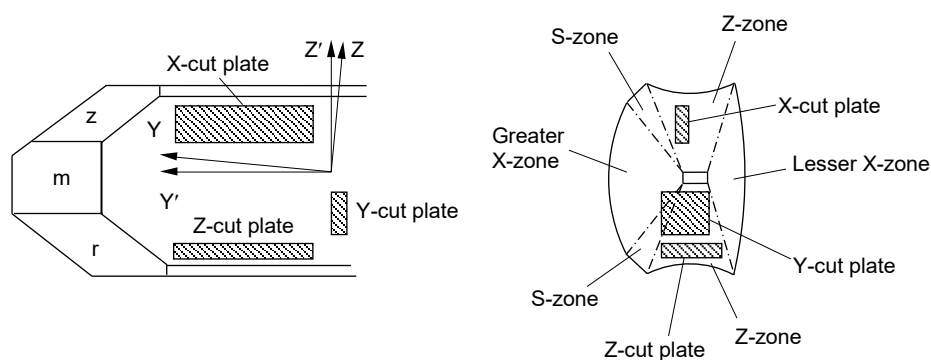
4.2.5.3.4 Volumes within a bar

A group of volumes within each sample bar is next chosen for inclusion counting. The boundaries of the volumes are defined by the area of the focal field of the microscope (or the outline of a square reticle) and either the height of the bar or the length range of the depth adjustment of the microscope chosen for use. It is necessary to determine and total the volumes throughout which counts are accumulated. The volumes selected for counting should include mainly regions (usually Z-growth zones as in Figure 3) whose material will be present and active in the finished devices and should not avoid dense inclusion volumes within these regions. The number of volumes per bar shall be at least six or more for reasonable statistical confidence.

The sample volume locations within a bar shall be appropriately distributed in its X-, Y-, Z-axes to include the variations of the inclusion density with these independent variables. Typical synthetic quartz bars (Figure 3a) are long in the Y- and small in the X- and Z-axes dimensions. Normally, the greatest variation of inclusion density appears over a zone's grown direction, for example the Z-dimension in the Z-zone (Figures 1a and 3). Thus, for large Z-crystals, the sampled regions shall be located at varied Z-distances from the seed to ensure that the bar's range of Z is well-represented by the group of sample volumes. Similarly, any noted variations over Y or X shall be sampled, if such variations are present.



a) – Location of AT-cut plate in right-handed synthetic



b) – Location of X-cut plate, Y-cut plate and Z-cut plate

Figure 3 – Typical example of cutting wafers of AT-cut plate, minor rhombohedral-cut plate, X-cut plate, Y-cut plate and Z-cut plate

To aid in distributing sample volumes within a typical bar, its lesser X-surface is marked with transverse Z'-lines, perpendicular to the seed at regular 10 mm intervals over the Y-length of the surface. Sample volumes for inclusion counting are chosen as needed from within each rectangle formed by the marks and the crystal surfaces. To locate the sample volumes at varied distances from the seed, in small crystals (where a Z-zone measures less than double the field diameter of the microscope), they should be alternated near and far from the seed. For larger crystals, the volumes should be sequenced in Z over its range to ensure that each inclusion band is represented in the sample volumes. Several frequently used sampling plans are illustrated in Annex A.

4.2.5.3.5 Inclusion counting

The circular or square field of view chosen for counting within its marked rectangle is scanned vertically over its chosen X-height within the microscope's range of depth adjustment, as follows.

If the sample is a Y-bar with a relatively small X-height, the scans will be at a series of sites varied in Z along its length (under side illumination, with its lesser X-surface up). Beginning in a rectangle positioned at one end of the usable zone, an inclusion count is taken in an X-cylinder (or parallelepiped) volume. Starting slightly below the lesser X-surface (and not counting surface material), all visible inclusions in focus are categorized and counted in each of the size categories required by the customer's order: 10 μm to 30 μm , 30 μm to 70 μm , 70 μm to 100 μm , and greater than 100 μm . The microscope is then lowered and the newly focused inclusions counted and added into their size categories. This process is continued through the chosen X-height; the procedure is repeated at the next sample position, and so on.

The counts from the bar's sample sites in each of the four size categories are summed by category and divided by the calculated total of the sampled volumes, to obtain an average count per cm^3 for each category in one bar. The count averages in each category from all the sample bars from a run are averaged and recorded as required to represent the size distribution for the run. Maximum and minimum bar averages may also be recorded, if desired or required. A numerical example is given in Annex B.

4.2.6 Evaluation of infrared quality by α measurement

4.2.6.1 General

The infrared absorption per centimetre at one or more of 3 500 or 3 585 wave numbers is measured in a Y-cut slice scan as the difference between the absorption at the chosen wave number and absorption in the background outside the band, at 3 800 cm^{-1} or at 3 979 cm^{-1} when using a single beam instrument. Since those α are known to vary directly with the total Z-growth size distribution of the crystals in one batch, a maximum Z-crystal (for worst-case maximum α measurement) is used to characterize the batch. Other crystals, the average Z-crystal for example, may be measured to further characterize the batch.

Two methods have been used for IR measurements in quartz. The first was dispersive, a set-up in which a monochromator results in the exposure of the test sample to a single beam spectrum of specified wavelength (wave number). Later, Fourier transform infrared spectrophotometers (FTIR) were developed allowing all relevant wavelengths to be sampled simultaneously. This method uses an interferometer to collect transmission across a wide spectrum. The detector signal is sent to a computer integrated into the test setup, and an algorithm called a Fourier transform is performed on the interferogram to convert it into a single beam spectrum, with the transmission data so obtained used to estimate the alpha extinction coefficient. Today, the FTIR is used more widely than dispersive IR equipment.

Both the dispersive Infrared Spectrometer and FTIR methods may be used to measure the whole sample at a single, fixed wave number. In this option, the sample is scanned about the Z direction using a narrow beam to measure infrared absorption point-by-point, then constructing a map of absorption versus Z-position.

4.2.6.2 Preparation of the Y cut slice

The synthetic quartz crystal to be sampled is mounted on a substrate then sliced with a quartz saw to yield at least one Y-cut slice whose thickness after lapping and polishing will fall in the range of 5 mm to 10 mm. The 5 mm thickness is appropriate for high α -material, to resolve its α -variations; the mid-range for medium α ; and the 10 mm thickness is appropriate for the lowest α -material to measure its small absorption.

After sawing, the slice is lapped on both major surfaces: first, with a homogenized mixture of 25 µm abrasive; second, with a homogenized mixture of 3 µm abrasive. Further lapping to polish is optional and is preferred for low α .

4.2.6.3 Calibration of a standard Y-cut slice in an infrared spectrophotometer

The infra-red spectrophotometer is turned on, allowed to warm and fully stabilize, then calibrated. The normal daily calibration includes its transmission (0 % to 100 %) or absorbance ($\log T = 1,0$ to 0) limit settings, chart speed and synchronized sample scanning arrangement. For evaluation and normal use, a 1,5 mm width aperture is located in the sample beam. For the lowest α value measurements, a 5 mm width aperture may be required. The aperture's height shall not exceed the X-dimension of the seed or 5,0 mm. A polished Y-cut standard reference slice is placed first in the sample holder, which is then mounted in the scanning device.

The wave number control is set at a background setting (outside, but near the OH absorption band), usually $(3\,800 \pm 3) \text{ cm}^{-1}$, and the sample is translated through the beam with synchronized chart advance at the fixed wave number. Such scanning is done only in the Z-growth zones of the Y-cut slice (illustrated in Figures 1 and 3). In certain cases where background noise may be a problem, such as single beam operation, a higher background wave number $(3\,979 \pm 3) \text{ cm}^{-1}$ may be used for lowered background noise. If the background scan trace is not reasonably flat outside the original seed's boundaries, a thin film of fluorolube grease shall be applied to both major surfaces of the semi-polished Y-cut slice. Baseline changes shall not exceed 1,0 percent absorption units during a background scan. After the sample has completed a successful scan at background, the wave number is adjusted to the chosen $(3\,500 \pm 3) \text{ cm}^{-1}$ or $(3\,585 \pm 3) \text{ cm}^{-1}$. The sample is returned to its original position and the chart paper rerolled to the position where its wave number scan began. The sample is then scanned to plot its infra-red absorption at this wave number in the absorption band.

The calibration α values (maximum and minimum) are calculated from this reference scan, using the equation below:

$$\alpha = \frac{\log_{10} I_1 - \log_{10} I_2}{t}$$

where

I_1 is the absorbance measured at the chosen reference wave number of $3\,800 \text{ cm}^{-1}$ or $3\,979 \text{ cm}^{-1}$,

I_2 is the absorbance measured at the chosen wave number of $3\,500 \text{ cm}^{-1}$ or $3\,585 \text{ cm}^{-1}$,

t is the thickness of the measurement sample (cm).

NOTE The absorbance is calculated from the sample transmission T using the logarithm base 10 according to $A = \log_{10}(1/T)$. To convert this value to linear absorption coefficient, as defined in IEC 60050-845:1987, 845-04-78, corresponding to the increase in absorption coefficient at the specified wavelength, α is multiplied with a factor $100 \cdot \ln(10) = 230,3$. The factor 100 converts from cm^{-1} to m^{-1} and $\ln(10) = 1/\log_{10}(e)$ from base 10 to e .

The spectrophotometer is considered in proper calibration if its $\alpha_{\max}$ and $\alpha_{\min}$ readings are repeatable within $\pm 0,004$ units of the standard's values for them. A standardization correction may be calculated as needed to bring the instrument's reading on a standard slice to an accepted value and used while current.

4.2.6.4 Test measurement of a Y-cut slice

After successful calibration, each prepared (preferably polished) unknown slice is scanned at the background and chosen OH absorption band wave numbers, using a thin film of oil as needed in cases where there is only a semi-polish. Their pertinent α values are calculated using the equation above. Regions excluded from this determination are $\pm 2,0$ mm from the seed centre and the excess growth beyond the appropriate pre-dimensioned bar diameter.

4.2.6.5 Compensation of α value by standard sample

Correlation between the test equipment of each manufacturer cannot be assured by strict adherence to uniform measuring conditions and procedures. Therefore, it is necessary to establish a compensation value for α . The recommended compensation value is determined by each manufacturer referencing the procedure described in Annex E.

4.2.7 Frequency versus temperature characteristics for piezoelectric applications

The specifications for the quartz crystal unit for evaluating the frequency-versus- temperature characteristics shall be as follows, and measurements shall be made as specified in IEC 60122-1:

- frequency 10 MHz $\pm$ 10 kHz (fundamental);
- location of specimen Z-zone;
- orientation of plate AT cut 35° 15' $\pm$ 30";
- shape of plate square flat and parallel plate with one edge along the X-axis;
- lateral dimensions of plate 8 mm $\times$ 8 mm;
- diameter of electrodes 4 $\pm$ 0,1 mm;
- plate back of frequency deviation 70 +20/-0 kHz;
- electrode material silver or gold;
- supporting points at two points on opposite corners;
- finish of surface lapped (average particle size of abrasive shall be less than 3 μ m), then etched by 200 kHz;
- flatness of surface when illuminated with monochromatic light through an optical flat glass and examined within a circular zone of 6 mm diameter, the product of the fringe curvature and the frequency expressed in MHz shall not exceed 5 if measured with yellow light or 6 if measured with green light;
- parallelism both surfaces of the plate shall be parallel within 10";
- seal hermetically enclosed in a dry nitrogen atmosphere at standard atmospheric conditions for temperature and pressure (see 4.1 of IEC 60068-1:2013).

The specification shall state the minimum and maximum slope of frequency-temperature characteristics (Figure 4).

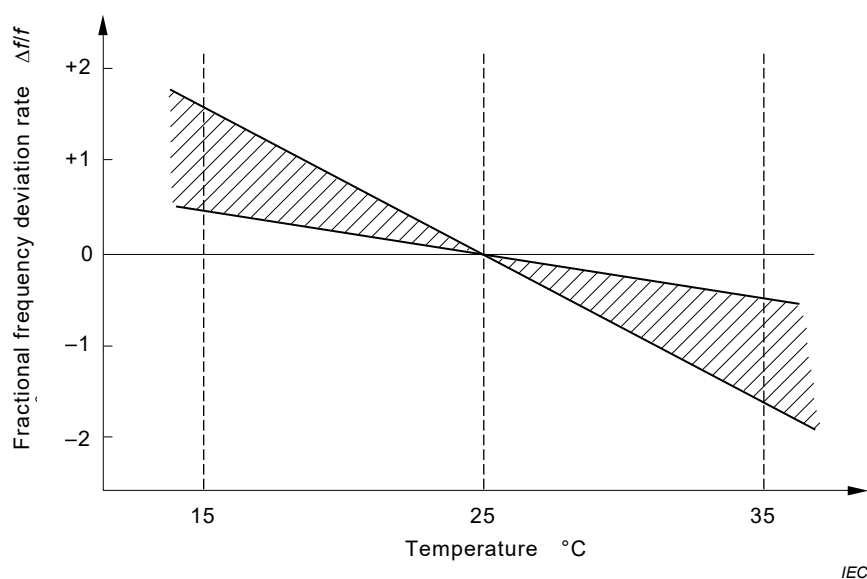


Figure 4 – Frequency-temperature characteristics deviation rate of the test specimen

4.2.8 Striae in synthetic quartz for optical applications

Striae in lumbered quartz crystal are observed in the white light with the liquid of which refractive index is almost the same as that of quartz, such as silicon oil. Lumbered quartz crystal is positioned between the cross nicol polarizers and striae can be observed from Z direction. The quantity, size and colour strength of the striae should be compared with those limit samples. They can be also observed in the polished Y-cut sample of several millimetres thickness by using schlieren or similar equipment. Typical optical set-up for testing of schlieren is shown at Figure 5.

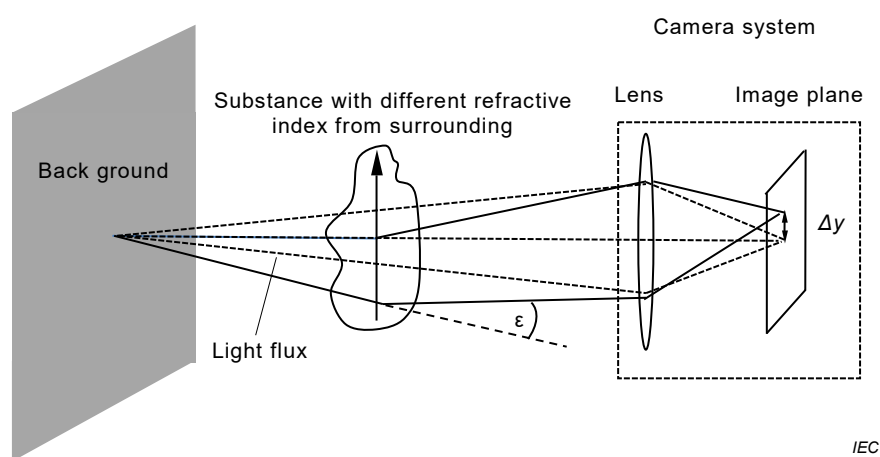


Figure 5 – Typical schlieren system setup

4.2.9 Growth band in synthetic quartz for optical applications

Sample's cutting direction is perpendicular to Y axis and its both Y face should be polished. By using schlieren or similar equipment growth band should be observed.

4.2.10 Etch channel density

4.2.10.1 General

The etch channel density is measured by counting channels in an etched AT-cut sample slice of the sampled quartz crystal as seen under a binocular microscope at up to 35× magnification.

4.2.10.2 Sampling the crystals from a batch

Sampling a batch to achieve a specified statistical confidence level requires the use of AQL confidence statistics appropriate in this, the etch channel case, because the etch channel population depends on both the channels in the seeds and the process that grows the material on the seeds. It is preferable to pre-sort and group seeds for their etch channel densities and record their group locations in autoclaves or ensure that there is an adequate sample of the seeds as well as the growth on them, because of the possibility that some of the seeds may differ greatly from the others, unless intentionally controlled during planting.

4.2.10.3 Preparation of AT cut slice for etching

A sample synthetic quartz crystal is cut to yield an AT-cut slice (at $35,25^\circ \pm 3^\circ$ to the Y-plane) of a thickness to finish at 10 mm. The slice should preferably contain full seed height, although thinner slices and lesser seed heights may be used in special cases, provided the specified amount of material is removed in each lapping. The slice's identity should be marked with a diamond scribe, preferably on its lesser X-surface, to an adequate depth to ensure that the identity will not be lost in lapping and etching.

After sawing and marking, the slice is lapped on both major surfaces; first, with a homogenized mixture of 25 µm abrasive, then on a cleaned lap with a homogenized mixture of 11,5 µm abrasive. A minimum of 0,25 mm total thickness of quartz shall be removed in the first abrasive lapping, and of 0,10 mm total thickness in the second abrasive lapping. The slice is then cleaned to ensure a uniform etching rate over its surfaces, as evidenced by its uniform matt appearance after etching.

4.2.10.4 Etching procedure for lapped AT-cut slice

Within a fume hood, using appropriate safety equipment (including apron, gloves, eyeglasses, and splatter shields), an excess of ammonium bifluoride is added to deionized water in a suitable container to make a saturated solution at $75^\circ\text{C} \pm 2^\circ\text{C}$. The ammonium bifluoride is maintained at the $75^\circ\text{C} \pm 2^\circ\text{C}$ temperature, with the use of a constant temperature liquid bath. A polytetrafluoroethylene (PTFE)-coated thermometer in the solution is used for monitoring the temperature. If different etch temperatures are preferred, the etch time will be adjusted appropriately to comply with the stock removal requirement below.

The lapped and cleaned AT-cut slice is immersed into the saturated ammonium bifluoride solution using PTFE-coated tongs or an inert etching slice holder. The solution should be agitated or the sample slowly moved (approximately 25 mm per second) during the etching process. An etching time of about 4 h should be sufficient, depending on the agitation and the number of slices, to remove not less than 0,10 mm (minimum) or more than 0,12 mm (maximum) total thickness. Uniform etch rate is a requirement. The quartz slice shall be checked periodically for etch uniformity both in time and over the area of the slice. After etching is completed, the slice is removed from the etch solution with the PTFE-coated tongs, rinsed with hot water (85°C), then cleaned with acetone and other cleaning agents, if needed, to prepare the sample for unobstructed visual inspection.

4.2.10.5 Etch channel counting procedure

A square grid of known dimensions in the range of 0,25 cm × 0,25 cm to 0,50 cm × 0,50 cm is marked on the AT-cut slice within the Z-growth zone as identified in Figure 3a. If preferred, the grid pattern may be drawn only over the rectangular portions of the Z-zones matching the

height of the seed. No grid squares should be counted that fall within ± 2 mm of the centre line of the seed.

Each slice is viewed with a binocular microscope using 30× to 40× magnification, and a strong light source directed into the slice in its lesser X-direction. Care shall be taken to focus the microscope on the upper surface of the sample, thus avoiding counting the etch channel intersections with the lower sample surface. The etch channels are counted and recorded in each and every square area with the microscope's zoom adjustment set for a convenient viewing magnification. After all the grid areas intended for counting are counted, their average count is calculated, and converted to an average per cm^2 , ρ , by multiplying by 16 for $0,25 \text{ cm} \times 0,25 \text{ cm}$, or 4 for $0,50 \text{ cm} \times 0,50 \text{ cm}$, etc., the number of grid squares in 1 cm^2 .

4.2.11 Internal transmittance for optical applications

Internal transmittance is measured by the following procedure.

- Two samples are cut from the same quartz bar;
- Direction of cutting is perpendicular to Y-axis $\pm 60^\circ$;
- Thickness of samples are 5 mm and 10 mm;
- Both Y-face of two samples is mirror polished;
- Transmission is measured through both samples using a spectrophotometer at the wavelengths 400 nm, 550 nm, 650 nm, and 1 550 nm. Internal transmittance is measured by a spectrophotometer at wavelength 400 nm, 550 nm, 650 nm and 1 550 nm.
- The internal transmittance for a 5 mm long sample is calculated using the following formula:

$$\tau = \frac{T_1}{T_2}$$

where

τ internal transmittance for sample thickness 5 mm;

T_1 transmittance including surface refraction loss of 5 mm sample;

T_2 transmittance including surface refraction loss of 10 mm sample;

Internal transmittance values for other thicknesses can be calculated using the following formula:

$$\tau(t) = \exp\left(\frac{t}{5 \text{ mm}} \times \ln(\tau)\right)$$

where

$\ln$ the natural log;

$\exp$ the natural exponent function;

5 mm the thickness for which τ was originally calculated;

t the thickness of sample for which the internal transmittance is calculated (e.g. 2 mm).

4.3 Marking

4.3.1 General

Each synthetic quartz crystal shall have the following information clearly marked on a major or lesser X-surface:

- manufacturer's name or trade mark;
- orientation of material;

- c) handedness of material;
RH stands for right-handed quartz;
LH stands for left-handed quartz.
- d) other information, if specified, to be marked such as;
 - 1) batch identification;
 - 2) α grade;
for piezoelectric applications: Aa, A, B, C, D or E;
for optical applications: OPT A, OPT B, OPT C or OPT D.
 - 3) inclusion density;
for piezoelectric applications: Ia, Ib, I, II or III;
for optical applications: OPT I, OPT II or OPT III.
 - 4) etch channel density;
for piezoelectric applications: 1aa,1a,1, 2, 3 or 4;
for optical applications: 1aa,1a,1, 2 or 3.

4.3.2 Shipping requirements

These requirements shall be specified upon agreement between the supplier and the user.

5 Specification for lumbered synthetic quartz crystal

5.1 Standard values

5.1.1 Tolerance of dimensions

Deviations from the specified dimensions along the X- and Z- or Z'- axes shall be less than 0,2 mm (see Figure 6).

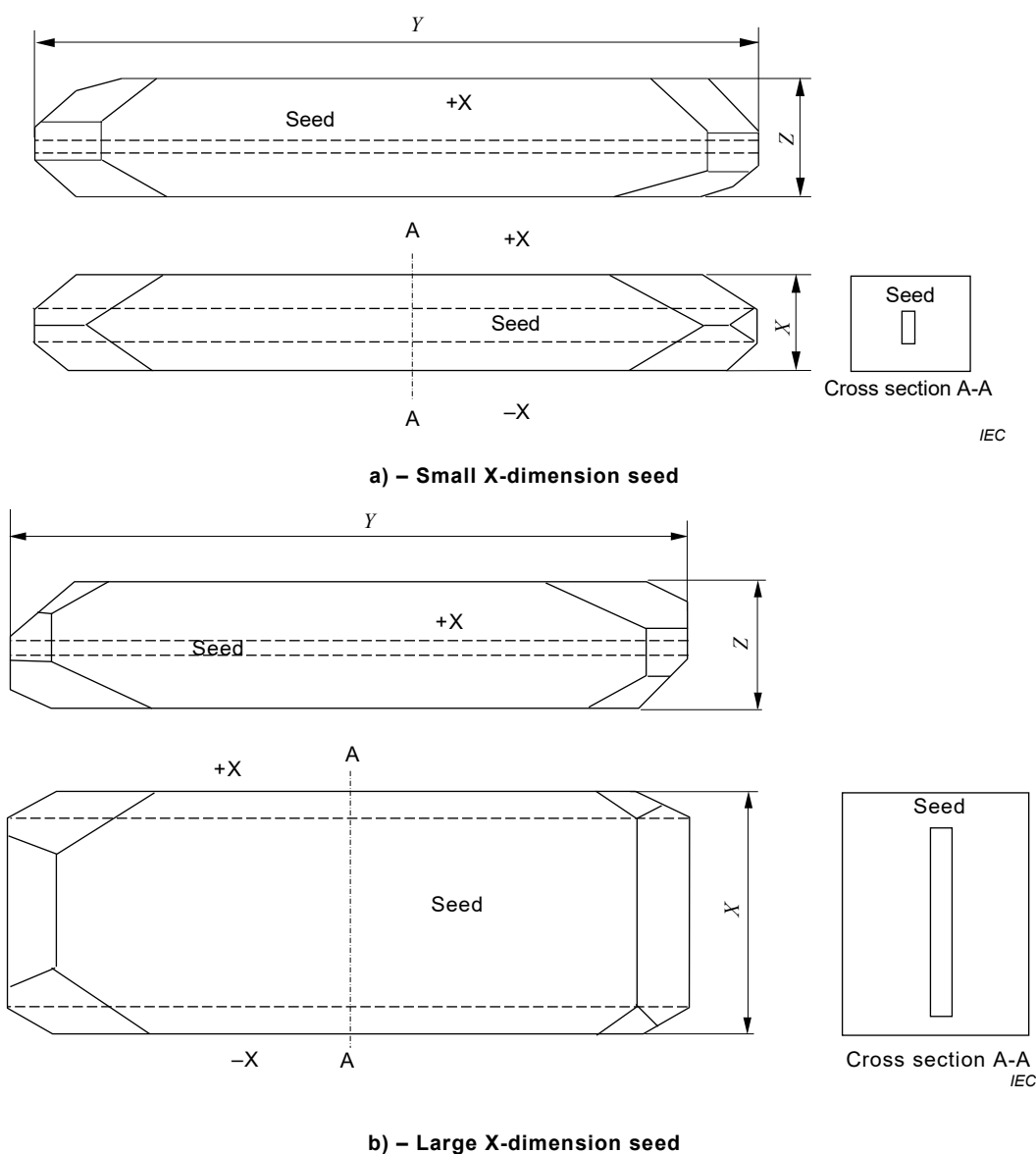


Figure 6 – Lumbered synthetic quartz crystal outline and dimensions along X-, Y- and Z-axes

5.1.2 Reference surface flatness

Reference surface shall be flat to within 0,2 mm or as specified. Care should be exercised in the selection of the method of measurement to isolate measures of reference surface deviations from those of the opposite side (parallelism).

5.1.3 Angular tolerance of reference surface

Angular deviations of the reference surface shall be less than 15' from the specified crystallographic direction (see Figure 7).

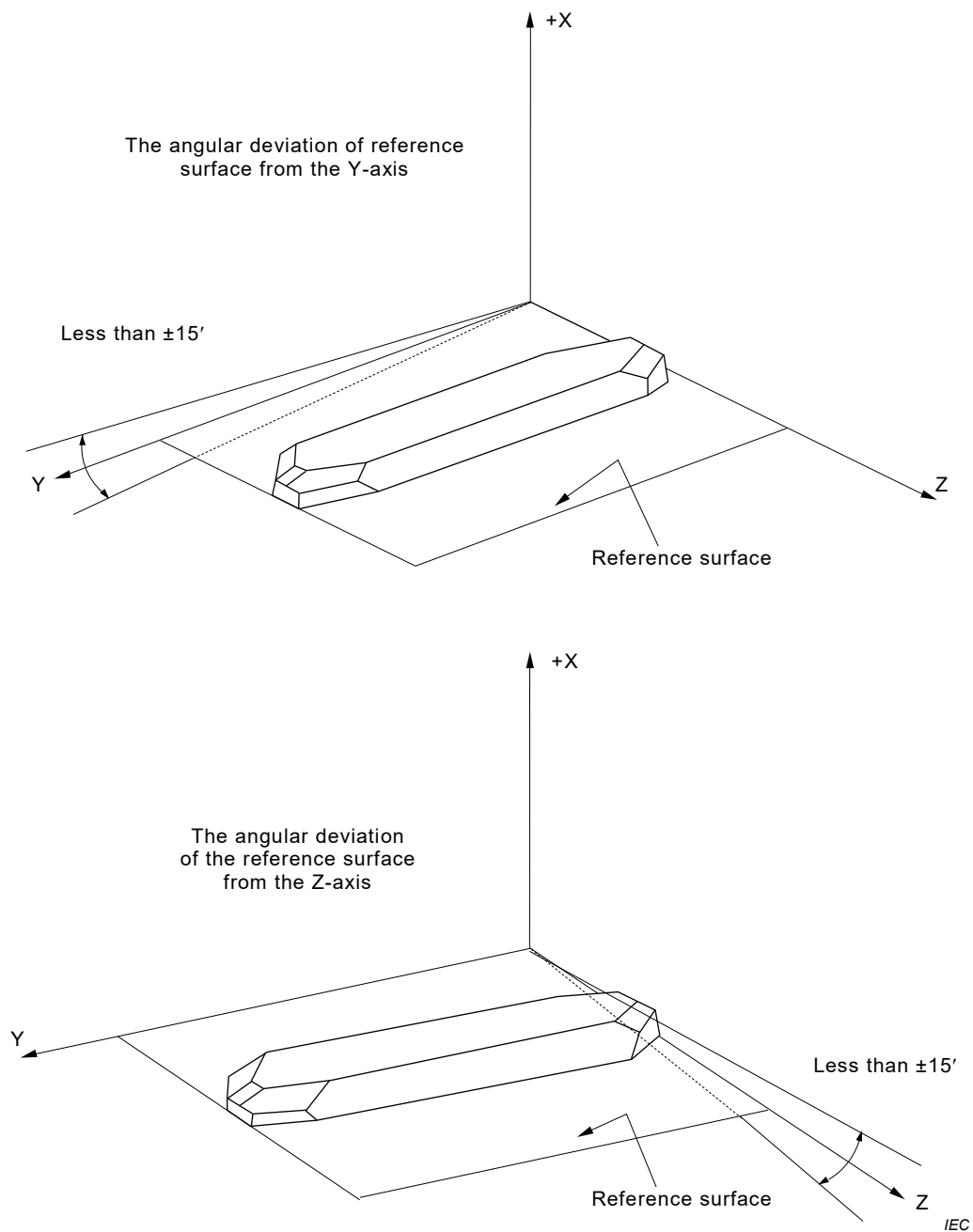


Figure 7 – Angular deviation for reference surface

5.1.4 Centrality of the seed

Seed position in lumbered synthetic quartz crystal is shown in Figure 8.

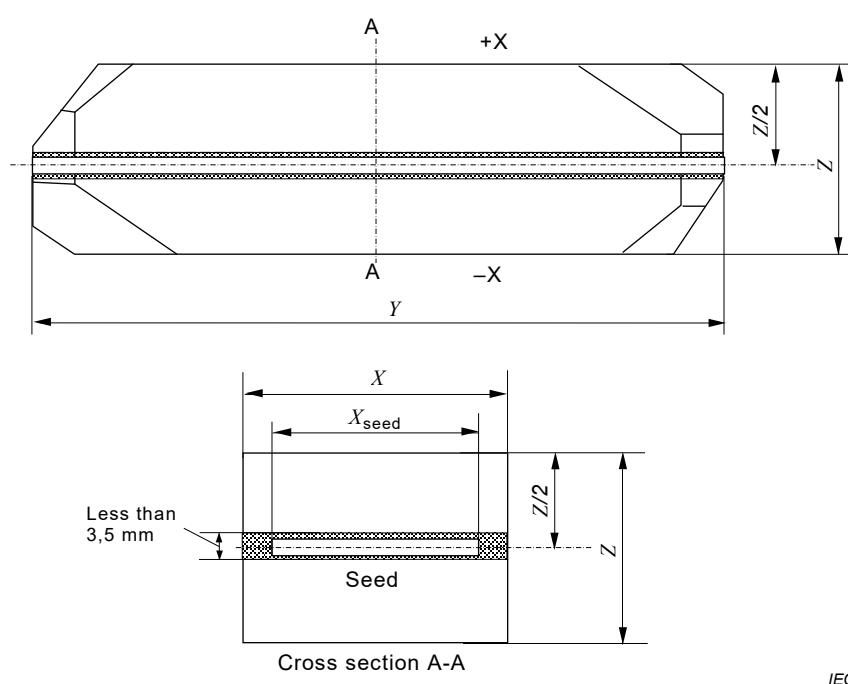


Figure 8 – Centrality of the seed with respect to the dimension along the Z- or Z'-axis

5.2 Requirements and measuring methods

5.2.1 As-grown quartz bars used for lumbered quartz bars

Raw materials for lumbered synthetic quartz crystal shall be in accordance with the standard values, related requirements and measuring methods of Clause 4 of this standard.

5.2.2 Dimensions of lumbered synthetic quartz crystal

Dimensions and tolerances shall be determined using callipers or micrometers by measuring at least at 3 points, one at the approximate midpoint of the Y-axis, and others symmetrically distributed along the Y-axis.

5.2.3 Identification on reference surface

Identification shall be marked on the reference surface and its presence confirmed by visual inspection. The content of the identification mark and the method of marking shall be determined by agreement between the supplier and the customer.

5.2.4 Measurement of reference surface flatness

A flatness of reference surface shall be measured by flatness measuring equipment as agreed between supplier and customer.

5.2.5 Measurement of reference surface angle tolerance

The deviation of the reference surface angle shall be measured by X-ray angle-measuring equipment.

5.2.6 Centrality of the seed

The centrality of the seed is determined by measuring the distance between lumbered surfaces and the nearest seed edge. An index matching liquid may be applied to the X surface to facilitate the measurement.

5.3 Delivery conditions

5.3.1 General

The delivery conditions are to be implemented by agreement between the manufacturer and the user.

5.3.2 Marking

In the marking code of lumbered synthetic quartz crystals, the crystal growth batch number shall be stated.

5.3.3 Packing

Quartz crystal users shall receive in a single package a quantity of lumbered synthetic quartz crystals having similar dimensions along the X- and Z- or Z'-axes and made from crystals of a single batch.

5.3.4 Making batch

It is recommended that, when users are supplied with lumbered synthetic quartz crystals in large quantities, they shall be made from quartz crystals of a single batch.

6 Inspection rule for synthetic quartz crystal and lumbered synthetic quartz crystal

6.1 Inspection rule for as-grown synthetic quartz crystal

6.1.1 Inspection

The inspection of synthetic quartz crystal comprises lot-by-lot tests.

6.1.2 Lot-by-lot test

6.1.2.1 General

The lot-by-lot test consists of group A and group B inspection.

6.1.2.2 Group A inspection

The test schedule for group A inspection is given in Table 6. The statistical sampling and the inspection shall be in accordance with IEC 60410 or as otherwise agreed between buyer and seller. The samples used for tests in group A may be used as the samples for group B tests.

Table 6 – Test conditions and requirements for the lot-by-lot test for group A

Test	D or ND	Test condition	IL	AQL %	Performance requirements
Minimum and maximum specified synthetic quartz crystal dimensions	ND	4.2.3.1	II	0,1	4.2.3.2
Twinning		4.2.5.1			4.2.5.1
Cracks and fractures		4.2.5.2			4.2.5.2
Inclusions	ND		II	0,4	4.1.3
NOTE Subclause numbers of test conditions and performance requirements refer to Clause 4 of this standard. D destructive ND non-destructive IL inspection level AQL acceptable quality level					

6.1.2.3 Group B inspection

The test schedule for group B is given in Table 7 or as otherwise agreed between the buyer and the seller.

The samples for group B may be selected from the samples tested in group A.

Table 7 – Test conditions and requirements for the lot-by-lot test for group B

Test	D or ND	Test condition	Sample size criterion of acceptability		Performance requirements
			<i>N</i>	<i>C</i>	
Evaluation of infrared quality by α -measurement	D	4.2.6.1	One or more as agreed between buyer and seller provided one sample represents the large size group intended for application	0	4.1.3
Etch channel density	D		One or more as agreed between buyer and seller	0	4.1.5
NOTE Subclause numbers of test conditions and performance requirements refer to Clause 4 of this standard. <i>N</i> sample size (Z-bar or Y-bar) <i>C</i> acceptance criterion (permitted number of defectives) D destructive ND non-destructive					

Etch channel and α -measurement may be performed on the same samples.

6.2 Inspection rule for lumbered synthetic quartz crystal**6.2.1 General**

The inspection of lumbered synthetic quartz crystal comprises lot-by-lot tests.

6.2.2 Lot-by-lot test

6.2.2.1 Inspection lot

An inspection lot shall consist of all the lumbered synthetic quartz crystals produced and offered for inspection at one time.

Raw material for lumbered synthetic quartz crystal shall be in accordance with the standard values, related requirements and measuring methods of this standard.

6.2.2.2 Inspection requirements

The schedule for the lot-by-lot test is given in Table 8.

The statistical sampling and inspection shall be in accordance with IEC 60410 or as otherwise agreed between the buyer and the seller.

Table 8 – Test conditions and requirements for the lot-by-lot test

Test	D or ND	Test condition	IL	AQL %	Performance requirements
Dimensions and tolerances	ND	5.2.2	II	0,4	5.1.1
Reference surface flatness		5.2.4			5.1.2
Angular tolerance of reference surface		5.2.5			5.1.3
Centrality of the seed		5.2.6			5.1.4
Verification of reference surface identification		5.2.3	II	0,1	5.2.3
NOTE Subclause numbers of test conditions and performance requirements refer to Clause 5 of this standard. D destructive ND non-destructive IL inspection level AQL acceptable quality level					

7 Guidelines for the use of synthetic quartz crystal for piezoelectric applications

7.1 General

7.1.1 Overview

The guidelines have been prepared in response to a generally expressed desire on the part of both users and manufacturers for guidelines to the best use of synthetic quartz crystal. These guidelines do not aim at explaining the practical techniques of manufacturing a crystal unit from quartz crystal, nor at attempting to cover all the properties of synthetic quartz crystal.

7.1.2 Synthetic quartz crystal

Synthetic quartz crystals are grown by the hydrothermal temperature gradient method.

A pressure chamber (autoclave) is partially filled with the alkaline (for example, Na_2CO_3 or NaOH) growing solution at room temperature. Seeds are placed in the upper space, and nutrient quartz fragments are placed in the bottom of the autoclave, which is then sealed and

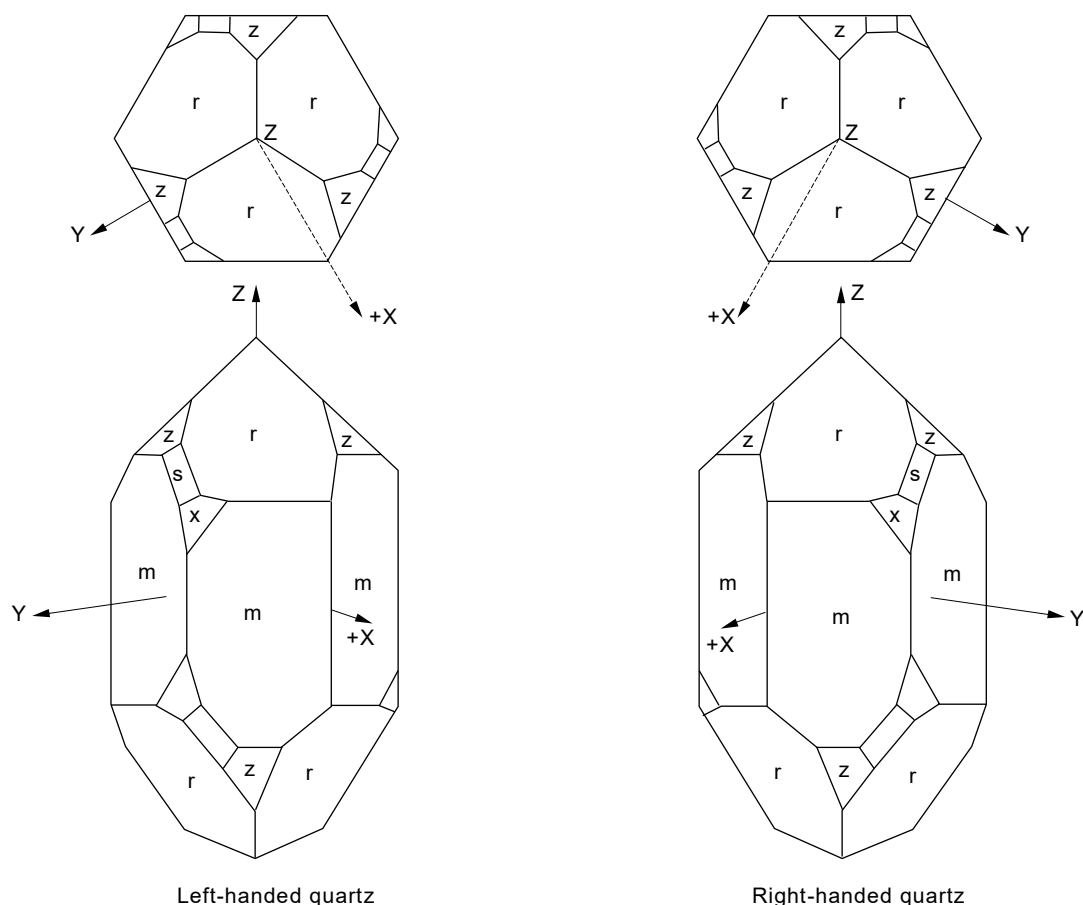
heated. The temperature in the upper space is kept lower than the temperature at the bottom. Hence, the solute nutrient is transferred by convection currents and deposited on the seeds.

Shapes, dimensions and physical properties of grown crystals depend on the orientation and dimensions of the seeds and on growing conditions. A good control of growing processes ensures uniformity in shapes and dimensions and homogeneity in quality.

7.2 Shape and size of synthetic quartz crystal

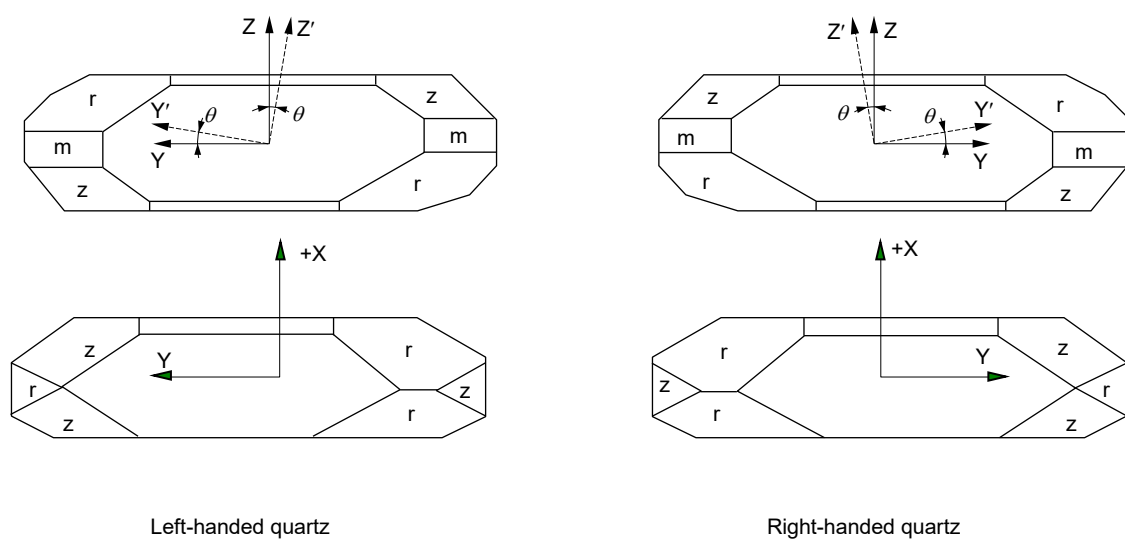
7.2.1 Crystal axis and face designation

In textbooks and in national standards, differences exist in the choice of axes, handedness and axial systems for describing a quartz crystal. Figure 9a shows a quartz crystal with all the natural faces. These are not always present on synthetic quartz. Figure 9b gives the corresponding synthetic quartz faces, but since these crystals are grown from specifically oriented seeds, their physical appearance differs materially from those shown in Figures 9a and 9b.



IEC

a) – Natural quartz crystals



IEC

b) – Synthetic quartz crystals

Figure 9 – Quartz crystal axis and face designation

7.2.2 Seed

Several standard orientations of seeds are chosen so that the most frequently used crystal units can be economically manufactured. Crystals made from Z-cut and minor rhomb (z-minor) cut seeds are mainly for manufacturing high-frequency crystal units vibrating in thickness shear modes and medium-frequency units vibrating in face shear modes. Crystals made from Z'-cut seeds are for manufacturing low-frequency crystal units vibrating in extensional or flexural modes. A seed in a grown crystal is usually surrounded by a thin veil, which consists of bubbles and inclusions.

7.2.3 Shapes and dimensions

As-grown synthetic quartz crystals are covered with the characteristic growth surfaces. Figure 10 shows the typical shape for a crystal grown on a Z-cut seed of small X-dimension. Crystals of other shapes are produced when the Z-cut seed is of other proportions or seeds of other cuts are used.

The size of synthetic quartz crystals is specified by three nominal dimensions: X, Y (or Y') and Z (or Z'). They are the dimensions along the X-axis, Y-(or Y'-) axis, and the Z- (or Z'-) axis respectively, as shown in Figure 10. The dimensions are so chosen that both economy in the growth process and good yield in the production of crystal elements may be achieved, but it should be appreciated that dimensions are a matter for discussion between the user and the manufacturer.

Since as-grown crystals exhibit such growth surfaces as m-face or z-face at the ends of the Y- or Y'-axis, the effective dimension which may be used for fabricating crystal elements is shorter than the nominal length.

An exact measurement of the minimum dimension Z' may be troublesome in practice, because of non-flatness and possible non-parallelism of the Z-surfaces. It is also difficult to achieve close dimensional tolerances. For these reasons, in cases where the user's tolerances on dimensions are closer than the manufacturer can supply, the use of partly processed material shall be considered (this is sometimes described as pre-dimensioned or lumbered quartz).