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Quartz

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Lithium Niobate
Lithium Tantalate
Langasite
Yttrium Vanadate
Gallium Orthophosphate
Gallium Nitride
Calcium Fluoride
Barium Fluoride
Lithium Fluoride
Silicon Carbide
Silicon on Sapphire
Silicon Windows
Germanium
Magnesium Fluoride

Quartz General Material Specification

1. Scope

This material specification concerns single crystal cultured quartz bars intended for use in the fabrication of piezoelectric elements for such applications as timing, frequency control and frequency selection.

2. Material Properties

2.1. Infrared α

Quartz resonators are often characterised by an electrical "quality factor" (Q) which is a measure of the efficiency of the resonator in converting between electrical and mechanical energy. Although this resonator Q depends heavily upon device considerations which are independent of the quality of the ***single crystal*** quartz material used in the device, the impurity levels in the quartz material do contribute to the overall resonator Q. A measure of this contribution is often referred to as the "material Q".
Inasmuch as material Q is not directly measured, manufacturer determines this value based on an established correlation between material Q measured with Warner-design 5 MHz resonators and infrared absorption measurements. The parameter of interest is the "infrared α" and is defined as:α_{3500} = 1/t Log T_{3800} / T_{3500})Where :α_{3500} = the extinction coefficient at wave number 3500 cm-1α = the thickness of the sample in centimetres T_m = the fraction of incident light of wave number v transmitted by the sample.
The correlation between infrared α and the material Q s then given by: 10^6 / Q = 0.114 + 7.47α -0.45α^2

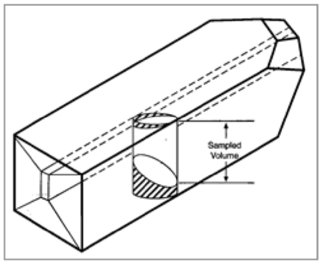
Manufacturer currently uses a Fourier Transform Infrared (FTIR) spectrometer with a custom sample handling and data collection systems to measure infrared α Based on an established degradation in infrared α with increasing crystal growth rate, the largest crystal from each autoclave run is sampled. Since it is assumed that the largest bar experienced the fastest overall growth rate, the measured α for that bar represents the worst (i.e. the highest) a for a given run. A nominal one centimetre thick Y-cut slice is taken from the bar and scanned from side to side (i.e. in the Z-direction) in the FTIR spectrometer to determine α. Reported values represent the maximum α reading across the width of the slice.

With the techniques described above, manufacturer cultured quartz crystal shall have a nominal infrared value defined by one of the following IEC grades:

Manufacturer Grade	IEC Grade	Infrared α	Material Q
Electronic grade	C	0.060	1.8 x 106
Premium Q	B	0.045	2.2 x 106
Special Premium Q	A	0.033	3.0 x 106

2.2. Inclusions

The process of culturing quartz is carried out in steel autoclaves which are not fully inert to the corrosive solution used for hydrothermal crystal growth. Chemical interactions between the dissolved quartz, the steel vessel surfaces and the mineralisers in the solution form complex alkali ferro-silicates which are present in a variety of phases. Some of these compounds become trapped in the growing crystal as inclusions which, if present in sufficient quantity and size, can have a detrimental impact on the final device performance. Depending upon the size of the autoclave run, a group of six to nine bars is selected at random to be inspected for inclusions. Sample size for a given autoclave run is based on resampling statistics compiled from 100% inspection of numerous runs over a period of time.



After marking the minus-X surface of each bar with six 6.35mm diameter circles, each bar is placed on a black background with the minus-X surface facing up. Using side illumination and index matching oil if necessary, each of the marked sites are examined under a stereoscopic microscope with calibrated reticle scales. The focal plane of the microscope is adjusted through the x-height of the bar such that the sampled volume is as indicated in the figure.

Inclusions in each size category are counted and recorded for each of the six sites. Overall inclusion densities are then calculated by dividing the total inclusions count for all sites by the total sampled volume and averaging the data by size category for all of the sampled bars.

Manufacturer cultured quartz is graded for inclusions based on the above procedures in accordance with IEC standards as summarised below:

Inclusion Mean Diameter (Im)	IEC Grade Ib (cm^{-1})	IEC Grade I (cm^{-1})	IEC Grade II (cm^{-1})
25to75	2	4	5
75to100	1	2	4
Over1OO	1	2	3

Note that IEC Grade 1b is sometimes referred to as "sweep quality". Material designated for "sweeping", a process by which the quartz is exposed to an electric field at elevated temperatures for a variety of purposes not addressed in this specification, requires ultra-low inclusion densities so that the sweeping process does not induce cracks originating from the inclusions.

2.3. Etch Channel Density

As is generally accepted within the quartz industry, manufacturer uses the etch channel density as a means of grading cultured quartz crystals for the presence of dislocations within the crystal structure. Like the infrared α test, the etching process is a form of destructive examination and, as such, is performed on a sample which is taken to be representative of the autoclave run. Since the largest bar from the autoclave run is used for the infrared α measurement, this same bar is normally used for the etch channel density measurement as well.

Typically, an AT-cut slice is taken from the sample bar and etched in ammonium biflouride. After marking the pure-Z region of the etched slice with a grid pattern, microscopic visual examination is used to determine the number of etch channels in each grid area. This data is averaged and converted to an etch channel density in the appropriate units.

In accordance with IEC standards, manufacturer quartz crystals are graded for etch channel density as follows:

IEC Grade	Maximum etch channel density (cm^{2})
1	10
2	30
3	100
4	300

2.4. Imperfections

manufacturer cultured quartz crystal are free of electrical and optical twinning, cracks, fractures and other gross imperfections in the useful volume of the crystal. The presence of such imperfections is detected by visual examination of a representative sampling of bars using a refractive index matching oil while the crystal is illuminated with a bright incandescent source.

2.5 Handedness

manufacturer cultured quartz crystal are, unless otherwise specified, right handed. Note that the definition of right handedness is in accordance with the IEEE convention which defines right handed crystals by the right rotation of light propagating along the z-axis.

3. Lumbered Properties

The above specifications address general material properties which apply to all manufacturer cultured quartz products, whether purchased "as-grown" or as lumbered bars. In addition, manufacturer lumbered products unless otherwise specified, adhere to the following typical dimensional specifications. Note that references to the dimensional axes in this section assume that the desired crystallographic orientation of the bar surfaces is 0° with respect to the z-axis, and that the length of the bar is in the direction of the y-axis. For alternate orientations, comparable tolerances would be maintained relative to the desired orientation.

Unless otherwise specified, all properties are verified by sampling in accordance with MIL-STD1055D.

3.1. Dimensional Tolerances

The lumbered X and Z dimensions of manufacturer cultured quartz crystal bars shall have tolerances of ± 0.13mm, and shall be within tolerance at all points along the bar.

3.2. Seed Centering

When required, seed centering between the two x-surfaces is verified using an optical comparator. When seed material is present in manufacturer lumbered quartz bars, the seed shall be fully contained within a 3.0mm band centered between the two z-surfaces, as verified visually using callipers.

3.3. Surface Orientation

The orientation of crystal surfaces is determined by means of x-ray diffraction based on the specific surfaces to be oriented and the known Bragg angles of the various atomic planes. Crystals are oriented prior to lumbering to establish proper cutting angles and then re-tested after lumbering to verify correct angular orientation.

3.3.1. Seed-Free Material

manufacturer seed-free lumbered bars shall have a maximum deviation of ± 15° in both the lumbered minus-x reference surface with respect to the y- and z- axes, as well as the lumbered z-surfaces with respect to the x- and y-axes.

3.3.2. Seed-Centered Material

manufacturer seed-centered lumbered bars shall have a maximum deviation of ± 15° in the lumbered minus-x reference surface with respect to the y- and z- axes and of ± 10 in the z-surfaces with respect to the x- and y- axes.

3.4. Surface Roughness

Surface roughness refers to the fine irregularities in the surface of a lumbered crystal surface that are a consequence of the machining process. One method by which surface roughness can be quantified is by measuring the distance between peaks and valleys over a representative sampling length on the surface. This is accomplished by means of a profilometer, which uses a stylus to trace the microscopic profile of the lumbered crystal surface over a characteristic length. The average roughness, Ra , is defined as the average deviation from the theoretical centreline of all of the peaks and valleys.

Unless otherwise specified, the minus-x reference surface of manufacturer lumbered quartz crystal bars shall have an Ra of not greater than 4 µm when measured over the useful surface of the crystal.

3.5 Surface Flatness

In order to assure ease of wafering, it is essential that the lumbered reference surface be maintained as a truly flat surface. In this manner, as long as the reference surface orientation is maintained, all but the principal wafer cutting angle is assured by the properties of this reference surface. Flatness measurements on lumbered surfaces are made relative to a ground granite reference flat. The crystal is suspended on two parallel blocks atop the reference flat such that the surface of interest is on the underside closest to the reference flat. A dial indicator is attached to stand which rests on the flat. Surface flatness is determined by adjusting the indicator to ride on the underside of the crystal and then sliding the stand on the reference flat so that the indicator traverses the surface of the crystal.

The minus-x reference surface of manufacturer lumbered bars shall be flat to within 0.1mm when measured over the useful surface of the crystal.

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