

## Synthetic Quartz

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### History of Synthetic Quartz Crystals

In 1905, George Spezia (Italy) announced the growth of quartz crystal using hydrothermal method and it was the first report in the world about the hydrothermal growth of sizable quartz crystal. In Japan, Yamanashi University started the research work on the hydrothermal growth of quartz crystal in 1953. After the research and development, high temperature/pressure vessel called autoclave was improved, and growth-technique was also improved, and finally, the industrial production of synthetic quartz crystals was started in 1973 with the amount of 7 kg of crystals per one autoclave.

After the progress in crystal growth-technique, the quartz crystals having the same quality level of natural quartz crystals were able to be grown in mass production, and furthermore, upscale of autoclave was performed to increase the amount of crystals. In 1973, an autoclave with a capacity of 1 m<sup>3</sup> was made and a full-fledged mass production was started. Now, an autoclave with a capacity of more than 4 m<sup>3</sup> is under operation, and yield of quartz crystal per autoclave is above 2,000 kg.

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### Characteristics of Quartz Crystal



<b>Hardness:</b>	7 (Mohs' hardness)
<b>Specific gravity:</b>	2.65 x 103 kg/m <sup>3</sup>
<b>Crystal system:</b>	Trigonal System
<b>Chemical composition:</b>	SiO <sub>2</sub>
<b>Relative permittivity:</b>	4.5
<b>Transition temperature:</b>	573 degrees
<b>Solubility:</b>	Soluble in fluorine compound

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### Piezoelectric effect

Since quartz crystal has piezoelectric property, applied force in some direction causes electrical charge. On the contrary, application of an electrical field across the quartz crystal causes deformation in the crystal. For instance, if a quartz crystal wafer which is perpendicular to X-axis (electric axis) is placed in an electric field as shown in the figure below, the wafer shrinks in X direction.

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### What is the growth of Synthetic Quartz Crystal?

Quartz crystal is grown using the phenomena that the solubility of quartz crystal in alkali-liquid is different at different temperature, and it is grown at high temperature under high-pressure. The vessel for growth is a pressure vessel called an autoclave. Raw material, many pieces of lasca (small piece of natural quartz crystal), is placed in the lower part of autoclave and seeds are suspended in the upper part. Then the vessel is filled with water and mineralizer such as Na<sub>2</sub>CO<sub>3</sub> or NaOH, and after encapsulating, it is heated up to gain high pressure. Finally, it reaches to supercritical state and the growth begins. By keeping the temperature of the upper part of autoclave lower than that of the lower part of

autoclave, convection occurs and dissolved quartz crystal is moved from the lower part to the upper part, then the dissolved quartz crystal precipitates on the seed crystal. The synthetic quartz crystal is grown with the repetition of this cyclic movement. Growth condition, size and orientation of seed crystal are adjusted to produce the synthetic quartz crystals, which have different shape, dimensions and properties, for various applications. Since the synthetic quartz crystals are grown under strict control, they have controlled shape, dimensions and properties.

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#### What is the seed crystal?

The orientation of seed crystal is determined considering the efficiency of production of quartz crystal unit. The synthetic quartz crystals grown using X-cut or r-cut seed are used to make thickness-share mode quartz crystal units for high frequency range application, and the contour-mode quartz crystal units for middle frequency range application. The synthetic quartz crystals grown using rotated X-cut seed are used to make extension or flexure-mode quartz crystal units for low frequency range application. In some cases, a thin layer, called seed veil, which consists of foreign object covers the seed in the grown crystal. And it deeply affects to the quality of quartz crystal.

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#### Quality evaluation of synthetic quartz crystal

The amount of crystal defect and impurity in synthetic quartz crystal depends on growth rate, mineralizer and raw material. The growth rate affects greatly to the important properties such as infra-red absorption coefficient  $\alpha$ , which correlates to Q value, and frequency-temperature characteristics. The larger growth rate causes increase in  $\alpha$ , decrease in Q value, and dispersion in frequency-temperature characteristics. The quality index of synthetic quartz crystal was originally a Q value, and a 5 MHz quartz crystal unit operated in 5th overtone mode was used to obtain the Q value. But it required laborious work to fabricate the 5 MHz crystal unit, so the index had been changed to the coefficient  $\alpha$  instead of the Q value.

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#### Standard specification for synthetic quartz crystal

##### Twining

There shall be no electrical or optical twinning in the usable region.

##### Strain

There shall be no strain contained both inside and surface of seed crystal as well as in a grown quartz crystal.

##### Cracks and fractures

There shall be no cracks, chippings or fractures in the usable region.

##### Inclusion density

The specification is in accordance with the IEC 60758.

Grade/size range $\mu\text{m}$	Desities per $\text{cm}^3$			
	10 to 30	30 to 70	70 to 100	> 100
Ia	2	1	0	0
Ib	3	2	1	1
I	6	4	2	2
II	9	5	4	3
III	12	8	6	4

##### Infra-red quality indication:

Grade	Maxima	Estimated Q values (x $10^6$ )
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α3585		
Aa	0.015	3.8
A	0.024	3.0
B	0.050	2.4
C	0.069	1.8
D	0.100	1.4
E	0.16	1.0

**Etch channel density**

Grade	Maximum number ρ per cm <sup>3</sup>
1	10
2	30
3	100
4	300
5	600

Tokyo Denpa guarantees the Grade 2 for all the products. The products of the Grade 1 are available upon request.

Seed orientation, dimensions and location:

Orientation: 0°00'±15'

Dimensions: 1.5 mm±0.5mm (along Z axis)

Location: Within the hatched area of 3 mm maximum centered as described in the figure below

**Specification for lumbered quartz crystal**

Angles:

Rotation angle of Z-surface around

X-axis:

00°00'±30'

Rotation angle of X-surface around

Y-axis:

00°00'±15'

Rotation angle of X-surface around

Z-axis:

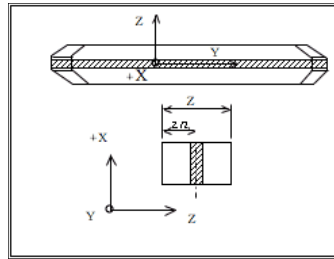
00°00'±15'

Dimensional tolerance:

±0.1 mm (along X or Z axis)

Surface roughness:

12.5µm (10-point average)



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**Typical synthetic quartz crystal**

**Quartz crystal for SAW devices**



Specification:

Inclusion density:	II or better
Infra-red quality indication:	C or better
Etch channel density:	2 or better

**Quartz crystal for miniature crystal units or filters**



## Specification:

Inclusion density:	II or better
Infra-red quality indication:	B or better
Etch channel density:	2 or better

#### Quartz crystal for SAW devices

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## Specification:

Inclusion density:	II or better
Infra-red quality indication:	B or better
Etch channel density:	2 or better

#### Quartz crystal for SAW devices

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The standard specification is shown in the table below.

The blanks for special requirements are available upon request.

Dimensions (mm) Tolerance: 0.05	Thickness (mm) Tolerance: 0.05	Surface finish	Major surface	Orientation flat2		Fringe finish
			Angular tolerance	Dimensional tolerance (h)	Angular tolerance	
Φ6.0φ	0.15 <sup>-</sup>	I·II	±15 <sup>''</sup>	±0.2	±15 <sup>'</sup>	#400
Φ8.0	0.15 <sup>-</sup>	I·II	±15 <sup>''</sup>	±0.2	±15 <sup>'</sup>	#400
Φ8.7	0.20 <sup>-</sup>	I·II	±15 <sup>''</sup>	±0.2	±15 <sup>'</sup>	#400
Φ79.0	0.50 <sup>-</sup>	I·II·III	±1 <sup>'</sup>	±0.5	±15 <sup>'</sup>	#400
Φ102.0	0.50 <sup>-</sup>	I·II·III	±1 <sup>'</sup>	±0.5	±15 <sup>'</sup>	#400
9 x 9	0.15 <sup>-</sup>	I·II	±15 <sup>''</sup>	-	-	#240
10 x 10	0.15 <sup>-</sup>	I·II	±15 <sup>''</sup>	-	-	#240
12 x 12	0.15 <sup>-</sup>	I·II	±15 <sup>''</sup>	-	-	#240
80 x 80	0.50 <sup>-</sup>	I·II	±1 <sup>'</sup>	-	-	#240
102 x 102	0.50 <sup>-</sup>	I·II	±1 <sup>'</sup>	-	-	#240

## Note:

1. Surface finish (I) : #1000 (II) : #2000 (III) : polishing
2. Thickness may be determined by a resonance frequency.
3. The character h in the column of orientation flat means the distance between two flats.

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#### Lumbered Synthetic Quartz Crystal



### Angular deviation

X axis-rotation of reference Z surface  $0^{\circ} 00' \pm 30'$   
 Y axis-rotation of reference - X surface  $0^{\circ} 00' \pm 15'$   
 Z axis-rotation of reference - X surface  $0^{\circ} 00' \pm 15'$

### Surface

Surface finish #80 for diamond tool.

### Cracks and fractures

There shall be no cracks or fractures in the usable region.

### Strain

Crystal does not have strain inside.

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### Standard specifications

Type	Type No.	Dimension (mm)			Etch channel density JIS Grade	$\alpha$ 3585 Maxima $\alpha$	Inclusion density JIS Grade	Orientation of theseed (0)	Remarks
		X	Z	Y					
		$\pm 0.2$	$\pm 0.2$	$\pm 0.5$					
Pure Z	PB-18	9.0	18.0	200	1	0.050	1a~II	$0^{\circ} 00'$	Z bar
	PB-19	9.5	19.0	200	1	0.050	1a~II	$0^{\circ} 00'$	Z bar
	PB-20	10.0	20.0	200	1	0.050	1a~II	$0^{\circ} 00'$	Z bar
	PB-23	23.0	18.0	185	1	0.050	1a~II	$2^{\circ} 00'$	Seedless
	PB-31	31.0	18.0	185	1	0.050	1a~II	$2^{\circ} 00'$	Seedless
	PB-38	65.0	38.0	185	1	0.050	1a~II	$2^{\circ} 00'$	Wristwatch
	PB-41	65.0	41.0	185	1	0.050	1a~II	$2^{\circ} 00'$	Wrist watch
	SB-02	53.0	46.0	200	1	0.050	1a~II	$0^{\circ} 00'$	2-inch SAW
	SB-03	76.0	65.0	200	1	0.069	1a~II	$0^{\circ} 00'$	3-inch SAW
	SB-04	100.0	85.0	200	1	0.069	1a~II	$0^{\circ} 00'$	4-inch SAW

※Custom-made items that are not included in the list are also available upon request.



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