

IEC/PAS 62276

Edition 1.0
2001-08

**Single crystal wafers applied
for surface acoustic wave device –
Specification and measuring method**

PUBLICLY AVAILABLE SPECIFICATION



INTERNATIONAL
ELECTROTECHNICAL
COMMISSION



Reference number
IEC/PAS 62276

IECNORM.COM : Click to view the full PDF of IEC PAS 62216:2001

Withdrawn



電子情報通信学会規格

The Institute of Electronics, Information and Communication
Engineers Standard

Single crystal wafers applied for surface acoustic wave
device 0

- Specification and Measuring method -

弾性表面波デバイス用単結晶ウェーハ
－規格と測定法－

IEICE/Std - 0002

2001 年 4月

April, 2001

(社)電子情報通信学会

The Institute of Electronics, Information and
Communication Engineers

IECNORM.COM : Click to view the full PDF of IEC PAS 62216:2001

Withdrawn

INTERNATIONAL ELECTROTECHNICAL COMMISSION

SINGLE CRYSTAL WAFERS APPLIED FOR SURFACE ACOUSTIC WAVE DEVICE – SPECIFICATION AND MEASURING METHOD

FOREWORD

A PAS is a technical specification not fulfilling the requirements for a standard, but made available to the public and established in an organization operating under given procedures.

IEC-PAS 62276 was submitted by the Japanese Institute of Electronics, Information and Communication Engineers and has been processed by IEC technical committee 49: Piezoelectric and dielectric devices for frequency control and selection.

The text of this PAS is based on the following document:

This PAS was approved for publication by the P members of the committee concerned as indicated in the following document:

Draft PAS	Report on voting
49/504/PAS	49/513/RVD

Following publication of this PAS, the technical committee or subcommittee concerned will investigate the possibility of transforming the PAS into an International Standard.

- 1) The IEC (International Electrotechnical Commission) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of the IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, the IEC publishes International Standards. Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. The IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
- 2) The formal decisions or agreements of the IEC on technical matters express, as nearly as possible, an international consensus of opinion on the relevant subjects since each technical committee has representation from all interested National Committees.
- 3) The documents produced have the form of recommendations for international use and are published in the form of standards, technical specifications, technical reports or guides and they are accepted by the National Committees in that sense.
- 4) In order to promote international unification, IEC National Committees undertake to apply IEC International Standards transparently to the maximum extent possible in their national and regional standards. Any divergence between the IEC Standard and the corresponding national or regional standard shall be clearly indicated in the latter.
- 5) The IEC provides no marking procedure to indicate its approval and cannot be rendered responsible for any equipment declared to be in conformity with one of its standards.
- 6) Attention is drawn to the possibility that some of the elements of this PAS may be the subject of patent rights. The IEC shall not be held responsible for identifying any or all such patent rights.

FOREWORD

Although the mobile communication is spreading rapidly with a global scale in recent years, the surface acoustic wave device is used, to the cellular phone that is used for these widely. Wafers of the various single crystal piezoelectricity materials are used for those devices. However, those standard is carried out individually between a manufacturer and user, that is actual situation.

As the demands of single crystal wafers are increasing, it is indispensable to standardize them such as the terms and definitions, test conditions, measurement methods of materials, and guide the use.

International Electrotechnical Commission located in Geneva is actively working for the international standardization in the electrotechnical field. Among many Technical Committees (TCs) in IEC, TC 49 is working on the Piezoelectric and Dielectric Devices for Frequency Control and Selection. TC 49 has ten Working Groups (WGs), and the Working Group 5 (WG 5) is working for the preparation and deliberation of the IEC standard on the piezoelectric single crystal.

The piezoelectric and dielectric devices for frequency control and selection in the Standard Committee of the Institute of Electronics, Information and Communication Engineers have gotten active as the interior deliberation party of IEC/TC 49. On the other hand, QIAJ (Quartz Industry Association of Japan) is the industry meeting that was organized with the maker of a crystal unit and the activity corresponding to TC 49/WG 5 is done by the material committee which belongs QIAJ. This document was issued by the material committee of QIAJ, under cooperation with the 10th work sectional meeting (WG 10) of TC 49.

When the Japanese National Committee for IEC/TC 49 proposed this documents as a new work item proposal (49/428/NP), however, was not approved (49/RVN/440), because only two countries; Germany and Japan, nominated experts to participate this project. According to the IEC rule for the New Work Items Proposal, it is required to start a new project that more than four P-member countries should nominate the name of experts and this proposal failed. But, the Japanese National Committee for IEC/TC 49 decided to continue the work to draft this standard, even though it was not approved, because we believed that this should be a very fundamental, useful and mandatory documents. Therefore, Technical Committee of QIAJ cooperated to make out this work and the draft was reviewed at the TC 49 Nara meeting, April, 2000 and recommended as a PAS document (49/RVN/474). Finally, this document has been completed

published as a standard of the Institute of Electronics, Informaion and Communication Engineers and a technical standard of QIAJ.

This standard is a fruit of collecting wisdom in the field of advanced technology in Japan and it is open for public as the Standard of the Institute of Electronics, Informaion and Communication Engineers. And it is expected that this standard will contribute to the development of technology in this fast growing field. This standard will be submitted to the IEC in the track of IEC PAS (Publicly Available Specification) for international circulation.

Finally, I would like to express my sincere appreciation to Mr. Kunihiro Nagai, Chairman, all members of the 10th work sectional meeting (WG 10) of the Japanese National Committee for IEC/TC 49 and Technical Committee of QIAJ, for their efforts develop this standard.

Mikio Takagi
Chairman

The Japanese National Committee for IEC/TC 49
in the Standard Committee of the Institute of
Electronics, Information and Communication Engineers.

Contributors to "Single crystal wafers applied for surface acoustic wave device
-Specification and Measuring method- "

Chief	Mr. Kunihiro Nagai (Toyo Communication Equipment)
Member	Mr. Kazushi Kamiyama (Kyocera)
Member	Mr. Masakazu Kurashige (Kinseki)
Member	Mr. Masami Mochizuki (Koike)
Member	Mr. Hajime Shimizu (Koto Crystal)
Member	Mr. Toshihiko Ryuou (Shinetsu Kagaku kogyo)
Member	Mr. Masayoshi Matsui (Sumitomo Metal Mining)
Member	Mr. Shinichi Shikada (Sumitomo Denki Kogyo)
Member	Mr. Susumu Maeda (Daishinku)
Member	Mr. Katsuma Muroishi (Tokyo Denpa)
Member	Dr. Sadao Matsumura (Toshiba)
Member	Dr. Junichi Kushibiki (Tohoku University)
Member	Dr. Masaaki Ono (Fujitsu Media Device)
Member	Mr. Junji Takahashi (Nihon Dempa Kogyo)
Member	Mr. Yutaka Mikawa (Fine Crystal)
Member	Heiji Takashima (Meidensha)
Member	Teitaka Sahashi (Yamaju Ceramics)

CONTENTS

Introduction	1
Section 1 : Specification for single crystal wafer.....	1
1 Scope.....	1
2 Reference documents.....	1
3 Terms and definitions.....	2
4 Symbols and abbreviated terms.....	5
5 Requirements.....	5
6 Sampling.....	7
7 Test methods.....	8
8 Identification, labeling, packaging, delivery condition.....	9
Section 2 Measuring method	10
1 Measuring method of Curie Temperature.....	10
2 Measurement of lattice constant(Bond method).....	11
3 Measurement of face angle by X-ray.....	12
4 Appearance inspections.....	14
Annex A(Normative) Expression using Eulerian angle for piezoelectric Single crystal.....	16
Annex B(Informative) Crystal growth method.....	18
Annex C(Informative) Manufacturing lot.....	22
Annex D(Informative) Explanation of TTV, LTV and Sori.....	22
Annex E(Informative) Standard mechanical wafer process flow chart.....	24
Annex F(Informative) Reference.....	26

IECNORM.COM : Click to view the full PDF of IEC PAS 62216:2001

Withdrawn

Single crystal wafers applied for surface acoustic wave device - Specification and measuring method

Introduction

A number of piezoelectric materials has become to use for surface acoustic wave (SAW) filters and resonators according to increasing wide electrical application and production demand. Until now, specifications for wafers were subjected to agreement between user and supplier. By these circumstances, IEC meeting in 1996 held at Rotterdam, Holland had announced to make proposals about new wafer standardization. This specification is respond to the standardization request in piezoelectric single crystal wafers for surface acoustic wave devices.

Section 1: Specification for single crystal wafer

1 Scope

This document applies to single crystal wafers intended for manufacturing substrates made of synthetic quartz crystal, lithium niobate, lithium tantalate, lithium tetraborate crystals for surface acoustic wave (SAW) filters and resonators

2 Reference documents

- IEC 60758:1993 Synthetic quartz crystal – Specifications and guide to the use
- IEC 60862-1:1989 Surface acoustic wave (SAW) filters. Part 1: General information, standard values and test conditions chapter I : General information and standard value –chapter II : Test conditions
- IEC 60862-2:1991 Surface acoustic wave (SAW) filters-Part 2:Guide to the use of surface acoustic wave filters (chapter III)
- IEC 60862-3:1986 Surface acoustic wave (SAW) filters. Part 3 : Standard outlines (Chapter IV)
- IEC 61019-1-1:1990 Surface acoustic wave (SAW) resonators-Part 1: General information, standard values and test conditions-Section1 General information and standard values
- IEC 61019-1-2:1993 Surface acoustic wave (SAW) resonators-Part 1: General information, standard values and test conditions-Section2:Test conditions
- IEC 61019-2:1995 Surface acoustic wave (SAW) resonators-Part 2: Guide to the use
- IEC 61019-3:1991 Surface acoustic wave (SAW) resonators-Part 3: Standard outline and lead connections.
- IEC 60410 Sampling plans and procedures inspection by attributes

3 Terms and definitions

3.1 Single crystals for SAW wafer

3.1.1 As-grown synthetic quartz crystal: Right handed or left handed single crystal quartz grown hydrothermally. As-grown refers to the state of processing and indicates a state prior to mechanical fabrication.

3.1.2 Lithium niobate: Single crystals described by chemical formula to LiNbO_3 grown by Czochralski method (crystal pulling method) or other growing method.

3.1.3 Lithium tantalate: Single crystals described by chemical formula to LiTaO_3 grown by Czochralski method (crystal pulling method) or other growing method.

3.1.4 Lithium tetraborate: Single crystals described by chemical formula to $\text{Li}_2\text{B}_4\text{O}_7$ grown by Czochralski method (crystal pulling method), vertical Bridgeman method or other growing method.

3.2 Manufacturing lot: Lot consists of one LN, LT or LBO crystal or one synthetic quartz crystal growth batch.

3.3 Terms and definition related synthetic quartz

3.3.1 Seed: A rectangular parallelepiped crystal plates or bar to be used as a nucleus for crystal growth.

3.3.2 Infrared absorption coefficient α -value: Measured absorption at specific infrared wavenumber by means of defect structures as OH-bond in crystal lattice.

$$\alpha = \frac{1}{t} \log \frac{T_1}{T_2}$$

α : Infrared absorption coefficient

t: Thickness of Y-cut sample in centimeter

T1: Percent transmission at wavenumber of 3800 or 3979 cm^{-1}

T2: Percent transmission at wavenumber of 3410, 3500 or 3585 cm^{-1}

3.3.3 Inclusions: Any foreign materials within a crystal, visible by examination of scattered light from a bright source.

3.3.4 Seed veil: The array of inclusions or voids on the surface of the seed upon which a crystal has been grown.

3.3.5 Etch channel: An etch channel is a roughly cylindrical void that is present along dislocation line after etching a quartz crystal.

3.3.6 Pinhole in seed: Pipe-like cavity along the dislocation (or line defects) within seed created at initial duration of quartz crystal growth.

3.4 Terms and definition related LN and LT crystal

3.4.1 Lattice constant: A length of one unit cell measured by X-ray using Bond method

3.4.2 Curie temperature: Phase transition temperature between ferroelectric and paraelectric by thermal analysis or dielectric measurement

3.4.3 Single domain: In a state of same electrical polarization in ferroelectric crystal (i.e. LN and LT).

3.4.4 Polarization (or Poling) process: Electrical process to single polarization of ferroelectric crystals.

3.4.5 Congruent composition: Chemical composition of single crystal consist with molten solution in growing process.

3.4.6 Twins: Twins follow laws of crystallography relating symmetrically to specific faces to axes.

3.5 Orientation flat: Cut surface intersecting around wafer objective to indication for crystal orientation. A direction of orientation flat almost agree with SAW propagation. It is also called as "Primary flat". (see figure 1)

3.6 Index flat: Cut surface intersecting around wafer objective to indication or polarization. It is also called as "Secondary flat" or "Sub orientation flat". (see figure 1)

3.7 Flatness

3.7.1 TV5 (Thickness Variation for five points): TV5 indicates the variation of thickness in a wafer and is the maximum difference of thickness. Thickness is measured at the center of the wafer and at the 4 points as shown in figure 1.

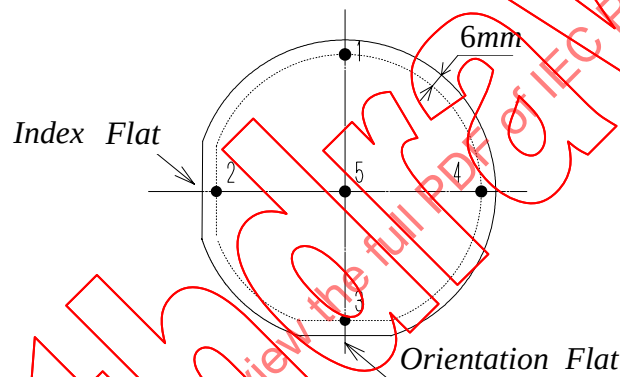


Figure 1. Wafer indication and measuring points for TV5

3.7.2 Warp¹⁾: Warp is one of indications that shown the deformation of a wafer and defined as the maximum difference of the median surface for an unclamped wafer from a reference plane as shown in figure 2. Reference plane is defined as 3-points focal plane. Warp is a bulk property of a wafer and is not a exposed surface .

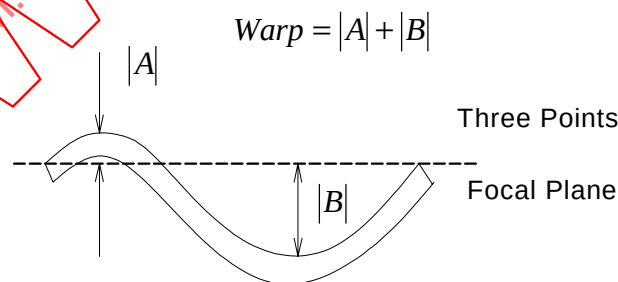


Figure 2 Schematic diagram of warp

3.8 Roughness of back surface: Definitions of Ra are given in ISO 468, "Surface roughness", and ISO 4287/1, "Surface and its parameters".

3.9 Surface orientation: Crystallographical orientation of the axis perpendicular to the surface of wafer.

3.10 Description of orientation and SAW propagation: Indicating the orientation for SAW propagation followed by surface orientation connected by “-“. In case of 0° orientation can abbreviate from explanation. Typical examples for these expression are as follows;

Table 1 Description of Orientation

Materials	LN	LT	Quartz crystal	LBO
Expression	128° Y-X Y-Z 64° Y-X	X-112° Y 36° Y-X	ST-X	45° X-Z

3.11 ST-cut: Although the original definition is 42.75 degree rotated Y-cut and X-propagation, actually cut angle has an available range of 20 –42.75 degree to be given zero temperature coefficient .

3.12 Tolerance of surface orientation: Tolerable difference between indicated surface orientation and measured orientation by X-ray goniometer. A larger value should adopt either parallel or perpendicular to the orientation flat.

3.13 Bevel: A slope or round shaping of wafer in a edge region.

3.14 Diameter of wafer: Diameter of circular portion of wafer except orientation-flat region.

3.15 Thickness of wafer: A thickness measured at a center of wafer.

3.16 Definitions of appearance defects

3.16.1 Contamination²⁾: Contamination can be classified into two groups, one is area contamination and another is particulate contamination. Those are surface contaminant that can not be removed by cleaning or are stained after cleaning. Those may be foreign matter on the surface such as localized area that are smudged, stained, discolored, mottled, etc., or large areas exhibiting a hazy or cloudy appearance resulting from a film of foreign materials.

3.16.2 Crack: Cleavage or fracture that extends surface of a wafer and that may or not through the entire thickness of the wafer.

3.16.3 Scratch: A shallow groove or cut below the established plane of the surface, with a length to width ratio greater than 5:1.

3.16.4 Chipping: Region where material has been removed from the surface or edge of the wafer. The size of chipping can be expressed by its maximum radial depth and peripheral chord length.

3.16.5 Dimple: A smooth surface depression larger than 3 mm diameter.

3.16.6 Pit: Pit means and hollow caused almost by bulk defects and manufacturing process.

3.16.7 Orange peel: Large featured, roughened type of surface visible to the unaided eye.

4 Symbols and abbreviated terms

4.1 SAW: An abbreviation of Surface Acoustic Wave.

4.2 LN: An abbreviation of Lithium Niobate.

4.3 LT: An abbreviation of Lithium Tantalate.

4.4 LBO: An abbreviation of Lithium Tetraborate.

4.5 OF: An abbreviation of Orientation Flat.

4.6 IF: An abbreviation of Index Flat.

4.7 Tc: An abbreviation of Curie Temperature.

5 Requirements

5.1 Material Specification

5.1.1 Synthetic quartz crystal: Synthetic quartz crystal grown by Z-cut seed having the orientation within +5 degree of arc, and wafer should consist of excepting -X growth region. Quality of synthetic quartz crystal is better than the following grades in accordance with IEC 60758.

Infrared absorption coefficient α value; Grade D:

Inclusion density (pcs./cm³); Grade II

Etch channel density (pcs./cm²); Grade 2

5.1.2 LN: Material properties having a Curie temperature within specified value and also treated to single domain by polarization process.

5.1.3 LT: Material properties having a Curie temperature or lattice constant within specified value and also treated to single domain by polarization process.

5.1.4 LBO: Material properties not including twins.

5.2 Specification for wafer

5.2.1 Diameters and tolerances:

76.2 mm $\pm$ 0.5 mm (For commonly called as 3 inches wafer)

100.0 mm $\pm$ 0.5 mm

5.2.2 Thickness and tolerance

0.3~0.5 mm $\pm$ 0.03 mm

5.2.3 OF

Dimensions of OF and tolerances

1) 22.0 mm $\pm$ 3.0 mm (For 76.2 mm wafer)

32.0 mm $\pm$ 3.0 mm (For 100 mm wafer)

2) Orientation tolerance

Orientation tolerance: $\pm 30^\circ$

Orientation of OF shall be perpendicular to SAW propagation. In case of wafer having orientation of OF parallel to SAW propagation, it should subject to agreement between user and supplier. Orientation of OF for quartz crystal wafer is X-plane (110) and -X side.

5.2.4 IF

1) Dimension of IF and tolerances

Dimensions and these tolerances of IF are specified as reference value

10.0 mm $\pm$ 5 mm (For 76.2 mm wafer)

12.0 mm $\pm$ 5 mm (For 100 mm wafer)

2) Orientation tolerance of IF

Orientation tolerance of IF are specified between user and maker. Nevertheless , a following is reference value. Orientation of IF is not specified.

Orientation Tolerance : $\pm 1.0^\circ$

3) A laser marking is permitted as an alternative method of IF.

5.2.5 Roughness of back surface: As specified in Table 2.**5.2.6 Warp:** As specified in Table 2.**5.2.7 TV5:** As specified in Table 2.**Table 2 Roughness, Warp, and TV5**

Materials	Diameter of wafer	Roughness of back surface(Ra)	Warp (μ m) Specified value	TV5 (μ m) Specified value
Quartz crystal	76.2 mm (3 inches)	0.5 μ m or over	30	10
		Less than 0.5 μ m	20	10
	100mm	0.5 μ m or over	40	15
		Less than 0.5 μ m	30	10
LN,LT	76.2 mm (3 inches)	2.0 μ m or over	50	15
		2.0~0.5 μ m	40	15
		Less than 0.5 μ m	40	10
	100mm	2.0 μ m or over	50	20
		2.0~0.5 μ m	40	15
		Less than 0.5 μ m	40	10
LBO	76.2 mm (3 inches)	0.5 μ m or over	40	15
		Less than 0.5 μ m	40	10

5.2.8 Surface finishing for propagation surface: Finishing of propagation surface shall be processed by mirror polish. Details of surface finishing subjected to agreement between user and supplier.

5.2.9 Defects of propagation surface³⁾

1) Scratch

No scratch by visual inspection .

2) Chipping

a) Edge chip:

Direction to center of wafer : less than 0.5mm

Parallel to tangential line at edge of wafer : less than 1.0mm

b) Surface:

There is no chipping by visual inspection.

3) Crack:

No crack by visual inspection.

4) Contamination:

No contamination by visual inspection.

5) Others

Other defects such as dimple, pit, and orange peel are occasionally detected on a wafer surface. No defect also specified by visual inspection.

5.2.10 Surface orientation and its tolerance

Surface orientation shall be specified by user and supplier.

Tolerance : Quartz crystal: $\pm 10'$
 LN • LT • LBO: $\pm 20'$

5.2.11 Inclusions

LN/LT/LBO wafer : No visible inclusion by naked eye inspection

Synthetic quartz wafer : Material satisfying the specification better than Grade II of IEC 60758 "Synthetic quartz crystal", 1.4.2 "Inclusion density".

5.2.12 Etch channel density and position of seed for quartz wafer

- 1) Etch channel within seed portion for quartz crystal wafer

Density of etch channel in a state of not passing through from propagation surface to back surface is less than 36 as per 76.2 mm wafer or less than 47 as per 100 mm wafer.

- 2) Position of seed

Position of seed shall be included within ± 3.5 mm center width of Z' direction and parallel to X-direction of center of wafer.

5.2.13 Bevel

Bevel shall be specified by user and maker.

5.2.14 Curie temperature and tolerance: This item is not request if lattice constant is specified.

LN : $1133 \sim 1145^{\circ}\text{C} \pm 3^{\circ}\text{C}$

LT : $598 \sim 608^{\circ}\text{C} \pm 3^{\circ}\text{C}$

5.2.15 Lattice constant : This item specified either Curie temperature or lattice constant.

LT: $0.51538 \text{ nm} \pm 0.00002 \text{ nm}$ for a-axis

6 Sampling

Sampling plan shall execute conforming with a effective statistical method by agreement between user and maker. Sample wafers must satisfy the quality assurance level by specified test and fully represent a population.

6.1 Sampling

Unless otherwise specified number of sampling, it shall specified in accordance with AQL 2.5%, single sampling as defined in IEC 60410.

6.2 Number of sampling

- 1)Dimensions

Diameter

2 wafers/manufacturing lot

Thickness

2 wafers/manufacturing lot

Length of OF

2 wafers/manufacturing lot

- 2)Surface orientation

2 wafers/manufacturing lot

- 3)Orientation of OF

2 wafers/manufacturing lot

- 4)Back surface finishing

2 wafers/manufacturing lot

- 5)TV5

2 wafers/manufacturing lot

- 6) Warp

2 wafers/manufacturing lot

6.3 All sampling

Following items shall be inspected for all wafers

- 1) Existing of OF and position of IF
- 2) Surface finishing
- 3) Wafer defects
- 4) Inclusions
- 5) Beveling

7 Test methods

7.1 Diameter

Measuring the diameter except OF portion by means of thickness meter having a accuracy of user's requirements.

7.2 Thickness

Thickness at a center of wafer measured by thickness meter having an accuracy of $1 \mu\text{m}$ in according to ASTM Test Method F533.

7.3 Dimension of OF

Measurements of OF length as straight cut line of intersect with circle by means of a thickness meter having an accuracy of user's requirements.

7.4 Orientation of OF

Angle difference between orientation flat and reference orientation of which specified lattice-plane reflected as Bragg's condition measured by X-ray goniometer. Explanation details of measuring method is in Section 2. "Measuring Method", 3. "Measurement of face angle by X-ray".

7.5 TV5

TV5 indicates the variation of thickness in a wafer. The variation is the difference between the maximum and minimum value of thickness of the wafer. Thickness is measured at the center and at the 4-points of 6 mm inside from the edge of the wafer.

7.6 Warp

Warp is one of indications that shown the deformation of a wafer and defined as the maximum difference of the median surface for an unclamped wafer from a reference plane as shown in figure 2. Reference plane is defined as 3-points focal plane. Warp is measured by optical flatness equipment.

7.7 Defects of propagation surface

Surface defects on wafer shall be inspected using the method which explained on Section 2. "Measuring method", 4. "Appearance Inspection".

7.8 Inclusions

Inclusions shall be performed using reflection light from wafer surface under illuminating high intensity optically condensed light in clean environment of dark background for preventing disturbing light.

7.9 Roughness of back surface

Measurement of surface roughness achieves by contact-needle type or appropriate optical equipment.

7.10 Orientation

Relative measurements between reference index plane and measured angle by X-ray goniometer.

7.11 Curie temperature

Explanation details in Section 2. "Measuring Method", 1. "Measuring method of Curie temperature".

7.12 Lattice constant

Explanation details in Section 2. "Measuring Method", 2. Measurement of lattice constant (Bond method)".

8 Identification, labeling, packaging, delivery condition

8.1 Packaging

All wafers should be packaged in a manner as to avoid any contamination, chipping and damages during transportation or keeping. Special packaging requirements shall be subject to agreement between user and supplier.

8.2 Identification, labeling

All wafers should be packaged in specified container on which labeled with following printed items.

- a) Supplier's name or trade mark
- b) A name of single crystal
- c) Orientation angle
- d) Manufacturing lot number
- e) Quantity

8.3 Delivery condition

Additional requiring items or documentation for inspection are negotiable by user and maker.

Section 2 Measuring method

1 Measuring method of Curie temperature

Curie temperature of crystal should be measured by thermal analysis (DTA: Differential Thermal Analysis, or DSC : Differential Scanning Calorimetric Analysis) or dielectric constant method. These methods occasionally may include discrepancy of values by different measuring condition and/or apparatus. General measuring method shall be specifying herein. Customers using wafers from several suppliers, checking of correlations shall be required between measured values.

1.1 DTA method

The DTA (Differential Thermal Analysis) method⁴⁾ is based on the endothermic or exothermic reaction which is observed when a single crystal transits from ferroelectrics to paraelectrics. The sample and the standard material are set symmetrically in a oven as shown in Figure 3 and heated up at constant speed, while measuring the differential temperature between the both material as a function of temperature.

In this case, the standard material should be thermally stable. α - Al_2O_3 is usually used because of its stability. The exothermic reaction occurs at the phase transition temperature of the sample, which produces the temperature difference between the sample and the standard material. The Curie temperature is defined with the temperature at which the temperature difference arises.

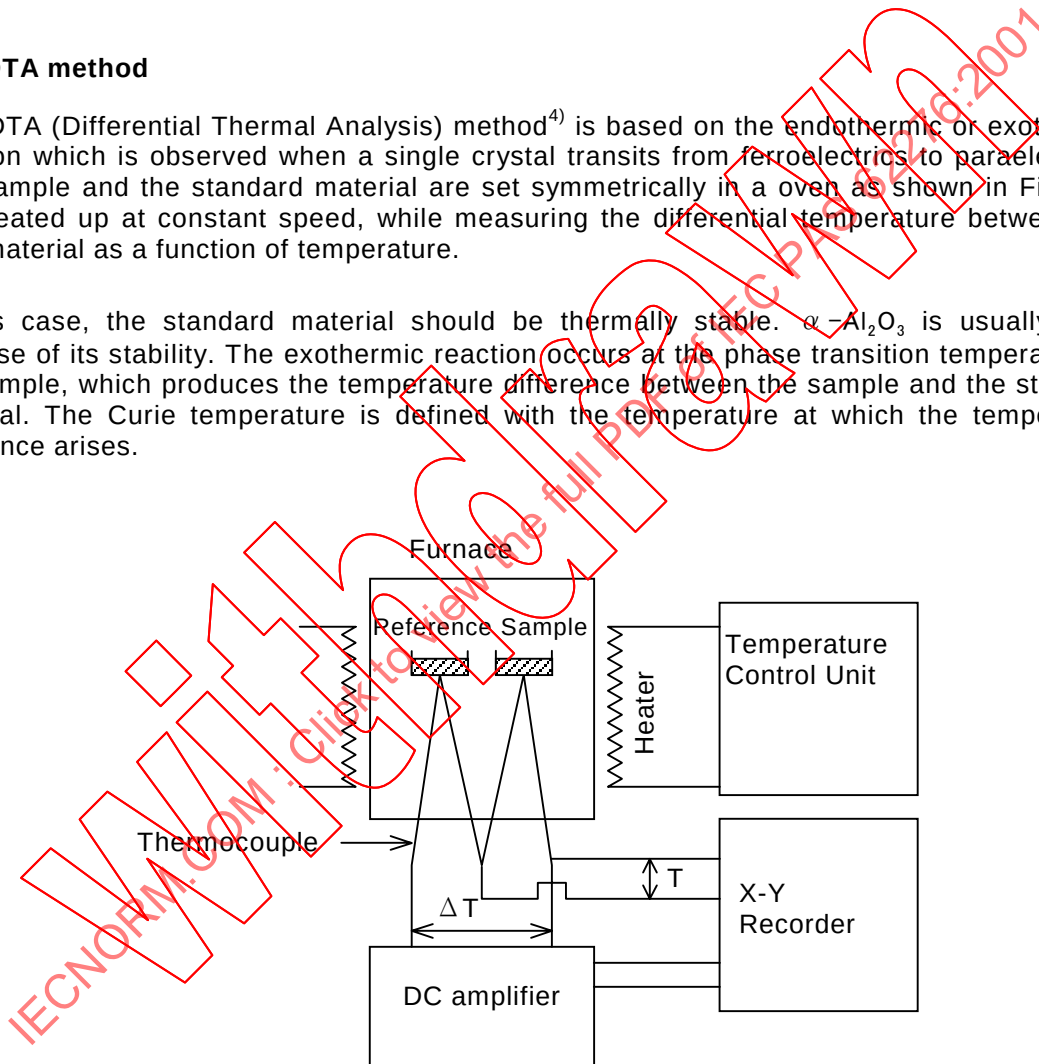


Figure 3 Schematic of DTA system

1.2 Dielectric constant method

The dielectric constant method utilizes the sharp change of the dielectric constant which occurs when a single crystal transits from ferroelectrics to paraelectrics. As shown in Figure 4 the electrode of Pt or Ag-Pd is formed on the sample in order that the electric field should be applied in the direction of C axis. As heating the sample in the oven at constant speed, the change of capacitance of the sample is measured by LCR-meter and the temperature profile as well. The temperature at which the capacitance shows maximum value can be defined as the Curie temperature.

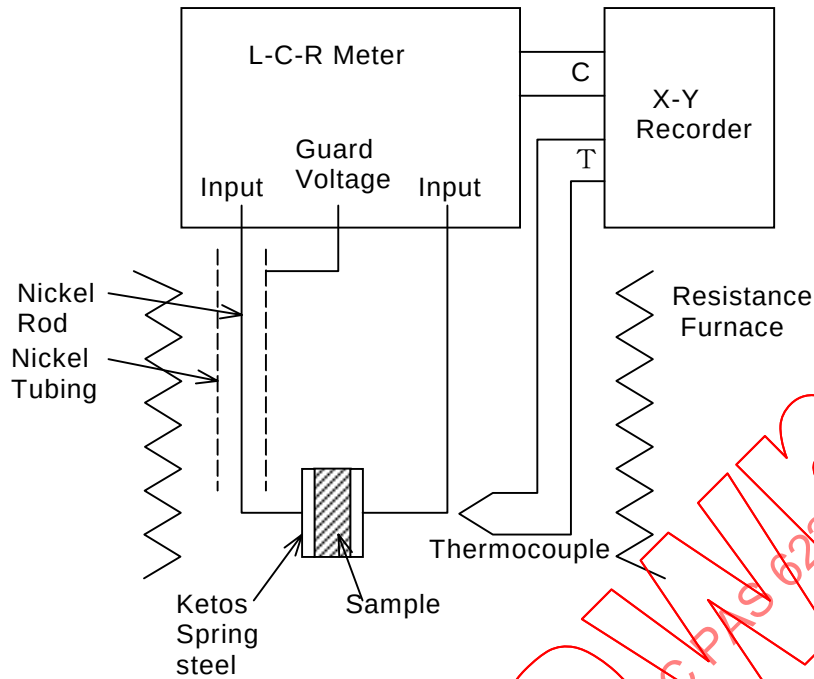


Figure 4 Schematic of dielectric constant measuring system

2 Measurement of lattice constant (Bond method)

Between SAW velocity and chemical compositions are correlated. Chemical compositions reflect lattice constants. If we control SAW velocity 10^{-4} range, lattice constants must control 10^{-5} range. The measurement of lattice constants is demanded the accuracy better than 10^{-6} .

The X-ray diffraction of crystals is used in order to their measure their lattice constants. The measurement is utilized the Bragg's law as follows

$$2d \sin \theta = n\lambda$$

where d is lattice spacing, θ is Bragg angle, and λ is wavelength of X-ray.

If λ is given, d and lattice constants are determined by measurements θ . $\Delta d/d$ is given in

$$\frac{\Delta d}{d} = -\cot \theta \times \Delta \theta$$

the form of the following differential of the Bragg's law with respect to θ

This equation shows that for a given precision of measurement $\Delta \theta$.

$\Delta \theta$ has to be measured correctly within a second in order to measure $\Delta d/d$ on a scale of 10^{-6} - 10^{-7} . In 1960, Dr. Bond developed to measure closely the value of lattice constants.

In Bond method, by making symmetric diffraction called plus-side and minus-side occurred in the same lattice face and finding the value of ω_1 and ω_2 from the peaks of rocking curve as the figure 5 shows, θ is measured as follows

Thus elements such as off-center error, absorption error zero error are eliminated

$$\theta = \left(\frac{1}{2}\right) \times (180 - |\omega_1 - \omega_2|)$$

theoretically. Furthermore we correct temperature correction, refraction correction, divergence correction, Lorentz-polarization correction.

In case of LiTaO_3 , We measured Miller index (330) by Bond method⁵⁾. Lattice constants of a axis is calculated as follows

$$a = 6d_{330}$$

All kinds of correction, lattice constants of LiTaO_3 is determined on a scale of 10^{-6} - 10^{-7} .

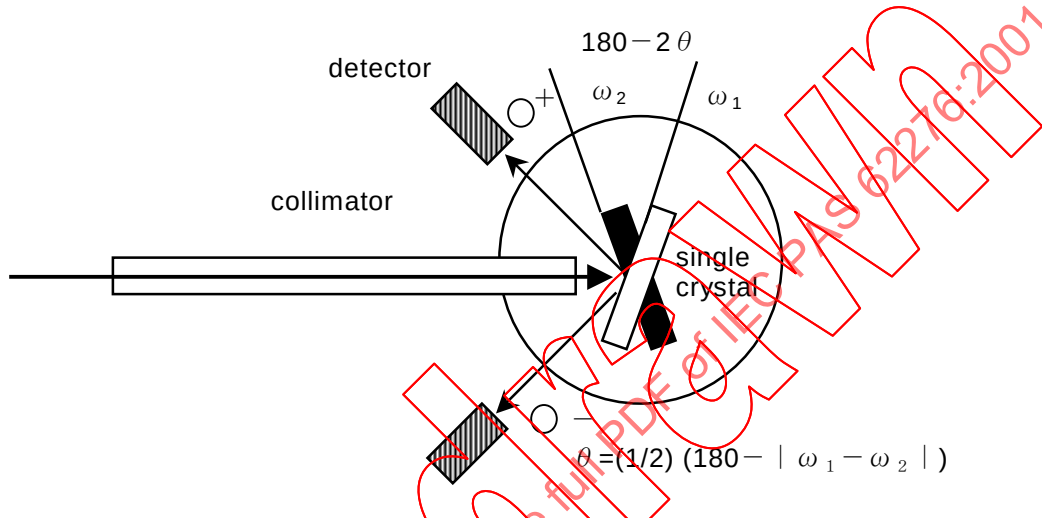


Figure 5 The principle of Bond method

3 Measurement of face angle by X-ray

3.1 Measurement

$$n\lambda = 2d \sin \theta$$

A face angle should be measured by X-ray diffraction goniometer. We assume that distance between each lattice face is d , wave length is λ , an optional integer is n . X-ray reflect by Bragg angle θ which is shown following formula.

X-ray from a source is made thin beam by a slit and a reflected crystal plate. A X-ray detector is attached at the position of Bragg angle 2θ . X-ray reflect when incidence angle is same as Bragg angle and detector can perceive X-ray. At the angle position which detector perceive we can measure the angle between a crystal plate surface and a lattice face by a goniometer.

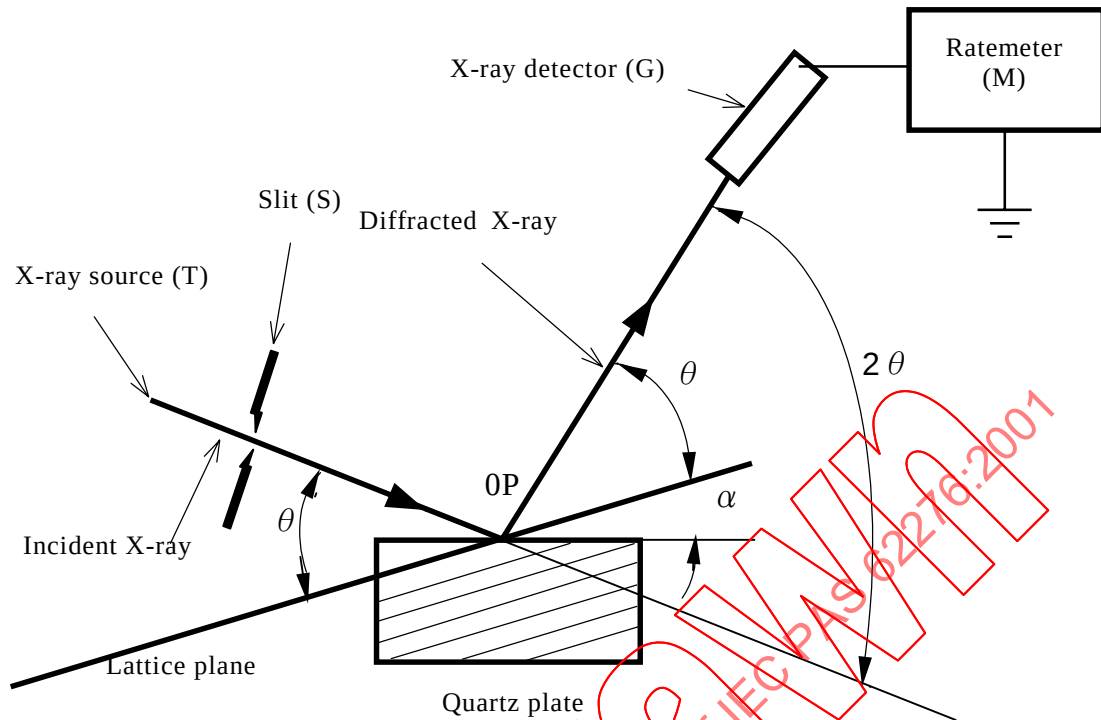


Figure 6 Measurement method by X-ray

3.2 Measuring method

The angle starting point determined by a standard sample before measuring.

The face angle of samples are measured by X-ray diffraction. The face angle of samples are calculated from the measuring values and the standard sample face angle.

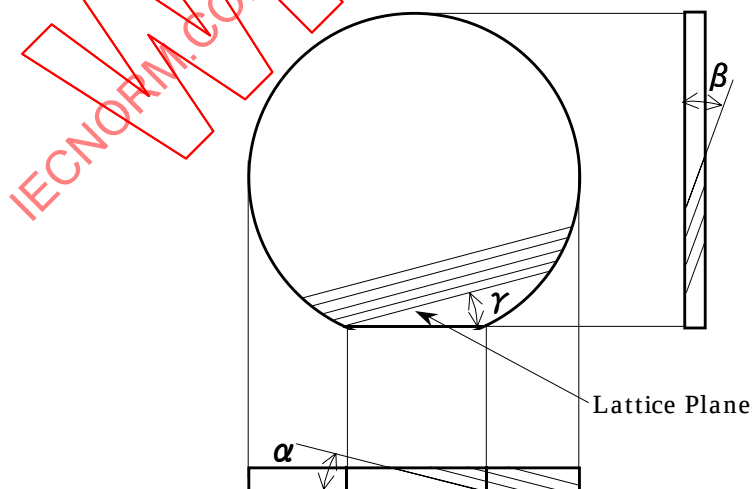


Figure 7 Relationship between cut angle and lattice face

3.3 Measuring direction of a wafer face

The angles should be measured about two directions as below.

Parallel to the OF (Y-direction): α (+ α direction is anticlockwise)

Perpendicular to the OF (X-direction) : β

3.4 Measuring direction of an orientation flat face

The angle (γ) should be measured.

3.5 Reference face of cutting face

Reference face of cutting face are shown as below.

Table 3 Reference face of cutting face

Materials	Cutting Orientation	Reference of Cutting Face	Face (α)	Cutting Face (β)	OF Reference Face	OF Face (γ)
LN	128Y° -X	(0 -1 4)hex	0	0	(2 -1 0)hex	0
LN	Y-Z	(0 3 0)hex	0	0	(0 0 6)hex	0
LN	64Y° -X	(0 1 8)hex	+4° 46'	0	(2 -1 0)hex	0
LT	X-112° Y	(2 -1 0)hex	0	0	(0 1 2)hex	+10° 44'
LT	X-112° Y	(2 -1 0)hex	0	0	(0 -1 10)hex	-5° 02'
LT	X-112° Y	(2 -1 0)hex	0	0	(0 3 0)hex	22° 12'
LT	36Y° -X	(0 1 2)hex	-3° 04'	0	(2 -1 0)hex	0
LT	42Y° -X	(0 1 2)hex	-9° 05'	0	(2 -1 0)hex	0
LBO	45X° -Z	(1 1 0)tetra	0	0	(0 0 1)tetra	0
Quartz	ST-X	(0 1 1)hex	+4° 32'	0	(2 -1 0)hex	0

4 Appearance inspections

4.1 The method of surface finish inspection

The wafer surface shall be mirrored polish such that the surface acoustic wave input or output transducer is formed successfully. As a general rule, the visual inspection of the surface finish shall be carried out according to the inspection items given below.

Scratches

Chipping

Cracks

Contamination

Others: Dimple, Pit, Orange Peel

The quality expression required in the visual inspection are, (1) numeral characteristics expression, (2) technical vocabulary expression, (3) illustration, photographic expression, (4) representative samples, these expression shall show clearly whether the product has passed or failed by the inspections according to defined acceptance criteria.

There are two methods for visual inspection by using naked eye or using microscope, the supplier shall use either or both method as required.

The visual inspection shall be carried out in the clean bench arranged so that the ambient scattered light is intercepted, and the light emitted from a high brightness white light tungsten or halogen source is condensed optically and focus it on the wafer, the scattering light from the above-mentioned defects existed on the wafer surface or edge is inspected by naked eye inspection.

The illumination intensity for light source, inspection area and boundary standard sample shall be stipulated in the clearest practical manner between user and supplier. When it is possible that the defect criteria is expressed by numeral characteristics expression, the supplier shall make better use of the defect criteria. In the meantime, if it is possible that the supplier make preparation for representative samples, it is desirable to use representative samples so that the dispersion and tolerance of inspection judgement can be improved.

When it is needed to fully inspect the defect which was detected by visual inspection and to find a minute defect which was not detected by visual inspection, the microscopic inspection shall be carried out concurrently. In the former case, the stereomicroscope shall be used in order to magnify the inspection object, and in the latter case, the metallurgical microscope shall be used by detecting the light reflected from wafer surface.

Annex A (normative)

Expression using Eulerian angle for piezoelectric single crystals

Material properties of piezoelectric single crystals, such as piezoelectric constants (e_{11}), elastic constants (c_{11}), dielectric constants (ϵ_{11}), have been generally determined in terms of the rectangular axes (X, Y, Z) which have been defined in place of the crystal axes (a, b, c) of crystallography for all crystal system of 23 point groups. On the other hand, SAW substrate show characteristic properties of SAW propagation according to cut angle of their substrate and direction of SAW propagation on the substrate. When theoretical treatments have been done for SAW propagating characteristics along x_1 direction on a given piezoelectric crystal substrate which is cut perpendicular to x_3 axes, as shown in figure 8, their characteristics have been usually calculated using the new rectangular coordinate system of the axes (x_1, x_2, x_3) which are converted from the rectangular axes (X, Y, Z) using Eulerian angles (ϕ , θ , ψ) as shown in Figure 8.

Figure 8 shows relationship between crystal orientations and Eulerian angles for typical SAW substrate in the table 4. SAW propagation system on piezoelectricity substrate is shown in Figure 9. Figure 10 shows the relationship between direction of piezoelectricity for typical SAW substrate and Eulerian angles.

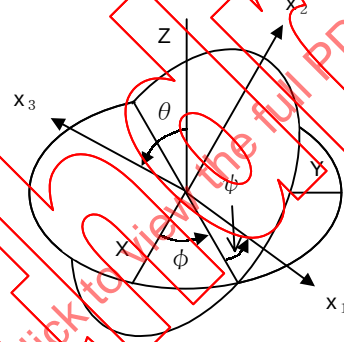


Figure 8 Eulerian angles of Right handed system

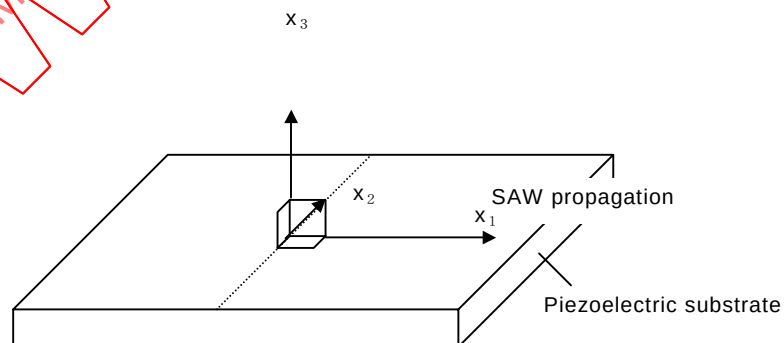


Figure 9 SAW propagation system on piezoelectricity substrate

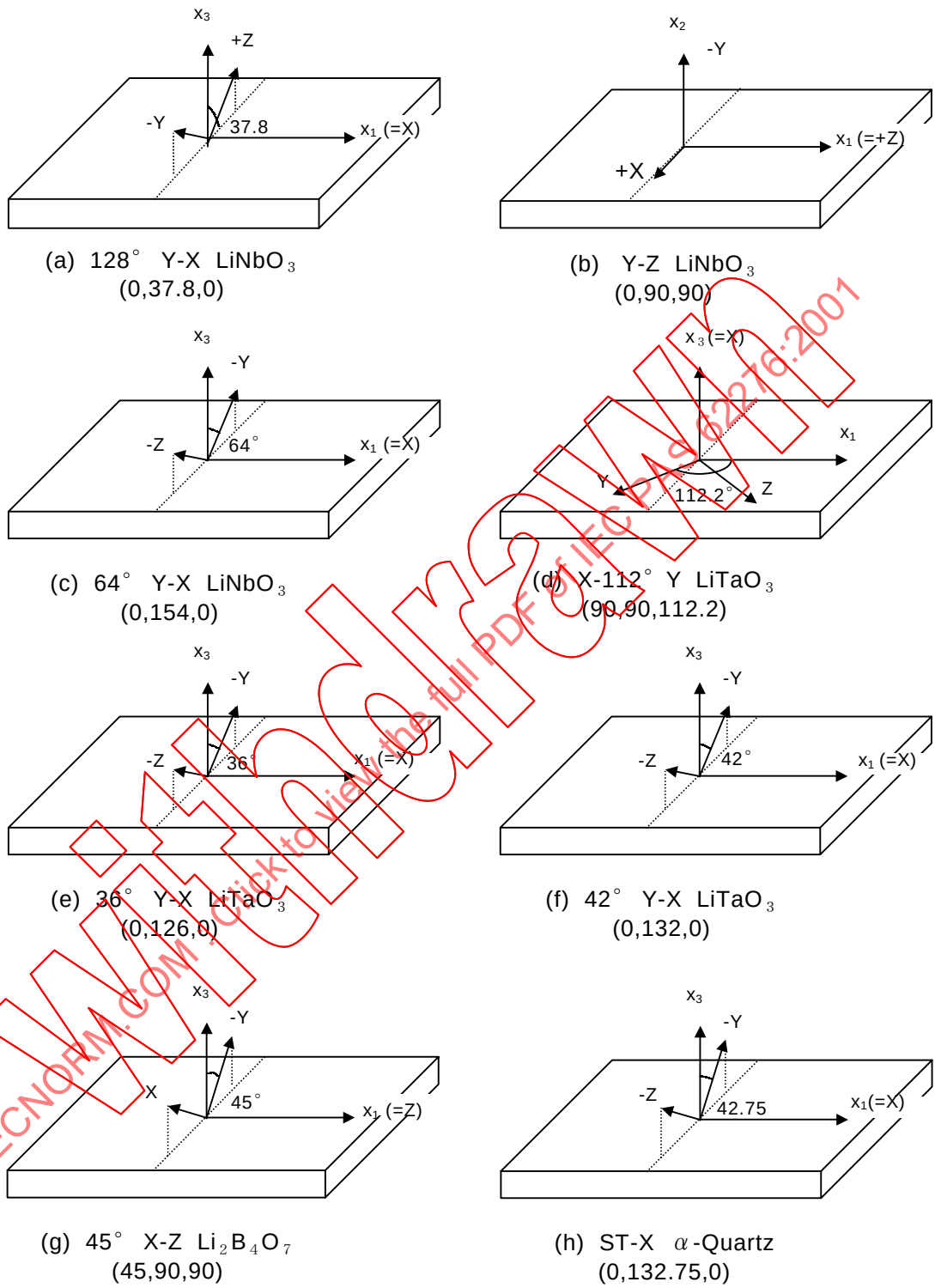


Figure 10 Direction of piezoelectricity representative substrate shown by Eulerian angles

Table 4 SAW wafer abbreviated terms for typical SAW substrate

Abbreviated terms	Cut angle and propagation	Chemical formula and Euler angle
128° Y-X LN	127.8° Rotated Y cut X SAW propagation Lithium niobate substrate	LiNbO ₃ (0, 37.8, 0)
Y-Z LN	Y cut Z SAW propagation Lithium niobate substrate	LiNbO ₃ (0, 90, 90)
64° Y-Z LN	64° Rotated Y cut X SAW propagation Lithium niobate substrate	LiNbO ₃ (0, 154, 0)
X-112° Y LT	X cut 112.2° Rotated Y SAW propagation Lithium tantalate substrate	LiTaO ₃ (90, 90, 112.2)
36° Y-X LT	36° Rotated Y cut X SAW propagation Lithium tantalate substrate	LiTaO ₃ (0, 126, 0)
42° Y-X LT	42° Rotated Y cut X SAW propagation Lithium tantalate substrate	LiTaO ₃ (0, 132, 0)
45° X-Z LBO	45° Rotated X cut Z SAW propagation Lithium tetraborate substrate	Li ₂ B ₄ O ₇ (45, 90, 90)
ST-X α -Quartz	ST cut X SAW propagation α -Quartz Crystal	SiO ₂ (α -Quartz) (0, 132.75, 0)

Annex B (informative)

Crystal growth method

Crystal pulling method (Czochralski method)

This is a method to grow a single crystal by dipping a seed crystal into the melt in a crucible, rotating a crystal or a crucible or the both of them, then pulling up a crystal from the melt. This method is called alias Czochralski method by the name of the person who tried it for the first time⁶⁾. Started industrially from germanium (Ge) and silicon (Si) single crystal, it has been used to manufacture various single crystals. Concerning LN and LT, a large-scale single crystal was manufactured for the first time by the Bell telephone lab and Old Soviet Union lab in 1965.

While there are r.f. induction heating and resistance heating as the heating method, regarding LN and LT, single crystals are grown generally by r.f. induction heating method. Figure 11 shows a sketch of an apparatus using r.f. heating.

It is general to utilize LiNbO₃ (LiTaO₃) polycrystals as a starting material, which are obtained by mixing Li₂CO₃ and Nb₂O₅ (Ta₂O₅) with Li/Nb (Li/Ta) mole ratio of 0.93 to 0.95, and then, calcinating the compound after press forming it. The polycrystals are made to melt by charging them into the crucible and heating inductively the crucible itself.

Next, after dipping the leading end of a seed crystal cut out in the direction of crystal axis of pulling-up onto the liquid surface where raw materials are melting, the seed crystal is rotated in the vicinity of the melting point. Then, after the temperature of the melt is controlled to the

state of necking down for the leading end and the state of equilibrium is confirmed, pulling-up is begun.

The temperature of the melt is generally reduced little by little until a shoulder part is formed and fixed diameter is obtained. And then controlling the diameter the crystal is pulled up until fixed length is obtained. Finally, the crystal is pulled off from the melt upward and the state as ingot of a single crystal is obtained.

Single-domain

Ferroelectric crystal can shift the state of multi-domain to single domain by external DC supply and this operation is called "poling". In typical ferroelectric material, a specific temperature exists at the level in which ferroelectric phase shifts to paraelectric phase and it is called Curie temperature. Below Curie temperature, the value of DC supply necessary for shifting to single-domain is so high that poling is in fact impossible. Therefore poling is generally operated above Curie temperature with external DC supply.

In the case of LN and LT, poling is done by forming an electrode on the face perpendicular to Z or Z' axis which is concretely the direction of polarization, heating up crystals to above Curie temperature then cooling down the crystals with holding external DC supply. To remove thermal stress from LN and LT which are grown by the crystal pulling method as a preparatory process for poling processing, anneal processing is carried out at the temperature below melting point. For LT, after anneal operation at about 1300°C, it is cooled down to room temperature to form an electrode by conductive metal paste. Then the temperature is raised to about 650°C with adding some mV/cm of DC supply and cooled down again to cause single-domain. Since Curie temperature for LN is high, it is general to conduct the both of anneal processing and poling processing simultaneously. Then single domain is obtained by forming an electrode on LN which is grown by the crystal pulling method after anneal processing at about 1200°C with adding some mV/cm of DC Supply.

Congruent composition

In general, there are two kind of crystal; one is the crystallization made by the stoichiometric composition and the other is the crystallization made within a certain range of the composition to form solid solution. In the latter case, when crystallization is made from melt, the composition of single crystal is determined by the composition of melt and the composition of solidified single crystal changes except a certain composition. In producing a single crystal, the raw material could be adjusted by bringing the composition of melt and solidified single crystal together, which is generally called congruent composition. However, congruency is strictly defined as compositional equilibrium in a thermodynamic equilibrium. We also can have substantial volatilization of certain compound (e.g. Li_2O). The axially uniform composition in a crystal therefore is not necessarily congruent, and depends on the particular growth apparatus used. The example given in the informative Annex shows a report for compositional problem of LiNbO_3 crystals.

The congruent composition is from 0.93 to 0.95 approximately in Li/Nb mole ratio and Li/Ta mole ratio for LN and LT. When LN and LT are grown in the composition which shifts from the congruent composition in the process of the crystal pulling method, the composition changes in the crystals and Curie temperature, lattice constants, and refractive index, etc. change similarly, and wave velocity which is important in SAW device also changes with the change of the composition. In Figure 12 which is an example of LN, as Li/Nb ratio increases, wave velocity quickens while wave velocity slows as Li/Nb ratio decreases. Moreover, on the top and the bottom of single crystal, except the congruent composition, the composition and the wave velocity change. The congruent composition here is ②.

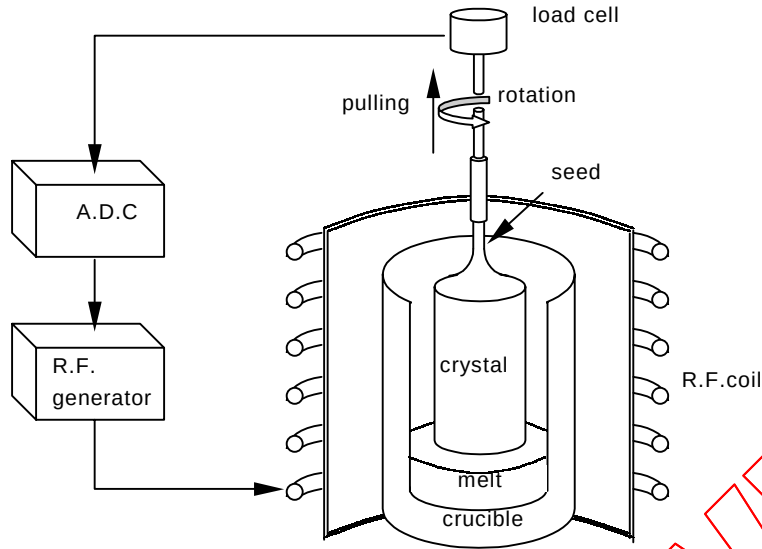


Figure 11 Czochralski method

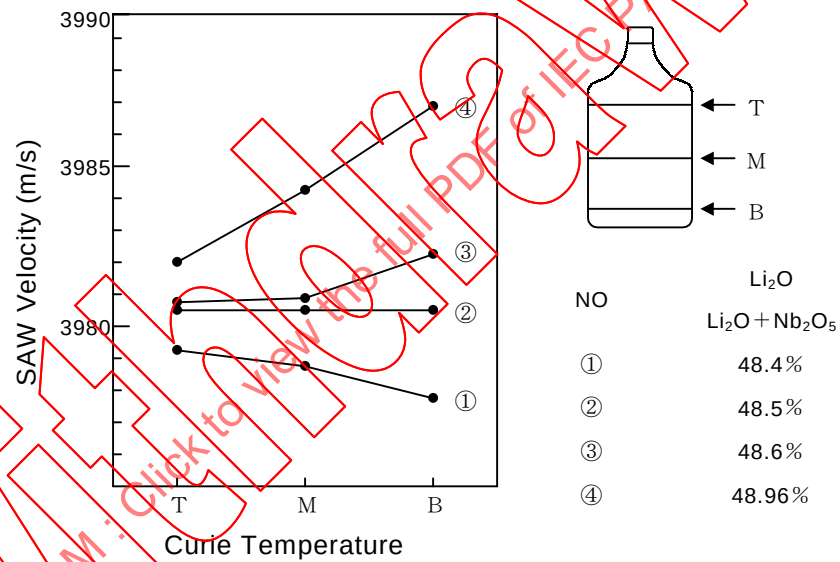


Figure 12 Relationship between SAW velocity and sample position⁷⁾

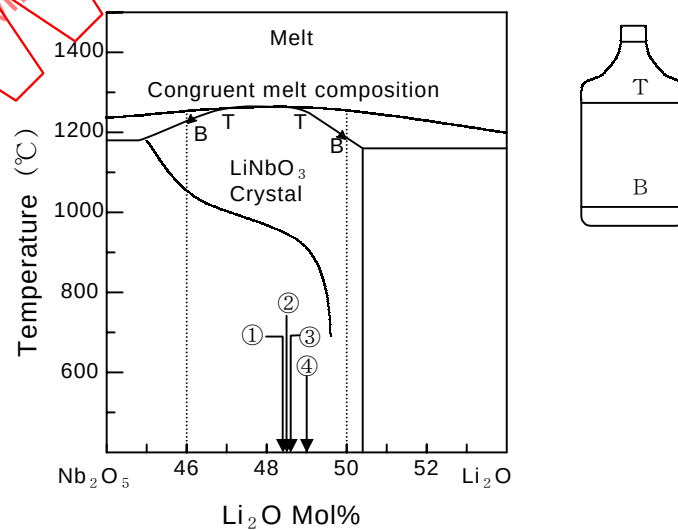


Figure 13 Relationship between LN composition and temperature⁷⁾

Vertical Bridgman method

Lithium tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$: LBO) is a stoichiometric composite, melting congruently, and can be crystallized by melt growth method as a result that no phase transition exists under melting temperature. Poling treatment is unnecessary because of not ferroelectrics.

In the first stage, Czochralski method applied to crystal growth, but it was difficult to obtain industrially usable large crystals. Recently, Vertical Bridgman method was induced in this field, and has been refined to provide large crystals for practical production of SAW application.

Vertical Bridgman method means a crystal growth method by unidirectional solidification with moving crucible filled with melted raw material through the furnace having a vertical temperature distribution. Easy for operation, simple and low cost facility construction comparing to other melt growth methods will be the most practical peculiarity. Crystal growth can be operated under unmanned environment, because diameter control should be unnecessary. By the hand, twinning, cracking, or polycrystalizing are occasionally happened by sticking between wall and crystal, or strain caused by difference of expansion ratio between crystal and crucible material.

Schematic drawings of furnace and temperature distribution for LBO crystal growth is shown in Figure 14 and Figure 15. Melting temperature is comparatively low, 917°C , and this allows to use resistance heating furnace for crystal growth. Cylindrical thin plate platinum crucibles are generally used for relaxing strains by anisotropy in thermal expansion. Stoichiometric polycrystalline blocks or powder are used as nutrient material.

It is reported that single crystals with 2 to 4 inches in diameter orientated $\langle 001 \rangle$, $\langle 100 \rangle$, $\langle 110 \rangle$ and $\langle 011 \rangle$ were obtained under the conditions of growth rates of 0.2 to 0.3mm/h, melting temperatures in the range of 950 to 1100°C , and temperature gradient of 10 to $20^\circ\text{C}/\text{cm}$ at solid-liquid interface.

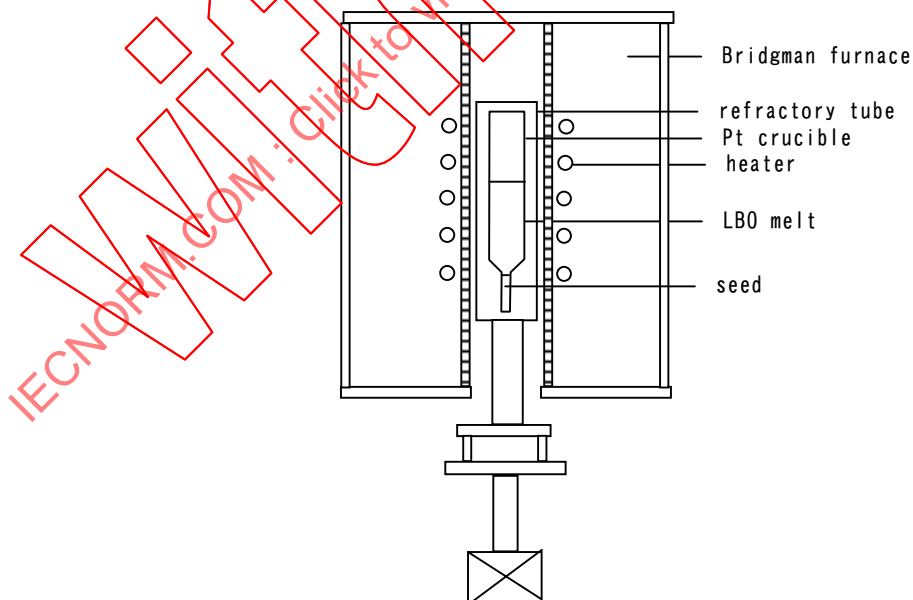


Figure 14 Schematic drawing of Vertical Bridgeman method

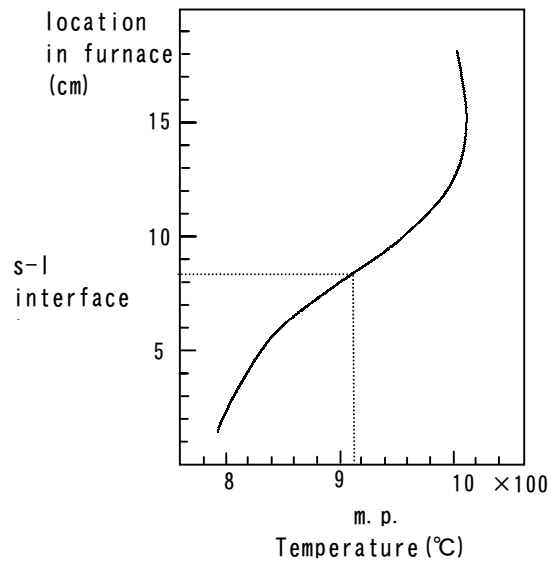


Figure 15 An example of temperature distribution in a furnace

Annex C (informative)

Manufacturing lot

The SAW wafers manufacturing process consist of main two processes. Former process is growing LN, LT, LBO and quartz single crystals which are materials of those wafers and latter is cutting, lapping, polishing process of wafers. By this reason, when a maker identify manufacture lot it is necessary to give a traceability of former process and latter process.

In the case of LN, LT, LBO single crystals only one crystal can be grown by one run and a cutting, lapping, polishing process lot correspond to growing lot generally. However in the case of quartz crystal, many quartz crystals can be grown by one run, a growing lot has several cutting, lapping, polishing lots. Lot identifications of former process and latter process should be given.

Annex D (informative)

Explanation of TTV, LTV and Sori

In recent years, various mobile communication systems have been developed world wide and the carrier frequency of 1 GHz is now extending to 2 GHz. RF filter which is one of SAW filters is used for the high frequency telecommunication systems. Production of RF filter is realized by adopting the fine lithograph technique and the fine electrode fabrication technique from LSI fabrication technology. Therefore the flatness of wafers such as LN and LT for high frequency applications appears to be a very important parameter to characterize the quality of wafers. Flatness of those wafers is often defined as TTV, LTV, Sori, and Bow other than TV5 and Warp, and the definition of those parameters are described below

TTV: Total Thickness Variation

Measurement of TTV is performed under clamped condition and its datum plane is defined as the back surface of a wafer. TTV is the difference between maximum height(A) and the minimum height(B) in reference with the back surface.