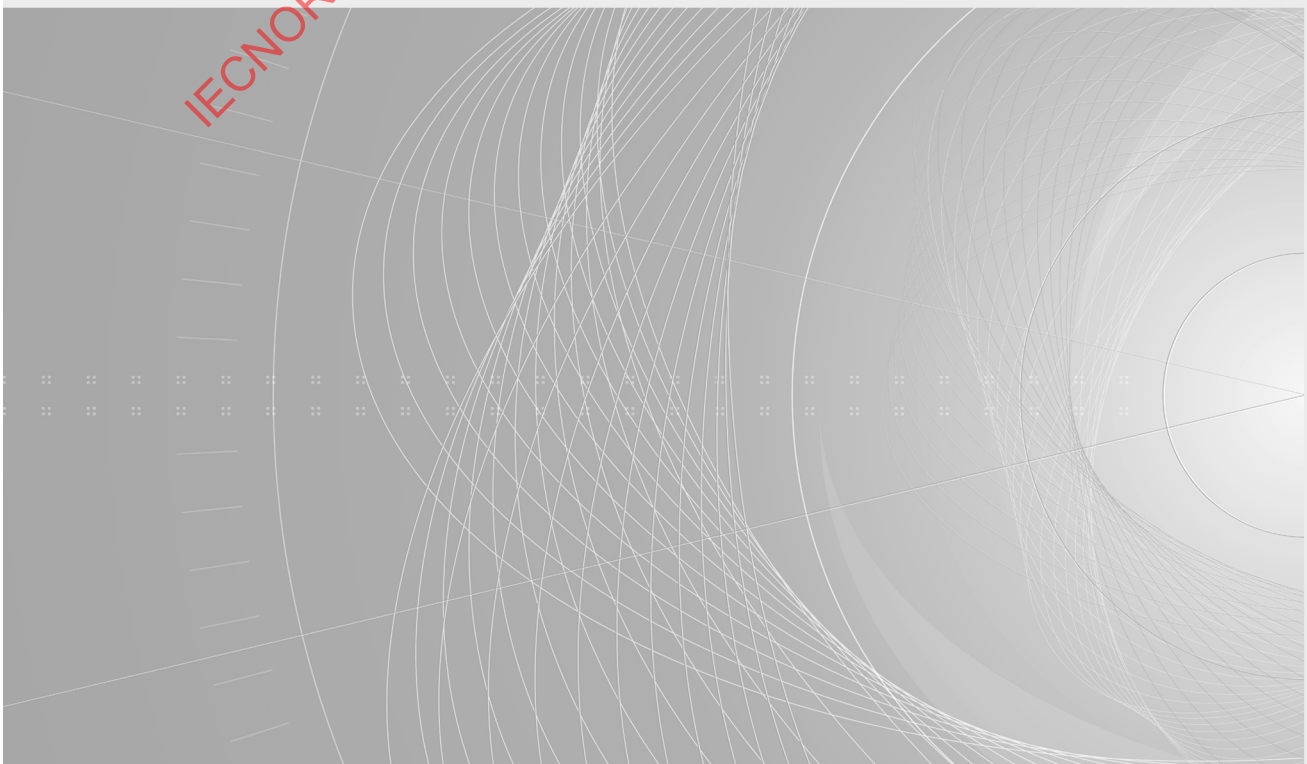
IEC 62276

Edition 3.0 2016-10

INTERNATIONAL STANDARD

Single crystal wafers for surface acoustic wave (SAW) device applications – Specifications and measuring methods



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INTERNATIONAL
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CONTENTS

INTERNATIONAL ELECTROTECHNICAL COMMISSION

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SINGLE CRYSTAL WAFERS FOR SURFACE ACOUSTIC WAVE (SAW) DEVICE APPLICATIONS – SPECIFICATIONS AND MEASURING METHODS

FOREWORD

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International Standard IEC 62276 has been prepared by IEC technical committee 49: Piezoelectric, dielectric and electrostatic devices and associated materials for frequency control, selection and detection.

This third edition cancels and replaces the second edition of IEC 62276 published in 2012. It constitutes a technical revision.

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

– Corrections of Euler angle indications in Table 1 and axis directions in Figure 3.
– Definition of "twin" is not explained clearly enough in 3.3.3. Therefore it is revised by a more detailed definition.
– Etch channels maximum number at quartz wafer of seed which do not pass through from surface to back surface are classified for three grades in 4.2.13 a). Users use seed portions of quartz wafers for devices. They request quartz wafers with less etch channels

in seeds to reduce defects of devices. The classification of etch channels in seed may prompt a rise in quartz wafer quality.

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

CDV	Report on voting
49/1144/CDV	49/1170/RVC

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

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- withdrawn,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.

A bilingual version of this publication may be issued at a later date.

INTRODUCTION

A variety of piezoelectric materials are used for surface acoustic wave (SAW) filter and resonator applications. Prior to an IEC meeting in 1996 in Rotterdam, wafer specifications were typically negotiated between users and suppliers. During this meeting, a proposal was announced to address wafer standardization. This standard has been prepared in order to provide industry standard technical specifications for manufacturing piezoelectric single crystal wafers to be used in surface acoustic wave devices.

SINGLE CRYSTAL WAFERS FOR SURFACE ACOUSTIC WAVE (SAW) DEVICE APPLICATIONS – SPECIFICATIONS AND MEASURING METHODS

1 Scope

This document applies to the manufacture of synthetic quartz, lithium niobate (LN), lithium tantalate (LT), lithium tetraborate (LBO), and lanthanum gallium silicate (LGS) single crystal wafers intended for use as substrates in the manufacture of surface acoustic wave (SAW) filters and resonators.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60758:2016, *Synthetic quartz crystal – Specifications and guidelines for use*

ISO 2859-1: 1999, *Sampling procedures for inspection by attributes – Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

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

Note 1 to entry: The term “as-grown” indicates a state prior to mechanical fabrication.

Note 2 to entry: See IEC 60758 for further information concerning crystalline quartz.

3.1.2
lithium niobate
LN
single crystals approximately described by chemical formula $LiNbO_3$, grown by Czochralski (crystal pulling from melt) or other growing methods

3.1.3
lithium tantalate
LT
single crystals approximately described by chemical formula $LiTaO_3$, grown by Czochralski (crystal pulling from melt) or other growing methods

3.1.4
lithium tetraborate
LBO
single crystals described by the chemical formula to $Li_2B_4O_7$, grown by Czochralski (crystal pulling from melt), vertical Bridgman, or other growing methods

3.1.5
lanthanum gallium silicate
LGS
single crystals described by the chemical formula to $La_3Ga_5SiO_{14}$, grown by Czochralski (crystal pulling from melt) or other growing methods

3.2 Terms and definitions related to LN and LT crystals

3.2.1
Curie temperature
T_c
phase transition temperature between ferroelectric and paraelectric phases measured by differential thermal analysis (DTA) or dielectric measurement

3.2.2
single domain
ferroelectric crystal with uniform electrical polarization throughout (for LN and LT)

3.2.3
polarization process
electrical process used to establish a single domain crystal

Note 1 to entry: The polarization process is also referred to as “poling”.

3.2.4
reduction process
REDOX reaction to increase conductivity to reduce the harmful effects of pyroelectricity

3.2.5
reduced LN
LN treated with a reduction process

Note 1 to entry: Reduced LN is sometimes referred to as “black LN”.

3.2.6
reduced LT
LT treated with a reduction process

Note 1 to entry: Reduced LT is sometimes referred to as “black LT”.

3.3 Terms and definitions related to all crystals

3.3.1
lattice constant
length of unit cell along a major crystallographic axis measured by X-ray using the Bond method

3.3.2
congruent composition
chemical composition of a single crystal in a thermodynamic equilibrium with a molten solution of the same composition during the growth process

3.3.3
twin
two or more same single crystals which are combined together by the law of symmetrical plane or axis

Note 1 to entry: Twins exhibit symmetry that may be classified as reflection across a mirror plane (twin plane), rotation around an axis (twin axis), or inversion through a point (twin center).

Note 2 to entry: Optical twins（growth twins） and electrical twins（transformation twins） are the most relevant to SAW wafers. Optical twins arise from defects related to growth. Electrical twins may result from extreme conditions (temperature and pressure, for example) during processing.

3.4 Flatness

3.4.1
fixed quality area
FQA
central area of a wafer surface, defined by a nominal edge exclusion, X, over which the specified values of a parameter apply

Note 1 to entry: The boundary of the FQA is at all points (e.g. along wafer flats) the distance X away from the perimeter of the wafer of nominal dimensions.

3.4.2
reference plane
plane depending on the flatness measurement and which can be any of the following:

a) for clamped measurements, the flat chuck surface that contacts the back surface of the wafer;

b) for without clamped measurememts, three points at specified locations on the front surface within the FQA;

for without clamped measurememts, the least-squares fit to the front surface using all measured points within the FQA;

3.4.3
site
square area on the front surface of the wafer with one side parallel to the OF

Note 1 to entry: Flatness parameters are assessed either globally for the FQA, or for each site individually.

3.4.4
thickness variation for five points
TV5
measure of wafer thickness variation defined as the maximum difference between five thickness measurements

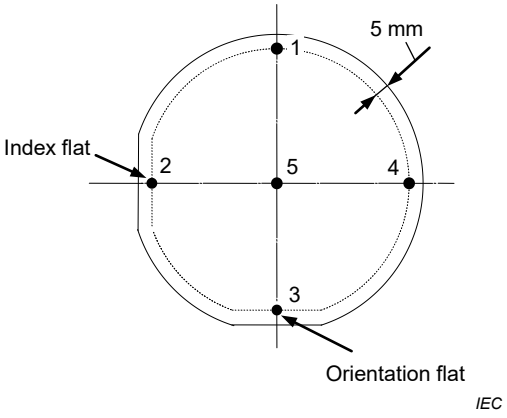


Figure 1 – Wafer sketch and measurement points for TV5 determination

Note 1 to entry: Thickness is measured at the centre of the wafer and at four peripheral points shown in Figure 1.

3.4.5
total thickness variation
TTV
difference between the maximum thickness and the minimum thickness

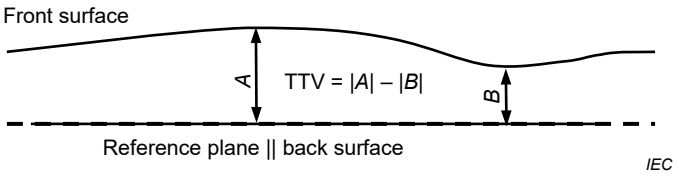


Figure 2 – Schematic diagram of TTV

Note 1 to entry: The maximum thickness is represented by the letter A and the minimum thickness is represented by the letter B in Figure 2.

Note 2 to entry: Measurement of TTV is performed under clamped conditions with the reference plane as defined in 3.4.2 a).

3.4.6
warp
maximum difference between a point on the front surface and a reference plane

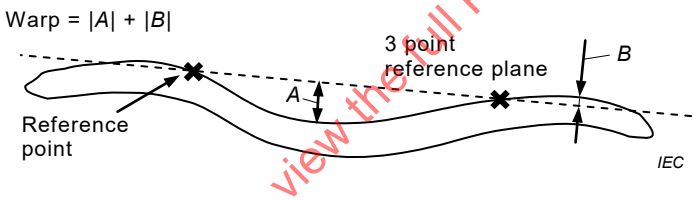


Figure 3 – Schematic diagram of warp

Note 1 to entry: Warp (shown in Figure 3) describes the deformation of an unclamped wafer.

Note 2 to entry: The reference plane is defined by 3-points as described in 3.4.2 b). Warp is a bulk property of a wafer and not of the exposed surface alone.

3.4.7
Sori
maximum difference between a point on the front surface and a reference plane

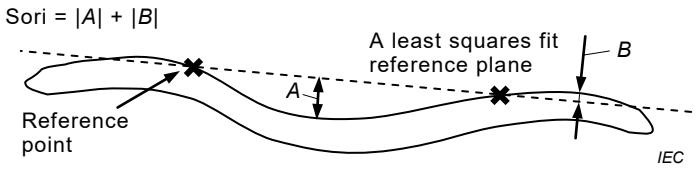
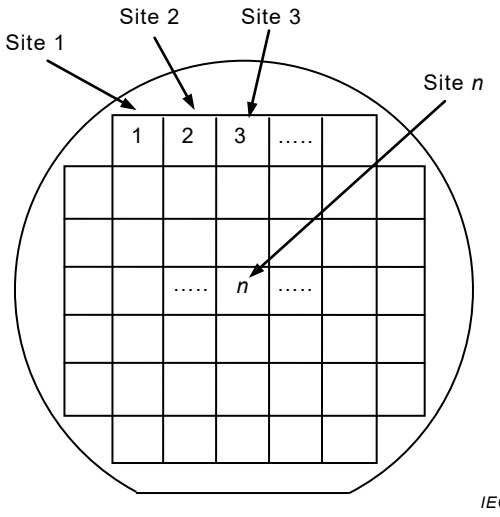


Figure 4 – Schematic diagram of Sori

Note 1 to entry: Sori describes the deformation of an unclamped wafer, as shown in Figure 4.

Note 2 to entry: In contrast to warp, in this case the reference plane is defined by a least-squares fit to the front surface (3.4.2 c)).

3.4.8
local thickness variation
LTV
variation determined by a measurement of a matrix of sites with defined edge dimensions



Note 1 to entry: All sites have their centres within the FQA.

Figure 5 – Example of site distribution for LTV measurement

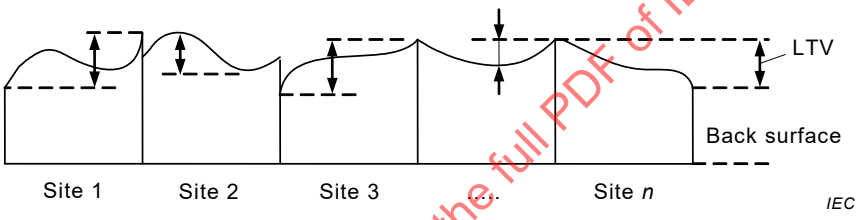


Figure 6 – LTV value of each site

Note 2 to entry: Measurement is performed on a clamped wafer with the reference plane as defined in 3.4.2 a). A site map example is shown in Figure 5. The value is always a positive number and is defined for each site as the difference between the highest and lowest points within each site, as shown in Figure 6. For a wafer to meet an LTV specification, all sites shall have LTV values less than the specified value.

3.4.9
percent local thickness variation
PLTV
percentage of sites that fall within the specified values for LTV

Note 1 to entry: As with the LTV measurement, this is a clamped measurement.

3.4.10
focal plane deviation
FPD
deviation measured relative to the 3-point reference plane

Note 1 to entry: The 3-point reference plane is defined in 3.4.2 b).

Note 2 to entry: The value obtained indicates the maximum distance between a point on the wafer surface (within the FQA) and the focal plane. If that point is above the reference, the FPD is positive. If that point is below the reference plane, the FPD is negative.

3.5 Definitions of appearance defects

3.5.1
contamination
foreign matter on a surface of wafer which cannot be removed after cleaning

3.5.2
crack
fracture that extends to the surface and may or may not penetrate the entire thickness of the wafer

3.5.3
scratch
shallow groove or cut below the established plane of the surface, with a length to width ratio greater than 5:1

3.5.4
chip
region where material has been removed from the surface or edge of the wafer

Note 1 to entry: The size can be expressed by its maximum radial depth and peripheral chord length.

3.5.5
dimple
smooth surface depression larger than 3 mm diameter

3.5.6
pit
non-removable surface anomaly

EXAMPLE A hollow, typically resulting from a bulk defect or faulty manufacturing process.

3.5.7
orange peel
large featured, roughened surface visible to the unaided eye under diffuse illumination

Note 1 to entry: This is also called pear skin.

3.6 Other terms and definitions

3.6.1
manufacturing lot
lot established by agreement between the customer and the supplier

3.6.2
orientation flat
OF
flat portion of wafer perimeter indicating the crystal orientation

Note 1 to entry: Generally, the orientation flat corresponds to the SAW propagation direction.

Note 2 to entry: Orientation flat is also referred to as the “primary flat” (see Figure 1).

3.6.3
secondary flat
SF
flat portion of wafer perimeter shorter than the OF

Note 1 to entry: When present, the SF indicates wafer polarity and can serve to distinguish different wafer cuts.

Note 2 to entry: Secondary flat is also referred to as the “suborientation flat” (see Figure 1).

3.6.4
back surface roughness
roughness which scatters and suppresses bulk wave spurious at back surface

3.6.5
surface orientation
crystallographic orientation of the axis perpendicular to the polished surface of wafer

3.6.6
description of orientation and SAW propagation
indication of the surface orientation and the SAW propagation direction, separated by the symbol "-"

Note 1 to entry: Specification of a 0° orientation is normally omitted.

Note 2 to entry: Typical examples for these expressions are shown in Table 1.

Note 3 to entry: Description of wafer orientation rule is shown at Annex A.

Table 1 – Description of wafer orientations

Material LT Quartz	LN	LT	Quartz crystal	LBO	LGS
Expression	128° Y-X Y-Z 64° Y-X	X-112° Y 36° Y-X	ST-X	45° X-Z	yxlt/48,5°/26,6°

3.6.7
ST-cut
cut direction of quartz to achieve zero temperature coefficient

3.6.8
tolerance of surface orientation
acceptable difference between specified surface orientation and measured orientation, measured by X-ray diffraction

3.6.9
bevel
slope or rounding of the wafer perimeter

Note 1 to entry: Bevel is also referred to as "edge profile".

Note 2 to entry: The process of creating a bevel is called "bevelling" or "edge rounding".

Note 3 to entry: The profile and its tolerances should be specified by the supplier.

3.6.10
diameter of wafer
diameter of circular portion of wafer excluding the OF and SF regions

3.6.11
wafer thickness
thickness measured at the centre of the wafer

4 Requirements

4.1 Material specification

4.1.1 Synthetic quartz crystal

A synthetic quartz crystal grown from Z-cut seed shall have an orientation within +5° of arc, and the wafer should consist of Z,+X,s growth region and seed (excepting –X growth region).

The quality of a synthetic quartz crystal conforms to or exceeds the following grades in accordance with IEC 60758.

– Infrared absorption coefficient α value Grade D
– Inclusion density (pieces/cm^3) Grade II
– Etch channel density (pieces/cm^2) Grade 2

4.1.2 LN

LN is a single domain material having a Curie temperature within the specified range.

4.1.3 LT

LT is a single domain material having a Curie temperature or lattice constant within the specified range.

4.1.4 LBO, LGS

Material not including twins.

4.2 Wafer specifications

4.2.1 General

The specifications listed in 4.2 apply in the absence of superseding agreements between user and supplier. These specifications are expected to evolve and change as existing processes are refined and new ones are developed. For wafers that are typically used in conjunction with a photolithographic stepper equipment, LTV is typically specified as one of the flatness criteria. When using projection lithography for full wafer exposure, FPD is often more relevant than TTV, as the system will perform a tilt correction referenced off the front surface. Sori is often more meaningful than warp since the least-squares derived reference plane used in that measurement typically provides a more accurate representation of the wafer surface.

4.2.2 Diameters and tolerances

– 76,2 mm ± 0,25 mm (Henceforth referred to as 76,2 mm wafer,commonly referred to as a “3 inch” wafer)
– 100,0 mm ± 0,5 mm (Henceforth referred to as 100 mm wafer)
– 125,0 mm ± 0,5 mm (Henceforth referred to as 125 mm wafer)
– 150,0 mm ± 0,5 mm (Henceforth referred to as 150 mm wafer)

4.2.3 Thickness and tolerance

Thickness is 0,18 mm to 0,80 mm. Tolerance for diameter of up to 100 mm is ± 0,03 mm. For diameter greater than 100 mm, thickness tolerance is to be agreed between the buyer and the manufacturer.

4.2.4 Orientation flat

a) Dimensions of OF and tolerances

22,0 mm ± 3,0 mm (for a 76,2 mm wafer)

32,5 mm ± 3,0 mm (for a 100 mm wafer)

42,5 mm ± 3,0 mm (for a 125 mm wafer)

47,5 mm ± 3,0 mm (for a 150 mm wafer)

57,5 mm ± 3,0 mm (for a 150 mm wafer)

b) Orientation tolerance

Orientation tolerance: ± 30’

Orientation of the OF shall be perpendicular to SAW propagation unless otherwise agreed upon by the user and the supplier. Orientation of the OF for quartz crystal wafers is X-plane (1 1-2 1) and an arrow pointing from the wafer centre to the OF is in the –X direction.

4.2.5 Secondary flat

The dimensions and tolerances are as listed below:

a) Dimensions of SF and tolerances

Dimensions and these tolerances of the SF are specified as reference values.

11,2 mm ± 4 mm unless otherwise agreed upon (for 76,2 mm wafer)

18,0 mm ± 4 mm unless otherwise agreed upon (for 100 mm wafer)

27,5 mm ± 4 mm unless otherwise agreed upon (for 125 mm wafer)

37,5 mm ± 4,5 mm unless otherwise agreed upon (for 150 mm wafer)

b) Orientation tolerance of SF

Orientation tolerances of the SF are measured with respect to the OF and are agreed on by the user and the supplier with a typical value being ± 1,0°.

Laser marking can be used as an alternative method to indicate the front surface.

4.2.6 Back surface roughness

As agreed upon by the user and the supplier (see Table 2).

4.2.7 Warp

As specified in Table 2.

4.2.8 TV5 or TTV

As specified in Table 2.

Table 2 – Roughness, warp, TV5 and TTV specification limits

Material	Diameter of wafer	Roughness of back surface (Ra)	Warp specified value μm	TV5 specified value μm	TTV specified value μm
Quartz crystal	76,2 mm (3 inch)	0,5 μm or greater	30	10	10
		Less than 0,5 μm	20	10	10
	100 mm	0,5 μm or greater	40	10	10
		Less than 0,5 μm	30	10	10
LN, LT	76,2 mm (3 inch)	2,0 μm or greater	50	15	15
		2,0 μm to 0,5 μm	40	15	15
		Less than 0,5 μm	40	10	10
	100 mm	2,0 μm or greater	50	20	20
		2,0 μm to 0,5 μm	40	15	15
		Less than 0,5 μm	40	10	10
	125 mm	2,0 μm or greater	60	20	20
		2,0 μm to 0,5 μm	50	15	15
		Less than 0,5 μm	40	10	10
	150 mm	2,0 μm or greater	60	20	20
		2,0 μm to 0,5 μm	50	15	15
		Less than 0,5 μm	40	10	10
LBO	76,2 mm (3 inch)	0,5 μm or greater	40	15	15
		Less than 0,5 μm	40	10	15
	100 mm	0,5 μm or greater	40	10	10
		Less than 0,5 μm	40	10	10
LGS	76,2 mm (3 inch)	0,5 μm or greater	40	15	15
		Less than 0,5 μm	40	10	10
	100 mm	0,5 μm or greater	40	20	20
		Less than 0,5 μm	40	10	10

4.2.9 Front (propagation) surface finish

The front surface shall be mirror polished. Surface finishing details are subject to agreement between the user and the supplier.

4.2.10 Front surface defects

a) Scratches

No scratches on visual inspection

b) Chips

1) Edge chips:

Radial depth: less than 0,5 mm

Peripheral chord length: less than 1,0 mm

2) Surface:

No chips on visual inspection

c) Cracks

No cracks on visual inspection

d) Contamination

No contamination on visual inspection

e) Others

Other defects such as dimples, pits, and orange peel: no such defects on visual inspection

4.2.11 Surface orientation tolerance

Surface orientation shall be specified by the user and the supplier.

Quartz crystal: ± 10’

LN, LT, LBO: ± 20’

LGS crystal: ± 10’

4.2.12 Inclusions

LN/LT/LBO/LGS: No visible inclusions on naked eye inspection.

Synthetic quartz: material satisfies the specification Grade II of IEC 60758:2016, 4.1.3.

4.2.13 Etch channel number and position of seed for quartz wafer

The etch channel number and the position of the seed are described below:

a) Etch channel within seed portion for a quartz crystal wafer

The number of the etch channel in a seed of not passing through from front surface to back surface is as shown in Table 3.

Table 3 – Maximum number of etch channels in seed position

Grade	76,2 mm wafer	100 mm wafer
1	6	8
2	12	16
3	36	47

b) Position of seed

The seed shall be included within ± 3,5 mm centre width of the Z’ direction and parallel to the X-direction of the centre of the wafer.

4.2.14 Bevel

The bevel shall be as agreed upon by the user and the supplier.

4.2.15 Curie temperature and tolerance

NOTE Only applies to LN/LT. The centre value for the specification is as agreed upon by the user and the supplier. Alternatively, the lattice constant can be specified.

LN: centre value within 1 133 °C and 1 145 °C. Tolerance ± 3 °C.

LT: centre value within 598 °C and 608 °C. Tolerance ± 3 °C.

4.2.16 Lattice constant

NOTE Alternatively, the Curie temperature can be specified.

LT: 0,515 40 nm ± 0,000 02 nm for an axis measured at 25 °C.

4.2.17 Bulk resistivity (conductivity) for reduced LN and LT

LN: $1,0 \times 10^{8}\ \Omega\cdot cm < BR < 1,0 \times 10^{12}\ \Omega\cdot cm$ ($1,0 \times 10^{-12}\ \Omega/cm < BC < 1,0 \times 10^{-8}\ \Omega/cm$).

LT: $1,0 \times 10^{10}\ \Omega\cdot cm < BR < 1,0 \times 10^{13}\ \Omega\cdot cm$ ($1,0 \times 10^{-13}\ \Omega/cm < BC < 1,0 \times 10^{-10}\ \Omega/cm$).

5 Sampling plan

5.1 General

A statistically significant sampling plan shall be agreed upon by the user and the supplier. Sampled wafers shall be randomly selected and representative of the production population, and shall satisfy the quality assurance criteria using the prescribed test methods.

5.2 Sampling

Unless otherwise specified, sampling shall be in accordance with AQL 2,5 %, single sampling as defined in ISO 2859-1. The specified AQL applies to the listed groups of defects considered collectively.

5.3 Sampling frequency

Appropriate statistical methods shall be applied to determine adequate sample size and acceptance criteria for the considered lot size. In the absence of more detailed statistical analysis, the following sampling plan can be employed:

a)	Dimensions	
	Diameter	2 wafers/manufacturing lot
	Thickness	2 wafers/manufacturing lot
	Length of OF	2 wafers/manufacturing lot
b)	Surface orientation	2 wafers/manufacturing lot
c)	Orientation of OF	2 wafers/manufacturing lot
d)	Back surface finishing	2 wafers/manufacturing lot
e)	TV5	2 wafers/manufacturing lot
f)	Warp	2 wafers/manufacturing lot
g)	TTV	2 wafers/manufacturing lot

5.4 Inspection of whole population

The following items shall be inspected for all wafers:

a) Existence and position of OF and SF
b) Surface finish
c) Wafer defects
d) Inclusions
e) Bevelling

6 Test methods

6.1 Diameter

Measurement of the wafer diameter (excluding OF and SF portions) using callipers of sufficient accuracy.

6.2 Thickness

Thickness at the centre of the wafer as measured by a sufficiently accurate (typically 1 μm) thickness meter, in accordance with ASTM test method F533.

6.3 Dimension of OF

Measurement of the OF length as a straight cut line of the intersection with the circle using callipers of sufficient accuracy.

6.4 Orientation of OF

Deviation of the geometrical orientation flat from the reference orientation of the lattice plane as measured with an X-ray diffractometer. The method is explained in detail in 10.4 and Figure 10.

6.5 TV5

TV5 is measured at the centre and at the four points located 6 mm from the edge of the wafer using callipers of sufficient accuracy (typically 1 μm) in accordance with ASTM test method F533.

6.6 Warp

Warp and other flatness parameters are measured using optical flatness equipment.

6.7 TTV

TTV is measured on clamped wafers using optical flatness equipment.

6.8 Front surface defects

Surface defects on the wafer shall be inspected using the method explained in Clause 11.

6.9 Inclusions

Inspection for inclusions shall be performed using light reflected from the polished wafer surface. Inspection should be carried out in a clean environment using a high intensity optically condensed light against a dark background to prevent interference from diffuse light reflections.

6.10 Back surface roughness

Surface roughness may be measured by either the contact or optical method. The average roughness (Ra) values listed in Table 2 were determined by contact profilometry. Measured values for a given wafer generally depend on the method (stylus radius, sampling interval, optical parameters).

6.11 Orientation

Crystallographic orientation is determined by X-ray diffraction (see 10.1 and Figure 10).

6.12 Curie temperature

The Curie temperature of a ferroelectric material may be determined by either calorimetric or dielectric measurement methods (see 8.1).

6.13 Lattice constant

The crystal lattice constant may be determined by XRD (see Clause 9).

6.14 Bulk resistivity

The bulk resistivity is determined by applying a voltage of 500 V across the wafer at room temperature and measuring the current one minute after the voltage has been applied. The inner-electrode shall have a diameter between 30 mm and 70 mm (see Clause 11).

7 Identification, labelling, packaging, delivery condition

7.1 Packaging

Wafers shall be packaged so as to avoid contamination or damage during shipping or storage.

Special packaging requirements shall be subject to agreement between the user and the supplier.

7.2 Labelling and identification

All wafer containers shall include labels with the following information:

a) supplier's name or trade mark;
b) material type;
c) wafer orientation;
d) manufacturing lot number;
e) quantity.

7.3 Delivery condition

Additional documentation or shipping requirements are to be negotiated between each user and supplier.

8 Measurement of Curie temperature

8.1 General

Curie temperature (T_c) determinations are performed on single crystal lithium tantalate (LT) and lithium niobate (LN). Both the DTA (differential thermal analysis) and dielectric constant methods used to determine T_c are destructive tests. Which measuring method should be used depens on the agreement between a user and a supplier.

8.2 DTA method

The DTA (differential thermal analysis) method is based on the endothermic or exothermic reaction observed when a single crystal transitions from the ferroelectric to paraelectric states. Typically, the sample and a reference material are symmetrically positioned in an oven (see Figure 7) and heated at a constant rate, while recording the temperature difference between the materials. Alumina (α-Al_2O_3) is often used as the reference when running DTA experiments on LN or LT. Heat is released at the LN or LT sample passes upward through the phase transition temperature, and the temperature profile relative to the alumina reference is recorded. The Curie temperature T_c is defined as the temperature at which the temperature difference arises.

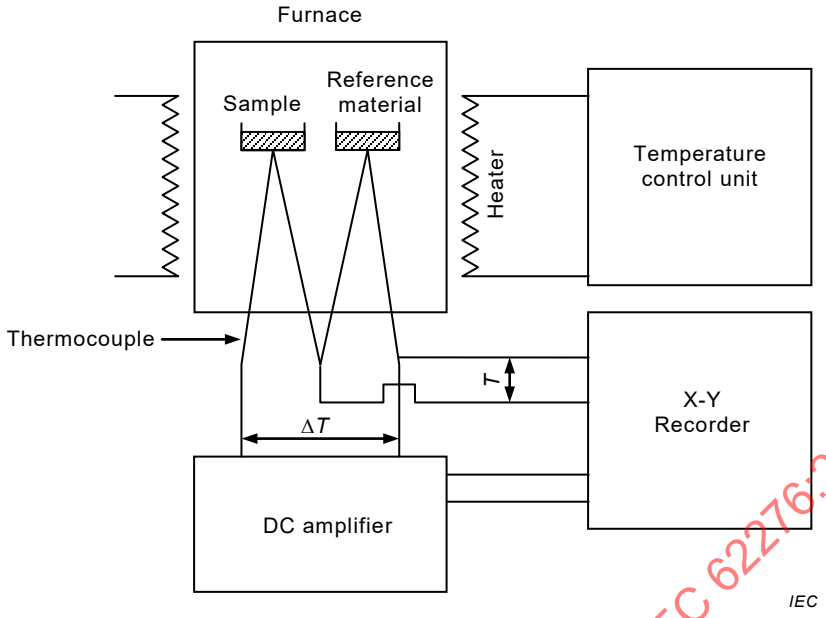


Figure 7 – Schematic of a DTA system

8.3 Dielectric constant method

The dielectric constant method relies upon observing the dielectric constant along the polar Z-axis of a ferroelectric crystal. The dielectric constant maximum is found to occur at the phase transition temperature. Since the dielectric constant, and thus the capacitance, for a given sample are a function of temperature only, the heating or cooling rates can be chosen to be small enough so as to minimize thermal hysteresis. In Figure 8, the electrode of Pt or Ag-Pd is placed on the sample so that the electric field runs along the polar Z axis. While scanning the temperature across the phase transition, the capacitance of the sample is measured by the LCR-meter. The temperature at which the peak capacitance is observed corresponds to the Curie temperature T_c.

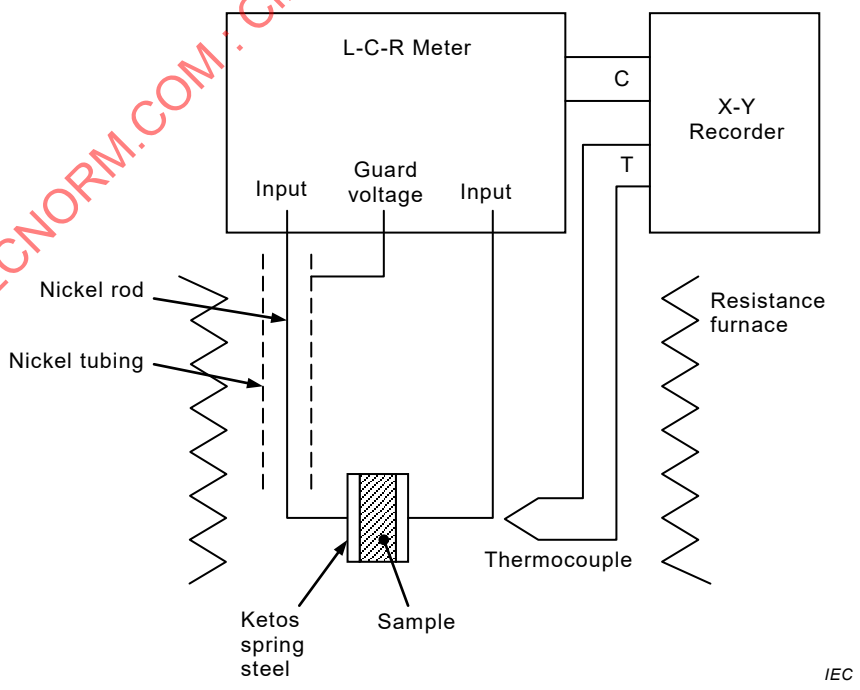


Figure 8 – Schematic of a dielectric constant measurement system

9 Measurement of lattice constant (Bond method)

As the chemical composition of a crystal changes, so do the SAW velocities and lattice constants. In order to control the SAW velocity to within one part per ten thousand (10^{-4}), the lattice constants shall be controlled within 10^{-5}. The measurement method in turn shall achieve part per million (10^{-6}) resolution.

X-ray diffraction is used to measure lattice constants. The method is based on Bragg's law as follows:

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

where

d is the lattice spacing,

θ is the Bragg angle,

λ is the X-ray wavelength, and

n is the integer diffraction order.

If λ is given, d and lattice constants are determined by measuring θ. A sensitivity analysis yields

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

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

In the Bond method, two measurements are made, (i.e. the 'plus-side' and 'minus-side'), located symmetrically around the same lattice face. The values ω_1 and ω_2 from the peaks of rocking curve are determined as Figure 9 shows and θ is calculated as:

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

This method eliminates off-centre error plus absorption and zero error is theoretically eliminated as well. Note that temperature, refraction, divergence and Lorentz-polarization corrections should be taken into account.

For the case of $LiTaO_3$, the Miller index (60-60) was evaluated by the Bond method. The a-axis lattice constant is calculated as follows:

$$a = 6d_{66,0}$$

After applying various corrections, the lattice constant of $LiTaO_3$ is determined to an accuracy of 10^{-6} to 10^{-7}.

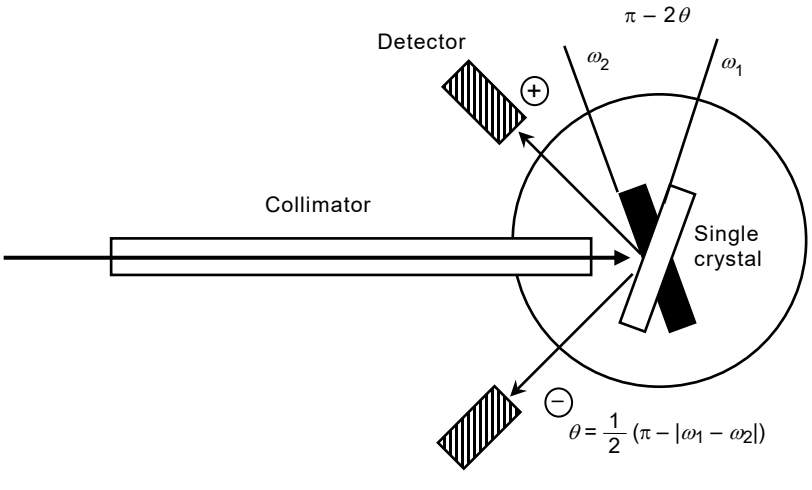


Figure 9 – The Bond method

10 Measurement of face angle by X-ray

10.1 Measurement principle

If the distance between each lattice face is d, the X-ray wavelength is λ and the diffraction order is n, the X-ray beam diffracts when the Bragg angle θ condition is satisfied as follows:

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

The X-ray source consists of a collimated beam and an optional reflecting crystal plate. An X-ray detector is positioned at an angle relative to the source. As the crystal is rotated, the detector will register signal maxima at the Bragg angle, and the goniometer will indicate the angle θ between the crystal plate surface and the crystal lattice planes, as illustrated below in Figure 10.

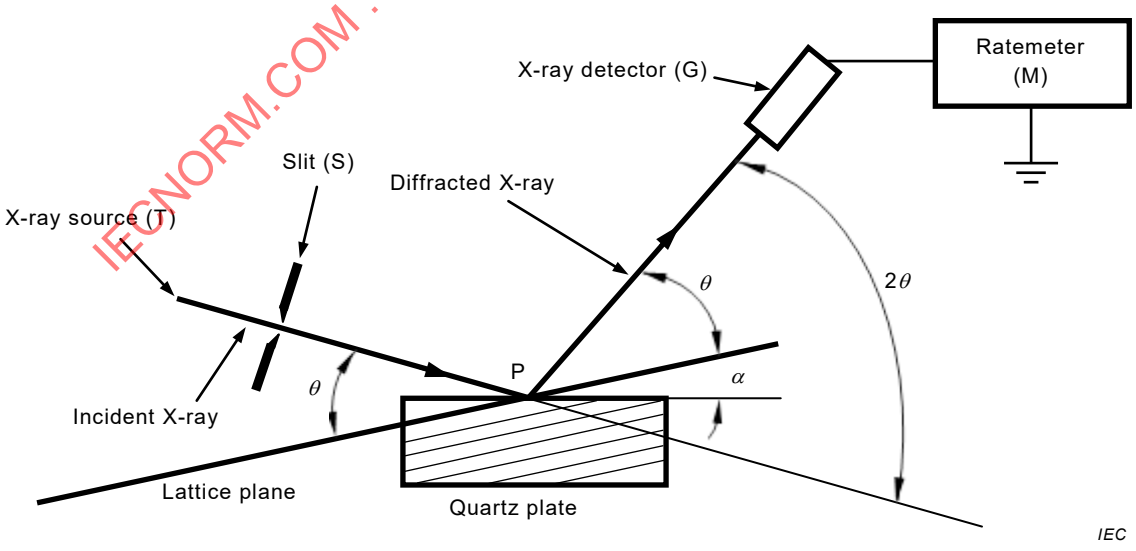


Figure 10 – Measurement method by X-ray

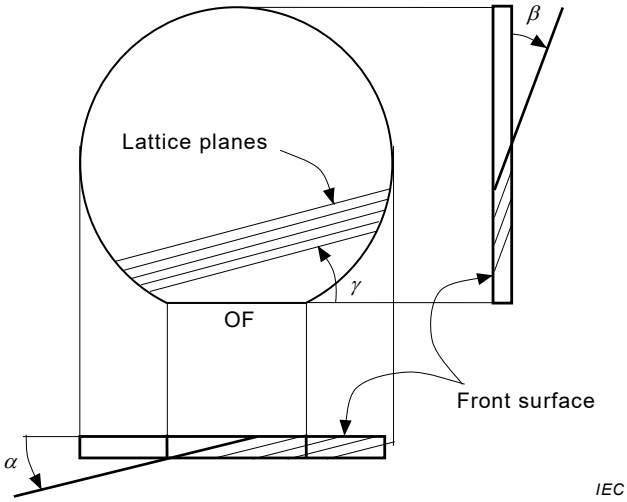


Figure 11 – Relationship between cut angle and lattice planes

10.2 Measurement method

Before measuring the face angle of the sample under test, the goniometer may need to be calibrated using a reference sample. The face angle deviations of the sample under test are then calculated based on comparison of the diffraction data obtained from the sample with data from the reference crystal.

10.3 Measuring surface orientation of wafer

The angles should be measured in two directions as follows:

- parallel to the OF: α (positive direction as shown in Figure 11 when looking at the OF);
- perpendicular to the OF: β (positive direction as shown in Figure 11).

NOTE Counter clockwise rotation of the angle α is positive,.measuring OF flat orientation.

10.4 Measuring OF flat orientation

The angle γ should be measured (positive direction as shown in Figure 11).

10.5 Typical wafer orientations and reference planes

Typical wafer orientations and reference planes are shown in Table 4.

Table 4 – Crystal planes to determine surface and OF orientations

Materials	Wafer description	Reference for cutting face	Face (α)	Cutting face (β)	OF reference face	OF face (γ)
LN	128° Y-X	(0 –1 1 4) hex	0	0	(2 –1 –1 0) hex	0
LN	Y-Z	(0 3 –3 0) hex	0	0	(0 0 0 6) hex	0
LN	64° Y-X	(0 1 –1 8) hex	+4°46’	0	(2 –1 –1 0) hex	0
LT	X-112° Y	(2 –1 –1 0) hex	0	0	(0 1 –1 2) hex	–79°16’
LT	X-112° Y	(2 – 1 – 1 0) hex	0	0	(0 –1 1 10) hex	–5°02’
LT	X-112° Y	(2 –1 –1 0) hex	0	0	(0 0 0 6) hex	–22°12’
LT	36° Y-X	(0 1 –1 2) hex	–3°04’	0	(2 –1 –1 0) hex	0
LT	42° Y-X	(0 1 –1 2) hex	–9°04’	0	(2 –1 –1 0) hex	0
LBO	45° X-Z	(1 1 0) tetra	0	0	(0 0 1) tetra	0

Materials	Wafer description	Reference for cutting face	Face (α)	Cutting face (β)	OF reference face	OF face (γ)
Quartz	ST-X	(0 1 –1 1) hex	+4°32′	0	(2 –1 –1 0) hex	0
LGS	yxlt/48,5°/26,6°	(0 1 –1 1) hex	–5°45′	0	(1 1 –2 0) hex	–26°36′

11 Measurement of bulk resistivity

11.1 Resistance measurement of a wafer

Resistance measurement equipment with insulation resistance $1{,}0 \times 10^{8} – 10^{14}\ \Omega$ and a circular electrode is used for measuring resistance of wafer. When a wafer is measured it is put between electrodes of the equipment. A voltage of 500 V is applied while the current through the wafer is measured and the reading is taken one minute after the voltage is applied. Measuring circuits and equipment are shown in Figure 12 and Figure 13.

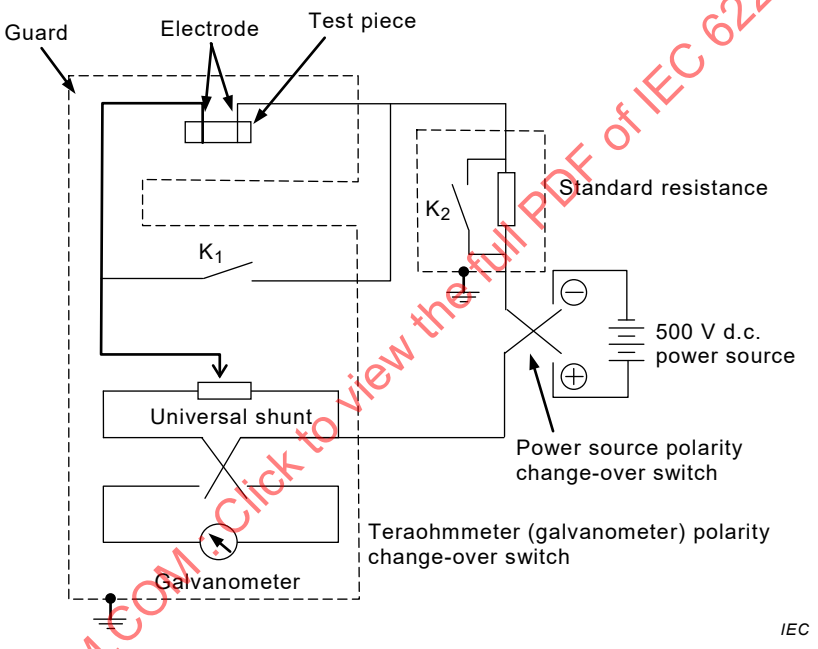


Figure 12 – Measuring circuit

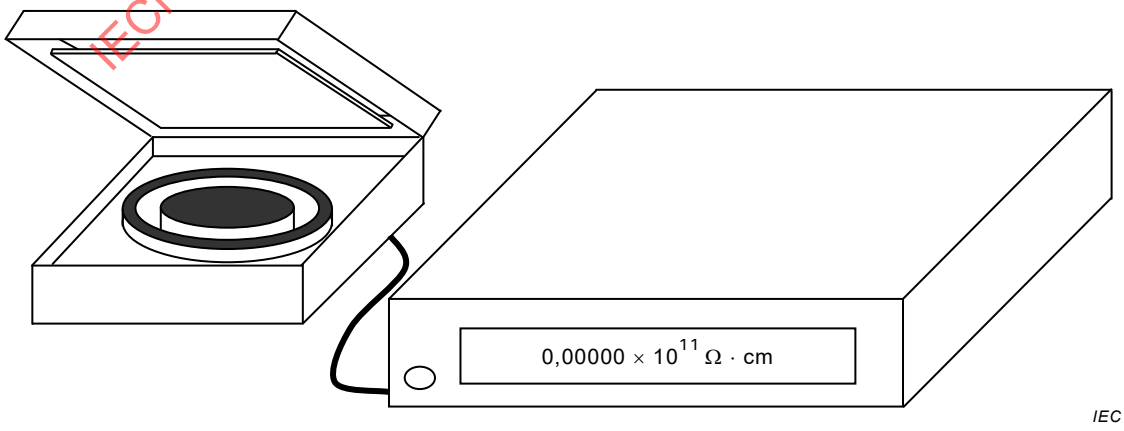


Figure 13 – Resistance measuring equipment

11.2 Electrode

A shape and size example of electrode is shown in Table 5 and Figure 14.

Table 5 – Electrode size

− electrode diameter mm			+ electrode diameter mm
D1	D2	d	D3
80 ± 0,5	70 ± 0,5	50 ± 0,5	100 ± 0,5

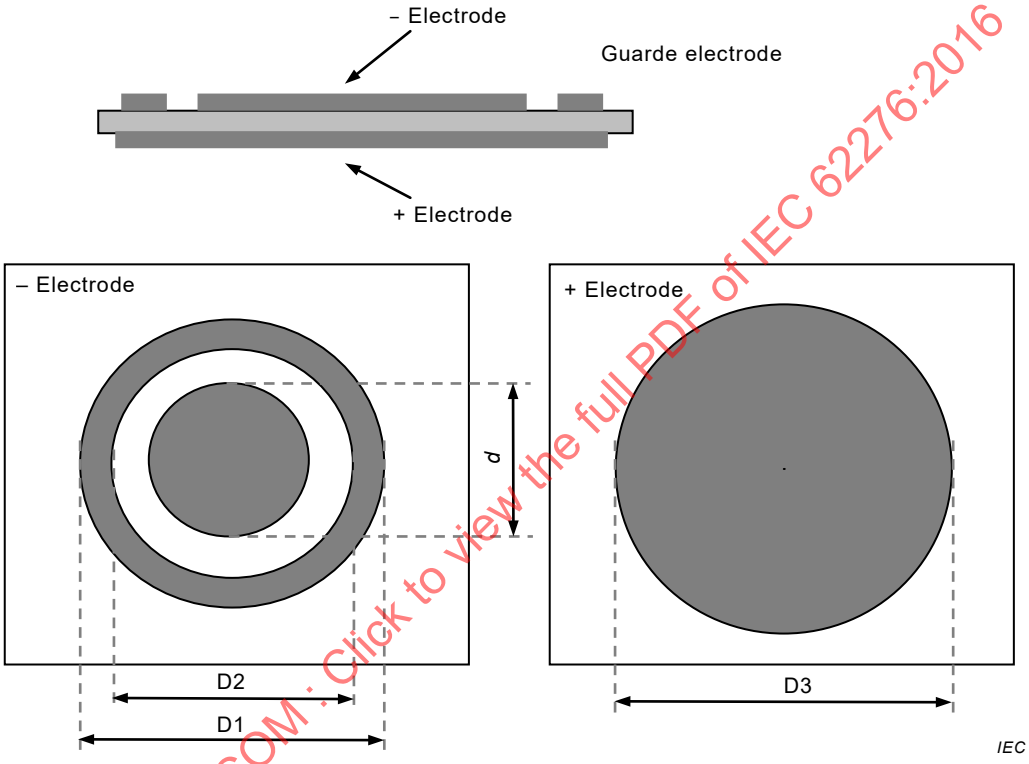


Figure 14 – Shape of electrode

11.3 Bulk resistivity

After measuring resistance of a wafer, bulk resistivity is calculated as follows.

$\rho_V = R\ (\pi d^2/4t)$

ρ_V: Bulk resistivity (Ω·cm)
d: Diameter of inner –face electrode
t: Thickness of substrate

$R = V/I$

R: Bulk resistance (Ω)
V: DC voltage (V)
I: Electric current (A)

12 Visual inspections – Front surface inspection method

A mirror polished wafer surface is required for fabrication of reliable SAW transducers. Routine wafer inspection should include visually checking for the following defects:

– scratches

– chips
– cracks
– contamination
– dimples, pits, orange peel, etc.

Visual inspection pass/fail criteria may be based on:

a) quantitative measurements;
b) qualitative descriptions;
c) visual documentation (e.g. illustrations, photographs), or
d) representative samples.

Inspection records shall clearly indicate whether the product has passed or failed the inspection based on the established acceptance criteria.

Wafers are typically sampled using the unaided eye, with a high intensity white light lamp providing illumination. Select wafers may also be examined under a microscope to better characterize small defects. Depending on the circumstances, different microscopy methods may be used (e.g. brightfield, darkfield, Nomarski). Unaided visual inspection is carried out in a clean environment, with the wafer suspended over a dark surface. The inspection area should be darkened so as to prevent stray ambient light from interfering with the inspector's ability to clearly see the surface.

In order to avoid bias and accurately gauge the number of defects present in a given sample population, wafers to be inspected should be chosen at random and, in the case of a production stream, with consistent sampling frequency. Variables such as the wafer surface area to be inspected, or details of the inspection light source (e.g. intensity, type or illumination angle) shall be negotiated between the user and the supplier. Quantitative defect criteria should be used when practicable.

Annex A
(normative)

Expression using Euler angle description for piezoelectric single crystals

A.1 Wafer orientation using Euler angle description

Material tensor properties of piezoelectric single crystals such as piezoelectric constants (d_{11}), elastic constants (c_{11}), and dielectric constants (ε_{11}) are generally described in a rectangular coordinate system (X, Y, Z) related to the crystal axes. Wafer cuts used for SAW device fabrication generally use rotated cuts. The Euler angle description gives a way to describe the crystallographic orientation of the wafer surface normal direction and the orientation flat direction which typically coincides with SAW wave propagation.

Figure A.1 shows the three rotations and their respective angles that transform the crystallographic axes (X, Y, Z) to the wafer coordinate system (x_1, x_2, x_3) as shown in Figure A.2. The SAW wave propagation direction typically is in the x_1 direction. The surface normal points are in the x_3 direction. The top surface is the polished surface on which the electrode patterning is done. x_2 is defined by forming an orthogonal, right-handed coordinate system with the other two vectors. To visualize the Euler angle rotations, start with a crystal having axes (X, Y, Z). The first rotation is around Z by the angle φ in the direction indicated in Figure A.1. The values of φ can range from 0° to 360°. This rotation maps the old X-axis onto x_{rot}. The next rotation is around this newly defined axis x_{rot} by the angle θ . This angle is restricted to values ranging from 0° to 180°. The rotation maps the Z-axis onto x_3, the wafer surface normal. The last rotation is around x_3 by angle ψ . The range for this angle can range from 0° to 360° and it will map x_3 onto x_1, the direction that coincides with the orientation flat. Using the angles within the range given here provides a way to completely describe any wafer orientation. It also allows to specify which wafer side is to be polished. In lithium niobate for example, the Y-face is polar and wet etching in hydrofluoric acid will have a different rate on the two opposite sides of the wafer. The angles (0°, 90°, 90°) designate a wafer with the –Y face polished and a flat on the +Z end of the wafer. The angles (180°, 90°, 90°) designate a wafer with the +Y face polished and the flat also at the +Z end of the wafer. While the SAW properties commonly do not depend on which face of the wafer is polished, other characteristics such as pyroelectric charging or etching often do and thus it is important to specify which surface to polish.

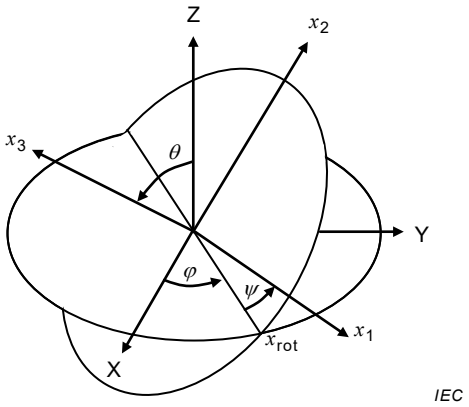


Figure A.1 – Definition of Euler angles to rotate coordinate system (X, Y, Z) onto (x_1, x_2, x_3)

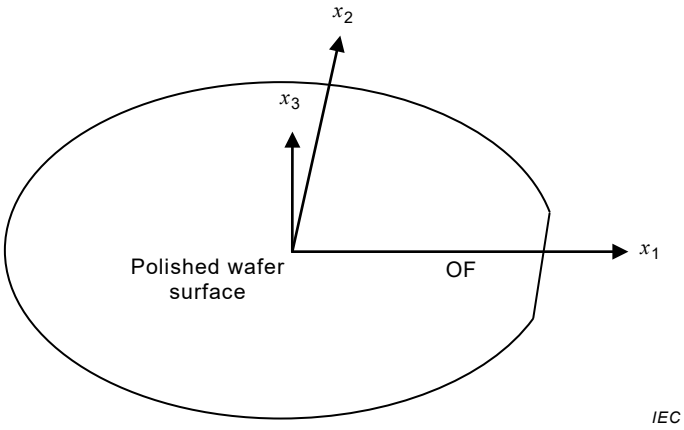


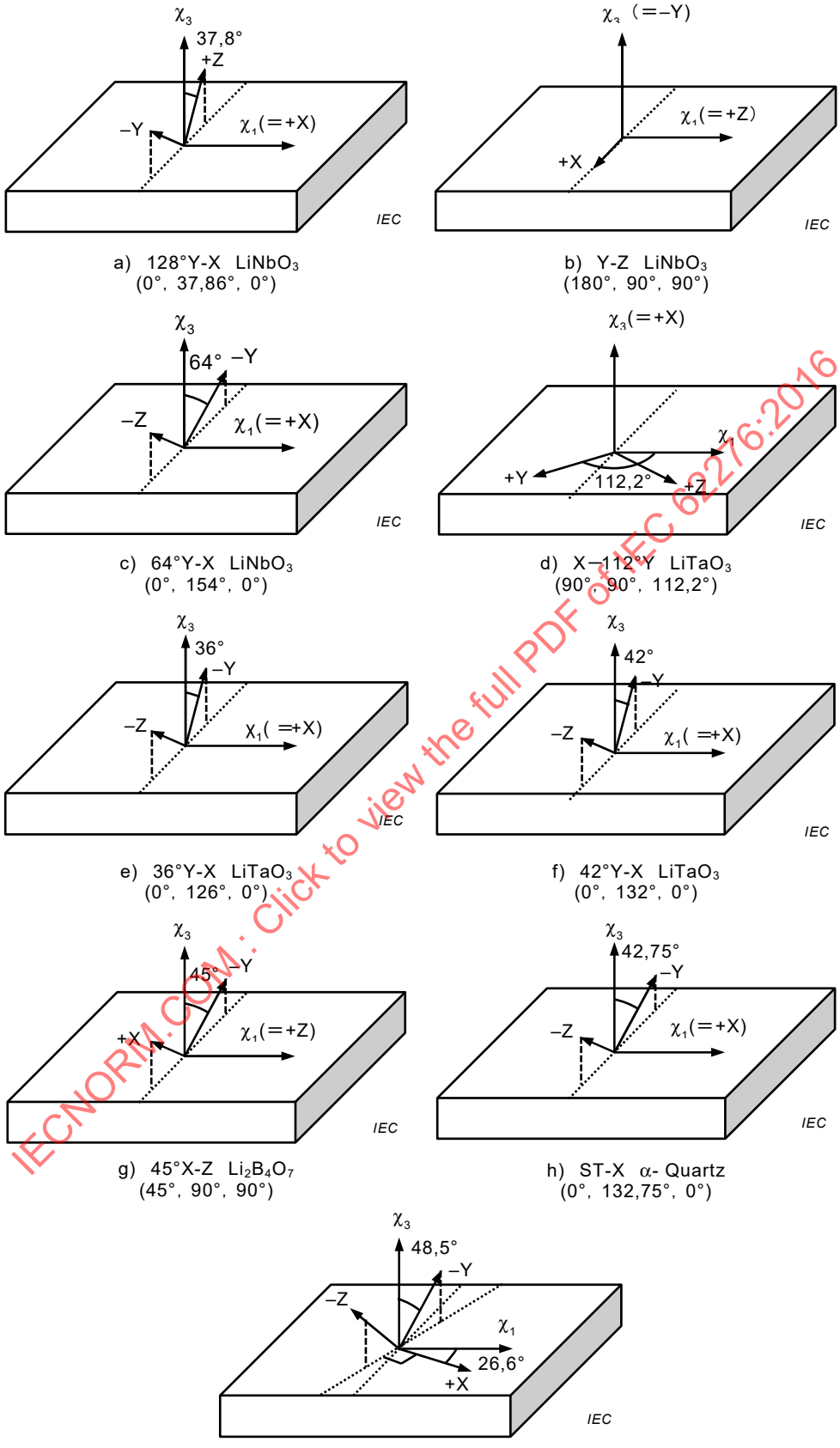
Figure A.2 – SAW wafer coordinate system

The Euler angle definition specified here applies only to crystals using right-handed crystal coordinate systems. In other cases, such as left-handed quartz, the nomenclature needs to be agreed on between users and suppliers.

Table A.1 lists typical SAW substrate orientations and Euler angles. The same substrate cuts are shown in Figure A.3.

Table A.1 – Selected SAW substrate orientations and corresponding Euler angles

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°, 37,86°, 0°)
Y-Z **LN**	Y cut Z SAW propagation Lithium niobate substrate	$LiNbO_3$ (180°, 90°, 90°)
64° Y-X **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	$Li_2B_4O_7$ (45°, 90°, 90°)
ST-X **α- Quartz**	ST cut X SAW propagation α- quartz crystal	SiO_2 (‹-Quartz) (0°, 132,75°, 0°)
yxlt/48,5°/26,6° **LGS**	48,5° rotated Y cut 26,6° rotated X SAW propagation Lanthanum gallium silicate substrate	$La_3Ga_5SiO_{14}$ (0°, 138,5°, 26,6°)



a) 128°Y-X $LiNbO_3$
(0°, 37,86°, 0°)

b) Y-Z $LiNbO_3$
(180°, 90°, 90°)

c) 64°Y-X $LiNbO_3$
(0°, 154°, 0°)

d) X–112°Y $LiTaO_3$
(90°, 90°, 112,2°)

e) 36°Y-X $LiTaO_3$
(0°, 126°, 0°)

f) 42°Y-X $LiTaO_3$
(0°, 132°, 0°)

g) 45°X-Z $Li_2B_4O_7$
(45°, 90°, 90°)

h) ST-X α- Quartz
(0°, 132,75°, 0°)

i) yxlt/48,5°/26,6° $La_3Ga_5SiO_{14}$
(0°, 138,5°, 26,6°)

Figure A.3 – Relationship between the crystal axes, Euler angles, and SAW orientation for some wafer orientations

Annex B
(informative)

Manufacturing process for SAW wafers

B.1 Crystal growth methods

B.1.1 Czochralski growth method

B.1.1.1 General

A single crystal boule is grown by dipping a seed crystal into a melt contained within a crucible. The seed and/or the crucible is rotated while slowly pulling the seed upward, thus drawing a boule from the melt as it cools and solidifies. This technique is named after Polish scientist Jan Czochralski who first used it in 1916 to grow single crystals of metals. Industrial volume production using this method started with germanium (Ge) and silicon (Si). The first crystals of LN and LT were manufactured in 1965 at Bell Labs and at a lab located in the former Soviet Union.

While heating can be applied either by RF induction or resistive heating, LN, LT and LGS single crystals are generally grown using RF induction heating. Figure B.1 shows a simplified sketch of an apparatus using RF heating.

The starting material is typically prepared as follows: Powders of Li_2CO_3 and Nb_2O_5 (Ta_2O_5) with a Li/Nb (Li/Ta) mole ratio between 0,93 and 0,95 are mixed and calcinated after press forming. The resulting polycrystalline ceramic of LN (LT) is placed into the crucible and melted by heating the crucible.

For LGS, initial starting material is obtained by mixing La_2O_3, Ga_2O_3 and SiO_2 in stoichiometric proportions. The mixture is pressed into pellets and annealed at temperatures higher than 1 200 °C for a few hours. The resultant polycrystalline LGS is used in the same way as for LN or LT.

The end of a seed crystal cut with the desired crystal orientation is carefully lowered to just make contact with the melt. This seed crystal is rotated to induce a controlled convection pattern in the melt, and a slow pull rate is initiated that withdraws the seed (and growing crystal) from the melt. The melt temperature needs to be carefully controlled near the melting point of the material so that the growing crystal displays the desired necking (narrowing of diameter at the top of the boule) during the early stages of growth.

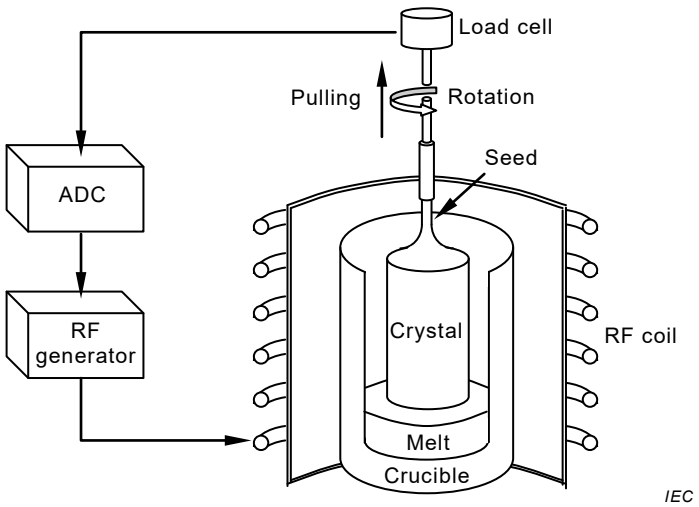


Figure B.1 – Czochralski crystal growth method

Once a proper neck is formed, the melt is next gradually cooled until a shoulder is formed and constant crystal growth diameter is achieved. The weight of the growing crystal is tracked with a load cell as shown in Figure B.1, and the present diameter is inferred from the time derivative of that signal. The automatic diameter control (ADC) electronics adjusts the RF power dynamically in order to maintain the proper diameter. When the desired crystal length has been achieved, the crystal is quickly pulled from the melt to stop growth and carefully cooled to room temperature.

B.1.1.2 Domain structure

LN and LT are ferroelectric crystals and exist in the non-polar paraelectric phase when grown. As they are cooled below the transition temperature, the Curie temperature, the structure changes from the ferroelectric phase to the paraelectric phase and a spontaneous polarization develops along the Z-axis. When this happens during cool-down after growth, the crystal typically forms many different regions, called domains, with opposite polarity in neighbouring domains. This multi-domain structure is undesirable, and a single domain crystal is obtained by applying an external d.c. voltage while the crystal cools through the transition. This operation is termed “poling”.

Because the spontaneous polarization aligns along the Z-axis in LN and LT, poling is done by forming an electrode pair on the faces perpendicular to the Z axis, heating up crystals above the Curie temperature, and then cooling down the crystals while applying a d.c. voltage. To remove thermal stress from LN and LT grown by the crystal pulling method, an annealing process is typically carried out before poling at a temperature below the melting point. For LT, the annealing operation is carried out around 1 300 °C, the crystal is then cooled down to room temperature to apply the electrodes by conductive metal paste. Then, the temperature is raised to about 650 °C, a d.c. voltage of some mV/cm of crystal length is applied, and the crystal is cooled through the phase-transition temperature of about 600 °C. As the Curie temperature for LN is around 1 140 °C, it is customary to conduct both the annealing (at about 1 200 °C) and poling processing at the same time.

B.1.1.3 Compositional uniformity

Crystals do not always grow with their constituents in stoichiometric elemental ratios as their formulae suggest, but instead may crystallize within a compositional solid solution range. For such a material, the composition of the growing crystal is determined by the composition of the melt. Generally, the composition of the solidified single crystal changes during growth. Some material systems have a certain composition, referred to as “congruent composition”, where the solid and the melt are in thermodynamic equilibrium. If absent, any constituent volatility (e.g. Li_2O or Ga_2O_3), a melt of congruent composition will produce a uniform single crystal of the same composition.

Crystals of uniform composition can be grown from melts with Li/Nb mole ratio (Li/Ta mole ratio) in the range from 0,93 to 0,95 in the case of LN (LT). When an LN (LT) crystal is grown from a composition different from the ideal composition, 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 devices also changes with the change of the composition. Figure B.2 shows some examples of LN growths. Consider for example case No. 4 where the starting composition was Li rich. As the growing crystal rejects some of the excess Li, the remaining melt Li/Nb ratio increases, and the crystal incorporates more Li as the growth proceeds. Consequently, the wave velocity quickens along the growth axis as the Li/Nb ratio increases. Only for crystals grown from the ideal composition will the wave velocity stay the same along the crystal axis.

This ideal composition may slightly deviate from the congruent compositions depending on crystallization speed, volatilization, etc. For the growth configuration considered here, the ideal composition is shown for case No. 2. Because of high volatility of Ga_2O_3 during LGS growth, these crystals tend to show some compositional nonuniformity, and growing crystals with good uniformity remains a challenge.