International Standard

ISO 5618-2

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for GaN crystal surface defects —

Part 2:
Method for determining etch pit density

Céramiques techniques — Méthode d'essai pour les défauts de surface des cristaux de GaN —

Partie 2: Méthode de détermination de la densité des piqûres

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Contents

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This document was prepared by Technical Committee ISO/TC206, *Fine ceramics*.

A list of all parts in the ISO 5618 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

GaN is a direct transition type of wide-bandgap semiconductor with superior physical properties, such as a higher breakdown electric field, saturated electron drift velocity, and thermal conductivity, compared with Si. GaN is expected to be applied not only in light-emitting devices that have been in practical use for a long time, such as ultraviolet and blue LDs and LEDs, but also in power devices that perform power conversion with high efficiency. In particular, the characteristics of GaN power devices are utilized in the fields of photovoltaics, automobiles, railways (electric motors and linear motors), communication base stations, and microwave power transmission.

The single-crystal GaN or single-crystal GaN film is the base material of many devices. However, the surface of the single-crystal GaN or single-crystal GaN film contains many dislocations that are introduced during crystal growth and defects introduced during wafer processing. These dislocations and/or defects cause a decrease in luminous efficiency for a light-emitting device and a decrease in performance and reliability for a power device. In particular, given the practical applications and market expansion of power devices that apply a high voltage and high current, it is indispensable to supply single-crystal GaN substrates and single-crystal GaN films with a low-density of dislocations and defects. Therefore, it is essential to have an international standard that defines and classifies the types of dislocations and processing-introduced defects that exist on the surface as an index for assessing the quality of a single-crystal GaN substrate or a single-crystal GaN film and determines the density of these dislocations and defects.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for GaN crystal surface defects —

Part 2: Method for determining etch pit density

1   Scope

This document describes a method for determining the etch pit density, which is used to detect dislocations and processing-introduced defects that occur on single-crystal GaN substrates or single-crystal GaN films.

It is applicable to the defects specified in ISO 5618-1 from among the defects exposed on the surface of the following types of GaN substrates or films: single-crystal GaN substrate; single-crystal GaN film formed by homoepitaxial growth on a single-crystal GaN substrate; or single-crystal GaN film formed by heteroepitaxial growth on a single-crystal Al_2O_3, SiC, or Si substrate.

It is applicable to defects with an etch pit density of $\leq 7 \times 10^7$ cm^{-2}.

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.

ISO 5618-1, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for GaN crystal surface defects — Part 1: Classification of defects*

ISO 19606, *Test method for surface roughness of fine ceramic films by atomic force microscopy*

ISO 21920-2:2021, *Geometrical product specifications (GPS) — Surface texture: Profile — Part 2: Terms, definitions and surface texture parameters*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

3   Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 5618-1 and the following apply.

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
primary orientation flat
flat part of the surrounding area of the substrate that is used to indicate the crystal orientation

3.2
secondary flat
flat part of the surrounding area of the substrate that is shorter than the primary orientation flat

3.3
optical microscope
microscope used to magnify and observe an object by using visible light

EXAMPLE   white, monochromatic, or laser light

3.4
numerical aperture
NA
sine of the vertex angle of the largest cone of meridional rays that can enter or leave an optical system or element, multiplied by the refractive index of the medium in which the vertex of the cone is located

[SOURCE: IEC 60050-731:1991, 731-03-85]

3.5
pixels per inch
ppi
individual pixels in a line or column of a digital image within a span of 25,4 mm (1 inch)

[SOURCE: ISO/IEC 39794-5:2019, 3.50]

3.6
chemical mechanical polishing
CMP
polishing that flattens the surface of a semiconductor substrate by means of a chemical action and a machine action

3.7
catalyst-referred etching
processing that induces chemical etching only on the reference plane to flatten the surface of a semiconductor substrate with high accuracy but without disrupting its crystallinity

3.8
etch pit
inverted hexagonal pyramidal or oval dent generated by etching

3.9
length of longer diagonal
length of the line that connects the opposite corners of the hexagonal shape at the bottom of the inverted hexagonal pyramid

3.10
coefficient of variation
relative variation calculated by dividing the standard deviation by the arithmetic mean

3.11
multiphoton excitation microscope
microscope used to observe the light emission distribution caused by simultaneous multiphoton absorption

3.12
photoluminescence
PL
luminescence caused by optical excitation

3.13
cathodoluminescence
CL
luminescence caused by electronic excitation

3.14
reference sample
sample manufactured by the same manufacturer or using the same method as that of the test sample

4 Principle

When a single-crystal GaN or single-crystal GaN film is immersed in etchant, etch pits are formed at locations where dislocations or processing-introduced defects are cropped out on the (0001)Ga polar face. These etch pits are formed because the etching speed at locations where dislocations or processing-introduced defects are cropped out on the (0001)Ga polar face is faster than that at locations where there are no dislocations. For threading dislocations, etch pits in the shape of an inverted hexagonal pyramid that consists of six faces of $\{1\bar{1}0n\}$ and has a vertex are formed. Dislocation-derived pits have this vertex. For basal plane dislocations, an oval etch pit is generated. In this case, one side is narrower and deeper than the other side.

Etch pits shaped like an inverted hexagonal truncated pyramid or etch pits shaped like a bowl that are caused by a corrupted inverted hexagonal truncated pyramid, are derived from pits caused by processing. Linear grooves are derived from scratches and latent scratches caused by processing.

5 Definition of substrate in-plane position

With the (0001)Ga polar face facing up, place the primary orientation flat at the bottom as shown in Figure 1. After that, draw a perpendicular bisector for the primary orientation flat. The position on the 1/2-centre side along the perpendicular bisector starting from the intersection point between the perpendicular bisector and the substrate upper outline is set as the origin point (0,0). The substrate in-plane position is expressed as (x, y) in units of millimetres (mm).
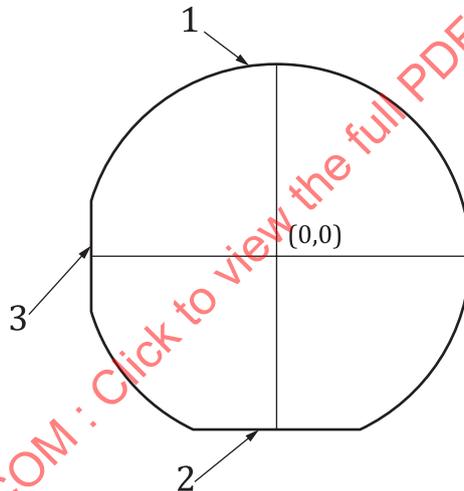
Key

1 substrate
2 primary orientation flat
3 secondary flat

Figure 1 — Definition of substrate in-plane position

6 Procedures for forming an etch pit

6.1 Pre-treatment of a sample

Set the average surface roughness of a sample R_a, as defined in ISO 21920-2:2021, to be $\leqq 1$ nm. When R_a exceeds 1 nm, flatten the surface by means of a method such as chemical mechanical polishing or catalyst-referred etching. Once the surface has been flattened, record this information in the report specified in Clause 10.

Measure the surface roughness in accordance with ISO 19606.

6.2   Etching process

Etch the sample under etching conditions that have been proven to be appropriate in accordance with Annex A.

Adjust the etch pit size to an appropriate size by changing only the immersion time, not any of the other etching conditions that have been proven to be appropriate (etchant composition, etchant temperature, and immersion time). See Annex B for reference.

Prepare the etchant for every batch etching.

Do not use an etchant once precipitates have been deposited on the sides of the crucible containing it during use.

Items exposed to the sample or etchant during etching: use the etchant container (e.g., crucible or beaker), sample carrier, and thermometer (thermometer or thermometer protective tube) only if they had the etchant removed from them after their previous use and were dried before storage.

Items exposed to the sample or etchant during etching: When performing the etching process for the sample, use the same materials for the etchant container (e.g., crucible or beaker), sample carrier, and thermometer (thermometer or thermometer protective tube) as those used during the etching process for the reference sample to prove their appropriateness.

Once a sample breaks due to thermal shock during etching, record this information in the report specified in Clause 10.

If an etchant containing KOH is used, a cover shall not be used for the crucible during the heating process.

If an etchant containing KOH is used, the etching shall be performed for 120 min or longer after the etching temperature has been reached.

If a sample has any cracks or internal stress, it can break due to thermal shock when it is input into the etchant.

6.3   Washing

Clean the surface of the sample with etch pits formed on it.

EXAMPLE   An example reference washing process is as follows:

—   Rinse under running deionized water for 10 min, cleaning in ultrasonic bath filled with deionised water for 10 min, boiling in 2 mol/l hydrochloric acid for 1 h, and rinsed under running deionized water for 10 min.

—   After the sample has been washed, dry it by blowing it with filtered air, nitrogen gas, or argon gas.

If the sample breaks during washing, record this information in the report specified in clause 10.

As shown in Figure 2, it is difficult to differentiate the crucible lysate and other substances that have adhered to the sample from the etch pits. Therefore, the etch pit density will be misestimated.

NOTE   Watermarks can remain if a sample is left to dry naturally after washing. Since it is difficult to differentiate such watermarks from the etch pits, the etch pit density will be higher than the actual value.

Samples that have cracks or internal stress can break during ultrasonic cleaning.
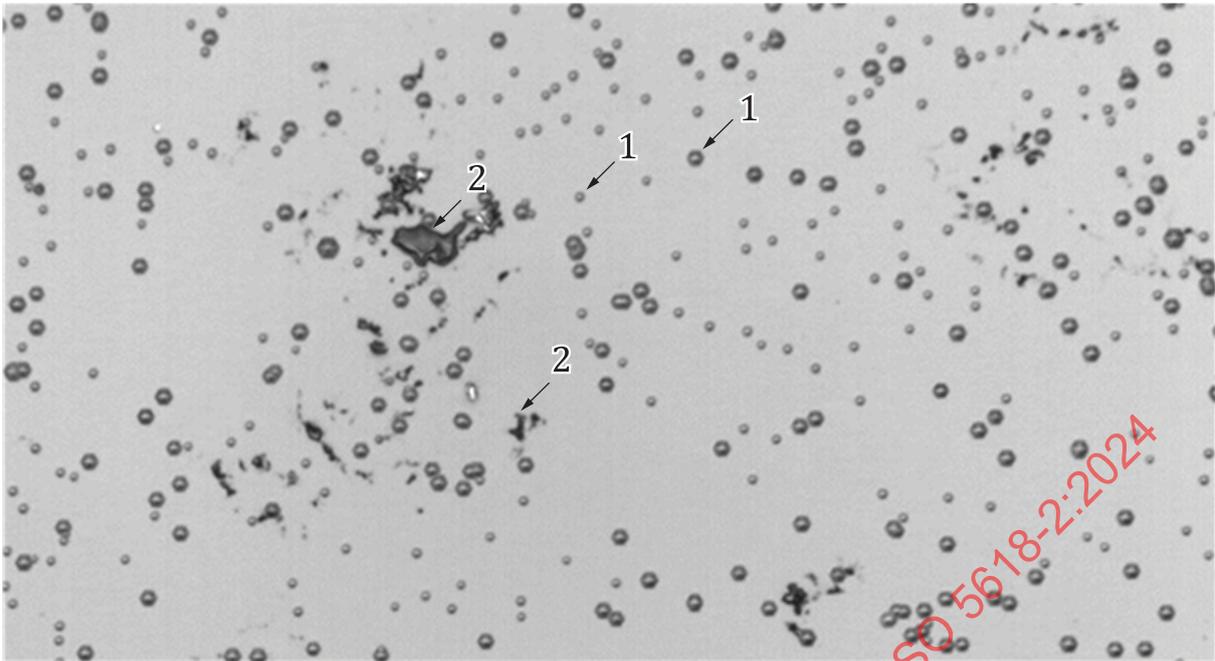
Key

1   etch pit
2   adhered matter

Figure 2 — Example of adhered matter on the etched surface of a GaN substrate

7   Method of capturing an etch pit image

7.1   Setting the observation conditions for an optical microscope

7.1.1   Objective lens

Use an objective lens with a numerical aperture (NA) that is greater than that of the objective lens used in 6.2.

7.1.2   Image resolution

Use an objective lens that satisfies the conditions specified in 7.1.1 to observe an area where 20 or more inverted hexagonal pyramidal pits can be seen at the (0,0) point on the sample, as shown in Figure 3. From these inverted hexagonal pyramidal pits, select five that you believe can be categorized into the smallest group and calculate the average of their lengths of longer diagonals, D μm.

Set the image resolution to $2{,}54 / D \times 10^4 \times 10$ ppi or more.

NOTE   If it is set to $2{,}54 / D \times 10^4 \times 10$ ppi or more, the length of the longer diagonal of the smallest pit is 10 pixels or more.

Key

1 inverted hexagonal pyramidal pit categorized into the smallest pit group

2 inverted hexagonal pyramidal pit categorized into the smallest pit group
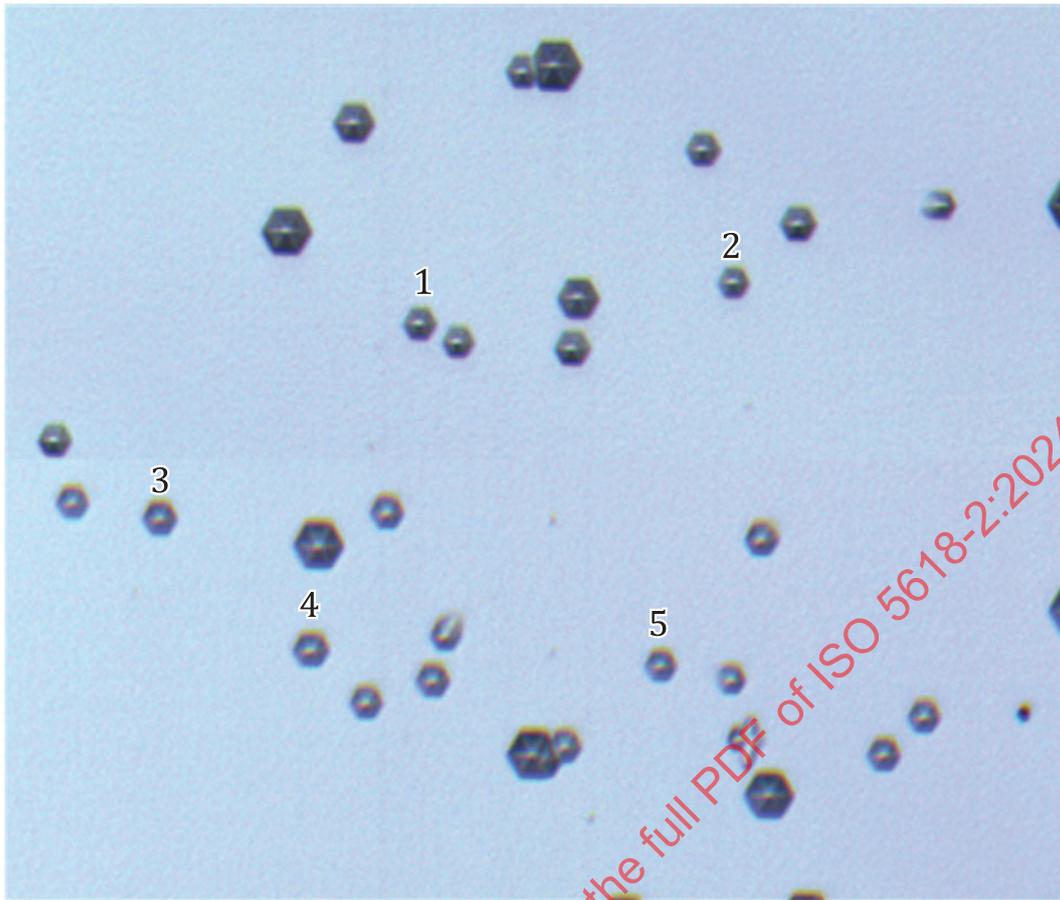
3 inverted hexagonal pyramidal pit categorized into the smallest pit group

4 inverted hexagonal pyramidal pit categorized into the smallest pit group

5 inverted hexagonal pyramidal pit categorized into the smallest pit group

Figure 3 — Example image of etch pits

7.1.3 Measurement area

Determine the measurement area as specified in Annex C so that approximately 500 or more etch pits can be counted at each measurement point.

Set the measurement area to be equal at all of the measurement points.

Set the measurement area to form a square.

In a sample with unevenly distributed dislocations, there can be less than 500 dislocations within the measurement area for one measurement point as determined above.

7.1.4 Measurement points

The measurement points are the nine positions specified in D.2.

If it is necessary to add measurement points to the nine positions specified in D.2, select them from the measurement points specified in D.3.

Align the centre of gravity for the measurement area determined in 7.1.3 with the measurement positions.

Although some substrates can have an uneven dislocation distribution, do not arbitrarily avoid high-density areas during the measurement.

7.2   Capturing an etch pit image by using an optical microscope

Capture digital images by using an optical microscope in accordance with the conditions specified in 7.1.

If the measurement area cannot fit in the field of view for the optical microscope, stitch the captured images while shifting the field of view to capture an image of the measurement area that you have measured.

To capture the area of the measured view accurately, use a standard scale that is in accordance with ISO/IEC 17025, a standard scale with certified calibration can be obtained from the by National Metrology Institutes (NMI's) or regional metrology organizations, such as Asia Pacific Metrology Programme (APMP), THE European Association Of National Metrology Institutes (EURAMET) and The Inter-American Metrology System (SIM), to measure it in advance.

8   Method of calculating the etch pit density

8.1   Etch pit counting criteria

8.1.1   Counting targets

Inverted hexagonal pyramidal pits and oval pits that have a core (i.e., the vertex of an inverted hexagonal pyramidal pit or the deep hole on one side of an oval pit; refer to Figure 4).

8.1.2   Counting criteria

Regardless of an etch pit's size and shape (i.e., oval or inverted hexagonal pyramid), count it as 1 etch pit. Refer to 1 and 2 in Figure 4.

If any etch pits overlap, count the etch pit cores. Refer to Key 3 in Figure 4. Key 3 is counted as four etch pits.

If an oval pit is combined with an inverted hexagonal pyramidal pit, count it as 1 etch pit. Refer to 4 in Figure 4.

Exclude etch pits located on the circumference of the measurement area from the count. Refer to 5 in Figure 4.

Key
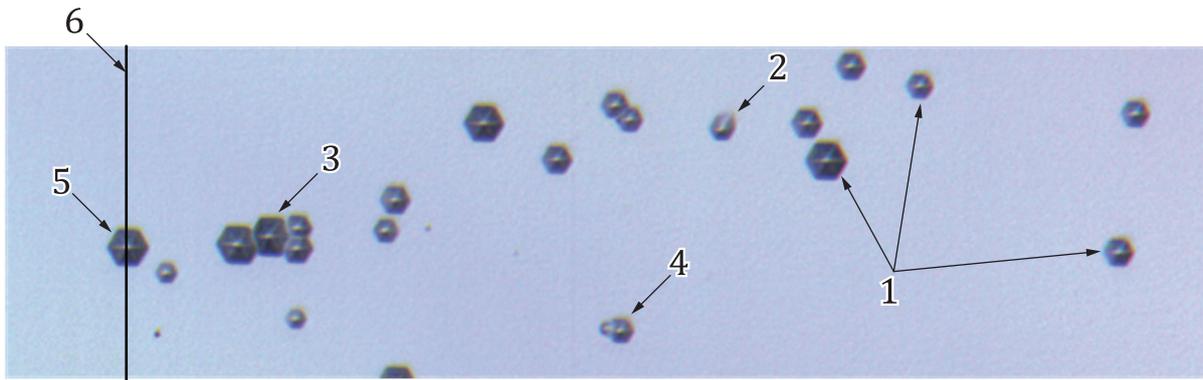
1	inverted hexagonal pyramidal pit	4	combination of an oval pit and an inverted hexagonal pyramidal pit
2	oval pit	5	etch pit to be excluded from counting
3	overlapped etch pits	6	boundary line

Figure 4 — Counting criteria for etch pits cropped out on the (0001)Ga polar face

8.2 Counting etch pits

Using the image captured in Clause 7, count the etch pits that 8.1.1 defines as countable in accordance with the criteria specified in 8.1.2.

Count the etch pits by using one of the following methods: visual counting, semi-automatic counting using counting software, automatic counting using image processing software, and automatic counting using image processing software and machine learning.

8.3 Method of calculating the etch pit density

Calculate the total number of etch pits as specified in 8.1.1 that have been counted in accordance with the counting criteria specified in 8.1.2 (D_{total}), the total number of inverted hexagonal pyramidal pits as well as combinations of oval pits and inverted hexagonal pyramidal pits (E_{total}), and the total number of oval pits (B_{total}).

The etch pit density is calculated by dividing the total number of etch pits by the area of their measurement areas. Set the unit to be cm^{-2}.

Express the etch pit density by using an index number with one decimal place. Round off the density value to one decimal point.

$$P_{EPD} = D_{total}/S_{area} \tag{1}$$

$$P_{TD\text{-}EPD} = E_{total}/S_{area} \tag{2}$$

$$P_{BPD\text{-}EPD} = B_{total}/S_{area} \tag{3}$$

where

P_{EPD} etch pit density caused by dislocations, in cm^{-2};

$P_{TD\text{-}EPD}$ etch pit density caused by threading dislocations, in cm^{-2};

$P_{BPD\text{-}EPD}$ etch pit density caused by basal plane dislocations, in cm^{-2};

S_{area} area of the measurement area.

8.4   Method of calculating the coefficient of variation for the etch pit density

Calculate the average of the P_{EPD}s measured at the respective measurement points.

With the P_{EPD} at position i set to $P_{EPD\text{-}i}$ and the number of positions set to n, the average of the P_{EPD}s ($P_{EPD\text{-}AV}$) is calculated by using Formula (4):

$$P_{EPD-AV} = \frac{1}{n} \sum_{=1}^{n} P_{EPD-i} \tag{4}$$

The standard deviation (S_d) is calculated using Formula (5).

$$S_d = \sqrt{\frac{1}{n} \sum_{=1}^{n} \left(P_{EPD-i} - P_{EPD-AV} \right)^2} \tag{5}$$

The coefficient of variation (C_{ov}) is calculated by dividing the standard deviation (S_d) by $P_{EPD\text{-}AV}$. See Formula (6)

$$C_{OV} = S_d / P_{EPD-AV} \tag{6}$$

9   Categorising the inverted hexagonal pyramidal etch pits by size and calculating their percentages

9.1   Judging whether inverted hexagonal pyramidal pits can be categorized by size

If the supplier and measurer agree that it is necessary, judge whether inverted hexagonal pyramidal pits can be categorized by size by using the image captured in Clause 7 in accordance with the procedure specified in Annex E. If it is judged to be possible, categorize the inverted hexagonal etch pit by following procedure.

9.2   Principle of categorizing dislocations by inverted hexagonal pyramidal pit size

Since the size of an inverted hexagonal pyramidal pit is proportionate to the size of the Burgers vector for threading dislocations[1,2], the proportion for each dislocation type can be calculated if the inverted hexagonal pyramidal pit size level can be categorized.

On a substrate where threading edge dislocations b = 1a exist, dislocations that form the inverted hexagonal pyramidal pits that fall under the smallest size level correspond to threading edge dislocations b = 1a[4]. Dislocations that form the inverted hexagonal pyramidal pits that fall under the second smallest size level correspond to threading screw dislocations b=1c and threading mixed dislocations b = 1a + 1c[4]. Dislocations that form the inverted hexagonal pyramidal pits that fall under the larger size levels correspond to threading dislocations $|b| > |1a + 1c|$[2].

In certain cases, even if threading mixed dislocations b = 1a + 1c are adjacent to each other at a distance of several dozen nanometres, an inverted hexagonal pyramidal pit that falls under the second smallest size level is formed[2]. This type of dislocation can be identified because it has two cores[2].

Depending on the production method, some of the GaN substrates that are produced do not have threading edge dislocations[3,4]. In this case, the inverted hexagonal pyramidal pits that fall under the second smallest level and the large size levels are possible to be present.

9.3 Determining the inverted hexagonal pyramidal pit sizes

Determine all the lengths of longer diagonals D for independent inverted hexagonal pyramidal pits within the measurement area of the image captured in Clause 7 that do not overlap (refer to Figure 3).

The lengths of longer diagonals can be determined by using one of the following methods.

— Measure the lengths of longer diagonals directly by using image processing software or the like.

— Use image processing software or the like to measure the area S at the bottom of the inverted hexagonal pyramidal pit and then calculate the length of the longer diagonal D by using $2\sqrt{2S/3\sqrt{3}}$

NOTE   The length of the longer diagonal of a regular hexagon with an area S can be calculated by using $2\sqrt{2S/3\sqrt{3}}$.

9.4 Creating a histogram for the inverted hexagonal pyramidal pit sizes

9.4.1 Setting data sections

Set the data sections as follows: L_{min}, $L_{min} + S_t$, $L_{min} + 2 \times S_t$, $L_{min} + 3 \times S_t$ …. , $L_{min} + n \times {}^*S_t$, $L_{min} + (n + 1) \times S_t$.

Here, n is a natural number, L_{min} is the minimum value for the lengths of longer diagonals of the inverted hexagonal pyramidal pits, and S_t is one-tenth of the average length of longer diagonal, which was calculated in 7.1.2 (one-twentieth if the length of longer diagonal was calculated based on the area).

In addition, set the data sections so that $L_{min} + n \times S_t < L_{max} < L_{min} + (n + 1) \times S_t$. Here, L_{max} is the maximum value for the lengths of longer diagonals of the inverted hexagonal pyramidal pits.

9.4.2 Generating a histogram

Calculate the number of pits for the sizes that correspond to the predefined data sections and generate a histogram as shown in Figure 5. Determine whether a histogram for each measurement point, a histogram for the sum of the measurement results for all the measurement areas, or both should be created based on an agreement between the measurer and supplier.

Key

X   the range of the length of the longer diagonal   ∘∘∘∘∘∘ Small: gaussian function with smallest b_i in convolution of the
     of inverted hexagonal pyramidal pit   gaussian function

$$\sum_{i=1}^{m} a_i \times \exp\left[-(x-b_i)^2 / 2c_i^2\right]$$ fitted to the histogram   for
the inverted hexagonal pyramidal pit sizes

Y   count of inverted hexagonal pyramidal pit   - - -   Large: gaussian function with second smallest b_i in
                                                          convolution of the gaussian function

$$\sum_{i=1}^{m} a_i \times \exp\left[-(x-b_i)^2 / 2c_i^2\right]$$ fitted to the histogram   for
the inverted hexagonal pyramidal pit sizes

☐ Count: histogram for the inverted hexagonal   Sum: convolution of the gaussian function
  pit size

$$\sum_{i=1}^{m} a_i \times \exp\left[-(x-b_i)^2 / 2c_i^2\right]$$ fitted to the histogram   for
the inverted hexagonal pyramidal pit sizes

Figure 5 — Example of a histogram for the inverted hexagonal pyramidal pit sizes and their fit with the sum of Gaussian functions

9.5   Analysing a histogram

9.5.1   Method of analysing a histogram

Fit the histogram by convolution of the Gaussian function $\sum_{i=1}^{m} a_i \times \exp\left[-(x-b_i)^2 / 2c_i^2\right]$.

Increment the Gaussian function convolution count m, starting from 1, until an appropriate fit is achieved.

Create a single graph of the Gaussian functions to which the histogram has been fitted. Create two types of this graph: a linear plot, as shown in Figure 5, and a semi-log plot, as shown in Figure 6 (frequency is plotted as the log).

Generally, there are two or more size levels. Since the third and subsequent smallest size levels do not appear very frequently, they can be overlooked in the fitting and linear plots. Therefore, a semi-log plot of the histogram for the inverted hexagonal pyramidal pit size is also required.

Key

X — the range of the length of the longer diagonal of inverted hexagonal pyramidal pit

∘∘∘∘∘∘ Small: gaussian function with smallest b_i in convolution of the gaussian function $\sum_{i=1}^{m} a_i \times \exp\left[-(x - b_i)^2 / 2c_i^2\right]$ fitted to the histogram for the inverted hexagonal pyramidal pit sizes

Y — count of inverted hexagonal pyramidal pit

- - - Large: gaussian function with second smallest b_i in convolution of the gaussian function $\sum_{i=1}^{m} a_i \times \exp\left[-(x - b_i)^2 / 2c_i^2\right]$ fitted to the histogram for the inverted hexagonal pyramidal pit sizes

▭ Count: histogram for the inverted hexagonal pit size

—— Sum: convolution of the gaussian function $\sum_{i=1}^{m} a_i \times \exp\left[-(x - b_i)^2 / 2c_i^2\right]$ fitted to the histogram for the inverted hexagonal pyramidal pit sizes

Figure 6 — Example of a histogram for the inverted hexagonal pyramidal pit sizes and their fit with the convolution of Gaussian functions (semi-log plot)
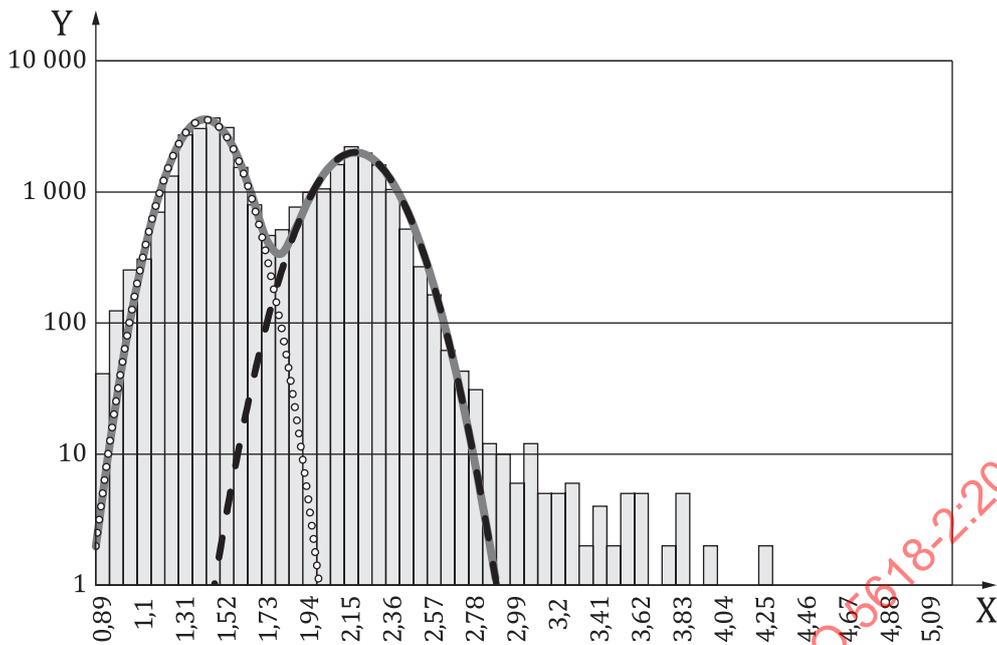
9.5.2 Number of inverted hexagonal pyramidal pit size levels

The number of inverted hexagonal pyramidal pit size levels is equal to the Gaussian function convolution count m.

It is possible to check whether any size levels have not been included in the statistics by using a histogram with a semi-log plot.

9.5.3 Inverted hexagonal pyramidal pit size levels

Each inverted hexagonal pyramidal pit size level is b_i for each of the Gaussian functions to which the histogram has been fitted.

9.5.4 Method of calculating the percentage of an inverted hexagonal pyramidal pit size level

The percentage of an inverted hexagonal pyramidal pit size level is calculated by S_{gi}/S_g. S_{gi} and S_g are expressed using Formula (7) and Formula (8), respectively:

$$S_{gi} = \int_0^\infty a_i \times \exp\left[-(x - b_i)^2 / 2c_i^2\right] dx \tag{7}$$

$$S_g = \sum_{i=1}^n S_{gi} \tag{8}$$

Round off the percentage to the nearest whole number and express it as a percentage (%).

10 Test report

Report the following items as etching results. Report items e) to g) only if it has been agreed that categorization based on the inverted hexagonal pyramidal pit size is required.

a) number of this document, i.e. ISO 5618-2:2024;

b) conditions for the etching process (etchant, temperature, and time);

c) measurement points, measurement areas, number of etch pits, and P_{EPD} at those measurement points
Sum of measurement areas at all measurement points, the sum of etch pits counted, and P_{EPD} for all measurement areas

Coefficient of variation for P_{EPD};

d) measurement points, measurement areas, and number of etch pits caused by threading dislocations and $P_{TD\text{-}EPD}$ as well as number of etch pits caused by basal plane dislocations and $P_{BPD\text{-}EPD}$ at those measurement points

Sum of measurement areas at all measurement points, the sum of etch pits counted, and $P_{TD\text{-}EPD}$ and $P_{BPD\text{-}EPD}$ for all measurement areas;

e) single graph showing a histogram for the lengths of longer diagonals of independent inverted hexagonal pyramidal pits and their fit with the Gaussian function. Both graphs with the linear scale and the semi-log scale (frequency plotted as the log).

Clearly describe whether the graph shows a histogram for the respective measurement points or a histogram for the sum of the measurement points so that they can be differentiated from each other;

f) number of inverted hexagonal pyramidal pit size levels n, each inverted hexagonal pyramidal pit size level b_i (i = 1 to n), and percentage of each inverted hexagonal pyramidal pit size level S_{gi}/S_g (i =1 to n);

g) details on whether threading edge dislocations b = 1a are included in the evaluated substrate as a dislocation type;

h) performed additional polishing or not before etching;

i) sample break or not during etching process;

j) any deviations from the procedure;

k) any unusual features observed;

l) measurement year/month/day and measurer name.

Annex A
(normative)

Verification of dislocation detection by etching

A.1 Purpose

Under some etching conditions, etch pits are not formed at the locations where dislocations are cropped out on the (0001)Ga polar face or the etch pits are too small to be detected with an optical microscope. Therefore, the purpose of this Annex is to prove that the etching process used in this inspection can form etch pits that are identifiable using an optical microscope at all of the locations where dislocations are cropped out on the (0001)Ga surface.

The results of an interlaboratory test indicate that even with the same etchant composition, etchant temperature, and etchant immersion time, etch pits of sizes that are easy to detect using an optical microscope are formed at all of the dislocation positions cropped out on the (0001)Ga for some implementing agencies and that such etch pits are not formed at some dislocation positions cropped out on the (0001)Ga for other agencies. In other words, because it is difficult to judge the appropriateness of the etching based on the etching conditions, the appropriateness should be judged based on the etching results for a reference sample.

A.2 Principle

When a sample's photoluminescence intensity distribution is observed using a multiphoton excitation microscope or its cathodoluminescence intensity distribution is observed using a scanning electron microscope, dark spots are observed at the locations where threading dislocations are cropped out on the (0001)Ga. If the positions of the dark spots show one-to-one correspondence with the positions of the inverted hexagonal pyramidal pits formed during etching after the photoluminescence intensity distribution or cathodoluminescence intensity distribution is observed, it is judged that the etching is appropriate and that all dislocations have been detected.

A.3 Procedures

A.3.1 Observing a reference sample's luminescence intensity distribution

Observe a reference sample's photoluminescence intensity distribution by using a multiphoton excitation microscope or its cathodoluminescence intensity distribution by using a scanning electron microscope within the area in which 100 or more dark spots are observed at the centre of the reference sample.

NOTE   If dust adheres to the surface of the sample, it can be detected as a dark spot.

EXAMPLE 1   The reference conditions for observing the photoluminescence intensity distribution by using a multiphoton excitation microscope are as follows.

After focusing on the surface of a crystal, scan the laser in the in-plane direction to detect light with a wavelength of between 360 and 400 nm emitted from the GaN crystal by using a photomultiplier tube and capture an intensity mapping image. Although clearer dark spots are observed with a higher excitation intensity, this intensity needs to be lower than the ablation intensity on the crystal surface. The appropriate excitation intensity will vary depending on the laser excitation wavelength and pulse width as well as other factors such as the objective lens' numerical aperture.

For example, dislocations were observed as dark spots under the conditions shown in Table A.1.

Table A.1 — Example of measurement conditions

Laser pulse width	120 fs
Laser cyclic frequency	80 MHz
Average laser power	2 mW
Laser excitation wavelength	800 nm
Objective lens	x50, NA 0,80
Observation range	$64 \times 64\ \mu m^2$
Number of pixels	1 024 × 1 024 px
Detection wavelength	360–400 nm
Detector	Photomultiplier tube (GaAsP photoelectric surface)

EXAMPLE 2   The reference conditions for observing the cathodoluminescence intensity distribution by using a scanning electron microscope are as follows.

Scan electron beams with an acceleration voltage of 5 kV or lower in-plane by using a field emission electron microscope to detect light emitted from the GaN crystal or light with a wavelength of between 360 and 400 nm by using a photomultiplier tube or CCD and capture a luminescence intensity mapping image. The appropriate current value will vary depending on the sample, the detection wavelength, and the detector. If dark spots cannot be observed clearly, it is advisable to increase the current of the electron beams by opening the condenser lens, for example, or to cool the sample to an extremely low temperature.

For example, dislocations were observed as dark spots under the conditions shown in Table A.2.

Table A.2 — Example of measurement conditions

Voltage of electron beam	5 kV
Current of electron beam	1 nA
Observation range	$240 \times 180\ \mu m^2$
Number of pixels	1 280 × 1 024 px
Scanning time/frame	80 s
Detection wavelength	Panchromatic mode
Detector	Photomultiplier tube

A.3.2   Etching the reference sample

Etch the observed reference sample to form etch pits on its surface.

Use the etchant composition, etchant temperature, and immersion time as parameters.

If an etchant containing KOH is used, do not use a cover for the crucible during the heating process.

If an etchant containing KOH is used, perform the etching for 120 min or longer after the etching temperature has been reached.

Do not reuse an etchant that has returned to room temperature after use.

Do not use an etchant once impure substances have been deposited on the sides of the crucible containing it during use.

Items exposed to the sample or etchant during etching: Use the same etchant container (e.g., crucible or beaker), sample carrier, thermometer (thermometer or thermometer protective tube), and other instruments throughout the etching process for the reference sample.

A.3.3   Checking the correspondence between etch pits and dark spots

Use an optical microscope to observe the area of the etched sample that was observed using a multiphoton excitation microscope or scanning electron microscope, as shown in Figure A.1. The etching conditions are

judged to be appropriate if the number of etch pits is within the range of 95 % to 105 % of the number of dark spots.
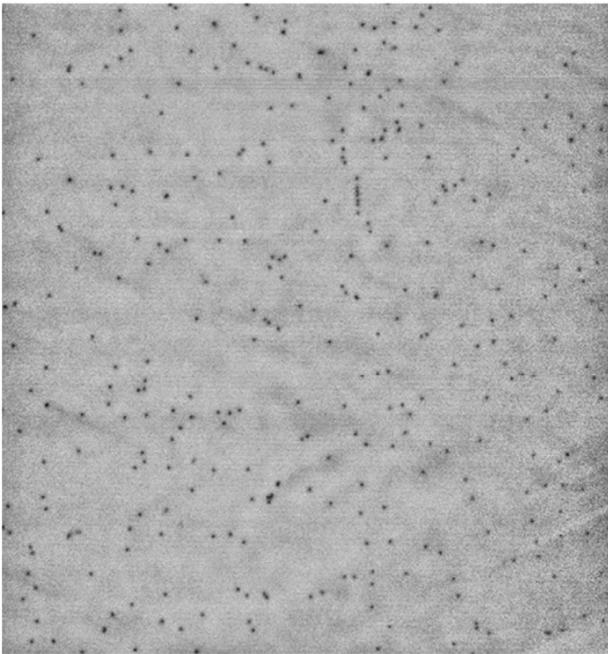
a) Image of the multiphoton excitation PL intensity distribution

b) Image of an etch pit

Figure A.1 — Example of the correspondence between the dark spots in an image of the multiphoton excitation PL intensity distribution and the etch pits

Annex B
(informative)

Appropriate inverted hexagonal pyramidal pit size for etch pit counting

B.1 Purpose

When the etch pit density is measured, the etch pits will overlap if they are too large in relation to the etch pit density, making it difficult to count them accurately. Conversely, if the etch pits are too small in relation to the etch pit density, the observation magnification needs to be increased to an appropriate level, thereby increasing the number of fields of view for observation and making the observation process more complicated. The purpose of this Annex is to provide a range of inverted hexagonal pyramidal pit sizes that will make it easy to count the etch pits with high accuracy.

B.2 Principle

Assuming that inverted hexagonal pyramidal pits of the same size are distributed evenly throughout a sample with a certain etch pit density, overlapping of the etch pits can be avoided with a high degree of probability if the size is limited to that required to secure sufficient space for a single inverted hexagonal pyramidal pit between the respective etch pits.

If the inverted hexagonal pyramidal pit size that corresponds to the smallest size level is double the resolution of the objective lens for the optical microscope used for observation of the etch pits, miscounting can be avoided with a high degree of probability.

B.3 Procedures

B.3.1 Calculating the etch pit density for judgment ($P_{tempEPD}$)

Observe an area measuring 100 μm × 100 μm at (0,0) on the sample by using a microscope and count the etch pits regardless of their sizes. Set this value to E.

$E > 3$ Calculate the etch pit density for judgment by using the following Formula (B.1):

$$P_{tempEPD} = E \times 10^4 \text{ cm}^{-2} \tag{B.1}$$

$E < 2$ Observe an area measuring 1 mm × 1 mm at (0,0) on the sample by using a microscope and count the etch pits. Set this value to F.

Calculate the etch pit density for judgment by using the following Formula (B.2):

$$P_{tempEPD} = F \times 10^2 \text{ cm}^{-2} \tag{B.2}$$

The etch pit density for judgment is not the etch pit density for a sample.

B.3.2 Upper limit for the inverted hexagonal pyramidal pit sizes

If there is only one size level for the inverted hexagonal pyramidal pits, set an upper limit for that size. If there are multiple size levels for the inverted hexagonal pyramidal pits, set an upper limit for the largest inverted hexagonal pyramidal pit.

The upper limit value of the lengths of longer diagonals D for the inverted hexagonal pyramidal pits should be less than half the square root of the inverse number of the etch pit density for judgment ($P_{tempEPD}$).

$$D(\mu m) \leq 1 \times 10^4 \sqrt{1/P_{tempEPD}} / 2 \qquad (B.3)$$

EXAMPLE   For the inverted hexagonal pyramidal pit size distribution shown in Figure B.1, key A is set an upper size limit.

### B.3.3   Lower limit for the inverted hexagonal pyramidal pit sizes

If there is only one size level for the inverted hexagonal pyramidal pits, set a lower limit for that size. If there are multiple size levels for the inverted hexagonal pyramidal pits, set a lower limit for the inverted hexagonal pyramidal pit of the smallest size level.

The lower limit value of the lengths of longer diagonals D for the inverted hexagonal pyramidal pits should be at least double the resolution of the objective lens used for observation of the inverted hexagonal pyramidal pits. The resolution based on the Rayleigh criterion is expressed using Formula (B.4):

Resolution = kλ/NA (λ = 550 nm k = 0,61)   (B.4)

EXAMPLE 1   If an objective lens with an NA value of 0,95 is used, the resolution is 0,35 μm. A value double that resolution (i.e., 0,7 μm) is set as the lower limit of the lengths of longer diagonals D for the inverted hexagonal pyramidal pits.

EXAMPLE 2   For the inverted hexagonal pyramidal pit size distribution shown in Figure B.1, the inverted hexagonal pyramidal pit that is close in size to key B is set a lower size limit. In this example, the inverted hexagonal pyramidal pits can be clearly categorized into four size levels.

Key
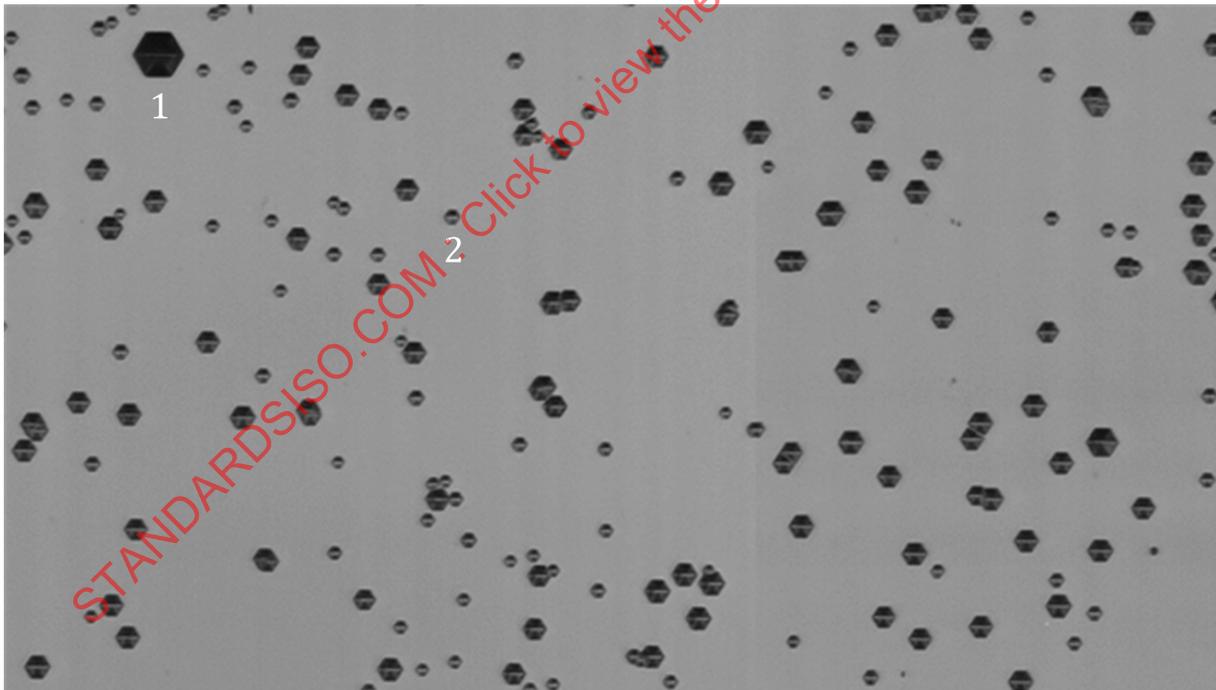
1   inverted hexagonal pyramidal pit of the largest size level
2   inverted hexagonal pyramidal pit of the smallest size level

Figure B.1 — Example image of etch pits

Annex C
(normative)

Measurement area

C.1   Purpose

When the number of etch pits is counted, the error in the etch pit density becomes large if the total number of pits counted is small. Conversely, the error in the obtained etch pit density becomes small if the total number of pits counted is large, but efficiency is reduced due to the effort required to do so. The purpose of this Annex is to provide a method of determining the minimum measurement area.

C.2   Principle

As the number of etch pits to be counted at one measurement point increases, the average coefficient of variation for the density becomes smaller and the measurement accuracy improves. A counting example is shown in Figure C.1. Along with the count quantity, the coefficient of variation decreases while the accuracy improves for crystals A and B. This document requires approximately 500 etch pits or more to be measured per measurement point. If the number of etch pits per 1 cm^2 is less than 500, a measurement area of 1 cm^2 is sufficient because the value is a true value.

The coefficient of variation is affected in not only the count quantity per measurement point, but also the unevenness of the dislocation distribution. As shown in Figure C.1, crystal A, where the distribution of dislocations is notably uneven, has a high coefficient of variation, while crystal B, where the distribution of dislocations is not quite as uneven, has a low coefficient of variation.