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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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Foreword

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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.

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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

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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 5618-2:2024

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

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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

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

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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

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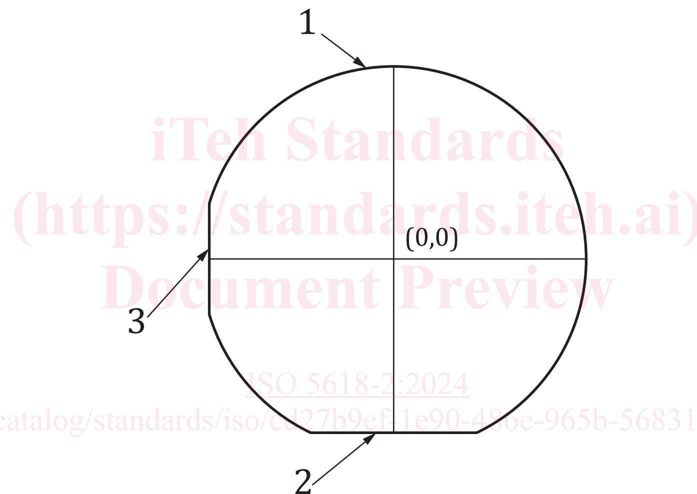
4 Principle

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).



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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 ≦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.

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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.

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6.3 Washing

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

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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.

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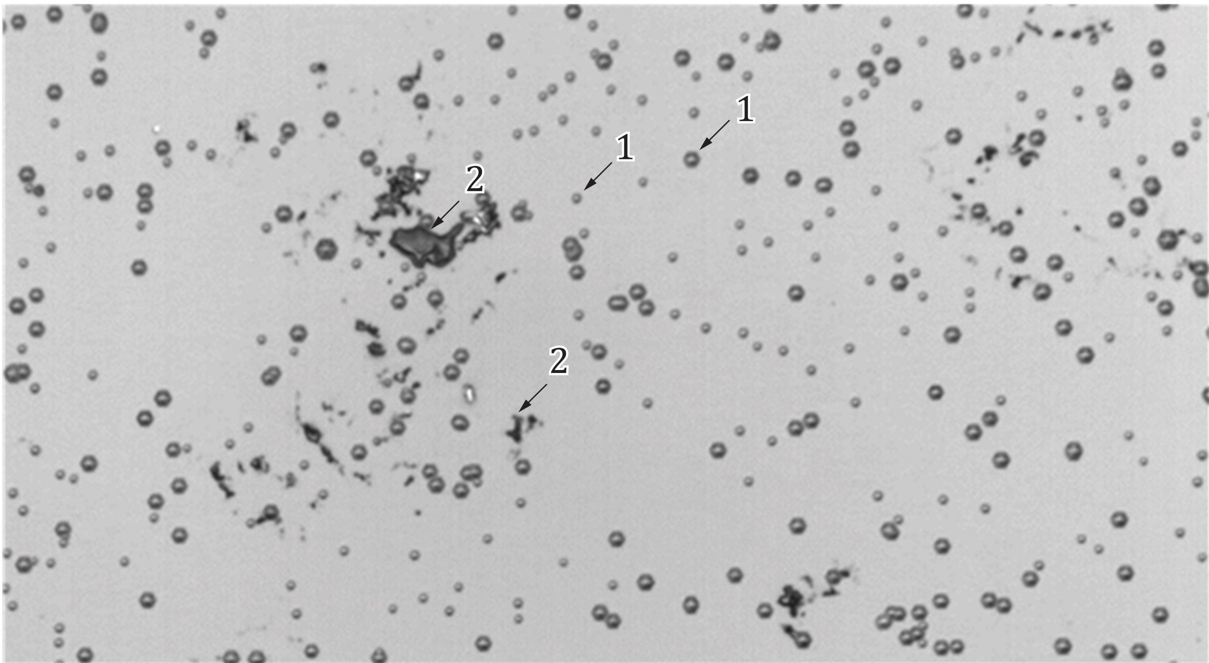

Key

1 etch pit

2 adhered matter

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

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7 Method of capturing an etch pit image

7.1 Setting the observation conditions for an optical microscope

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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.

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