



## Test Method for Crystallographic Perfection of Gallium Arsenide by Molten Potassium Hydroxide (KOH) Etch Technique<sup>1</sup>

This standard is issued under the fixed designation F 1404; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method is used to determine whether an ingot or wafer of gallium arsenide is monocrystalline and, if so, to measure the etch pit density and to judge the nature of crystal imperfections. To the extent possible, it follows the corresponding test method for silicon, Test Method F 47. Test Method F 47 also presents the definition of many crystallographic terms, applicable to this test method.

1.2 This procedure is suitable for gallium arsenide crystals with etch pit densities between 0 and 200 000/cm<sup>2</sup>.

1.3 Gallium arsenide, either doped or undoped, and with various electrical properties, may be evaluated by this test method. The front surface normal direction of the sample must be parallel to the <001> within  $\pm 5^\circ$  and must be suitably prepared by polishing or etching, or both. Unremoved processing damage may lead to etch pits, obscuring the quality of the bulk crystal.

1.4 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 8.

### 2. Referenced Documents

#### 2.1 ASTM Standards:<sup>2</sup>

D 1125 Test Methods for Electrical Conductivity and Resistivity of Water

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

F 26 Test Methods for Determining the Orientation of a Semiconductive Single Crystal

F 47 Test Method for Crystallographic Perfection of Silicon

by Preferential Etch Techniques<sup>3</sup>

### 3. Summary of Test Method

3.1 The determination of the etch pit density is only meaningful for monocrystalline material. After a mechanical or chemical polish, or both, of the sample surface, the sample is etched in molten KOH. This agent preferentially attacks the gallium arsenide surface in regions of crystal imperfections, such as low angle grain boundaries, twin lamellae, precipitates, slip lines, and dislocations. The etched surface is examined microscopically to characterize these imperfections, and determine their density.

3.2 Viewed through an optical microscope, etch pits appear as dark elongated hexagonal pits. The etch pit density (EPD) is determined by counting these pits at nine different standardized locations across the sample along <011> and <001> directions. A lens micrometer or a grid installed in the microscope is used to define the sampling area. The reported EPD is obtained by averaging the EPD values in the nine counted areas.

3.2.1 The orientation of the elongated KOH etch pits may also be used to determine the crystal orientation prior to the addition of flats to gallium arsenide (GaAs) wafers or crystals.

### 4. Significance and Use

4.1 The use of GaAs for semiconductor devices requires a consistent atomic lattice structure. However, lattice or crystal line defects of various types and quantities are always present, and rarely homogeneously distributed. It is important to determine the mean value and the spatial distribution of the etch pit density.

### 5. Characteristics of Revealed Imperfections

5.1 The KOH etch of the specimen surface reveals patterns that are characteristic for several of the crystalline defects described in detail in Test Method F 47.

5.1.1 Dislocations on {100} GaAs surfaces are characterized by microscopic anisotropic six-sided etch pits. The size of the pits depends on the consistency of the etch and the etching time and will be typically 25 to 50 μm for the procedure

<sup>1</sup> This test method is under the jurisdiction of F-1 on Electronics and is the direct responsibility of Subcommittee F01.15 on Gallium Arsenide.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Discontinued; see 1997 *Annual Book of ASTM Standards*, Vol 10.05.

described in Section 9. Because the sides of these pits are not normal to the incident light, they appear dark under vertical field illumination. The use of a Nomarski microscope is optional.

5.1.2 *Lineage*, a precursor to a low-angle boundary, appears as a linear array of etch pits with a density greater than 25 pits/mm. For this test method, linear arrays less than 0.5 mm in length are not considered lineage. The individual etch pits are aligned end to end, or side to side. The lineage does not necessarily follow a  $\langle 110 \rangle$  direction.

5.1.3 *Slip* is evidenced by a pattern of one or more straight lines of etch pits that do not necessarily touch each other. The ends of the anisotropic etch pits will be on a common line. This line of etch pits will be in a  $\langle 110 \rangle$  direction.

5.1.4 A grain boundary appears as a grooved line of any length in which individual etch pits cannot be resolved microscopically at 200 $\times$  magnification. The grooved lines may enclose an area of the etched surface or extend to the periphery of the specimen.

5.1.5 A twin boundary appears as a straight line at the intersection of a crystallographic plane (usually a  $\langle 111 \rangle$  plane) and the etched surface under examination. Two parallel twin boundaries that are separated by only a few crystal lattice planes form a twin lamella that appears as a straight grooved line.

## 6. Apparatus

6.1 *Slicing Equipment*—Typically an inside diameter (ID) saw. Such a saw produces a minimum amount of cutting damage.

6.2 *Wafer Preparation Equipment*—This equipment includes lapping and polishing facilities capable of removing a minimum of 12  $\mu$ m from the surface to be characterized. A polishing etch may be used in place of the wafer polisher, but will require substantially more stock removal (50  $\mu$ m minimum).

6.3 *Laboratory Equipment*—Nickel crucibles and tweezers are necessary to work with molten KOH. Platinum or zirconium have also been used successfully and can be substituted for the nickel tools.

6.4 *Device*, capable of heating the crucible with the samples to 500°C.

6.5 *Microscope*, provided with 10 $\times$  and 20 $\times$  magnification objective lenses, a 10 $\times$  magnification eye piece, a 0.5-mm pitch micrometer, and a metric stage micrometer.

## 7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Where available, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.<sup>4</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity that it will not reduce the accuracy of the test.

<sup>4</sup> "Reagent Chemicals, American Chemical Society Specifications," Am. Chem. Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

7.2 *Purity of Water*—Reference to water shall be understood to mean either distilled or deionized water with a resistivity greater than 2 M $\Omega$ ·cm at 25°C, as determined by the nonreferee method of Test Methods D 1125.

7.3 *Chemical Polish*—One of the following:

7.3.1 *Polishing Etch*, (such as bromine/methanol, or sulfuric acid/hydrogen peroxide).

7.3.2 *Sodium Hypochlorite*.

7.4 *Lapping Abrasive*—Alumina, Size 5 (0.06 to 0.3  $\mu$ m).

7.5 *Degreasing Chemicals*—As required according to previous process such as:

7.5.1 *1,1,1-trichloroethane (TCA 1-1-1)*,

7.5.2 *Acetone*,

7.5.3 *Isopropanol* (2-propanol), and

7.5.4 *Other Wax-Removing Solvent*.

7.6 *Defect Etch*:

7.6.1 *Potassium Hydroxide (KOH)*, anhydrous.

## 8. Hazards

8.1 The chemicals used in this evaluation procedure are potentially harmful and must be handled with the utmost care at all times. Read the most current copy of the Material Safety Data Sheet (MSDS) for each chemical used. Wear protective gloves and a safety mask so that molten KOH cannot contact your skin. Safety glasses must be worn at all times. Observe common laboratory safety precautions. Dispose of all chemicals properly.

## 9. Sample

9.1 The wafer to be measured must be free of inclusions, large grains and twins. Those would interfere with the determination of the average EPD value.

9.2 The procedure applies to crystals grown by any method, such as Liquid Encapsulated Czochralski (LEC), Horizontal Bridgman (HB), and Vertical Gradient Freeze (VGF). The sample surface must be oriented within 5° parallel to a  $\langle 100 \rangle$  plane.

## 10. Procedure

10.1 Orient the ingot so that the front surface normal direction of the sample is parallel to the  $\langle 001 \rangle$  within 5°. Either the X-ray or the optical method of Test Methods F 26 can be applied. Cut a wafer at least 0.025 in.-thick from the crystal. If the crystal has no flats, notch a  $\{110\}$  edge of the wafer. This will later permit locating areas for etch pit counting. LEC crystals grown on  $\langle 100 \rangle$  result in round wafers. HB wafers are D-shaped, unless processed into round wafers.

10.2 Polish the wafer. Afterwards, the wafer must be cleaned and dried. Make sure that a minimum of 0.0015 in. has been removed from each side. If the wafer appears contaminated or not fully polished, repeat the polishing process.

10.3 If the wafer was exposed to wax during previous processes, it must be fully degreased. Immerse the wafer for five min in hot (60°C) 1,1,1-trichloroethane, followed by 5 min in cold 1,1,1-trichloroethane, followed by an acetone dip and by an isopropanol dip. Finally, immerse the wafer for five min in hot (60°C) isopropanol; remove the wafer and allow to air dry.

10.4 Place the wafer in the center of the bottom of a nickel crucible. If several wafers are treated simultaneously, the wafers should not touch each other or the walls of the crucible. A large, flat crucible may be necessary.

10.5 Cover the wafers with KOH completely. Use KOH sparingly and avoid skin contact; remember to wear eye protection.

10.6 Preheat the heating device to 450°C. Place the lid on the crucible. Place the crucible on the heating device. Check after 3 min to verify that KOH is completely molten; if not, increase the heat-up time. Leave the crucible on the heating device for additional 7 min.

10.7 Using long tongs, remove the crucible from the heating device and place it on a hot pad nearby. Remove the lid.

10.8 Pour the molten KOH into a second nickel crucible. The molten KOH can be used once more for a second batch of wafers.

10.9 Using the nickel tweezers, place the wafer(s) nearly upright along the wall of the crucible so that most of the molten KOH drips off. This will also allow for easy wafer removal once the KOH freezes. Allow the wafers to cool for 5 min.

10.10 Place the wafer(s) under running deionized water until the remaining solid KOH is fully removed. Any KOH remaining in the crucible may be removed the same way.

10.11 Examine the wafer. The previously polished surface should now have a dull, matte appearance. It may exhibit some cellular structures. Examine it under the microscope with a 10× objective lens to determine if the proper development of etch pits has occurred. If no pits have developed, repeat 10.4 through 10.11 with an adjustment of the heating period, for example, to a total time of 15 min.

#### 10.12 *Adjustments for Overetched Wafers:*

10.12.1 If large and overcrowded pits are present the sample may have been etched too long, or at too high a temperature.

10.12.2 Change the objective lens to 20× magnification and check again. If the pits are still too difficult to count, repeat 10.1 to 10.11 for a shorter etch time, for example, to a total time of 7 min.

10.13 For etch pit densities less than 500/cm<sup>2</sup>, choose a field of view that results in a minimum of 20 pits and a maximum of 150 pits in each counting area. The field selection should remain as representative as possible.

10.14 The etch pit density is to be established in 9 locations. These locations are defined in Table 1 for round wafers such as produced by the LEC process and in Table 2 for D-shaped wafers produced by the HB method. Table 2 has been taken from Table 1 of Test Method F 47.

10.14.1 For LEC GaAs the EPD distribution does not have circular symmetry. The average EPD does not fully describe the condition of the entire wafer.

10.14.2 For specimens with EPD greater than 500/cm<sup>2</sup>, a 10× objective lens is recommended. A20× objective is recommended when counting EPDs in the range of 30 000 to 200 000/cm<sup>2</sup>.

10.15 *Round LEC Wafers*— To count the EPD, place the etched specimen on the microscope stage so that the major flat faces towards the operator. If the sample has no flats, orient it so that the long axis of the pits point toward the operator. Note the location of the reference notch from 10.1 for test records.

10.15.1 Measure the diameter of the wafer using the Vernier scale of the microscope stage. Determine the nine counting positions according to Table 1 and Fig. 1.

NOTE 1—The order of the counting locations differs from Test Method F 47 to avoid interference with the flats on GaAs wafers.

10.15.2 With the wafer flat facing the operator, move the wafer so that the 0.5-mm micrometre disk is centered in Position 1. Count the etch pits and record the results as well as the microscope objective magnification. Repeat the procedure for Position 2 etc. Upon reaching Position 5, rotate the wafer 45° clockwise and continue for Positions 6 through 9. In calculating the average EPD, be sure to count Position 3 only once.

#### 10.16 *{100} Oriented D-Shaped Wafers From Boules Grown by the Bridgman Method:*

10.16.1 The etch pit density of these wafers will be counted at 9 locations that are different from the locations used for LEC wafers. Because of the wafer shape asymmetry, and because the KOH etch pits on D-shaped wafers have a more uniform distribution, the two counting axes are chosen at 90° to one another as shown in Fig. 2.

10.16.2 To count the etch pits, construct the two axes of Fig. 2. The first axis is perpendicular to the flat part of the wafer. The second axis is perpendicular to the first axis, and bisects it.

10.16.3 Measure the length of each axis to the nearest millimetre.

10.16.4 Locate the nine counting positions along both axes according to Table 2. With the flat side of the wafer pointing away from the operator, begin with Location 1 and continue to Position 5. Rotate the wafer 90° counterclockwise and proceed with Positions 6 through 9. Record results and conditions as described in 10.15.2.

#### 10.17 *Very High and Very Low Etch Pit Density Procedure:*

10.17.1 If for an LEC sample the total number of pits counted for the 9 positions is less than 50, double the counted area by rotating the specimen clockwise 90° from Position 9 and repeat the counting process. Alternatively, the whole area of the specimen can be counted, and the average EPD would be calculated using the total specimen surface area. The latter method is also applicable to HB samples.

**TABLE 1 Test Locations for Round GaAs Wafers**

NOTE 1—Reference point is the wafer edge opposite to the major flat.

Wafer Diameter	Positions 1 and 6	Positions 2 and 7	Position 3	Positions 4 and 8	Positions 5 and 9
2 in. (50.8 mm)	4 mm	15 mm	26 mm	37 mm	48 mm
3 in. (76.2 mm)	5 mm	21.5 mm	38 mm	54.5 mm	71 mm
3.94 in. (100 mm)	7 mm	28.5 mm	50 mm	71.5 mm	93 mm

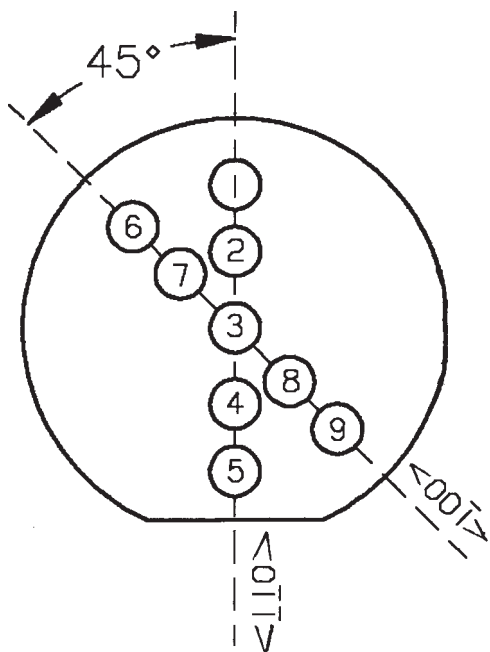


FIG. 1 Position of Count Locations for Round Wafers

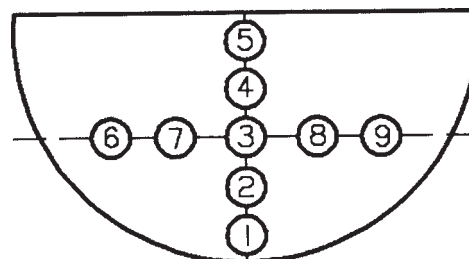


FIG. 2 Position of Count Locations for D-shaped Wafers

10.17.2 If there are too many pits to be counted with the 10× objective lens, change the object lens to a 20× magnification, or count ¼ of the grid. If the pits are still difficult to count, the etch has probably been too severe or the EPD of the sample is too high to obtain a reliable estimate.

10.18 Microscopically scan the entire etched surface of the sample and record on a diagram area of high etch pit densities, grain boundaries, slip, or other crystalline imperfections.

TABLE 2 Test Locations for D-Shaped GaAs Wafers

NOTE 1—Locations taken from Test Method F 47, Table 1.

Slice Diameter, mm	Count Position, mm from edge					Slice Diameter, mm	Count Position, mm from edge				
	Field 1, 6	Field 2, 7	Field 3	Field 4, 8	Field 5, 9		Field 1, 6	Field 2, 7	Field 3	Field 4, 8	Field 5, 9
10	1.5	2.7	5.0	7.3	8.5	61	4.5	13.5	30.5	47.5	56.5
11	1.5	2.9	5.5	8.1	9.5	62	4.5	13.7	31.0	48.3	57.5
12	1.6	3.1	6.0	8.9	10.4	63	4.6	13.9	31.5	49.1	58.4
13	1.6	3.3	6.5	9.7	11.4	64	4.7	14.1	32.0	49.9	59.3
14	1.7	3.5	7.0	10.5	12.3	65	4.7	14.3	32.5	50.7	60.3
15	1.8	3.7	7.5	11.3	13.2	66	4.8	14.5	33.0	51.5	61.2
16	1.8	4.0	8.0	12.0	14.2	67	4.8	14.7	33.5	52.3	62.2
17	1.9	4.2	8.5	12.8	15.1	68	4.9	14.9	34.0	53.1	63.1
18	1.9	4.4	9.0	13.6	16.1	69	5.0	15.2	34.5	53.8	64.0
19	2.0	4.6	9.5	14.4	17.0	70	5.0	15.4	35.0	54.6	65.0
20	2.1	4.8	10.0	15.2	17.9	71	5.1	15.6	35.5	55.4	65.9
21	2.1	5.0	10.5	16.0	18.9	72	5.1	15.8	36.0	56.2	66.9
22	2.2	5.2	11.0	16.8	19.8	73	5.2	16.0	36.5	57.0	67.8
23	2.2	5.4	11.5	17.6	20.8	74	5.3	16.2	37.0	57.8	68.7
24	2.3	5.6	12.0	18.4	21.7	75	5.3	16.4	37.5	58.6	69.7
25	2.4	5.9	12.5	19.1	22.6	76	5.4	16.6	38.0	59.4	70.6
26	2.4	6.1	13.0	19.9	23.6	77	5.4	16.8	38.5	60.2	71.6
27	2.5	6.3	13.5	20.7	24.5	78	5.5	17.1	39.0	60.9	72.5
28	2.5	6.5	14.0	21.5	25.5	79	5.5	17.3	39.5	61.7	73.5
29	2.6	6.7	14.5	22.3	26.4	80	5.6	17.5	40.0	62.5	74.4
30	2.7	6.9	15.0	23.1	27.3	81	5.7	17.7	40.5	63.3	75.3
31	2.7	7.1	15.5	23.9	28.3	82	5.7	17.9	41.0	64.1	76.3
32	2.8	7.3	16.0	24.7	29.2	83	5.8	18.1	41.5	64.9	77.2
33	2.8	7.5	16.5	25.5	30.2	84	5.8	18.3	42.0	65.7	78.2
34	2.9	7.8	17.0	26.2	31.1	85	5.9	18.5	42.5	66.5	79.1
35	3.0	8.0	17.5	27.0	32.0	86	6.0	18.8	43.0	67.2	80.0
36	3.0	8.2	18.0	27.8	33.0	87	6.0	19.0	43.5	68.0	81.0
37	3.1	8.4	18.5	28.6	33.9	88	6.1	19.2	44.0	68.8	81.9
38	3.1	8.6	19.0	29.4	34.9	89	6.1	19.4	44.5	69.6	82.9
39	3.2	8.8	19.5	30.2	35.8	90	6.2	19.6	45.0	70.4	83.8
40	3.2	9.0	20.0	31.0	36.8	91	6.3	19.8	45.5	71.2	84.7
41	3.3	9.2	20.5	31.8	37.7	92	6.3	20.0	46.0	72.0	85.7
42	3.4	9.5	21.0	32.5	38.6	93	6.4	20.2	46.5	72.8	86.6
43	3.4	9.7	21.5	33.3	39.6	94	6.4	20.4	47.0	73.6	87.6
44	3.5	9.9	22.0	34.1	40.5	95	6.5	20.7	47.5	74.3	88.5
45	3.5	10.1	22.5	34.9	41.5						
46	3.6	10.3	23.0	35.7	42.4	96	6.5	20.9	48.0	75.1	89.5



TABLE 2 Continued

Slice Diameter, mm	Count Position, mm from edge					Slice Diameter, mm	Count Position, mm from edge				
	Field 1, 6	Field 2, 7	Field 3	Field 4, 8	Field 5, 9		Field 1, 6	Field 2, 7	Field 3	Field 4, 8	Field 5, 9
47	3.7	10.5	23.5	36.5	43.3	97	6.6	21.1	48.5	75.9	90.4
48	3.7	10.7	24.0	37.3	44.3	98	6.7	21.3	49.0	76.7	91.3
49	3.8	10.9	24.5	38.1	45.2	99	6.7	21.5	49.5	77.5	92.3
50	3.8	11.1	25.0	38.9	46.2	100	6.8	21.7	50.0	78.3	93.2
51	3.9	11.4	25.5	39.6	47.1	101	6.9	21.9	50.5	79.1	94.1
52	4.0	11.6	26.0	40.4	48.0	102	6.9	22.2	51.0	79.9	95.1
53	4.0	11.8	26.5	41.2	49.0	103	7.0	22.4	51.5	80.6	96.0
54	4.1	12.0	27.0	42.0	49.9	104	7.0	22.6	52.0	81.4	97.0
55	4.1	12.2	27.5	42.8	50.9	105	7.1	22.8	52.5	82.2	97.9
56	4.2	12.4	28.0	43.6	51.8	106	7.2	23.0	53.0	83.0	98.8
57	4.2	12.6	28.5	44.4	52.8	107	7.2	23.2	53.5	83.8	99.8
58	4.3	12.8	29.0	45.2	53.7	108	7.3	23.4	54.0	84.6	100.7
59	4.4	13.0	29.5	46.0	54.6	109	7.3	23.6	54.5	85.4	101.7
60	4.4	13.3	30.0	46.7	55.6	110	7.4	23.8	55.0	86.2	102.6
111	7.5	24.1	55.5	86.9	103.5	161	10.4	36.2	80.5	124.8	150.6
112	7.5	24.3	56.0	87.7	104.5	162	10.5	36.4	81.0	125.6	151.5
113	7.5	24.4	56.5	88.6	105.5	163	10.5	36.7	81.5	126.3	152.5
114	7.6	24.7	57.0	89.3	106.4	164	10.6	36.9	82.0	127.1	153.4
115	7.7	24.9	57.5	90.1	107.3	165	10.7	37.1	82.5	127.9	154.3
116	7.8	25.1	58.0	90.9	108.2	166	10.7	37.3	83.0	128.7	155.3
117	7.8	25.3	58.5	91.7	109.2	167	10.8	37.5	83.5	129.5	156.2
118	7.9	25.5	59.0	92.5	110.1	168	10.8	37.8	84.0	130.2	157.2
119	7.9	25.7	59.5	93.3	111.1	169	10.9	38.0	84.5	131.0	158.1
120	8.0	26.0	60.0	94.0	112.0	170	11.0	38.2	85.0	131.8	159.0
121	8.1	26.2	60.5	94.8	112.9	171	11.0	38.4	85.5	132.6	160.0
122	8.1	26.4	61.0	95.6	113.9	172	11.1	38.7	86.0	133.3	160.9
123	8.2	26.6	61.5	96.4	114.8	173	11.1	38.9	86.5	134.1	161.9
124	8.2	26.8	62.0	97.2	115.8	174	11.2	39.1	87.0	134.9	162.8
125	8.3	27.0	62.5	98.0	116.7	175	11.3	39.3	87.5	135.7	163.8
126	8.3	27.2	63.0	98.8	117.7	176	11.3	39.5	88.0	136.5	164.7
127	8.4	27.4	63.5	99.6	118.6	177	11.4	39.8	88.5	137.2	165.6
128	8.5	27.6	64.0	100.4	119.5	178	11.4	40.0	89.0	138.0	166.6
129	8.5	27.9	64.5	101.1	120.5	179	11.5	40.2	89.5	138.8	167.5
130	8.6	28.1	65.0	101.9	121.4	180	11.5	40.4	90.0	139.6	168.5
131	8.6	28.3	65.5	102.7	122.4	181	11.6	40.6	90.5	140.4	169.4
132	8.7	28.5	66.0	103.5	123.3	182	11.7	40.9	91.0	141.1	170.3
133	8.8	28.7	66.5	104.3	124.2	183	11.7	41.1	91.5	141.9	171.3
134	8.8	28.9	67.0	105.1	125.2	184	11.8	41.3	92.0	142.7	172.2
135	8.9	29.1	67.5	105.9	126.1	185	11.8	41.5	92.5	143.5	173.2
136	8.9	29.3	68.0	106.7	127.1	186	11.9	41.8	93.0	144.2	174.1
137	9.0	29.6	68.5	107.4	128.0	187	12.0	42.0	93.5	145.0	175.0
138	9.1	29.8	69.0	108.2	128.9	188	12.0	42.2	94.0	145.8	176.0
139	9.1	30.0	69.5	109.0	129.9	189	12.1	42.4	94.5	146.6	176.9
140	9.2	30.2	70.0	109.8	130.8	190	12.1	42.6	95.0	147.4	177.9
141	9.2	30.4	70.5	110.6	131.8	191	12.2	42.9	95.5	148.1	178.8
142	9.3	30.6	71.0	111.4	132.7	192	12.3	43.1	96.0	148.9	179.7
143	9.4	30.8	71.5	112.2	133.6	193	12.3	43.3	96.5	149.7	180.7
144	9.4	31.0	72.0	113.0	134.6	194	12.4	43.5	97.0	150.5	181.6
145	9.5	31.2	72.5	113.8	135.5	195	12.4	43.7	97.5	151.3	182.6
146	9.5	31.5	73.0	114.5	136.5	196	12.5	44.0	98.0	152.0	183.5
147	9.6	31.7	73.5	115.3	137.4	197	12.6	44.2	98.5	152.8	184.4
148	9.7	31.9	74.0	116.1	138.3	198	12.6	44.4	99.0	153.6	185.4
149	9.7	32.1	74.5	116.9	139.3	199	12.7	44.6	99.5	154.4	186.3
150	9.8	32.3	75.0	117.7	140.2	200	12.7	44.9	100.0	155.1	187.3
151	9.8	32.5	75.5	118.5	141.2	201	12.8	45.1	100.5	155.4	188.2
152	9.9	32.7	76.0	119.3	142.1	202	12.9	45.3	101.0	156.7	189.2
153	9.9	32.9	76.5	120.1	143.1	203	12.9	45.5	101.5	157.5	190.1
154	10.0	33.1	77.0	120.9	144.0	204	13.0	45.7	102.0	158.3	191.0
155	10.1	33.4	77.5	121.6	144.9	205	13.0	46.0	102.5	159.0	191.8
156	10.1	33.6	78.0	122.4	145.9	206	13.1	46.2	103.0	159.8	192.9
157	10.2	33.8	78.5	123.2	146.8	207	13.1	46.4	103.5	160.6	193.9
158	10.2	34.0	79.0	124.0	147.8	208	13.2	46.6	104.0	161.4	194.8
159	10.3	34.2	79.5	124.8	148.7	209	13.3	46.9	104.5	162.1	195.7
160	10.4	34.4	80.0	125.6	149.6	210	13.3	47.1	105.0	162.9	196.7



## 11. Calculations

11.1 Compute the average etch pit density for the specimen by averaging the etch pit density for the nine counting positions. The EPD in each field of view is the number of pits counted divided by the area.

## 12. Report

12.1 The report characterizing the perfection of a gallium arsenide crystal shall include:

- 12.1.1 The crystal and slice identifications,
- 12.1.2 A statement as to whether the specimen is a single crystal or is polycrystalline,
- 12.1.3 A sketch showing the location and distribution of twin boundaries, lineage, slip lines, or other crystal imperfections such as regions of unusually high EPD,
- 12.1.4 The average etch pit density,
- 12.1.5 The maximum and minimum etch pit densities found in the nine locations, and their locations,
- 12.1.6 The long direction of anisotropy of the etch pits,
- 12.1.7 The total number of etch pits counted on the entire slice, and
- 12.1.8 The temperature and the duration of the etch in molten KOH.

## 13. Precision and Bias

13.1 *Precision*—When averaging the densities from the 9 (or 18) specified counting positions, the multi-laboratory pre-

cision as defined in Practice E 177, as applied to measurement of a property of a material, is  $\pm 2000/\text{cm}^2$  (2 standard deviations) or  $\pm 35\%$  (R2S %) of the average dislocation etch pit density, whichever of these two values is greater.

13.2 The precision is limited largely by the nonrandom distributions of etch pits in GaAs crystals combined with the inaccuracy of the count locations, and the resulting inherent difficulties in obtaining a statistically significant representation of the etch pit density.

13.3 *Round Robin Results*—Results will be added, after the round robin tests have been completed.

13.4 *Bias*—The bias of this test method is largely limited by the sampling of the specimen. Because this test method is destructive, it is impractical to take a more representative sample from the crystal. For this reason, the sample specimen is taken from that portion of the crystal that solidifies last where normally the crystal imperfection is the greatest. However, considerable axial variations are common. It is also common to report data from a seed and a tail sample.

13.4.1 The bias of this test method is also limited by factors that can contribute to larger EPD, such as work damage.

13.4.2 The bias of this test method may be improved by using the same operators consistently.

## 14. Keywords

14.1 crystal perfection; etch pit density; gallium arsenide; potassium hydroxide etch; semiconductor; single crystal

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