

JIS

JAPANESE
INDUSTRIAL
STANDARD

JIS H 0609 : 1999

**Test methods of crystalline defects in
silicon by preferential etch
techniques**

ICS 31.200 ; 77.120.99

Descriptors : silicon, metalloids, crystals, crystal defects, substrates (insulating),
integrated circuit technology, semiconductor devices, etching,
non-destructive testing

Reference number : JIS H 0609 : 1999 (E)

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at the Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently JIS H 0609 : 1994 is replaced with this Standard.

Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have technical properties. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have the said technical properties.

Date of Establishment: 1966-12-01

Date of Revision: 1999-11-20

Date of Public Notice in Official Gazette: 1999-11-22

Investigated by: Japanese Industrial Standards Committee
Divisional Council on Electronics

JIS H 0609 : 1999, First English edition published in 2004-07

Translated and published by: Japanese Standards Association
4-1-24, Akasaka, Minato-ku, Tokyo, 107-8440 JAPAN

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Printed in Japan

Test methods of crystalline defects in silicon by preferential etch techniques

Introduction This Japanese Industrial Standard has been established to specify the test methods of determining crystalline defects in silicon using preferential etch techniques. Defects in silicon single crystalline affect yield and performance seriously in semiconductor integrated circuits. Traditionally, etching solution which include hexavalent-chromium in large quantities had been used for detection of such defects in silicon single crystal. However, we decided to change direction to etching solution which is hexavalent-chromium-free for environmental protection in 1994. Though this etching solution without hexavalent-chromium was unable to be applied to single crystalline of low resistivity, we established an optimized standard for such low resistivity ones because we had forecast the increase of the utilization of epitaxial wafers using single crystalline of low resistivity substrates.

1 Scope This Standard specifies the detection of crystalline defects in silicon by preferential etch techniques with hexavalent-chromium-free etching solution. The objects are single crystal wafer, epitaxial wafer, and its thermally oxidized wafers. The surface crystal orientations are {100}, {111} and {511}.

Remarks : The types of wafers dealt with in this Standard are as follows:

- a) **single crystal wafer** sliced, lapped, and mirror-polished single crystal wafers
- b) **epitaxial wafer** epitaxial-grown wafers
- c) **thermal oxidized wafer** wafers of a) and b) that are thermally oxidized

For above wafers, applicable etching solutions differ by the resistivity. Preferential etching solution (I) in 5.3 b) applies to high resistivity wafers of more than 0.02 Ω cm, and preferential etching solution (II) in 5.3 c) applies to low resistivity wafers not exceeding 0.02 Ω cm.

2 Normative references The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. If the indication of the year of publication is given to these referred standards, only the edition of indicated year constitutes the provision of this Standard but the revision and amendment made thereafter do not apply. The normative references without the indication of the year of publication apply limiting only to the most recent edition.

JIS K 8355 : 1994 *Acetic acid*

Remarks : ISO 6353-2: 1983, R1 is equivalent to the said standard.

JIS K 8541 : 1994 *Nitric acid*

Remarks : ISO 6353-2 : 1983, R19 is equivalent to the said standard.

JIS K 8550 : 1994 *Silver nitrate*

Remarks : ISO 6353-2 : 1983, R28 is equivalent to the said standard.

JIS K 8819 : 1996 *Hydrofluoric acid*

Remarks : ISO 6353-3 : 1987, R67 is equivalent to the said standard.

JIS K 8913 : 1996 *Potassium iodide*

Remarks : ISO 6353-2 : 1983, R25 is equivalent to the said standard.

3 Definitions For the purposes of this Standard, the following definitions shall apply.

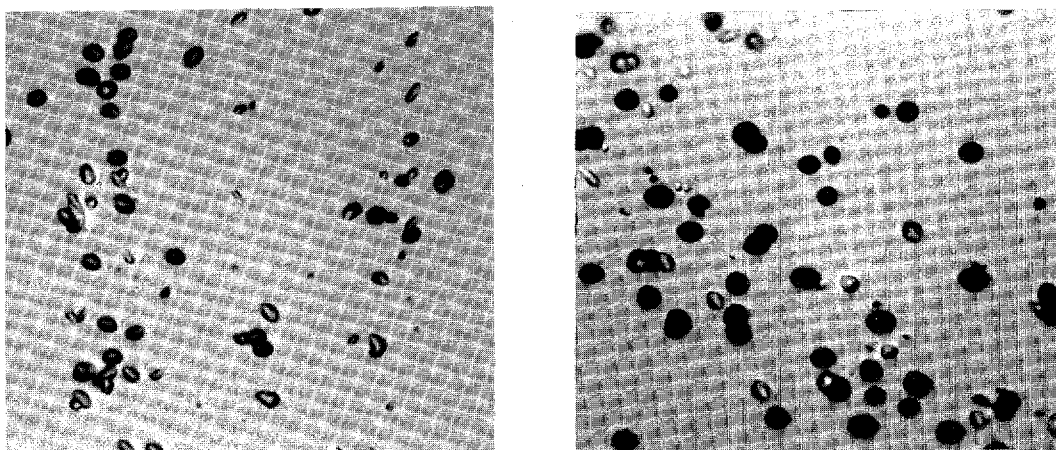
Corresponding English words from ASTM F 1241 : -95 *Standard Terminology of Silicon Technology* were used for consistency.

- a) **dislocation** A kind of lattice defect. The boundary line between slip and non-slip area is called the dislocation line. Dislocation line is confined to the crystal or extended to surface. Dislocation line that intersects the surface can be observed as etch pits after preferential etching solution has been applied (see Photo 1). A slip induced dislocation is called a slip dislocation (see Photos 14, 21 and 26).
- b) **lineage** A low-angle grain boundary resulting from an array of dislocations. This angle is the angle deviation between one part of a crystal and another part. The angle deviation is not more than 1(one) minute. Lineage can be observed as an array of pits (see Photo 2).
- c) **slip** A process of plastic deformation in which one part of a crystal undergoes a shear displacement relative to another in a fashion that preserves the crystallinity of the material. Shear displacement occurs on specific planes along specific directions. On silicon crystal, slip occurs on {111} surfaces along a {110} direction. After preferential etching, slip lines can be observed as parallel lines of dislocation pits. On {111} surfaces, groups of lines are inclined at 60° to each other; on {100} surfaces, they are inclined at 90° to each other (see Photos 3 and 4).
- d) **striation** Helical features on the surface of a silicon wafer observable by the unaided eye after preferential etching. These are continuous striped patterns under 100× magnification. The striations are caused by local variations in resistivity. The phenomena can be explained as a periodic crystalline phenomenon of the rotating solid-liquid interface. Striations may also arise from periodic deviations of dopant concentration, interstitial oxygen, and concentration of unintentionally mixed impurity (see Photo 4).
- e) **swirl** Helical or concentric features that are visible to the unaided eye after preferential etching, and appear as pits forming a discontinuous line under 100× magnification (see Photo 5).
- f) **stacking fault (SF)** A two dimensional defect that results from the abnormal stacking sequence of atoms in a crystal. These exist within the crystal or may be induced by oxidation (oxidation induced stacking fault : OSF). As defect planes propagate parallel with {111} surfaces, they appear as bar-like ditches in certain

directions. On {111} wafers, they appear in three directions and on {100} wafers, in cross directions. (see Photos 6, 11, 13, 15, 17, 19, 22 and 24)

On epitaxial layer, stacking faults originate from contamination or lattice defects on the initial surface layer and grow with the epitaxial layer. On a {111} surface, a stacking fault appears as an equilateral triangle with one, two or three side-walls after preferential etching. On a {100} surface, a stacking fault appears as a square or as two sides of a right angle or as one side only (see Photo 7).

- g) **shallow pits** Etch pits that are small and shallow in depth, observable by eye under high magnification $\geq 200\times$. The cause is metallic impurity contamination or other on silicon surface (see Photo 8).
- h) **precipitates** Regions of silicon compound contained within the silicon lattice frequently manifested as etch pits under high magnification $\geq 100\times$ (see Photo 9).
- i) **bulk micro defects : BMD** Micro defects such as dislocations, stacking faults, and precipitates inside wafer. Usually these appear under high magnification $\geq 100\times$ as {110} cleavage plane of after preferential etching (see Photo 10).
- j) **denuded zone : DZ** A region of the crystal in which precipitation is suppressed by constructing a region of low oxygen concentration under the surface resulting from outward diffusion of lattice oxygen by heat (see Photo 10).



(1) $\{100\}$, C Etch 2 min. ($\times 1000$)

(2) $\{111\}$, C Etch 2 min. ($\times 1000$)

Photo 1 Etch pits (slip induced)

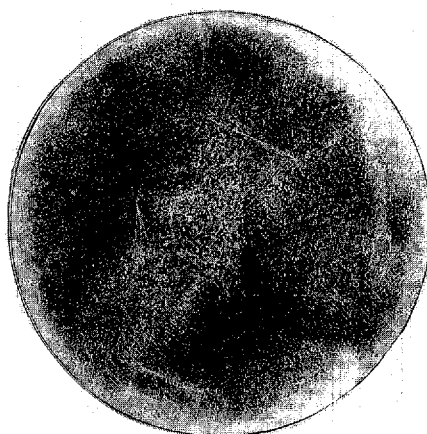
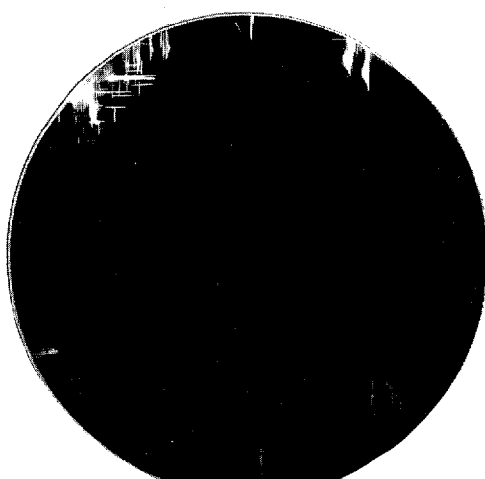


Photo 2 Lineage and etch pits FZ crystal D Etch 10 min.



(1) $\{100\}$, B Etch 2 min.



(2) $\{111\}$, A Etch 2 min.

Photo 3 Slip