Background Statement for SEMI Draft Document 5723A

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Background Statement
Single crystal sapphire is used as a substrate material for manufacturing HB-LED wafers. Despite the importance of single crystal sapphire, however, there currently is no industry-consensus standard in the LED industry for single crystal sapphire. Such a standard would improve communication between users and suppliers, reduce costs, and increase productivity, so it is critical for the LED industry to come to consensus in the near future.

Review and Adjudication Information

<table>
<thead>
<tr>
<th>Task Force Review</th>
<th>Committee Adjudication</th>
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</thead>
<tbody>
<tr>
<td><strong>Group:</strong> Single Crystal Sapphire Task Force</td>
<td>China HB-LED Committee</td>
</tr>
<tr>
<td><strong>Date:</strong> TBD</td>
<td><strong>Date:</strong> Oct. 14th, 2016</td>
</tr>
<tr>
<td><strong>Time &amp;Timezone:</strong> TBD</td>
<td><strong>Time &amp;Timezone:</strong> 9AM—4PM, Beijing time</td>
</tr>
<tr>
<td><strong>Location:</strong> TBD</td>
<td><strong>Location:</strong> Friend Plaza Hotel Dandong</td>
</tr>
<tr>
<td><strong>City, State/Country:</strong> China</td>
<td><strong>City, State/Country:</strong> Dandong, Liaoning, China</td>
</tr>
<tr>
<td>Leader(s): Lena Qi(GHTOT)</td>
<td>Yong Ji(GHTOT), XinHong Yang(AURORA)</td>
</tr>
<tr>
<td>Leader(s): Xuejun Zhang(AURORA)</td>
<td></td>
</tr>
<tr>
<td>Standards Staff: Kris Shen(SEMI China)</td>
<td><strong>Standards Staff:</strong> Kris Shen(SEMI China)</td>
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</table>

Meeting date and time are subject to change, and additional TF review sessions may be scheduled if necessary. Contact the task force leaders or Standards staff for confirmation. Check www.semi.org/standards for the latest schedule.

If you have any questions, please contact the Single Crystal Sapphire Task Force.

   Lena Qi (GHTOT)  
   Tel: +86 18785035580  
   E-mail: lyqi@ghtot.com

Or contact SEMI Staff, Kris Shen at kshen@semi.org
SEM Draft Document 5723A

1 Purpose
1.1 The purpose of this standard is to define the specification of single crystal sapphire intended for use in manufacturing HB-LED wafers.

2 Scope
2.1 This specification includes the physical and chemical properties of single crystal sapphire, and the defects of sapphire.
2.2 The physical and chemical properties include hardness, light transmission, full width at half maximum and the purity.
2.3 The defects include EPD, bubbles, cloud, grain boundary, inclusion, color, and micro crack.

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3 Limitations
3.1 This standard does not define the physical shape or geometry of the sapphire.

4 Referenced Standards and Documents
4.1 SEMI Standards
SEMI M36 — Test Method for Measuring Etch Pit Density in Low Dislocation Density Gallium Arsenide Wafers

4.2 ASTM Standards
ASTM F1316 — Standard Test Method for Measuring the Transmissivity of Transparent Parts
ASTM D5357 — Test Method for Determination of Relative Crystallinity of Zeolite Sodium A by X-ray Diffraction
ASTM C0978 — Standard Test Method for Photoelastic Determination of Residual Stress in a Transparent Glass Matrix Using a Polarizing Microscope and Optical Retardation Compensation

4.3 ISO Standards
ISO 10110-3 — Optics and optical instruments -Preparation of drawings for optical elements and Systems - Part 3: Material imperfections -Bubbles and inclusions

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology
5.1 Abbreviations and Acronyms

1 American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: 610.832.9585, Fax: 610.832.9555, http://www.astm.org

2 International Organization for Standardization, ISO Central Secretariat, 1 rue de Varembe, Case postale 56, CH-1211 Geneva 20, Switzerland; Telephone: 41.22.749.01.11, Fax: 41.22.733.34.30, http://www.iso.ch
5.1.1 FWHM — full width at half maximum
5.1.2 EPD — etch pit density
5.1.3 T% — light transmission
5.1.4 GB — grain boundary

5.2 Definitions

5.2.1 bubble — a crystal defect that exists in single crystal sapphire that presents itself as a void in the crystal, and includes gaseous and vacuum voids. These defects can be observed by projecting bright light through the material and may appear as spherical, pear-shaped, dumb-bell shaped, fiber-shaped, and hexagonal disk-like structures that scatter light.

5.2.2 inclusion — a crystal defect that results from the capture of foreign solid particles in the crystallization growth front (interface) or in the phase segregation during the crystal growth, anneal or cooling stage. These defects can be observed by projecting bright light though the material and appear as irregular shaped light scatterers.

5.2.3 grain boundary — a crystal defect, which is the interface between grains or crystallites of the same phase in monocrystal or polycrystal material. These two grains differ in mutual orientations.

5.2.4 cloud — dense micro bubbles that diminish the clarity of the crystal and appear as fog when the material is illuminated with bright light.

5.2.5 color — crystal defects, which may or may not be eliminated, that cause a selective absorption of light caused by impurity elements or crystal internal defects, and the crystal presents a different color.

6 Ordering Information

6.1 Purchase orders for sapphire goods shall include the following items:

6.1.1 Growth method
6.1.2 Crystal orientation
6.1.3 Orientation target and tolerance
6.1.4 Diameter and tolerance
6.1.5 Bubble and inclusion requirement
6.1.6 Grain boundary requirement

6.2 In addition, ordering information shall include other specific requirements for specific goods, such as length for ingots, thickness for wafers.

7 Requirements

7.1 Physical properties

7.1.1 Hardness — Vickers-hardness of single crystal sapphire should be \( \perp \) C-axis1800~2500HV, // C-axis1800-2300HV according to the method specified in ASTM E384

7.1.2 Light transmission — For HB-LED wafers, the light transmission should be >60% at 250~400nm wavelength, and more than 85% at 400~900nm wavelength according the test method specified in ASTM F1316. The light transmission of single crystal sapphire should be similar to Figure 1.

7.1.2.1 Light transmission depends on the wavelength, sample thickness and the test sample surface quality. Therefore, transmissivity of sapphire wafers shall be measured under the following conditions.

1) Thickness of test part 0.5mm, parallelism of surfaces <30°.
2) Surface finish – polished, roughness <0.3nm.
3) Incident angle \(-0°\pm1°\).
7.1.3  *FWHM*—The full width at half maximum (FWHM) of sapphire for HB-LED wafers shall be no more than 15". According to the XRD test method specified in ASTM D5357, the X-Ray Diffraction FWHM chart similar to the shape as shown as in Figure 2. The curve should be bell shape, there should be only a single peak.

![Figure 1](image1.png)

**Dependence of Transmission on Wavelength**

![Figure 2](image2.png)

**Figure 2**

**X-Ray Diffraction FWHM Chart Example**

7.2  *Defects*

7.2.1 *Purity* — Al2O3 purity of single crystal sapphire intended for use for manufacturing HB-LED wafers must be greater than 99.996%. Table 1 gives the maximum impurities allowed for specific elements. Purity is to be tested using the GDMS(Glow Discharge Mass Spectrometry) test method specified in ASTM F1593.
7.2.2 **EPD** — EPD for single crystal sapphire for HB-LED wafers must be less than 1000 according to the method specified in SEMI M36.

![Figure 3](image)

**Figure 3**  
EPD Sample

7.2.3 **Bubble/inclusion** — It is impractical to avoid bubbles and inclusions in the process of sapphire crystal growth. Sapphire for HB-LED wafers grade 4 levels per diameter of bubble and inclusion, according to the method specified in ISO 10110-3, take a maximum length as diameter for noncircular bubble and inclusion. Bubble and inclusion of single crystal sapphire grade shall be as shown in Table 2.

Table 2 Bubble and Inclusion of Single Crystal Sapphire

<table>
<thead>
<tr>
<th>Grade</th>
<th>Defect Specification</th>
<th>Detection Tool</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>The maximum bubble or inclusion is smaller than 5µm, located not closer than 10mm.</td>
<td>High intensity lamp, 6000~10000LUX</td>
</tr>
<tr>
<td>2</td>
<td>The maximum bubble or inclusion is smaller than 10µm, located not closer than 10mm.</td>
<td>High intensity lamp, 6000~10000LUX</td>
</tr>
<tr>
<td>3</td>
<td>The maximum bubble or inclusion is smaller than 20µm, located not closer than 10mm.</td>
<td>High intensity lamp, 6000~10000LUX</td>
</tr>
<tr>
<td>4</td>
<td>The maximum bubble or inclusion is smaller than 20µm, located not closer than 2mm.</td>
<td>High intensity lamp, 6000~10000LUX</td>
</tr>
</tbody>
</table>

Table 1 Chemical Impurities (unit:ppm wt)

<table>
<thead>
<tr>
<th>Na</th>
<th>Si</th>
<th>Fe</th>
<th>Mg</th>
<th>Ni</th>
<th>Ti</th>
<th>Cr</th>
<th>Mn</th>
<th>Ca</th>
<th>Cu,Mo,W</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;8</td>
<td>&lt;4</td>
<td>&lt;2</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>&lt;2</td>
<td>&lt;1</td>
<td>&lt;2</td>
<td>&lt;2</td>
<td>&lt;3</td>
</tr>
</tbody>
</table>
7.2.4 Grain boundary (GB) — It is impractical to avoid grain boundary in the process of sapphire crystal growth. The grain boundary includes low angle grain boundary and large angle grain boundary, twin crystal, polycrystalline, and crystal lattice distortion. Due to the same test method and similar map, single crystal sapphire for manufacturing HB-LED wafers are graded into 4 levels. Grain boundaries are observed and graded according to the test method specified in ASTM C0978. GB of single crystal sapphire grade shall be as shown in Table 3.

Table 3 GB of Single Crystal Sapphire Grade

<table>
<thead>
<tr>
<th>Grade</th>
<th>Defect Specification</th>
<th>Reference Figure</th>
<th>Detection Tool</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Free of GB</td>
<td>Figure 5.1, 5.2, 5.3</td>
<td>Polarization light 1000~3000LUX</td>
</tr>
<tr>
<td>2</td>
<td>No obvious GB, slight deformation circle and Maltese cross</td>
<td>Figure 5.4, 5.5, 5.6</td>
<td>Polarization light 1000~3000LUX</td>
</tr>
<tr>
<td>3</td>
<td>No more than one sharp GB, the length less than 8mm at each diameter 2 inch ingot, or moderate deformation circle and Maltese cross</td>
<td>Figure 5.7, 5.8, 5.9</td>
<td>Polarization light 1000~3000LUX</td>
</tr>
<tr>
<td>4</td>
<td>Total obvious GB length more than 8mm, or serious deformation circle and Maltese cross</td>
<td>Figure 5.10, 5.11, 5.12</td>
<td>Polarization light 1000~3000LUX</td>
</tr>
</tbody>
</table>

Figure 4.1 Bubble in Ingot  
Figure 4.2 Bubble in Double Polished Wafer

Figure 5.1 GB Grade1 OHT Image  
Figure 5.2 GB Grade1 OHT Image  
Figure 5.3 GB Grade1 OHT Image
7.2.5 Cloud — Perfect sapphire crystal is colorless, and transparent. However, large quantities of micro bubbles that are densely packed will diminish the clarity of the crystal and appear cloudy in the crystal. Single crystal sapphire for HB-LED wafers does not allow any cloud defects visible to the naked eye, with same detection tool and condition for bubbles and inclusions.
7.2.6 Color — Color can be introduced to the crystal by micro impurities or elements captured in the lattice structure during growth or by the oxygen vacancies. These phenomena can cause the sapphire to appear yellow, pink or others color. Sapphire with color is suitable for HB-LED production if the color is removed during the production process (typically during the annealing process). Single crystal sapphire for HB-LED does not allow colors if the color cannot be removed.

7.2.7 Crack — Single crystal sapphire for HB-LED does not allow any crack defects visible to the naked eye.

8 Certification

8.1 Upon request of the customer in the contract or order, a manufacturer’s or supplier’s certification that the material was manufactured and tested in accordance with this specification, together with a report of the test results shall be furnished at the time of shipment.

9 Packing and Package Labeling

9.1 Special packing and package labeling requirements shall be per the agreement between the supplier and the customer. Otherwise all sapphire goods shall be handled, inspected and packed in such a manner as to avoid chipping, scratches, and contamination, and in accordance with the best industry practices to provide enough protection against damage during shipment.

9.2 The shipping package shall have a label placed upon the exterior of the package stating “Fragile”.

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