Cleaving Method for Sample Prep

Cleaving Breakthrough: A New Method Removes Old Limitations

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Manual cleaving, the oldest sample-preparation technique, is still the most efficient and broadly used approach in industries around the world. How cleaving is performed depends on whether the sample has a crystalline substrate or not, although the technique used and the finished results are quite similar. Tools used for cleaving typically include a diamond scribe, paper clip or pin, and tweezers or pliers.

For single-crystal substrates, a line a few millimeters long is scribed near the sample edge that is best aligned with the target. This introduces a weak point, which, along with slight downward pressure, initiates the break (cleave) along the direction of the crystal plane. For non-single-crystal samples (or when attempting to cleave off a crystal plane), the line must be scribed from edge to edge, and then a three-point cleave is performed.

The cleaving process outcome depends on several key factors. These include sample material size and shape; the length, width, and depth of the scribe itself; the pressure, speed, and location of the force used to create the cleave; and the size and location of the target area.[1]

Table 1 illustrates some of the challenges of the scribe-and-cleaving method. In fact, numerous challenges make scribe-and-cleaving less than optimal for a number of samples other than traditional single-crystal silicon wafers or where accuracy is prized. A new approach, called Indent and Cleave (LatticeGear, LLC), results in long, straight, clean cleave lines, even with small, thin, or irregular-edged samples as well as some multicrystalline and some amorphous substrates, such as glass. Table 2 shows how this approach extends and improves on the traditional scribe-and-cleaving capabilities.

Key breakthroughs observed with the indent-and-cleaving method include:

• The smaller and shallower the indent, the stronger the weak point (the “cleave initiator”) will be. (The optimum depth will vary by sample.) This shallower indent results in a straight, clean cleave directly from the leading edge.

• A slow, controlled three-point cleave with equal pressure on both sides of the indent is an important contributor to the cleave quality.

Table 1 Scribe-and-cleaving challenge factors

<table>
<thead>
<tr>
<th>Factor</th>
<th>Challenges</th>
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<tbody>
<tr>
<td>Sample material</td>
<td>Thin samples or brittle ones, such as III-V compound semiconductors, can chip or break unpredictably. For noncrystalline samples, scribing near the edge only chips it and does not initiate a cleave. A sample with many or strong surface structures or one made of a compound material can result in poor cleaves.</td>
</tr>
<tr>
<td>Sample size</td>
<td>Very small or very large samples are tricky to handle and cleave accurately.</td>
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<tr>
<td>Scribe size</td>
<td>A larger, wider, or deeper scribe line makes it harder to predict the direction and quality of the cleave.</td>
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<tr>
<td>Cleaving force</td>
<td>Unequal pressure during the cleave results in reduced surface quality.</td>
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<tr>
<td>Target location/size</td>
<td>When the target is farther away from the edge and/or is smaller, it is more difficult to align the edge scribe with it.</td>
</tr>
<tr>
<td>Cleave quality</td>
<td>Obtaining a high surface quality suitable for SEM imaging</td>
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• Integrated software allows one to draw cleave lines under high magnification from the target to the leading edge. The angled view and integrated indenter tip positioner allow one to perfectly line up the target (or cleave line) with the 10 μm diamond tip.

Figure 1 illustrates the differences in the size of the tips for both the manual scribe tool and the diamond indenter.

The indent-and-cleave apparatus offers a straightforward, repeatable outcome—even with inexperienced users—and takes less than 5 min. It can handle samples 5 mm wide to one-half of a 300 mm wafer and can target a feature to within 10 μm. In many cases, the cross-sectional face will be ready for direct imaging in the scanning electron microscope (SEM) without further preparation.

This precise indent and slow, controlled cleaving technique, integrated with a vision system and software, extends the cleaving sample-preparation method beyond the typical single-crystal sample to samples and applications previously perceived as “noncleavable.” These include semiconductor backend devices, microelectromechanical systems, and 3-D packaging.

Figure 2 shows the results of the two techniques when cleaving sapphire, the third-hardest mineral behind diamond and moissanite. The scribe-and-cleaving manual method produced an irregular, uncontrolled fracture (Fig. 2a); using the indent-and-cleaving method produced a sharp, clean, controlled cleave (Fig. 2b).

In Fig. 3,[3] a 30-μm thin die was depackaged and attached to a host substrate (a standard silicon wafer)

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### Table 2 Comparison of cleaving techniques

<table>
<thead>
<tr>
<th>Factor</th>
<th>Scribe-and-cleaving</th>
<th>Indent-and-cleaving</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample handling</td>
<td>Placed on a surface and handled manually</td>
<td>Placed on a stage, held by vacuum against a positioning guide</td>
</tr>
<tr>
<td>Scribing apparatus</td>
<td>Handheld scribe with a &gt;250 μm hard material tip</td>
<td>A fixed shaft holds a diamond indenter with a 10 μm tip controlled by a precision knob for a repeatable indent.</td>
</tr>
<tr>
<td>Weak-point formation method</td>
<td>Scribing the surface</td>
<td>Indenting the surface—the precision knob moves the indenter tip in Z to control the indent depth on the sample surface.</td>
</tr>
<tr>
<td>Weak-point size from leading edge</td>
<td>Length: a few millimeters Width: &gt;250 μm Diameter: variable</td>
<td>Indent: Length: 1 mm Width: 10 μm Diameter: a couple of microns—controlled and adjustable</td>
</tr>
<tr>
<td>Three-point cleave</td>
<td>Finger tension, pliers, or tweezers are used to press against the scribed surface for a forced cleave.</td>
<td>The sample rests on a pin incorporated in the stage and aligned with the target indent. A knob lowers a bar with two small breaking pins to ensure equal pressure on both indent sides for a slow, controlled cleave.</td>
</tr>
<tr>
<td>Cleaving accuracy</td>
<td>Hand-eye coordination (with or without optical microscope) creates an edge scribe aligned with the target with &gt;100 μm accuracy[2]</td>
<td>Integration of vision system and software coupled with precision stages allow for alignment between the indent and the target with 10 μm accuracy.</td>
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*Fig. 1 Comparison of manual scribe and diamond indenter tip sizes*  
(a) >250 μm scribe tip  
(b) 10 μm indenter tip

*Fig. 2 Results of cleaving sapphire with both methods. In (b), both left and right sides are cleaved edges.*
prior to indenting the host and cleaving through the die. The cleaving direction is dominated by the crystallographic axis of the thick silicon piece. The indent takes place on the support silicon side (either the front or the back), and the thin-die cleaving will always follow the dominating thick silicon direction. Precision cleaving was required in this case to bring the die size down to $7 \times 7$ mm, which is a suitable size to mount on a nanomanipulator sample holder for electrically probing sub-100 nm features.

In a case study at Tescan,\textsuperscript{[4]} the indent-and-cleaving process was used to cross section a solder bump sample, where each bump was 90 $\mu$m wide and 80 $\mu$m deep.

The 5-min indent-and-cleaving process positioned the target 10 to 20 $\mu$m from the edge without additional preparation. The sample was then rough milled at an FIB current of 1 $\mu$A for 7 min, followed by a first polishing with an FIB current of 300 nA for 7 min and a second FIB polishing at 100 nA for 3 min while rocking the sample $\pm 10^\circ$ in the cross-sectional plane to eliminate a curtaining effect. The total preparation time for the indent-and-cleaving method with plasma FIB-SEM (P-FIB) processing was less than 20 min per bump (Fig. 4).

The indent-and-cleaving technique was critical to sample-preparation success because:

- The accuracy of the indent location and a slow cleave resulted in a high-quality cross section with no risk of damaging the structure.
- The resulting cleave was straight and long and created an opportunity to use the P-FIB to prepare more than one bump, as needed.
- Accurate cleaving was important to reduce the rough milling time as well as eliminate an additional polishing step.

Tomas Hrncir, senior researcher and physicist at Tescan, noted that the “precision of the indent-and-cleaving method significantly reduced the milling time, especially for such a large target. It allowed a $90^\circ$ mill and view and made FIB operation easier.”

In a second case study, at the Fraunhofer Center for Applied Microstructure Diagnostics,\textsuperscript{[5]} the goal was to cross section top to bottom through a row of through-silicon vias (TSVs), from the largest to the smallest. The
cross section must be straight, clean, and accurate so that the process variations could be characterized, including thickness of the seed layer, type of insulator layer or barrier material, modified chemical vapor deposition recipes, and temperature regimes.

The TSV sample diameters were oriented horizontally, with nominal diameters of 2, 3, 4, 6, 8, and 10 µm. The depths also varied, from 20 to 30 µm. The TSVs were filled with copper and polished to remove overburden from the surface.

Using the indent-and-cleaving apparatus, the specimens were placed in the correct orientation and held by a vacuum chuck. Using the integrated vision system and software, a cleave line was drawn from and through the TSV row and aligned with the diamond tip. A shallow indent was created at the leading edge, followed by a controlled cleave. The sample was ready for SEM imaging in less than 5 min.

The cleaving separated the copper plug from the titanium/titanium nitride (Ti/TiN) barrier layer or the silicon dioxide (SiO₂) isolation layer in such a way that the copper plug stayed on one side, and the Ti/TiN or SiO₂ could be analyzed on the other side (Fig. 5).

The indent-and-cleaving technique was useful in this case study because:

• The high-quality, accurate, perpendicular cross section allowed high-resolution SEM analysis of the TSV filling, sidewall isolation, and barrier layers from top to bottom.

• Cross-sectional preparation was a time-efficient method suitable for process characterization of multiple TSVs.

• Both cross-sectional sides demonstrated perfect SEM conditions for additional analysis by electron backscatter diffraction or energy-dispersive x-ray spectroscopy.

In a third case study, at X-FAB’s Failure Analysis Lab,[6] the goal was to cross section a monitor wafer with 400 µm bulk silicon with SiO₂ on top and to use design of experiments for the correct TSV etching process to evaluate the copper coating inside the TSV.

Formerly, the sample-preparation workflow included mounting the sample, embedding it in epoxy, and then mechanically grinding and polishing it prior to SEM inspection. While this allowed inspection of the complete TSV in one cross-sectional view, the epoxy embedment was necessary and the complete process was time-consuming (90 min to SEM). In addition, the epoxy could not fill the entire TSV, which resulted in grinding-preparation artifacts, as shown in Fig. 6.
After using the indent-and-cleaving method on similar samples, both sides could be seen in the SEM view, and no material was lost, as shown in Fig. 7.

In Fig. 8, note how the TSV remains connected by the copper layer at the top. The indent-and-cleaving technique facilitated fast preparation for evaluation of the copper coating (under 20 min to SEM), and it allowed inspection of the surface of the copper coating inside the TSV as well as both sides of the TSV for a better overview. The resulting lack of artifacts was another key benefit over the mechanical polishing preparation method.

One drawback with this approach is that the exact thickness of the copper coating cannot be measured. Also, because one cannot rely on hitting a specific TSV, this method is not ideal for single devices. However, for single structures, it becomes an efficient process if this method is followed with additional preparation in a P-FIB.

The three case studies, along with earlier figures, show how the nature of the indent-and-cleaving technique extends the cleaving sample-preparation method to samples and applications previously perceived as “noncleavable.” The resulting high-quality cross sections can preserve important sample information, reduce artifacts, and eliminate additional processing time in further analysis equipment.

The accurate and repeatable indent, the slow and controlled cleaving, the speed of preparation time, the high accuracy, and the high-quality results—regardless of user experience—all combine to broaden cleaving from research to industrial production.

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4. T. Hrncir, Tescan Brno, Czech Republic.


6. E. Brandstädt, X-FAB Semiconductor Foundries AG, Failure Analysis Lab, Erfurt, Germany.

About the Authors

Efrat Moyal has been involved in the semiconductor industry for over 18 years, prior to which she had a successful career as a registered nurse specializing in hyperbaric medicine. Her career in the semiconductor industry has focused on innovative technologies, including equipment, tools, and accessories used by analytical laboratories for process monitoring, failure analysis, and R&D. During her tenure in the industry, Efrat has held positions that have included technical sales and marketing, business development, and upper management at startups such as Sela, Sagitta, and Omniprobe as well as with established corporations such as Gatan and...
Oxford Instruments. Recently, Efrat co-founded a new startup dedicated to innovative and highly accurate cleaving solutions for a wide set of applications and markets. Efrat has co-authored several publications with leading academic institutions, including the University of Central Florida and the Technion, as well as with successful corporations, including FEI, Atmel, and Sematech. She is an active member of the EDFAS community, chairing the ISTFA Expo Committee for the past six years. Efrat resides in the Silicon Valley with her husband Gal and her three children.

Ekkehart Brandstädt received a Master’s degree in chemistry from the University of Halle, Germany, in 1981, whereupon he began working in the microelectronics industry. His first job was in wafer process control in Erfurt, Germany. In 1992, he entered into failure analysis with the newly founded enterprise X-FAB, where he became the failure analysis team leader in 2009.
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