Wafer mounting provides researchers with a configurable platform for electrical characterization of devices and components without the need to pursue the often time-consuming and sometimes costly endeavor of dicing and packaging. Cryogenic probe stations further equip researchers with a system to carry out similar wafer-level characterization with the addition of a vacuum environment and variable temperature. For most room temperature probers, a vacuum sample holder typically holds a wafer or device die to the sample stage during probing operations; however, for a cryogenic station, the vacuum environment surrounding the sample space demands different approaches to sample mounting. In addition to mechanically securing the sample for probing, the mounting methodology must also meet other potential measurement requirements such as high thermal conductivity at cryogenic temperatures in order to achieve the lowest possible device temperature or electrical conductivity for back-side electrical gating. In this application note, several mounting methods for cryogenic probing, summarized in Table 1, will be discussed and compared.
Preferred mounting methods will depend on the user’s needs and the application. Below is a detailed discussion of the most common methodologies employed by Lake Shore probe station users.

**GE varnish**

GE varnish (IMI 7031, formerly GE7031) is a phenolic compound used commercially to coat wires and encapsulate devices. Properly thinned with a 50/50 solution of denatured alcohol and toluene, it has long been used for mounting sensors and wires for cryogenic applications. For removable wafer and die mounting for cryogenic applications, GE varnish provides one of the highest thermal conductances (~0.1 to 1 W/K for a 1 cm$^2$ area)$^1$ and therefore is recommended for applications where an extremely low device temperature is critical. Cure time, vacuum compatibility, and removal process are the principle detractions for wafer mounting with varnish and limit wide applicability. While the varnish tends to harden in 5 to 10 minutes after application, full cure time typically takes 12 to 24 hours at elevated temperatures. When not fully cured, residual solvent can make its way into the probe station’s pump and cause damage or could potentially alter the functionality of surface-sensitive devices under test. Demounting a varnished wafer typically requires soaking the sample holder and wafer in solvent and subsequently applying mechanical stress until the wafer releases. Because GE varnish is electrically insulating, it is not recommended for applications where a back-side voltage (gating) is needed. Cured varnish can be used up to 470 K.

**Vacuum grease and clamping**

Without solvents or curing time, sample mounting with vacuum grease is one of the most popular and flexible approaches used in cryogenic probe stations. With low vapor pressure and higher thermal conductivity, Apiezon® N-grease is a common choice for low temperature applications. At room temperature, the grease is easily spread between the sample wafer and sample holder; below 250 to 280 K, N-grease begins to crystallize$^2$ — mechanically locking the wafer to the stage. In order to maximize thermal performance when mounting samples with vacuum grease, two rules apply — less is more, and force is better. Heat from radiative load, dissipation in the device, and the heat transfer from the probes thermally loads the sample and must be dissipated by conduction from the sample wafer to the sample holder via wafer-metal contact. Vacuum grease fills the uneven, microscopic voids in the sample holder surface so that there are more conduction pathways for heat exchange between the holder and wafer. When the grease layer gets too thick, metal-wafer contact is diminished — resulting in less efficient heat conduction through the grease layer.

While the wafer will be mechanically stabilized by crystallized N-grease at low temperature, grease alone often provides less than satisfactory thermal performance. Figure 1 compares the temperature offset of a probed device compared to the sample stage temperature for a clamped and unclamped device mounted on the sample stage of a Lake Shore CPX-VF probe station. Details of the measurement can be found in the caption of Figure 1 and elsewhere$^3$. Near liquid helium temperatures, the device will be warmer than the sample stage due to heat load from the probe arms (7 to 15 K, typically) and from the radiation shield.

<table>
<thead>
<tr>
<th>METHOD</th>
<th>ADVANTAGES</th>
<th>DISADVANTAGES</th>
</tr>
</thead>
<tbody>
<tr>
<td>GE varnish (IMI 7031)</td>
<td>High thermal conductance</td>
<td>Long cure time, soaking sample and holder in aggressive solvent to remove</td>
</tr>
<tr>
<td>Vacuum grease and clamping</td>
<td>Quick sample exchange, good thermal conductance</td>
<td>Probe interference with clamps, wipe clean with Xylene</td>
</tr>
<tr>
<td>Silver paint and paste</td>
<td>Backside electrical contact, reasonable thermal conductance</td>
<td>During removal with water, mobile silver particles can short or damage devices</td>
</tr>
<tr>
<td>Carbon paste</td>
<td>Backside electrical contact, reasonable thermal conductance, can be used at higher temperatures</td>
<td>During removal with water, mobile carbon particles can short or damage devices</td>
</tr>
<tr>
<td>Cyanocrylate (thin layer)</td>
<td>Quick cure, easy removal, suitable for small surface area</td>
<td>May not endure repeated thermal cycling</td>
</tr>
</tbody>
</table>

Table 1. Summary of recommended sample mounting methods for cryogenic wafer probing
At elevated temperatures, a device in this station will be colder than the sample stage as the probe arms are anchored to a 4.2 K magnet stage and remain near helium temperatures. Here the device temperature offset is determined by the quality of thermal contact of the device to the sample stage. With a clamped wafer, the sensor is less than 0.5 K warmer than the sample stage near helium temperatures and less than 2 K colder than the sample stage at warmer temperatures. With only N-grease and no clamp, the device will be nearly 5 K warmer than the sample stage at helium temperatures and will be almost 30 K colder than the sample stage near 200 K. In both cases, the N-grease begins to melt above 200 K; once melted, the device temperature offset shrinks considerably. The device temperature offset in Figure 1 is indicative of the CPX-VF probe station and will vary across probe station models, probe arm configuration, choice of sample stage, and operating conditions such as cryogen flow rate or power line frequency.

The mechanisms behind this contrast between clamped and unclamped samples are two-fold. Foremost, clamping reduces the thermal contact resistance (TCR) across the solid-solid, wafer-sample holder interface; TCR scales with the contact force not pressure\(^1\); more force, lower contact resistance and better heat transfer. The secondary effect lies in the N-grease layer — unless pressure is applied during pumping and thermal cycling, N-grease layers will develop voids which can further limit thermal conduction\(^4\).

Most users typically fabricate their own clamps tailored to their wafer characteristics and utilize the M3 tapped holes on the edge of the grounded sample holder for wafer attachment. For most probe stations, the tapped holes align with two of the arm locations and, depending on the device pad orientations, can cause interference between the clamp and probe arm. Clever offsetting of the device probing pads from the interior of the clamp can help avoid this interference. Flat-head and other low-profile fasteners are recommended for use with clamps as these decrease the risk of probe arm interference compared to a socket cap machine screw. A mechanical drawing of a generic sample clamp is shown in Figure 2, and this basic design can easily be adapted to a user’s mounting needs. Clamps are typically fabricated from stainless steel or PEEK machinable plastic; 3D printed clamps are also a possibility as additively manufactured components are already finding utility in cryogenic applications\(^5\).

N-grease should be avoided for applications in which the sample stage will exceed 303 K. Above this threshold, the grease liquifies and can migrate around the sample stage and potentially coat electrical contacts or active areas on the top side of wafers. Additionally, the grease will develop a finite vapor pressure, evaporate, and condense on the coldest nearby surface — typically the radiation shield.
Electrically conductive mounting methods

For applications where backside gating or grounding are necessary, samples need to be mounted to the sample holder using an electrically conductive medium such as silver paint, silver paste, or carbon paste. These compounds typically consist of micron or sub-micron scale silver or carbon particles with a binder and dispersed in a liquid solvent. For both adhesion and optimization of thermal transfer, thin bonding layers are critical. When the time comes for sample removal, the wafer can usually be released with application of a shear force to the wafer and then the residue removed from sample and sample holder by wiping with a solvent such as acetone for paint and water for pastes. Due to the resin binder and solvents used in silver paint, application and clean-up is often much easier compared to pastes which may require scraping or polishing to fully remove the residues. With pastes, the constituent conductive particles are often mobile in water and, with soaking, can redeposit on other potentially more critical areas of the wafer die.

Silver paint should be avoided for bonding wafer samples larger that approximately 50 mm$^2$. Likely a result of dissimilar thermal contraction between the sample, paint, and sample holders, microfractures in and delamination of large area silver paint layers have been observed. Microfractured paint layers impair the thermal conductivity of the interface at cryogenic temperatures compared to silver pastes and manifests in warmer device temperatures.

In addition to electrical contact to the backside of the wafer, conductive paints and pastes extend the high temperature range of a measurement when compared to N-grease. Many silver paints can be used up to 375 K while high performance silver and carbon pastes can be used beyond 500 K. It should be noted that at elevated temperatures, these pastes can cure and more permanently adhere substrates the sample holders. Check paint and paste data sheets for safety, cryogenic compatibility and curing information.

Cytanoacrylate adhesives

For small area device die less than a few square millimeters, many of the previously discussed mounted methods are impractical. Provided there are only top-side contacts, smaller dies can be mounted to sample holders or chip carriers with thin layers of cyanoacrylate adhesives for cryogenic characterization. If possible, light pressure should be applied to the die while the glue is still soft in order to achieve a thin glue layer. Thicker glue layers tend to be intolerant of the stresses induced by thermal contraction, and, on fracturing, can cause loss of die in the probe station vacuum chamber.

Conclusions

Wafer mounting methodologies are a key element of wafer-level characterization at cryogenic temperatures. The mounting materials and approach will influence device temperature, cycle time, and even the ability to post process a wafer after characterization. In this application note, several recommended and commonly used methods have been compared.

References


