TEM Specimen Preparation Technique For Small Semiconductor Devices

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One of the methods of preparing samples for analysis by Transmission Electron Microscopy (TEM) is the well-known procedure using a Tripod polisher and the wedge technique (1-3), developed in our laboratory. Though developed explicitly for preparing samples of integrated circuit structures built on silicon, the technique has been used in our lab for a wide variety of other materials, including metals and ceramics. In general when working with silicon samples, we have the luxury of starting with large wafer pieces or chips that are generally at least a couple of millimeters square or larger. Recently, however, we needed to work for an extended period on GaAs based lasers, where the devices requiring analysis were individual lasers extracted from individual packages. These small samples, measuring 100x100x50 microns, are too small for the manual handling involved in routine mechanical cross sectioning methods. A typical example of such a device can be seen in Figure 1 where the actual device, indicated by the arrow, has been detached from the package and is being held by the bond wire. Furthermore, we found the GaAs used in these lasers to be less robust mechanically than silicon during mechanical polishing. This article describes the method we developed for dealing with these tiny devices.

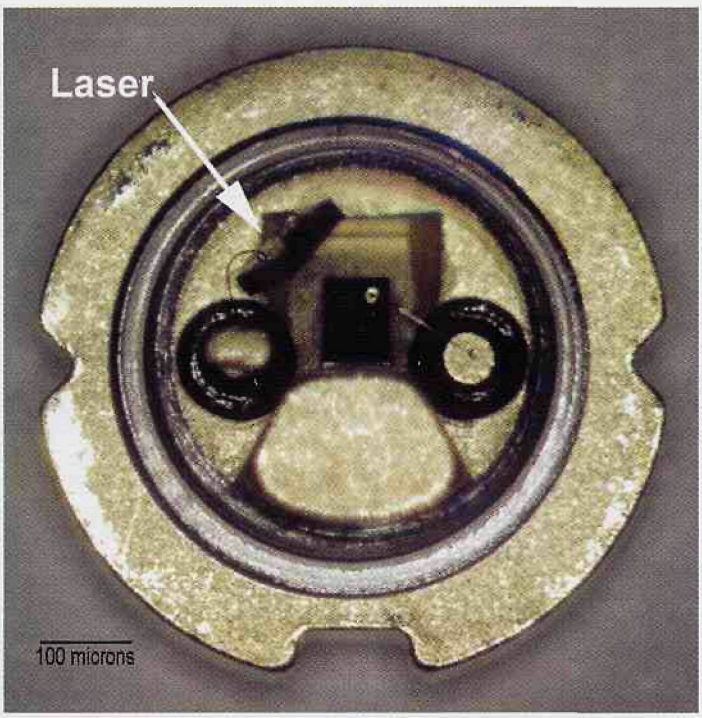

Figure 1 -- Depackaged laser device still attached to package by the bond wire.

A larger sample, for easier manual handling, is made by gluing the original small sample between a larger piece of silicon and a larger piece of dimpled quartz. The dimpled depression in the quartz is just large enough to surround the original sample. The sample is then mechanically thinned down using a Tripod polisher and the wedge technique. The quartz piece, glued to the top of the original sample, allows the progress of the polish to be monitored as the first side of the cross section is being mechanically polished. The silicon piece, glued to the bottom of the original sample, is used to gauge the final thickness of the wedge produced when polishing the second side of the cross section using the wedge technique. A final brief ion mill is used to remove any mechanical damage and to bring the device to electron transparency.

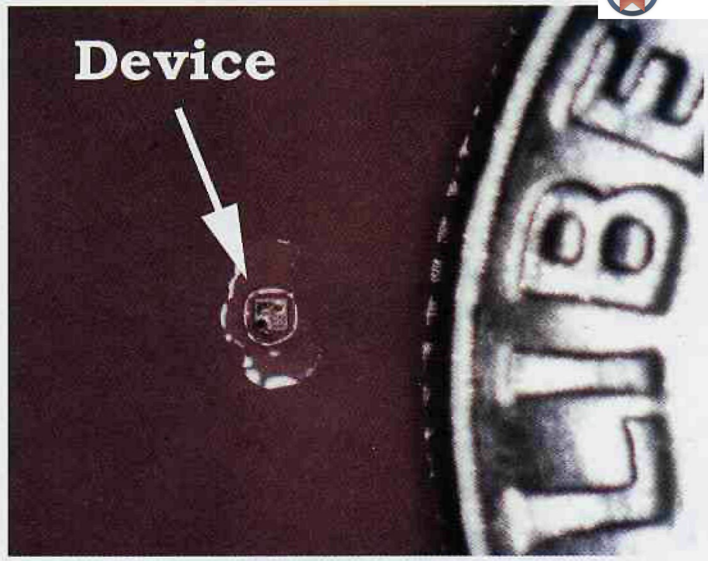

Figure 2 -- Laser device glued to silicon support piece next to a dime.

Before polishing, a pre-glue process is necessary where the three parts: the dimpled quartz piece, the silicon piece, and the sample are glued together in a stack. The first step is to mount the device to a piece of silicon, typically 10x10 mm for easy handling. The gluing process starts with a very small drop of epoxy (we use Gatan G-1) that is applied to the rough, backside, of the silicon piece using the small tip of a toothpick. It is best to use a minimal amount of epoxy for this step, just enough to extend completely under the laser device. The laser is gently pressed down into the glue and the glue is allowed to cure at 80°C for 4 hours. The device, mounted on silicon, is seen in Figure 2, with a dime as a reference for size.

Next, a 10x10x1 mm square is cut from a piece of quartz. The quartz we used was a large block purchased from a local Science and Hobby store. The quartz block had to be cut down to a 10x10mm rod, then sliced into 1mm thick pieces. This square is then mounted on the bottom of a Tripod polisher using glycol pthalate. The quartz is polished flat on both sides using diamond lapping film down to a 3 μm finish, ending with a final thickness of ~0.75 mm. These polishing operations were required to remove the damage from cutting the quartz and make it possible to see through the quartz during the specimen preparation steps. After polishing, the quartz is taken off of the Tripod polisher and a dimple made in the center of the quartz piece using an ultrasonic cutting tool. We used a standard, solid, ultrasonic tip that we modified by fileing the tip down to a size slightly larger than the device we would

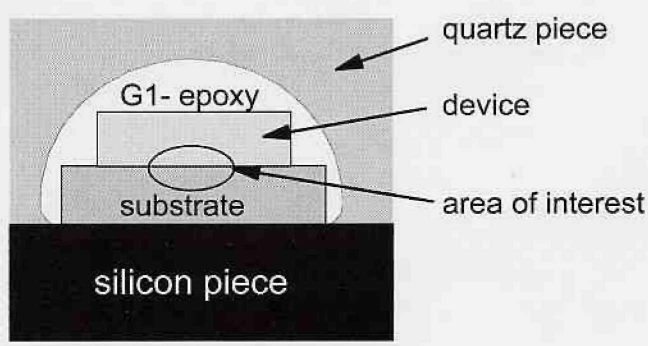

Figure 3 -- Diagram showing encapsulation structure.

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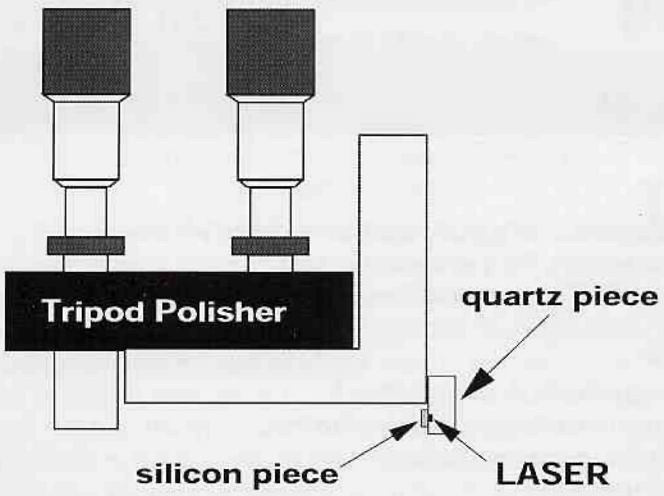

Figure 4 -- Tripod mounting configuration for first side polishing.

be mounting. The depth of the hole was gauged by placing the quartz over the mounted device, and cutting repeated until the quartz and silicon surfaces would meet without the quartz resting on the device. The quartz piece is now cleaned off in acetone and allowed to air dry.

After curing, the dimple in the quartz piece is filled with G-1 epoxy and a small drop of epoxy applied to the top of the device package with a toothpick. Filling the cavity and coating the device with fresh drops of epoxy minimizes the likelihood that an air bubble will be trapped around the device, causing difficulties during mechanical polishing. The dimple is then carefully inverted over the device package and the quartz is pressed down on to the silicon piece with the epoxy drops filling the cavity around the device and extruding out between the quartz and silicon, bonding the pieces together, as seen in Figure 3. The dimple is then inspected to make sure that there are no air bubbles in the epoxy and the epoxy is allowed to cure overnight at 80°C.

This silicon/device/quartz stack is now polished with a Tripod polisher. (South Bay Technology, Inc.) (4)Glycol phthalate is used to attach the stack to a Tripod polisher, with the quartz layer facing towards the front of the polisher Figure 4. The stack is then polished down to the device with progressively finer grits of diamond paper, using only 3 micron or finer to polish the device. The final polishing is done by using 0.25 micron diamond slurry on a "Final A" polishing cloth (Allied High Tech Products Inc.) for thirty seconds, followed by ten seconds of colloidal silica on the same cloth. Longer final polishing times cause rounding of the sample edges and should be avoided. The polished surface is then carefully wiped clean using soap and water, followed by acetone.

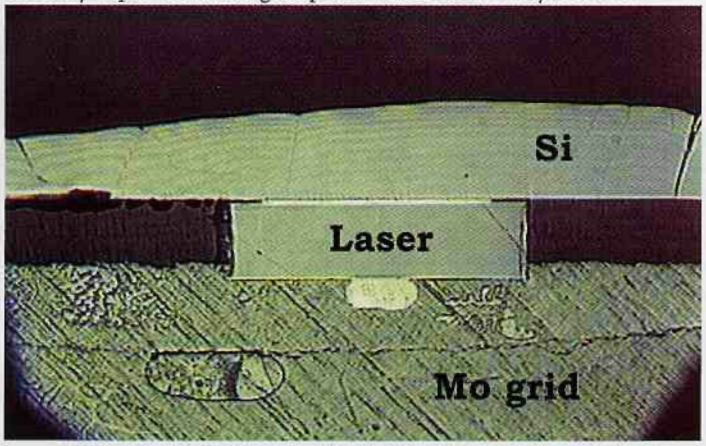

Figure 5 -- Optical image of final polished laser structure. Note the thickness fringes in the silicon support.

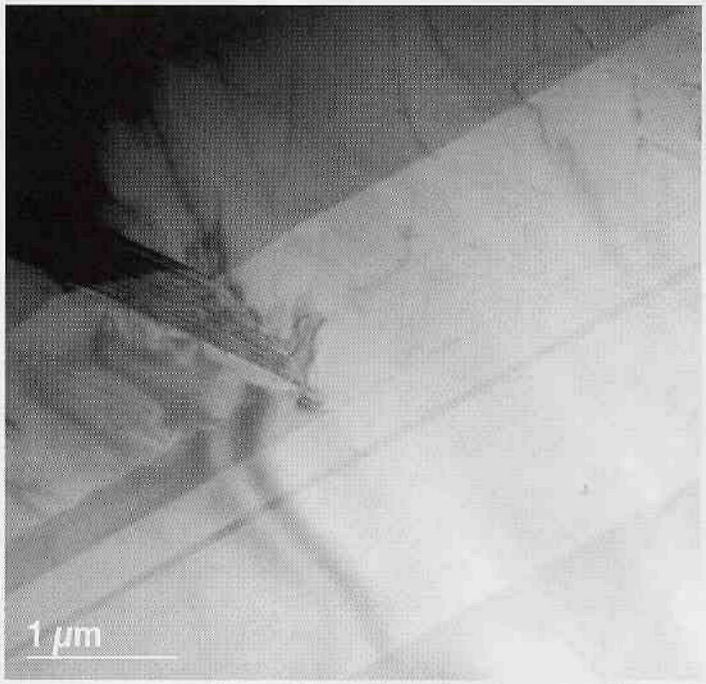

Figure 6 -- TEM image of a typical laser after a few minutes of Ar ion milling to remove polishing damage.

Before second side polishing, a grid is mounted to the first side polished surface. M-Bond 610 epoxy (Measurements Group Inc.) is painted on one side of a 0.4 X 2 mm slotted molybdenum grid. The epoxy side of the grid is then carefully pressed down on the polished surface, aligning one edge of the grid with the back side of the laser device so that the laser/silicon interface is in the grid opening. The M-Bond epoxy is now cured for two hours at 80°C. The Tripod is prepared for wedge polishing using the method discussed in Benedict, *et al.* (1996). A small drop of cyanoacrylate (super) glue is placed in the slot of the grid and on the sample post of the Tripod polisher to minimize the incursion of air bubbles during mounting. The stack is oriented over the sample post so now the silicon layer is facing the front of the polisher. The grid is then carefully pressed down in the glue on the sample post and the excess glue around the grid is removed with a piece of filter paper. The cyanocrylate glue is cured overnight at 80°C.

The rear feet of the Tripod are extended out an additional 100 microns to compensate for the thickness of the grid and provide sufficient wedge for the structural integrity of the sample. The stack is wet polished using diamond paper while monitoring the thickness of the wedge using the changes in transmitted light color of the silicon layer. The stack is mechanically polished until the transmitted light forms a multicolored fringe along the silicon-to-device interface. In cases where the laser substrate cannot be depackaged from the laser, the transmitted light fringes from that part of the device can be used to gauge the final thickness. This can be seen in Figure 5, with the various parts of the specimen identified. The device is thinned to TEM transparency using low angle Ar ion milling and monitoring the thinning process with the TEM. A typical example of a final thinned laser is seen in Figure 6.

Using this technique, we have successfully prepared a large number of laser devices, and have adapted the technique to other structures too small to be prepared by Tripod polishing directly. ■

References

1. S.J. Klepeis et al., Mater. Res. Soc. Proc. 115 (1988) 179.
2. J.P. Benedict et al., Mater. Res. Soc. Proc. 199 (1990) 189.
3. J.P. Benedict et al., Microstructure Science, ASM International 23 (1996) 277.
4. The Tripod Polisher as well as glycol phthalate wax and other supplies is available from South Bay Technology, Inc. (www.southbaytech.com).