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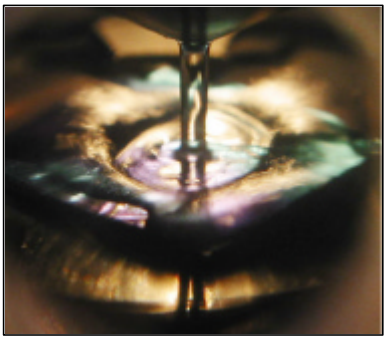
Chemical Thinning of Silicon for TEM with Model 550C Jet Thinning Instrument

When thinning materials that require the use of chemicals such as hydrofluoric acid that will attack stainless steel, quartz and glass, the Model 550C is an excellent tool used for preparing materials in this manner. This instrument can be adapted from the standard electrolytic version using a simple kit that can easily be implemented into the instrument for chemical thinning applications. The conversion kit uses sapphire light rods instead of standard quartz, a PVC reservoir for holding the acid solutions, and PVC or Teflon™ parts in place of metal parts typically used in the electrolytic unit.

The following is a basic step by step procedure describing the basic operation of the Model 550C for chemical thinning applications. It is meant as a guide to users planning on implementing chemical polishing techniques using the Model 550. Materials that can be chemically polished using this instrument include Si, GaAs, and other semiconductor materials. This report uses Si as an example for the process.

1. Cut silicon into standard 3mm diameter discs or squares.
2. Place a mylar disc (p/n: 01-01897) on the recessed area on top of the PVC or Teflon™ pedestal mount. Melt a small amount of mounting wax (MWM070) so that it flows under the mylar disc. Melting of the wax is easily accomplished by using a low temperature hot plate.
3. Center the silicon sample on the mylar disc and melt more wax on the top of the mylar disc to form a fillet of wax which will protect the edges of the sample.
4. Using a cotton swab and acetone, clean the top surface of the silicon sample. Be careful not to remove the wax at the edges as this will protect the edges of the sample during chemical etching.
5. Carefully scrape off the excess mounting wax on the bottom of the mylar disc. This is required to eliminate any misalignment of the sample surface relative to the etching solution.
6. A chemical polishing solution consisting of 360 ml of nitric acid and 90 ml of hydrofluoric acid is used. When mixing these solutions, ALWAYS add the hydrofluoric acid to the nitric acid to prevent any reaction.
7. Using an air hose or air can, blow as much water and fluid out of the jet head assembly and pump assembly as possible. Attach an air hose line (very low pressure line, just requires air flow) to the nozzle located on the pump head assembly. This provides air flow into the pump shaft assembly and will protect the motor and motor assembly from the acidic fumes.
8. Place the pedestal assembly (PVC or Teflon™ combined with the mounted disc) onto the Model 550. Adjust the jet nozzle to a distance of approximately 4mm from the surface of the sample. Jet flow should be as slow as possible without the jet flow breaking into droplets or beads of solution.
9. With sensitivity set to a maximum value, the Model 550 should shut off the jet flow prior to perforation of the sample material. The polishing rate with a fresh solution of acid is approximately 100µm / minute. When the instrument triggers off and shuts down, quickly remove the pedestal assembly using rubber gloves. Rinse off the sample in deionized water and then in methanol.
10. Check the thickness of the sample by viewing in transmitted light using an optical microscope. If the sample is not perforated and a perforation is required, further etching can be accomplished by dipping into the solution for 5 seconds.
11. Dissolve the sample from the mylar disc using acetone and alcohol. Rinse the sample to clean off excess wax.

Model 550 Jet Thinning Instrument.



A view of a properly aligned pump head.