Characterization of the Model 38001 Clamping Jig

1.0: Purpose

A TEM clamping jig has been developed for the rapid production of high quality TEM specimens. This report will cover the basic steps of the procedure, the materials and equipment necessary for the preparation of specimens, and possible improvements and uses for the new jig. The jig is designed for preparing cross sections of any specimens by preparing stacks of material to further reinforce the mechanical strength of the specimen. This "stack method" is used for the preparation of virtually all materials, and will be tested on a select few of materials systems chosen to represent the broad range of materials currently in common use today.

2.0: Experiment

The following materials and equipment are required for the preparation of TEM cross section stacks:

Equipment / Consumable	Description
Model 380 Ultrasonic Cutter	Used to cut out templates for stacks; cutting out tube assembly
Model 3800x TEM Clamping Jig	Includes: 1 clamp (Model 110); Bonding jig; cutting assembly; tube mounts
Model 650 Diamond Wheel Saw	Used to cut the mounted specimen tubes into slices for grinding and dimpling.
Epoxy (MBond 610 or Epotek 353)	Epoxy is used for the bonding of the stack together in the bondig jig.
8 micron Boron Carbide abrasive powder	Powder is used for the cutting of the Si templates as well as the tube
MWH 135 Mounting Wax	Mounting wax is used for mounting the specimen stack into the cutting assembly
Model 360 Disc Cutter	Used in place of the 380 to cut out the specimen tube.
Model 650 Low Speed Diamond Saw	Used to slice out the small sections of the brass tube/specimen assembly for dimpling.

The preparation of these specimen stacks for TEM specimen preparation requires several stages. To begin, several square pieces of a Si wafer are cut out using the Model 380 Ultrasonic Cutter. These squares are 4x5mm in dimension and are about 1 mm thick. They are the stack materials which will be used to provide the specimen with mechanical strength and stability when the specimen is thinned below 20 microns or less. The Si wafer is first mounted to the mounting plate of the Model 380 using a low melting point wax (MWH 135). Next, using the 4 x 5mm cutting tool and 8 micron boron carbide abrasive powder (BC), several squares are cut. (See figure 1 below)

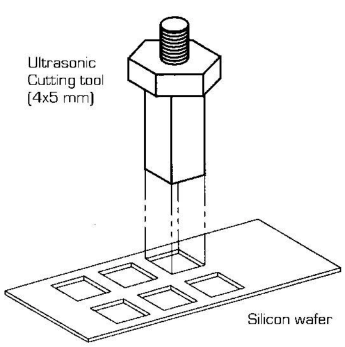

Figure 1: Illustration of the cutting tool used on the Model 380 for the production of the stacking materials for preparing stacked specimens. These 4 x 5 mm Si pieces will then be used as the bonding material for making the actual specimen thicker. Cutting times for each individual square was ~ 45 seconds.

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Once the square material has been cut from the Si wafer, the specimen itself must now be cut. The same procedure used in the above steps is used. All of the pieces should now be cleaned in acetone and ethanol. This will remove any debris which may cause problems in the bonding process.

For bonding, several choices for epoxy are available to the user to choose from. Important factors in this decision are hardness, durability, ability to withstand solvents, mechanical strength, resistance to ion milling, cure time, and thickness. The two best choices for this are the M Bond 610 epoxy and the Epotek 353 epoxy. The M Bond is a slow curing, two part epoxy that maintains strength and stability at elevated temperatures along with high resistance to solvents. The draw back to M Bond is the high temperatures and times needed for curing the adhesive (typically about 200° C and 2 hours curing time). Epotek, on the other hand, has similar properties and cures much faster at lower temperatures. The Epotek epoxy can cure at much lower temperatures (50-80° C) at much faster rates (30-5 minutes). This makes the Epotek a good choice for the rapid production of these specimens.

Once the desired epoxy has been chosen, in this case the Epotek, the specimen is ready to be glued into a thick stack to create a stable specimen structure. To do this, each piece of Si is placed into the teflon Bonding Jig which is used for the stacking process. One by one the Si is placed into the jig, each time applying a drop of epoxy to the surface of the Si piece. Each piece must have a drop of epoxy to ensure that the entire stack is uniformly bonded and strong. Two sacrificial pieces are placed in first, then the actual specimen of interest, then two more sacrificial pieces of Si are then layed on top. The final structure is placed into the Model 110 Specimen Clamp and is used to create a thin, uniform glue line. See Figure 2 below for the illustration of this procedure.

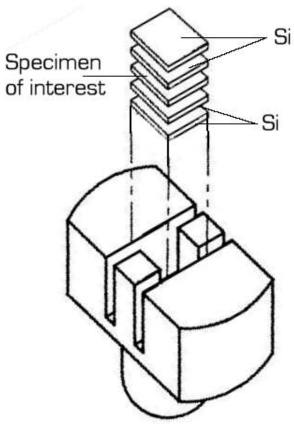

Figure 2: Illustration of the use of the bonding jig for constructing the specimen stack for cross section specimen preparation. The sacrificial stacks of Si are placed both on top and below the actual specimen of interest. In between each square piece of material is where the epoxy is applied. The entire jig is then placed into the specimen clamp to create a thin layer of epoxy in the specimen.

The curing time for this stack to completely cure is around 15-30 minutes on a hot plate set at 100° C. The stack was then removed from the jig and the excess epoxy scraped off with an Exacto knife. Once the stack has been completed, the tube of material must be cut out for assembly into the brass tube. The specimen stack was first mounted into the cutting assembly designed for cutting on the Model 380 Ultrasonic cutter. The specimen stack is oriented into the holder with the stacks of material facing up in cross section. These stacks are then cut into a long tube shape which will be around 2.8 mm in diameter.

Cutting using the Ultrasonic cutter is done as follows: A small amount of 8 micron Boron Carbide powder is placed onto the surface of thematerial. The cutting tool is brought down into contact with the specimen and the amount of material to be cut is measured out on the dial indicator at the top of the unit. A small drop of water is then added to the powder and cutting is started.

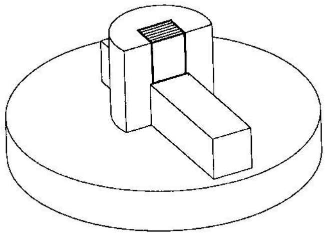

Figure 3: Illustration of the cutting assembly used for mounting and cutting the specimen stacks into a long rod. The specimen rod is then mounted into a brass tube used as the support ring and to provide mechanical support to the stack when thin.

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Once the cutting has been completed, the specimen rod is then ready to be affixed into the brass tube using the same epoxy as was used for creating the specimen stack. This brass tube is placed into the brass support holder to allow the specimen to be sectioned, ground, polished, dimpled, and ion milled all within the specimen tube. The brass tube is first inserted into the teflon ring support holder which will allow the specimen rod to be glued into the brass tube. Drop 2-3 drops of the Epotek epoxy into the tube and then insert the specimen rod. Excess epoxy will either rise to the surface or be forced between the teflon pieces. Allow the specimen to cure for about 30 minutes at 100° C.

The specimen is now permanently affixed inside the brass tube and is ready to be sectioned using a Model 650 Diamond Wheel Saw or equivalent. Sections of 300 micron thick specimens can easily be sliced out using the Model 650.

3.0: Results

Based on the requirements for the successful preparation of specimens which include: Repeatability, ease of use, expense, and success rate, the following was found:

1. Cutting of the bare Si templates is an easily duplicated, fast process. The Si squares are easily cut and the desired shapes are easily obtained.
2. Use of the clamping jig for creating the specimen stacks is an easy, repeatable process as well.
3. Cutting of the specimen tube was not successful for the creation of the tube assembly. Cutting using the Ultrasonic cutter was not successful due to it's lack of ability to cut thick materials > 2 mm in thickness. Cutting time drastically reduces as the specimen thickness is increased.
4. Cutting of the specimen rod was successful using the Model 360 Abrasive Slurry Disc Cutter. The cutting was done using a diamond tipped brass tool of 2.8 mm diameter. The cutting time was 5 minutes per rod.
5. Glueing of the brass tube and sectioning was successful. The specimens are easily created into the 3mm shapped discs required for dimpling, ion milling, and insertion into the electron microscope.

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