Electron
Microscopy
Sciences

INSTRUCTIONAL MANUAL
CAT. 50170-500i Dimpler – Model D500i

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Introduction

The Dimpler is specially designed for the preparation of standard and mechanical pre-thinning in TEM specimens. The use of the Dimpler can be utilized across a variety of applications that involve many different materials such as:

- Germanium
- Carbides
- Silicon
- Gallium arsenide
- Carbon and carbon composites
- Aluminum alloys
- Sapphire
- Glasses
- III-V semiconductors
- Oxides
- Multi-phase materials
- Borides
- Silicides

Specifications

Dimensions	
Length	27” (68.6 cm)
Width	14” (35.6 cm)
Height	13” (33.0 cm)
Weight	70 lbs. (32 kg)
Power	
Standard	120 VAC, 60Hz
Optional	240 VAC, 50Hz
Replacement fuse	
Standard	120 VAC 2A
Optional	240 VAC 1A
Tolerances: Electronic	
Z offset	Accuracy µm
Range	2000µm
Z termination accuracy	
Tools 1i/3i	≤±1µm
Tools 2i/4i	≤±2µm
Tool force	1-200 gm/ 1 gm steps
Balance	Sensitivity < gm
Tool speed	100 to 600 RPM, constant speed
Specimen platen speed	10 RPM, constant speed
Tolerances: Mechanical	
Tool drive shaft	≤0.5µm TIR
Tools	
1i/3i	±1µm TIR
2i/4i	±2µm TIR
Platen surface	Vertical excursions ≤0.5µm TIR

Parts checklist

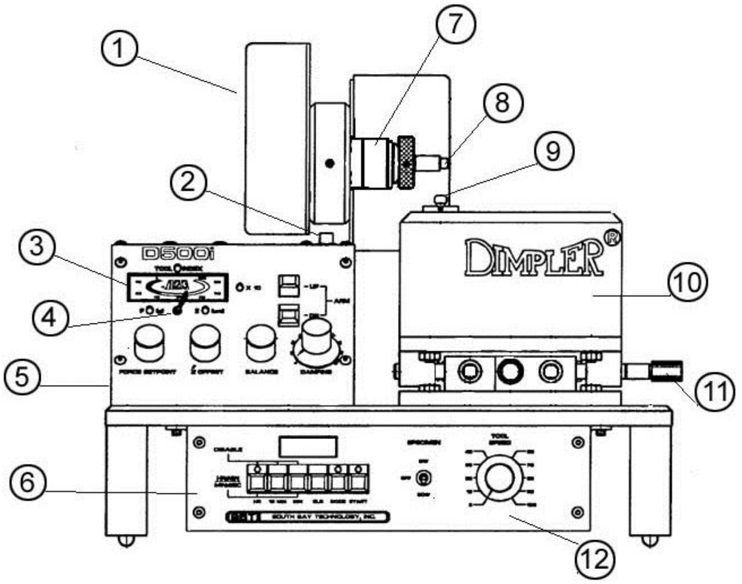
The following parts are included with the instrument:

- Tool retaining screw (1)
- Allen wrench set (1)
- Power cord for D500i (country specific) (1)
- Installation and setup DVD (1)

Instrument layout

The Dimpler is a precision electro-mechanical metallographic lapping instrument that constantly monitors dimpling levels as well as precisely halts at present specimen thicknesses. Due to this revolutionary product, the dimpling process, now automatic, has become much easier in that specimens can now be dimpled very thin. This unit also contains a non-contact Z sensor that works in conjunction with an arm position by a closed feedback loop. This feature allows the tool surface to remain positioned against the specimen surface where the interface is measured precisely. The Z location of the tool surface against the specimen surface is electronically measured and displayed with an accuracy of ±1µm. Similar to precision dial indicators, this system enables the Dimpler to make initial thickness measurements of specimens. The automated features of this product make dimpling of specimens more consistent with less interference of the operator. The fulcrum arm is raised and lowered by push buttons. Aspects of the unit such as the Z offset, dimpling force, damping force, and fine balance can all be set and adjusted from the front panel. When the initial specimen thickness is measured and the specimen is mounted, the operator can then select the dimple depth and begin the application process. The exact Z position of the tool/surface interface is constantly displayed while using any of the tools. Please note than an alarm will sound once the desired dimple depth has been reached. It is at this time that the fulcrum arm will raise, and the tool rotation as well as the timing will stop. Note that if the preset time is reached first, the same sequence for shutoff will occur.

Figure 1. Schematic illustration of the Dimpler Model D-500i.



1 – Fulcrum arm
2 – Non-contact Z position/termination sensor
3 – Digital/analog display of Z position
4 – Toggle switch for Force/Z position
5 – Upper front panel
6 – Lower front panel
7 – Shaft spindle and motor
8 – Tool mounting position
9 – Specimen platen lower
10 – X-Y positioning label
11 – Micrometer
12 – Speed control

Unpacking

Please pay close attention to these unpacking directions as the unit can easily be uncalibrated if not unpacked properly.

1. Remove the instrument from the packing box.

NOTE: Do not pull the instrument out of the box by the arm – doing so will damage the instrument and will cause the unit to go out of calibration. Be sure to remove the instrument from the base plate.

2. Remove the wooden shipping block and save for future use. This supports the fulcrum arm.

3. Lower the rubber stop at the back of the fulcrum arm.

NOTE: Whenever the unit is moved, the block should be used to support the fulcrum arm. The rubber stop at the back of the fulcrum arm should be raised.

4. Connect the power cord to mains supply.

5. Level the base plate by adjusting the left rear leveling foot.

Controls

There are four distinct aspects of the Dimpler that improve the accuracy of termination:

1. Non-contact position sensor
2. Magnetically coupled damping
3. Tool phase sensor
4. Specimen measurement in place

The non-contact position sensor is used to measure the location of the fulcrum arm in relation to the thickness of the specimen. It has better accuracy than that of a micrometer. Due to its magnetically coupled nature to the fulcrum arm, there are no serious effects on the action of the tools on the specimen. The reaction time of the sensor is fast enough to terminate the dimpling process the first time the tool phase sensor indicated that Z termination set point has been reached. The thickness of the specimen is measured directly on the unit with the tool surface as the measuring point. The dimple depth and the Z termination set point are measured with the same reference point – this avoids error in measurement as well is termination error. The combination of the Z offset control and the timer gives the operator two ways of monitoring and terminating in the dimpling process. Another aspect of the Z offset is that is allows material to be removed with all tools before termination occurs. With the timer in present time mode, the process will terminate when the preset time is reached. If, however, the tool reaches the Z offset before the preset time has expired, the process will terminate.

Upper front panel

The table below shows the various modes of the unit as well as their description and function.

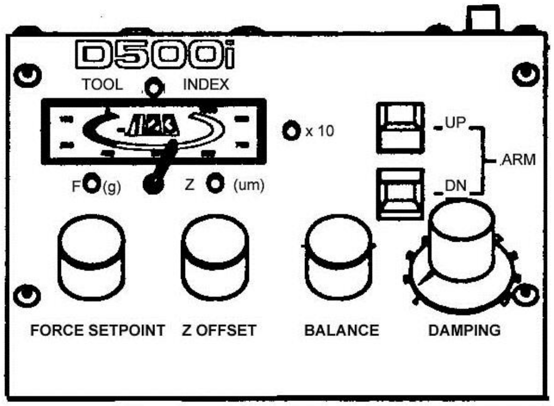
F(g)/Z(μm) Display	The F(g)/Z(μm) Display allows monitoring in two different modes. The F(g), or force mode, provides a digital display of dimpling force in grams. The Z(μm) mode provides a digital and analog display, indicating the Z location of the tool/specimen surface interface for dimple depth in microns, and Z termination.

	The digital display is updated by the tool phase once every revolution of the tool, using the same point on the tool each time. This method of measuring Z termination negates errors due to tool eccentricities. The analog elliptical bar displays the Z position of the tool in real time so tool run out and the effect of different damping settings can be monitored.
F(g)/Z(μm) Toggle	When the arm is in the up position the F(g)/Z(μm) toggle is disabled and force is always displayed. When the arm is lowered the F(g)/Z(μm) toggle is enabled and either mode may be selected.
Tool Index LED	Thickness measurements are made when the TOOL INDEX LED is illuminated. Turn the knurled knob of the Tool Drive Shaft slowly until the LED comes on. The LED flashes once each tool rotation as the F(g)/Z(μm) Display is updated by the tool phase sensor.
F(g) LED	When the F(g) LED is on, force is displayed as a negative number on the digital meter. Zero to 200gm. can be preset with the FORCE SETPOINT potentiometer.
Z(μm) LED	When the Z(μm) LED is on, Z position is displayed.
X10	When the X10 LED is on Z value is 10 times the meter display.
Arm Up-Dn	The ARM-UP/ARM-DN buttons raise and lower the fulcrum arm. ARM-DN also enables the F(g)/Z(μm) toggle allowing the mode of choice to be selected once the tool comes within range of the contactless Z detector.
Force Setpoint	This 10-turn potentiometer provides continuously adjustable calibrated tool force from 0 to 200gm. in 1gm. increments. Force is displayed as a negative number.
Z Offset	The Z OFFSET is a 20-turn potentiometer used to make initial specimen thickness measurements and set the amount of material to be removed in each dimpling step.
Balance	BALANCE is a one turn potentiometer used to make precise adjustments when different tools are used. It is usually not necessary to adjust the BALANCE potentiometer once it has been set.
Damping	The one turn DAMPING potentiometer provides continuous adjustment of tool damping from zero to stiff. Damping of the arm is accomplished by using position information from the Z sensor, processing it, and feeding it back to the arm motor which controls the up/down force of the arm. This circuit actively dampens any vibration frequencies that the arm may experience. Since the damping force is magnetically coupled to the arm, there is no mechanical play in the system providing damping response on the sub-micrometer level.

It is important to note that in addition to the aforementioned functions of damping are three other significant features:

1. Allow tools to carefully come into contact with the specimen when the arm is lowered.
2. Reduces shock that can crack thin specimens.
3. Keeps tools round, thus improving Z position measurements.

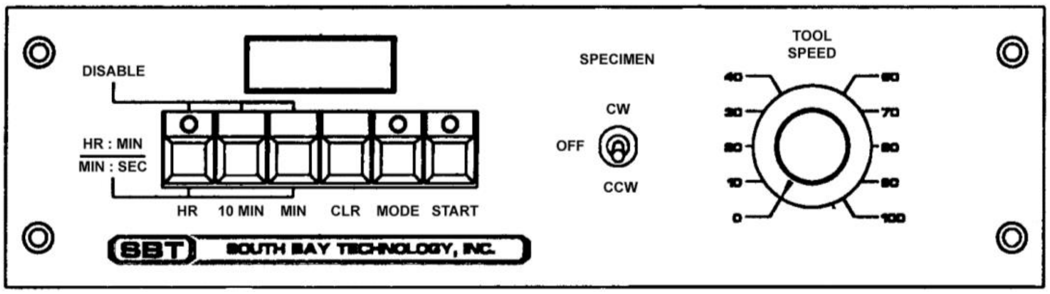
Figure 2. Illustration of the upper front panel



Lower front panel/timer

Tool speed	A one turn potentiometer provides continuously adjustable tool speed from 100 to 600 RPM. Each division indicates a change of tool speed of approximately 50 RPM.
Specimen rotation toggle	A three-position toggle permits either CW or CCW rotation of the specimen. The OFF position halts rotation.
Timer display	The Timer Display can be set for hours and minutes or minutes and seconds. By pressing the HR and MIN push buttons simultaneously, the display will alternate from hours and minutes to minutes and seconds. Depressing the **START** button will start and stop the timer, supplies power to the tool drive shaft, and provides power to the platen drive shaft. Depressing **CLR** resets the Timer to zero.
The timer operates in three different modes:	
(1) Elapsed time mode	The Elapsed Time Mode is used to time a dimpling step and is most frequently used. This mode counts up during the dimpling process until the process is terminated.
(2) Preset time mode	The Preset Time Mode is used when doing repetitive steps where the amount of time is known to perform a particular step. The dimpling process will terminate after a preset time or when the tool reaches the Z offset setting, whichever occurs first. This can be used to calculate removal rates of materials and to monitor slurry life.
(3) Disable mode	The Disable Mode will disable automatic termination when activated, and is not usually used.

Figure 3. Illustration of the lower front panel/timer.



Rear panel

The rear panel consists of the following parts:

- ON/OFF main power switch
- FUSE holder for 120 VAC, 2A fuse; for 220VAC, 1A fuse
- 110 VAC socket for the mains power cord

X-Y Specimen Stage

The X-Y Specimen Stage is designed to position the center of rotation of the Specimen Platen directly under the tool. The precision threaded screws permit orthogonal adjustment and have already been preset. Reset the screws only if you notice that donuts rather than dimples are formed when using Tool 3i or 4i. Readjustment will also be required if the Platen Tower is removed to replace the specimen bulb. When it becomes necessary to make this readjustment, the alignment procedure is described in section “Maintenance”.

The X-Y Specimen Stage also allows adjustments in X or Y to dice bulk specimens with a thin diamond cut off blade for cross-sectional specimen preparation.

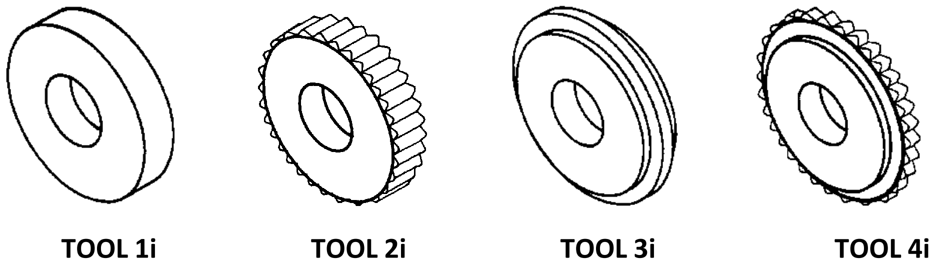
Dimpling tools

These tools are different dimpling tools in that they are specially designed for various stages of specimen preparation. The various dimpling tools are described below.

Tool 1i, flattening	Tool 1i is a flatting tool just wider than a 3mm specimen disk so it will thin a properly centered specimen disk uniformly. Because of eventual wear this tool will lose its flat surface and should be replaced. Using a non-flat Tool 1i will lead to non-planarities on the specimen surface. This can create a concave specimen that will cause errors in thickness measurements and weakness or rocking of the disk when it is flipped over for double sided dimpling.

Tool 2i, flat polishing	Tool 2i is a padded tool used to final polish the side of the specimen that will not be dimpled. The pad mounted on the tool at the factory is normally either Chemotex® (Tool 2iT) or Rayon-Fine® (Tool 2iM). The Chemotex® pad is white and a bit firmer. The Rayon-Fine® pad is beige and a bit softer and spongier. Before using the padded tools, and between steps, soak the tools in extender fluid. Thorough cleaning is mandatory and should be done thoroughly. The pad and bonding will eventually break down and the tool should be replaced at that time.
Tool 3i, dimpling	Tool 3i is a dimpling tool generally used after flatting the specimen with Tool 1i. The cross-sectional profile of the dimpling tool determines the shape of the dimple produced on the specimen. Tool 3i will frequently produce 380µm diameter dimpled areas. It should be replaced when abnormalities are visible on the dimple surface.
Tool 4i, dimple polishing	Tool 4i is a padded tool used to increase the dimple depth, polish and remove working damage from the specimen surface. Tool 4i will allow the operator to approach electron transparency with the least amount of damage and greatest sensitivity. The pad mounted on the tool at the factory is normally either Chemotex® (Tool 4iT) or Rayon-Fine® (Tool 4iM). The Chemotex® pad is white and slightly firmer. The Rayon-Fine® pad is beige and a little softer and spongier. Before using the padded tools, and between steps, soak the tools in extender fluid. Thorough cleaning after use is mandatory. The pad and bonding will eventually break down and the Tool should be replaced.

Figure 4. Schematic illustration of the tool set used on the Dimpler.



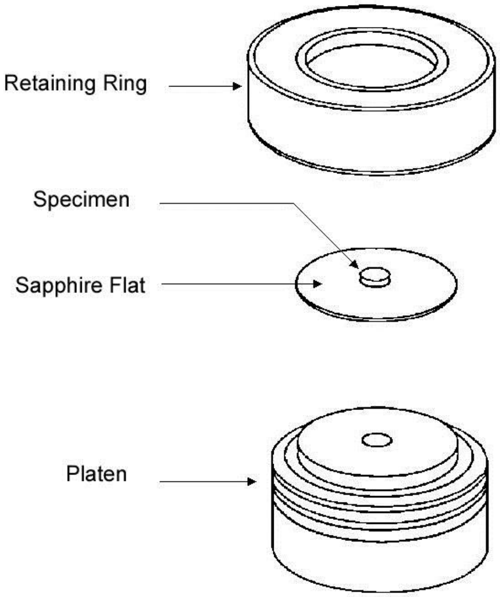
1. All tools are designed to perfectly fit the tool drive shaft, avoiding any errors in measurement.
2. Tools 2i and 4i are pre-padded and have a TRI approaching Tools 1i and 3i. This design increases the ending termination accuracy, free of major surface deformities.

Specimen platen assemblies

The specimen platen is used for holding the specimen during the dimpling process. There are many types of specimen platens available for use with the Dimpler. Their descriptions and functions are listed below:

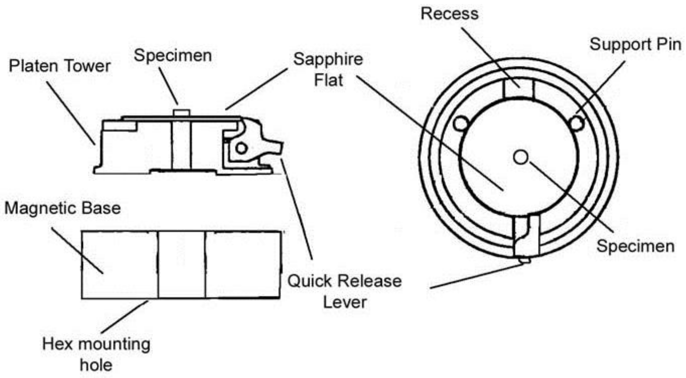
Standard specimen platen assembly	The Dimpler comes equipped with a standard specimen platen assembly consisting of a specimen platen, retainer ring, and a sapphire flat. The specimen platen is threaded for the retainer ring and machined with a hex mounting hole so it fits snugly on the platen drive shaft. The platen drive shaft rotates the platen either CW or CCW depending upon the desired rotation set by the user. The retainer ring holds the sapphire flat in place and acts as a reservoir for the lapping medium. It has an O-ring which allows the sapphire flat to be seated properly in the platen. The sapphire flat serves as the mounting surface for the specimen. It is optically flat for ease of specimen removal, clear to allow inspection of the specimen in transmitted light, and hard in order to minimize damage from over dimpling. If one surface is accidentally dimpled it can be flipped over providing another surface. See Figure 5.

Quick release magnetic eccentric platen assembly	An optional magnetic eccentric platen is available for locating and dimpling off-center dimple sites. The MEP assembly consists of the platen tower, with a quick release mechanism holding the sapphire flat; the sapphire flat, the magnetic base, and the centering stand. The platen allows for the rapid adjustment of the specimen for centering the area of interest during dimpling. The centering stand, because of its standard 1" x 3" laboratory slide footprint, can be placed on an existing optical microscope X-Y table to allow positioning the desired dimple site under the microscope cross hairs. It has a hex shaped shaft extending from the slide to allow for the mounting of the platens onto the stand. Two different magnetic base sizes are used depending upon the tools being implemented. The procedure for using the platen is described below. 1. Mount the specimen onto the sapphire flat. 2. Place the magnetic base onto the centering stand. 3. Place the platen tower onto the magnetic base and roughly center by eye. 4. Pull back on the quick release lever and slide the sapphire flat onto the platen tower. Make sure that the sapphire flat slides up against the support pins. 5. Allow the quick release to clamp the sapphire flat into place. 6. Center the entire assembly under the microscope. See Figure 6.
Quick release platen	The quick release platen is almost identical to the magnetic eccentric platen. The quick release platen is fixed and therefore does not allow any adjustment of the specimen for off-center dimpling procedures. The quick release platen consists of a platen tower, with a quick release mechanism holding the sapphire flat; the sapphire flat, and the base. The platen allows for the rapid removal of the sapphire flat for inspection during the dimpling process. Two different base sizes are used depending upon the tools being implemented. The procedure for using the platen is described below. 1. Mount the specimen onto the sapphire flat. 2. Pull back on the quick release lever and slide the sapphire flat onto the platen tower. Make sure that the sapphire flat slides up against the support pins. 3. Allow the quick release to clamp the sapphire flat into place. 4. Place onto the Model D500i. See Figure 7.



← Figure 5. Schematic illustration of the standard specimen platen. First, the specimen is mounted to the sapphire flat, which is then placed on the platen and secured by the retaining ring.

Figure 6. Schematic illustration of the quick release magnetic eccentric platen. The platen is made up by a magnetic base, platen tower, and sapphire flat. →



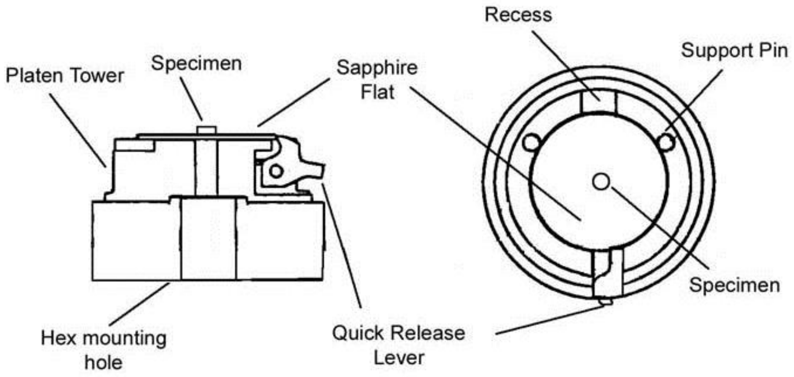


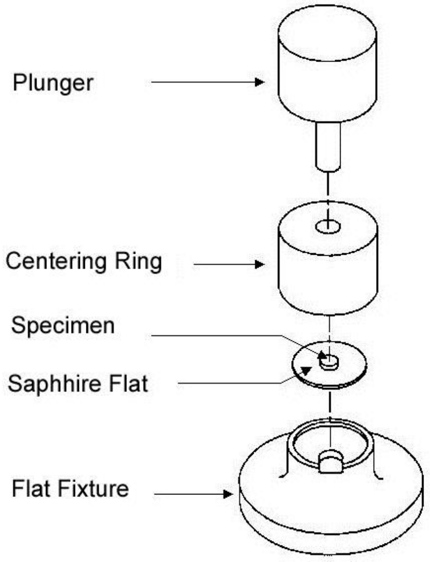
Figure 7. Schematic illustration of the quick release platen. The platen is made up of the hex mounting base, the platen tower, and the sapphire flat.

The specimen mounting jig assembly comes with a flat fixture, centering ring, and plunger. The assembly confirms that specimens are mounted evenly on the sapphire flat.

The flat fixture is kept on a hot plate so specimens can be mounted almost immediately after applying wax to the sapphire flat. It has a centrally located 3mm. circle for specimen alignment and a tweezers recess for removing the sapphire flat.

The centering ring locates the plunger and keeps it in a vertical placement. The plunger is inserted into the centering ring with gentle pressure and a slight twist pressing the specimen against the sapphire flat. This insures that the specimen remains coplanar and forms a firm bond.

Figure 8. Schematic illustration of the specimen mounting jig assembly.



Description	**Qty.**
Specimen platen assembly	1
Specimen mounting jig	1
Circular cover slips	2
Mounting wax, quickstick 135	1
Diamond suspension, 1µm, 1oz. bottle	1
Diamond suspension, 3µm, 1oz. bottle	1
Colloidal silica, syton®, 0.05µm, 4oz. bottle	1
Diamond paste extender and lubricant, 4oz. bottle	1
Spare fuse (2 a for 110 vac; 1 a for 220 vac)	1

Accessories

There are a variety of accessories that can be used for the Dimpler model D500i. See below.

Model D500i accessory package

Description	**Qty.**
1i flatting tool	1
2im flat polishing tool, pre-padded w/rayon-fine® cloth	1
2it flat polishing tool, pre-padded w/chemotex® cloth	1
3i dimpling tool	1
4im dimple polishing tool, pre-padded w/rayon-fine® cloth	1
4it dimple polishing tool, pre-padded w/chemotex® fine cloth	1

Diamond tools. 1iD and 3iD	These tools can be used instead of standard tools 1i and 3i. Due to the diamond-embedded tool surface, slurry is unnecessary. A liquid lubricant is used to carry away the specimen material as it is removed.
Large area dimpling tools	These tools are larger diameter tools (0.615" diameter and 1" diameter) that are used for producing a larger dimpled area on specimens. They are used in the same manner as the standard tools used for processing.
Quick release magnetic eccentric platen assembly	An optional magnetic eccentric platen is provided for locating and dimpling off-center dimple sites. The MEP assembly is made of the platen tower, a quick release mechanism holding the sapphire flat, the sapphire flat, the magnetic base, and the centering stand. The platen allows the rapid adjustment of the specimen for centering the desired area during the dimpling process. Due to its standard 1" x 3" laboratory slide footprint, the centering stand can be placed on an existing optical microscope X-Y table to facilitate the positioning of the desired dimple site under the microscope cross hairs. It has a hex shaped shaft extending from the slide to allow for the mounting of the platens onto the stand. There are two different magnetic base sizes that can be used depending upon the tools being implemented. A dedicated alignment microscope is also available if a laboratory microscope is not provided for this purpose.
Precision coring tools	Electron Microscopy Sciences offers two types of coring instruments: Model 360 Abrasive Slurry Disc Cutter and Model 380 Ultrasonic Disc Cutter. The Model 360 is provided with a soft metal coring drill or an optional diamond coring drill. This produces 3mm. disks (optionally 2.3mm. disks) for essentially any material without chipping, breakage, or any other damage of any kind. The drill table holds a moveable plate and slurry reservoir. The cutting tool speed can be adjusted from 0-600 RPM and has an electronic shutoff to prevent overly damaging the unit. The Model 380 is an ultrasonic cutting tool used for coring out various sized specimens. The instrument cuts extremely fast, especially for brittle, hard materials such as Si and sapphire. Different shapes can be produced depending on the shape of the tool.
Hand lapping and polishing fixture	The Model 150 Lapping and Polishing Fixture is made to lap and polish specimens up to 1.25" in diameter. Its general use is for thinning materials to less than 50 microns in thickness, however, for TEM applications it can be used for polishing several 3mm. discs to less than 200 microns in thickness prior to dimpling.
Diamond dicing tool	The Diamond Dicing Tool is used to cut small pieces of bulk specimen material for cross sectional specimen preparation. It consists of a thin, circular, diamond impregnated blade, and two driving flanges. The blade is mounted on the Model D500i tool shaft between the driving flanges. 1. Adhere the specimen to be cut to a cover slip and then glue the cover slip to the surface of the platen. The glue should be a low melting point wax.

	NOTE: Be sure to clean the platen surface thoroughly before dimpling. Do not use the sapphire flat as the substrate. Cutting into the sapphire flat will cause damage. 2. Dispense a small quantity of extender fluid or water when dicing material. The fluid will keep the kerf clear of cut off particles and cool the blade. 3. Establish the thickness of the material to be diced by lowering the blade onto the cover slip. Set the zero point into the ZERO OFFSET. Measure the thickness of the material to be diced like measuring the specimen thickness 4. Begin with low force and moderate tool speed until familiarity is achieved with the cutting ability of the blade.
Specimen vise	The Specimen Vise is designed to reduce the epoxy layer thickness during the preparation of cross-sectional specimens. The vise is constructed of brass with interchangeable aluminum jaws. Optional Teflon™ jaws are available. Tightening a thumbscrew applies co-planar pressure onto the specimen. The vise can be placed on a hot plate for accelerated curing of the epoxy.

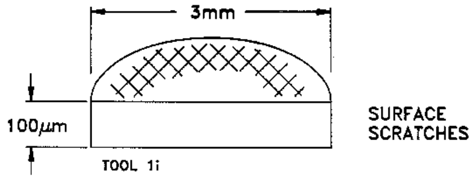
Instrument operation

Dimpling produces an ultra-thin region necessary for successful, artifact-free ion thinning while maintaining a robust peripheral thickness to prevent specimen damage. Pre-thinning with the Model D500i also minimizes specimen preparation time in an ion mill from 20-50 hours to as little as five minutes. Surface variations and artifacts produced by long milling times, chemical etching or electro-polishing, are practically eliminated. Local thickness variations produced by extended milling are reduced by at least an order of magnitude. The effects of differential milling rates often found in the preparation of cross-sectional specimens or multi-layer specimens are reduced. The ultimate thickness reached at the bottom of the dimple is dependent on the nature of the material being thinned, however, hard specimens and many cross-sectional specimens typically can be dimpled to <5μm with a 100μm thick periphery for specimen support.

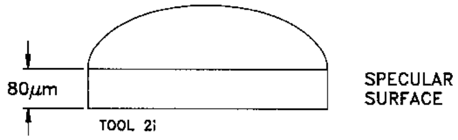
An example of the general protocol for specimen preparation can be found in Figure 9.

Figure 9. Schematic illustration of the 4 basic steps used for dimpling a TEM specimen.
Step 1: Make the specimen flat to approximately 100μm.
Step 2: Flat the specimen and polish to less than 80μm.
Step 3: Dimple the specimen to a center thickness of about 25μm, followed by a final dimpling step to create the final thickness.

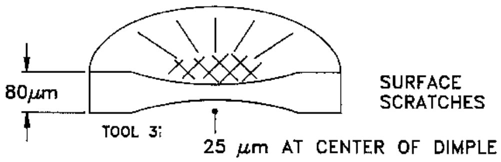
STEP 1



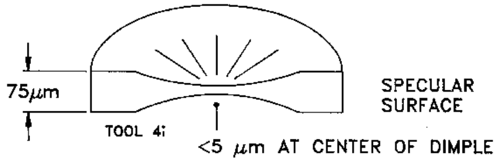
STEP 2



STEP 3



STEP 4



Initial setup

Before using the Dimpler, please follow the procedures below prior to preparing a specimen.

1. Follow the steps under the section “ Instrument layout: Unpacking”
2. Switch on the MAINS power switch at the rear of the instrument.

NOTE: Allow for the instrument to warm up for about 1 hour. The instrument requires this in order to reach thermal equilibrium before operating.

3. Balance the fulcrum arm as follows.

The fulcrum arm is pre-balanced and should only need minor adjustments. The electronic balance adjustment is sensitive enough to compensate for different tool weights, but making further adjustments is not recommended for normal use. Readjusting the balance is not necessary unless the unit has been moved.

a. Turn MAINS power switch at the rear of the instrument to the OFF position.

b. Mount the specimen platen assembly on the platen drive shaft and Tool 3i on the tool drive shaft.

c. With the power OFF the fulcrum arm should balance approximately level. If it does not, adjust the counter weight located at the rear of the fulcrum arm. Do this very carefully.

d. Turn MAINS power to ON position.

e. Set FORCE to zero and DAMPING to 3 or 4.

f. Toggle the F(g)/Z(μm) switch to Z(μm) and rotate the tool drive shaft with the knurled knob until the TOOL INDEX LED comes on.

g. Press ARM-DN push button. As the arm descends and the tool nears the specimen platen, the Z(μm) LED will come on and an arbitrary Z position will be displayed. Manually push the arm down so the tool is against the sapphire flat and adjust Z to 0 and release.

h. Slowly adjust the BALANCE potentiometer so the tool balances within ½mm. above the sapphire flat. When the F(g)/Z(μm) Display stabilizes and reads +500μm the adjustment is correct.

The contactless Z sensor electronics require 10-12 minutes to reach thermal equilibrium. During this time, do not make initial measurements. Drastic changes in ambient temperature will affect the accuracy of Z readings.

Dimpling guidelines

Using tools 1i and 3i

Tools 1i (flatting tool) and 3i (dimpling tool), are unpadded, will remove material from the specimen rapidly, and require similar operational procedures. With the use of a lapping plate, the abrasive particles become partially imbedded in the soft lap and are held in place to abrade the work piece. Note that with flatting Tool 1i and dimpling Tool 3i, the material removal forces when dimpling are small. The particles are carried along the moving tool surface against the specimen without the abrasive particles being deeply embedded into the tools.

When flatting or dimpling with tools 1i or 3i, the Z sensor is measuring the same point on the tool as on the specimen surface, exactly at the point where the tool touches the specimen. The abrasive particles do not affect the accuracy of the Z position. The ΔZ between the tool and specimen surface is zero. When the Z OFFSET setting reaches zero, the dimpling process stops exactly when the specimen has been reduced by this amount. Termination accuracy with Tools 1i and 3i is ±1μm. It is unnecessary to remove the specimen platen from the platen drive shaft to confirm dimple depth measurements when using Tools 1i and 3i.

If the Tool 3i OD is reduced during the dimpling process, the dimple depth will be less by this amount, however the termination error will be on the safe side. This may happen if the specimen material is softer than the tool. The abrasive becomes embedded in the specimen and as a result, becomes the lap. This effect is reduced, however, as any reduction in Tool 3i is normalized by the initial measurement made with this tool at the outset of dimpling process.

The active damping force and constant dimpling force applied will allow specimens to be reduced by large amounts in a single step without introducing specimen damage. Minimizing the number of dimpling steps actually results in a more accurate final dimple depth measurement. Many different materials can be dimpled to less than 10μm in one step with Tools 1i or 3i. Silicon, for example, can be thinned to less than 10μm in just five minutes with Tool 1i.

NOTE: It is important to leave enough material for removal in the next step to polish away scratches from the previous step. (See Section "Abrasive Selection").

The diamond particles in the 1μm and 3μm slurries supplied in the Accessory Package are polycrystalline. Polycrystalline diamond has a spherical shape so the particles' average diameter is reduced evenly as the diamond breaks down. This characteristic of polycrystalline diamonds decreases surface scratches.

The specimen platen should be charged sufficiently with diamond slurry from the beginning to end of the step until the target dimple depth has been reached.

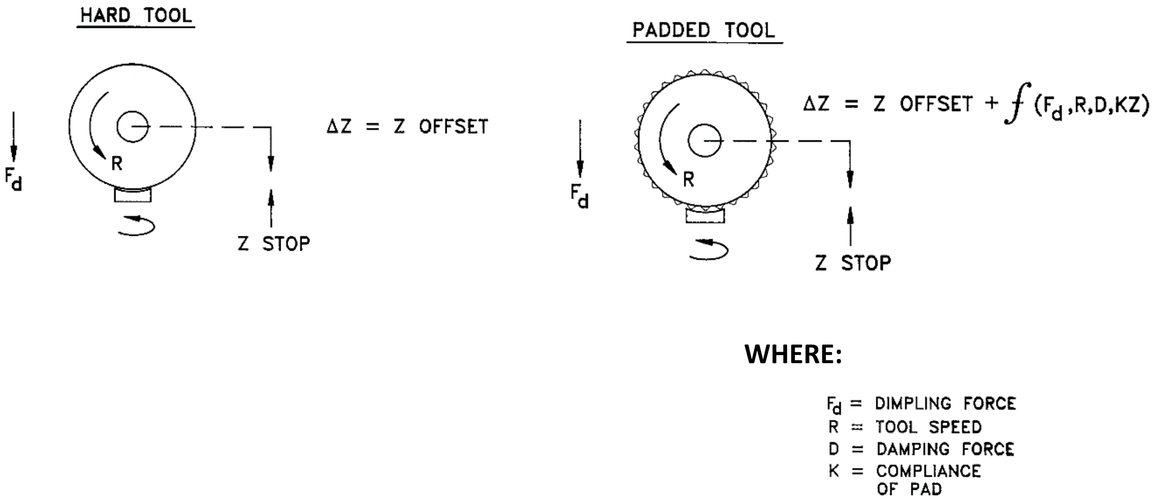
Using tools 2i and 4i

Tools 2i (flat polishing) and 4i (dimple polishing), are padded tools designed to remove material by polishing rather than by abrasion. The results of material removal using the padded Tools 2i and 4i have not been collected on a consistent quantitative basis, but the difference is that the abrasive particles are held and carried by the nap of an affixed pad. The particles become embedded in the nap and are thrusted against the specimen with abrasive force that is dependent upon pile resilience, particle size, dimpling force, and tool speed.

We recommend these tools be conditioned by pre-soaking in extender fluid for 10 minutes to soften the nap. Running the tool in extender fluid with the selected force and tool speed before charging the platen with the abrasive slurry will improve the set amount of material removal and termination repeatability.

Unlike Tools 1i and 3i, the Z location where the padded tool surface contacts the specimen is variable. The ΔZ between the specimen and the unpadded tool surface is virtually zero, while ΔZ between the padded tool surface and the specimen is a function of the Dimpling Force (F_d), Tool Speed (R), Damping Force (D), and Pad Compliance (K).

Figure 10. The relationship between the two tools. Schematic illustration of the Z parameters and how they differ from the other tool types. As shown, the hard tools show no variation in the Z offset setting, whereas with a padded tool, the Z offset is a function of four different parameters.

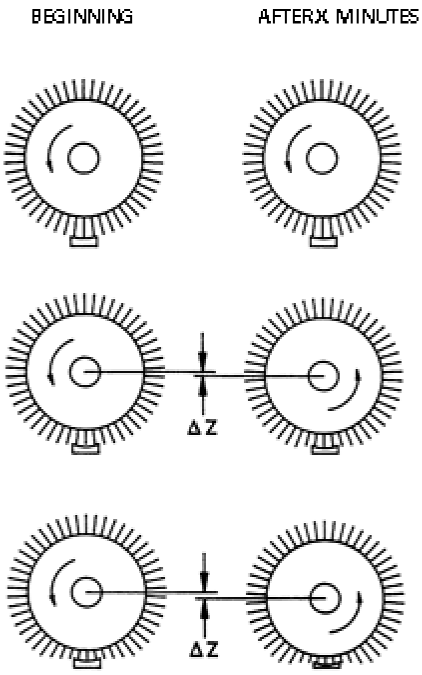


When sufficient dimpling force is applied, the pad will compress and material will be removed from the specimen. This is the working interface. The working interface of the padded tools is reached when the F(g)/Z(μm) Display stabilizes while the tool is rotating against the specimen. This becomes the reference location from which the specimen is dimpled further. Because the pad is compliant after the dimpling parameters are selected, they should not be changed.

Locate the working interface first, using the extender fluid rather than the abrasive slurry. This will condition the nap and simulate the viscosity, buoyancy, and surface tension of the slurries, but not abrade the specimen. Puddle a small amount of extender fluid in the platen reservoir and lower the tool onto the platen.

The display will stabilize to ≤ ±2μm. Set the Z OFFSET to the amount of material to be removed. Vary F_D, F_d, and R to get a sense of how changing the working interface location affects both the analog and digital Z(μm) Display. It is suggested to set F_d, Damping Force, to maximum.

After conditioning the tool, charge the Platen reservoir with the abrasive slurry by adding it directly to the extender fluid.



Force = 0 GM
Working interface is at the full extension of the cloth nap of the tool.
No material removal.

Force = 10 GM
Working interface is within the cloth and nap of the tool.
Moderate material removal.

Force = 20 GM
Working interface is well within the cloth nap of the tool.
Greater material removal.

As the working interface moves down into the dimple, the ΔZ is precisely measured and displayed. When the working interface removes material set into the Z OFFSET control, the dimpling process is terminated.

Figure 11. Schematic illustration of the working interface and its relationship with cloth nap, force, and ΔZ.

The table below of padded tool compression rates demonstrates that operating in the force range of 30-100gm. will yield the greatest repeatability.

FORCE (GM)	TOOL 2iM		TOOL 2iT		TOOL 4iM		TOOL 4iT	
	A	B	A	B	A	B	A	B
5-10	75	7	7	3	25	7	6	3
10-15	35	10	5	3	10	7	4	3
15-20	20	8	3	3	8	7	3	3
20-25	17	8	2	2	7	5	3	3
25-30	10	6	2	2	5	5	3	3
30-35	8	8	2	2	5	5	3	3
35-40	5	4	2	2	5	4	2	2
40-45	5	4	2	2	3	4	2	2
45-50	5	4	2	2	3	4	1	2
50-55	4	4	2	2	2	4	1	2
55-100	3	3	1	1	1	3	1	1

Compression rates of selected padded tools
Settings: Tool speed = 40 / Damping force = MAX
A = Compression (µm) / B = Setting after two minutes (µm)

Single sided dimpling for ion milling

If a specimen initially has a specular finish on one side, Steps 1 and 2 can be skipped.

1. Tool 1i: Flat Lap until the specimen is 110 to 135µm thick. Make sure lapping scratches cover the entire specimen surface.
2. Tool 2i: Flat Polish until a specular surface is obtained. Remove about 10µm.
3. Remove the specimen, flip it over, then measure the initial thickness. Remount specimen.
4. Tool 3i: Dimple to 10µm.
5. Tool 4i: Dimple Polish to < 5µm.
6. Ion Mill from both sides until perforation.

Double sided dimpling for ion milling

With double sided dimpling, the goal is to reach a specimen thickness of 150µm at the periphery, and a dimple depth from both sides of 75µm. This geometry ensures mechanical strength of the specimen.

Although the example (150 - 75 – 75µm = 0) suggests a perfect dimpled specimen the amount of material left between dimples depends on the characteristics of the specimen and operator proficiency.

1. Tool 1i: Flat Lap until lapping scratches cover the entire specimen surface.
2. Tool 3i: Dimple until 45µm is removed.
3. Tool 4i: Dimple Polish until 30µm is removed. The dimple depth is now 75µm from the periphery of the specimen.
4. Remove, flip over and remount.

NOTE: Extreme caution should be taken to remount in exactly the same position.

5. Tool 1i: Flat Lap until the specimen thickness at the periphery is 150µm.
6. Tool 3i: Dimple until 45µm is removed.
7. Tool 4i: Dimple Polish until 30µm is removed.
8. Ion Mill from both sides until perforation.

Single sided dimpling for chemical/electrochemical polishing

1. Tool 1i: Flat Lap to 200-250μm.
2. Tool 3i: Dimple to 75-100μm.
3. Tool 4i: Dimple Polish to 60-85μm.
4. After removing the specimen from the sapphire flat, mask the surface area to be preserved with black wax or lacquer, then chemically or electro-chemically polish the dimpled side until perforation.

Initial specimen preparation

Before engaging in the dimpling process, the specimen must be prepared in such a way that allows for efficient dimpling procedures. This generally consists of cutting the specimen from a bulk form into a disc, pre-thinning the specimen material to a given thickness, and polishing to provide a smooth, uniform surface to begin dimpling.

Miscellaneous supplies

The preparation of specimens, both dimpling and other methods, requires some basic laboratory supplies. See the list below:

1. Laboratory hot plate
2. Tweezers
3. Solvents: acetone, methanol
4. Deionized water
5. Cotton swabs and decontaminated soap or mild dish detergent
6. Pressurized dry nitrogen gas or air
7. Optical microscope with cross hair reticle
8. Coring device (Model 360 or Model 380)
9. Hand lap (Model 150)

Disc cutting

Before dimpling, specimens are usually formed into 3mm. diameter disks to fit the TEM holder. Older TEMs may use a 2.3mm. diameter disk format. Disc cutting should be performed with caution to provide a specimen disk with a periphery that is smooth and not oversized. The Model 360 Abrasive Slurry Disc Cutter, or Model 380 Ultrasonic Disc Cutter, available from Electron Microscopy Sciences, are designed especially for this purpose. It is a rugged yet precise coring instrument appropriate for all kinds of specimen materials. (See Section "Precision Coring Tools").

Pre-thinning

Specimens can be pre-thinned with Tools 1i and 2i, or with a hand lap. We suggest that specimens not be thinner than 100μm before dimpling. A gravity feed hand lap is available from Electron Microscopy Sciences and are especially designed to hold specimens mounted on the sapphire flat. Multiple specimens of 3mm. diameter can be lapped simultaneously using the Model 150 Lapping Fixture, allowing several specimens to be ready for dimpling. (See Section "Hand Lapping and Polishing Fixture").

Standard operating procedures (Single sided dimpling)

Consider the following information below describing the basic steps required for producing a TEM specimen, using a single sided dimpling method. Check for:

1. Instrument has been carefully removed from the packing material.
2. Instrument power cord has been installed.
3. Wooden support blocks have been removed from the arm.
4. Rubber stop at rear of fulcrum arm has been lowered.
5. Instrument has been switched on for at least one hour.
6. Fulcrum arm has been balanced.

Initial Z measurement

1. The initial thickness measurement is made while the tool and specimen are stationary. Mount Tool 3i and rotate the tool drive shaft with the knurled knob until the TOOL INDEX LED is on.

NOTE: The TOOL INDEX LED must be on to make static Z measurements.

2. Place specimen platen assembly on the hex shaft. Be sure the sapphire flat is clean and mounted squarely under the retainer ring.

3. Select a dimpling FORCE high enough to press the specimen firmly against the sapphire flat but not so high to compress a specimen with a concave back surface. This typically is about 20gm.

4. Toggle to Z(μm).

5. Press ARM-DN to lower the tool onto the platen. The Z(μm) LED will come on and an arbitrary number will be displayed.

6. Zero the F(g)/Z(μm) Display with Z OFFSET control.

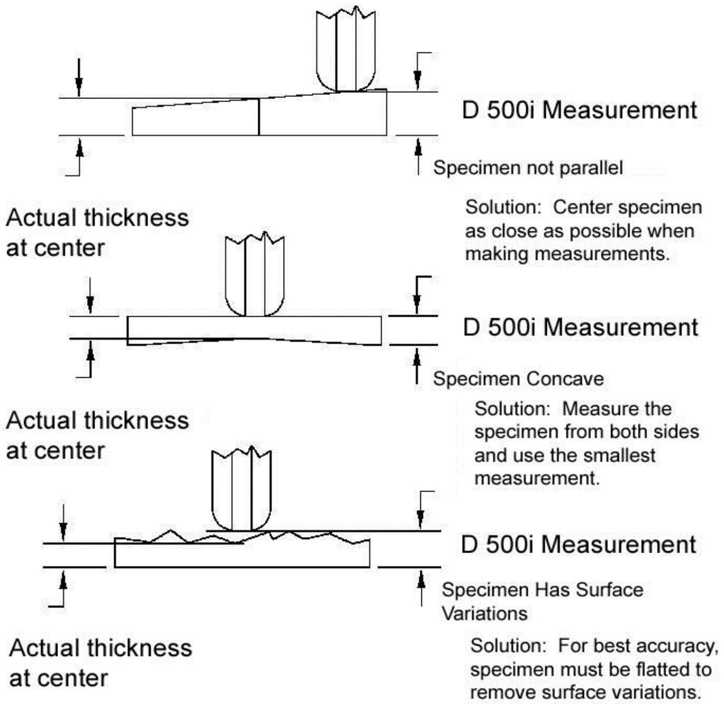
7. Press ARM-UP.

8. Place a clean specimen on the sapphire flat directly over the 3mm. hole in the platen.

9. Press ARM-DN to lower the tool onto the unmounted specimen surface. The Z(μm) LED will come on and specimen thickness will be displayed in micrometers. This is the initial specimen thickness and should be logged on the Specimen Log Sheet. If you suspect that the specimen is bowed, measure it from both sides with a force large enough to flatten out the specimen.

NOTE: Since specimen thickness measurements are made by a single point on the periphery of Tool 3i, more than one measurement is recommended. If specimens have irregular surfaces and the average thickness is not determined, termination errors will occur. See Figure 12.

Figure 12. Possible causes of measurement errors when measuring specimen thickness with Model D500i. Errors are generally a result of the specimen not being parallel, the specimen having a concave shape, or the specimen having a rough surface.



Solution: Center specimen as close as possible when making measurements.

Solution: Measure the specimen from both sides and use the smallest measurement.

Solution: For best accuracy, specimen must be flatted to remove surface variations.

Wax thickness measurement

All measurements are taken from the top surface of the specimen, so it is not necessarily required to know the exact wax thickness. If you wish to gauge proficiency in minimizing the wax thickness, another measurement can be made after the specimen has been mounted. Subtract the initial thickness from this measurement to get the wax thickness.

Specimen mounting

Many different mounting adhesives can be used to adhere a specimen to the sapphire flat such as beeswax, quick setting glue, or mounting wax. The following directions and examples describe the mounting technique using acetone soluble thermoplastic mounting wax.

Specimens are mounted on optically flat sapphire flats. The low thermal mass of the flat minimizes mounting and dismounting time and the smooth surface allows the delicate dimpled specimen to be gently slipped off. Sapphire hardness limits will become damaged if specimens are accidentally dimpled due to operator error. The retainer ring that secures the sapphire flat also serves as a slurry reservoir. The new quick release platens do not have a retaining ring, however slurry will not drip all over the platen tower.

The magnetic eccentric platen is used for dimpling off-center dimple sites. It can be adjusted under a laboratory microscope or a special alignment microscope available from Electron Microscopy Sciences.

1. Warm the flat fixture on a hot plate to 135°C; do not overheat. Place a sapphire flat on the flat fixture.

2. With tweezers place a very small flake of wax within the 3mm locating circle of the flat fixture. Adjust the temperature so the wax will melt but not bubble. Bubbles in the wax decrease specimen support, especially in double sided dimpling. Avoid excess wax and try to make the wax layer about 5μm.

3. With tweezers place specimen directly over the 3mm. circle when the wax goes clear.

4. Place the centering ring of the specimen mounting jig assembly on the flat fixture and insert the plunger. Apply gentle downward pressure to the plunger using a rotational motion to ensure a uniform wax layer and co-planarity of the specimen with the platen. Avoid pushing too hard and forcing the disk out of the circle.

5. Using tweezers remove the sapphire flat from the flat fixture keeping it level and place on a cold surface. The wax will harden in less than a minute.

6. If mounting wax gets on top of or around the specimen, remove with acetone by gently rubbing with a cotton swab. Follow by swabbing with mild detergent, rinse with deionized water and methanol, and blow dry with air or nitrogen.

7. Now place the flat with the specimen on the specimen platen and secure with the retainer ring. Observe that when retainer ring is tightened the specimen is over 3mm. hole in the platen. If it is not, repeat specimen mounting.

Setting parameters

Dimpling force set point and tool speed

The optimum dimpling force and tool speed will vary for different materials. Increasing the dimpling FORCE and TOOL SPEED results in faster material removal, but also increases the risk of specimen damage. Until familiarity is gained with different specimen materials, a moderate dimpling FORCE of 20-50gm. and a TOOL SPEED of 30-50 are recommended.

Damping force

Selecting the optimum DAMPING force also depends on the type of specimen material. The effect of changing the DAMPING force from fully counterclockwise (virtually no damping), to fully counterclockwise (maximum damping), can be monitored on the F(g)/Z(μm) Display.

Damping keeps the tool surface from impacting the specimen as the tool rotates on the specimen due to any eccentricities of the tool or high spots that develop on the specimen as it is dimpled. Higher damping will reduce tool excursions and help minimize the chances of specimen cracking.

When using Tools 1i and 3i, moderate to maximum damping is suggested. With the padded Tools 2i and 4i, maximum damping will stabilize the digital Z data and improve Z termination accuracy while dimpling at the working interface of padded tools.

Specimen rotation

The clockwise or counterclockwise rotation will not affect the dimpling process. The OFF position is only used when adjusting the X-Y parameters and for special applications such as grooving a specimen or when using the dicing tool.

Timer

If the Timer is not used in the Preset Time Mode, it is suggested that the elapsed time be noted after each dimpling step. Knowing the time to perform a dimpling step will aid in optimizing the tool speed and dimpling force settings.

Elapsed Time Mode

1. Press CLR to zero the timer display.
2. When the amber light is on, the timer is in the preset time mode. Press MODE to enter elapsed time mode.
3. Press START to begin monitoring elapsed time. The green LED will come on and the colon on the Timer Display will flash. Power is now supplied to the tool drive shaft and the platen drive shaft.
4. Press START again to halt the timer, tool, and platen motors. The green LED goes off.

Preset Time Mode

1. Press CLR to zero timer display.
2. Press MODE to select the Preset Time Mode and the amber LED will come on.
3. Enter the desired amount of time. Pressing HR and MIN simultaneously will alternate the display from hours and minutes, to minutes and seconds. If just seconds are to be preset it is necessary to go back to the Elapsed Time Mode and press START until the desired number of seconds is displayed, then press START again. Now return to the Preset Time Mode and continue.
4. Press START to begin preset timing.
5. To stop the process manually, press START again.

Disable Mode

1. To disable the termination functions press three push buttons simultaneously: HR, 10 MIN and MIN. The red LED will come on when in the Disable Mode.
2. To re-enable the termination function, again press the three push buttons simultaneously.

Z offset

The amount of material to be removed is set with the Z OFFSET control as a positive number. Always set the maximum amount of material to be removed in one step to increase termination accuracy. For example, when using Tool 3i and starting at an initial thickness of 100μm with a target thickness of 10μm, set Z OFFSET to 90μm.

NOTE: Dimpling parameters should only be changed between steps and not once a process has begun. Changing parameters when using soft tools severely affects the compliance of the pad and causes Z measurement discrepancies.

Specimen processing

In the example below, a 3mm. silicon disk 200 µm thick is dimpled to 5 µm.

Step 1: Remove 90µm from Side 1 of a 200µm Thick Specimen with Flatting Tool 1i

1. Mount Tool 1i on tool drive shaft and secure with the tool retaining cap.
2. Place specimen platen assembly with mounted specimen on platen drive shaft.
3. Press ARM-DN and toggle to Z(µm). The Z(µm) LED will come on and indicate a random Z Offset.
4. When the tool touches the specimen set Z OFFSET to 90µm.
5. Press ARM-UP. The F(g) LED will come on.
6. Charge specimen with 3µm diamond slurry by puddling the slurry in platen reservoir. Use an ample supply of slurry from the beginning.
7. Press CLR to zero the timer. Operate the timer in the Elapsed Time Mode to monitor the time necessary to perform this step.
8. Press START to begin timing, as well as tool and platen rotation.
9. Press ARM-DN. The Z(µm) LED will come on and the tool will contact the specimen surface to begin the flatting process.
10. If during the run the material removal slows, recharge the reservoir by carefully dropping slurry on top of rotating Tool surface.
11. As the 90µm is removed the Z (µm) Display decrements to zero. When 65µm is displayed, 2 µm has been removed and dimple depth is 25µm. When 0µm is displayed, 90µm has been removed and the process is terminated; the three-beep alarm sounds, the timer stops, the arm lifts the tool off the specimen, and the platen ceases to rotate.

    200µm – 9 µm = 110µm

12. Document all parameters on Specimen Log Sheet.
13. Clean specimen for examination using DI water and Kimwipes.
14. Remove and clean tool.

Step 2: Remove 10µm from Side 1 with Flat Polishing Tool 2i

The specimen is now 110μm thick, with 10μm to be removed by flat polishing for a resulting thickness of 100μm. If any dimpling parameters are to be changed, make adjustments now.

1. Mount Tool 2i on the tool drive shaft after it has soaked in extender fluid for 10 minutes.

2. If the specimen platen assembly was removed, remount on the hex drive shaft. Insure that the specimen platen is replaced on hex drive shaft in the same circular position (orientation) as it was removed.

3. Confirm the F(g)/Z(μm) toggle is still in the Z(μm) position.

4. Puddle a small amount extender fluid in platen reservoir. It will simulate the abrasive slurry but will not remove material while the working surface Z position is located.

5. Turn the Z OFFSET control a full turn and then some. This will prevent false termination as the pad is compressed when it contacts the specimen.

6. Press START on the timer to start tool and platen rotation.

7. Press ARM-DN. The Z(μm) Display will come on and tool will contact specimen. The Z(μm) Display should settle with a deviation ±2μm. If the padded tool goes through zero as the arm is lowered, the Tool and Platen will stop rotating, and the arm will automatically raise. If this happens turn the Z OFFSET control another turn and try again until the arm settles without triggering termination.

8. Set the Z OFFSET control to 10μm, the amount of material to be removed in this step. This should be done carefully to avoid triggering termination. Add the slurry to the reservoir by dropping it onto the top of the tool.

9. As the material is removed, the Z(μm) Display will decrement to zero. When the 10μm has been removed, the process will terminate; 0μm will be displayed and the arm will raise.

   $110\mu m - 10\mu m = 100\mu m$

10. Document all parameters on Specimen Log Sheet.

11. Clean specimen for examination.

12. Clean padded Tool by rinsing several times in deionized water. Blow dry. If the Tool will be used subsequently, let it soak in extender fluid.

Step 3: Remove 90μm from Side 2 with Dimpling Tool 3i

Since this an example of single side dimpling, it is necessary to remove, clean, remeasure, and remount the specimen. The flatted specimen should be remeasured on the D500i with Tool 3i.

1. Mount Tool 3i on the tool drive shaft. Measure the thickness from both sides. Enter the smaller of the two measurements on the Specimen Log Sheet and use this number as the

new initial thickness. Remount the specimen on the sapphire flat with side one down.

2. Mount the specimen platen assembly with mounted specimen on the hex drive shaft.
3. Press ARM-DN and toggle to Z(μm). The LED will come on and indicate a random Z OFFSET reading.
4. When the Tool touches the specimen, set the Z OFFSET control to 90μm.
5. Press ARM-UP. The F(g) LED will come on.
6. Charge specimen with 1μm diamond slurry by puddling the slurry in the platen reservoir. Use an ample supply of slurry from the beginning.
7. Press CLR to zero the timer. Operate the timer in the Elapsed Time Mode to monitor the time necessary to perform this step.
8. Press START to begin tool and platen rotation.
9. Press ARM-DN. The Z(μm) LED will come on and the tool will contact the specimen surface to begin the dimpling process.
10. Document all parameters on Specimen Log Sheet.
11. As the 90μm is removed, the Z(μm) Display decrements to zero. When 90μm is removed the process will terminate; 0μm will be displayed, and the arm will raise. The dimple depth is 90μm and the specimen thickness is now 10μm.

    100μm – 90μm = 10μm

12. Document all parameters on Specimen Log Sheet.
13. Clean specimen for examination.
14. Remove and clean tool for storage.

Step 4: Remove 5μm from Side 2 with Dimple Polishing Tool 4i

The specimen is now 10μm thick, with 5μm to be removed by Dimple Polishing. If any dimpling parameters are to be changed, make adjustments now. Syton® or Alumina is recommended for the final Dimple Polishing step. Syton® is frequently used when dimpling silicon because it chemically etches the silicon while abrading the specimen.

1. Mount Tool 4i on the tool drive shaft after it has soaked in extender fluid for 10 minutes.
2. Confirm that the F(g)/Z(μm) toggle is still in the Z(μm) position.

3. Puddle extender fluid in platen reservoir. The extender fluid simulates the abrasive slurry but will not remove material while the working surface Z position is located.

4. Turn the Z OFFSET control a full turn and then some. This will prevent false termination as the pad is compressed when it contacts the specimen.

5. Press START on the timer to start tool and platen rotation.

6. Press ARM-DN. The Z(μm) Display will come on and tool will contact the specimen. The Z(μm) Display should settle with a deviation ± 2μm. If the padded tool goes through zero as the arm is lowered, the tool and platen will stop rotating, and the arm will automatically raise. If this happens turn the Z OFFSET control another turn and try again until the arm settles without triggering termination.

7. Set the Z OFFSET control to 5μm, the amount of material to be removed in this step. This should be done carefully to avoid triggering termination. Add the slurry to the reservoir by dropping it onto the top of the tool.

8. As the material is removed, the Z(μm) Display will decrement to zero. When the 5μm is removed, the process will terminate; 0μm will be displayed and the fulcrum arm will raise.

   10μm – 5μm = 5μm

9. Document all parameters on Specimen Log Sheet.

10. Clean specimen for examination.

11. Clean padded Tool by rinsing several times in deionized water. Blow dry.

Specimen removal

METHOD A

1. Remove the specimen by placing the sapphire flat on the flat fixture. The wax will liquefy quickly and the specimen can be removed with tweezers.

2. With tweezers place the specimen in acetone to dissolve any excess wax.

3. Rinse the specimen with methanol and blow dry with dry air or nitrogen gas.

METHOD B

This method is recommended when the specimen is heat sensitive or too delicate to remove by Method A. Frequently Method B is suggested after the second side has been dimpled and polished.

1. Immerse the sapphire flat in acetone for about an hour, or until the specimen becomes unbonded and slides off the flat. Decant the acetone and repeat if necessary. This rinse should be done several times until the specimen slides off the flat easily. This avoids unnecessary handling which can crack the thin, fragile, dimpled center.

2. After emptying the acetone from the final rinse, and if the specimen characteristics allow, set the beaker with the specimen on a low temperature hot plate to evaporate any residual acetone from the specimen. Do not leave on the hot plate for an extended time. This could damage the specimen.

Applications guide

This section serves to give a basic guidelines for the use of the Model D500i for various applications. Also included in this section is a discussion on abrasives and their typical uses during processing. These sections are served only as a basic outline to the approach taken for these specific materials. Similar materials may be processed in a similar fashion using these same types of guidelines.

Gallium arsenide (Single or double sided)

1. Core a 3mm. specimen or cleave a square of GaAs and mount on the sapphire flat of the specimen platen assembly.
2. Tool 1i: Flat to 100μm with 3μm diamond slurry. Set the dimpling FORCE to 20gm.
3. Tool 3i: Dimple specimen to approximately 30μm (remove 70μm) with 1μm diamond slurry.
4. Tool 4i: Dimple Polish specimen with Syton® to 25μm (remove 5μm). Make sure that the specimen is submerged in the Syton® and that the TOOL SPEED is very slow. After approximately 15-20 minutes, the center of the specimen should be polished. There may be scratches on the thicker periphery of the specimen, but they will be out of the electron-transparent region and are not a concern. Very shallow scratches in the central area may be removed by chemical polishing and/or ion milling. Deep scratches in the central area, however, indicate that the polishing pad should be thoroughly cleaned or replaced and the specimen re-polished.

   Dimple depth can be verified with a quality optical microscope. In order to ensure mechanical strength of the specimen, the periphery of the disk should be 75μm thicker than its center, i.e., the dimple should be 75μm deep.

5. For double-dimpled specimens, mount the specimen with the dimpled side down. Be sure that the dimple cavity is filled with mounting wax and that no air bubbles are trapped under the thin area. Repeat the dimpling procedure Steps 1 through 4 above. Leave at least 25μm of material before ion milling or chemical thinning. Double-dimpling produces a robust specimen and reduces the amount of back-sputtered material deposited on the disk during ion milling.

6. If a smoother polish than can be obtained by ion milling is required, a bromine/methanol chemical polish can be used. Follow Steps 1-4, then transfer the disk to a glass microscope slide using mounting wax as before. Mix about 200ml. bromine/methanol solution. Always mix the solution under a chemical hood while stirring with a magnetic stirrer. Rubber gloves and protective glasses are required while handling the solution. The dissolution rate of GaAs is directly proportional to the bromine content, so a weak concentration is desirable for controlled polishing. Never use bromine concentrations greater than 10%. A 2-3% bromine solution is adequate if only a few specimens are being polished.

7. Place the solution in a clear beaker with a strong light source behind it. Insert the slide with mounted specimen into the beaker so that the light hits the specimen backside. The backside of the specimen is masked by the mounting wax and will not be attacked by the solution. A small pinhole should be produced in not more than 10 minutes for a 25μm thick specimen. The

dimpled area will appear a dense red just before perforation. It is best to remove the slide and specimen at this time, i.e., before you can see the transmission of white light through the pinhole.

8. Stop the polishing action by quenching the specimen in methanol. Next, place the slide in acetone and let the specimen float off as the wax is dissolved. Examine the specimen under an optical microscope with transmitted light. All the light red areas should be electron transparent. The polishing solution may be saved for future use, but it must be stored in a dark bottle since bromine evolves from the solution when it is subjected to light.

   Experience will dictate the mix of dimpling, chemical polishing, and ion milling to be used in any given situation. The electron transparent area created by dimpling is always exactly in the center of the specimen with no preferential polishing effects, whereas the bromine/methanol polish is uneven and does not work on specimens with certain crystallographic orientations, e.g., (111)B. These specimens must be dimpled before ion milling.

Tungsten carbide (Single side)

Initially a moderate TOOL SPEED of approximately 30, and an average dimpling FORCE of 10-20gm. is recommended. As experience with the technique accumulates, speed and force can be increased for greater specimen throughput.

When proficiency in dimpling is attained, tungsten carbide samples can be dimpled to approximately 4μm because of the strength of the material. The resulting reduction of milling time will produce smoother, more uniformly thin specimens.

1. Core a 3mm. disk of polycrystalline tungsten carbide and mount on the sapphire flat of the specimen platen assembly.
2. Tool 2i: Flat Polish using 15μm, 3μm, 1μm, and ½μm diamond slurry, then finish with Syton® to 100μm.
3. Remove, flip specimen over, and remount.
4. Tool 1i: Flat Lap to 75μm using 15μm, 3μm, and 1μm diamond slurry.
5. Tool 3i: Dimple to a thickness of 15μm with ½μm diamond slurry.
6. Tool 4i: Final Polish with Syton® to a thickness <10μm.
7. Ion mill specimen on both sides until a few small holes appear. Prolonged milling of polycrystalline specimens like tungsten carbide often result in a rough surface that shows as a matte finish in the central portion of the disk. This occurs because the milling rate depends on grain orientation, presence of a second phase, etc.

PB/SN solder

All dimpling steps are performed with Tool 4i. No other tools are used. Oil based diamond slurries are suggested. The diamond slurries supplied in the Accessory Package are water based.

1. Core a specimen and mount on the sapphire flat of the specimen platen assembly.
2. The suggested dimpling force is 50gm. at slow tool speed.
3. Tool 4i: With silicon carbide Dimple Polish to 100μm.
4. Tool 4i: With 15μm oil based diamond slurry Dimple Polish to 30μm. Remove 70μm.
5. Tool 4i: Clean all the 15μm diamond from the tool pad or use another tool. With 3μm oil based diamond slurry Dimple Polish to 15μm. Remove 15μm.

6. Tool 4i: Clean all the 3µm diamond from the tool pad or use another tool. Dimple Polish to 1-5µm with 0.3µm alumina mixed with water. Remove 14-10µm, or until small holes appear.
7. Final thin in an ion mill until perforation.

Abrasive selection

Dimpling is a modification of the metallographic techniques used for grinding and polishing materials and requires the use of abrasive media to prepare a specimen. Different abrasives yield different results and are described below.

Diamond slurries

The diamond slurry supplied by Electron Microscopy Sciences is specially formulated for the dimpling process. These slurries are H_2O soluble with polycrystalline diamond particles blended to the proper viscosity. The appropriate extender is supplied and can be used to thin the slurry if slower material removal rate is desired.

The viscosity of the slurry should be thick enough to puddle, but not flow. Pasty slurries with inhomogeneous viscosity should be avoided. Replenish the slurry on the specimen if it evaporates, or progress is significantly retarded. This slowing occurs when polycrystalline diamond breaks down and the average particle diameter decreases. The surface finish on the specimen, however, improves.

Diamond abrasive guide

Particle size (µm)	Applications
0.1	Provides the highest precision polish for critical surfaces.
0.25	Produces extremely high polish on soft metals and minerals. Used for final material removal of TEM specimens with thickness approximately 25µm or less.
0.5	Typical used for final polishing of TEM specimens.
1	Final polish for carbides, sapphire, ruby; material removal for TEM specimens with thickness approximately 25-30µm.
3	Good polish on carbides, hard minerals; good finish for thin sections; material removal for TEM specimens with thickness approximately 30-60µm.
6	Fast fine finishing for hard materials; material removal for TEM specimens with thickness approximately 60-100µm.
9	Controlled quick material removal; pre-finish on many materials; shape adjustment on hard materials; rapid material removal for TEM specimens with thickness >100µm.
15	Rapid removal of material and flatting of surfaces on thin sections; lap cutting; loose grain lapping.
30	Rapid material removal and shaping; pre-forming; lap cutting; loose grain lapping.

60	Fast stock removal and roughing on hardest materials.

Syton® HT50

Syton® HT50, a 400 Å slurry of SiO_2 particles, is acceptable for final polishing especially when dimpling silicon since it chemically etches silicon while it mechanically polishes. Syton® will at times become imbedded in samples and must be ion milled away. In the case that crystals form in the bottle, the contents should be filtered to prevent scratching and further damage. Keep container tightly capped and avoid freezing temperatures.

Alumina

Alumina slurry is available in grades from 500Å up to 3µm. It is a permanent suspension of Al_2O_3 in a neutral, non-toxic base. The base will not dry out on the Specimen Platen, is completely water soluble, and is non-irritating to the skin. Tool clean-up is easy and fast with Alumina.

Notes on the Use of Abrasives:

1. A metallographer's rule of thumb is that particle size of abrasive slurry will produce scratches and working damage to a depth of approximately 3 times the particle size.
2. Enough material should remain on the sample to be removed in the next step. So if one begins with a particle size of 3µm, approximately 9µm should be removed in the next step. If the next smaller particle size used is 1µm, then 9µm should be removed with the 1µm slurry.
3. Since 3 x 1µm = 3µm then this amount of material should be left to be removed in the subsequent step and so forth.
4. Slurries of different particle sizes and oil based diamond slurries are available from Electron Microscopy Sciences.

Maintenance

The Model D500i is a precision instrument and must be properly maintained to keep the instrument calibrated and working efficiently.

General Maintenance

1. Do not allow water to reach the electrical components.
2. Allow the instrument to remain on most of the time. This keeps the electronics in thermal equilibrium.
3. Do not allow abrasives to dry around the platen tower.
4. Do not allow the tools to dry onto the tool shaft. Always remove the dimpling tools when finished using them.

5. Do not drop the instrument onto a hard surface. This could damage the system.

6. Do not pull up on the arm or use it as a handle. This will cause the instrument to go out of calibration.

Dimpling tool maintenance

Cleaning Tools 1i and 3i

Clean Tools 1i and 3i in hot deionized water. Sonicate in acetone and then methanol. Blow dry.

Cleaning Tools 2i and 4i

Padded Tools 2i and 4i *must* be cleaned immediately after use. Syton® will crystallize on the pad making the tool unusable. Clean by rinsing several times in deionized water. Blow dry.

Re-padding Tools 2i and 4i

Re-padding Tools 2i and 4i in the laboratory is an option, and is described in detail below. It is recommended, however, that tools supplied pre-padded by Electron Microscopy Sciences be used. The pad life is longer, and the resultant specimens experience less breakage, and are of better quality.

The polishing pad should be cut precisely with the two long sides accurately parallel. The length of the strip should be approximately ¼ inch longer than the circumference of the tool to allow for accurate trimming, and the ends trimmed at a 45° angle.

Although the polishing pad has an adhesive back, additional bonding strength is achieved by applying a very thin layer of a rapid cure liquid glue to the circumference of the tool. This can be done after the pad is placed around the tool by wicking a small amount of glue from a toothpick. Capillary action will draw the glue between tool and pad. Do not use too much glue, as it can soak through to the polishing side of the pad and scratch the specimen when in use. Press the pad evenly and firmly on the tool circumference, a small section at a time.

Give the glue sufficient time to set. The curing process can be accelerated by placing the tool on a low-heat hot plate for extended periods of time.

Trim the last section of the pad to make a precise fit where the two ends come together. The union should not create a bump in the pad, but be sure there is sufficient glue under the two ends of the strip to prevent them from lifting during polishing.

Other than pad replacement, polishing tools should last indefinitely.

Figure 13. Polishing pad template.

Changing specimen bulb (For units prior to 1999)

There are two ways to reach the specimen back lighting bulb in the platen tower:

1. Remove the electronics control module, then unscrew the four bolts holding the X-Y specimen stage to the base plate. Carefully cradling the assembly, place it upside down on a soft padded bench. Be sure the leads are disconnected from the terminal block. Note the original X-Y micrometer settings, then adjust the X-Y specimen stage micrometers so that the four corner

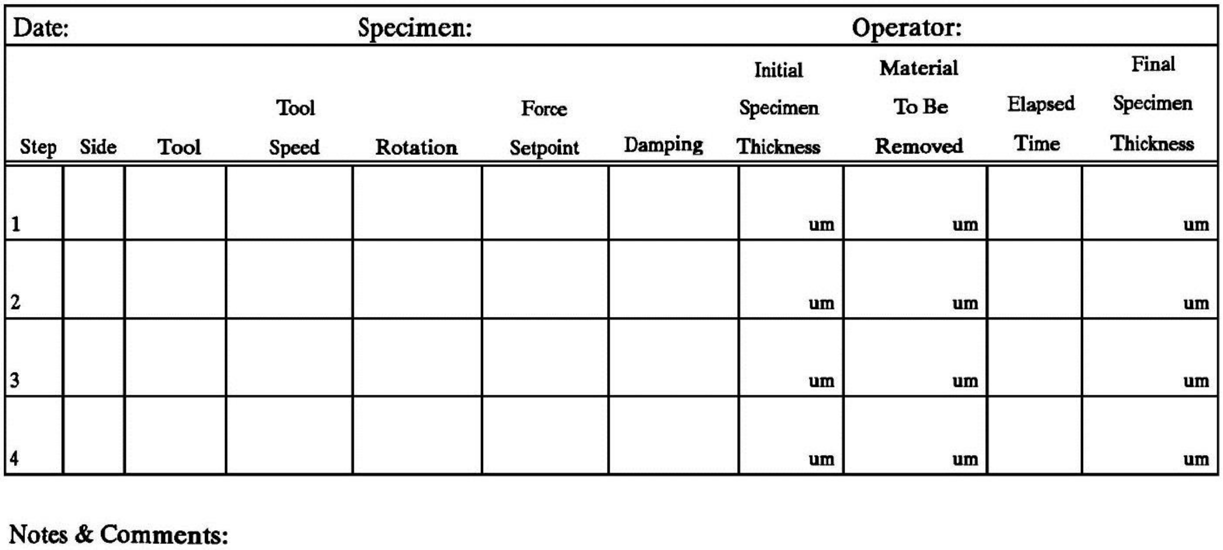
holes line up and the hex-head bolts holding the specimen mount are visible. Insert a 3/16" ball driver through each of the four holes and remove the four bolts. The specimen mount can now be separated into two pieces. The bulb is inside the larger, top piece. Replace the bulb carefully. It is a bayonet mount and should be pushed and turned gently. Readjust X-Y micrometers.

2. The second way to reach the bulb is to remove the specimen mount from X-Y specimen stage directly, without removing the electronics control module. There are 1/4"-20 bolts holding the specimen mount to the X-Y specimen stage. Remove these bolts with a small wrench. Patience is required to insert the short end of a 5/32" Allen wrench upside down into the four hex-head bolts.

X-Y Specimen stage adjustment

1. Mount Tool 3i on the spindle shaft.
2. Mount the specimen platen onto the hex spindle shaft without the retaining ring and the sapphire flat.
3. Rotate the tool drive shaft until the Tool Index LED is on.
4. Set the FORCE to 25g.
5. Press ARM-DN and Tool 3i will seat into the 3 mm hole.
6. Slowly adjust the X adjusting screw drive until the lowest Z reading is displayed.
7. Slowly adjust the Y adjusting screw until the lowest Z reading is displayed.
8. When lowest readings, (or most negative number), is displayed the X-Y stage is aligned.

Specimen log sheet



Date: Specimen: Operator:

Step	Side	Tool	Tool Speed	Rotation	Force Setpoint	Damping	Initial Specimen Thickness	Material To Be Removed	Elapsed Time	Final Specimen Thickness
1							um	um		um
2							um	um		um
3							um	um		um
4							um	um		um

Notes & Comments:

For any questions or for ordering information, please contact Customer Service at 1-800-523-5874.

Thank you for choosing Electron Microscopy Sciences!

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