MTEC MicroLap Manual

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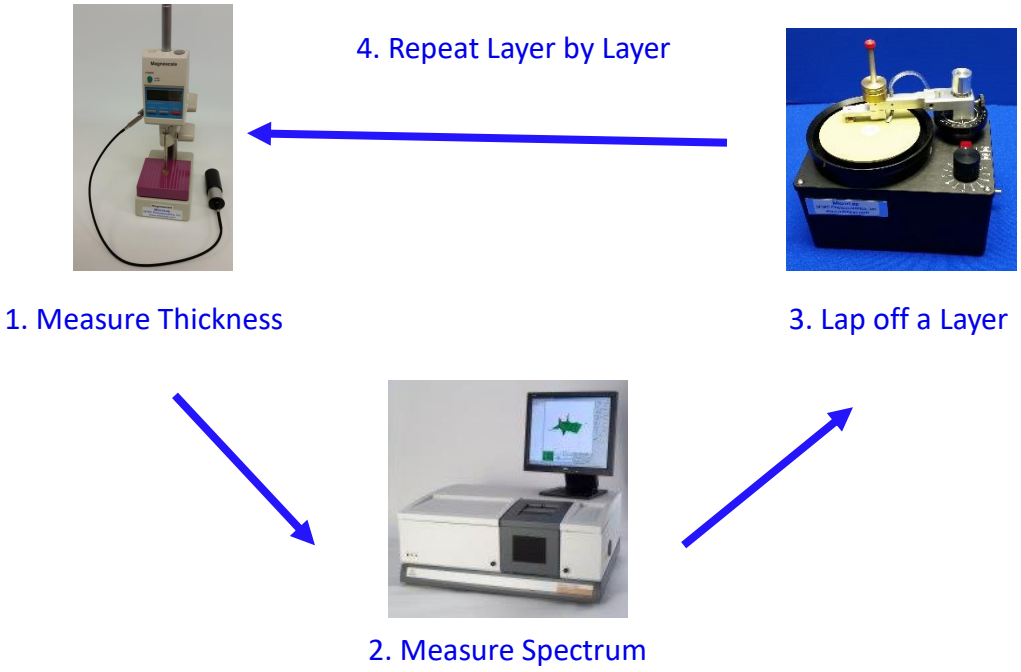
Table of Contents

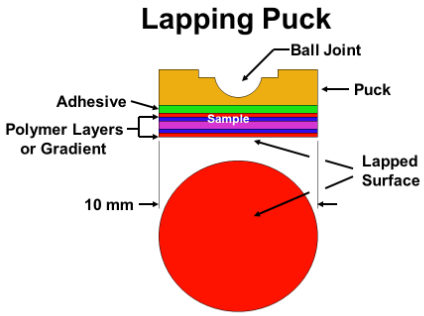
Introduction

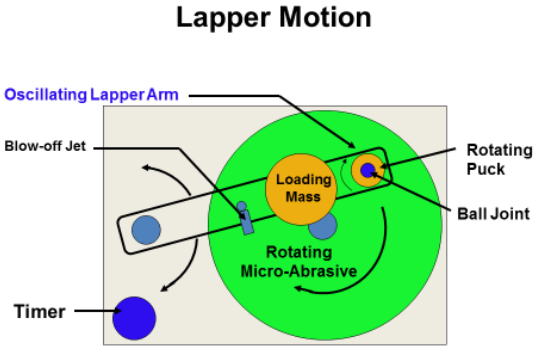
MicroLap allows planar materials with gradient or layered compositions to be analyzed as a function of depth, layer by layer, using FT-IR spectroscopy. MicroLap employs a rotating lap operated with a choice of abrasive sizes, lapping times, and mass (force) loadings to precisely remove thin layers from the surface of a sample. A precision electronic gauge is used to measure the thickness of the removed layer while FT-IR photoacoustic (PAS) or attenuated total reflectance spectroscopy (ATR) determines the sample chemistry as a function of depth as each layer is removed.

Depth Profiling Using the MicroLap Process



Lapping Process





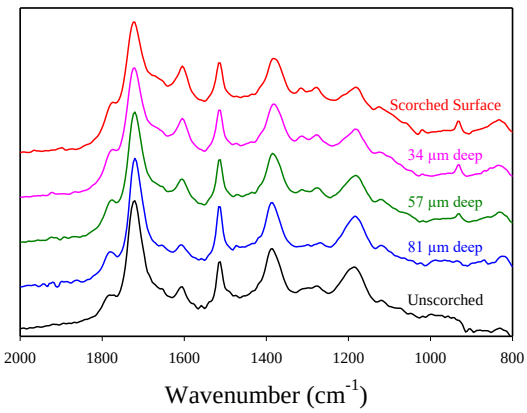
Samples are mounted on the Lapping Puck with 3M Removable Double-Stick Tape.

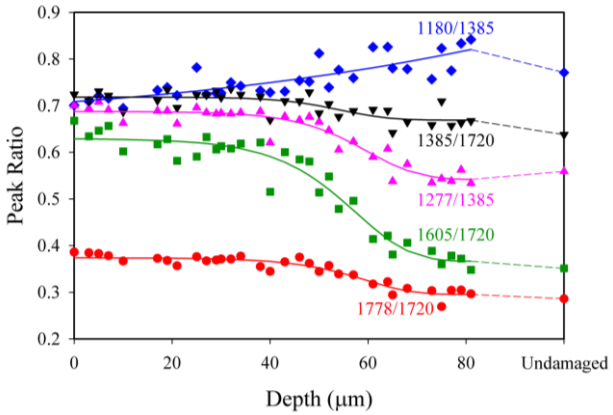
The Lapping Puck is placed on the Lapper for removing a layer of the sample.

Most planar materials, ranging from polymers to coated paper samples, can be dry lapped using an appropriate abrasive type and grit size. Aluminum oxide abrasive works well for general purposes and disks with this abrasive are supplied with MicroLap in 1, 3, 12, 30, and 60 micrometer alumina abrasive grades. Diamond abrasives are available from MTEC if needed. Lapping times typically range from 10 to 120 seconds using the timer built into the lapper. An additional more precise external countdown timer is also provided which allows the lapping time to be set on a second scale. The lapper design allows for wet lapping but this is not advisable in most cases because moisture may make the sample swell and water bands to appear in the spectra.

Since the lapping operation creates fine particles, precaution should be taken if the material is toxic in terms of respiration of particulates. Concerns in this regard are best addressed by operating the lapper in a hood where particulates will be swept out of the occupied space. Wet lapping, when possible, can be used to limit airborne particulates.

Typical MicroLap results are displayed in these plots that show spectral variations as a function of depth in a composite material damaged by exposure to a stream of 617 F air for 15 seconds.





MicroLap Parts List

Lapper and auxiliary parts

Lapper with splash pan
Power supply
Hard and resilient surface lapping discs with aluminum centering piece for lapping films
Tubing for blow-off air
6 Lapping weights
Abrasive lapping film (20 sheets of 1, 3, 12, 30, and 60 micron alumina grit sizes)
Precision external timer
Manual for MicroLap at: http://www.mtecpas.com/Docs/MicroLap%20Manual.pdf

Magnascale Gauge

Digital Gauge
Power supply
Measuring stand with ceramic stage
Tip lifter with cable
Manual for Gauge at:
http://www.mgscale.com/mgs/language/english/product/Useries.html

Sample Handling

4 Lapping pucks and masking ring (masking ring provided if photoacoustic rather than ATR spectroscopy is to be used)
3M double-stick removable tap
Scissors and razor blade for trimming samples after mounting on pucks
Adhesive release paper
Q-tips
Tweezers

Initial Setup

A source of oil free compressed air or nitrogen is useful for blow off to prevent material from building up on the lapping pad. A valve to adjust the flow rate and a flip valve to turn the flow on and off once the flow rate has been set is desirable.

The lapper and digital gauge should be placed in close proximity to allow convenient checking of material removal.

Mounting Samples

Sample Puck

Samples are mounted using 3M double-stick removable tape. The removable tape is adequate to hold most samples to the puck but more aggressive versions of double stick tape can be used if necessary but the tape will be more difficult to remove from the puck.

First, apply the tape to a piece of adhesive release paper and then to the puck. The flat surface of a microscope slide is a good surface to press against to establish an even contact with the adhesive across the puck's surface. The excess tape should be trimmed around the puck's perimeter using the scissors first and following up with the razor blade so that no adhesive extends beyond the perimeter. The release paper is then removed. No air bubbles should be present between the tape and puck. If any are observed they can be rubbed out by putting another piece of release paper on the tape and rubbing the handle of the scissors across it. At this point, the puck is ready for sample attachment.

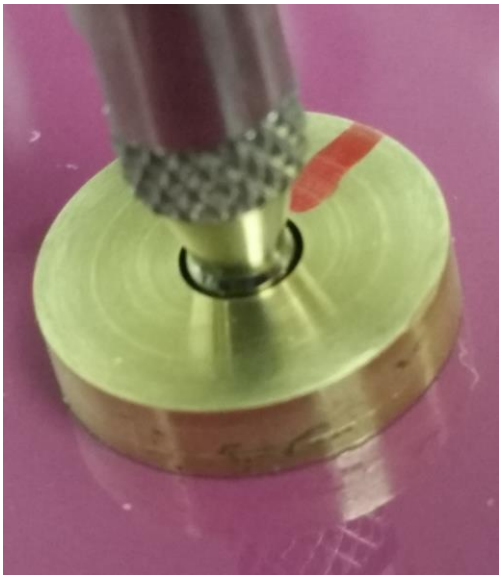
Samples that can be trimmed with the scissors and razor blade can be applied directly to the puck and then carefully trimmed so that no material extends beyond the puck perimeter. Trimming often raises a microscopic ridge of sample material around the edge of the puck which can be minimized by pressing the puck mounted sample firmly against a microscope slide.

Samples that cannot be trimmed with the scissors and razor blade should be punched (http://www.mtecpas.com/samplepunch.html) or cut in some other way. It is important that the planarity of the sample not be destroyed when samples are cut. An EDM machine will cut metal backed samples without disturbing planarity. If the punching or cutting operation produces a ridge at the edge of the sample's perimeter so that the sample's surface will not contact the puck across its surface, this ridge must be removed prior to mounting on the puck. Once the sample is mounted, it should be pressed firmly against the slide to be sure it is well seated.

Using the Gauge

Please consult the Magnascale Instruction Manual at: http://www.mgscale.com/mgs/language/english/product/Useries.html , involving initial setup and operation.

It is important that the gauge ball contact and ceramic stage surface be kept free of dirt of any kind and that the ball well on the puck be also free of any contamination. Kimwipes, cotton Q-tips, and a can of duster gas are useful for this purpose. Periodic blow off of these surfaces should be done during measurements.



Digital Gauge Gauge Tip Contacting The Sample Puck

The contact between the stage and sample and between the gauge probe and the puck's ball well should be established in a reproducible way. No air layer should be present in the former case and, in the latter it, is important that the gauge ball be fully seated in the puck's well. These are best accomplished by gently tapping the gauge contact ball in the puck ball well approximately 10 times before each measurement using the hand plunger that lifts the gauge probe.

Use this technique to set the zero on the gauge prior to lapping and to check reproducibility by removing and replacing the puck. It is best to use the tweezers to handle the puck.

The initial gauge readings during lapping often will reflect the removal of material on the perimeter of the sample that is above the planar surface of the sample. PAS and single bounce ATR measurements focus on the center area of the sample and may be insensitive to the lapping process until the perimeter and central area are coplanar.

Faster material removal usually occurs until all of the sample's surface is completely planar. The spectra measured during this initial process often show little variation but are important because they provide a means of knowing the effective zero point in the lapping process at which layers are actually being removed that reflect changes in chemical composition with depth.

Gauge readings should be recorded before and after each spectroscopic measurement. With many samples the "after" measurement will show a shift indicating less material removal than indicated by the "before" measurement due to a relaxation of the sample after the removal process. The "after" measurement is best to use when constructing composition versus depth plots. It is important to do the "ten tap" procedure before all measurements.

FTIR Spectroscopic Measurements

FTIR spectra can be measured using either photoacoustic or ATR spectroscopy. The photoacoustic method is preferable because it is a noncontact measurement whereas ATR requires pressure on the sample to establish optical contact that may result in compression and disturb the gauge measurements.

Photoacoustic Spectroscopy Method

It is important to pick FT-IR measurement parameters that do not over resolve either spectrally (usually 8 cm-1 to 32 cm-1) or depth wise (2.5 kHz to 10 kHz on the laser fringe or 0.1 cm/s to 0.6 cm/s OPD mirror velocity) because overall measurement time will be significantly increased when these parameters exceed necessary values.

Consult the Applications Library, http://www.mtecpas.com/applicationslibrary.html , regarding sampling depth and other information on depth profiling. Due to the role of exponential decay of optical and thermal waves in photoacoustic signal generation, the sample layers closest to the surface of the sample always have the strongest contribution to the spectrum. Hence, it may be acceptable to do measurements with a photoacoustic thermal sampling depth which is somewhat large relative to the layer increments that are being removed. For instance, one might be removing 3 to 5 micrometers during each lapping step but measuring with an L value of 6 or 8 micrometers or even greater for the spectral band of interest.

Another spectroscopic probe depth consideration is the absorbance strength of the band being monitored in order to depth profile a particular component. If the band is very strong, such as a C-H band, it may be the limiting factor in probe depth at high concentration rather than L. As concentration changes with depth, however, L could become the dominant factor. In view of this consideration it may be desirable to monitor weaker rather than stronger absorbance regions of spectra so that L is the dominating factor throughout.

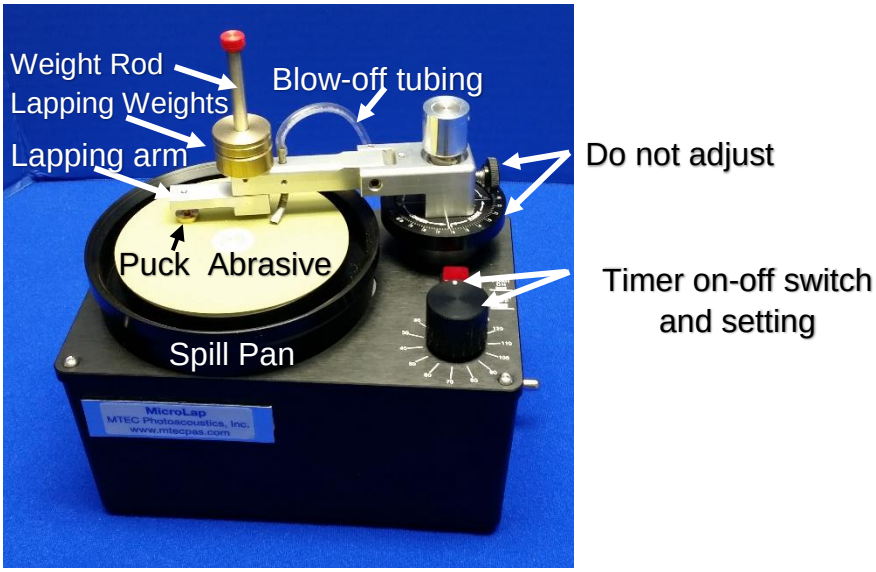
The sample should be placed in the sample cup with a desiccant filled cup at the bottom of the brass sample holder followed by spacers under the sample. The stainless steel ring that is supplied to PAS users should be positioned on top of the sample. The spacers should be adjusted so that the ring is just below the brass cup's rim and never above the ring to avoid breaking the window.

ATR Spectroscopy Method

If ATR measurements are used, an accessory with a large diameter crystal (http://www.harricksci.com/sites/default/files/pdf/data_sheets/Data_Sheet_FastIR.pdf) is needed to avoid compressing softer samples. Compression will interfere with both the lapping depth quantitation and spectroscopy processes. If the samples are harder, a small contact area crystal accessory may be acceptable, http://www.harricksci.com/sites/default/files/pdf/data_sheets/Data_Sheet_MVP_Pro.pdf

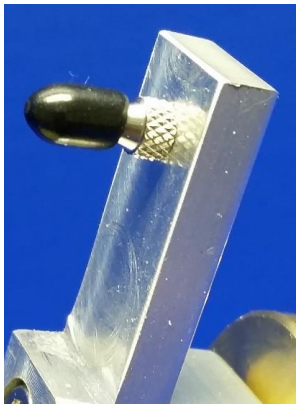
Lapper Description

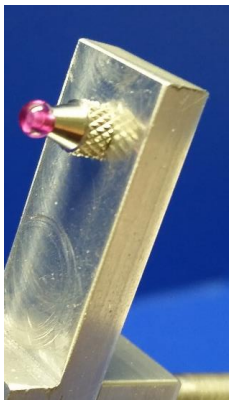
These photographs show the lapper components and controls.



Lapper Overview

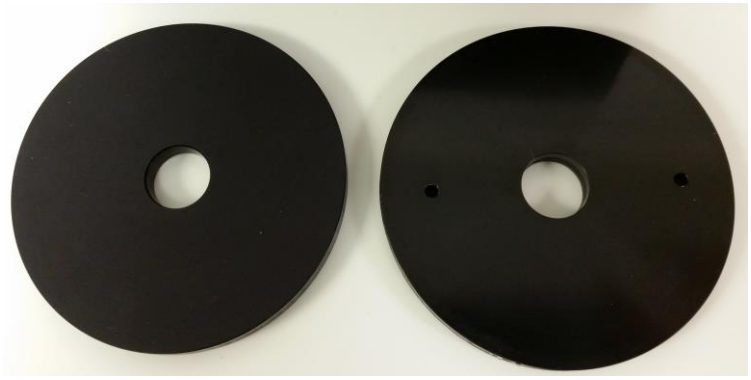
Lapper Power Switch



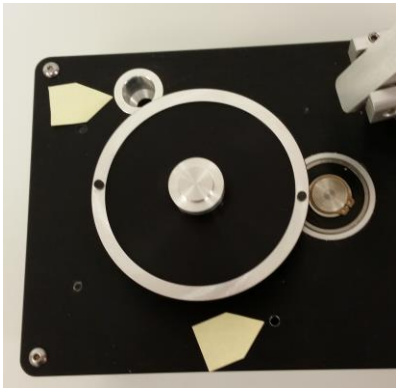


Lapper Arm with protective cap and Ruby Ball Joint exposed. This ball should be protected from shock and abrasion

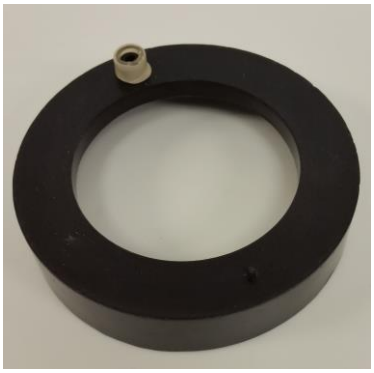
Engagement Pins for Discs



Lapping Discs Mount into Pins



Pan Holes



Spill Pan

Lapper Setup

A lapping disc installs on the lapper with the pins mating into the bottom side of the disc. Normally the hard disc is used. The weight rod is installed on the lapping arm. The power supply is connected. The power switch has three positions. In the **Timer On** position, the 0 to 120 sec. timer on the lapper can be used. In the middle position, all power is off. In the lower position, the precision digital timer can be used for more accurate control.

Electrical Connections

110 V Operation

220 V Operation with 220 V to 110 V Converter

220 V Operation with External Timer (Timer Plugs Directly for 110 V)

Setting the Lapping Time Duration

There are two methods for setting the lapping time,

1. A rotary switch on the lapper can be set for a maximum time of 120 sec. by setting the switch on the side of the lapper to **Timer On** and the rotary switch to the desired time. Make the electric power connections as shown in the preceding photographs. Press the **red button** twice to start the lapper.

2. For a more accurate and reproducible lapping time with one second time resolution use the external white timer. Set the switch on the side of the lapper to **Power Off.** Make the electrical connections as shown in the preceding photographs.

   To program the timer:
   1. Press its **Switch** button.
   2. Press the **Timing Circulation** button for 3 seconds and you see the **On** and **Off** time settings on the screen.
   3. The timer is now operating in the “seconds mode” and the button labeled **Minute** is controlling settings in seconds. The button labeled **Hour** is controlling settings in minutes.
   4. Use the **Minute** button to set the desired **On** time duration of the lapping in seconds on the upper screen. If the lapping time needed is in minutes, use the **Hour** button.
   5. Once the **On** time is entered and displayed on the flashing upper screen, press the **Timing Cycle** button and the upper screen will stop flashing and the lower screen will commence flashing.
   6. Enter an **Off** time of typically 5 sec. and press the **Confirm** button. The timer will now continuously countdown through the **On** and **Off** cycles. During these cycles the **On** status or **Off** status will be displayed as shown in the pictures below.

On Cycle

Off Cycle

7. Watching the repeating **On-Off** cycle of the timer, switch the switch on the side of the lapper to **Power On** during the **Off** cycle of the timer. At the end of the **Off** cycle the lapper will turn on automatically for the specified **On** time. When the lapper stops, switch the lapper switch to **Power Off.** Repeat Step 7 to continue to the depth profile the sample.

Caution: The blue buttons on the external timer occasionally stick after being pressed. They can be released using your finger nail.

Setting the Clock

1. Press the **Switch** button.
2. Press the **Confirm** button for 3 seconds.
3. Enter the **hour** and **minute** numbers using their buttons.
4. Press the **Confirm** button.

Lapper Operation

Be sure to zero the gauge and measure the first spectrum before beginning the lapping operation. Operate the lapper in a hood if the particulates generated are considered an inhalation hazard.

Different samples lap at different rates and the rate for a given sample usually changes during the lapping operation. Initially, material removal often is more rapid until the sample surface becomes completely planar. Later in the operation the rate decreases due to wear and loading of the abrasive disk. Usually gradually increasing the lapping time and mass on the lapping arm can be done to compensate for this before changing to a new abrasive pad. If slow material removal indicates that the abrasive pad should be replaced or that a coarser abrasive would be better, it is important to reduce the lapping time and arm mass so as not to remove too much material under the new conditions. Lower mass loadings with longer lapping times will reduce sample compression and improve the accuracy and repeatability of gauge measurements.

Experience is the best indication for determining the initial alumina abrasive grade (1, 3, 12, 30 and 60 micrometers), lapping time (30-120 sec.), and arm mass (50, 40 x 3, 20, and 10 gm). Lapping time is controlled by the knob on the lapper or with the digital timer. A typical initial parameter set for a polymer sample might be 3 micrometer abrasive, approximately 10 sec. lapping time, and 30 gm arm mass with lapping time being the first parameter to adjust followed by arm mass.

The dull side of the abrasive pad has the abrasive coating whereas the shiny side should be mated against the black lapping plate. Both the plate and the shiny side of the pad should be rubbed with a water-moistened Kim wipe prior to applying the pad to the plate. No adhesive should be used nor should any moisture be on the abrasive surface. The lapping is done dry to avoid contaminating the sample with water.

After the pad is attached and the power supply and air supply connected, the sample/puck should be positioned under the ball of the lapper arm (remove black plastic cover first) so that the ball is engaged in the ball socket of the puck. Set a fairly brisk airflow rate that will keep the pad free of powder from the lapping. During lapping, the puck will be seen to be rotating by watching the red mark on the top of the puck.

After the first lapping, flip up the arm and allow the air jet to blow off the lapped sample surface by holding both sides of the puck in front of the jet before placing it on the gauge to check the amount of material removed.

Repeat the sequence of lapping and spectroscopic measurements to generate a series of spectra indicative of the sample's depth-varying composition.

Publications:

MicroLap Depth Profiling of a Coated Paper:
http://www.mtecpas.com/Docs/MicroLap1.pdf

MicroLap Depth Profiling of Automobile Paint Weathering:
http://www.mtecpas.com/Docs/MicroLap2.pdf

Quantitative Depth Profiling Using Saturation-Equalized Photoacoustic Spectra:
http://www.mtecpas.com/Docs/MicroLap3.pdf

Degradation of ROMP-Based Bio-Renewable Polymers by UV Radiation:
https://research.wsulibs.wsu.edu/xmlui/bitstream/handle/2376/5570/95-degradation.pdf?sequence=1&isAllowed=y

Influence of UV weathering on corrosion resistance of prepainted steel:
file:///C:/Users/John/Downloads/UV%20weathering%20of%20Painted%20Steel.pdf