

MTEC MicroLap Manual



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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 or reflectance spectroscopy determines the sample chemistry as a function of depth as each layer is removed.

Most planar materials can be lapped using an appropriate abrasive type and grit size ranging from polymers to coated paper samples. 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 grit sizes. Typical lapping times range from a 10 to 120 seconds.

Set Up

The lapper and components should be removed from the box and the lapping arm attached to the lapper shaft using the Allen wrench supplied.

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.

A source of oil free compressed air or nitrogen is required 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 gauge should be placed in close proximity to allow convenient checking of material removal.

Mounting Samples

Samples are mounted using 3M double stick tape. First, apply the tape with its release paper 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 the microscope slide. Be sure that the slide is on a clean flat surface to avoid cracking it.

Samples that cannot be trimmed with the scissors and razor blade should be punched 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 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

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 and Q-tips are useful for these purposes. Periodic wiping of these surfaces should be done during measurements.

The contact between the stage and sample and between 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 check reproducibility by removing and replacing the puck. It is best to use the tweezers in handling the puck.

The initial gauge readings during lapping often will reflect the removal of material above the planar surface of the sample which is not in the center 6 mm dia. area of the sample which is probed spectroscopically by PAS. The center area is defined by a stainless steel masking washer that is placed on the sample during the spectroscopic measurement. Faster removal usually occurs until all of the sample's surface is planar. The spectra measured during this 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.

Spectroscopic Measurements

It is important to pick FT-IR measurement parameters that do not over resolve either spectrally or depth wise because overall measurement time will be significantly increased when these parameters exceed necessary values.

Consult the MTEC literature supplied with your photoacoustic detector regarding sampling depth. 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 photoacoustic thermal sampling depth as defined by $L=(D/\pi f)^{1/2}$, where the thermal diffusivity and modulation frequency are given by D and f , respectively, which is somewhat “coarse” 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.

Spectral resolution should be set no higher than necessary to reduce the spectrum acquisition time required for an adequate signal-to-noise ratio. In many applications, 16 cm^{-1} or even 32 cm^{-1} may be sufficient.

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 should be positioned on top of the sample. The spacers should be adjusted so that the ring is flush with or just below the brass cup’s rim.

Lapping Operation

Be sure to zero the gauge and measure the first spectrum before beginning the lapping operation, and to 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 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 finally 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.

Experience is the best indication for determining the initial abrasive grade (1, 3, 12, 30 and 60 micrometers), lapping time (30-120 sec.), and arm mass (50, 40 x 4, 20, and 10 gm). Lapping time is controlled by the knob on the lapper or manually with the switch while watching a clock or watch. Shorter lapping times than 30 sec. will require switching off the lapper manually. 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 well-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. The lapping operation is started by pressing the red button once or twice (depending on the conditions under which the lapper was last stopped). 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 the sample in front of the jet before placing it on the gauge to check the amount of material removed. This begins the sequence of lapping and spectroscopic measurements that will provide depth-varying information on the sample.