ULVAC-PHI, INC.

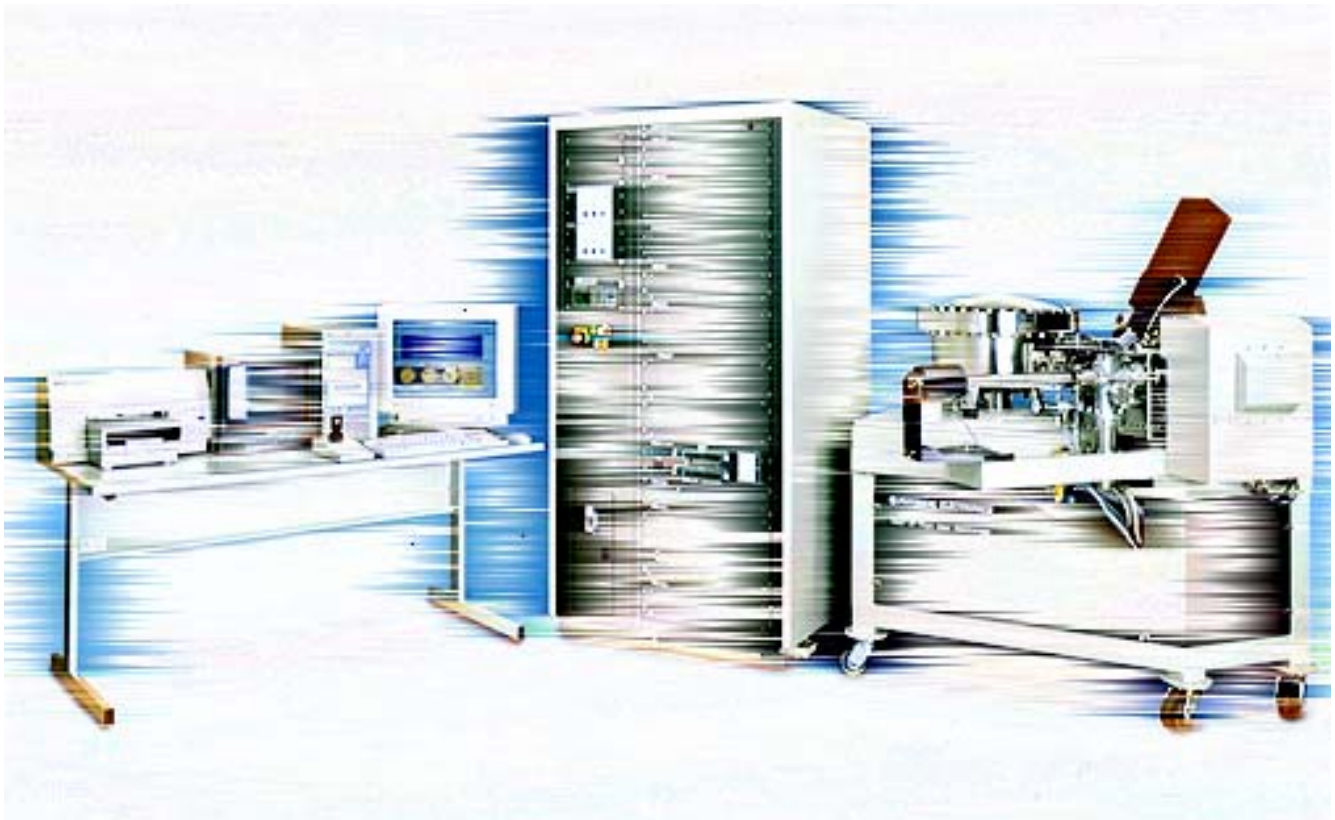**TRIFT III TOF SIMS**

Operator's Guide

Part No 649277 Rev. C

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• Has read and understood the PHI technician's manual for the equipment,

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PHI's products are installed with international-style or **ANSI**†-style safety notices, according to site requirements. International notices are symbols within triangles (alerts) or circles (mandatory actions). PHI's ANSI-style safety notices contain:

• One of three signal words (in all capitals) preceded by the general danger symbol ($\triangle$);

• One of PHI's safety symbols along with a brief description of the hazard and the risk or injury that could occur;

• Short message that observes ANSI's Hazard Alert Trilogy Rule by identifying the hazard, the possible result of ignoring the notice, and how to avoid the hazard.

The three signal words are defined as follows:

• **DANGER**—imminently hazardous situation that, if not avoided, will result in death or serious injury;

• **WARNING**—potentially hazardous situation that, if not

avoided, could result in death or serious injury;

• **CAUTION**—potentially hazardous situation or unsafe practice that, if not avoided, may result in minor or moderate injury or damage to equipment.

SEMI‡ standards require identification of type 3, 4, and 5 electrical maintenance tasks in equipment manuals:

• **Type 3** electrical maintenance tasks involve energized equipment, exposed live circuits, and possible accidental contact; potential exposures are less than 30 V RMS, 42.2 V peak, 240 V-A, and 20 J.

• **Type 4** is the same but potential exposures are greater than 30 V RMS, 42.2 V peak, 240 V-A, and 20 J or radio frequency is present.

• **Type 5** tasks involve energized equipment and measurements and adjustment require physical entry into the equipment, or equipment configuration will not allow the use of clamp-on probes.

Only experienced, trained technicians should attempt to perform type 3, 4, or 5 electrical maintenance tasks.

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* Many of PHI's safety symbols are provided and copyrighted by Hazard Communication Systems, Inc., Milford, PA.

* American National Standards Institute, 1430 Broadway, New York, NY 10018.

‡ Semiconductor Equipment and Materials International, 805 E. Middlefield Rd., Mountain View, CA 94043-4080.
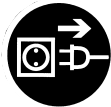
PHI Safety Notices

Voltages may be present that could cause death or personal injury.

A risk of death, personal injury, and/or damage to equipment exists (and a more specific label is not available).

Pulling the plug from its power source before servicing is mandatory.

A pinching point is present that could cause personal injury.

A risk of explosion or implosion may be present that could cause personal injury.

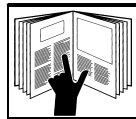Lifting with assistance or equipment could cause personal injury.

Visible or invisible radiation may be present that could cause personal injury.

Hot surfaces may be present that could cause personal injury.

Turning off the power switch before servicing is mandatory.

Refer to the manual(s) before proceeding.

Contents are under pressure.

A harmful or irritant material may be present that could cause personal injury.

Extremely low temperatures may be present that could cause personal injury.

A potentially dangerous magnetic field may be present.

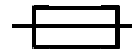Wearing protective gloves is mandatory.

Wearing eye protection is mandatory.

Wearing foot protection is mandatory.

This is the location of the protective grounding conductor terminal.

This is the location of the fuse.

This is the location of an earth (ground) terminal.

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Limited Warranty

Except as otherwise provided herein, the Seller warrants to Buyer that the equipment sold hereunder, whether it is new equipment or remanufactured (reconditioned) equipment, is, at the time of shipment to Buyer from Seller, free from defects in material and workmanship. As Buyer's sole exclusive remedy under this warranty Seller agrees either to repair or replace, at Seller's sole option and free of part charge to Buyer, any part or parts of such equipment which, under proper and normal conditions of use prove to be defective within 12 months from the date of receipt by the Buyer. Warranty period for equipment requiring installation by Seller will commence on completion of standard installation services. If customer delays installation beyond 45 days after delivery, the warranty period will commence to run 45 days after delivery. After installation, any realignment, readjustment, recleaning or recalibration, provided it does not relate to a proven defect in material or workmanship, shall be performed only at Seller's then current rates for service.

Exclusions and Limitations

It is recognized that some parts by their nature (expendable items) may not function for one full year; therefore, excluded from the foregoing warranty are filaments, anodes, cathodes, multipliers, retard grids, special ceramics, ionizers, along with other such parts mentioned in the applicable operating manual.

The foregoing warranty excludes certain major items or accessories specifically indicated on applicable price lists or quotations, as to which Seller passes to Buyer whatever warranty is provided to Seller by the manufacturer or the specific warranty indicated by the price list or quotation.

This warranty does not cover loss, damage, or defects resulting from transportation to the Buyer's facility, improper or inadequate maintenance by Buyer, buyer-supplied software or interfacing, unauthorized modification or misuse, operation outside of the environmental specifications for the equipment or improper site preparation and maintenance.

Product Service

All claims must be brought to the attention of Seller within 30 days of the failure to perform.

Seller at his option may require the product to be returned to the factory, transportation prepaid, for repair.

Refund of Purchase Price

In lieu of the foregoing, Seller may at any time elect, in its sole discretion, to discharge its warranty by accepting the return of such equipment and refunding any portion of the purchase price paid by Buyer.

Software and Firmware Products

The sole exclusive warranty applicable to software and firmware products provided by Seller for use with a processor will be as follows: Seller warrants that such software and firmware will conform to Seller's program manuals current at the time of shipment to Buyer when properly installed on that processor. Seller does not warrant that the operation of the processor software or firmware will be uninterrupted or error free.

No other warranty is expressed or implied. Seller expressly disclaims the implied warranties of merchantability and fitness for a particular purpose.

Section 1:
Introduction

Physical Electronics, Inc. (PHI) provides three main system manuals which pertain to the TRIFT III system—this operator's guide, and the *Operator's WinCadence Version 3.5 Software Reference Manual*, several individual OEM and PHI-based components, including the system computer, printer, turbo pumps, monitors, gauges, and some electronics units.

This manual should be used in conjunction with the WinCadence Software Reference manual and the Hardware manual for the TRIFT III TOF-SIMS system. The software manual describes the user interface and details all functions within the software. The hardware manual describes the system hardware, periodic service procedures, and contains a complete set of system cable diagrams.

This *TRIFT III Operator's Guide* takes the system operator through operation sequences, from system startup to data acquisition, to system log off and shutdown. It is intended as a resource for getting familiar with how to operate the *TRIFT III* system under routine conditions.

Physical Electronics' TRIFT III system is operated using one main interacting software application, namely WinCadence. This software operates in conjunction with the Microsoft Windows NT operating system. The operator uses the WinCadence software to set all instrument parameters, perform data acquisition, data reduction, and publishing of the results. An additional software utility known as *Watcher* is also used on the TRIFT III system for controlling various pneumatic valves on the vacuum console.

This section presents an overview of Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) and data acquisition using the TRIFT III TOF-SIMS:

- Operator's Guide: Purpose and Contents.
- Introduction to SIMS and TOF-SIMS.
- Brief Introduction to the System.
- Data Acquisition Modes.
- How To Reach PHI Customer Service.

Operator's Guide: Purpose and Contents

The TRIFT III, once installed in the analytical laboratory, can be operated in a "push and go" mode, using pre-recorded "instrument states", with little knowledge of primary gun or spectrometer tuning. However, as the user becomes more familiar with the operation of the instrument, it is inevitable that he or she will want to delve deeper into how the instrument functions, so as to explore the full capabilities of this powerful analysis tool. This guide has been written with that aim in mind. Many of the sections are extremely detailed and it is certainly not necessary to read and understand all that is written for you to make good use of the instrument initially. Like all users' guides, you will probably begin to consult it after you have had some "hands on" experience. The guide is used as a basis for your instrument training, and can be used thereafter as a reference to remind you of certain principles and methods that you learned during training.

The layout of the guide follows the logical sequence in which you come across various aspects of the instrument as you move from loading a sample through to data acquisition. Some of the sections, such as LMIG fine tuning and spectrometer theory and tuning may appear not only daunting, but rather overly detailed, especially as we have noted that the instrument is designed for routine "push and go" analysis without having to worry about such tuning. While it is intended that most analyses be conducted in this way, we do not wish to withhold detailed descriptions on how to proceed if you have to conduct the tuning yourself, and thus the details in these sections are for those who wish to understand *why* the instrument defaults have been set the way they are.

The operator's guide attempts to follow the order in which various aspects of the instrument are encountered when performing a typical analysis:

Section 2—Start your analysis sequence by first mounting and introducing a sample. This section details how to mount various size sample carriers for the two different types of sample introduction systems (50 mm and 200 mm).

Section 3—Calling up the instrument files from the computer to setup the system in the required 'state' is the next step prior to analysis.

Section 4—Tuning the TRIFT primary beams is the third step in the analysis process. This should be set automatically by the instrument file and need no adjustment. However, if necessary, detailed tuning of the LMIG, the Cesium gun, and the low energy electron neutralizer are all covered in this section.

Section 5—The next required step is to perform a simple, software-assisted alignment of the primary beam(s) to the mass spectrometer acceptance area. This section details this alignment, as well as adjustment of the light optics, and the positive and negative fields of view.

Section 6¾ The next step involves matching the energy of the secondary ions to the mass spectrometer. The mass spectrometer is normally not adjusted from day to day, but a detailed explanation is provided in this section should it be necessary to check the spectrometer tuning.

Section 7—This section explores the various types of analysis which can be performed, including mass spectral acquisition, imaging, depth profiling, retrospective analysis, and ROI (Region-of-Interest) analysis.

Section 8—This section covers charge neutralization of insulators.

Section 9—This section is an extensive treatment of sample preparation techniques including all different types of samples (i.e., powders, slurries, fracture samples, cross sections, fibers etc.)

Section 10—This section outlines routine instrument evaluations and general maintenance including any procedures of a non-service nature (i.e., those tasks that do not require a field service engineer or technician).

Introduction to SIMS and TOF-SIMS

When energetic "primary" ions (typically, several keV) bombard a sample surface in vacuum, various atoms and molecules are 'sputtered' from that surface. A small percentage of those sputtered species are ionized naturally and may be extracted by an electric field and then transferred into a mass spectrometer, where their mass-to-charge ratios are measured. The "secondary ions" (to distinguish them from the primary bombarding ions) are emitted from only the top few atom layers, and thus secondary ion mass spectrometry, or "SIMS", belongs to the family of surface analytical techniques which include Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) for characterization of material surfaces.

Energy from the primary ion is transferred into the sample surface in such a way that, in the immediate vicinity of the impact site, considerable fragmentation of the original surface occurs, resulting in the release primarily of atomic and low mass molecular species. Further out from the impact site the energy imparted to the substrate results in the release of higher mass molecular species. This is illustrated in Figure 1-1 below.

DE$^+$ M$^+$
ABC$^+$
ABCDE$^+$

Primary Ion
10^{12} - 10^{13} ions/cm^2
(Static SIMS Limit)

ABCDE
M
ABCDE

Molecular
Desorption
Area

Sputtering
Region

20 - 100 Å
10^{15} atoms / cm^2
Single monolayer coverage

Figure 1-1.   *An Illustration of the SIMS Process: Atomic View of Individual Primary Ion Impact.*

There is a regime of primary ion bombardment, known as the "static" SIMS regime, during which the number of primary ions impacting the surface per unit area is so low that each successive primary ion strikes a virgin area of the surface. This "static" SIMS regime does not end abruptly, but it is generally accepted to be limited to ~10^{12} - 10^{13} primary ions/cm^2. The density of atoms on a silicon surface is ~10^{15}/cm^2 for comparison. This regime is said to be "static" because less than one monolayer is typically removed from the sample surface. It is in this regime that organic surface characterization is achieved. Beyond this ion beam dose, incoming primaries begin to sample already sampled (damaged) material, and the amount of undamaged high mass molecular material remaining begins to fall off rapidly.

Since there is a limited amount of secondary ion information available within this "static" regime, any SIMS instrument which is intended to be used for surface organic characterization must make optimum use of this limited material. SIMS instruments with quadrupole- or magnetic sector-based spectrometers make use of the atoms (mainly) and low mass molecules to make surface characterization measurements, and are well suited to depth profiling applications in a "dynamic" SIMS mode. In the "dynamic" SIMS mode the surface is deliberately eroded to determine the concentration of various species as a function of depth into the solid, and in fact little attention is paid to the organic information available at the

outer surface. To be fair, quadrupole SIMS instruments have been used successfully for many years to perform static SIMS measurements, although the quadrupole is a relatively inefficient analyzer considering the limited information available in the static SIMS regime. More recently, instruments based on time-of-flight mass spectrometers have been added to the SIMS family, increasing the range of secondary ion detection out to many thousands of mass units, and making optimum use of those molecular secondary ion species released from the outer periphery of the primary ion impact zone.

Unique properties of TOF-SIMS

In a quadrupole or magnetic sector SIMS analyzer, the spectrometer scans through the mass range to record a mass spectrum. If the mass spectrometer is analyzing Fe it is *not* analyzing Al or Cu or indeed any other secondary species. This is not a serious restriction if only one or two species are being monitored in a depth profile, but it is a problem for surface studies within the static SIMS regime. By contrast, the TOF spectrometer is a parallel detection analyzer which does not scan through the mass range and thus no information is lost when recording a mass spectrum. As well as parallel detection, a well-designed TOF spectrometer has extremely high transmission even when operating at high mass resolution. In the TRIFT III analyzer, approximately 80-90% of organic molecular ions entering the mass spectrometer are recorded at the detector.

Since the basis of the TOF-SIMS mass measurement is the accurate measurement of secondary ion flight times from start to finish, it is clear that there must be a start time. This is achieved by pulsing the primary ion beam. This is very different to quadrupole and magnetic sector SIMS instruments in which the primary beam(s) operate in a continuous (d.c.) mode. As well as pulsing, if the primary beam is also focused and scanned over the sample surface, the output of the secondary ion detector may be synchronized with the primary beam raster to produce SIMS images (and also secondary electron images). Owing to the efficient use of secondary ion signal via parallel detection and high transmission, mass spectra and SIMS images can be recorded during the static SIMS regime, and thus the technique is unique as a surface organic microprobe.

During each analytical 'cycle', there are relatively long periods when the primary beam is off and the secondary ions are traveling around to the detector. For insulator analysis, it is possible to collapse the extraction field to zero during this time and inject low energy (20eV) electrons to perform charge neutralization. Thus, unlike continuous primary beam SIMS instruments, insulator analysis in TOF-SIMS is relatively trivial.

Although most efficient in the static SIMS regime, SIMS may be acquired at depth also using a second, continuous beam sputtering phase to advance the analyzed surface into the material. Depth profiling is thus readily achievable with TOF-SIMS, although not with the same efficiency as the pulsed-only experiment in which (almost) all ions emitted are passed through the analyzer and detected.
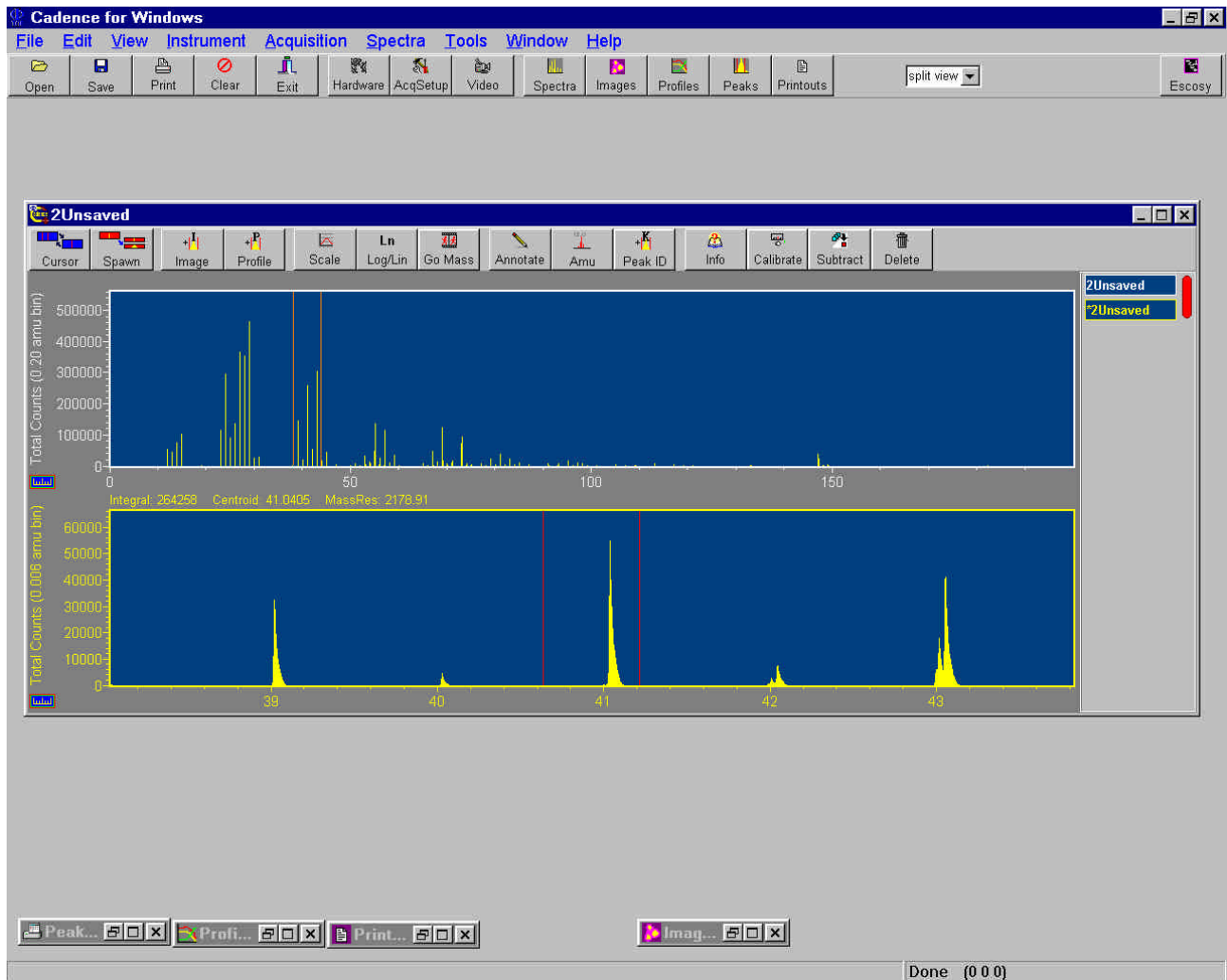
Figure 1-2.   An Example of a TOF-SIMS Mass Spectrum.

Brief Introduction to the System

The PHI TRIFT III (<u>TRI</u>ple <u>F</u>ocusing <u>T</u>ime-of-Flight) is a high transmission, parallel detection instrument that been designed to make optimum use of *all* the secondary ion species leaving the sample surface, and can thus be used for both organic surface characterization and elemental analysis. The instrument has a unique and patented time-of-flight analyzer which has the highest angular and energy acceptance of all commercially available TOF-SIMS instruments. The analyzer continually re-focuses the secondary ions within the spectrometer, and this permits a number of powerful experiments to be performed with the secondary ion beam. The primary ion beams are also designed with state-of-the-art technology to permit imaging at high lateral resolution and with high mass resolution, and are under computer control. A variety of instrumental settings for different analytical needs are simply re-called at the loading of a single file. The software package, WinCadence, has been developed and evolved with the extensive interaction of TOF-SIMS analysts to make operation of the instrument simple and effective.

Considering the sophistication of the TRIFT mass spectrometer's secondary ion optics, the mass spectrometer is extremely simple to use, and is almost never adjusted once the instrument has been installed. The mass spectrometer has been set during installation to operate at high performance when passing secondary ions which have an energy of ~3000eV. Thus, the only parameter which the analyst may need to adjust (in the case of insulating samples) is the sample potential, which effectively matches the energy of the secondary ions to the mass spectrometer. An angular filter and an energy filter are available, and are easily moved into place if required. For most analyses, the spectrometer is operated "fully open", i.e. with no filters in place.

With regard to the primary ion beams, the liquid metal ion gun (LMIG) is an extremely powerful and versatile tool, which can operate over 3 orders of magnitude of primary beam current, from 20nA down to 20pA. The gun is typically operated either at 15kV for optimization of high mass resolution spectroscopy (9000 FWHM) simultaneous with a 2μm imaging capability, or at 25kV for small probe sizes (0.5μm down to ~100nm) with low to medium mass resolution (up to ~5000 FWHM). The high current mode (20nA) is used primarily for spectroscopy, while the low current modes ($\leq$ 60pA) are used exclusively for small probe applications. The broader beam Cs gun is typically used either for spectroscopy at 10kV, or as a low energy ($\leq$ 1keV) beam for optimization of TOF-SIMS depth profiling. The electron gun used for low energy ($\leq$ 20eV) electron charge compensation of insulators is focused into a static probe of ~3mm diameter. Operation is automatic and self stabilizing.

Data Acquisition Modes

Mass Spectra

Generation of a mass spectrum (intensity vs. mass-to-charge ratio) from a sample surface is at the heart of the TOF-SIMS technique. Mass spectral data can be acquired in either positive or negative SIMS (but not both simultaneously), and can be acquired from all vacuum compatible materials, including insulators. Volatile samples may be analyzed via the cold stage option. Mass spectral fragments from mass 1 (H) to many thousands of mass units (e.g. polymeric oligomer distributions) can be recorded at the detector. Mass spectral acquisition occurs using either the Cs or LMIG gun to provide the primary ion beam, and a variety of beam energies from ~5-10kV (Cs) and ~5-25kV (LMIG) are available. Mass spectral analysis may or may not involve simultaneous imaging of the sample surface.

Imaging

When lateral distributions of surface species are required, the LMIG is the ion gun of choice, since it provides sub-micrometer probes. A variety of primary currents from 20nA to 20pA are available, and, as with any charged beam technique, there is a trade off between probe size and primary beam current (equivalent to data rate). At the highest available beam energy (25kV), short primary beam pulses (high mass resolution) are achieved by *reducing* the primary pulse width in software. This preserves the primary beam probe size, but reduces the secondary ion count rates. To obtain high mass resolution without loss of secondary ion count rates, the LMIG is operated at 15kV and the primary beam is "bunched" (electrodynamically compressed). One consequence of this method of maintaining a short (sub-nanosecond) pulse without chopping the beam is that the probe size increases significantly. The TRIFT III LMIG provides a 2-3µm probe simultaneous with a sub-nanosecond, bunched pulse.

Region of Interest (ROI) Analysis

The primary beam raster is under full computer control, and it is possible to extract mass spectral information from any 'region(s)-of-interest' visible in an image. The system can acquire full images and selectively port out mass spectral data when the ion beam is in the user-defined 'region-of-interest'. Region-of-interest data can also be acquired retrospectively (see Retrospective Analysis: Raw Data Files below).

Depth Profiles

In a pulsed-only mode of operation, the 'duty cycle' (time on: time off) of the primary beam is such that the analysis typically occurs in the 'static' SIMS regime. If the primary current is increased to ≥20nA, the repetition rate in increased to >25kHz, and the primary beam raster reduced, it is possible to sample to depths of ~100A in acquisition times of 20-30 minutes with a pulsed beam. For greater depths, it is necessary to invoke a separate d.c. sputtering phase (with no mass measurement) in order to expose new material at depth. This can be achieved with a single gun system, or with a dual beam system. If a d.c. phase is used, then the sensitivity falls, since most of the material removed is not analyzed. Such TOF-SIMS profiles are useful, but the inherent sensitivity of the technique is sacrificed to make such measurements.

Retrospective Analysis: Raw Data Files

Because of the parallel detection capabilities of the technique, it is possible during data acquisition to record (store) the x and y position, the time-of-flight (mass) and appearance time (within the acquisition) of every secondary ion recorded at the detector. Such files can be extremely large, but this facility makes it possible to analyze data retrospectively. This can be invaluable (for instance) if the analyst is analyzing an unknown sample, conducting failure analysis or analyzing a unique and limited amount of material. In such cases it is often difficult at the beginning of the analysis to anticipate what species are important to monitor and therefore which images will prove to be most informative. In retrospective analysis of 'raw' files, mass spectral data can be reconstructed from 'regions-of-interest' visible in an image, or images can be re-created from any peak in the mass spectrum. Depth profiles may also be acquired in this mode.

How to Reach PHI Customer Service

If any PHI manufactured or supported controls or equipment fail or other problem-solving is called for, contact PHI Customer Service as follows:

By mail:

Physical Electronics, Inc.

PHI Customer Service, M/S G11

6509 Flying Cloud Drive

Eden Prairie, MN 55344

USA

ULVAC-PHI, Inc.

ULVAC-PHI Customer Service Organization

370, Enzo, Chigasaki

253-0084 Japan

By e-mail:

service@phi.com	(USA)
ykitamura@phi.com	(Japan - Domestic)
jgorgol@phi.com	(Japan - International)

By telephone or fax:

Region	Telephone	Fax
U.S.	1-800-922-4744	1-952-828-6325
Outside U.S.	1-952-828-5831	1-952-828-6325
Japan	81-467-85-6522	81-467-85-4411
Europe	49-89-96275-0	49-89-96275-50

Section 2:
Mounting & Introducing Samples

This section of the operator's guide discusses the process of mounting samples on the sample carriers, then steps through the process of introducing the mounted samples into the analysis chamber using the sample introduction system.

Cleanliness of the Sample Preparation and Mounting Area

When mounting samples for TOF-SIMS analysis, <u>always</u> maintain a clean preparation area: remember the technique is surface sensitive. The following steps will help in maintaining clean preparation surfaces when mounting samples.

1. Cut a square of aluminum foil and tape the edges to a flat preparation bench or preferably to a small square of thick window glass. The surfaces of most commercially available Aluminum foil are extremely clean. Replace the foil surface based on usage.

2. Obtain a supply of lens tissue paper, such as SPI lens tissue from Structure Probe, Inc., P.O.Box 656, Westchester, Pennsylvania, PA 19381-0656. (www.2spi.com). Use fresh sheets of this lens tissue each and every time new samples are prepared and mounted. Again, a hard and flat surface such as a square of thick window glass is ideal as a substrate for this operation.

Cleaning Sample Holders and Accessories

Sample holders and accessories (including tweezers, scissors, hemostats, etc.) should be cleaned regularly. This prevents the spread of organic compounds from one sample to another. The recommended sequence for cleaning is as follows:

1. Ultrasonic rinse in methylene chloride;

2. Ultrasonic rinse in hexane;

3. Ultrasonic rinse in acetone; and

4. Ultrasonic rinse in methanol.

If holders come into contact with samples having liquid fluorocarbons such as 'Krytox' or 'Fomblin' oils on their surfaces, the holders and accessories should be rinsed thoroughly with trifluoroethylene <u>prior</u> to performing the standard cleaning process outlined above.

After analyzing samples that contain surface silicones, it is advisable to repeat the hexane rinsing two or three times to insure removal of the silicones that will inevitably have transferred to the sample holders.

Store clean sample holders to minimize the risk of contamination. Most plastic products (sample drawers, sample boxes) have been molded with the aid of a mold-release agent such as polydimethyl siloxane. Clean storage is assured in clean petri dishes within a drying oven, or in boxes or drawers lined with Aluminum foil.

Always remember that TOF-SIMS will detect compounds on sample surfaces that are only a few monolayers thick. The user should always maintain scrupulously clean preparation methods, so that detection of compounds can, in confidence, be linked to sample chemistry only.

Back Mounting for Fixed Z Position

The PHI TRIFT III TOF-SIMS sample stage has extensive x and y movement, but no z movement. This simplifies the subsequent tuning process, for reasons that will be discussed later.

The stage's z position is where the primary ion beams, the electron beam and the axis of the TOF-SIMS optics all intersect. Ultimately, when mounting samples, the sample surface to be analyzed should be at that optimum z position. This is achieved by "back mounting" samples within a sample carrier (50 mm systems and small sample holders in 200 and 300mm systems), or by clamping the sample against a 150 mm, 200 mm or 300mm sample diameter holder (200 mm and 300mm systems). The carriers have been designed so that sample surfaces to be analyzed will be at the correct z position when the sample carrier is within the analytical chamber.

Quadrant Sample Carrier and Sample Holders

In the 150mm, 200mm and 300mm TRIFT systems, a quadrant sample carrier is used to position small sample holders (up to four) within the TRIFT analytical chamber. The quadrant carrier is, however, different for each system.

The full sample mounting kit is comprised of:

- Carrier and holders
  - A sample carrier with four sample holder positions, arranged as quadrants
  - Sample holders, 25mm diameter, with 13mm circular openings

- Sample holder inserts
  - Recessed sample 'pucks', with a variety of recess depths
  - Masks with different windows to permit mounting of small samples
  - Coarse mesh grids
  - Cross-section holder
  - Aluminum backing plates
  - Be-Cu springs
  - Be-Cu sample clips.

Place the circular sample holder face down on a clean surface, as described earlier in this section. Assume, for example, that the current sample is a single, thin and flat sample. Place the surface to be analyzed face down over the open window in the sample holder. Provided that the preparation surface is flat, the sample surface will not touch it, as it will be separated by the thickness of the window in the sample holder. Choose a window size to suit the area over which analysis is to be performed. Next, lower one of the aluminum backing disks onto the rear of the sample. Using tweezers, compress a beryllium-copper sample clip and lower it inside the sample holder until the three legs of the clip lightly compress the backing disk. Release the compression and leave the clip in place within the sample holder.

If mounting more than one sample in a single holder, select one of the multiple window masks as appropriate and insert into a sample holder. Load the samples as before, and place the small Cu-Be springs on each sample. Lower an aluminum backing plate into the holder, taking care not to displace any sample sideways. The disk should now be resting on a symmetrical arrangement of conical springs. Finally, compress a sample clip as before and lower into the holder until the backing disk is under load and each sample is held in place by compression from its own conical spring. Note that the use of the conical springs is essential if samples of different thickness' are to be mounted together and retained in place.

With the sample carrier face down on the preparation area, lower each sample holder into the desired quadrant of the carrier. Using the caphead allen screws provided, lightly grip the holder within the carrier. Turn each screw gently until no further movement is possible, and then turn approximately one sixteenth of a turn at the most. <u>Do not overtighten</u>.

For information on when it may be appropriate to use stainless steel grid meshes, refer to Section 8 "Charge Neutralization of Insulators".

For information about how to prepare and mount difficult geometry samples, refer to Section 9 "Sample Preparation Techniques".

Large, Single Window Sample Carrier (50 mm TRIFT Systems)

For large area samples, a separate sample carrier is provided which allows a maximum sample size of 25 mm x 25 mm x 5mm.

This sample mounting kit consists of:
−   Sample carrier;
−   A variety of front plates to allow different sample sizes to be mounted;
−   Four tapered screws for affixing the front plates to the carrier; and
−   Five beryllium-copper sample clips.

Place the sample carrier (with the front plate removed) onto the clean preparation area. Place the sample to be analyzed face up within the carrier. Next, select a cover plate with the most appropriate window size and place onto the carrier. Using the tapered screws provided, lightly grip the cover plate to the carrier. Do not overtighten. Invert the carrier assembly as carefully as possible. With the carrier face down, move the sample to the desired position and use as many sample clips as is necessary to hold the sample firmly in position.

300 mm, 200 mm and 150 mm Sample Carriers (300 mm and 200 mm TRIFT Systems)

There are different sample carrier types for the 300 mm and 200 mm systems: carriers that hold 150 mm diameter (6 inch) samples; carriers that hold 200 mm diameter (8 inch) samples; carriers that hold 300 mm (12 inch) samples; and a small sample holder which has four (quadrant) positions and receives the small sample holders used in the 50 mm TRIFT III systems. These first three carrier types have been designed primarily to carry silicon wafers, although the carriers have also been designed to be versatile and can be configured to hold other large samples such as hard disks, for instance. Sample mounting is relatively simple. 300 mm and 200 mm system sample introduction assemblies can be rotated 90 degrees so that the large sample carriers are held horizontally while the sample is being loaded onto the carrier. Once mounted, the introduction rod is rotated 90 degrees back to the vertical position prior to sliding the introduction chamber door forward ready for pump down. The quadrant, small sample carrier for the 300 mm and 200 mm systems does not require such rotation.

Load Lock and Sample Carrier Introduction/Removal - (All Systems)

To load a sample carrier into the load lock, first check visually that there is no carrier already within the sample chamber.

Perform the following steps in the System Vacuum Control's (SVC) Watcher software.

1. It is good practice (although not strictly necessary) to ensure that the spectrometer gate valve is closed (V5-Spectro Gate).

2. Select the "Backfill Intro" button.

3. There will be a few seconds delay, after which the introduction gate valve will close, and then a pneumatic valve will open and vent gas will enter the load lock. Always use a 'dry' vent gas such as nitrogen or argon. Observe that the convectron gauge reading in the load lock (the central reading on the Granville-Phillips controller display, or "Intro Convectron" in Vacuum Watcher) rises until ~760 Torr , signifying that the load lock is now at atmospheric pressure.. Note that if the turbo pump is brought up to atmospheric pressure too quickly, the overall lifetime of the intro turbo pump may be reduced. The intro chamber pressure should rise to near atmospheric pressure in a *minimum* of 30 seconds. If the chamber vents too quickly, please consult PHI customer service.

4. It is not necessary or advisable to turn the introduction turbo pump off at the controller prior to venting the load lock.

Sample Carriers for 50mm Systems

1. Screw the sample carrier handle into the sample carrier and transport the carrier over to the load lock. Lift the lid of the load lock and lower the carrier inside. Using the large circular handle (or the magnet in the case of magnetic rod systems) on the external end of the sample introduction rod, rotate the introduction rod clockwise (CW) and screw the threaded end into the side of the sample carrier. Do not overtighten, but rather rotate clockwise (CW) until no further rotation is possible and then 'back-off' one quarter of a turn counterclockwise (CCW).

2. Remove the sample carrier handle by unscrewing counterclockwise (CCW). Before closing the load lock lid, insure that the o-ring surface is free of dust and/or particulate. Remove the o-ring and wipe with a methanol-soaked tissue, if necessary. Reinstall the o-ring and close the load lock lid. Select "Pump Intro Chamber" in the Vacuum Watcher control software. The backing line (rotary) pump will now begin to evacuate the load lock, and the convectron gauge reading will begin to fall, until it is offscale low, displaying 1.0×10^{-4}. No ion gauge exists within the load lock, but the Vacuum Watcher software gives feedback to determine the length of time required to

leave samples pumping within the load lock prior to sample introduction. The series of statements is as follows: "waiting for intro turbo to reach speed with good roughing," "waiting for good pressure in load lock," "task pump intro complete." To ensure a good vacuum level, the load lock can be left pumping for another 10 minutes.

Before attempting sample introduction, check that the sample stage is in the "Exchange Position". If not, manipulate the stage until it is. In WinCadence, under Instrument/Stage Control, select "Exchange Position" (button in the center of the window).

3. To open the introduction gate valve, select "Transfer Sample" in the Vacuum Watcher. If the analytical chamber pressure, measured by an ion gauge and displayed in the upper section of the Granville-Phillips controller (or the "Chamber Ion Gauge" in Vacuum Watcher), rises above 2×10^{-6} and remains there, close the introduction gate valve. The gas load inside the load lock is too high and more pumping is required. To close the intro gate valve, simply push the intro rod part of the way in, then retract it all the way out and the gate valve will automatically shut.

    When the introduction gate valve can be opened without the chamber pressure rising and remaining in the low 10^{-6} range, the sample carrier can be introduced into the chamber.

4. With the introduction gate valve open, push on the introduction rod handle and slide the carrier a few centimeters towards the analytical chamber. Immediately, a switch is activated, and the sample stage voltage (if it is on) will begin to drop. Continue sliding the carrier forward. An internal guide system will prevent misalignment, and will guide the carrier onto the sample stage.

5. When the introduction rod is as far forward as possible, it should be possible to see that the carrier is engaged within the sample stage, by viewing through one of the external viewports.

6. Now begin turning the sample introduction rod handle counterclockwise (CCW). Eventually the rod will disengage from the thread of the carrier, allowing the sample introduction rod to be retracted without withdrawing the sample carrier. Just before you finally withdraw the introduction rod, use it to push gently on the carrier to ensure that it is as far onto the stage as it can go. There is an Allen screw stop which the carrier should touch. By doing this, you can ensure a repeatable correlation between stage co-ordinates and position on the carrier. As the rod is withdrawn, check through a viewport or on the optical viewing screen, to ensure that the carrier is not being withdrawn also. Withdraw the introduction rod until the magnetic handle passes over the magnetic switch. The introduction gate valve will automatically close.

7. Sample carrier removal:

a. When removing a sample carrier from the analytical chamber, check that the stage is in the "Exchange Position," and select "transfer sample" in the Watcher software. The load lock gate valve is interlocked to prevent accidental loss of vacuum in the analytical chamber. If for any reason the load lock is **not** under vacuum when the transfer button is pushed, the gate valve will not open and no harm will come to the analytical chamber. If the interlock condition has been met, the load lock gate valve will open when "Transfer Sample" is selected.

> **Transfer Sample**

b. Slide the introduction rod forward slowly, as before, until the threaded end of the rod can be felt pushing up against the threaded hole in the sample carrier. If there is any doubt, gently rotate the introduction rod counterclockwise (CCW) and maintain a slight forward pressure. The user should feel the rod 'click' once every 360 degrees, as the thread of the rod passes over the thread in the sample carrier.

c. After confirming the alignment of the rod with the sample carrier, rotate the introduction rod clockwise (CW), maintaining a slight forward pressure to engage the thread. Begin turning the introduction rod handle (or magnet) clockwise (CW). When no more rotation is possible and the rod is fully engaged, 'back-off' at least one quarter of a turn counterclockwise (CCW). It should now be possible to withdraw the introduction rod and sample carrier into the load lock. When the introduction rod is fully retracted, the introduction gate valve closes automatically. Vent the load lock by selecting "Backfill Intro" in Vacuum Watcher.

> **Backfill Intro**

Sample carriers for 200mm and 300mm systems

1. Once the load lock is vented to atmospheric pressure, you may slide back the intro lock door on its guide rail. To do this, depress the spring-loaded lever arm located near the guide rail which releases a mechanical locking mechanism. Slide the assembly back, and depress the lever arm once again to lock the assembly in place at the furthest point of travel.

2. Select the appropriate sample carrier, and insure that the spring loaded "latch" (which holds the carrier in place within the main chamber) is *perpendicular* (90°) to the plane of the sample carrier. Slide the carrier into place on the intro rod, gently matching the holes in the carrier with the prongs on the rod. With a gentle but firm action, push forward on the coupling magnet and twist the magnet 90 degrees <u>counterclockwise</u> (CCW) so that the latch swings from a horizontal position to a vertical one, now parallel to the plane of the sample carrier. The rationale for this is as follows: when the sample carrier is transferred into the analytical chamber, these steps will be reversed so that the latch will rotate clockwise (CW) and the carrier will be in place on the sample stage.

3. A "flat" exists on the coupling magnet. It can be used to aid this process to help load and unload with confidence. The magnet flat should be upwards with respect to the instrument when the carrier is locked in place on the rod. The magnet flat should be at a "3 o'clock" position when the carrier is loaded.

4. Once the carrier is in place on the intro rod, raise a second lever, located at the intro door above the intro rod, and rotate the intro rod and carrier so that the face of the sample carrier is horizontal. Note that this only applies to the 150mm, 200mm and 300mm sample carriers. Allow the lever to lock into place. Load the sample by sliding back the single, extendible sample grip, placing the sample onto the carrier in the correct orientation (flat or notch on the wafer against flat or notch on the carrier), and allowing the grip to retract gently.

5. Lift the rotary locking lever once again and rotate the whole assembly counterclockwise (CCW) so that carrier and wafer are now vertical.

6. Depress the guide rail locking lever and slide the intro door assembly forward until the door adjoins the o-ring seal in the opposite face of the intro chamber.

7. Within the watcher software, select "pumpdown". See previous notes for details about pump down.

8. When the intro chamber has pumped sufficiently, open the intro gate valve by pressing selecting the "Transfer Sample" button within the Watcher software and then sliding the sample intro rod forward using the coupling magnet. Maintain the rotational position of the magnet. The magnet flat should be horizontal and on top. Until the action of "latching" the sample carrier in place on the stage becomes a routine task, proceed carefully during the next step. Bring the magnet as far forward as it will go with gentle pressure. The magnet should be ~5-10 mm away from a locking ring around the intro rod tube to prevent pushing too far. If more than ~10 mm away from this ring, stop. Check the carrier position by going around to the other side of the instrument to look through the viewport before proceeding.

9. With a firm action, push the coupling magnet forward to the locking ring _and then_ rotate the magnet 90 degrees clockwise (CW). Try to visualize what is happening inside the system: the vertical latch on the sample carrier is rotating in place onto the stage to grip the carrier in place. If any mechanical abrasion of any sort is felt during this procedure, do not force the action. With the magnet flat now on its side and at a "3 o'clock" position, use your finger tips only and gently slide the coupling magnet back along the intro rod tube. The carrier will remain in place on the stage while the intro rod slides back into the intro chamber.

10. Once the magnet is fully retracted, the intro gate valve is closed automatically. The analytical chamber vacuum should improve more rapidly.

11. To remove the sample carrier, reverse the procedure described above. The coupling magnet will be sliding forward with its "flat" in a vertical orientation and at a "3 o'clock" position. Gently engage the intro rod onto the sample carrier, and then firmly but gently _first_ push forward _and then_ rotate 90 degrees counterclockwise (CCW) to bring the sample carrier latch vertical. This will release the carrier from the stage and allow for sliding the magnet back, pulling the carrier into the intro chamber. Sample introduction and removal will become routinely fast and easy.

Digital Scanner Option

Improved stage navigation capability is available with the digital scanner option. The entire sample holder can be scanned and the image saved to disk. The digital sample holder image can then be used to drive the sample stage to specific areas of interest. In addition, the current location of the stage is always indicated on the digital image of the sample holder even if the joystick is being used to move around. This feature has been implemented in the *Stage Control* Window. The following procedure describes how to scan the sample holder and then use the image to drive the sample stage.

1. Place the sample holder containing a set of samples onto the special plastic plate sitting on the scanner bed.

2. Open the Stage Control window under the Instrument pull down menu. The set of icons across the top of the right half of the window are the functions available. To begin, click on the arrow between the first and second icon and select 'configure'. This will open two new windows that allow the sample holder to be scanned. One is a Preview Window and the other is the standard Epson Twain Pro window.
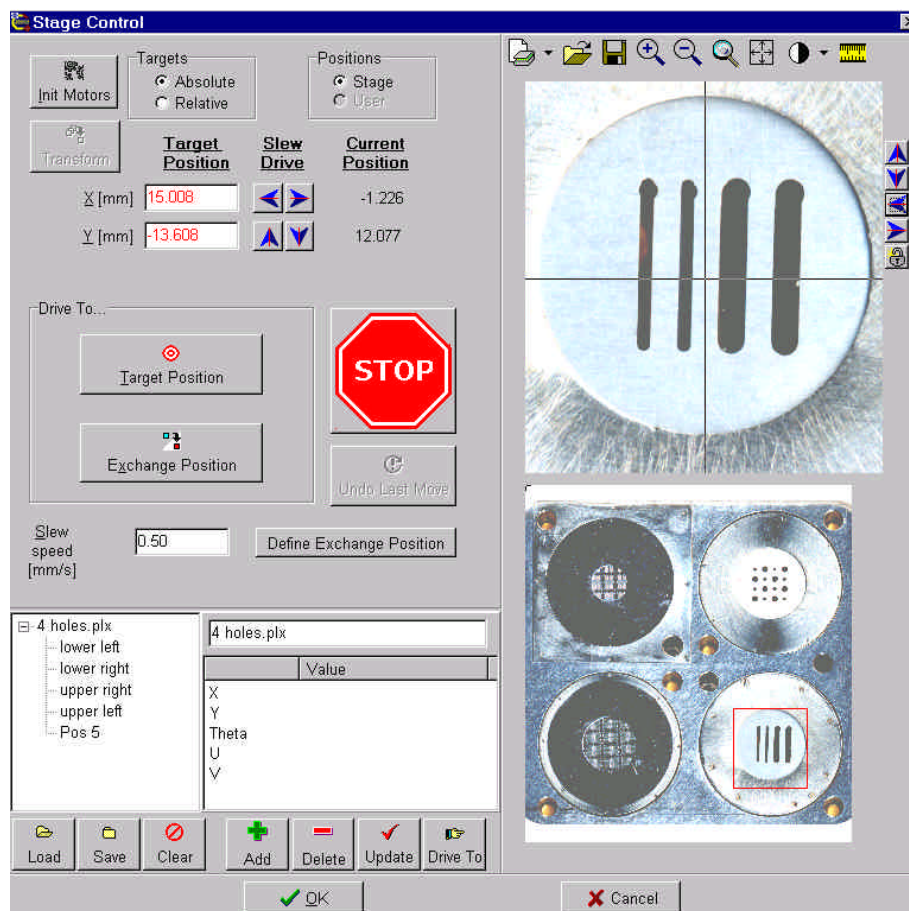
Figure 2-1.   Example of the Stage Control window using scanned images.

3. By selecting the preview button, a low-resolution image is scanned for setup. Select an area to be scanned by clicking a dragging a rectangle and select the desired resolution (e.g. 1200 dpi).

4. Click on the Scan button. A high resolution scan will be done and the resulting image will be automatically placed in the Stage Control Menu as shown in Figure 2-1 above.

5. The image on top is intended to show a magnified view of the area outlined in the red square in the image on the bottom. The magnification can be adjusted by left click-and-drag with the mouse on the top image. The red square on the bottom image will be adjusted immediately. The 'magnify' and 'de-magnify' icons can also be used to change the magnification.

6. Left clicking with the mouse on the red square and dragging it around on the bottom image can be used to explore the sample holder. This will cause the magnified view to change in real-time. The up, down, left, and right arrows to the right of the top image can be used to fine tune the position of the cross hairs. The Target Position X (mm) and Y(mm) values are continuously updated to reflect the location of the cross-hair in the top image.

7. Once the desired location is centered on the cross hairs in the top image, hit the Target Position button and the stage will drive to that location.

Finding the Analysis Position

The analytical chamber may need to be illuminated to view the sample holder inside. If an instrument file (*.ins) is not loaded, the chamber will be dark. To illuminate the chamber, in WinCadence, go to Hardware Menu/Instrument tab, and fill in a value of 10 for Illuminator Power. Note the sample holder can be viewed at two different magnifications. In the SVC's *Watcher* software program, the Camera Field option can be set to small or large.

Moving to the analysis position can be done in several ways. The stage can be moved directly using the joystick. Or, in WinCadence, go to Instrument/Stage Control. A designated position can be entered in the X(mm) and Y(mm) values; then, when "Target Position," is selected, the stage will automatically drive to this position. Also, positions from memory can be called up (load file *.plx). Highlight the position desired, then select "Drive To."

TRIFT systems running the "ESCOSY" wafer navigation software can also move the stage into the exchange position from within ESCOSY.

Typical Procedures in ESCOSY (200mm, 300mm Systems)

The ESCOSY software is a separate software application apart from the WinCadence software application, however, the two applications are heavily integrated. ESCOSY allows control of the TRIFT system's 8 inch sample stage. The following section is an overview of the commonly used features of ESCOSY.

Stage Directions

X,Y, Rotation Orientation

- The point of view is looking at the sample from the SPECTROMETER.

- + X direction is towards the INTRO

- + Y direction is UP

- + Rotation direction is clockwise

- The ROTATION ANGLE is assumed to be 0 at the EXCHANGE POSITION.

Stage Movements

- + X movement will cause the stage to move AWAY from the INTRO

- + Y movement will cause the stage to move DOWN

- + R movement will cause the stage to move COUNTER CLOCKWISE

Methods of Moving the Stage

Command Window

The Command Window allows the operator to control stage movement by sending single commands to the motor control. Commands are entered in the Command text box. To repeat the last command, choose the "Repetition" button besides the Command text box.

Table 2-1.   *Escosy commands and Corresponding Stage Movements.*

Commands:	Resulting Stage Movement:
-5.5x	moves the stage to the ABSOLUTE x coordinate -5.5mm
2.5X	moves the stage over 2.5mm in the positive x direction RELATIVE to the current position
e30r	does a eucentric rotation to the ABSOLUTE R coordinate 30°
e-30R	does a eucentric rotation of -30° RELATIVE to the current R coordinate position
1b	does a backlash correction in the X-axis
2b	does a backlash correction in the Y-axis
4b	does a backlash correction in the R-axis
x	drives the stage to 0.0 in the X-axis
y	drives the stage to 0.0 in the Y-axis
r	drives the stage to 0.0 in the R-axis
15g	moves the stage to position no. 15 in the last opened position list
i15g	moves the stage to position ID 15

Automated Drive

At the bottom of the Command Window is a pulldown menu of stage locations, including "Origin", "Exchange", "Notch" and "4 POS ALIGN MARK". To automatically drive to any of these locations, choose the desired location from the pulldown menu, then press the "Drive" button.

Joy Stick Control

The joystick can be used to move the stage for fine adjustment within a local area. However, when using the joystick, be careful not to run out of bounds. If it is necessary to move large distances, it is better to use the mouse to define the desired location on the wafer map.

Addressing Modes on the Wafer Map

There are 6 different addressing modes available in the wafer map window, depending on the lasso status and the usage of the "Shift" key. If the target position is within the allowed X-Y travel, point to the target and click the right mouse key. To reach positions within the forbidden zone, you must use stage rotation. This can be done automatically by using the "Shift" key and clicking the desired location in the forbidden zone with the right mouse key.

Table 2-2.   *Operation of the Mouse and Corresponding Modes and Stage Movements.*

Operation	Lasso Mode	Resulting Stage Movement
right mouse	Lasso off "X"	Move to exactly the mouse pointer position
right mouse	Lasso on	Move to the coordinate inside the selected chip, which has been defined by setting the "Chip Catch Offset".
right mouse	Lasso w/ dot	Move to the defined position nearest the mouse pointer position.

If "Shift" right mouse key is used, the above 3 modes can be performed on a position within the forbidden zone.

Addressing from the Position List

By highlighting one of the positions in the position list, then clicking with the right mouse key, a menu is displayed. One of the options in the menu is "scan". By selecting "scan", the stage will drive to that position.

Pre-Alignment

The purpose of pre-alignment is to calculate the rotation angle and offset vector between the XY and the UV coordinate system. Whenever you change to a new wafer map with a different rotation angle, you must drive to the alignment position (e.g. 4-pos alignment) and execute the pre-alignment routine.

Loading a Project

Each of the 200 mm TRIFT III sample holders has an associated "project" which can be loaded in Escosy. Project files contain data concerning screen configuration, software limits, and associated position lists and wafer maps. To get the wafer map to align with the actual sample holder, one has to do a pre-alignment after loading the project. For the 3 different sample holders use the following steps:

4-position holder:

1. Load the 4-position project.

2. Drive to the 4-position alignment mark.

3. Click on the wafer map window.

4. Under Edit choose "prealign".

5. Click on OK.

12-position holder:

1. Load the 12-position project.

2. Drive to the 12-position alignment mark.

3. Click on the wafer map window.

4. Under Edit choose "prealign".

5. Click on OK.

8" Wafer holder

1. Load the 8-inch wafer project.

2. Drive to the notch position.

3. Click on the wafer map window.

4. Under Edit choose "prealign".

5. Click on OK.

Position Lists

Each PROJECT file contains a position list of the locations of each of the sample analysis areas. A position list is defined and stored as an ASCII data file with an extension ".PLS". To drive to one of the sample positions using the position list, select the position of interest and choose SCAN ACTUAL. The operator can also click on a position line in the list with the right mouse key and chose SCAN.

Many different position lists can be saved on disk for different auto-analysis routines. The number of analysis points and the pattern of analysis points can be varied and saved as separate position lists. To load a new position list choose *Open Positionlist* under the File menu.

Wafer Maps

A wafer map layout file is used to define a specific sample holder layout. These are ASCII files with an extension ".WLO". Each position list will have a corresponding wafer map associated with it.

Once a position list has been read from disk, the corresponding wafer map can be brought to the desktop by clicking on the 'wafer map' icon in the top toolbar. The defined positions can be identified on the wafer map (red dots) by clicking the appropriate icon in the toolbar at the top of the Escosy window.

Coordinate Window

This window provides the X,Y,R,U,V values for the current position of the stage.

Adjust Three Points

This routine is performed when the prealign is not accurate enough. When doing defect analysis, one performs the adjust three point routine on three defects. Some defect review systems (Tencor, KLA) provide alignment mark coordinates which will be automatically transferred to the related position list during the import routine.

Open the Adjust 3 Points window.

1. Enter the UV coordinates of the first position either digitally or by setting flag #1 within the wafer map or the defect map.

2. Choose the **GO** button referring to label #1. The stage will go to the specified UV coordinates.

3. Use the joystick to move the stage exactly to the position of label #1 and execute backlash for the X and Y axes. This position may either be a defect or a die corner.

4. Choose the **READ** button referring to label #1 to display the current stage (XY) coordinates.

5. Repeat the above steps for label #2 and #3.

6. Choose the **ADJUST** button to calculate and store the parameters.

Importing KLA Coordinates and Finding Defects

The Data Import module allows conversion of defect coordinate lists from external defect inspection systems into standard ESCOSY positionlists. If available, the import filter also creates the corresponding wafer layout.

Escosy Plus supports the following:

- Tencor SFS-6000 and 7000 series;

- KLA 21xx series;

- INSPEX TPC 8500;

- DefectFinder 2000.

Refer to the unabridged Escosy manual (pp. 8-32 to 8-48) for a description of how to import these specific file formats.

After importing a defect position list and corresponding wafer map, use the alignment marks which come with the file to do a 3 point alignment.

Stage Initialization

1. Within WinCadence, select "Instrument" pulldown menu.

2. Select "Stage Drive".

3. *>stage requires init.*

4. Press OK.

5. Select INIT MOTORS.

6. *>init stage.*

7. Select YES.

8. *>Turn motors off & on.*

9. Go the back of the Motor Chassis and turn power off and back on.

10. Select OK.

11. All three axis will move to preset positions, then search for HOME.

12. Press OK.

Align Exchange Position

ESCOSY assumes that the ROTATION ANGLE at the exchange position is 0.

The actual ROTATION ANGLE will not end up at 0, therefore, ESCOSY provides an OFFSET ANGLE to account for this difference.

Install the 4 position sample holder in system.

1. Click COMMAND WINDOW to assure all pulldown menus appear.

2. Choose PROJECT.

3. Choose OPEN.

4. Open the 4 POSITION PROJECT.

5. Select MODULE STATUS from the menu bar.

6. Select COMPU MOTOR CONTROL form the list.

7. Click EDIT VARIABLES.

8. Select OFFSET R form the list.

9. Click EDIT.

10. Enter 0 and click OK.

11. Click OK.

12. Click OK.

13. Exit ESCOSY and reenter to assure the change takes effect.

14. Using the COMMAND SHELL manually move the stage to the exchange (load) position making sure to use a ROTATION value between -180 and 180 degrees.

15. Multiply the ROTATION value in the COORDINATE WINDOW by 2000 to determine the ROTATION OFFSET.

16. Select the MODULE STATUS from the menu bar.

17. Select COMPU MOTOR CONTROL from the list.

18. Click EDIT VARIABLES.

19. Select OFFSET R from the list.

20. Click EDIT.

21. Enter ROTATION OFFSET and click OK.

22. Click OK.

23. Click OK.

24. Exit ESCOSY and Reenter to assure the change takes effect.

25. Make sure the ROTATION VALUE in the COORDINATE WINDOW should now read 0.00.

26. Select EXCHANGE from the COMMAND WINDOW pull down list.

27. Click EDIT.

28. Manually enter the X,Y,R values or Click READ to read the values from the COORDINATE WINDOW.

Align the 4-Position Marker

1. Select the 4 POS ALIGN MARK from the COMMAND WINDOW pull down list.

2. Click DRIVE.

3. Sample will rotate 180 degrees.

4. Sample will move to the ALIGN MARK machined in the sample holder (+)

5. Using the COMMAND SHEL4L BOX move the stage in X and Y to align the center of the MARK to the viewing optics

6. Select 4 POS ALIGN MARK from the COMMAND WINDOW pull down list.

7. Click EDIT.

8. Manually enter the X,Y,R values or Click READ to read values from COORDINATE WINDOW.

Note: The location of this alignment mark is also used to set the PRE ALIGNMENT values that will allow the 4-POS Position List (set up in U,V coordinates) to be properly oriented to the stage coordinates.

9. Click MODULE STATUS form the pull down menu.

10. Select ADJUST MODULE from the list.

11. Click EDIT VARIABLES.

12. Scroll down the variable list to PREALIGN CENTER X.

13. Click EDIT.

14. Enter 4 POS ALIGN MARK X value from COORDINATE WINDOW.

15. Click OK.

16. Click PREALIGN CENTER Y.

17. Click EDIT.

18. Enter 4 POS ALIGN MARK Y value from COORDINATE WINDOW.

19. Click OK.

20. Click POSITION LIST WINDOW.

21. Save the 4-POS PROJECT.

22. Exit ESCOSY and reenter to assure values are used.

Note: You should now be able to use the 4-POS POSITION LIST and the WAFER MAP to navigate around the 4-Pos Sample Holder.

Note: If the sample height is adjusted you should recheck the position of the ALIGNMENT MARKER and reset it in the 4-POS ALIGN MARK position in the COMMAND WINDOW and also the PREALIGN CENTER X and Y in the ADJUST MODULE in the MODULE STATUSS WINDOW.

Find Rotation Center

The rotation center of the stage is saved as the "origin" in the pulldown menu of stage locations in the Command Window. It should only be necessary for this to be set once and it will be used for all projects. To check, perform the following steps.

1. Drive to the "origin".

2. Click FIND ROTATION CENTER icon.

3. Click START ROTATION.

4. Adjust X-Y position of stage to find the CENTER.

5. Click STOP ROTATION.

6. Click SET ROTATION CENTER.

The new coordinates will be written in the coordinate.

Section 3:
Instrument State Files

This section discusses the "Instrument State" files. These files reside on the system computer and contain all of the software settings, parameters, and information required to setup and control the TRIFT system.

System Setup prior to Analysis

The Concept of Instrument States: "INS" Files

An instrument file, which uses the file extension ".ins", contains all the information required to set-up and control the system in a single file, for both positive and negative SIMS. All the information concerning voltages, the sequencer and calibration files are now contained in a single file, along with information to control the high mass blanker and post-ESA blanker (pre- and post-spectrometer blanking).

The only parts of the system not under instrument file control are: the secondary ion apertures (Contrast diaphragm or "angular filter", and the energy filter); and the primary beam apertures on the LMIG and Cs gun. The Cs and the LMIG guns are slightly different: the Cs column has both a "slit" and an aperture, while the LMIG has a single, mechanically variable aperture (MVA).

NOTE: Computer control of the LMIG MVA is offered as an option on the TRIFT system.

The TRIFT system comes with a set of instrument files prepared at the factory. These files are specific to both the system and to its configuration as well as the analytical modes that the configuration supports. During on-site installation, these same files may need some adjustments. At the end of the TRIFT installation, the system should contain a full set of instrument files. After becoming familiar with routine system operation and tuning, the system user is free to create more instrument files that are optimized for specific analyses.

Inspecting the Library of Instrument State Files

Certain important parameters from the instrument state file are passed through to a file header and appear when a file is highlighted prior to reading in the file. These parameters include primary beam, beam energy etc. A two-line description of the file, entered at the time that the file was created, is also displayed. Thus, the description and the basic properties of the file can be reviewed *prior* to loading it. For instance, an instrument file may have been saved that was specifically tuned to analyze a difficult glass sample for a client,

and now a repeat analysis of the same material is required. Unless the "ins" file had been saved with an obvious filename such as "GLASS1.INS", the "ins" files may need to be searched for the properties: i.e., charge compensation; 15kV energy; Ga beam; bunched; post-acceleration. This will greatly reduce the number of files to be searched before locating the correct one.

Creating New Instrument State Files

After becoming familiar with the routine operation of the system, creation of customized instrument files will be necessary. If any modifications to an existing instrument file are made and the new conditions need to be saved as a new instrument state, simply save the instrument file with an appropriate new filename. Remember, however, that an instrument state file should contain *all* the information required for both SIMS polarities. If the file is saved in one polarity, but the user neglects to switch to the other polarity to create the other information for the file, the "ins" file will not be complete, although it will load correctly in the polarity where the files were created. When creating a new instrument file, use the following checklist to verify that the new file has the following parameters set correctly *in each polarity*:

- Mass calibration, with relevant species;

- Raster size;

- Polarity offset values (- to +);

- Buncher voltage (Mass resolution LMIG or Cs);

- Buncher delay (Mass resolution LMIG or Cs);

- Raster scale factor for correct image calibration (LMIG or Cs);

- High mass blanker calibration (LMIG or Cs);

- Post ESA blanker calibration (LMIG or Cs);

- Tilt (LMIG or Cs)

- Chicane amplitude (LMIG files);

- Pulse width (LMIG files).

Filter-Loading Part of the Instrument State Files

It is possible to load specific values of interest from a saved instrument state file. For instance, if the charge compensation parameters (Egun steering, lens and filament bias) were optimized in one INS file, and the operator wants to use these same values in another INS file, filter-loading part of the instrument state file is useful. To do this, first load the INS file with the parameters that you want to modify. Then in the Hardware Utility, select the INS file with the specific values of interest (the INS file with the optimized charge compensation), check the box for "Filter-load part of the INS file," and select Open. In the new window, move to the page with the values of interest (in this case the Semi-perm/Cal page) and select the boxes with the values of interest (Egun steering, lens and filament bias). When OK is selected, these values will replace the values already loaded in the Hardware Utility. The INS file can be resaved with the new values if applicable.

Another reason to use the Filter-Load option is during ion gun tuning. For example, when all the voltages are optimized in the positive polarity at a particular current and energy, the voltages required for the negative polarity are quite similar. It is possible to transfer the positive polarity voltages to the negative polarity mode; then tuning in the negative mode will require minor tweaking of these values. To transfer voltages from one polarity to the other, select the "Load voltages into opposite polarity" box located at the bottom of the filter-load window, and select all the voltages to transfer (for example all the voltages on the LMIG tab in the positive polarity). When OK is chosen these values replace the ones in the current Hardware Utility.

The Filter-Load option also comes in handy for creating dual beam INS files. For example, in depth profiling one often needs to analyze with the LMIG at 15keV and sputter with the Cs at 1 keV. For this mode, the operator would load the 15keV LMIG INS file. Next, the Cs voltages could be added using filter-load. Select the "filter-load part of the INS file box" and select the 1 keV Cs INS file, then choose open. In the filter-load window select all the voltages that are applicable: in the Cs tab, include all the voltages; in the Sources tab select all the relevant Cs column values (e.g. Cs Beam Energy, Cs Control Electrode, Cs Frit Power, Cs Oven Temp, Cs Extractor, Cs Emission and Cs Frit Bias); in the Semiperm/Cal tab, select the relevant Cs column values (e.g. uBeam Rotation, Cs Raster Scale Factor, uBeam Tilt, uBeam Quad/Oct Ratio, and Microbeam Vref). When OK is chosen the Hardware Utility will contain voltages for both Ga and Cs sources. The new INS file should be saved. Note that for depth profiling, the operator must ensure that the Cs and Ga guns are aligned for the sample of interest (refer to Section 7).

Section 4:
Tuning the Primary Beams

This section discusses the tuning process for the LMIG, the Cs ion gun, the O_2 ion gun (on the dual source option), and the Gas Gun . Tuning of the low energy electron beam is also covered in this section. The TRIFT III instrument is delivered from the factory with the following LMIG instrument files:

Filename	Description
15Ga3.ins	15kV, 600 pA high mass resolution (bunched) file
15Ga2.ins	15kV, 2nA high mass resolution (bunched) file
15Ga1.ins	15kV, 20nA high mass resolution (bunched) file
25Ga4.ins	25kV, 60pA high spatial resolution (unbunched) file
25Ga3.ins	25kV, 600pA high spatial resolution (unbunched) file
25Ga2.ins	25kV, 2nA high spatial resolution (unbunched) file

If the system includes a Cs ion beam, additional files will be included for 10kV Cs spectroscopy and for 1kV dual beam depth profiling. If the system includes the Gas Gun, additional files will be included for "phase depth profiling". The factory set instrument files can be loaded and used without tuning the ion beams. However, there may be times, such as after replacing any of the ionizer assemblies, that re-tuning of the ion beams will be necessary. The procedures outlined in this section describe how to optimize the LMIG for both the bunched and unbunched mode of operation. It also covers tuning of the Cs gun and O2 gun for both spectroscopy and depth profiling. Tuning of the Gas Gun is covered for optimizing "phased depth profiling" settings.

Liquid Metal Ion Gun (LMIG)

The latest generation liquid metal ion gun (LMIG) on the TRIFT features:

- Primary D.C. beam currents that range over three orders of magnitude from 20nA to 20pA;

- A pulsing scheme that minimizes beam aberrations which arise from the pulsing process, producing analytical probes down to 100 nm and 200 nm analytical probes at high mass resolution;

- An optimized electrodynamic buncher which permits high primary pulse compression with two micrometer spatial resolution; and

- High current (20 nA) spectroscopy mode at high mass resolution.

The ion gun is computer-controlled, resulting in a whole host of differing analytical conditions which can be recalled at the click of a mouse button. The following sections provide in-depth details to help the user to understand how the gun can be set up and optimized. Standard 'instrument files' will be loaded and used throughout this text as examples. Although the following sections may seem unnecessarily detailed, the more the user understands how the ion gun functions, the more he/she will be able to harness the gun for their own special analytical circumstances.

Firing the Source

The first required step is to fire the source as outlined below.

1. Load the desired instrument file. The source may or may not fire, since the 'onset' voltage for emission from a liquid metal ion source is always higher than the 'extinction' voltage.

2. If the source fires at the desired voltage, adjust the suppressor voltage until the emission current registers approximately 1 microamp.

3. If the source does not fire at the desired extraction voltage, lower the suppressor voltage until the source does fire. Again, adjust the suppressor voltage until the correct emission current is obtained.

4. If the source will not fire with the extractor at the desired value and the suppressor voltage at maximum, increase the extractor voltage until the source does fire. Sometimes, the resulting emission current can be instantaneously high (several microamps), and the extraction voltage should be quickly reduced to lower the emission current. If the source cannot be fired as described above, it may be necessary to heat the source. For instructions on how to perform this step, refer to 'Heating the Source' below.

Heating the Source

There are different circumstances in which it will be necessary to apply heating to the gallium source.

If the source cannot be fired, or the extraction voltage cannot be reduced to the desired voltage without extinguishing the source, it may be necessary to heat the source until the appropriate emission current can be achieved with the 'usual' extraction and suppressor voltage settings.

In time, as the source reaches the end of its life, it will be necessary to move to higher extraction voltages to extract the last of the gallium. This is expected after several months of steady operation, when heating the source is ineffective in maintaining the extraction voltage.

The circumstances in which heating may be necessary are listed below, ranked according to the severity of heating required.

1. In its native state, gallium is normally solid at standard room temperature. Although gallium freezes below 29.4° C, it 'supercools' in vacuum and remains in a liquid state well below this temperature. Consequently, it is normally not necessary to apply any heating to ensure that the gallium is liquid. Characteristics of a frozen source are that no emission occurs at the maximum extraction and suppressor voltages, despite the fact that the source had run at normal settings the day before.

    In order to fire the source, set the extractor and suppressor voltages to their normal values. Raise the filament current to approximately 1 amp for approximately 30 seconds. If the gallium was frozen, it will now be molten and the source should fire as normal.

2. The source is firing, but it is not possible to run with the extractor voltage at its standard setting, even with maximum suppressor voltage. A liquid metal ion source is an 'electrohydrodynamic' source which relies on unrestricted capillary flow of the liquid metal to the emitting 'Taylor cone'. In time, an oxide can form on the surface of the thin liquid gallium film, restricting its flow and requiring higher extraction voltages for a given emission current. Fortunately, the Ga_2O_3 oxide that forms will sublime in vacuum at modest temperatures. Do not attempt to clean the source excessively by heating at too high a current. An extremely clean source has a steep current vs. voltage curve, and this is not particularly desirable (refer to Figure 4-1 below)
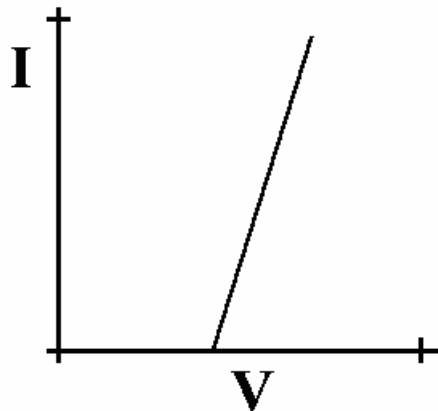
Figure 4-1.   *Diagram of the Current versus Voltage Characteristics for a Clean Liquid Metal Ion Source.*

In this state, small changes in the extraction field result in large changes in emission current. The steep current / voltage curve also coincides with the beginning of a period during which the liquid metal will undergo relatively rapid changes in its metallurgical surface condition. The steep curve is due to the fact that the capillary metal film experiences no restriction to flow on its outer surface until a thin surface oxide forms. On an ultra clean surface of liquid Ga, oxidation begins to occur, even in vacuum, reforming a native oxide on its surface. As it does so, the gradient of the current/voltage curve begins to reduce, owing to increased resistance to the flow of liquid. Because of the kinetics of the oxidation of most metals and alloys, oxide formation occurs rapidly at first, and then slows down significantly as the growth of new oxide becomes limited, typically by the diffusion barrier through the new oxide. Refer to Figure 4-2 below.
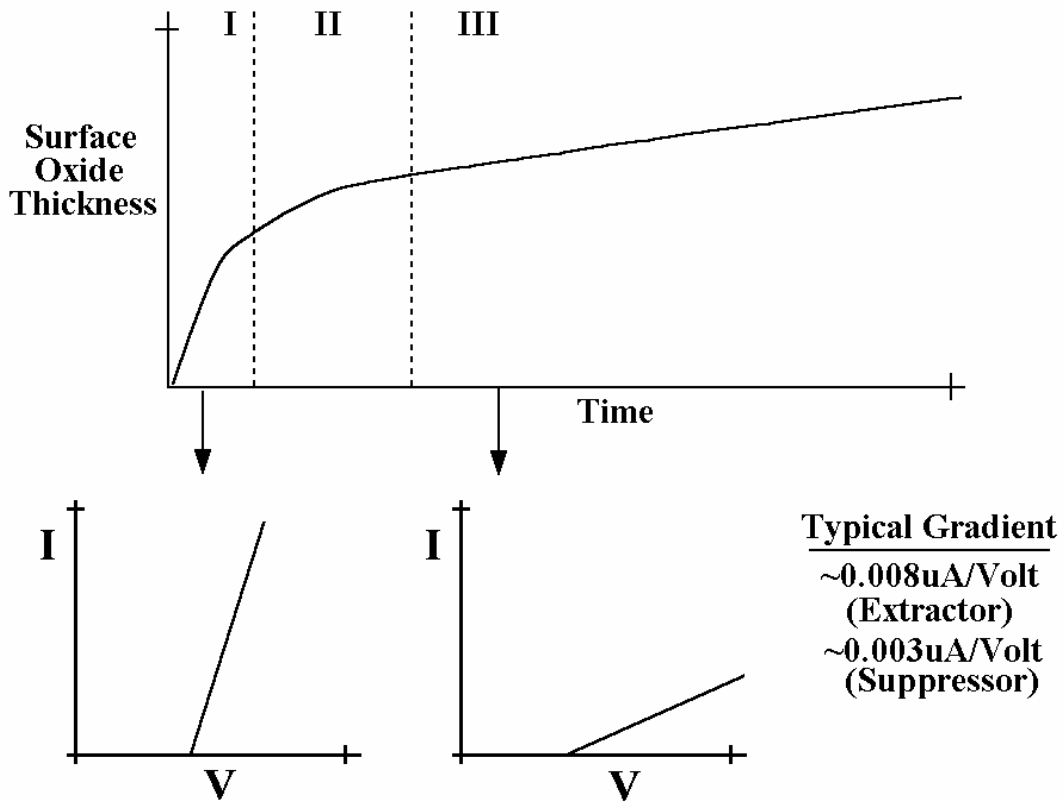
Figure 4-2.   *Diagram of the Surface Oxide Thickness versus Time on a Liquid Metal Source.*

The point of all this metallurgical thermochemistry is that if the source is cleaned excessively by subliming away all of the surface oxide on the liquid metal, the source will inevitably have a steep I/V curve. Once again, it will probably emit too high an extraction current at your 'normal' instrument state settings. As well as this, the liquid metal will inevitably begin re-oxidizing, causing the I/V characteristics of the source to change such that the extractor voltage will need to be changed relatively frequently to maintain a constant emission current (variation of the suppressor alone will be insufficient).

The preferred approach to source cleaning takes all of the above into account. The source should be heated at the lowest possible filament current to sublime just enough of the Ga_2O_3 so that the full range of the suppressor is recovered without needing to alter the 'normal' value of the extractor voltage. By doing this, the source liquid metal will always be maintained with a controlled surface condition, namely a thin surface oxide.

CAUTION*: Two different ^{69}Ga Liquid Metal Ion Sources (LMIS) are used on TRIFT systems: a 500 hour source and a 1500 hour source. The 500 hour sources have serial number 11604, while the 1500 hour sources have serial number 16830. The 1500 hour sources require LESS filament current to achieve the equivalent amount of source heating. Verify which source is internal to the Ga column <u>before</u> heating the source.*

A typical sequence of events would be as follows:

- At the standard extractor voltage, the suppressor voltage is now at its maximum negative value, and the emission current is <1µA.

- With the source still firing, set the filament current to ~1.8 amps and wait 25-30 seconds (it takes this time for the maximum temperature to be achieved at the source tip).

- Hopefully, some increased emission current will be observed, indicating that some of the Ga2O3 is subliming. Adjust the suppressor voltage to achieve an emission current of ~1.5 - 2.0µA. When the suppressor value is a positive value, turn off the filament current.

If the above procedure did not result in a significant change in the source characteristics, go back to the second step above, increase the applied filament current by a further 0.1 amps, and repeat the procedure. Keep increasing the filament current until you have achieved the desired amount of controlled cleaning (refer to Figure 4-3 below). Remember, if starting with a low filament current such as 1.8 amps, no harm is done and time is available to assess the changing source characteristics. If, however, a "speed things up" approach is adopted by setting a high filament current initially, the sublimation reaction may occur too rapidly for the operator to be in control, and may inadvertently overclean the source with the various inconveniences outlined above.

If the operating extraction voltage cannot be reduced by this method, the source may be nearing the end of its life. If this is the case, a higher extraction voltage is required for operation with the appropriate lens voltages.
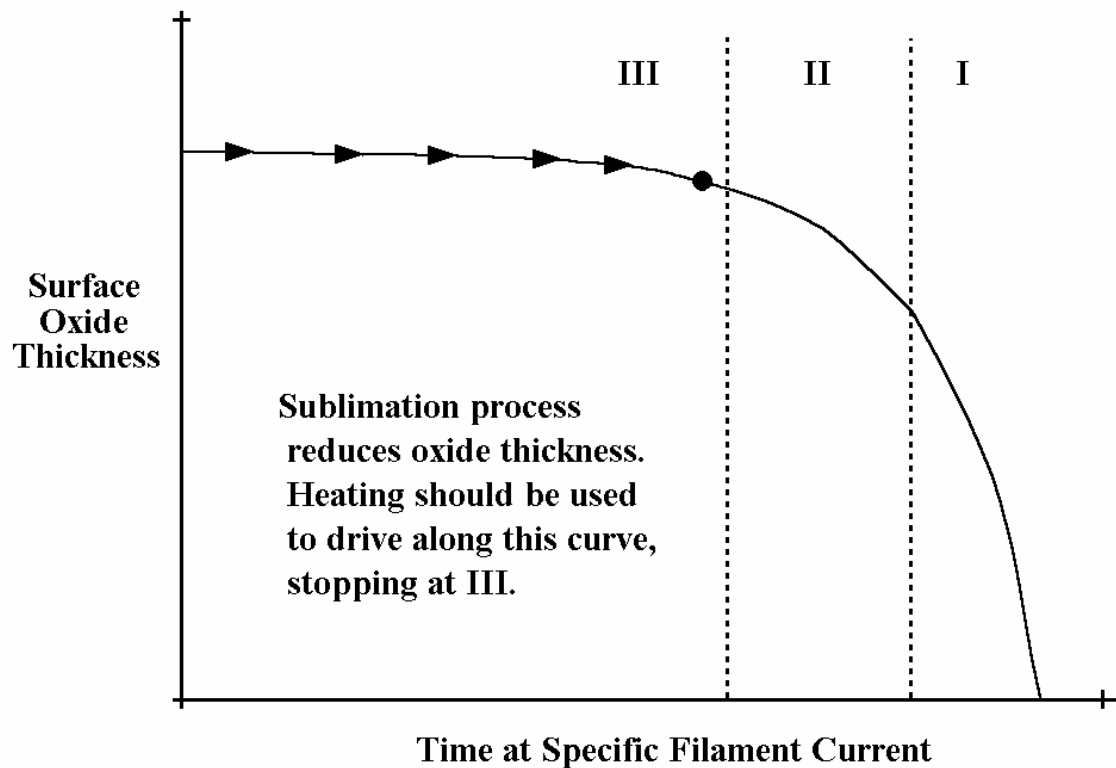
Figure 4-3.   *Diagram of the Surface Oxide Thickness as a Function of the Sublimation Cleaning Process.*

3. If the source will not fire, even after performing the heating steps described above, it may be necessary to heat the source until the gallium boils, thereby removing whatever is causing the source not to fire. This extreme heating will inevitably reduce the lifetime of the source because there is a limited amount of gallium in the source. This lifetime can be reduced dramatically if the heating process is not carried out extremely carefully. It is recommended that a service Engineer perform the steps outlined in Appendix A.

Tuning the LMIG

For routine system operation, the range of instrument files provided in the 'instrument state' library should meet nearly all analytical needs. However, if different files are desired, the following describes how this procedure may be carried out.

Choose a primary beam aperture

a. On the mechanically variable aperture (MVA) at the side of the gun, rotate the linear motion drive to a new aperture position. The convention for naming these apertures is as follows:

The first visible white ring on the manipulator corresponds to the first aperture, the 20 nA beam position; the second ring to the second aperture, the 2 nA beam position; etc. The sequence is: 20 nA; 2 nA; 600 pA; 60 pA and 20 pA, representing apertures 1-5. The manipulator should be set so that the x and y vernier readings are close to 0 when an aperture is in place. The convention for reading these scales on the MVA is as follows: aperture 1 = position 1.0, 0.0 (ideal setting); aperture 2 = position 2.0, 0.0 (ideal setting); and so on. Readings are taken at the white marks provided on the x and y drives.

b. Produce an SEM image by pressing the "DC" button at the bottom of the LMIG page of the Hardware window. Under the "Deliver beam to sample" option, choose "Liquid Metal Ion Gun", then select the OK button. Adjust the SED gain and the Raster Size to obtain a good SEM image.

c. Move to the LMIG control page. Wobble lens 1 using the automatic wobbling tool. This is done by highlighting the Lens1 field and pressing the wobble button at the bottom of the LMIG page.

Suppressor and LMIS ———

Extractor Cap ———

Acceptance Aperture ———
(AA)

Lens 1 ———

Mechanically Variable ———
Aperture (MVA)

Buncher ———

Lens 2 Steering Quadrupole ———

Blanker/Pulser Plates ———

Chicane Blanker ———

Lens 2 ———

Scanning Octupole ———
and Stigmation

Specimen ———
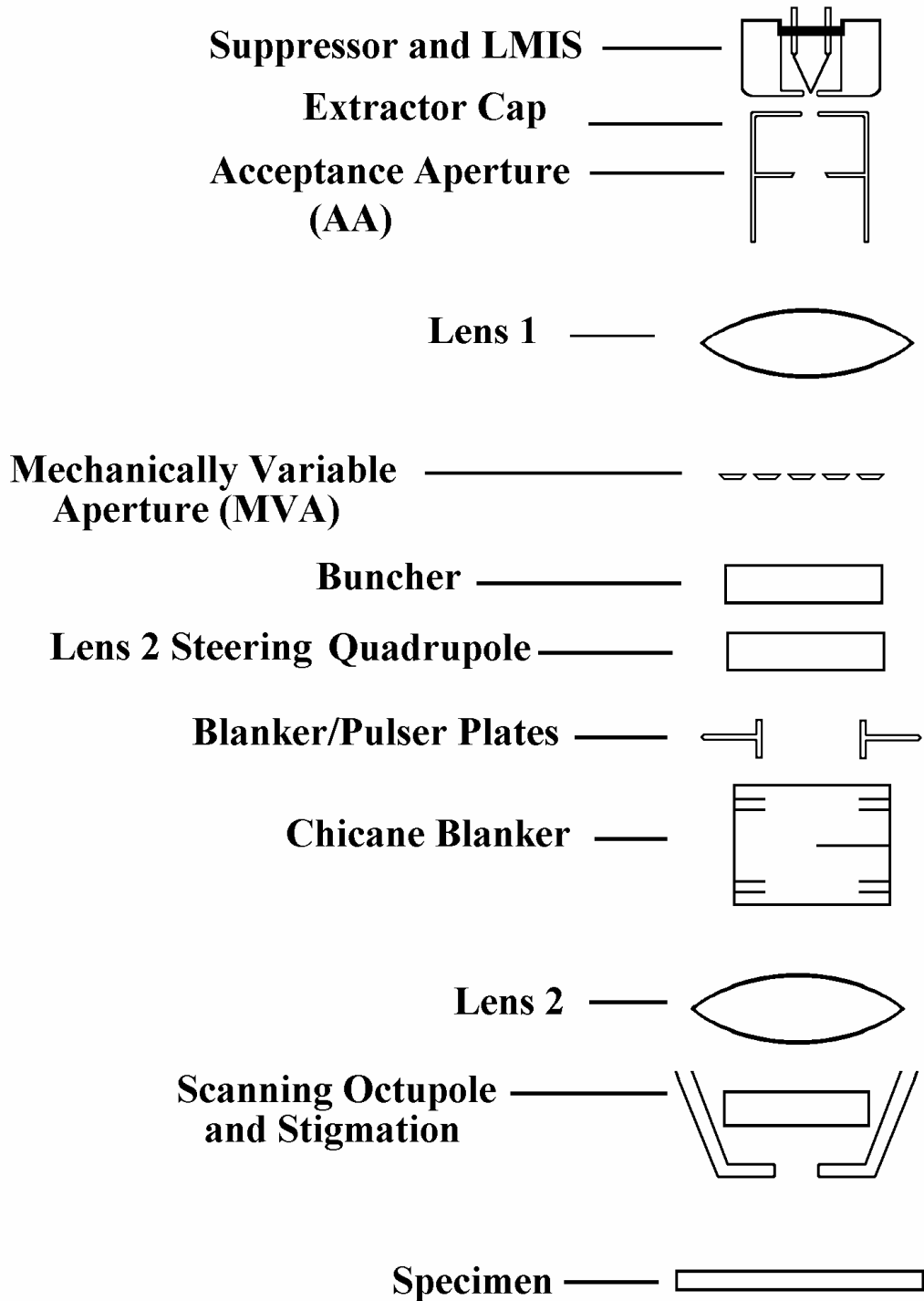
Figure 4-4.   Schematic Diagram of the LMIG Gun.

d. Move the x and y adjusters on the MVA to minimize any observable motion in the D.C. beam image. In order to reduce the effects of 'backlash' (mechanical uncertainty) on the accuracy of positioning, approach the aperture position from the same direction each time, for instance, a clockwise (CW) direction. When satisfied that the image is motion-free at a low magnification, zoom in and check at a higher magnification. Write down the final MVA readings as these settings will be keyed into the 'instrument file' header in the software when this particular gun tuning is saved as a file.

e. Remove the lens 1 wobble with the Remove Wobble button.

f. Now add a wobble to lens 2 using the software wobble tool detailed above. If the automatic, fixed amplitude wobbler is too much for this lens, try performing the wobble manually with the + and - keys on the keyboard. Adjust the x and y beam steering for lens 2 to reduce the wobble observed in the image. As a general rule, the lower these x and y values the better, although it is not realistic to expect them to be zero. Remove the lens 2 wobble.

g. Now add a wobble to the "blanker" voltage, and adjust the values of lens 1 and lens 2 so as to minimize the motion and yet still maintain a sharp image. This determines the "conjugate pair" that sites the beam crossover in the center of the blanking plates for motionless blanking. This is best done by first adjusting Lens 2 to minimize the wobble, the adjust Lens1 to refocus the image. Repeat this procedure until the image is wobble-free. Remove the wobble when finished.

h. Now comes the setting of the chicane amplitude. Until now, the beam has passed through the chicane and has not been near to the blanking "knife edge" owing to a high chicane amplitude. As a crucial part of achieving motionless blanking, it is now necessary to begin lowering the chicane amplitude so as to bring the beam closer to the knife edge. Try lowering the chicane amplitude in 10 volt steps until a clear reduction of the image intensity is perceived. Now raise the amplitude in 5 volt steps until the brightness reaches a plateau. The correct setting of the chicane amplitude for a given beam energy and beam current (aperture setting) is the one where the beam is just clear of the knife edge, i.e., has <u>just</u> reached the maximum image brightness. From this position, the minimum displacement to completely blank the beam is required. Generally, the blanker voltage required to just blank the beam at 25kV beam energy is as follows: the 2 nA beam requires ~15 volts; the 600 pA beam requires ~10 volts; the 60 pA beam requires ~5 volts; and the 20 pA beam requires ~3-5 volts. If the blanking required to blank the beam is significantly greater than these values, the chicane amplitude may still be a little high.

i. As the beam current is changed, the physical size of the beam at the knife edge changes, and so for each beam current there will be a different value of the chicane *amplitude* that brings the beam as close to the knife edge as possible. Note also that the magnitude of the chicane amplitude is proportional to the beam energy. Therefore, when setting the gun for 25kV operation, a chicane amplitude >360 (260 x 25/15) volts may be needed to allow a 2 nA beam to go well past the knife edge for tuning purposes.

Stigmator balancing and stigmation

a. Before stigmation of the beam can take place, make sure that the beam is centered as best as possible within the octupole stigmator. To do this, add a wobble to each of the two stigmators in turn, adjusting the x and y "balance" for each of them to reduce the wobble in the image, i.e., wobble stigmator 1 first, adjusting balance 1x and balance 1y in the gun menu. Once the beam is centered in the octupole stigmator, adjust each of the stigmators in turn to give the sharpest image possible. It may be helpful to operate at lower raster speeds at this point. To change the raster clock speed press the Raster Specs button in the LMIG page. Select a slower scan speed (i.e., 0.5 or 0.25 MHz). This will allow for easier observation of the fine tuning with the stigmators. Low scan speeds are mandatory when tuning the gun with smaller beam currents. Iterate between the stigmators and Lens 2 to produce the sharpest image.

b. Verify that the image size obtained corresponds to the one requested in the menu. If it does not, adjust the parameter "raster scale factor" in the Semiperm/Cal page. Note that this value will be different for positive SIMS and will need to be determined separately.

c. Beam tuning is now complete in this polarity (-SIMS). Except for the stigmator settings, the beam position and the raster scale factor, the gun parameters are the same for the opposite polarity (+SIMS). Printout the LMIG page in readiness for tuning the other polarity (+SIMS) by pressing the Print Page button.

d. During data acquisition, the blanker voltage remains at a constant D.C. voltage. A matching voltage is applied to the opposite blanking plate <u>only</u> when the pulser unit fires resulting in the beam pulsing on. The beam has now just been tuned in D.C. condition with the blanker plate voltages at zero. This means that the best image has been obtained at a specific place with reference to the knife edge in the chicane. When the beam is pulsing during acquisition, the values of the blanker and the pulser (amplitude) will need to be set so that the pulsed beam appears at the same position. This will not happen automatically. The values obtained at PHI, which appear in your master instrument files, should be used.

e. If it is necessary to determine the pulser value yourself, the following method can be used. Start an acquisition, setting the pulser deliberately low and then begin raising the pulser voltage. No counts are obtained until the beam begins to pulse past the knife edge, after which a rapid increase in the count rate is observed as a function of pulser amplitude. Once the beam is pulsing clear of the knife edge, the rise in the count rate will slow down, and any further increase in the count rate will be due to a longer on-time for the beam due to an increased transit time past the knife edge and then back again. Make a graph of the count rate vs. pulser amplitude, and then set the pulser at the point where the rate of change in the count rate just turns over.

Saving "Instrument files"

a. Once satisfied that the LMIG menu parameters are correct, save this instrument state as a file if the primary beam is:
    -pulsing correctly, with an appropriate pulse width;
    -steered with beam position x and y to center it to the spectrometer acceptance area;
    -an appropriate default raster size (>200μm recommended); and
    -provided that the other system parameters are acceptable.
    The concept of the "instrument file" is to be able to read and write a single file which contains all the relevant information to return the system to that particular state, e.g. imaging at 15 kV, etc. At the present time, the only parameters <u>not</u> under computer control are: the mechanically variable aperture (MVA) on the LMIG (although a computer control option is available); the contrast diaphragm, and the energy filter. All other parameters are stored in the "instrument file".

b. Remember that an "instrument state" file needs all relevant parameters for <u>both SIMS polarities</u>. A helpful way to think of an instrument file is to consider that it contains two "pages" of information to control the system: a +SIMS "page" and a -SIMS "page". For instance, following the preceding tuning instructions above has resulted in only one page (-SIMS) of this instrument file.

Changing polarity

a. To change polarity, press the Instrument pull-down menu and select the desired polarity. A check mark (√)will be shown next to the current polarity. Changing polarity takes about 10 seconds and is complete once the user hears the click of a relay within the system.

b. The LMIG values for the newly tuned polarity now can be transferred to the opposite polarity. This can be done by selecting the filter-load option from Load Ins File in the Hardware Menu. Then select "Load voltages into the opposite polarity" and select all the parameters that have just been optimized (also refer to Chapter 3). When OK is chosen the values will be transferred to the opposite polarity. Remember that the beam position x and y will be incorrect, since the "polarity offset" parameters have not yet been determined. Also, the exact stigmator settings will need to be determined. The beam position values stored in the master instrument state files for a given beam energy and polarity should be used as a first approximation.

c. Put the LMIG into a D.C. tuning mode, and fine tune the stigmators in +SIMS using a continuous image at the SED detector. Check that the raster scale factor is correct by adjusting it until the requested field of view on a known, patterned sample such as the Cu tuning grid matches the field of view actually obtained in +SIMS. Now save the instrument file again with the same name used initially for the -SIMS page. This completes a full instrument file with all the relevant LMIG parameters set correctly. All the time invested in creating this first, detailed file will pay off as more files are created with different primary currents at the same energy. Effectively, the only difference between these files in a given SIMS polarity will be the Chicane Amplitude setting, and possibly a small change to the stigmators.

Polarity offsets and beam alignment

The appropriate offset values have been determined at PHI and are stored with the instrument files. If new instrument files are created at energies for which files already exist by modifying a file, the correct polarity offset values will automatically be copied into the new file. However, if an understanding of how to determine the polarity offsets at any beam energy is necessary, refer to Section 5, "Determining the Polarity Offset Values".

Optimizing the LMIG for High Spatial Resolution

The highest spatial resolution is achieved at 25keV with the buncher off. In this mode, the pulse width can be adjusted to trade-off between sensitivity (long pulse width) and mass resolution (short pulse width).

The chicane LMIG allows data to be acquired at high spatial resolution no matter what the primary pulse width. This is because the chicane beam blanking system permits rapid blanking of the beam with the minimum of beam motion. In order to obtain the best spatial resolution while pulsing the ion beam, the chicane amplitude should be set so that the beam just clears the knife edge when the beam is pulsed on. By doing this, the beam has the minimum travel before it is blanked, minimizing aberrations introduced by pulsing. Refer to the previous section for details on how this is achieved.

The minimum analytical beam size (<120nm) is achieved with the smaller of the available D.C. beam currents (20, 60 pA), and at longer pulse lengths where slight residual beam shifts due to pulsing represent only a negligible fraction of the beam 'on' time.

High mass resolution simultaneous with probe sizes of ~200 nm is achievable at all primary beam currents, but in practice is performed at the higher beam currents of 600 pA or 2 nA. This is because mass resolution at high spatial resolution (200 nm) is achieved by *reducing* the primary pulse width, not by electrodynamic bunching. As a consequence of *reducing* the primary pulse width, the primary beam flux is reduced. This in turn reduces the secondary ion count rate. For this to be high enough for practical data acquisition, begin with a high primary beam current. When reducing the pulse width to increase mass resolution in a 25 kV beam energy instrument file, a point is reached at very short widths where the pulser amplitude begins to also fall. This requires compensation through increasing the value of the pulser (amplitude) for pulsing the beam just past the chicane's knife edge once again. Remember to be realistic about acquisition times. If 5 minutes in needed with a pulse width of ~10ns to acquire sufficient data at a 1000 m/z range, it will take 25 minutes to acquire the same data using a 2 ns (reduced) pulse width. In such cases, set the maximum mass range as low as possible to increase the repetition rate of the experiment. By doing so, the data rate is increased by a factor of 3 which reduces the acquisition time in the above example to ~8 minutes.

Optimizing the LMIG for High Mass Resolution

This section discusses the factors influencing mass resolution performance in TOF-SIMS. High mass resolution performance in a TOF system depends upon three factors, as shown in Figure 4-5 below. In the low mass range, the primary beam pulse length is the dominant factor. In the medium mass range the spectrometer tuning has the major factor. Finally at high mass, electronic effects become significant to the ultimate mass resolution. Since there are no adjustments available of the fundamental electronic characteristics of the timing electronics and the power supplies, this leaves two instrumental factors available for tuning.

To obtain high mass resolution performance with the TRIFT, there are two distinct tuning phases: first, optimizing of the primary ion beam parameters to minimize the pulse length, and secondly optimizing of the spectrometer parameters to maximize the angular and energy compensation of the secondary ions. These have been set in the instrument files. However, if you wish to understand these two aspects of instrument optimization, detailed instructions are now provided. Note that since spectrometer optimization is independent of primary beam species, in this (LMIG) section we will describe *only* the first phase, namely optimization of the LMIG parameters. To continue with high mass resolution tuning after optimizing the LMIG, refer to "Practical Method of Tuning the Mass Spectrometer for High Mass Resolution Performance" in Section 6.

A = Length of Primary Ion Pulse
B = Mass Spectrometer Characteristics
C = Time Resolution of Detection System
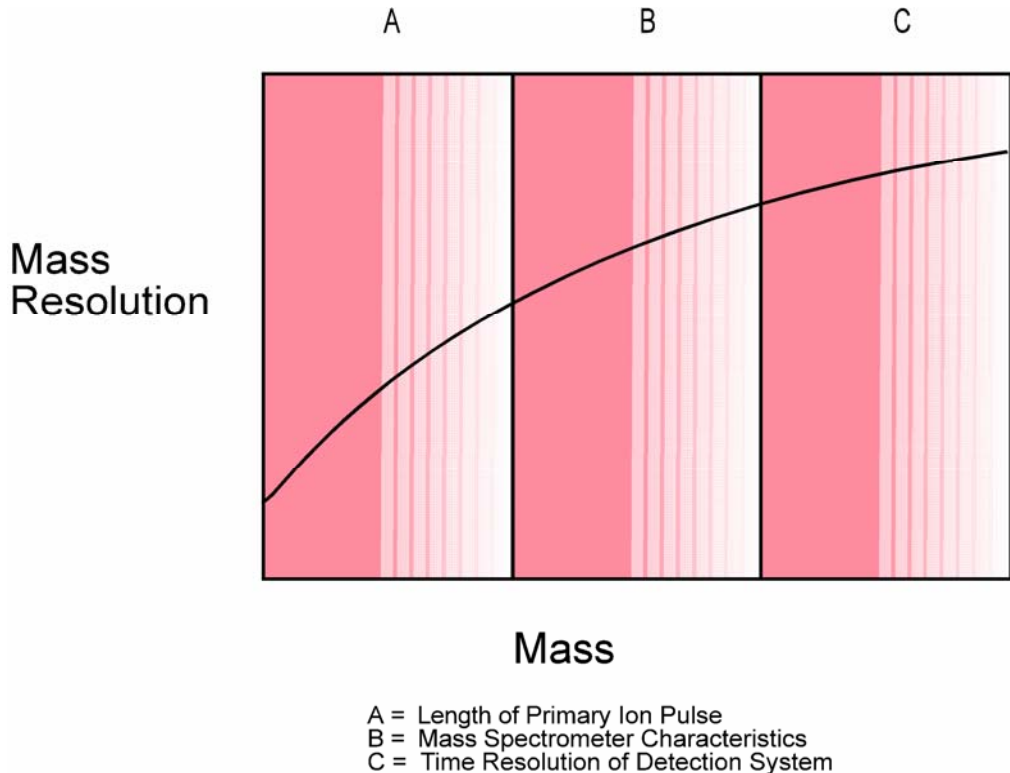
Figure 4-5.   *Diagram of the Principal Effects of Mass Resolution versus Mass in TOF-SIMS.*

Preliminary Tuning

a. Tune the LMIG for 15kV operation as described in the LMIG tuning section. Select the 600pA aperture. When attempting to set up files for mass resolution, *tune the gun with the buncher voltage on.* If you have not yet determined the optimum buncher voltage, set this parameter to ~800 volts. Remember to check all alignments: lens 1 centering; lens 2 centering; a crossover in the center of the blanking plates (wobble blanker and set lens1 and lens2 for minimum motion); set chicane amplitude, and finally the stigmator "balancing" (centering) and stigmation itself. The chicane amplitude should be set so that the minimum voltage is required on the blanker to turn the beam "off" (see LMIG tuning). Set the stigmation *after* you have determined the correct chicane amplitude for any given beam parameters (energy, current).

b. Note that, even with a perfectly tuned column, if you make the blanker an increasingly *negative* value, the image will not only become less focused, but it will move as well. Don't worry. This is the expected behavior. You are moving the beam *away* from the knife edge into a position which results in greater aberrations. The important thing is how the image appears as you take the blanker from zero to *positive* values. If you do not achieve a motionless blanking at low blanking voltages, consult the LMIG tuning section once again.

c. The gun has just been tuned with the buncher voltage on, in a D.C. condition, i.e. the buncher voltage *was constant and not pulsing*. During data acquisition however, the buncher is pulsed, and this results in an "overfocusing" of the beam. To compensate, you will need to determine a new lens 2 voltage which, during pulsing, will compensate for this overfocusing effect.

d. The way to determine this lens 2 value is to reduce lens 2 in 50 volt steps, acquiring an image at each lens 2 value from a copper grid in positive SIMS mode. Since the buncher overfocuses the beam, lens 2 will need to be lower to compensate. Each LMIG column is slightly different in its behavior, and the appropriate drop in lens 2 will have to be determined experimentally. Move to the next step when a SIMS image with 2 - 3 um resolution in the image has been obtained.

Minimizing the bunched primary pulse length

a. In this first phase of tuning the system for high mass resolution, only two
    LMIG parameters will be dealt with: the buncher voltage and the buncher
    delay. The buncher voltage is accessed directly in the Hardware menu; the
    buncher delay is accessed under Instrument, Set Delay Lines, Buncher. To
    perform this next task, have a silicon wafer in position, and set the
    spectrometer to "fully open" (no C.D. or energy filter). For convenience, a
    blank tuning grid is provided in an appendix to help in note taking. Although
    this is the *full* tuning grid including the spectrometer parameters, entries for
    the relevant LMIG parameters are also discussed below.

b. Before adjusting the bunching parameters, make sure to not attempt to bunch
    too much primary beam. In general, a 15ns pulse can be compressed
    efficiently, although in practice a 12ns pulse or less is more forgiving. To
    check the input pulse going into the buncher, set the buncher voltage to zero,
    set the time channels to 1ns and acquire data. Count the number of channels
    at FWHM at hydrogen. Make sure that the input pulse is appropriate. If you
    want to have the maximum input pulse into the buncher, the buncher timing
    will need to be set very carefully. If a lower input pulse is chosen, there will
    be some 'play' in the buncher timing.

c. Turn the buncher voltage back on and arrange the mass spectral display so
    that hydrogen occupies the full screen. Insure that the minimum time
    resolution, namely 138ps, is being used. Begin altering the delay until the
    beam is clearly being compressed, and then, for a given buncher voltage,
    optimize the buncher delay (2ns steps) so as to minimize the number of
    channels which define the full width half maximum (FWHM) of the
    hydrogen peak. When this is minimized, switch to the buncher voltage, and
    try altering this in 25 volt steps until the FWHM of hydrogen is minimized.
    With no C.D., a FWHM of 6-8 channels should be achievable. Note that if
    this experiment is performed with the 1st C.D. (400um) in place, the FWHM
    will be less because the angular acceptance of hydrogen is being reduced. If
    tails are observed at either side of the peak, the buncher is attempting to
    bunch more ions than it can accommodate.

d. Monitoring hydrogen is an excellent way to minimize the primary pulse
    width. However, for final determination of the correct buncher and delay
    values, repeat the above experiment on an organic peak (i.e. C_3H_5 at mass
    41), again minimizing the FWHM in terms of the number of channels which
    constitute the peak. This is because, unless the spectrometer is completely
    misaligned, the molecular ions are much less sensitive to spectrometer tuning
    than the atomic ion species. Good mass resolution can be achieved on
    molecular species as long as the primary pulse length is short, even if the
    spectrometer tuning is not fully optimized. This is not true of atomic species
    such as Si.

e. If you have achieved 6-8 channels on hydrogen (no C.D.), and if the mass
resolution at C_3H_5 is ~8000-9000, this concludes the LMIG parameter tuning.
It is still likely that the atomic species such as Si will not yet be optimized,
and so now move to the second phase of instrument optimization, namely
spectrometer tuning which is referenced in Section 6.

Primary Beam Dose Calculation for LMIG Experiments

A brief explanation below centers on two topics: the first principles on
calculating the number of ions delivered (and thereafter the dose calculation);
and secondly, how the dose calculation is implemented in the software from a
practical approach.

First Principles

a. In a continuous beam system, this is a simple parameter to determine. Only
the primary beam current and the experiment time are needed to calculate
this. Then:

Primary ions = Beam Current (Amps) x 6.25 E18 (ions/Amp.sec) x
Experiment Time (seconds).

For instance, a beam current of 1 nanoamp on for 100 seconds will give:

1 E-9 (Amps) x 6.25 E18 (ions/Amp.sec) x 100 (seconds) = 6.25 E11
Primary Ions delivered

b. In a TOF system, the beam is not on continuously, and so in addition, the
frequency of pulsing and the duration of the beam 'on-time' for each primary
pulse is needed. Then:

Primary ions = Beam Current (Amps) x 6.25 E18 (ions/Amp.sec) x
Experiment Time (seconds) x Frequency (Hz) x Primary Pulse Width
(nanoseconds).
The frequency is dependent upon the mass range. It can be determined for a
specific mass range in the Hardware menu, in the Sequencer. The Load value
of Prate 0 must be multiplied by 200×10^{-9} to obtain the 1/frequency. Invert
to obtain Hz.

The Primary Pulse Width was determined in the previous section by
measuring the FWHM of the H signal. Multiply the number of channels at
FWHM by the time resolution of the acquisition (the nanoseconds/channel
value found in Acquistion Setup/Advanced Settings).

Here is an example, for a beam current of 1 nanoamp, an experiment time of 100 seconds, a repetition rate of 10kHz and a beam 'on-time' of 10 nanoseconds per pulse of primary beam, the yield is:

1 E-9 x 6.25 E18 x 100 x 1 E4 x 10 E-9 = 6.25 E7 primary ion delivered (i.e., down by 10,000 on D.C. case).

c. Dose calculations are simple once the number of primary ions delivered is known. Express the area of analysis in cm^2, and divide it into the total number of primary ions delivered, e.g.

Number of Primary Ions = 6.25 E7;

Area of Analysis 100um x 100um;

Dose = 6.25 E7 ions/1 E-4cm^2 = 6.25 E11 ions/cm^2

WinCadence Dose Calculator

The dose of primary ions for each acquisition will automatically be calculated through the software once the dose standardization is set up. The dose standardization is found on the Instrument Settings page of Acquisition Setup, and can be performed using the simple or automatic options. For the simple method, click on this option and choose Go. You will be prompted to enter the primary ion current (can be measured by choosing the D.C. option in the Hardware Menu and "monitoring beam inside primary column") and the pulse width (measure the FWHM of H and multiply the number of channels by the time resolution of the acquisition (the nanoseconds/channel value found in Acquisition Setup/Advanced Settings)). Then the ions per pulse will be automatically calculated. For the automatic method of dose standardization, you will be prompted to position the sample and accept an acquire time of 7 sec. The system will then automatically measure the primary beam current in the Faraday cup, acquire a spectrum in the unbunched mode, measure the CH$_3$ (positive polarity) or OH (negative polarity) peak width and intensity, measure the peak intensity in the bunched mode, and finally make a dose estimate. If the estimate sounds reasonable Accept when prompted. After the dose standardization has been performed, subsequent spectra will have their total dose property set. Check the Info Tool in the Spectral Viewer. Remember, the dose rate must be determined each time a different current or pulse width is used for the dose calculation to be accurate.

Cesium Ion Gun

Precautions

Cesium is a highly reactive alkaline element, that melts at 28 C and undergoes a violent exothermic reaction when brought into contact with water or moist air. New cesium vials are hermetically sealed, but once the vial has been cracked the 'Nupro' valve on the ion source should always be closed before bringing the rest of the system up to atmospheric pressure. The replacement of the cesium vial should only be attempted by a PHI service engineer.

Ion Gun Overview

The cesium source is a surface ionization type source. A reservoir of cesium (approximately 5g) is heated by an oven (inside the 'mail box' cover at the back of the column) to produce cesium vapor. The vapor diffuses to the back of a tungsten frit that is heated to >1100C with a refractory metal resistance heater. The cesium diffuses through the porous frit and is auto-ionized at the front surface of the frit and extracted away from the source. The cesium atoms are auto-ionized because the work function of the tungsten surface is 4.52eV, compared to the ionization potential of cesium, which is 3.88eV.

There are, therefore, only two key source parameters that need to be set: the oven temperature and the frit power. Generally, the supply of cesium vapor should be sufficient to provide enough cesium to the front of the frit, but should not be so great that the frit becomes clogged with cesium. Likewise, the frit power should be enough to provide efficient auto-ionization of the cesium at the front of the frit, or else, once again, the frit will become clogged.

In practice, it has been found that an oven temperature between 105C and 120C (typically 110C) is a good match to a frit power setting of between 30-35 watts. A higher oven temperature will require a higher frit power to maintain a dynamic equilibrium flow of cesium vapor. Remember however, that increases in frit power do not result in linear increases in frit temperature.

Ion Source Operation

All the optical elements in the ion column are controlled from the "Cesium" page of the Hardware Utility window: (WinCadence: Instrument, Hardware Utility, Cesium page). The important parameters for the ion source operation are found on the "Sources" page of the Hardware Utility.

- **Cs Oven Temperature:** This is the value (in °C) which determines the operating temperature of the oven surrounding the Cs reservoir. It may be changed by the user and typical values are 100° - 120° C. To read the actual temperature, use the F11 function key on the keyboard.

- **Cs Frit Power:** This is the power value in Watts applied to the filament that surrounds the Cesium frit assembly. Cesium ionization occurs when the surface of the tungsten frit is high enough for the Cesium (diffusing up from the reservoir) to auto-ionize. Typical settings are 30 - 35 watts.

- **Cs Emission:** This is a 'readable' value to allow the user to measure the total ion emission from the frit, and is in units of microamperes. It can be read back using the F10 function key on the keyboard.

- **Cs Control Electrode:** This is a 'field-shaping' electrode immediately in front of the frit, for the purpose of optimizing the lens properties of the source region.

- **Cs Frit Bias:** This negative voltage floats on top of the beam energy. A negative voltage on the frit bias permits the stable operation of the control electrode. The frit bias has no ion optical purpose.

- **Cs Extractor:** This 'floating extractor' supply is downstream from the frit and control electrode, and allows more ion current to be drawn from the source. Its main use is to increase the probe current from low energy beams (typically ≤2,000 volts).

Operation

Oven Set Temp Function

Older, manual controllers for the Cesium source had "On", "Off", and "Revive" settings. This is now controlled by the simple expedient of the oven set temperature in the Sources tabbed page of the Hardware menu within the WinCadence software.

- **On**: When the oven set temp is >40° C, the oven heater is turned on, the frit (ionizer) heater is turned on, as well as a minimum bias voltage on the frit (500V). The fan is set to off.

- **Off:** If the oven set temperature is <20° C (for instance when a zero volt file is loaded), the fan is turned on (until oven temp is sensed to be <40° C), and the oven heater is turned off. The frit heater and frit bias are turned off once the oven temperature has fallen below 60° and 40° C respectively (Cs extracted from source while Cs vapor is inside ionizer tube).

- **Revive:** If the oven temperature is set between 20° and 40° C, the oven is turned off, but the frit heater and frit bias remain on. This is the mode that you use to clear a 'clogged' frit. For instance, if the source had been run with too high an oven temperature, it is possible that there could be too much Cs in the tungsten pores, blocking smooth diffusion of Cs vapor (supply rate of Cs > removal of Cs by ionization). By running the frit heater and frit bias at a minimum value with the oven off, excess Cs can be cleared out of the source. This is the mode known as "revive", for obvious reasons.
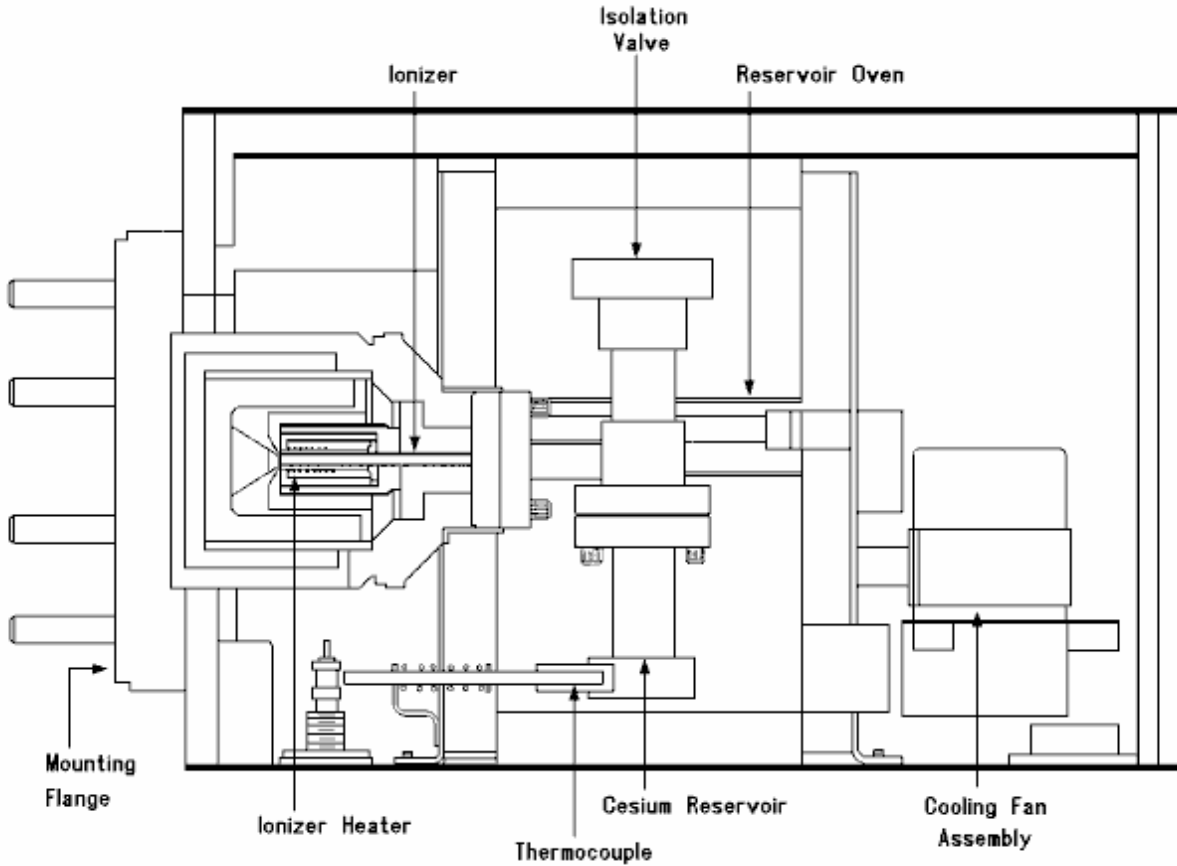
Figure 4-6.   *Cutaway View of the Duoplasmatron Source.*

Table 4-1.   *Listing of Source Modes and Corresponding Settings or Conditions.*

Mode:	Oven:	Bias:	Frit:	Frit Bias	Control Electrode	Fan:
Oven Set Temp. > 40° C	On	On *	On *	On	On	Off
Oven Set Temp. < 20° C	Off	Off @ Oven Temp. <40° C	Off @ Oven Temp. <60° C	Off	Off	On @ Oven Temp. >40c
Oven Set Temp. Between 20° C – 40° C	Off	On *	On *	On	On	On
Frit Interlock	Off	Off @ Temp. <40° C	Off @ Oven Temp. <60° C	Off	Off	On @ Oven Temp. >40° C
Vacuum Interlock	Off	Off	Off	Off	Off	On @ Oven Temp.>40c

* Bias value will set to 500 volts if no value is set in 15 seconds.
* Frit power will set to 30 watts if no value is set in 15 seconds.
* Frit interlock mode occurs when frit value differs from set value by 20 watts.

Tuning for Spectroscopy at 10kV Beam Energy

For spectroscopy performed at a beam energy of 10kV, the Cesium ion gun is first tuned for a D.C. beam current of ~1-2 nA (5ml slit / 5ml aperture) producing a probe diameter of ~50 µm. For spectral acquisition at high mass resolution, the beam is electrodynamically bunched to give a pulse width of ~1.25-1.5 ns. The following description starts with a discussion of how to tune the ion gun in a D.C mode, followed by a discussion on how pulsing and bunching is achieved within this column. Finally, tuning for a pulsed, bunched and focused beam is described. A schematic of the Cesium ion column with all its optical elements is provided in Figure 4-6 below to assist the operator in becoming familiar with these operations.

Once the reservoir has reached its operating temperature, the total emission current from the source can be monitored by read-back of the emission current in the Source page of the hardware menu. With a beam energy of 10 keV, the emission current should be somewhere between 5-15 µA. The Control Electrode element in the Cesium page floats at the beam energy potential and is given an offset voltage to establish ion emission from the source (typically 0-100V) and provide some control over ion trajectories in the extraction region. This parameter is power supply-dependent and typically does not require any alteration.

Frit ——————————————

Control Delta ——————————

Extractor ——————————

Lens 1 Quadrupole Deflection ———

Blanking Aperture ——————

Lens 1 ——————————

Mechanical Gate Valve —————

Lens 2 Quadrupole Deflection ———

Variable Slit Aperture —————
(0.005, 0.010, 0.100)

Lens 2 ——————————

Buncher ———————————

Blanker 2 —————————— ....(1.4° Bend)

Variable Blanking Aperture ———

Faraday Cup Deflector —————

Faraday Cup ————————

Lens 3 Quadrupole Deflection ———

Scanning Octupole and Stigmation —

Lens 3 ——————————

Specimen ———————————
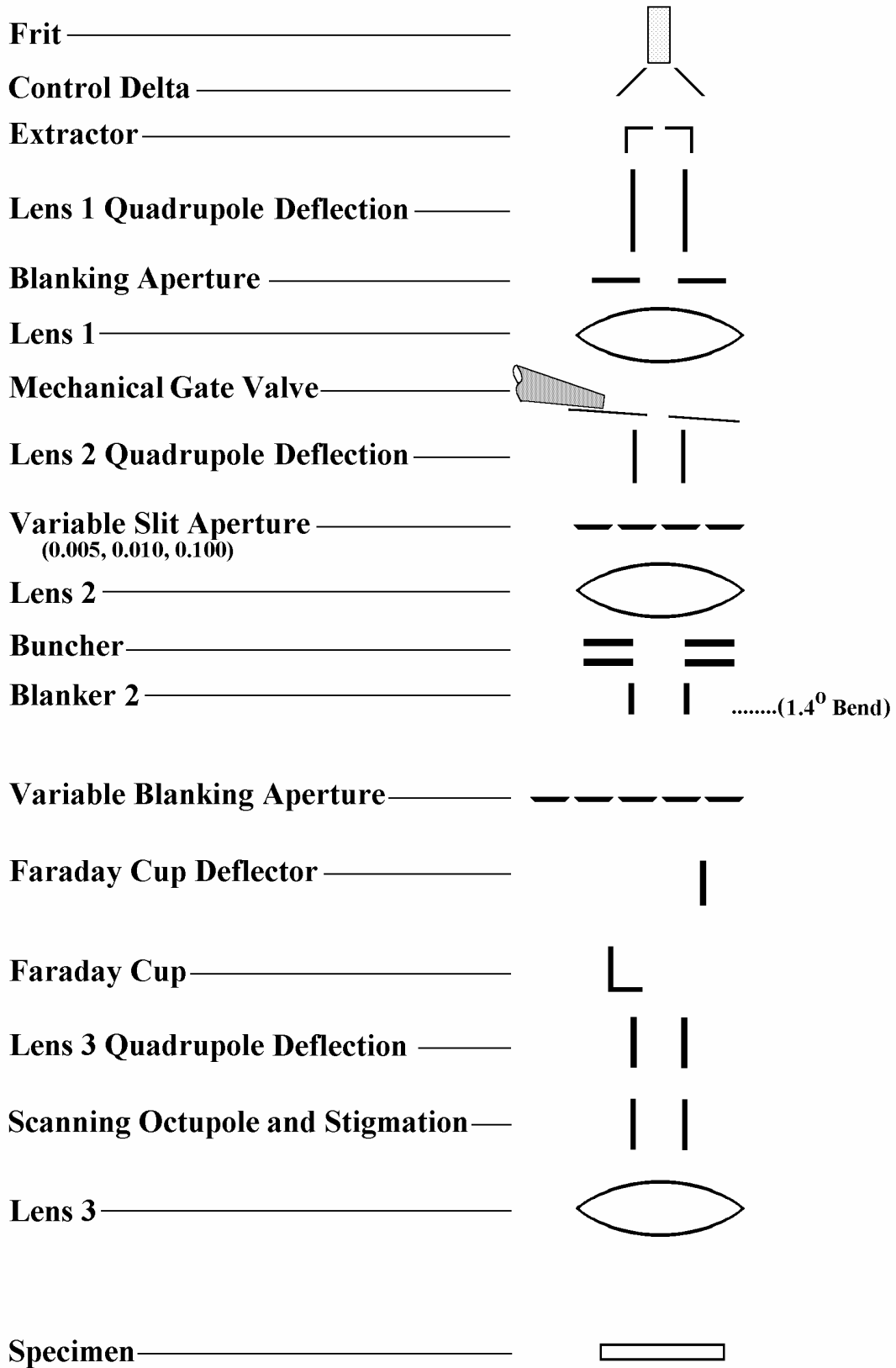
Figure 4-7.   *Schematic Diagram of the Cesium Ion Gun.*

D.C. Beam Alignment

Once ion emission has been established, it is possible to monitor D.C. beam current either on the sample (with a picoammeter) or further upstream using the ion column Faraday cup. The beam is set to D.C. in WinCadence on the Cesium control page by clicking on the "D.C." button at the bottom of the page. Under "Monitor beam inside primary column", choose Cesium Gun on and select the OK button. Using the Faraday cup monitor, the column should now be aligned for maximum beam current.

For high energy (ie $\geq$ 4 keV) operation, the 'extractor' electrode is maintained at 0 V, giving an extraction potential defined only by the beam energy voltage. The three lens column has a set of beam alignment plates in front of each lens in order to steer the beam through the center of each lens. As with most ion / electron guns, the alignment procedure is an iterative process that is described in the flow chart shown below (refer to Figure 4-7). Each of the elements should be sequentially adjusted for maximum beam current, until the measured beam current reaches a plateau.
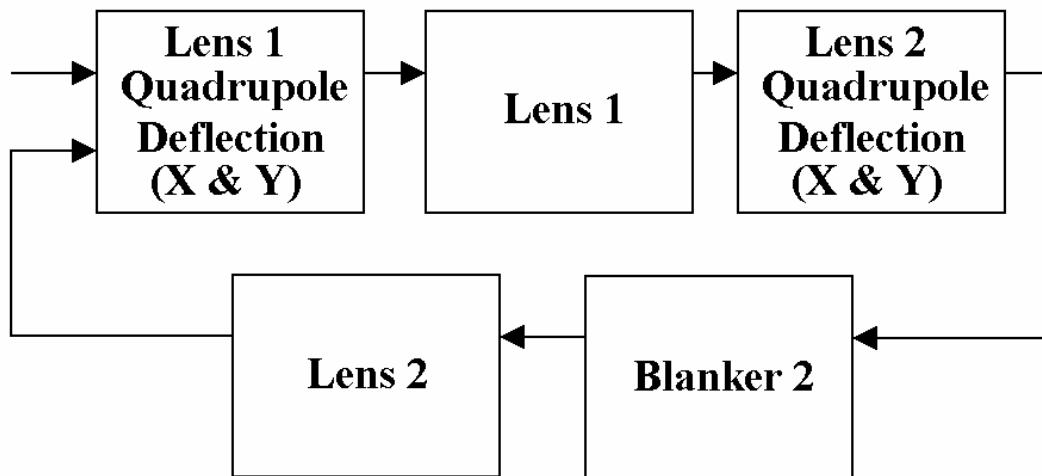
Figure 4-8.   *Flowchart Diagram of the Cesium Ion Gun Alignment Procedure.*

Pulsing and Bunching Operations Yields Short Primary Pulses

There are two beam blanking deflectors in the cesium ion column. In pulsed operation, blanker 1 will pulse down from nominally +200V to 0V, to unblank the beam for a period of 4 μs (long pulse during each acquisition cycle. A period of approximately 2 μs elapses to allow the front of the pulse to pass through the buncher. At this time, the front buncher electrode is pulsed down from the preset voltage (typically, +1000 V), such that the 7mm section of pulse that is inside the buncher when it fires (~ 60 ns pulse at 10 kV) experiences the bunching pulse. Immediately after the buncher, blanker 2 chops out all but the 60 ns bunching pulse, allowing it to proceed down to the sample. Thus, the time delay between the buncher firing and blanker 2 unblanking is critical and requires fine tuning. A depiction of the pulsing sequence is shown in Figures 4-9 through 4-12 below.
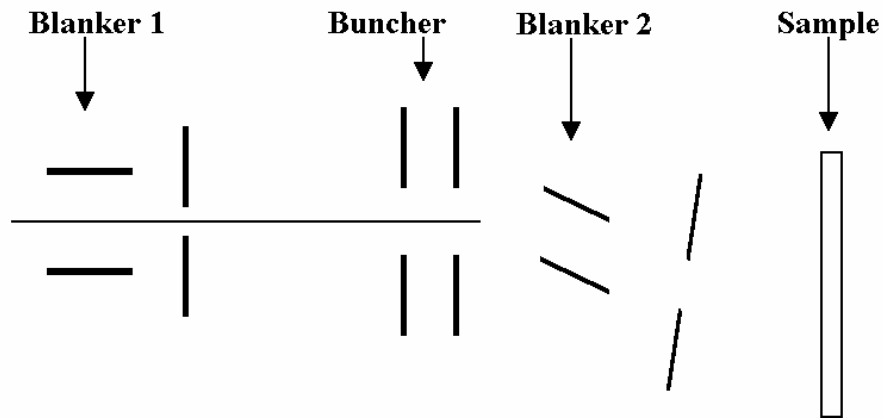
Figure 4-9.   *The long pulse produced by blanker 1 enters the buncher region.*
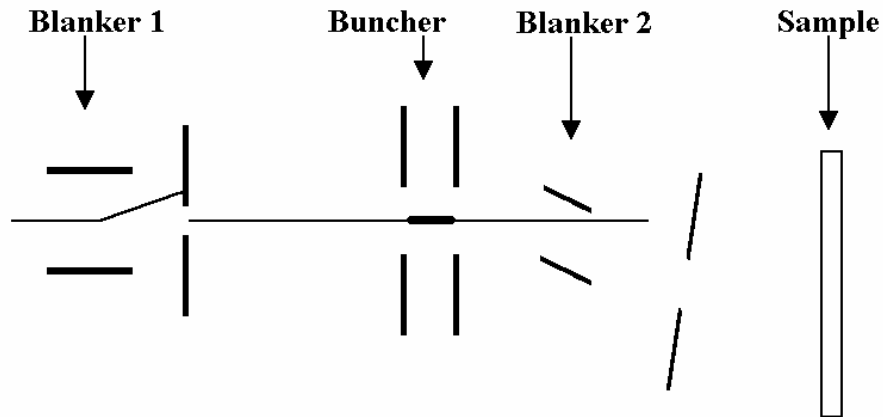
Figure 4-10.   *The buncher pulses down to ground and starts to bunch the part of the pulse that is present within the buncher.*

Blanker 1   Buncher   Blanker 2   Sample
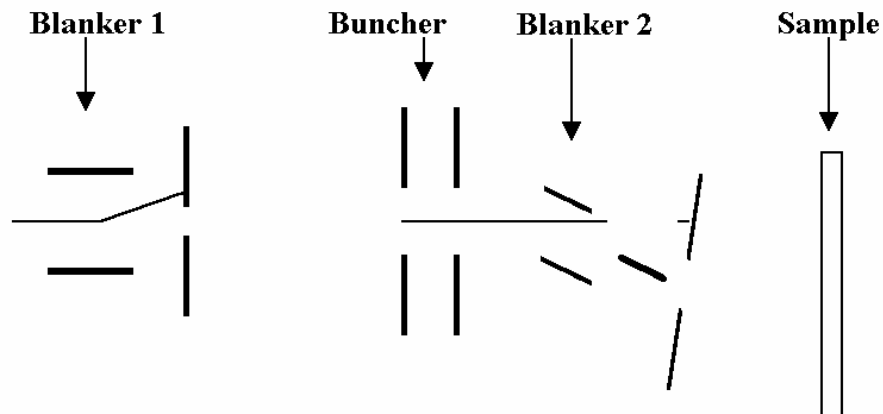
Figure 4-11.   *Blanker 2 turns on for ~ 60 ns to deflect the bunching part of the pulse past the 3 degree bend in the column and on towards the sample.*

Blanker 1   Buncher   Blanker 2   Sample
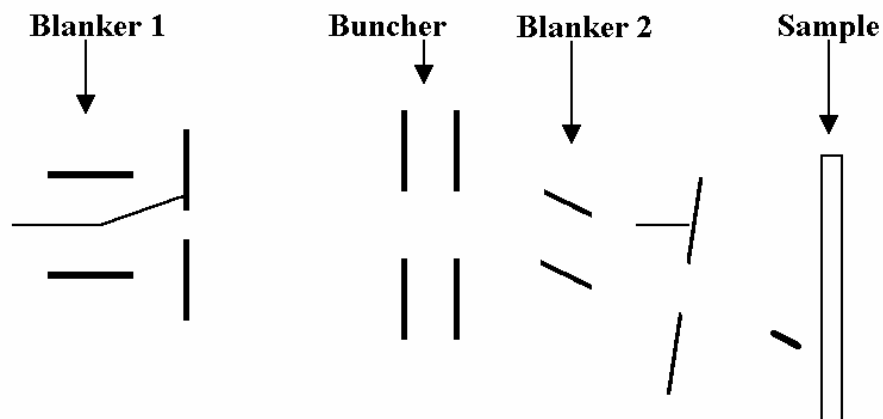
Figure 4-12.   *The bunching pulse compresses in time as it approaches the sample.*

Tuning the Pulsing and Bunching Operations

The tuning procedure for optimizing the pulsing and bunching is described below.

1. In -SIMS mode and with 138 ps per channel selected, set the spectral display window around the hydrogen peak such that the individual channels within the peak are visible. This is also possible in +SIMS mode, but the hydrogen signal is normally much lower.

2. If the optimum buncher voltage is not yet known, then set to +1000 V.

3. Acquire data from a planar sample, such as a silicon wafer.

4. In the delay line page (WinCadence: 'Instrument, Set Delay Lines') the parameter called 'Buncher' allows the buncher timing to be adjusted in 1 ns increments. The parameter referred to as 'Blanker 2' sets the pulse width of blanker 2 in units of nanoseconds. Set the pulse width to 40-60 ns and the delay to anything between 0 to 255ns.

5. Lower the blanker 2 voltage by about 10 % from the D.C. value, to allow for the reduction in mean beam energy brought about by the bunching operation.

6. Using the image on the DMCP, adjust the bucher delay until some bunching is evident. Bunching is recognized by a defocusing of the beam viewed on the DMCP. Typically, lens 3 will focus ~300 V lower in the pulsing / bunching operation compared to the D.C. beam condition. Re-focus Lens 3 for the best probe under bunched conditions.

7. Slight adjustment of the lens 2 voltage may now be necessary to obtain the brightest image at the DMCP. This will optimize the position of the beam cross-over in the blanking aperture. This should result in the minimum change in the blanker 2 voltage to turn the beam off (~20 V reduction in the blanker 2 voltage should blank the beam). Focus the beam again with lens 3.

8. The timing of the buncher and the setting of blanker 2 should now be optimized while acquiring a negative ion spectrum and observing the H⁻ peak width. It is possible to compress a 40-60 ns pulse down to 1.25-1.5 ns FWHM (i.e. 9-11 channels at 138 ps / channel) with optimum buncher voltage and timing.

Low Energy Cesium Beam Alignment

For dual beam depth profiling, an instrument file containing both the LMIG settings <u>and</u> the Cs beam settings is necessary. New instruments ship with an instrument file for depth profiling with a 1 keV Cs beam and a 15 keV bunched LMIG for acquisition. This section is useful for setting up other low energy Cs beam settings or for re-tuning the 1 keV file. A variety of dual beam depth profiling instrument files can be setup by using the filter load function to match the desired LMIG settings with the desired Cs beam settings.

All three lenses and lens 1 and 2 steering are expressed as a percentage (%) of the beam energy. This makes it easier to generate new Cesium beam files at different energies by simply changing the beam energy value. The three lenses and lens 1 and 2 steering should scale close to the optimum value. Notice that the settings for lens 3 steering will have to be set manually by scaling to the new beam energy.

The D.C. tuning procedure is almost the same as the high energy D.C. beam alignment described above, with the exception of tuning the extractor electrode in order to maintain an approximately 3kV extraction potential. Set the Extractor voltage so that the Cesium beam energy plus the Extractor potential = 3kV. For example, if the Beam Energy is 1,000, set the Extractor to 2,000. Fine tuning of the lens 3 voltage does, however, require a different method to that described for 10keV operation. At beam energies suitable for high depth resolution depth profiling ($\leq$ 3keV), the column needs to be tuned with a sample bias as close to zero as possible.

A method of setting the lens 3 value is described below using SEM imaging.

SED-based method for tuning the low energy D.C. Cs beam

1. Use as a sample one of the coarse metallic grids in a sample holder, with no backing plate and no true sample. Imaging the coarse grid bars will give good image contrast.

2. Close the spectrometer gate valve as a precaution, since you do not need to use the mass spectrometer for this operation.

3. In -SIMS mode, turn the Cesium beam to DC using the DC button at the bottom of the Cesium page in Hardware Utilities. Select Cesium Beam under "Deliver Beam to Sample" and select the OK button. Now set the following:
     -The sample bias to approximately -200 V.
     -The immersion lens to 550.
     -The high mass blanker to 7.

4. Set the cesium raster size to 1,000 µm, and increase the SED gain until you begin to see an image. Fine-tune the immersion lens and the high mass blanker voltages for the most intense and uniform image intensity. It should now be possible to focus lens 3 for the best grid image.

5. Wobble the lens 3 voltage by approximately 50 V and minimize the translation of the image with the lens 3 (quadrupole) deflectors.

6. Switch the video so that the optical view is on the monitor, and position the sample so that a recognizable part of the grid is in the center of the light optic's field of view (closely corresponding to the center of the spectrometer's field of view during analysis).

7. Switch the video to display the SED image once again and adjust the Cesium beam x and y position voltages to image the same field of view as the light optics. While this will not necessarily position the cesium beam exactly, it will bring it very close. More accurate alignment of the LMIG and cesium probes is described in the 'Interleaved Depth Profiling' section.

8. Once the best focus is obtained, press the DC button again to blank the beam. Set the sample bias and immersion lens to their original values. Resave the instrument file.

Dual Source Column Option

General Description

Some TRIFT systems are equipped with a dual source ion column for depth profiling. The dual source column consists of the Cs gun as described above, along with the addition of a duoplasmatron ion source for producing an oxygen ion beam. In general, the Cs^+ ion beam is used for –SIMS depth profiling while the O_2^+ ion beam is used for +SIMS depth profiling. In many cases, the O_2^+ beam is used together with the oxygen leak valve to ensure full oxidation of the material being depth profiled.

The duoplasmatron source is mounted in-line on the end of the column while the cesium source is mounted at a 45 degree angle to the column. The Cs^+ primary ion beam is steered around the 45° bend with an electrostatic sector in order to bring it down the optical center of the column. A schematic of the dual source column is shown in Figure 4-13 below.

The column should be tuned up first with the O_2^+ beam with the ESA's set to 0 volts. The column is tuned up in the normal way as described above for the cesium ion beam. Once the column is tuned up for the oxygen beam, Lens 1, Lens 1 steering, and the ESA's can be set for the Cs^+ beam to bring it down the same path through the column. A description of the duoplasmatron source is given below.
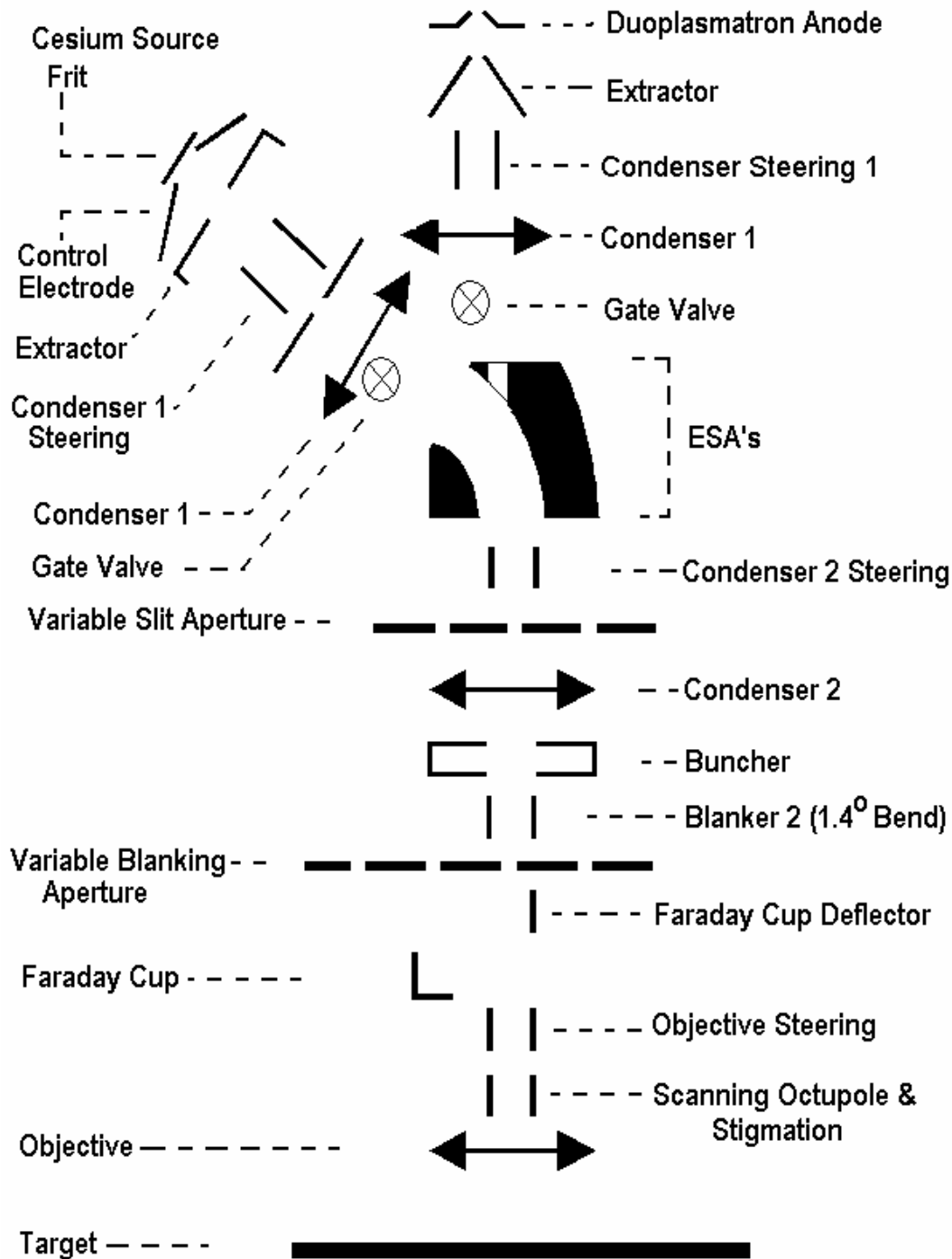
Figure 4-13.   Dual Source Ion Column Schematic.

Duoplasmatron Ion Source

General Description

The duoplasmatron (DP) source has four primary components. They are the cathode, intermediate electrode, anode and coil. The DP employs a cold cathode ion source that operates by striking and maintaining an arc, creating ionized gas species. A gas is introduced into the source (typically oxygen, but can also be argon, nitrogen, or xenon) as a high voltage potential (approximately 4,500 V D.C.) is developed between the cathode and the anode. An electrostatic arc is struck between anode and cathode, and sustained via a constant current power supply. Typical source operation occurs with ~60 millitorr of gas pressure and ~60 milliamps of arc current. The anode aperture and coil work in tandem to constrict the beam and contribute in great part to the small beam size and high source brightness. The "duo" in duoplasmatron stems from this dual beam constriction.
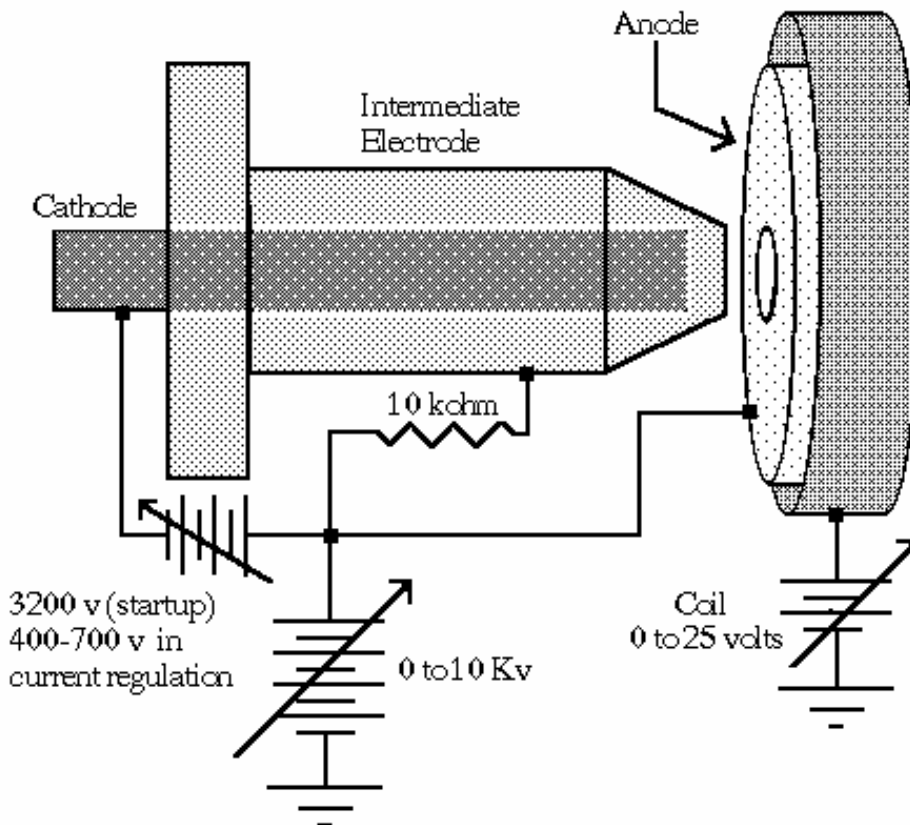
Figure 4-14.   *Schematic diagram of the duoplasmatron ion source.*

The arc supply floats on the Beam Voltage supply, and runs in a constant current mode. With no gas in the source the supply will go to the maximum permissible voltage (the read back will display around 4,500), Next, the voltage on the oxygen source valve will be increased automatically. The valve opens to let gas in to the source, driving down the arc voltage to the value entered in the "ArcVoltage" value in the 'Sources' page. Once the pressure is high enough to cause an arc, (dropping the voltage from 4,500 to around 460). The control will then start regulating the gas pressure by raising or lowering the voltage on the valve in order to maintain a constant arc voltage set in the menu.

Source Operation using the Model 20-740 Source Controller

The Sources Tabbed Page Menu

A number of Duoplasmatron source control parameters are used to control the functions within the source. These parameters are listed in the table below with a brief explanation of their function.

Table 4-2.   *Control parameters and functions.*

Parameter:	Function:
O2 ArcVoltage	This is the voltage that the control will try to maintain across the arc (measured in volts).
O2 ArcCurrent	This is the constant current that you wish to maintain in the source (measured in mA).
O2 CoilCurrent	This regulates the current through the coil.
O2 GasControl1	This is the applied voltage on the gas valve when the Duo_PID_ValveSelect is set to 0.
Duo_PID_ValveSelect	This parameter determines whether the gas valve is under computer control (set to 1) or under manual control (set to 0).
Duo_ProportionalGain	Controls how hard or how fast the valve will ramp up until it opens.
Duo_DerivativeGain	As above.
Duo_IntegralGain	Controls how fast & how much it varies the voltage on the valve to maintain the Arc Voltage set in the O2 ArcVoltage.
Duo_IntegralLimit	As above.

Starting values (first time firing)

*Note: The values and steps listed below should be used any time after the
following conditions to verify that the valve characteristics have not changed:*
- anytime that a new Piezo valve is installed; or
- just after a bake.

1. Start with a zero volt (zerovolt.ins) instrument file.

2. Verify (within Watcher) that gate valve V4 is open and that the Duo Source
    is being differentially pumped. **There is no safety interlock.** Also insure
    that the isolation valve is open on the ion gun column.

3. Before starting to let in gas, measure the O2 ArcVoltage. It should be in a
    range between 4,000 to 4,500. If it is around 500 – 700, an Intermediate-to-
    Cathode short exists and the source will require cleaning. This is a very
    unambiguous diagnostic.

    Similarly, the O2 ArcCurrent readback should be around 1 – 4 (without
    oxygen gas flowing), if it reads 60, there is a short (probably caused by a
    flake of oxide).
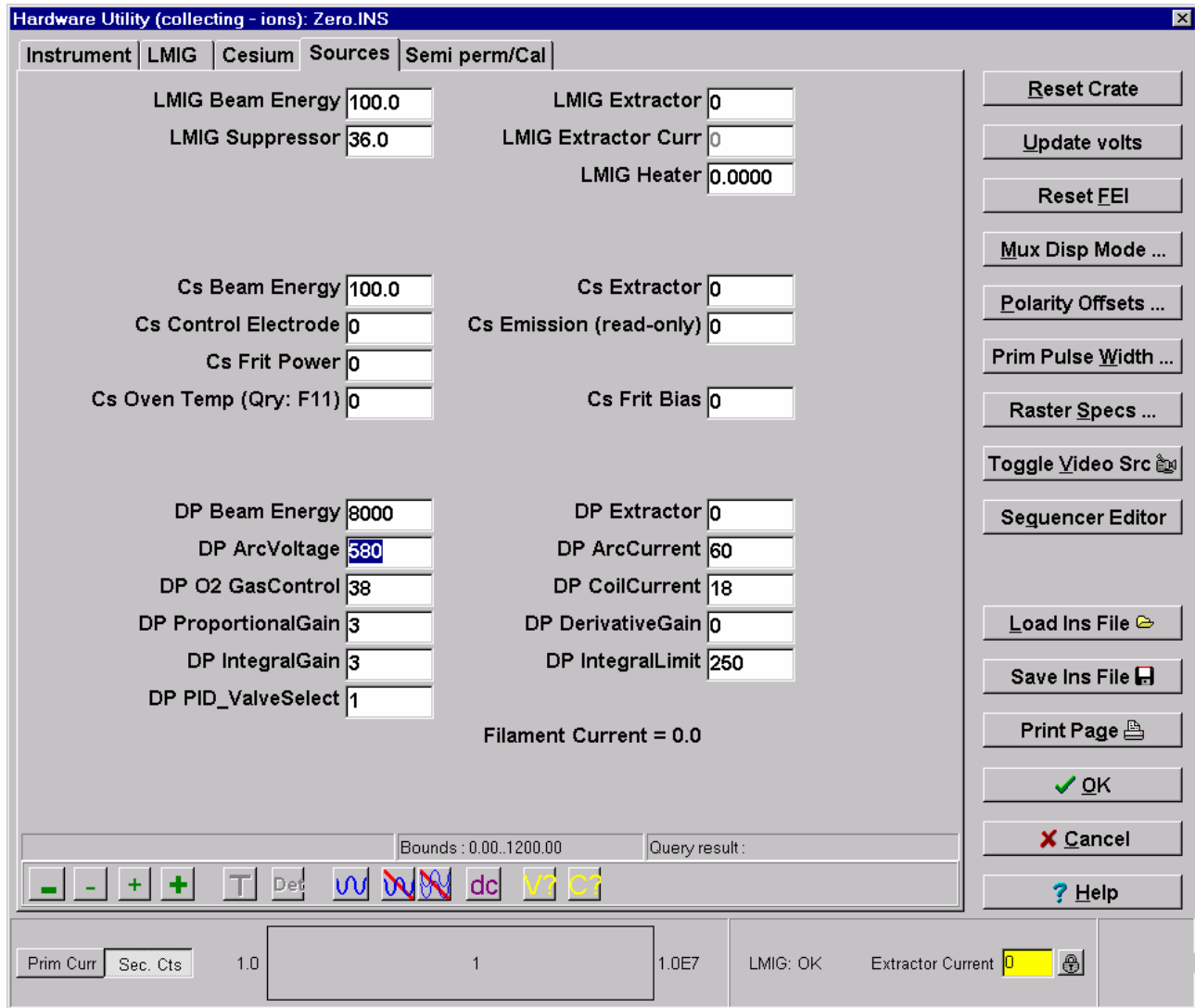
Figure 4-15.   *Illustration of the Sources tabbed page within the TRIFT's WinCadence Hardware Utility menu including typical DP operating parameters.*

4. Now, slowly start raising the O2 GasControl1, watching the roughing line monitored by the Granville Philips gauge. As soon as a slight increase in pressure is observed, note this value of the gas control. (The value that will be saved with the instrument files will be this value minus 15). Continue raising this value to start the arc by measuring the O2 ArcVoltage. Once the arc is struck, this voltage will be around 400 – 460. (If the value is ~ 1200, the anode is open in the source).

5. Now set the O2 ArcVoltage to around 480 – 490. Then set the Duo_PID_ValveSelect to 1. This will turn control of the valve over to the source electronics module. It will control the pressure to maintain the Arc Voltage that you had entered.

6. Now set the O2 GasControl1 to the value from step 3 (value of just opening minus 15 volts).

7. Record these values. Change polarity and enter them on the other side of the instrument file, (in case polarities are changed accidentally, the arc will not be lost). Now save this Instrument file as 'StartUpDuo.ins', or some other clearly named file. This will result in a good file to get the source going. It will still be running a gas load of approx. 100 – 120 millitorr in the source, but should start the arc and go into pressure regulation fairly quickly.

Normal Source Operating Condition

Best Starting Conditions

We have just discussed how to create a file with duo parameters that allow the source to start easily. Unfortunately, such a duo condition will result in a poor probe, no matter how hard you try to tune the ion column subsequently. Consequently, the source will start with a lower arc voltage, but then the arc voltage will need to be raised to a higher value, as discussed in the next section.

Best Probe-Forming Conditions

For best performance (source brightness), it is well known that a duoplasmatron should be run 'lean', i.e. with a lower gas pressure. On a well-conditioned source with a nickel cathode, running a value of around 570 – 600 for the arc voltage should be close. The higher the voltage you set, the less will be the gas pressure. (refer to following chart in Figure 4-16 below). A parameter value is required that is not set too close to a level at which the source will lose its arc.

Invest a few moments and perform the following experiment: on a conditioned source, raise the arc voltage in 10 volt steps, monitoring an ion beam into the column Faraday, or frequently reading back the arc voltage. Raise the arc voltage until the arc is extinguished. A good operating arc voltage will be ~50-75 volts *down* from that extinction value. Verify that you have a stable source at such a value, and then save an instrument file with a suitable name, such as 'BestDuo.ins' or some such name.

Start-up versus Best Operation

One strategy that is implied by the above discussion is to load the 'StartUpDuo.ins' instrument file to start up, allow the source to stabilize, and then raise the arc voltage up to the value determined from ***Best Probe-Forming Conditions*** section above, for normal operation.

Alternatively, the 'BestDuo.ins' instrument file could be loaded and then go to the Source tabbed page of the Hardware menu and lower the arc voltage to the start-up value.
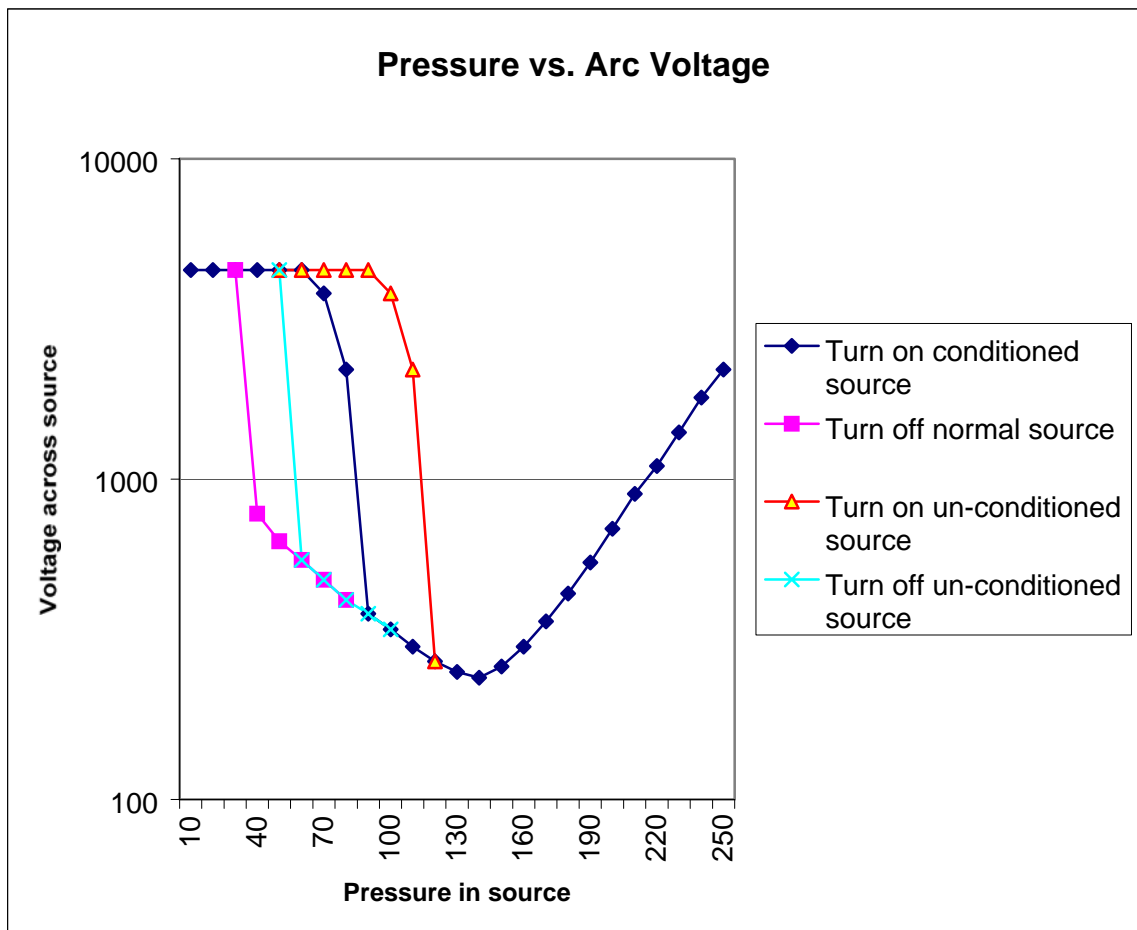
Figure 4-16.   Diagram of the Correlation between Pressure in the Ion Source and Arc Voltage.

The Electron Neutralization Gun

Important features of the neutralization scheme employed in the PHI TRIFT III TOF-SIMS system are its simplicity of operation and the infrequency of tuning. There are no front panel tuning knobs, and the parameter variations that are available within the WinCadence software program rarely require any adjustment. The benefits of this scheme are that, under most experimental conditions, the only requirement is to remember to select "Analytical with chg. comp" within <u>A</u>cquisition setup in WinCadence.

There are, however, circumstances in which the tuning of the e-gun will require checking. These occur:

- After replacing the filament;

- If a deterioration in the neutralization performance is detected; or

- If system performance is to be monitored on a routine basis.

Testing for Filament Current and Filament Continuity

The filament current to the electron gun is applied either by:

- Setting the value of Egun Filament Power in the Instrument page of the Hardware menu to some value such as 4.5 (0-6 max), or

- Selecting "Analytical with chg. comp" in the acquisition setup menu. In this latter case, a default value stored in the registry is used.

If a sudden deterioration in the neutralization performance is detected, the most likely cause is loss of filament current, probably due to filament failure.

To determine if the filament is intact, check the readback voltage (V) for the E-gun Filament Power on the Instrument page of the WinCadence Hardware menu. Note that the E-gun Filament Power is normally set to 4.5 (for the "On" state of the E-gun) and that this should be the readback value if the filament is intact.

Setting the Correct Filament Current

The curve of electron emission current versus filament current for a thermionic emission source is 'S-shaped', exhibiting three major regimes:

- No electron emission: temperature of the filament insufficient for thermionic emission.

- Rapid rate of change of electron emission with filament current: temperature for onset of thermionic emission exceeded.

- Decreasing amounts of extra electron emission for incremental changes in filament current: the filament is said to be "saturated", owing to 'space-charge' effects.

The correct filament current setting occurs when the curve of electron emission versus filament current has just "turned over", e.g., just as the filament begins to 'saturate'. If the filament current is higher than this, the filament's lifetime is significantly reduced for no significant gain in electron emission. If the filament current is less than this, small fluctuations in current result in large variations in the electron emission.

Figure 4-17.   Illustration of Ion Gun Emission Current v.s. Filament Current.

The correct filament current for the tungsten filaments used in the electron gun is typically 2.4 amps, or a measured filament voltage of ~1.2 volts. The actual value may vary slightly from this, and should be determined by the saturation curve.

Measuring/Maximizing Pulsed Electron Current

This section outlines the steps that are necessary both to measure and then maximize the pulsed electron current to the analytical area. If for any reason the factory-set values must be changed, record these factory settings first so they can be returned to the original values if necessary. For this next stage, measurements need to be made with a Faraday cup.

A Faraday cup is provided in the Sample Mounting Kit, but one can also be made. To make a Faraday cup, cut two standard length microscope slides so that both fit horizontally within the single window sample holder. Attach the large window front plate as described in Section 2. Use four Cu-Be sample clips to hold the glass slides in place against the window. Next, cut a strip of aluminum foil with dimensions 4mm x 50mm. Fold the strip to produce a more rigid strip with dimensions 2 mm x 50 mm. Insert the strip in the gap between the two glass slide, and with the aid of tweezers, fold the strip to produce a square with dimensions 2 mm x 2 mm on one of the glass slides. The remainder of the metal strip should now be behind the glass slides. Now connect this tail to the sample carrier by trapping it with a fifth sample clip, in the center of the holder. Refer to the schematic diagram below. For 200mm systems (which has no 50mm single window sample holder), take a 150mm or 200mm wafer and place a clean, adhesive-backed plastic square, approximately 50mm x 50mm, at the center of the wafer. Cut out a 2mm x 2mm square of the plastic in the center of the wafer. This serves very well as a Faraday cup sample for electron gun alignments for the 200mm instrument. Refer to Figure 4-18 below.

To monitor and/or maximize the pulsed electron current to the Faraday cup, proceed as follows:

1. Load the Faraday cup assembly described above and centralize the aluminum square within the optical field of view.

2. Select "Analytical with chg. comp" in Acquisition Setup to ensure the filament is ON.

3. Set the instrument for Negative SIMS.

4. Set the sample voltage to 0 (zero). Lower the LMIG Extractor to turn off the filed emission so that the LMIG extractor current reads 0 (zero).

5. Remove the high voltage cable to the sample stage and connect an electrometer using the PHI-supplied lead which has an MHV connector at one end for the stage and a BNC connector at the other end for the electrometer.
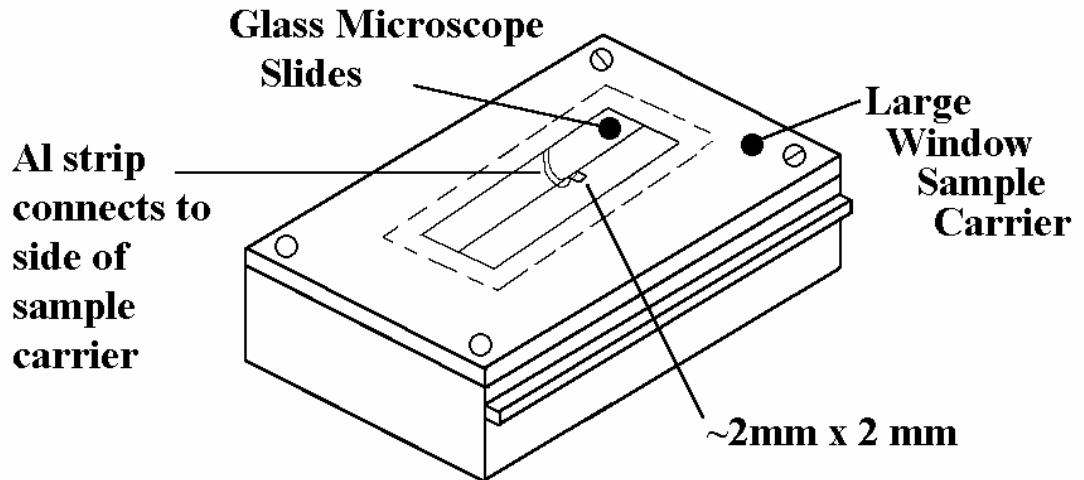
Figure 4-18.   Schematic of the Faraday cup assembly for 50mm TRIFT systems.

> *NOTE: Do not use a BNC-BNC lead, as this will spread the metal legs of the connector at the stage. In time this can result in arcing and corrosion of the stage high voltage connector.*

6. To obtain a pulsed electron beam, set a "dummy" acquisition running by requesting a new acquisition.

7. If no electron current is measured, first check the range on the electrometer. Begin by adjusting the Egun lens and the Egun x and y deflection potentiometers in sequence to maximize the pulsed electron current. These parameters are found in under Instrument, Hardware Utility, Semi perm/Cal tab. Continue the adjustments until the maximum has been determined.

8. The absolute value of the pulsed current depends upon (a) the steering of the beam and (b) parameters in the sequencer menu which determine the "duty cycle" of the electron gun (time on-to-time off ratio). A value greater than 40nA is typical for the pulsed current in negative SIMS. If after maximizing all parameters 40nA or greater cannot be achieved, consult PHI field service.

9. To stop the electron gun pulsing, stop the "dummy" acquisition.

This concludes the alignment of the electron beam to the analytical area. Once the steering and focusing of the electron gun has been set to maximize the pulsed electron current, no changes should be necessary unless the filament is replaced. This procedure is conducted during check-out at Physical Electronics and is not required to be re-done.
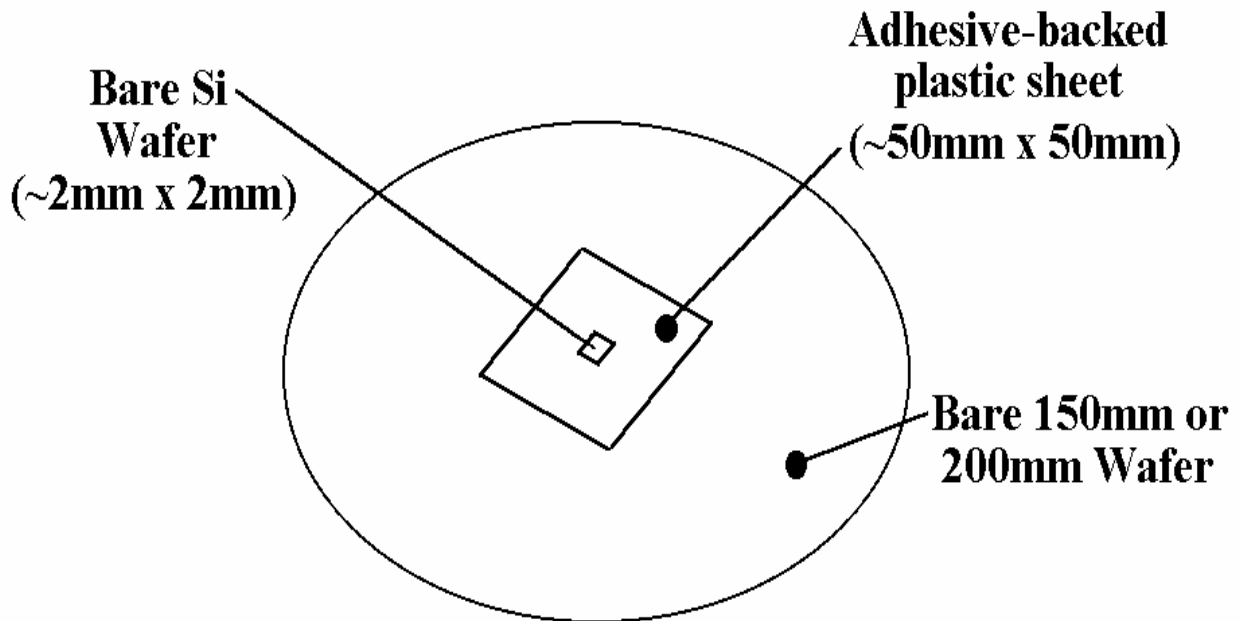
Figure 4-19.   Schematic of the Faraday cup assembly for 200mm TRIFT systems.

Sputter Gas Gun

The TRIFT III instrument is delivered from the factory with the following Gas Gun instrument files:

Filename	Description
GasGun_5kV_Max.ins	5kV, 3uA max sputter current file
GasGun_5kV_Min.ins	5kV, minimum beam size file
GasGun_5kV_1uA.ins	5kV, 1uA sputter current file
GasGun_2kV_400nA.ins	2kV, 400nA sputter current file
GasGun_1kV_200nA.ins	1.5kV,-500Float, 200nA sputter current file
GasGun_500V_100nA.ins	1kV,-500Float, 100nA sputter current file

These files have been saved as Gas Gun only instrument files and are meant to be filter loaded with a desired primary gun file to customize the desired profiling performance. Please refer to Section7 Dual Gun Profiling.

This section describes the operation of the sputtering ion gun, which can be used to sputter clean samples and to perform "phase depth profiles". Included will be information needed to tune and operate the ion gun efficiently for best results and to prolong the lifetime of the ionizer/filament. The subsections in this section include:

Gas Gun Overview

Gas Gun Operation

Conditioning the Gas Gun Ionizer and High Voltage

Setup and Calibrating the Argon Gas Regulation

Creating New or Tuning Existing Gas Gun Parameters

Gas Gun Ionizer Shutdown

Gas Gun Overview

The 06-360 is a low energy argon sputter gun. The ion gun has been developed for high depth resolution, rapid sputtering and low impact energy. It can be used as an alternative to the micro-focusing (duo/cesium) column in "phased depth profiling" applications. The Gas Gun does not operate in a pulsed/bunched mode and will not be used as the primary spectroscopy gun.

The ion source is a hot filament, electron impact ionizer that is configured for ionizing argon. The argon ionizer uses a tungsten filament to operate the ionizer at 15mA emission, while a pneumatic controller and flow restriction aperture delivers a source pressure of 15mPa. New ionizers should be conditioned prior

to operating. An adjustable grid voltage is provided to optimize ionization and extraction of ions from the source into the gun column. The Gas Gun is differentially pumped by the turbo pump mounted to the condenser section of the gun column.

The ion gun has a continuously variable energy range from 0-5keV. At low beam energies (impact energies < 1keV) the ion column is "floated" up to -500V to improve beam transport through the ion column. A condenser lens focuses ions extracted from the source to a position at or before the objective (pressure step) aperture. The objective lens focuses the beam onto the sample. The higher the condenser lens setting, the smaller the spot size and smaller the sputter current. With varying condenser lens settings, a slight adjustment is required of the objective lens for best focus.

To blank the beam and also prevent neutrals from hitting the sample, a set of deflector plates are located just upstream of the objective aperture to deflect the beam around a 5º bend. During sputter operation, the blanking voltage is optimized to steer the beam through the bend and through the center of the objective lens/octupole region. During blank operation, the condenser lens is automatically set to provide a crossover of the beam at the blanking plates and the blanker voltage is automatically set to deflect the beam into a beam trap to minimize column current from reaching the sample.

A set of octupole deflectors are located at the end of the ion column to scan and shift the beam across the sample.

The Gas Gun housing is equipped with a set of X/Y port alignment adjustor. These are used to mechanically align a non-deflected beam to the center of the spectroscopy position of a sample, when the sample is at "zero" potential.

Gas Gun Operation

All the optical elements in the ion column are controlled from the "Gas Gun" page of the Hardware Utility window: (WinCadence: Instrument, Hardware Utility, Gas Gun page). Additional parameters for the blanking operation and ionizer usage are found on the "Gas Gun" page of the WinCadence Options window. The sputter raster pattern during external raster operation is found on the Raster Specification window. The following page contains examples of WinCadence menus associated with the Gas Gun.
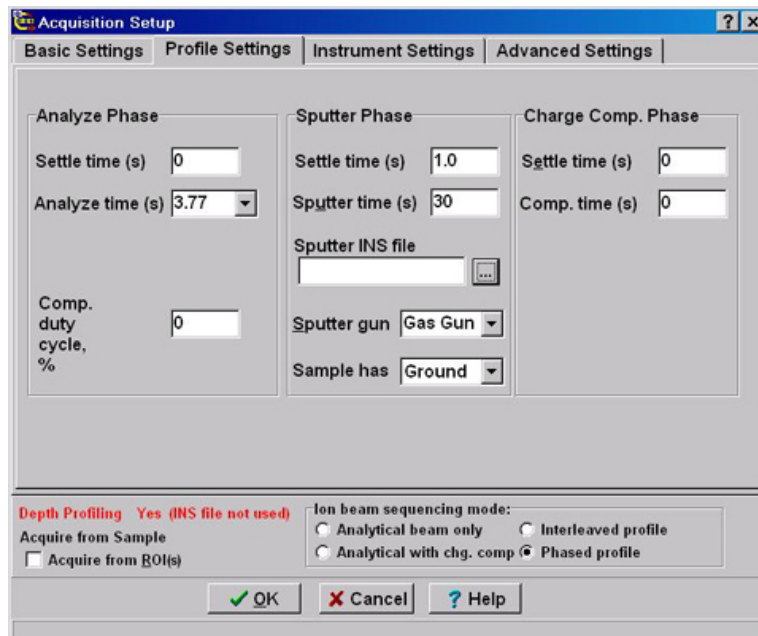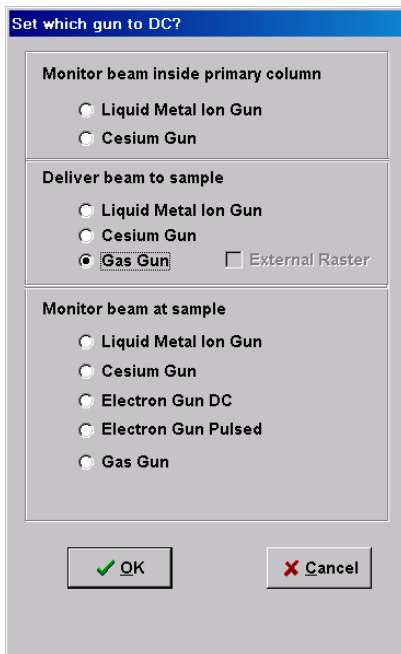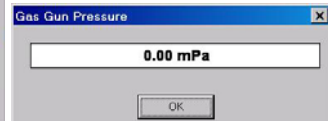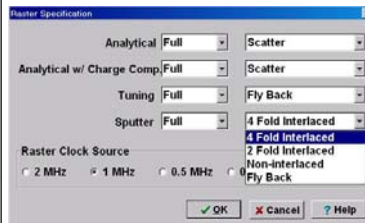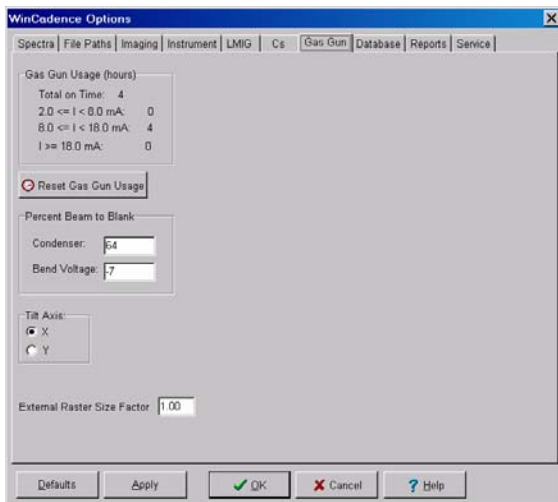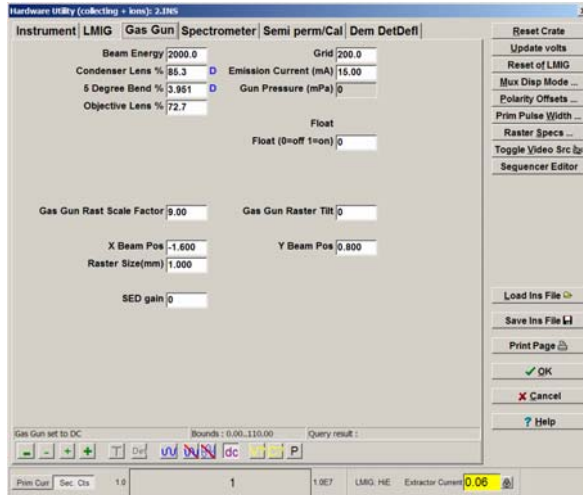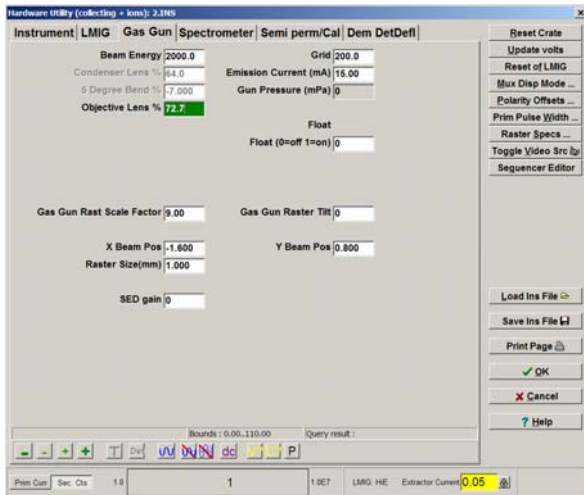
- ▪ **Emission Current (mA):** This value determines the voltage applied to the ionizer filament to maintain this target emission current. The recommended value of 15mA will extend the life of the filament and provide required emission from the source. Higher values may provide additional emission current but will greatly reduce the life of the filament. To read the actual current, use the F10 function key on the keyboard while this field is highlighted.
  Note: This field is not saved with an instrument file. When an instrument file is loaded, the emission will need to be manually adjusted as well as manual operation of V10 to initiate the flow of Argon into the ionizer. Once this value has been entered, it will be used to regulate the ionizer until a new value or 0mA has been entered. This input will be incremented in 1mA steps by the software when a new value is entered.

- • **Grid voltage:** The grid voltage is set to 200 during calibration of the argon pressure control. In the DC mode, it is adjusted to maximize the sample current.   Typical settings are 120-160 volts in the DC mode. To read the actual supply voltage, use the F10 function key on the keyboard while this field is highlighted.

- • **Gun Pressure (mPa)**: This field is a readback only display of the pressure measured in the ionizer. This reading is accurate at 0.1kev Beam Energy with a 200v Grid voltage. During DC operation, lower Grid voltages and higher Beam Energies will appear to reduce this value as fewer electrons are available in the ionizing region and more ions are being accelerated from the ionizer. Clicking the "P" icon at the bottom of the Gas Gun window will pop-up an auxiliary window displaying the pressure reading and bar graph of pressure intensity. See section on calibrating gas flow.

- • **Beam Energy and Float:** The beam energy is used in conjunction with the Float to select the impact energy of the sputtering ions. Higher values of Beam Energy will deliver a smaller beam diameter with higher sputter rates. Higher values will also result in increased impact energy and reduced "depth resolution". Lower Beam Energies are desired to improve "depth resolution". With desired energies of <1keV, the Float supply can be used in conjunction with the Beam Energy to increase the efficiencies of the transport of the lower energy ions down the ion column. The actual impact energy can be determined by adding the -Float voltage to the +Beam Energy. To read either of the actual supply voltages, use the F10 function key on the keyboard while either of these fields is highlighted.
  Note: External rastering is not available if a Float Voltage is selected. The default is an internal triangular raster pattern.
  Note: When choosing a Beam Energy and Float value, keep the final impact energy => the Float value. Choosing a combination resulting in an impact energy < the Float value will alter the operation of the Objective Lens focusing.

- **Condenser Lens:** The condenser lens is adjusted to set the desired sample current for a given Beam Energy. Typically a value of 68-70% of the Beam Energy will maximize the sample current. Increasing this value towards 100% will reduce the beam size and current. To read the actual supply voltage, use the F10 function key on the keyboard while this field is highlighted.
  Note: Normally this field is dithered and a fixed value 64% of the Beam Energy is applied to optimize the beam blanking state. When the "dc" icon is chosen, this field will display the previously saved Condenser Lens value selected to set the sputter current. In addition, a "D" will be displayed next to the field to indicate the DC operation mode.

- **5º Bend:** The 5º Bend is adjusted to steer the beam through the center of the Objective Lens and Octupole; as well as to optimize sample current for a given Beam Energy. Typically a value of 3.5% of the Beam Energy will optimize operation.   To read the actual supply voltage, use the F10 function key on the keyboard while this field is highlighted.
  Note: Normally this field is dithered and a fixed value -7% of the Beam Energy is applied to optimize the beam blanking state. When the "dc" icon is chosen, this field will display the previously saved 5º Bend value. In addition, a "D" will be displayed next to the field to indicate the DC operation mode.

- **Gas Gun Rast Scale Factor:** This value is used to calibrate the Raster Size in the Y axis. This calibration can be accomplished at 5KeV operation [where it is easier to image] and will be accurate for all selected Beam Energies.
  Note: a slightly different value may be needed between +SIMS and – SIMS.
  Note: This parameter will also affect the X axis size, but the **Gas Gun Raster Tilt** will provide the final calibration of the X axis scanning.

- **Gas Gun Raster Tilt:** This value is used to calibrate the Raster Size in the X axis [due to the mounting angle of the sputter gun with respect to sample normal]. This should be optimized after the Y axis has been calibrated. This calibration can be accomplished at 5KeV operation and will be accurate for all selected Beam Energies.
  Note: a slightly different value may be needed between +SIMS and – SIMS.

- **X Beam Pos:** This value is used to steer the sputter pattern to coincide with the Primary Gun's analysis area on the sample. This value will need to be optimized for different trajectories in –SIMS vs +SIMS and float vs. non-float operation. A more + value will move the sputter position to the right on the sample as viewed on the optical camera or LMIG SED image.

- **Y Beam Pos**: This value is used to steer the sputter pattern to coincide with the Y position of the Primary Gun's Analysis area on the sample. This value will track the Beam Energy supply and will need minimal optimization with varying Beam Energies. A more + value will move the sputter position down on the sample as viewed on the optical camera or LMIG SED image.

- **Raster Size(mm):** This value when calibrated with the Scale and Tilt Factors will track the Beam Energy supply and will determine the size of the sputter crater. This value should typically be set to 4x the analysis area dimension.

Conditioning the Gas Gun Ionizer and High Voltage

The ionizer needs to be conditioned (to outgas the filament) whenever the Gas Gun column has been vented. Verify that differential pumping is on and that the chamber pressure is less than $1x10^{-9}$ torr. Verify V10 is closed.

Load the GasGunMaintenance.INS file.
Note: if this has not been created yet, perform the following: Create an instrument file filtering in a typical Ga setting. Set the Gas Gun Beam Energy to 100V, the Emission to 0mA, and the Grid to 200V. Save this as GasGunMaintenance.INS.

Select the P icon and monitor the pressure during this procedure. Set the Emission to 1mA. Wait for the pressure to recover to <0.5mPa. Increase the Emission in 1mA steps up to 15mA, keeping the Extractor Pressure reading below 50mPa. Wait for the pressure to recover to <0.5mPa before going to the next emission Current setting. After proper outgassing, the Extractor Pressure should reach a value below 0.5mPa with the Emission set to 15mA. Set the Emission back to 0mA.

Increase the Beam Energy to 500v. Monitor the Extractor Pressure and chamber pressure while increasing the Beam Energy in 500v increments up to 5000v. With the Beam Energy at 5000v, repeat this procedure increasing the Condenser and Objective Lens values to match the Beam Energy value of 5000v. If there is a jump in pressure, reduce the voltages and repeat the procedure.

Setup and Calibrating the Argon Gas Regulation

The gas handling system has been designed to provide a constant pressure delivered to the argon ionizer. Once it is manually calibrated, on/off control is provided through Watcher control of V10.
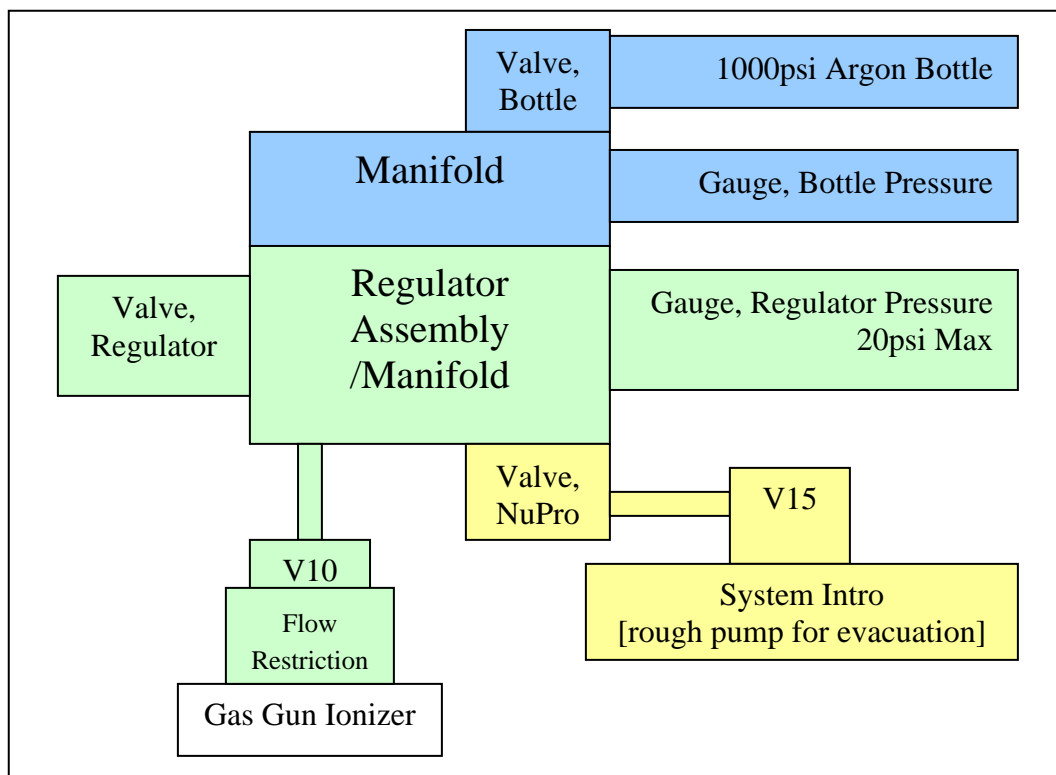
The gas handling system consists of a pressurized gas bottle (with gauge), a gas regulator (with gauge and regulator valve), a gas line and flow restriction aperture delivering a constant pressure to the Gas Gun ionizer assembly, and a manual isolation valve and rough line connected to the intro assembly through V15(for initial roughing of the regulator assembly).

Roughing Out the Regulator and Gas Line

In Watcher: Close V4 & V7 to isolate the rough pump from the system turbo. Close V3 to isolate the intro turbo pump from the intro chamber. Verify that V10, V12 and V14 are closed. Verify that the gas bottle valve is closed. Fully screw in (clockwise) the regulator valve to open it. Open the bypass manual roughing valve on the regulator assembly. Open V15 and V6 to pump out the regulator and manifolds through the intro assembly and rough pump. The regulator pressure should drop to -30 inches of Hg and the Intro pressure reading should drop below 1×10^{-3}.

Close the bypass manual roughing valve. Briefly open the gas bottle valve to re-pressurize the regulator and gas line. The regulated pressure will be 20-30PSI. Open the bypass manual roughing valve on the regulator assembly. Pump out the regulator through the intro assembly. Once the Intro Convectron reaches <1e-^{3} Torr, close V6 and open V3 and V7. The regulator pressure should drop to -30 inches of Hg and the Intro pressure reading should drop below 1×10^{-3}.

Close the bypass manual roughing valve and V15. Verify that the pressure remains at -30 inches of Hg for 10 minutes. Open V4 to continue roughing the Main turbo. Turn the regulator valve fully counter-clockwise to close it. Open

V10 to pump out the small volume between the pressure step aperture and the Gas Gun. Close V10. Open the gas bottle valve.

Setting the Regulator Pressure for Ion Gun Operation

Load the GasGunMaintenance.INS file.
Note: if this has not been created yet, perform the following: Create an instrument file filtering in a typical Ga setting. Set the Gas Gun Beam Energy to 100V, the Emission to 0mA, and the Grid to 200V. Save this as GasGunMaintenance.INS.

Open V10 in Watcher. Set the Emission to 15mA. [Note: WinCadence will increment the emission in 1maA steps]. Select the P icon to monitor the pressure. (J11 on the back of the 11-066 Gas Control will also provide a 5mPa/V reading). Verify that the pressure is <0.5mPa before continuing. Slowly turn the regulator valve clockwise until the pressure reading is 15mPa or ~3V on J11. Allow the regulator and ionizer to stabilize for 1 hour and then readjust if necessary for a reading of 15mPa or 3V on J11.

Close V10. The pressure reading should drop to <0.5mPa. Open V10 and the pressure reading should stabilize at ~15mPa within 5 minutes. Close V10. Set the emission to 0mA.

Creating New or Tuning Existing Gas Gun Parameters

For "phase depth profiling", an instrument file containing both the LMIG settings and the Gas Gun settings is necessary. New instruments ship with instrument files for the Gas Gun for "phase depth profiling". This section is useful for setting up new low energy Gas Gun settings or for re-tuning the existing Gas Gun instrument files. A variety of Gas Gun depth profiling instrument files can be setup by using the filter load function to match the desired LMIG settings with the desired Gas Gun settings.

Setting new instrument files or tuning saved files:

To modify an existing "phase depth profile" setting; open an existing instrument file. To generate new Gas Gun files at different energies, filter load the desired LMIG settings to create a new instrument file OR open an existing "phase depth profile" instrument file and modify it.

The Gas Gun Raster Scale Factor and Gas Gun Raster Tilt have been calibrated and should not need adjustment. Notice: if changing to a new Beam Energy, the settings for Condenser Lens, 5º Bend and Objective Lens will have to be set manually by scaling to the new beam energy.

The following description starts with a discussion of how to tune the Gas Gun in a D.C mode. A schematic of the Gas Gun column with all its optical elements is provided in Figure 4-19 below to assist the operator in becoming familiar with these operations.
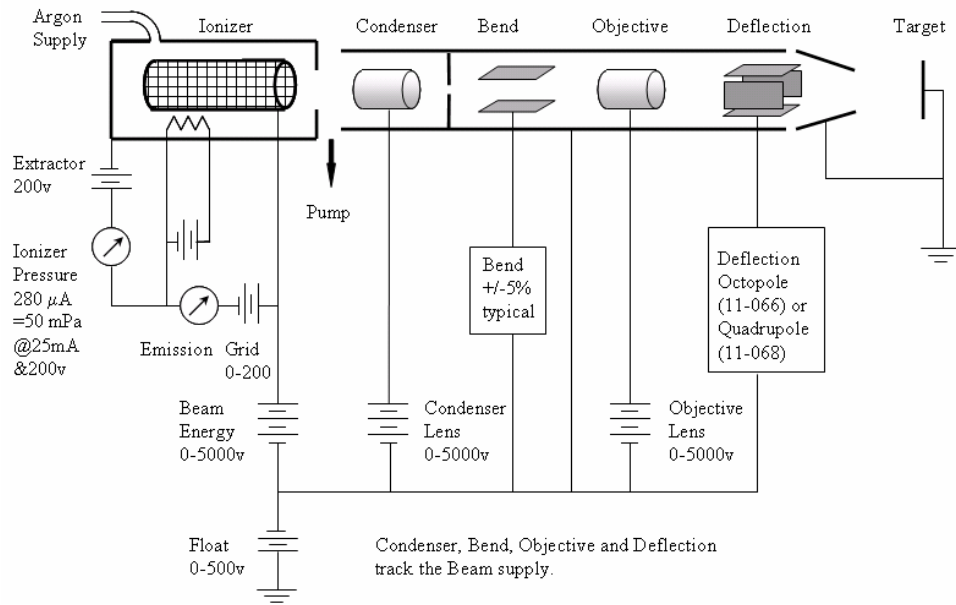
Figure 4-20.   Schematic Diagram of the Sputter Ion Gun.

D.C. Beam Alignment

File	Beam Energy	Float Voltage	Impact Energy	Grid	Bend 3.5%	Current	Condenser	Objective 73%
GasGun_5kV_Max.ins						3uA	68-70% 3400-3500v	
GasGun_5kV_1uA.ins	5000v	0v	5000v	110v	175v	1uA	80-84% 4000-4200v	3650v
GasGun_2kV_400nA.ins	2000v		2000v		70v	400nA	80% 1600v	1460v
GasGun_2kV_1400nA.ins						1.4uA	70% 1400v	
GasGun_1kV_200nA.ins	1500v	-500v	1000v	130v	52v	200nA	76-83% 1150-1250v	1095v
GasGun_500V_100nA.ins	1000v		500v		35v	100nA	75-85% 750-850v	730v

SED-based method for tuning the D.C. Gas Gun beam with 0v float

1. Use one of the coarse metallic grids in a sample holder, with no backing plate and no true sample. Imaging the coarse grid bars will give good image contrast.

2. Tuning is best done in –SIMS, which provides the optimum SED image for tuning the Objective Lens. Objective Lens values will then transfer to +SIMS settings later.

3. Close the spectrometer gate valve as a precaution, since you do not need to use the mass spectrometer for this operation.

4. In the Gas Gun page, select the desired Beam Energy and verify that the Float Voltage is set to 0v. Set the Emission to 15mA. [Note: if changing this value from 0mA, incrementally set the value in 5mA steps] Open V10 in Watcher.

5. Once ion emission has been established, it is possible to monitor D.C. beam current on the sample (with a picoammeter) The beam is set to D.C. in WinCadence on the Gas Gun control page by clicking on the "D.C." button at the bottom of the page. Select the Gas Gun button under "Monitor beam at sample".

6. Set the 5º Bend to ~+3.5% of the Beam Energy. Adjust the Grid voltage to maximize the current. Adjust the Condenser Lens for the desired sample

current at this Beam Energy. Maximum current will occur with a condenser setting of 68-70% of the Beam Energy. Increasing the Condenser between 70% and 100% will provide a smaller beam size and reduced sample current.

7. Turn off the DC operation

8. In -SIMS mode, turn the Gas Gun beam to DC using the DC button at the bottom of the Gas Gun page in Hardware Utilities. Select Gas Gun Beam under "Deliver Beam to Sample" and select the OK button. Now set the following:
    -The sample bias to approximately -200 V.
    -The immersion lens to 550.
    -The high mass blanker to 7.

9. Set the Gas Gun raster size to 2mm, and increase the SED gain until you begin to see an image. Fine-tune the immersion lens and the high mass blanker voltages for the most intense and uniform image intensity. Reduce the sample bias to minimize its deflection affect on the primary beam; but still provide a good SED image. It should now be possible to focus the Objective Lens for the best grid image. Adjust the Bend voltage to center the beam in the Objective Lens and minimizing wobble of the lens in the "Y" axis. This should coincide with maximum current. You may need to return to the picoammeter mode to readjust sample current with the Condenser Lens.

9. Switch the video so that the optical view is on the monitor, and position the sample so that a recognizable part of the grid is in the center of the light optic's field of view (closely corresponding to the center of the spectrometer's field of view during analysis).

10. Switch the video to display the SED image once again and adjust the Gas Gun beam x and y position voltages to image the same field of view as the light optics. While this will not necessarily position the Gas Gun beam exactly, it will bring it very close. More accurate alignment of the LMIG and Gas Gun probes is described in the "Phased Depth Profiling: Dual Gun" sub-section of Section7: Data Acquisition.
    Note: The following alignment section pertaining to low energy "float" setting alignment can also be used to optimize X,Y and Focus for these higher energy files.

11. Adjust the objective lens for best focus. Once the best focus is obtained, press the DC button again to blank the beam. Set the sample bias and immersion lens and high mass blanker to their original values. Resave the instrument file.

Method for tuning the D.C. Gas Gun beam with a float voltage

1. Use a piece of Silicon Oxide or Silicon Nitride.

2. Close the spectrometer gate valve as a precaution, since you do not need to use the mass spectrometer for this operation.

3. In the Gas Gun page, select the desired Beam Energy and associated Float Voltage (ie:1500V,-500V or 1000V,-500V). Set the Emission to 15mA. [Note: if changing this value from 0mA, WinCadence will incrementally set the value in 1mA steps] Open V10 in Watcher.
Note: When choosing a Beam Energy and Float value, keep the final impact energy => the Float value. Choosing a combination resulting in an impact energy < the Float value will alter the operation of the Objective Lens focusing.

4. Once ion emission has been established, it is possible to monitor D.C. beam current on the sample (with a picoammeter) The beam is set to D.C. in WinCadence on the Gas Gun control page by clicking on the "D.C." button at the bottom of the page. Select the Gas Gun button under "Monitor beam at sample".

5. Set the 5º Bend to ~+3.5% of the Beam Energy. Adjust the Grid voltage to maximize the current. Adjust the Condenser Lens for the desired sample current at this Beam Energy. Maximum current will occur with a condenser setting of 68-70% of the Beam Energy. Increasing the Condenser between 70% and 100% will provide a smaller beam size and reduced sample current.

6. Turn off the DC operation.

7. Switch the video so that the optical view is on the monitor, and position the sample so that an un-sputtered part of the sample is in the center of the light optic's field of view (closely corresponding to the center of the spectrometer's field of view during analysis).

8. Set the Sample voltage to 0v

9. Set the Gas Gun raster size to 0mm

10. In -SIMS mode, turn the Gas Gun beam to DC using the DC button at the bottom of the Gas Gun page in Hardware Utilities. Select Gas Gun Beam under "Deliver Beam to Sample" and select the OK button.

11. Adjust the Gas Gun beam x and y position voltages to align the sputtered area to the center of the field of view of the light optics. A more + X will move the sputter area to the right. A more +Y will move the sputter area down.

12. Adjust the objective lens for best focus or use table on previous page. Once the best focus is obtained, press the DC button again to blank the beam.

13. Set the sample bias back to its original value.

14. Set the raster size to 2mm or desired size.

15. Resave the instrument file.

Timed Sputter

To clean an area of the sample before taking an acquisition, perform the following:

Select Instrument/Sputter Tool

Choose the Gas Gun. Select a desired raster size (4 times the analysis area) and sputter time in seconds. Select the Start Sputter button. The sample bias during sputtering will be the value in the SPUTTER field on the WinCadence Hardware Utility, Semi perm/Cal page. This field should be set to 0.

Gas Gun Ionizer Shutdown

After using the Gas Gun for profiling operation, it is recommended to turn off the ionizer if it will not be used in the next 1-2 hours. This will help prolong filament lifetime.

1. On the Gas Gun page, set the emission to 0mA.

2. Close V10 in Watcher.

3. Load the zero.ins file

4. Load desired LMIG instrument file to maintain proper operation of the LMIG.

Section 5:
System Alignments

This section discusses the alignment process of key system components and stresses the importance of having all critical portions of the system in alignment with one another (i.e., beam steering, viewing optics, spectrometer, etc.)

Alignment of Primary Beam Analysis Area with SIMS Optics

Concept of SIMS Acceptance Area

In any SIMS instrument, a limited area exists on a sample from which secondary ions can be collected efficiently. In stigmatic imaging SIMS systems, this area is effectively determined by the de-magnified image of the detector at the surface of the sample, as a consequence of the magnification properties of the microscope. Even though the primary ion beam may be illuminating or scanning a large area, the area from which secondary ions are extracted is thus restricted or 'gated'. In the PHI TRIFT III spectrometer, the maximum SIMS acceptance area at the surface of the sample is ~600 microns at maximum secondary ion transmission. Larger fields of view can be achieved at the loss of some transmission, refer to 'Defocusing the Immersion Lens: Analytical Trade Offs' in Section 6.

Effect of Sample Height Variations

In the TRIFT system, the SIMS optics are normal to the sample surface, and thus the position on the sample from which secondary ions are extracted does not vary with z position (sample height changes). What <u>does</u> vary with sample height variations are the points of intersection of the primary beams and electron gun with the sample surface. A sample at the optimum z height is positioned to be at the intersection of the SIMS optical 'gate', the primary ion beams and the electron neutralization beam. When the sample height changes, however, the primary beam impact point moves by z.tan (theta), where z is the height variation, and theta is the primary beam impact angle with respect to normal incidence.

The exact primary beam impact angle at the sample surface depends on three parameters:

- The primary beam column orientation;

- The primary beam energy; and

- The sample potential.

In the TRIFT system, the LMIG and Cesium columns are set at 35° and 42° from sample normal, respectively. The Ga+ beam voltage can be varied continuously from +1 to +25 kV, and the Cs+ beam energy is continuously variable from approximately +2 kV through to +11 kV. Typically, however, the Cs+ beam is set at +10 kV and the Ga+ beam is usually switched between operation at +15 kV and +25 kV. The sample potential is likewise continuously variable between 0 and 5 kV (positive or negative), although it is typically set at 3 kV. If the sample is at a negative potential, then the primary beam is accelerated towards the sample, reducing the impact angle from the nominal 35° or 42°. The lower the primary beam energy, the greater is this effect. The converse is true when the sample potential is positive.

As a general rule, each variation of 1 micron in sample height (z) corresponds to a movement of the center of primary beam impact point of approximately 1 micron.
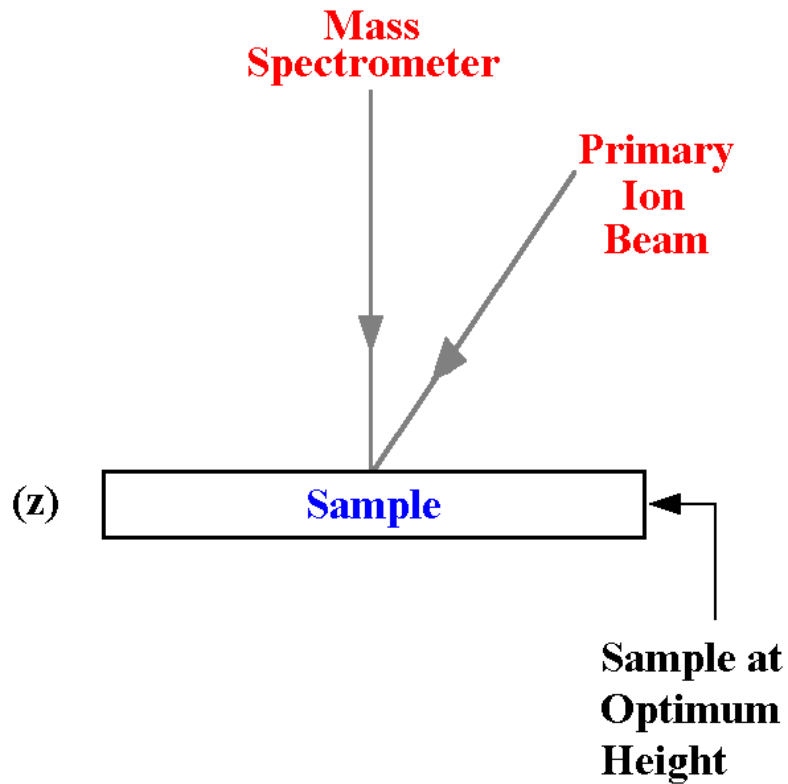
Figure 5-1.   Illustration of the Sample at the Optimum Height within the Analysis Chamber.
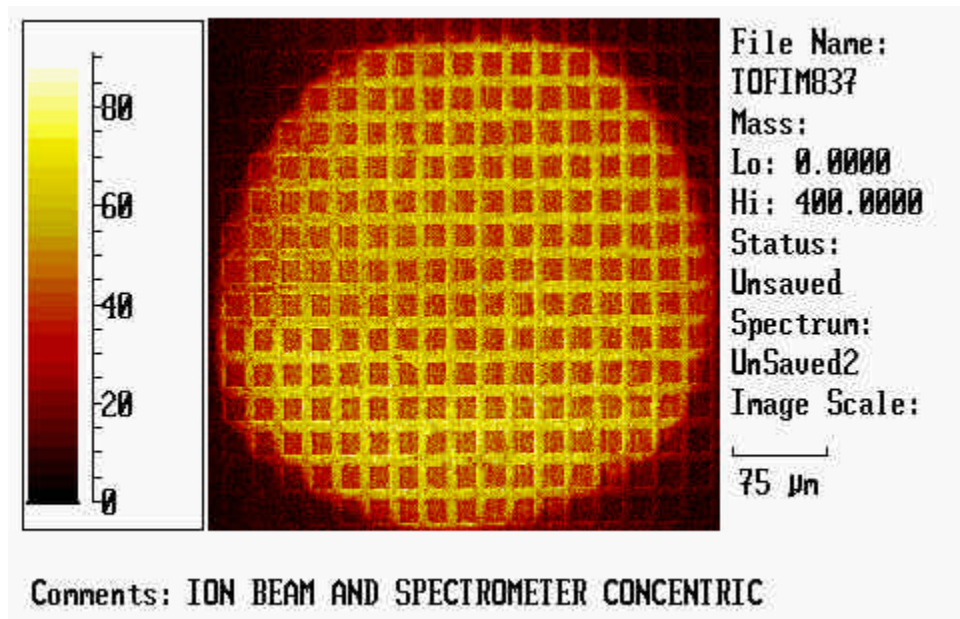
*Figure 5-2.   TOF Image of the Acceptance Area as seen by the Spectrometer with the Sample
Position at the Optimum Height.*
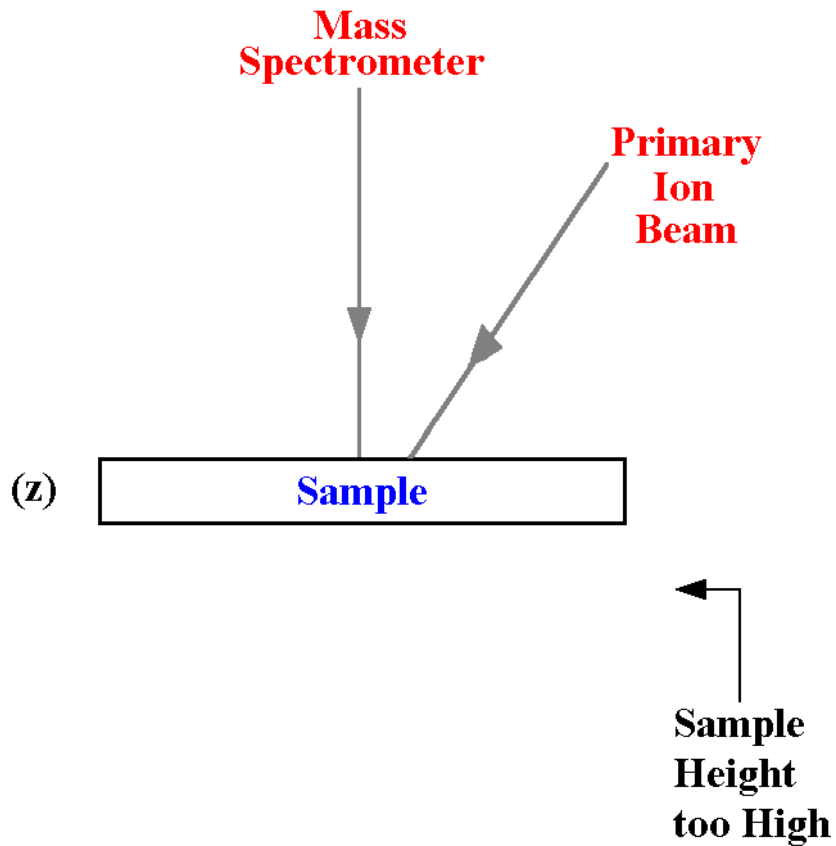
Figure 5-3.   *Illustration of the Sample Height being too High within the Analysis Chamber.*
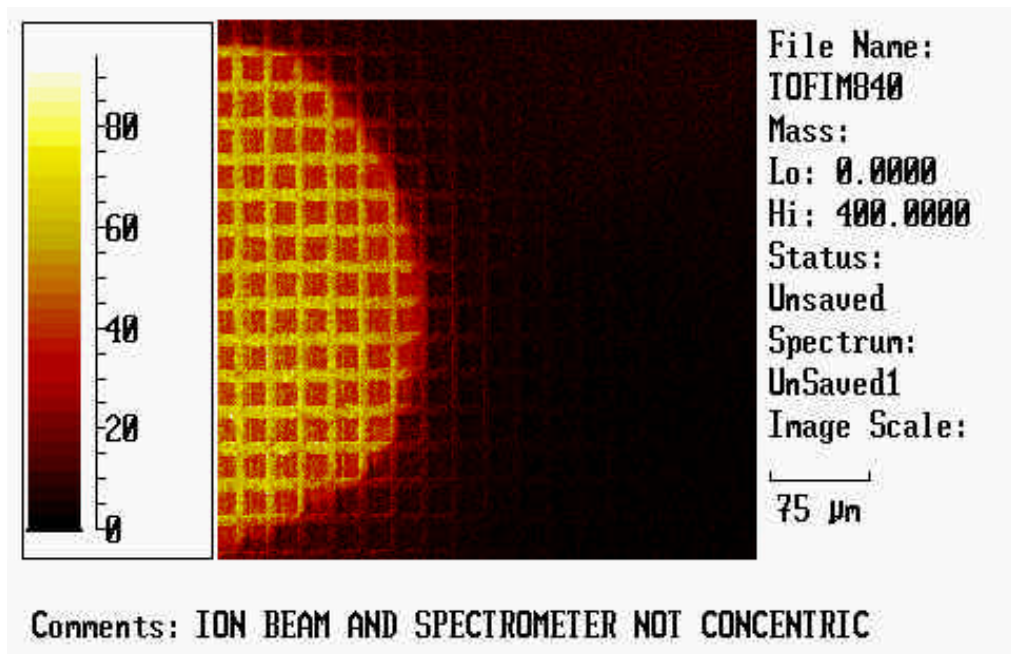
Figure 5-4.   *TOF Image of the Acceptance Area as seen by the Spectrometer with the Sample Position too High.*
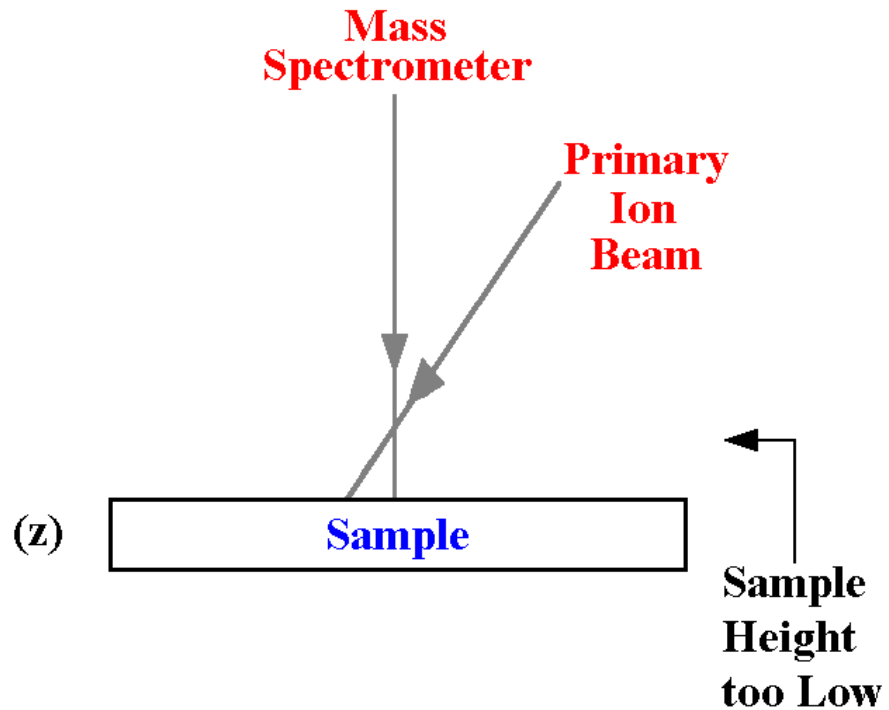
Figure 5-5. *Illustration of the Sample Height too Low within the Analysis Chamber.*
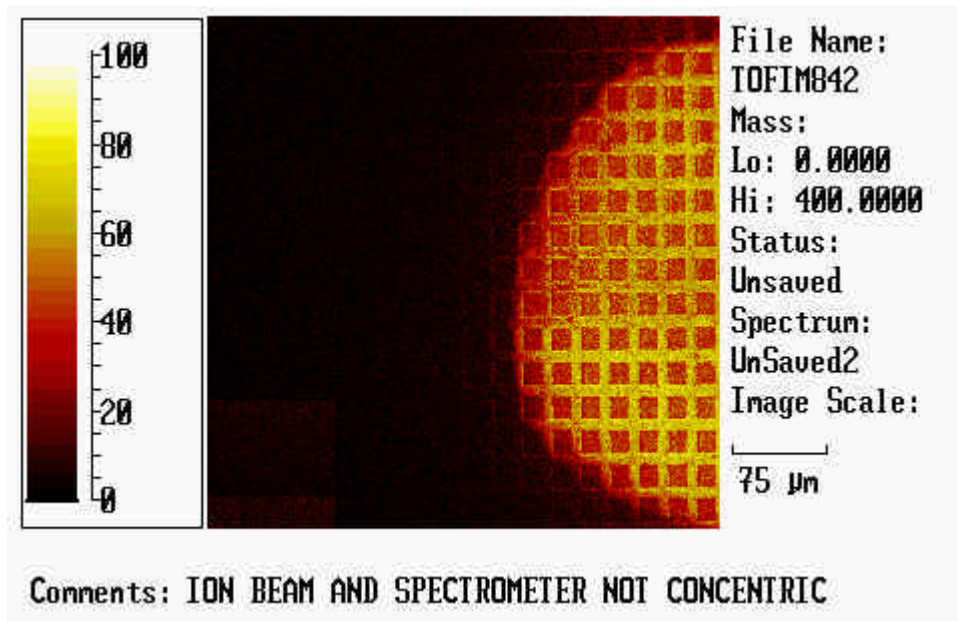
Figure 5-6. *TOF Image of the Acceptance Area as seen by the Spectrometer with the Sample Position too Low.*

Use of Primary Beam Steering (Beam Position)

The final deflectors in the primary columns of both the Cesium and the LMIG are used not only to raster the primary beams on the sample surface, but also to steer the position of impact of the primary beams on the sample surface. The raster signals effectively 'float' on top of D.C. steering voltages. These primary beam steering voltages are changed by altering the x and y values of "beam position" in the LMIG control page.

The usefulness of this beam steering should now be evident: it is possible to compensate for sample height variations by simply steering the primary beam until it is concentric with the secondary ion optical gate on the sample surface.

The distance moved by the primary beam per unit of "beam position" x and y varies linearly with the primary beam energy, since the available steering voltages are fixed (a beam of higher energy is less affected by a given steering voltage than a beam of lower energy). Typically, a 25 kV Ga+ beam will move approximately 50 microns in the x and y directions for every unit of beam position x and y. Since the beam positions values have ±10 units variation, this implies that the maximum height variation away from optimum that the system can accommodate electronically is ±500 µm, or a total of 1 millimeter. At lower beam energies this value is greater.

It is not difficult to mount samples to this positioning accuracy. If a sample is flat to begin with, then back-mounting against the sample holder window will automatically position the sample at the correct height. If the sample is not flat, employ one of the techniques outlined in Section "Sample Preparation Techniques", and use a low magnification binocular microscope to assist in aligning the z position by eye. Optical microscopes typically have a short depth of field, and it is quite easy to calibrate your microscope to determine if a sample is mounted within ±500um of the optimum z. Remember that a little care and attention to sample mounting saves considerable time once the samples are inside the analytical chamber.

Setup, Verification of Primary Beam Alignment to the Spectrometer

It is good practice to steer the primary ion beam so that it is concentric with the secondary ion acceptance area. This concentricity is vital for scanning a large field of view and yields good illumination of the entire area.

Verification of this alignment can be performed either in a TOF-SIMS imaging mode or directly on the secondary ion detector.

TOF-SIMS Imaging Mode

A simple way to set the concentricity of the primary beam and the secondary ion acceptance area is as follows:

1. Select a primary beam raster size that is significantly larger than the acceptance area, e.g., $\geq 1{,}000\mu$m.

2. Begin an acquisition and acquire a total ion image (automatic in WinCadence)

3. Now examine the total ion image. An image should now be forming of part or all of the secondary ion acceptance area. Once you have acquired sufficient signal, stop the acquisition.

Right click on the image using the mouse and select "center beam at cursor". Position the arrow at the center of the acceptance area and left click on the mouse.

This will adjust the beam position x and y values so that the primary beam will now be concentric with the acceptance area. Repeat the procedure, if necessary, and then reset the raster size to the desired analytical value.

Direct imaging on the secondary ion detector

It is even simpler to center the primary beam to the acceptance area using the image obtained at the secondary ion detector. To do this, set the video screen to monitor the detector, set a raster size of ~100μm, and begin an acquisition. Steer the primary beam x and y position so that the secondary ion raster is centered on the detector.

Alignment of Viewing (Light Optics) to SIMS Optics

The light optical field of view of the TRIFT is designed to provide a maximum magnification of ~600 at the video screen, with a field of view of ~450 µm and a lateral resolution of ~2 µm. The TRIFT III system is fitted with a variable field of view light optics, permitting ~1 mm field of view. For systems with dual-magnification capabilities of light optics, the alignment of light optics to SIMS optics should be done for the view of highest magnification (~450 µm). In Vacuum Watcher, the Camera Field option should be set to Small.

Alignment of Viewing Optics

The viewing optics, like the primary ion beams, are designed to be set concentric with the secondary ion acceptance area. This is achieved by small adjustments of an angled mirror at the base of the microscope assembly mounted on top of the analytical chamber. The procedure to set and verify this alignment is as follows:

1. Mount and load a sample into the system containing a unique feature that can be easily located within the optical field of view. The corner of the standard 25 micron Cu grid sample is perfect for this procedure.

2. Start an acquisition and using the X and Y beam steering, align the LMIG with the spectrometer acceptance area.

3. Turn the LMIG to D.C. and view the SEM image.

4. Move the sample stage until the corner of the grid (or some other feature) is centered in the SIMS field of view. Decrease the image size, adjusting the stage coordinates to keep the feature centered. When finished, blank the beam once again.

5. Center the feature in the optical field of view by adjusting the two diagonally opposed knobs which control the tilt of the angled mirror at the base of the optical microscope assembly. Rotate each knob until the corner of the grid (or other feature) is centered in the optical field of view.

6. The viewing optics are now aligned to the SIMS optics.

The procedure just explained aligns the viewing optics to the SIMS optics for <u>most</u> samples. Once again, however, large displacements of the sample in the z direction will result in some displacement of the viewing optics with respect to the SIMS optics. If considerable time is invested in the analysis of a sample which is clearly displaced in the z direction, it is often worth setting the viewing optics for that sample, since the procedure is simple. Remember to exercise caution if using a DC primary beam depending on the sample type. If a DC primary beam cannot be used, a standard SIMS image should be used to identify a feature and then set the viewing optics for that sample.

Alignment of + and - SIMS Fields of View (Polarity Offsets)

Primary Beam Steering Effects in Different Polarities

The sample potential causes the beam to steer away from the nominal impact angles of 35° and 42° for the Ga+ and Cs+ beams, respectively. When the sample potential is negative, the beam is steered towards the sample, and when the sample potential is positive, the beam is steered away from the sample. The exact steering values vary, but for a given primary column (LMIG or Cesium), a given beam energy and a given sample potential, the beam steering between negative and positive SIMS is always constant.

This is an important result, because the system software allows the user to apply a fixed polarity offset (delta x, delta y in the beam position) such that upon changing polarities, the feature in the SIMS field of view is always retained.

In WinCadence, the polarity offset values are determined at a specific energy for either the LMIG and Cs gun, and are stored with each instrument file. These values do not vary for a given set of conditions. Thereafter, whenever the polarity is changed, the polarity offset values will be applied to the current beam position values.

Determining the Polarity Offset Values

The procedure below determines the polarity offset values for a given primary species and experimental conditions (beam energy, sample potential).

1. Mount and load a sample into the system containing a unique feature easily located within the optical field of view. The corner of the standard 25 micron Cu grid sample is ideal for this procedure.

2. Locate the grid corner (or other feature) in the optical field of view.

3. Assuming that the viewing optics have been set correctly, switch to <u>negative SIMS mode</u>.

4. Center the primary beam raster to the secondary ion acceptance area as outlined in a previous section above entitled "Setup and Verification of Primary Beam Alignment to the Spectrometer".

5. Obtain an image using a DC primary beam, either at the SED detector, if using Ga+ beam, or at the DMCP detector if using the Cs+ beam. For details on how to do this, refer back to "Alignment of the Viewing Optics" within this section.

6. Zoom into the corner feature, <u>using stage adjustments only</u> to center the feature in the image. When the corner is centered within approximately 100 x 100 microns field of view, blank the primary beam.

7. Note the x and y beam position values. Obtain a printed hardcopy of the primary beam control page.

8. Change to positive SIMS. Select a large raster size and unblank the beam once again.

9. The grid corner will now be somewhere in the field of view. <u>Using changes to the beam position x and y only</u>, bring the grid corner into the center of the field of view. Zoom in until it is centered within approximately 100 x 100 microns field of view and blank the beam.

10. Note the new beam position values, and calculate the difference in going from negative to positive SIMS.

    Offset value = (+SIMS value) - (-SIMS value)

    These are the values that are entered into the polarity offsets in the instrument file.

The signs of the values are important, so double-check your calculation. For instance, if an x value in negative SIMS was -2, and an x value in positive SIMS was +3, then the entered value would be +5. [(+3) - (-2) = +5].

It's good practice to check the result by taking a positive SIMS image of the feature, changing polarities, and acquiring a SIMS image. **Hint**: use Cl- or CN- and Cu+ signals for the best contrast. This insures that the offset can be trusted at a later time on a real sample.

Section 6:
Tuning the Mass Spectrometer

This section is intended to give an introduction to the stigmatic secondary ion optics of the PHI TRIFT TOF-SIMS mass spectrometer and familiarize the operator with the most important ion optical features of the instrument. For a more detailed treatment of this subject, the operator should refer to the paper "Microscope imaging by time-of-flight secondary ion mass spectrometry", by B. W. Schueler in Microscopy Microanalysis Microstructures 3 (1992) p. 119-139.

Theoretical Aspects of Spectrometer Operation

The basic principles of TOF-SIMS are very simple. A pulsed primary ion beam bombards the sample surface, causing the emission of atomic and molecular secondary ions. The secondary ions are then electrostatically accelerated into a field-free drift region with a nominal kinetic energy of:

$$(1)\ E_{kin} = e\,V_o = mv^2/2,$$

where V_o is the accelerating potential, m the mass of the ion, and e its charge. Since lighter ions will have higher velocities than the heavier ones, they will arrive at the detector at the end of the drift region earlier than the high masses. The mass separation is thus obtained in the flight time *t* from the sample to the detector. This is given approximately by:

$$(2)\ t = L_o/v = L_o\,(\,m\,/\,2\,e\,V_o\,)^{1/2},$$

where L_o is the effective length of the mass spectrometer. Since different mass ions arrive sequentially at the detector, the operating conditions of a TOF-SIMS instrument can be chosen such that (virtually) all secondary ions of a given polarity are detected and recorded in the mass spectrum. This efficient collection and detection of secondary ions by TOF-SIMS instruments means that significant numbers of secondary ions can be produced from a sample surface with the minimum primary ion dose and results in a very sensitive technique for organic and inorganic surface analysis. Typical primary ion doses in this "static SIMS" analysis mode are $\leq 10^{12}$ primary ions / cm^2 for mass analysis and $\leq 10^{13}$ primary ions / cm^2 for imaging.

Neighboring masses m_o and $m_o + \Delta m = m_o (1 + \gamma)$ can only be resolved as such if their time width and separation Δt is sufficient. For small mass differences it follows from equation (2) that the relationship between flight times and mass resolution is:

(3) $m_o / \Delta m = \gamma^1 = t_o / 2\Delta t.$

It is obvious from equation (3) that in order to obtain high mass resolution a short primary ion pulse duration, t_p, has to be used since the smallest measurable time difference $\boldsymbol{D}t$ is t_p. The mass measurement in a TOF-SIMS instrument is achieved by converting the flight time into the exact mass by determining the flight time differences of ions through the spectrometer.

Physical Effects Influencing High Mass Resolution TOF-SIMS Analysis

Although TOF-SIMS is conceptually very simple, there are a number of physical processes which require a more sophisticated design for a TOF-SIMS spectrometer than merely having a long flight tube and fast detection electronics at the end. The two most extenuating phenomena are:

1. Secondary ions can be generated with non-zero initial kinetic energy and will thus acquire total kinetic energies which are larger than those imparted by the accelerating potential. For sputtered atomic secondary ions, the initial kinetic energy distribution is typically centered around 5 - 10 eV with a tail of the distribution extending out to greater than 100 eV. Molecular ions are ejected with lower mean kinetic energy and are distributed over a much smaller energy range. The narrower energy distribution is a result of the kinetics of the sputtering process: collisions which impart high kinetic energy to a molecule will also result in high internal energy, thereby resulting in molecular fragmentation. Only those molecules which have sustained "gentle" collisions will survive and be detected. Inorganic molecular ions are thus observed to have kinetic energies in the 1 - 5 eV range with distributions rapidly falling off towards higher energies. Generally, the more complex the detected molecule, the lower is its initial kinetic energy. Larger organic molecular ions, as the extreme, are typically emitted with kinetic energies in the low or sub-eV region.

2. Secondary ions are emitted from the sample surface with an angular distribution with respect to the surface normal. Ions extracted by the accelerating field may enter the mass spectrometer with angular momentum, i.e. kinetic energy, which causes them to diverge from the desired flight path through the mass spectrometer.

The TOF mass spectrometer must therefore not only separate ions according to their mass to charge ratio but must also compensate for the initial kinetic and angular distribution of the secondary ions, caused by the sputtering and ionization process. This task would be trivial if the influence of the very small acceleration section at the beginning of the mass spectrometer could be neglected (as this is usually done in the literature). The fact is that the flight time errors caused in the accelerating section are typically the limiting factor for the high mass resolution performance of any TOF-SIMS mass spectrometer. The following discussion refers primarily to the analysis of atomic secondary ions.

When ions are created at the sample, their velocity spread will be dominated by the initial kinetic energy distribution as they are just leaving the solid's surface. Ions with higher kinetic energy will spend less time in the accelerating space than ions that are ejected with near zero initial energy. As mentioned above, this effect is most pronounced for atomic/inorganic secondary ions and relatively unimportant (but not negligible) for higher molecular organic species. The flight time error caused in the accelerating space alone will be smaller the shorter the total acceleration time, e.g., the shorter the acceleration section and the higher the magnitude of the acceleration potential. For the case that the ions are ejected only in the direction of the spectrometer axis, the mass spectrometer can typically be tuned to provide the proper energy compensation over some range of initial ion kinetic energies. The TRIFT spectrometer is capable of compensating for the (axial) kinetic energy distribution ΔE of secondary ions over a range of $\Delta E/eU_o \geq$ 20% (e.g., ≥ 60 eV at 3,000 V acceleration potential).

As well as emission along the spectrometer axis, secondary ions will also have a distribution of emission angles. Again, the dominating flight time errors will be introduced within the acceleration space. It should be intuitively clear that an ion emitted with 10 eV kinetic energy in the direction of the spectrometer axis will have a shorter flight time through the acceleration section than an ion emitted parallel to the sample surface with the same initial kinetic energy. The TOF mass spectrometer will, of course, provide the appropriate compensation for the total kinetic energy but it cannot distinguish between different emission directions. In order to achieve high mass resolution, the angular acceptance of the spectrometer has to be limited (or otherwise corrected) so that the effect of flight time differences in the accelerating space is minimized. This effect is only noticeable in TOF spectrometers of high angular acceptance which provide the maximum ion transmission for atomic secondary ions where ultimate mass resolution is not imperative.

The Secondary Ion Focusing System: Stigmatic Secondary Ion Optics

A schematic diagram of the PHI TRIFT III is shown in Figure 6-1 below. A pulsed primary ion beam strikes the sample surface causing the emission of secondary ions. Secondary ions of a selected polarity are electrostatically extracted in the acceleration section and focused by the "immersion lens". Strictly speaking, the "immersion lens" extraction optics consists of (a) the acceleration section (sample surface and extraction plate), followed by (b) an Einzel lens (the extraction plate forms the grounded entrance aperture of the lens). Elsewhere in this manual a loose interpretation is used by calling the voltage applied to the Einzel lens of the extraction system the "immersion lens" Voltage. Strictly speaking, the sample bias is also a control voltage for the "immersion lens".
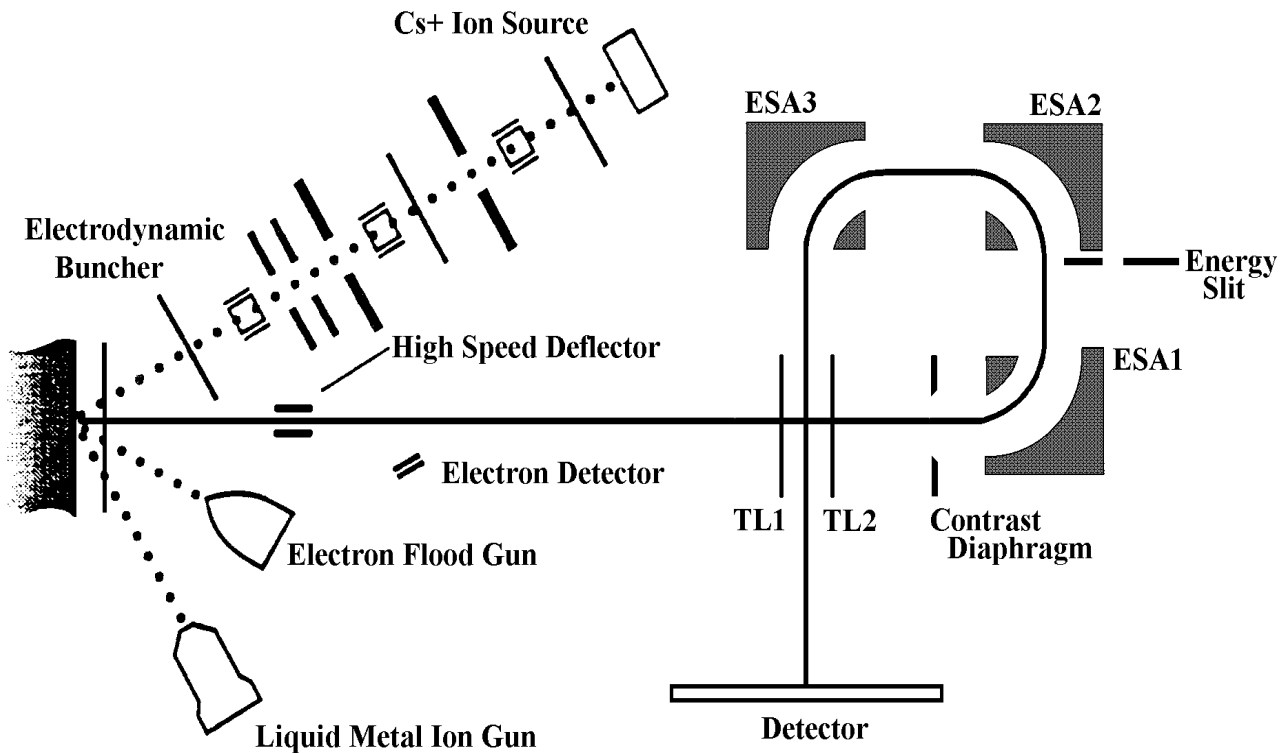
Figure 6-1.   *Schematic Diagram of the TOF-SIMS Mass Spectrometer.*

The immersion lens in conjunction with the transfer lenses determines the total image magnification of the secondary ion optics. A stigmatic imaging system of three 90° (quasi) hemispherical electrostatic analyzers (ESA) forms part of the secondary ion optics and provides "first order" energy focusing for imaging and ion flight times with small residual "second order" aberrations. The final, magnified secondary ion image is then projected onto an imaging secondary ion detector. The ESAs are fitted with field termination plates known as "Herzog shunts", and the center ESA is fitted with "Matsuda Plates" above and below for electrostatic field shaping.

The secondary ion mass spectrometer optics always projects a magnified image of the ion emission profile from the sample surface to an imaging detector. The ion arrival position at the final secondary ion detector is correlated with the emission point at the sample surface. A schematic diagram of the secondary ion image transport through the TOF spectrometer is shown in Figure 6-2. The extraction optics consists of the accelerating section formed by the sample surface and the extraction plate. The immersion lens projects a magnified image of the ion emission profile into the first field-free region of the mass spectrometer. This image is transported via the transfer lens into the first ESA, where an image is formed close to its exit aperture. Another intermediate image is formed close to the entrance of the third ESA, which projects the final magnified ion image onto the imaging secondary ion detector.

For continuous beam tuning of the LMIG, a set of deflectors (high mass blanker) is located after the extraction lens in order to deflect ion-induced secondary ions and/or secondary electrons into a channeltron secondary electron detector (SED). The high mass blanker is also used during mass spectral acquisition to blank certain ranges of high or low mass secondary ions from the mass spectrum by deflecting them out of the ion optical path. The high mass blanker, as its name suggests, is used principally to remove high mass ions with masses greater than the requested mass range of the experiment. Without this blanking, these high mass ions would "wraparound" and appear as noise at the low mass end of the mass spectrum.

An angle-defining aperture, the "contrast diaphragm" or "angular filter", is placed into the secondary ion "crossover" in front of the first ESA. The physical size of this crossover is primarily dependent on the secondary ion emission angle (and energy) for a given lens system but not by the size of the analysis area. The contrast diaphragm can thus be used to limit the transmitted angular distribution of atomic secondary ion distribution. The contrast diaphragm does not affect the transmission of organic secondary ions if the ion optical system is properly focused. The crossover position is primarily determined by the transfer lens voltage, whereas the image focus of the secondary ions on the final detector is primarily determined by the immersion lens voltage. Further information on this filter is provided in: "The Angular Filter or "Contrast Diaphragm".
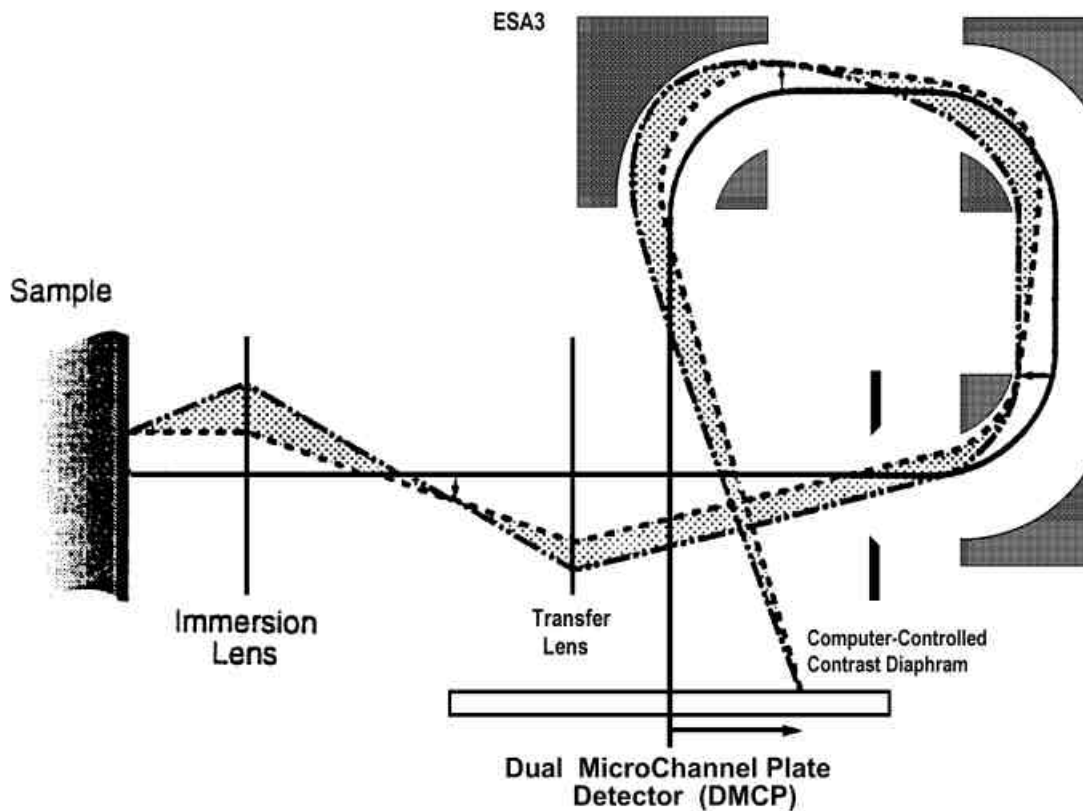
Figure 6-2.   Magnified Secondary Ion Image Path within the TOF-SIMS Mass Spectrometer.

An energy filter can be brought into position just in front of the entrance of the second ESA. The filter consists of a rectangular plate which has a knife edge and a fixed slit set back from the knife edge. For most analytical purposes, this assembly is completely retracted from the secondary ion path. The outer knife edge of this slit assembly is usually placed such that the accepted maximum (axial) energy is some 60 - 120 eV, mainly to reject very high ion energy components and increase the *abundance sensitivity* in atomic secondary ion analysis. In SIMS experiments, the energy slit is used principally to remove 'metastable' ions from the secondary ion stream (refer to the section entitled "The Energy Filter" later in Section 6).

After the third ESA, there is another focus plane or "cross-over" where a second set of mass blanking plates is located. This blanker is known therefore as the "post ESA" blanker. At this position in the secondary ion flight path the ions are well separated in time, and it is possible to use the post ESA blankers, for instance, to remove mass 28 from the secondary ion stream without removing masses 27 and 29. The post ESA blanker is used when high primary ion currents are used at high repetition rates. For more details, refer to Section 7.

Secondary ion images are obtained using a pulsed, micro focused primary ion beam from a liquid metal ion gun (LMIG). The tuning of the mass spectrometer is (usually) unchanged but an ion image is formed by synchronizing the secondary ion flight time with the primary beam raster position, ignoring the secondary ion arrival position on the detector. This imaging mode is referred to as "microprobe" imaging. In this imaging mode, lateral image resolutions of 1,000 Angstroms may be obtained in the TOF-SIMS

Mass spectra are recorded by measuring the time difference between pulsing of the primary ion gun and the arrival of secondary ions on a fast dual microchannel plate (DMCP) detector at the end of the spectrometer by means of a multistop (1024) time-to-digital converter (TDC) of 138 ps time resolution. A phosphor screen may be placed behind the DMCP to observe the secondary ion arrival position at the detector, although the (x y) information is not stored in the data acquisition computer system. The collection of image information is thus correlated to, but decoupled from, the time measurement. Ion imaging is achieved by registering the (x y) raster position of the primary ion beam (microprobe).

Practical Aspects of Spectrometer Operation

Dynamic Emittance Matching (DEM)

One of the biggest changes of the TRIFT III compared to previous generation TRIFT's is the addition of dynamic emittance matching (DEM). The main motivation of adding DEM was to improve the mass resolution that could be achieved from large analysis areas. Without DEM, the mass resolution falls as the raster size is increased. This is because secondary ions that are generated from points off the central ion optical axis take a different path through the spectrometer to the detector. This is illustrated in Figure 6-2. The goal of the DEM optics is send all secondary ions through the TRIFT analyzer along the ion optical axis. In doing this, the peak broadening which came from large raster sizes is nearly eliminated.

On way to conceptualize DEM is to imagine that the extraction field-of-view is rastered perfectly in sync with the LMIG raster. Therefore, from the spectrometer point of view, it appears as if the LMIG is not being rastered. Rather than seeing a square pattern on the detector that is proportional in size to the LMIG raster size, secondary ions always hit the same spot on the detector, independent of the LMIG raster size. The direct ion image viewed at the detector always appears like the raster size is set to zero.

Although this accomplishes the goal of maintaining high mass resolution over raster sizes up to 600 μm, it would result in rapidly producing a "dead spot" on the detector. Therefore, in addition to DEM, a set of scanning plates has been added to scan the secondary ion beam on the detector. These are called the 'detector deflectors'. The size of the scanned area on the detector can be set in the software and is independent of the LMIG raster size on the sample.

When using the TRIFT III, the DEM and detector deflectors should always be turned on. However, there are times when tuning of the instrument it may be necessary to turn them off. When they are turned off, the TRIFT III operates in the direct imaging mode just like the TRIFT I and TRIFT II. The instrument files will come with the values for DEM and the detector deflectors properly calibrated. These values have been determined specifically for your instrument. The DEM tabbed page within the Hardware Utility of the WinCadence software is shown in Figure 6-3 below.

To turn the DEM and detector deflectors OFF, use Table 6-1 below. To turn them back ON, re-enter the values that are in the instrument file.

Table 6-1.   *Table of Typical Settings Required for DEM Operation.*

Parameter:	DEM On Settings:	DEM Off Settings:
Dem_Deflec_Sensi	~ 13330	1
DetectorScanSize X	4	0
DetectorScanSize Y	4	0
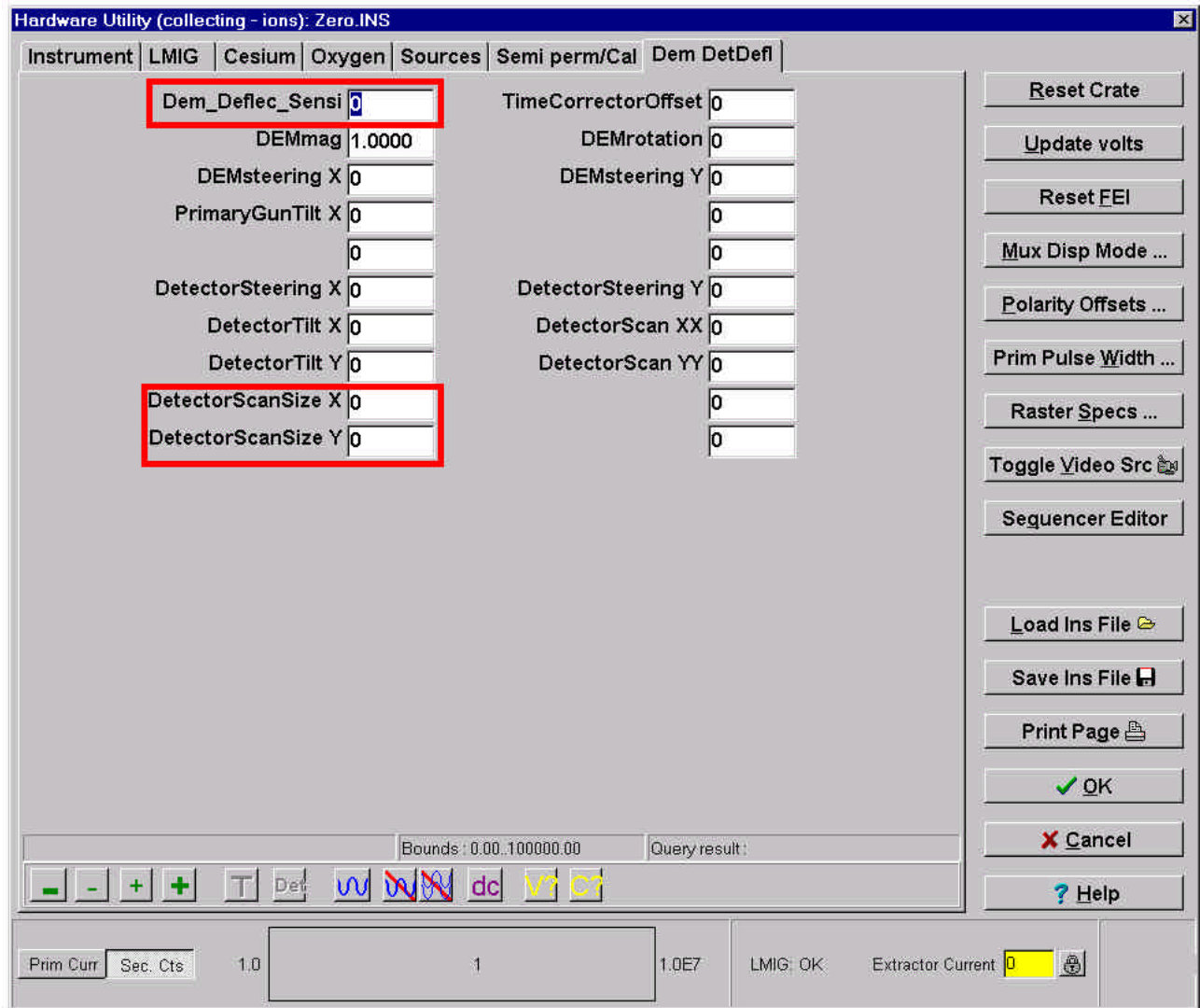
Figure 6-3.   *Example of the DEM tabbed page within the Hardware Utility menu of WinCadence.*

Secondary Ion Filters

The TRIFT is unique among TOF mass spectrometers because it continually refocuses the secondary ion beam at various crossovers within the system. This allows the system to be run stigmatically, and permits the placement of secondary ion filters at appropriate points in the secondary ion flight path. The system's *normal* state is "fully open" (no filters), and under these circumstances the spectrometer's energy acceptance is ~240eV, and the angular acceptance is ~90mrad. This last number is better understood by stating that, with an angular acceptance of 90mrad, an ion of up to 5eV with an angular emission of 89 degrees will be extracted into the mass spectrometer.

The two principle filters, angular and energy, are described in the following two sections. These sections also discuss when it is appropriate to use these filters and the consequences associated with using each.

The Angular Filter or "Contrast Diaphragm"

Secondary ions are extracted through the immersion lens and are then passed on to the transfer lens. This lens forms a focus plane or "crossover" in front of the first ESA, where ions with the same angle of emission are focused. Ions emitted normal to the sample surface (zero angle) pass through the center of this cross-over, while ions with increasing angles of emission are dispersed toward the outside of the cross-over. By placing a circular aperture at this crossover, high angle ions may be removed from the secondary ion stream. These high angle ions contribute significant aberrations to the stigmatic image, and the use of an aperture at this point increases the contrast in the subsequent stigmatic ion image (hence the name "contrast diaphragm"). More importantly for the routine operation of the TRIFT, it is these high angle ions which limit the achievable high mass resolution, particularly on atomic species. In order to achieve high mass resolution (>7000 FWHM) on atomic species, all TOF spectrometers must limit the angular acceptance into the system, either in the initial extraction optics, or, as in the TRIFT, by use of an angular filter at a crossover position.

Angular Aperture

The angular aperture manipulator is computer controlled and has two positions; in or out. With the aperture "out", the spectrometer solid angle of acceptance is wide open. For this setting, we use a 2 mm aperture size. This setting is ideal for imaging rough surfaces where the solid angle of collection is more important than the ultimate mass resolution.

When the angular aperture is "in", a 400 μm diameter aperture is used. This eliminates the atomic secondary ions which are emitted from the surface at high emission angle from sample normal and at high kinetic energy. The result of removing these specific ions from the secondary ion beam is a significant improvement in both mass resolution and peak shape on atomic species. The transmission of atomic species is reduced to approximately 40%, while the transmission of molecular secondary ions is not effected because of their narrow energy distribution. The mass resolution of atomic species increases from ~5,500 - 6,000 with the angular aperture out to ~ 9,000 with the aperture in.

In general, the angular aperture should only be put in when the maximum mass resolution needs to be achieved on atomic species. This could be because the analysis requires the user to deal with well known mass interferences, such as ^{56}Fe /$^{28}Si_2$, ^{63}Cu/$^{28}Si^{35}Cl$, or ^{31}P/^{30}SiH, where high mass resolution is critical to resolve peaks fully. The mass spectrometer does not need any adjustment whether the angular aperture is in or out.

The Energy Filter

As well as pupils or "crossovers" in the spectrometer, there are dispersions. A dispersion of *energy* occurs before the second ESA (in fact it is at a maximum in the center of this ESA). Just before the entrance to the second ESA is located the plane of the energy filter. When the energy filter is fully retracted, the energy acceptance is defined by the Herzog shunts in front of the electrostatic sectors, and is approximately 240eV. Just in front of the second ESA, ions with high energies appear on the outside of the secondary ions stream. By bringing in the knife edge of the energy filter, these high energy ions may be progressively filtered. As well as a knife edge, the energy filter has a slit which may be moved in to pick off a particular band of secondary ion energies. The energy band width is determined by the fixed slit size, and is typically 50eV. Users have exploited this energy band width filtering by making customized energy slits to make fundamental studies on organic secondary ions detected in the TOF-SIMS experiment. For further details, please refer to the work of Delcorte and Betrand in Nucl. Instrum. and Methods B100 (1995) p. 213, ibid. B115 (1996) p. 235, and ibid. B117 (1996) p. 246.

The principal use of the energy filter is to place the slit over the secondary ion beam so as to reject *low* energy ions which are created by the process of metastable decay. In the TOF-SIMS experiment, metastable decay typically takes the form of the loss of hydrogen from a neutral molecule. This creates a charged ion with a mass one unit less than the parent (neutral) molecule. The newly formed ion begins to accelerate in the acceleration (extraction) space, but does not acquire the same energy, and thus velocity, as the other identical ions which were created at the sample surface. Because metastable decay occurs at a range of times and thus distances in the acceleration space, most organic peaks will show a metastable "tail" (interestingly, intense metal ions have metastable tails also, due to the metastable decay of the neutral species MH^o). Because of the way that the energy compensation is performed in the TRIFT, these lower energy metastables typically appear on the low mass side of the peak, and not on the high mass side as one might expect for lower velocity ions. The metastable ions contribute significant background noise in the narrow mass region next to the organic mass, and can mask the appearance of other low level secondary ions. Some transmission losses do of course occur on atomic species when using the energy filter, since the fully open position is rather large at ~240eV acceptance. Transmission losses on molecular organic ions are associated with the removal of the metastable ions as described above, and this loss is normally one of the reasons for employing the energy filter anyway.

The energy filter has only one axis of movement, the x-axis. The energy filter is correctly located over the secondary ion dispersion when the maximum count rate is observed on the ratemeter, coinciding with the brightest stigmatic ion image. Like the angular filter, experimenting with the energy acceptance of the mass spectrometer does not involve any re-tuning of the spectrometer settings, and is thus quick and easy to try.

Practical Method of Tuning the Spectrometer for High Mass Resolution

As a way of introducing this topic and section, refer to Figure 6-4 below. Three factors determine the mass resolution performance in a TOF-SIMS system. The user has control over two of these factors: the primary beam pulse width, and the spectrometer tuning.

The primary pulse length *must* be minimized first to be able to optimize the mass spectrometer. In this section, a practical method to tune the spectrometer is presented, and therefore, before proceeding with this section, insure that the primary beam pulse width has been minimized. For more details pertaining to ion gun tuning, refer to the relevant discussions for either the LMIG or Cs gun.

A = Length of Primary Ion Pulse
B = Mass Spectrometer Characteristics
C = Time Resolution of Detection System
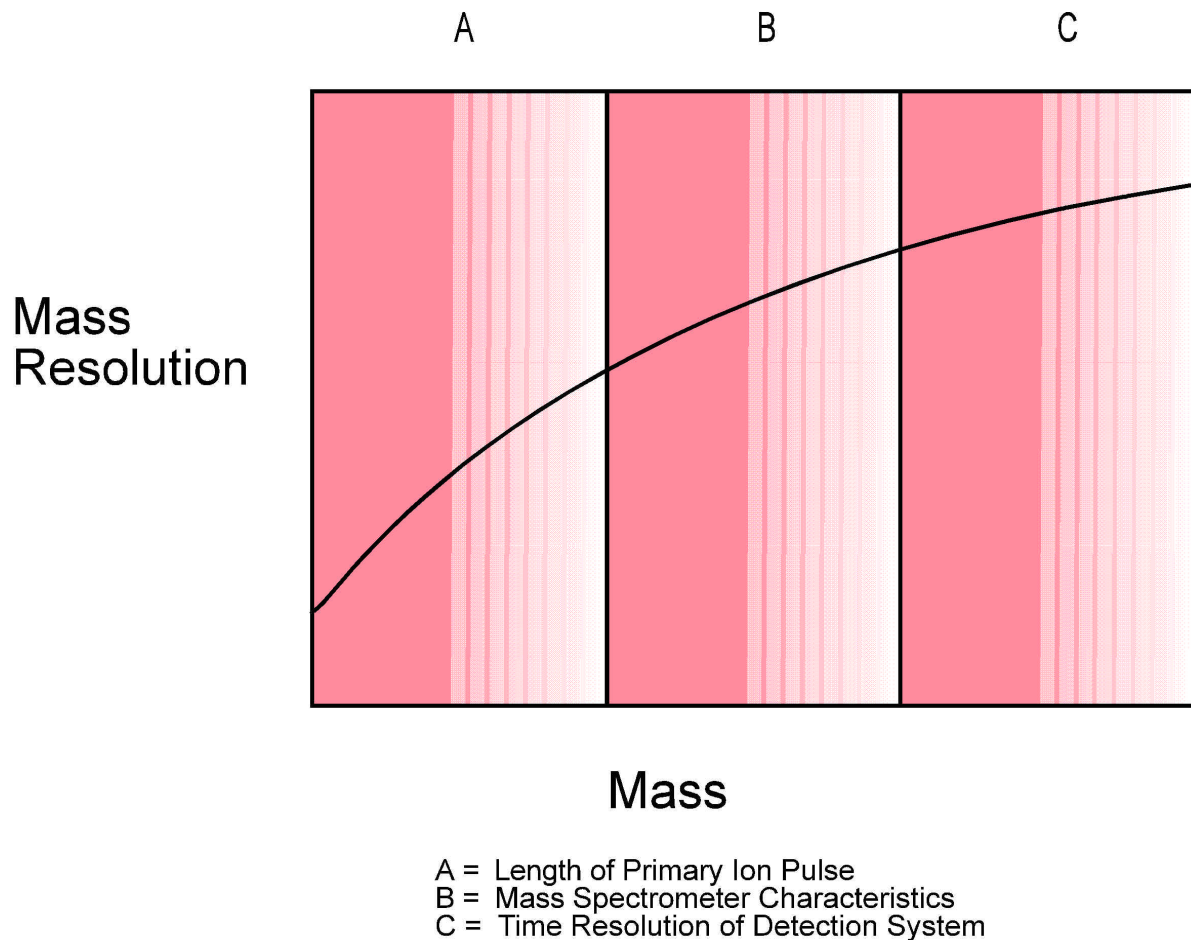
Figure 6-4.   *Diagram of the Principal Effects of Mass Resolution versus Mass in TOF-SIMS.*

Mass Spectrometer Tuning

If the mass spectrometer is already optimized, high mass resolution should be achieved on both molecular and atomic species the moment the primary pulse length is minimized. Typically, high mass resolution will be achieved on molecular species, but less than optimum performance is achieved on atomic species.

If this is the case, the spectrometer should now be optimized for atomics. Once the atomic mass resolution has been optimized, the molecular ions are still at high mass resolution. As a general rule when using the TRIFT, "if you take care of the atomics, the moleculars will look after themselves". This is because molecular species have such narrow kinetic energy spreads compared to atomic species, and therefore, it is much easier to compensate these species and provide high mass resolution performance.

Optimizing the spectrometer for mass resolution on atomic species

When using the LMIG, the mass resolution specification is achieved with a 600pA beam at 15kV beam energy, with appropriate electrodynamic bunching. Therefore, the spectrometer tuning should be performed with that LMIG beam energy and current.

Using a clean silicon wafer, arrange the data acquisition screen so that the peak at Si, with the individual 138ps time channels, can be seen clearly. Without the angular aperture out and with a "fully open" spectrometer, the goal is to achieve ~5,500 - 6,000 mass resolution with a sub-nanosecond primary pulse width.

Since there are a number of parameters which must be set correctly to achieve high mass resolution on a species such as Si, it is advisable to make a table, change one parameter at a time and write down the results. A good log of the results will make it easier for PHI personnel to assist with any difficulties encountered. An example of a blank mass resolution tuning grid is included in Appendix A at the back of this guide for the user's convenience. The relevant parameters to note are: Sample potential; Matsuda Voltage; Raster Size; Detector Voltage; Post Acceleration; C.D. Number; Primary Beam Buncher Voltage; Primary Beam Buncher Delay; Primary Beam Pulse Width; LMIG Emission Current; LMIG Blanker Voltage; LMIG Pulser Voltage, and the Mass Resolution achieved on Atomic and Molecular species.

A series of notes follows below on each of the "tunable" parameters (those parameters in the software which can be altered by operator in order to maximize their settings):

1. **Setting the correct sample potential.** With the Matsuda plate voltage OFF, perform the following experiment: create a plot of <u>channel number</u> vs. sample potential for C_2H_5, using the channel which best represents the peak center. Start at 3100 volts and raise the sample potential in 10 volt steps until the almost flat line begins to "dip" downwards and show significant curvature . Reset the sample potential to 3100, apply -70/-80 volts on the Matsuda plates, and repeat the experiment. Observe a line with a positive gradient, which again begins to fall off at higher sample potential (refer to Figure 6-5 below). The correct setting of the sample potential is the value approximately 10 volts below the point at which the line begins to exhibit a downward curvature when the Matsuda plates are on. The best energy compensation for the atomic species is at this voltage. Observe that the atomic peaks have significant tails and their mass resolutions are lower at sample potentials significantly below this value.
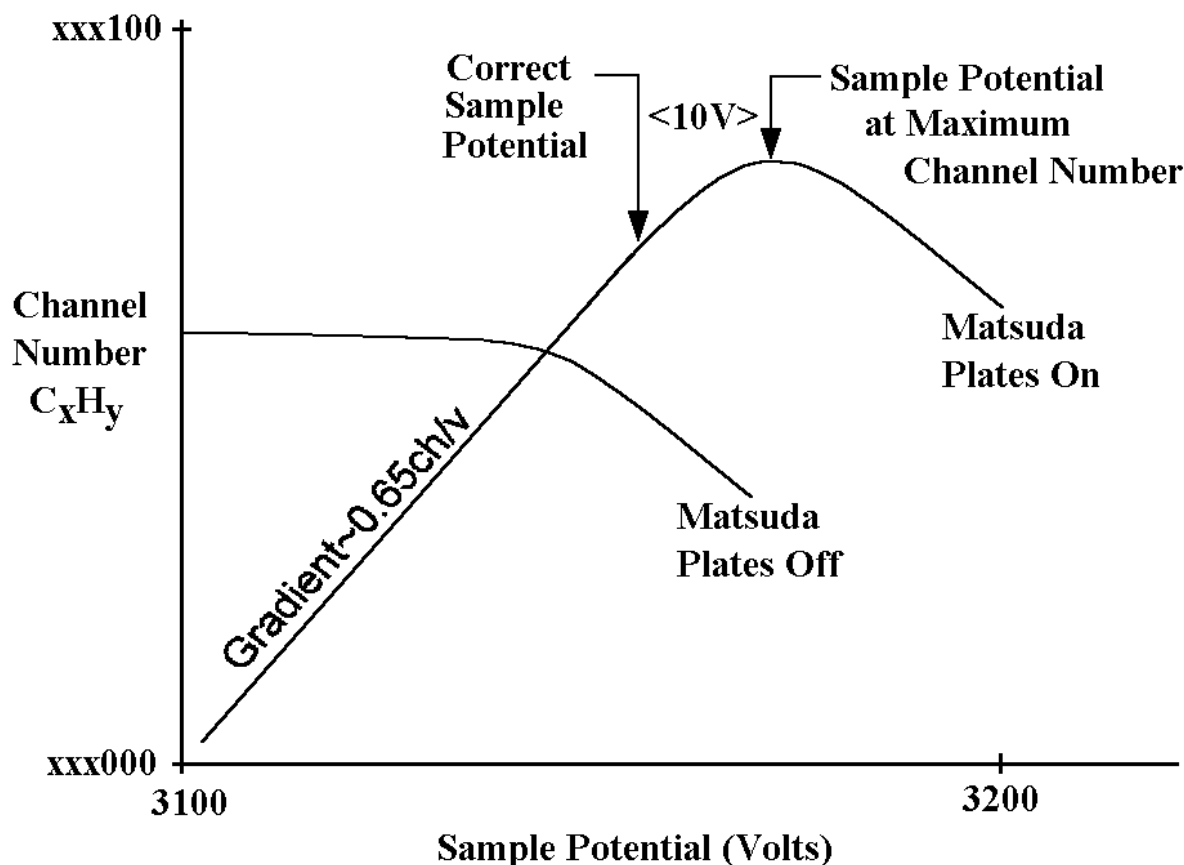
Figure 6-5.   *Diagram of a Plot of Channel Number (at Maximum Intensity) of Organic Species C_xH_y as a Function of Sample Potential with Matsuda Plates On or Off.*

2. **Matsuda Plate Voltage**. For PHI TRIFT III systems, the theoretical value of the Matsuda plates is approximately 80 volts. However, explore the effect of varying the Matsuda plate voltage on the mass resolution of Si, using whichever value gives the optimum mass resolution and a sharp rising edge on the low mass side of the peak.

3. **Raster Size**. For the purposes of maximizing the mass resolution, the beam should be set in a probe mode, i.e. zero (0) raster on the beam.

4. **Detector Voltage**. The correct setting for the detector is when the secondary ion count rate no longer increases significantly with applied voltage on the detector. To be more thorough, plot the peak integral for a given peak as a function of detector voltage, and plot the shape of the curve. At some detector voltage, the peak integral will stop rising dramatically, and begin to level out. This is the "knee" of the curve. Note that if this experiment is performed with zero post-acceleration voltage, the detector will saturate at a higher value than it does when post-acceleration is on (typically, 5000 volts or more). Keep this in mind when deciding the appropriate detector voltage. Set the voltage just past the "knee". Over a period of time (i.e., a few weeks or months) with a new detector, the detector voltage will need to be increased slightly to maintain correct detector efficiency.

   Also explore the effect of detector voltage on the achieved mass resolution at Si, looking for a voltage past which there are no significant improvements in mass resolution.

5. **Detector position**. This really means "the position of the secondary ion beam on the detector". The best energy compensation of the secondary ions occurs when the ions travel down the centerline of the ESAs. Provided the detector is centered to the exit plane of the last ESA, this will result in the beam arriving in the center of the detector. However, explore how steering the beam on the detector affects the mass resolution (if at all) to maximize the resolution.

6. **Post acceleration**. In general, the application of post acceleration increases the mass resolution slightly. However, changing the post acceleration often requires a slight adjustment of the Matsuda Plate voltage.

7. **C. D. position**. The C.D. is at a fixed position and cannot be adjusted.

8. **Primary Beam Buncher Voltage and Buncher Delay**. Ideally, these parameters were optimized earlier when minimizing the primary pulse length during bunching. If the input pulse length is changed significantly, re-examine the buncher delay value because it will probably need to be changed also.

9. **LMIG Emission Current**. For obtaining the optimum mass resolution when using the LMIG, the emission current from the LMIG should be kept at or below 1uA in order to limit the energy spread of the primary beam. For this reason, tune the gun for mass resolution only when a liquid metal ion source is stable and does not have a steep current/voltage (I/V) curve (refer to the LMIG section for more details).

10. **LMIG Blanker and Pulser Voltage**. These two voltages should be considered as a pair, since the blanker voltage shuts the LMIG beam off and the pulser brings it back on. If the pulser voltage is not matched to the blanker voltage, the best mass resolution may not be achieved when using the LMIG (refer to the LMIG section for more details).

Section 7:
Data Acquisition

This section of the guide discusses all aspects of acquiring TOF-SIMS data including imaging, depth profiling, retrospective analysis, and regions of interest.

System Configuration

The typical PHI TRIFT III system falls into one of two common system configurations: a) a single gun (LMIG), or b) a dual gun (an LMIG and a Cs, O_2 or Ar+ ion gun. The various analyses routinely performed on the TRIFT can be separated according to the required properties of the primary beams. Table 7-1 below illustrates how these requirements dictate the usage of the primary ion guns (primary ion gun selection, species, and conditions).

Table 7-1   A correlation between Analysis Conditions and the Primary Beam(s) \ Modes.

Analysis Conditions:	Primary Beam(s) and Modes:
Small probe (sub-μm) imaging, $\leq$ at medium m/δm, high data rate.	LMIG: High beam energy (25kV); primary currents 2nA; medium-long primary pulse lengths.
Small probe (sub-μm) imaging, at high m/δm, low data rate.	LMIG: imaging with 25kV "chopped" beam, 200nm probe @ low primary ion flux/cycle, 1ns primary pulse widths.
Medium probe (~2μm) imaging, at high m/δm, high data rates.	LMIG: imaging with 15kV "bunched" beam, ~2μm probe at high primary ion flux / cycle; sub-nanosecond primary pulse widths.
High depth resolution, high m/δm dual beam (interleaved) depth profiling.	Bunched LMIG for analysis (15kV beam energy): 1keV Cs beam for high depth resolution sputtering. Or low energy Ar+ beam for high depth resolution sputtering
Medium depth resolution, high m/δm, single beam (phased) depth profiling.	Bunched LMIG for analysis (5kV beam energy): 2-5keV LMIG beam for sputter removal.
High current, high m/δm, surface spectroscopy (no imaging).	Bunched LMIG (15kV beam energy); *OR* Bunched Cs (10kV beam energy).

The highly versatile LMIG developed by PHI for TOF applications means that an LMIG-only system will perform most of the routine analyses on the TRIFT system. Dual beam depth profiling at ultimate depth resolution requires a two-gun system operating in what is known as "interleaved" mode, and general spectroscopy may be performed with *either* the LMIG or the Cs gun. The user should be aware that the choice of primary beam species can affect the appearance of the mass spectra obtained from organic materials.

Mass Spectra

The sections below deal with features which the user should become familiar with from an instrumental point of view. These features are important because they represent hardware tools which have a significant impact on the way in which mass spectra are acquired. However, specific details on how to acquire, display, manipulate and store mass spectral data using the system software are discussed in a separate software manual.

Setting and Varying the Primary Pulse Width of the LMIG

If the LMIG beam is electrodynamically bunched, there is little point varying the primary pulse width, which has been set to provide an efficiently bunched beam with a pulse length of <1ns at the sample surface. However, if the beam is not being bunched, for instance at 25kV operation, it can be quite useful to vary the primary pulse width, either to achieve a specific mass resolution, or perhaps as a means of adjusting the secondary ion count rate.

The pulse width of the signal originating from the pulser unit for the LMIG can be varied from 100 ns down to approximately 1.5 ns. Below 1.5 ns, the pulse amplitude falls off resulting in an ion beam that no longer pulses back on. When attempting to produce extremely short pulses (<2ns) by chopping back the pulse width, it will be necessary to reduce the magnitude of the blanker voltage to match the reduced amplitude of the pulser signal.

When the pulse length is set at 100 ns, the primary ion flux and the secondary ion signal are maximized. In the absence of bunching, however, the primary pulse length becomes the limiting factor in achieving mass resolution. Below mass 50, atomic and molecular species of the same nominal mass will not be separated when the primary pulse length is set to >20 ns.

In order to achieve a mass resolution which permits peak separation, it is often sufficient to reduce the primary pulse length to 10 ns or less. Naturally, the acquisition time will be increased because the dose rate is reduced. There are many occasions where the increased acquisition time is more than offset by the ability to conduct SIMS imaging with good mass spectral resolution.

When the primary pulse length is reduced, it is important to know what the new pulse length is, for example, to enable the dose calculator to function. A very close estimate of its value can be obtained by measuring the time width of the hydrogen peak at full width half maximum (FWHM) when reducing the pulse length. To do this, simply note the number of time channels defined by the FWHM, and multiply that number by the TDC timing resolution set in the acquisition, acquisition setup menu. For instance, if the hydrogen peak is 8 channels wide at half its height, and the TDC time resolution is 1.25 ns, the primary pulse length is close to 10 nanoseconds (ns).

Imaging

SIMS Images: Peak Window

The Peak Window allows the user to view and edit the list of mass-windows which define what images or profiles will be built during data acquisition, as well as the mass range(s) which will be blanked by the post-ESA blanker. The initial list of defined mass-windows is called the master list (in Wincadence it is termed (AllPeaks)). If the imaging option is selected for these mass-windows, an image will be created for each mass-window during acquisition. Secondary lists of mass-windows can also be created. The secondary lists (termed ImagePks#) allow the data from all the mass-windows in the list to be summed into one image. The mass-window defined in the secondary list will automatically be added to the master list, thereby, an individual image of the peak will be generated, as well as its summation with the other peaks in the secondary list. In practice, the summation of signals from several mass-windows into one image allows the fragments from one compound in a sample to be combined; it gives a more realistic view of the compound's distribution as well as improved signal-to-noise.

Creating Peak Lists

A master list can easily be created in the Peak Window. From the toolbar, in the upper left, select New Peak. On the right side of the Peak Window, fill in the information about the mass-window desired (for example type Cu into the Formula box, then select Imaging from the list below it). This results in the mass-window being added to the "(All Peaks)" master list (see lower left of the Peak Window). Additional mass-windows can be added in a similar manner.

If a secondary list of peaks is desired, the Image Pks button should be selected from the toolbar. A new list will be begun, labeled ImagePks#1 (see upper left of the Peak Window). Now select New Peak from the toolbar, fill in the information about the new mass-window desired (right side of Peak Window), and the mass-window will be added to the secondary list and the master list. Subsequent peaks can be generated in a similar manner. Remember that all the peaks in the secondary list will be summed into a single image. In addition, their individual images will be generated because they also reside in the master list.

Note that the peak lists are maintained in a "tree" format in the upper left of the Peak Window. The titles of the master list and the secondary lists are shown. They can be double clicked to show the mass-windows defined within each list. It is possible to drop-and-drag peak windows among different peak lists. A mass-window can be deleted from a secondary list, but it will remain in the master list. If a mass-window is deleted from the master list it will be automatically be deleted from the secondary list.

After the mass-windows of interest have been defined, imaging data acquisition can begin.

The default primary ion beam raster for SIMS imaging is known as the "scatter" raster. This raster was originally designed to be used only for insulating samples. Although there is no harmful effect on conducting samples, this raster mode is used as a default for all data acquisition. A variety of raster modes are available and can be set in the "hardware" section of the software (Instrument, Hardware Utility, Raster Specs).

Raster Types and Description

The five (5) various raster types are listed below followed by a brief description of each type.

Scatter: Each pixel point is located as far from the previous and next pixel point as is possible so as to spread the primary beam charge homogeneously. A frame is finished when all 256 x 256 points have been addressed once, when the sequence begins again.

Flyback: The frame is completed as a series of lines, with the beam "flying back" at the end of each line. In each line scan, the beam position increments by one x value, until that value has reached 255, whereupon the x value is set back to zero and the y value is increased by 1. At the end of the last line, the beam flies back to the starting pixel position.

Non-interlaced: Similar to flyback, except that at the end of the line scan, they value changes by one, moving to the next line, and the x value starts at 255 and decrements until it reaches zero. The non-interlaced raster thus does not fly back at the end of each line, but zig-zags through the frame. At the end of the frame, the beam does then flyback to the starting pixel position.

2-Fold interlaced: Similar to non interlaced, except that at the end of each line the y value increments by two. At the end of the sweep, only half the pixel points have been addressed, and the beam then zig-zags back, covering the pixels that were not addressed in the first sweep. At the end of the frame, all pixels have been addressed, and the beam is close to the starting point for the next frame, and thus does not flyback at any point.

4-Fold interlaced: Similar to 2 Fold interlaced, except that the y value increments by 4 at the end of each line.

"Scatter" is typically used as the default for acquisition, "flyback" for tuning the LMIG in a D.C. beam mode, and the "2 fold" or "4 fold interlacing" are used for depth profiling and other D.C. beam sputtering applications where homogeneous ion beam flux density is required.

Provided that the LMIG has been tuned correctly, the remaining aspects of acquiring secondary ion images that are not covered in the LMIG section are defined by software tools, and are not covered in this manual.

SED Images

The TRIFT system produces secondary electron images when the sample is at negative potential (-SIMS mode). Secondary electrons are extracted from the surface with high efficiency because of the high extraction field. Consequently, it is possible to acquire SED images in a *pulsed* primary beam (as opposed to D.C. primary beam) mode, under "static" SIMS conditions. These images are acquired simultaneously with negative secondary ion images, provided that the high mass blanker is set accordingly. If the high mass blanker is set for a mass range of 0.5 to (say) 400, during each pulsing cycle the high mass blanker will send electrons to the channeltron detector, but then change state in time to allows secondary ions to pass into the mass spectrometer, where images are produced at the DMCP.

To acquire a pulsed SED image into the computer you must instruct the software to acquire such an image by requesting "SEM image" in the acquisition menu (WinCadence: Acquisition, Setup acquisition menu). Normally, the SED gain is set for tuning of a *D.C.* primary beam, and so it is necessary to increase the SED gain by approximately 300 volts to compensate for the reduction in secondary electron flux in a pulsed beam mode. Pulsed SED imaging allows SEM images to be acquired from organic samples with ultra low primary ion beam doses ("static" SIMS condition) without destroying the sample. Also, because the instrument is in a pulsed state, it is possible to use low energy electron charge compensation *simultaneously* with pulsed SED imaging! This allows the user to acquire SEM images from highly insulating surfaces, and under "static" SIMS conditions. For these reasons, the pulsed SED imaging is also known as "static" SEM imaging.

Acquiring an SED image under *D.C.* primary beam conditions is not commonly done, because the act of bombarding the sample with a *D.C.* primary ion beam will often damage the sample (organic surfaces) or result in charge build-up (insulators). However, it is simple to acquire a *D.C.* ion beam-induced SED image. Choose DC in the menu Instrument, Hardware Utility, LMIG tab. If the video monitor is set to show the output of the SED, you will now observe an image. Adjust the SED gain accordingly. The scan speed defaults to T.V. rate, and can be reduced to 0.25 MHz for slower, clearer scans.

Mosaic Mapping

The Mosaic Map data acquisition function creates an ion image of a large area by stepping the sample stage over the area and stitching together the set of individually acquired ion images into a single Mosaic Map image.
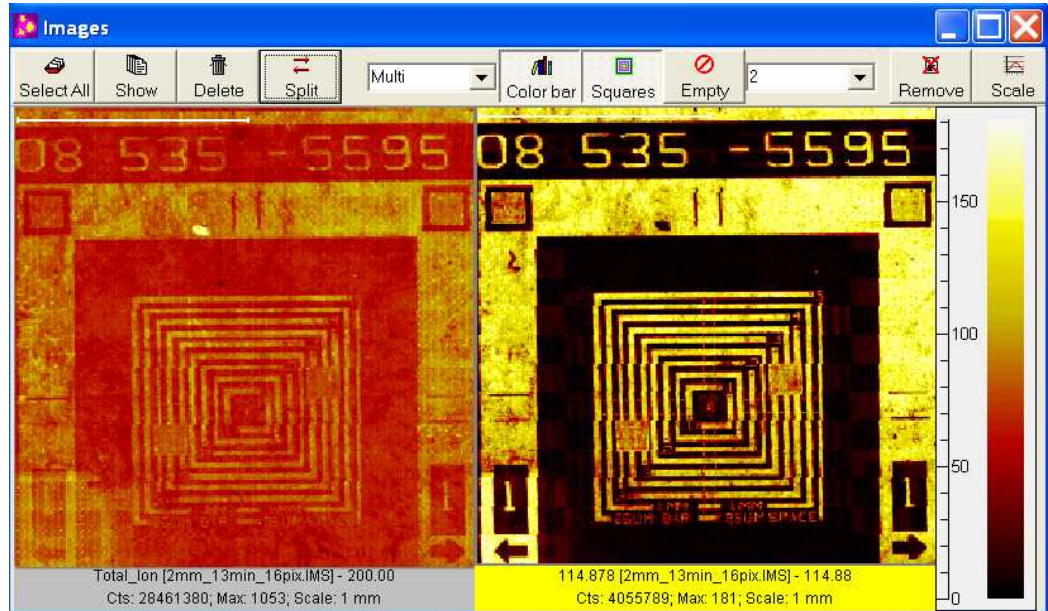
Figure 7-1.   *Mosaic Map Ion Images*

As part of Acquisition Setup, the user specifies the size (e.g. 8x8mm) and stage coordinates of the large area, as well as the desired acquisition time per step (e.g. 3s). WinCadence will then calculate the optimal number of steps and, if necessary, adjust the analytical raster size appropriately. All these values are shown on the Mosaic Map tab of the Acquisition Setup Dialog as shown in Figure 2 below.
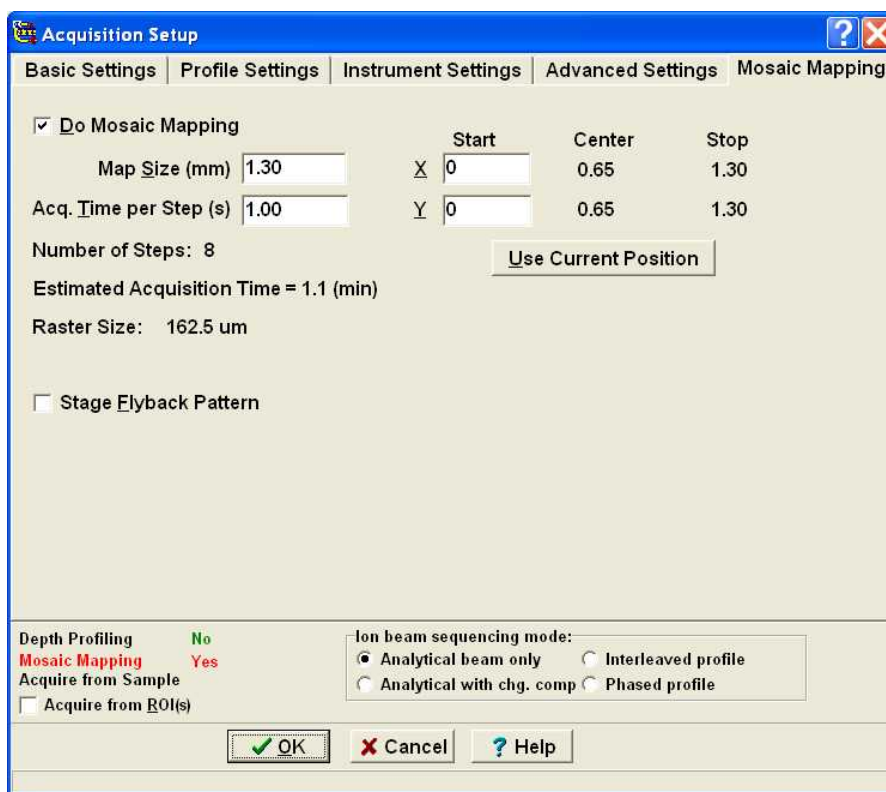
Figure 7-2   *Mosaic Mapping tab of the Acquisition Setup Dialog*

The following is a description of the options available on this tab:

Do Mosaic Mapping Checkbox

This checkbox should be checked when the user wishes to perform a Mosaic Map acquisition. When the next acquisition is started, the stage will begin moving in order to create the Mosaic Map.

Map Size (mm)

This edit field indicates the size of the analytical area for the Mosaic Map. This value determines the extent in mm of one side of the square area. For example, a value of 2mm indicates that a square area of 2mm by 2mm will be analyzed during the Mosaic Map acquisition. Note that this value, in conjunction with the maximum raster size defined (typically 250um — see Mosaic Max. Raster Size below), will determine the Number of Steps value (visible on the user interface as shown above). The Number of Steps value is the number of individual stage steps that will occur along one side of the square area. For example, a Map size of 2mm in conjunction with a raster size of 250um will result in a Number of Steps of 8 per side (i.e. a total of 8x8=64 steps for the complete square area). The Number of Steps per side will always be a power of 2 and can range from 2 to 256.

Acq. Time per Step (s)

This edit field indicates the amount of acquisition time that will be spent at each step of the Mosaic Map (i.e. at each individual stage position). This value, along with the Number Of Steps, determines the Estimated Acquisition Time (visible on the user interface as shown above). Note that the calculation of the Estimated Acquisition Time includes an additional amount of time per step required for stage movement and related timing overhead.

Start X and Start Y Stage Coordinate edit fields

These edit fields are used to determine the starting stage position (in mm) of the Mosaic Map. The center and end of travel are also indicated on the user interface as shown above.

Use Current Position Button

This button is used to set the current stage position as the starting point for the Mosaic Map. When this button is clicked, the Start X and Start Y values should update appropriately with the current stage position.

Stage Flyback Pattern Checkbox

This checkbox is used to ensure that the primary axis of stage travel is followed in the same direction on every row of stage movement. When a row of stage movement is completed, the stage will 'flyback' to the other side before beginning the next row. This flyback option lengthens the total time of acquisition, but may improve Mosaic Map alignment in some cases where stage hysteresis is of concern. Note: if this checkbox is not checked, the stage will follow a serpentine pattern, without flybacks.

Mosaic Size	2	4	8	10	16	20	32	50	64
#Steps	8	16	32	64	64	128	128	256	256
RasterSize	250	250	250	156	250	156	250	195	250
Mosaic Pixels	32	16	8	4	4	2	2	1	1
Mosaic Pixel Resolution (µm)	8	16	31	39	63	78	125	195	250
Total # Steps	64	256	1,024	4,096	4,096	16,384	16,384	65,536	65,536
Minutes for each 1sec/acq	1.1	4.3	17.1	68.3	68.3	273.1	273.1	1092.3	1092.3
Hours for each 1sec/acq	0.0	0.1	0.3	1.1	1.1	4.6	4.6	18.2	18.2

Mosaic Map Acquisition

A mosaic map acquisition is initiated in the same way as any other in WinCadence, by choosing the Acquisition Start menu option. After a brief period of calculation, the stage should begin to move and data should begin to appear in the Mosaic Map images shown in the Image Window, see Figure 7-3.
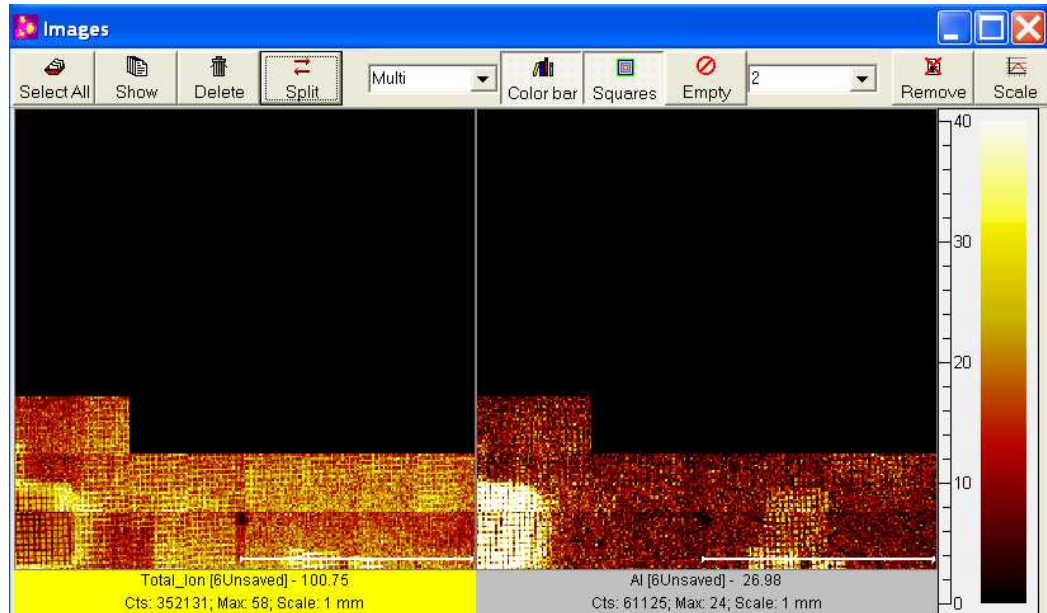
Figure 7-3   *Data appears in a stepwise fashion during Mosaic Map Acquisition*

Mosaic Map images are displayed and manipulated in exactly the same way as standard ion images. For example, mass selected Mosaic Map images may be defined via the Peak Window in the same way as is done for standard ion images. Raw Data File Mosaic Map acquisitions may also be saved and replayed in the standard fashion.

Mosaic Map Maximum Raster Size

The Mosaic Map Maximum Raster Size is set in the Tools|Options dialog on the Imaging tab as shown below in Figure 7-4
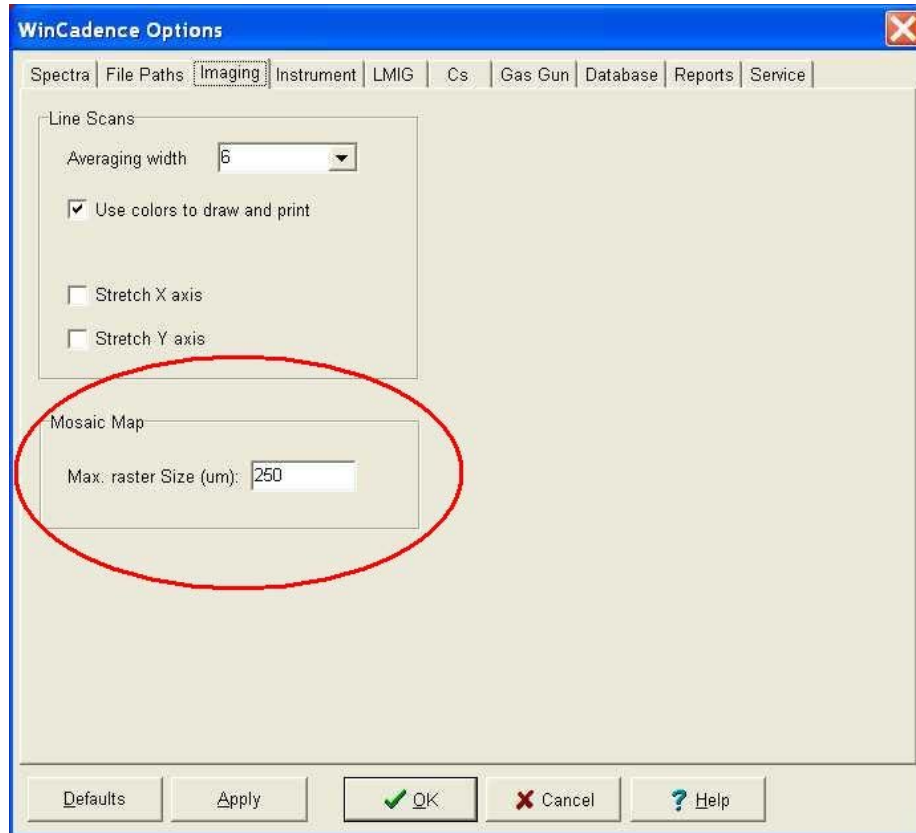
Figure 7-4   *The Imaging Tab of the Tools/Options Dialog*

The Mosaic Map Maximum Raster Size is limited to 250um or a similar value in order to ensure that the relative lateral ion intensity for a given 'tile' (individual stage position) of a Mosaic Map is highly uniform. Increasing this value will speed up the total acquisition time for a Mosaic Map of any given size (because the Number of Steps will be reduced), but may have an undesirable effect in the perceived uniformity of the Mosaic Map image.

TOF-SIMS Depth Profiling

Definitions and Descriptions

When introducing TOF-SIMS depth profiling, it is important to clarify how this form of depth profiling differs from conventional dynamic SIMS depth profiling using either quadrupole- or magnetic sector-based instrumentation. In a conventional dynamic SIMS instrument, a <u>continuous</u> primary ion beam erodes the sample surface while the mass spectrometer is tuned to monitor a specific secondary ion signal as a function of sample depth. Even though conventional mass spectrometers may not have as high a transmission rate as TOF spectrometers, they are able to measure a secondary ion mass of interest <u>continuously</u> as the primary beam erodes the surface. If the magnet or quadrupole within a conventional mass spectrometer is scanned to measure more than one secondary ion species, overall sensitivity is reduced due to 'serial detection". Serial detection (versus parallel detection in TOF) allows the monitoring of one mass but cannot measure other secondary ion masses entering the spectrometer simultaneously. Dynamic SIMS achieves remarkable sensitivities for various analyses in a given volume of sputtered material, which justifies its unique place in surface analysis. Obtaining 'SIMS at depth' can be achieved in a number of ways in the TRIFT system. These modes are defined and described below. The section entitled "TOF-SIMS Profiling: Practical Details for Instrument Setup" then describes the practical details for implementing each mode.

Automatic depth profiling routines can be programmed for either the Cs or Ga - LMIG primary ion guns. These depth profiling routines make use of three cycles:

- The *analysis cycle* is operated as in any standard analysis, utilizing the pulsed primary ion beam. In this cycle, a mass spectrum is acquired for the predefined time period and selected mass range.

- The *sputter cycle* is (typically) utilized for material removal by means of sputtering with a continuous (unblanked) primary ion beam.

- The *charge compensation cycle has* been primarily intended for use with depth profiles on insulating samples.

Near surface, TOF-SIMS profiling with high current pulsed beams

TOF-SIMS can perform depth profiling by pulsing only, in which case, the profile has all the benefits of the inherent high transmission of a TOF spectrometer combined with parallel detection capabilities. Unfortunately, such profiles are limited to the near surface (approximately 30-50Å), even if high current primary beams ($\geq$ 20nA) are used. This is because the 'duty cycle' (the time that the primary beam is pulsed on compared to the time that the beam is off) is extremely low, typically, 10^{-4}. In other words, for a set primary beam current, it will take a TOF instrument typically 10,000 times longer to reach the same depth as a non-pulsing SIMS instrument. As a result, it is only in performing near surface profiles that all the unique features of a TOF instrument are fully utilized. One such experiment is the measurement of low level metals in the surface oxides on silicon wafers.

TOF-SIMS at depth with analysis / sputter sequence

When the system operator wishes to profile deeper than, for example, 50Å in a reasonable time with a TOF-SIMS, the analysis must consist of two separate phases:

- an analytical phase, and

- a dynamic SIMS phase.

In the analytical phase, the secondary ions sputtered by the primary beam are measured with the full capabilities of the TOF in "static SIMS" mode.

In the dynamic SIMS phase, the primary beam is used to reveal material at depth in the sample, but the spectrometer does <u>not</u> measure any of the sputtered secondary species.

In the TRIFT III, there are two modes in which this form of TOF-SIMS profiling may be achieved:
- 'Phased' Depth Profiling
- 'Interleaved' Depth profiling

A description and comparison of these two modes follows.

Phased Depth Profiling

In 'phased' profiling, the analysis begins with an acquisition phase within the static SIMS regime (typically, 10-30 seconds). Following this, the analytical conditions are stored to temporary memory, and a sputter phase is invoked.

After a set sputter time is defined by the user (typically, 1-30 seconds), the analytical phase is re-loaded from temporary memory. The next analysis is a static SIMS analysis, but it occurs at some depth into the sample. The cycle of analysis/sputter/analysis/sputter... continues until either the countdown timer times out, or to a point where the profile is terminated by the user. This mode of profiling is very similar to that used for Auger or XPS profiling: an ion beam reveals a new surface which is then probed by the analyzing beam.

In addition to an analytical phase and a sputter phase, the user can invoke two other phases:
1. A charge compensation phase; and
2. A 'settling' phase.

The charge compensation phase invoked in profiling is different from the typical pulsed charge compensation which occurs during acquisition. The charge compensation phase pauses the profiling, lowers the extraction field to zero (0), and then turns the electron gun on in a *continuous* mode to equalize any charge build-up that may have occurred during the sputtering phase. This phase may also be used to assist with imaging of insulators at high magnification, as described in another section.

A settling phase permits optimization of profiling conditions for single gun systems by allowing the system to settle in between phases. It is designed for profiles in which a single gun is switching beam energy between the acquisition phase (higher energy) and the sputter phase (low impact energy). Settling phases can be programmed prior to each of the other phases: acquisition; sputter; and charge compensation.

Phased profiling can involve the use of one primary ion beam only, or can use two beams to optimize the depth resolution of the profile with a second low energy beam.

Phased depth profiles are useful for deep structures in which the concentration changes are either continuous, or where the layer structures are quite thick.

The disadvantage of 'phased' depth profiling is that it is possible to miss shallow, buried layers bypassing through them during a single sputter phase. For such structures, a different profiling mode known as interleaved depth profiling is preferred.

Interleaved Depth Profiling

The principle of interleaved depth profiling is shown in Figure 7-5 below and can be understood by considering what happens after a single primary pulse from the primary ion beam strikes the sample surface. After some number of microseconds, the secondary ions are all extracted from the surface and are injected into the drift section of the mass spectrometer, followed by a period of time (typically 100 microseconds) where those secondary ions are dispersing in space and traveling through to the detector. The primary beam is normally dormant until all the secondary ions have been counted and the next cycle begins the sequence all over again.

In interleaved depth profiling, instead of waiting until the heaviest of the secondary ions are counted at the detector, the primary ion beam is switched to a *D.C.* beam as soon as the secondaries have been extracted and injected into the drift space, eliminating the need for 'extra' time spent on a separate sputter phase. The sputtering is 'interleaved' with the analysis at 5 kHz. It is not feasible for a single gun to switch beam energies at the speeds required during interleaved profiling, therefore, two primary beams are required to optimize this profiling method: one for the analyzing pulse and one for the sputter pulse (typically at low energy for depth resolution).

The advantage of interleaved profiling is that fine structures are not missed during the profile. The disadvantage is that the two primary beams must be carefully aligned. The analytical pulse raster must be concentric with, as well as within, the sputter crater.
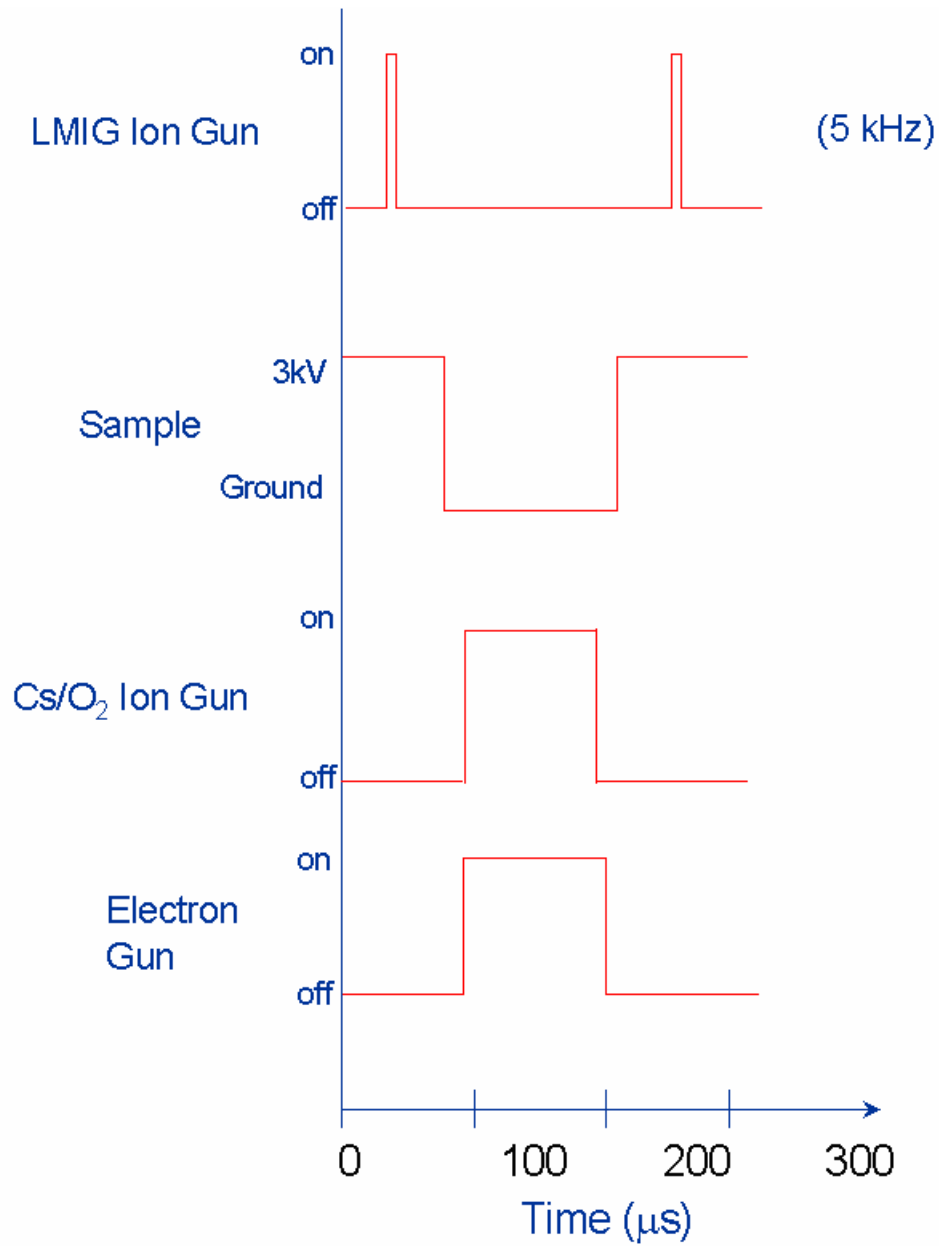
Figure 7-5.　　*Timing schematic of the Dual-Beam Interleaved Mode of depth profiling. The electron beam is only used for insulating materials. In this mode, sputtering is done in 100-150 ms intervals between LMIG pulses. As a result, data is integrated continuously as function of depth. In the phase mode, data is collected only at specific depths.*

Practical System Set-up

The previous section defined the different profiling modes. This section will consider the *practical* details for implementing profiles in each of the different modes, such as setting a single gun LMIG system for low energy profiling, and how to use the profiling tools in software to finesse other experiments, (e.g., imaging insulators at high magnification, etc.)

Near surface, TOF-SIMS profiling with high current pulsed beams

High sensitivity, near surface profiling (typically, 30-50Å) can be achieved in a pulsed-only mode in 'reasonable' times (e.g., 15-20 minutes) using primary beam currents of ≥20nA. The beam energy for the Ga beam is typically 15kV, and 10 or 11kV for the Cs beam (for high mass resolution in bunched mode). These beams could be tuned to a lower beam energy, except the probe size increases and the efficiency of the primary beam transport is reduced at these lower beam energies, resulting in lower probe currents. The only drawback to using the higher energy beams is reduced depth resolution.

The principal use for these pulsed-only profiles has been to make quantitative measurements of the surface metal concentrations in thin surface oxides on silicon wafers. The following notes in this section reflect this. In these measurements, it is important to <u>not</u> confine the measurement to the top surface only, but to consume the entire native oxide. In some cases, metal species are segregated to either the outer surface or the oxide/silicon interface. Concentration profiles help to determine such segregations, although it is the final integrated mass spectrum which is used to derive the metal concentrations.

Post-ESA blanking

When using the high current probes of either the Ga or the Cs beam, insure that the post-ESA blanker is set to remove intense matrix peaks right from the outset. There are three reasons for doing this:

1. The most intense peaks will often suffer 'dead time effects' which distort their relative peak intensities. Dead time effects are encountered when the secondary ion count rate is significantly higher than the primary beam repetition rate. For example, consider a secondary count rate of 1 x 10^5, and a primary beam repetition rate of 1 x 10^4. An average of 10 secondary ions arrive at the detector in every cycle. If these secondary ions are significantly different in mass, spread apart in time, the detector will register all of the ions. If these ions, as is typically the case, represent only one or two masses, then some of the ions will be ignored due to detector dead time effects.

    Consider the specific example of a Si wafer analysis. Most of the secondary ion signals are due to ^{28}Si, ^{29}Si, ^{28}SiH and ^{30}Si. In the case of 10 secondary ions per cycle, let's say that four of them are ^{28}Si. When the first of the batch of four arrives at the detector, the detector registers the ion, and there is a charge transfer within the detector. During that period of approximately 10ns

(or ~70 channels at 138ps/channel), the detector is unable to count any more ions and is experiencing a period of 'dead time'. The second of the ^{28}Si ions arrives at the detector while the detector is dormant, and is not counted. In the case of ^{29}Si, statistically there will be a smaller number per cycle compared to ^{28}Si, and the dead time effect on that peak is not as pronounced (e.g, less ions lost). Therefore, the isotope ratios for Si appear not to be in accordance with the natural abundance of the isotopes.

2. Secondly, high current pulsed probes operating at high repetition rates can, in some cases, result in instantaneous secondary count rates of ~10^{10}/second at the detector. By using the post-ESA blanker to remove these intense matrix peaks (which are often hampered by dead time effects anyway), the detector lifetime is extended.

3. Extremely high count rates tend to slow the computer as it sorts and plots the incoming data. For the case cited above, the ^{28}Si, ^{29}Si and ^{28}SiH peaks often account for >95% of the secondary ion population. By removing these, the secondary ion count rate falls by a factor of 20, making data acquisition much more manageable (and also very beneficial when saving 'raw' data to disk!)

Since post-ESA blanking windows can be stored with an instrument file, instrument files can be modified and re-stored according to your particular analytical needs. For instance, a file could be prepared which blanks mass 28 and 29, but leaves mass 30 detected for quantification purposes, and then store this out with a customized name (e.g., SIWAFER.INS, etc.)

Crater Edge Effects: Gating

There is no automatic secondary ion 'gating' when performing pulsed-only profiles. This can limit the dynamic range of secondary signals due to crater edge effects. Gating may be achieved by taking an acquisition as a 'region of interest' from the center of a raster, provided the beam size is much smaller than the total raster size.

Profiling Species

The goal of near surface profiling is normally to integrate the counts for various species in the near surface. For a given set of conditions, the beam will profile to a standard depth in a set time on a specified substrate, and the experiment can be run with a spectrum (only) as the final product. Typically, the goal is to produce a concentration profile as well as a total, integrated mass spectrum, or at least, allows for determination of a suitable end point for the experiment. If concentrations of various species are to be monitored as a function of depth (elapsed time), a list of these species needs to be set up in the Peak window and profile selected. If 0 seconds is entered for the sputter time, the profile window will show the intensity of the selected peaks as a function of acquisition time.

Phased Depth Profiling: Single Gun

Instrument Files need to be created for the analytical phase and for the sputter phase. The analytical phase is accomplished by aligning and tuning the pulsing primary beam as per a normal mass spectral acquisition. The instrument state does not need to be saved specifically because it is stored and recalled automatically from a temporary memory during profiling. If the sputter phase is to be accomplished with the same primary beam *and* energy as the analytical phase, setting up the sputter phase is simple. To prepare the sputter file, increase the raster size of the beam in the control page (typically >4 times the size of the analytical area). If necessary, alter any settings for the beam which affect its focus when in the *D.C.* mode (for instance, raise Lens 2 in the LMIG back to its focused value for *D.C.* tuning as opposed to pulsed and bunched focus). Note that the beam blanker does not need to be set to zero. The software automatically sets the beam blanker to zero during the sputter phase of profiling. Now, save the sputter file with a unique identifier (e.g. sputter.ins). Reset all values back to their original settings for the acquisition phase (e.g. raster and Lens 2).

To setup the single gun depth profile, go to Acquisition Setup, Basic Settings tab. Select Phased Profile from the bottom of the page. This automatically opens the Profile Settings tab, which is divided into sections for the Analytical Phase, Sputter Phase and Charge Compensation phase.

Choose the analyze time. Note that the analyze time is dependant upon the mass range chosen (repetition rate), and that the time periods listed are multiples of a complete frame (data acquired at each of the 256x256 pixels). If the species of interest are present at low concentrations, the time for the analytical phase may need to be increased so that a statistically significant number of counts is generated for the species of interest per cycle.

In the Sputter Phase, load the sputter file previously created (sputter.ins), select the sputter gun (i.e, LMIG), choose the sputter time (20 sec. = typically a good intermediate time for an "unknown" sample), and choose the settle time (normally 1 sec.) Specify the sample potential during the Sputter Phase: high voltage, at ground or with charge compensation (implies at ground). Note: if either "at ground" or "charge comp" is chosen, the ion beam will shift in the x-axis. This will have to be accounted for in the x beam position value saved in sputter.ins in order to keep the analysis phase and sputter phase aligned.

For single gun depth profiles, the sample is typically set at high voltage; in this manner the impact energy of the sputter ions will be lower in the positive ion polarity (5 keV Ga sputter file and 3 keV sample potential results in 2 keV impact energy). In the Charge Comp phase, enter a time (typically a few seconds) if charge compensation is desired in a continuous mode between the analytical and sputter phases. This extra phase drops the secondary extraction field to zero and then turn on the electron gun continuously. Note that if charge compensation is desired during the analytical phase, a value should be entered in the charge comp duty cycle box in the Analyze Phase (typically 64%). Also note

that this value is automatically entered if the operator selects Analytical with Chg. Comp in the Basic Settings tab, before selecting Phased Profile.

The species to be monitored must be designated. A peak table should be created with the peaks of interest and the "Profile?" option selected. It may also be desirable to blank matrix peaks of high intensity and the peak of the sputter source species. This table should be open in the Peak Viewer. The depth profile acquisition can then be started: select Start Acquisition.

Phased Depth Profiling: Dual Gun

An Instrument File that contains parameters for both guns must be used (typically LMIG for analysis and Cs, O2 or Gas Gun for sputtering). If an instrument file (*.ins) does not already exist, one can be created by filter loading the values for the sputter gun into the open instrument file for the analytical gun (15 keV LMIG). Refer to Section 3: Filter-Loading Part of the Instrument State Files.

NOTE: If the Gas Gun has not been used recently, it will be necessary to set the emission to 15mA [the software will ramp the emission setting in 1mA steps per second]. Open V10 in Watcher to introduce the flow of Argon gas into the ionizer.

The sputter and analytical ion beams must be aligned. This can be done manually on a sample where sputter craters can be observed visually (for example, a thin Au film on Si). Use the following steps to align the two beams:

1. Start a spectral acquisition, switch to the detector view, and using the LMIG X and Y beam position, center the secondary ions on the detector. The LMIG is now aligned with the spectrometer.

2. Set the LMIG raster to 25µm and turn the beam to D.C. on the sample. Note the location of the sputtered crater.

3. Adjust the optical microscope to center the LMIG sputter crater. Now the optical microscope is aligned with the LMIG and the spectrometer.

4. Set the sample voltage to zero (0). Set the sputter gun raster to zero (0). Turn the sputter gun beam to D.C. on the sample. Then adjust the X and Y beam position to align the sputter gun sputtered spot with the LMIG crater. Once this is done, the sputter gun beam is now aligned with the LMIG, optical microscope and the spectrometer. Return the sample voltage and sputter gun raster size to their previous values and save the file.

Once a file has been created containing parameters for two ion guns, and the guns have been aligned on a particular sample, the beam alignment can be done automatically on subsequent samples. The automatic alignment takes into consideration the z-height shift of the sample and moves the X/Y beam positions of the guns accordingly. Move to the sample of interest. In the Acquisition Setup, in the Instrument Settings tab, choose "Re-align analysis beam on detector

for z-height shift (non-destructive)." Then select Go. The distance of the resulting LMIG Beam Position shift will be shown. If the value is greater than 5 micron, the auto alignment should be repeated until the resulting X/Y Beam shift is less than 5 micron. Care must be taken to only use this method if the off-set is due to a z-height change. If it is due to some other type of phenomenon, such as extraction field curvature near the sample's edge, then a propagation error will result.

If the operator wishes to verify the alignment of the beams and has ample sample for a destructive test, this can be done as follows.

For the Cs sputter gun:
Implant Cs into the sample in the D.C. mode using a zero raster size and zero sample potential. Image the Cs implant with the LMIG in the analytical mode. If the Cs is not in the center of the analytical area, it can be moved there by right clicking on the image and choosing "Move sputter beam from cursor." Now move the pointer to the center of the Cesium beam spot and left click on the mouse. The guns are now aligned.

For the O_2 sputter gun:
If O_2 is used as the sputter source, the location of the O_2 source can be determined by D.C. sputtering with O_2 (zero spot size, zero sample potential) and then imaging some surface species that will be sputtered away so as to make a dark spot (e.g., C_2H_3). If the O_2 beam is not in the center of the analytical area, it can be moved there by right clicking on the image and choosing "move sputter beam from cursor." Now move the cursor to the center of the sputtered area in the image and click the left mouse button.
This test can be done automatically by choosing "Center sputter beam on image field (destructive!)" in Acquisition Setup, Instrument Settings tab. The automatic mode works only in positive SIMS, for Cs as the sputter source and the LMIG as the analytical source.

For the Ar+ sputter gun:
If Ar+ is used as the sputter source, the location of the Ar+ source can be determined by D.C. sputtering with Ar+ (zero spot size, zero sample potential) and then imaging some surface species that will be sputtered away so as to make a dark spot (e.g., C_2H_3). Any correction required will need to be done manually by adjusting the Xpos or YPos on the Gas Gun page to center the dark spot. A more +X will move the spot to the right. A more +Y will move the spot down.

Once the dual beams are aligned, set the raster size for both the Ga and sputter ion beams. The sputter gun should be at least 2mm or 4x larger than the primary gun raster size. Make sure the sample potential has been set back to its original value in the hardware page. The acquisition can now be set up. Go to Acquisition Setup, Basic Settings tab. Select Phased Profile from the bottom of the page. This automatically opens the Profile Settings tab, which is divided into sections for the Analytical Phase, Sputter Phase and Charge Compensation

phase. Choose the analyze time. Note that the analyze time is dependant upon the mass range chosen (repetition rate), and that the time periods listed are multiples of a complete frame (data acquired at each of the 256x256 pixels). If the species of interest are present at low concentrations, the time for the analytical phase may need to be increased so that a statistically significant number of counts is generated for the species of interest per cycle.

In the Sputter Phase, do not load a sputter file. The software will use the sputter gun settings in the current instrument file. Set the Settling time (s) to 3. Choose the sputter time (20 sec. is typically a good intermediate time to try on an "unknown"sample). Select the desired sputter gun. Specify the potential of the sample during the Sputter Phase: high voltage, at ground or with charge compensation (implies at ground).
Note: If a low energy Cs sputter gun energy is being used as the sputter source or if the Ar+ Gas Gun is being used as the sputter source, the sample potential must be at ground during the sputter phase.

In the Charge Comp phase, enter a Settle time (s) of 1, and choose a Comp time (s) (typically a few seconds) if charge compensation is desired in a continuous mode between the analytical and sputter phases. This extra phase drops the secondary extraction field to zero and then turns on the electron gun continuously. Note that if charge compensation is desired during the analytical phase, a value should be entered in the charge comp duty cycle box in the Anayze Phase (typically 64%). Also note that this value is automatically entered if the operator selects **Analytical with Charge Comp** in the Basic Settings tab, before selecting **Phased Profile**.

The species to be monitored must be designated. A peak table should be created with the peaks of interest and the "Profile?" option selected. It may also be desirable to blank matrix peaks of high intensity and the peak of the sputter source species. This table should be open in the Peak Viewer.

The depth profile acquisition can be started: select **Start Acquisition**.

NOTE: Perform the following if the Gas Gun is used for sputtering:

Close the Spectrometer Gate Valve
Setup the GasGun Parameters
    Start with known setting
    Select desired **Beam Energy**, **Float State**, **Float Voltage** and **Grid Voltage**
    Launch the GasGun DC mode in the Hardware Menu
        Monitor beam at sample—GasGun
            Adjust GasGun **Condenser** for desired current
            Select GasGun **Objective**
            Set GasGun **Raster Size** to **0.001**
Align the GasGun to LMIG
    Move to new area
    Launch the GasGun DC mode in the Hardware Menu
        Monitor beam at sampleThis will turn on the GasGun & burn a spot

        Select DC to sample OFF
        Launch the LMIG DC mode in the Hardware Menu
            LMIG--DC image with 1000um
            [this will display image of rastered area by GasGun]
        Adjust GasGun X&Y to recenter
        Launch the GasGun DC mode in the Hardware Menu
            Monitor beam at sample-This will turn on the GasGun & burn a spot
        DC to sample OFF
        Launch the LMIG DC mode in the Hardware Menu
            LMIG--DC image with 1000um
            [this will display image of rastered area by GasGun]
        Iterate until centered raster

Create the Final settings and SAVE *desired_name*.ins setting
    Select GasGun **Raster Size** for crater
    Select LMIG **Raster Size** for analysis
    SAVE GasGun setting with *desired_name*.ins
    Change GasGun **Beam Energy** to **100**
      [this will minimize the ion current at the sample during the analysis phase]

Acquisition Setup
    Open Spectrometer Gate Valve
    Move to desired analysis area
    Setup Profile **Acquisition Setup**
        Settle time(s) = **(BeamEnergyDelta+1) (eg. 5kv > =6)**
        sputter time = desired
        sputter file = ***desired_name*.ins**
        sputter gun = **GasGun**
        sample has = **Ground**

Acquire

If the Gas Gun is used during the sputter cycle, turn off the ionizer after the profiling is complete. This is important to prolong ionizer lifetime.

1. On the Gas Gun page, set the emission to 0mA.

2. Close V10 in Watcher.

3. Load the zero.ins file

4. Load desired LMIG instrument file to maintain proper operation of the LMIG.

Interleaved Depth Profiling: Dual Gun

An Instrument File that contains parameters for both guns must be used (typically LMIG for analysis and Cs for sputtering).   If an instrument file does not already exist, create one by filter-loading the values for the sputter gun (1 keV Cs) into the open instrument file for the analytical gun (15 keV LMIG). Refer to Section 3: Filter-Loading Part of the Instrument State Files. The sputtter and analytical ion beams must be aligned (see suggestions above in Phased Mode Dual Beam Depth Profiling).

Once the dual beams are aligned, the acquisition can be set up. In Acquisition Setup, in the Basic Settings tab, choose Interleaved Profile at the bottom right. Select the cycle time of the analytical phase. Note that in the interleaved mode, the repetition rate is automatically set to 5 kHz, and that the cycle times are based on multiples of the time it takes to acquire a complete frame at this rate. Enter the duty cycle of the sputter pulse (typically 50%). Enter the duty cycle of charge compensation, if desired (typically 64%). Note that this value is automatically entered if the operator selects Analytical with Charge Comp in the Basic Settings tab, before selecting Interleaved Profile.

The species to be monitored must be designated. A peak table should be created with the peaks of interest and the "Profile?" option selected. It may also be desirable to blank matrix peaks of high intensity and the peak of the sputter source species. The depth profile acquisition can be started. Select Start Acquisition.

Low impact energy, LMIG only depth profiles

The material in this section, (depth profiling with an LMIG-only TRIFT system), could have been covered above, but instead is being considered separately in this section.

1. For positive SIMS depth profiling, a 2keV impact energy can be achieved by tuning the LMIG beam energy to 5kV. In +SIMS, a +5kV beam bombards the +3kV sample with a net energy of 2keV, making it quite suitable for depth profiling at high depth resolution. To tune the gun at 5kV beam energy, first make a plot of the lens 1 and lens 2 values against beam energy from the existing instrument files on the system. From this, estimated values for lens 1 and lens 2 can be extrapolated that will be approximately correct at 10kV and 5kV. Typically, for an extractor value of 7.5kV, the values for lens 1 and lens 2 at 10kV are ~2.1kV and ~5.9, and at 5kV, they are ~ 1.6kV and 2.9kV, respectively. Because it is typically easier to tune the LMIG using secondary electrons, consider switching to negative SIMS mode in order to set up the gun. If a focused image cannot be produced at an energy level of 5kV, go up to 10kV. The LMIG should be tuned with the buncher voltage off initially, even if the intention is to bunch at a later stage.

2. Once the beam voltages are in negative SIMS, switch to positive SIMS. Since the LMIG is now tuned, the only new detail left is where the beam is

striking the sample with respect to the mass spectrometer acceptance area. At 2keV impact energy, the x beam position will require considerable adjustment for the beam to reach the acceptance area of the spectrometer, because the beam is steered significantly by the decelerating sample potential. Note that the x beam position may need to be moved significantly before secondary ions can detected at the DMCP detector.

3. A 5kV beam can be bunched quite effectively to permit high mass resolution mass spectroscopy. The buncher voltage will need to be reduced when compared to the value used for the 15kV beam, and the buncher firing (delay line) will need to change slightly (typically some tens of nanoseconds compared to the 15kV case). Experiment until some narrowing of any mass peak is observed, and then fine tune the values of the buncher and the delay until the highest mass resolution is obtained. Do not be alarmed if the application of the buncher voltage causes the beam to shift slightly on the DMCP. Also, remember that the application of the buncher will cause the beam to be over focused, and lowering the lens 2 value to compensate (as happens at 15kV also) will be required. The lens 2 value for the best bunched imaging at 5kV beam energy is best determined empirically on a copper grid sample.

4. In the case of negative SIMS, the lowest impact energy for a sputter beam that can be realistically achieved is 5keV, using a 5kV LMIG beam onto a sample at zero potential. This is not as low an impact energy as the positive SIMS case, but it is useful.

5. When switching between analytical files and sputtering files tuned for 5kV beam energy, two things can occur: if the beam is being bunched, the value of lens 2 needs changing to keep the beam focused in both phases; and the beam may shift position due to one of two reasons: a) the dynamic steering of the buncher when it is firing (a smaller effect); or b) due to a change in the beam impact energy between the phases in the profile, as is the case for negative SIMS (a larger effect). This is the more likely reason for the shift.

6. The first effect is easily corrected on the computer-controlled LMIG by setting the appropriate lens 2 value in the analytical phase file, and by storing the higher value of lens 2 for the D.C. beam condition in the sputtering phase file. As for the second effect, if the crater is not concentric with the analysis area, appropriate x and y beam positions must be determined for the sputter beam to insure that it is concentric with the analytical beam. When the shift is due to a sputter phase with zero (0) sample potential, proceed as follows. With the sample at zero potential, steer the low energy D.C. beam until the observed crater in the optical view of the sample is concentric with a mark that was made previously with the analytical file in a D.C. condition, with the sample potential on.

Profiling Tools: Enhancing Small Area Imaging of Insulators

Although a discussion on the imaging of insulators may seem out of place when discussing profiling, the profiling tools can be most helpful when attempting to image insulators at high magnification (<50μm x 50μm area) and at high spatial resolution (high primary beam current density). As seen after understanding the method, the proposed analysis uses the phased acquisition capability of the profiling software. It is phased acquisition with no sputtering.

Below a certain ion beam raster size, when a focused probe is rastered over an insulating sample, the flux *density* of neutralizing electrons from the pulsing electron neutralizer will no longer be sufficient to balance the positive flux *density* due to the ion beam. A slow, but inevitable, shift in the sample surface potential occurs, causing peak broadening and blurring of the SIMS image. The smaller the ion beam raster below this critical raster size, the more rapid the sample potential drift. This is represented in the figure below.
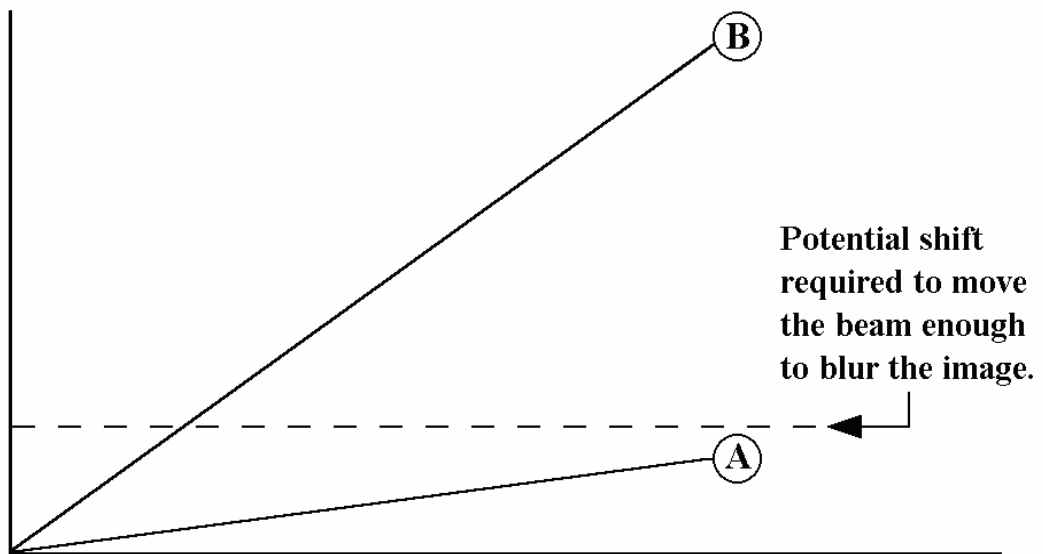
Figure 7-6.   *Change in sample surface potential as a function of acquisition time.*

Refer to Figure 7-6 above. In A, the beam raster size is just less than the critical value. The change in surface potential with time is low. In B, the beam raster size is considerably less than the critical value. The change in surface potential with time is high; image blurring occurs quickly.

The aim of the method described in this section is to prevent the surface charge from building up too much by using periodic flooding of the sample surface with D.C. electrons. This is achieved by harnessing some of the tools available for profiling: acquisition; settle; compensation; settle, and acquisition. This method is not necessarily sufficient to achieve the highest high mass resolution from a small area on an insulator, but it is effective for imaging applications where very small surface potential variations may be tolerated with no noticeable shift in the

primary beam position. The duty cycle between acquisition and charge compensation phases can be adjusted according to requirements. This method is shown schematically in the following diagram.
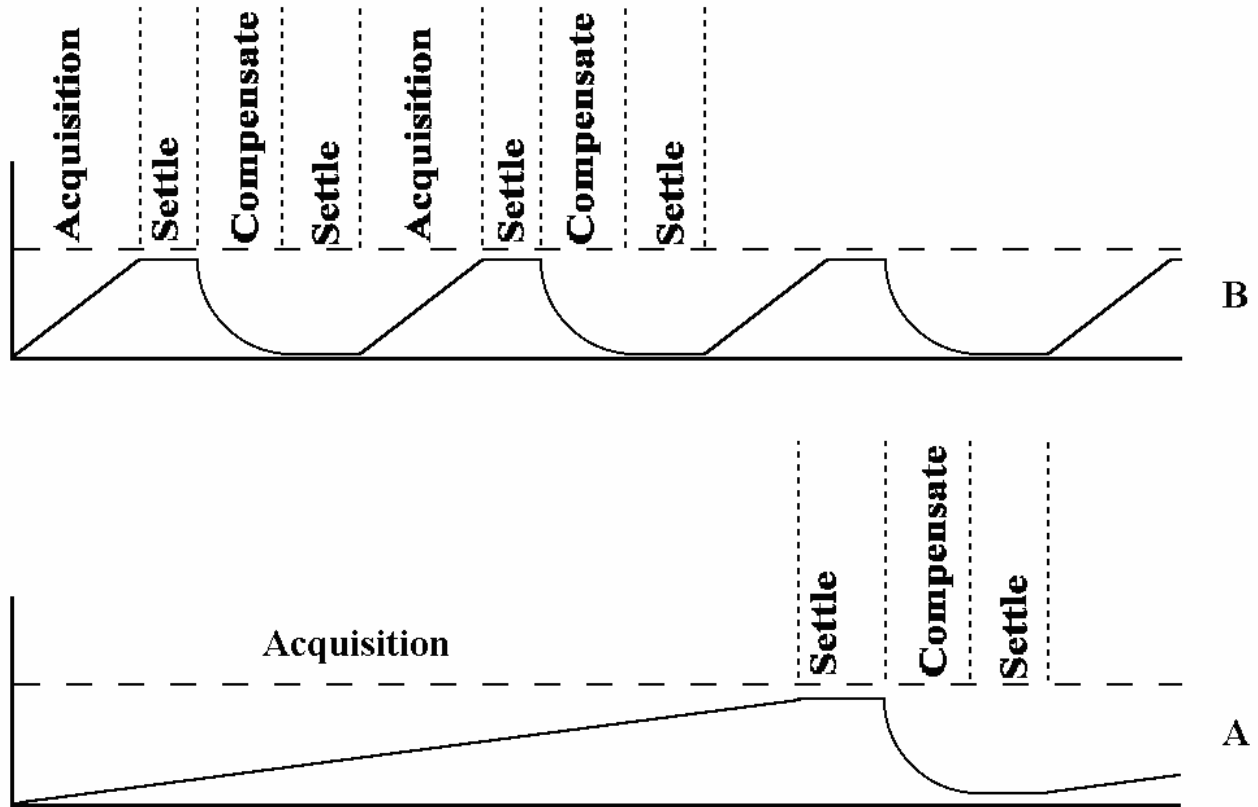
Figure 7-7.   *Using phased acquisition to minimize the effect of surface charging upon imaging of insulators at high magnification.*

Refer to Figure 7-7 above. In A, where the raster size is only just below a critical value, charging is slow and a compensation phase is required infrequently. In B, the surface potential is changing more rapidly, and more frequent compensation phases are required.

As noted in the above diagrams, the duration of acquisition before the extra charge compensation phase needs to be invoked will depend upon the dielectric properties of the materials being analyzed, and more particularly on the beam current and raster size of the primary ion beam. The simplest way to determine this time of acquisition is to allow an acquisition to run as normal (without any extra compensation), and to note at what time the images begin to blur noticeably. If the acquisition time is set in the phased experiment to ~50% of this time, imaging blurring should be eliminated over prolonged experiment durations. Charge compensation is more exacting in negative SIMS when using positive ion beams, and so the ratio of times for the acquisition phase and charge compensation phase will have to be smaller than for positive SIMS.

The following instructions assume that charge compensation has been selected in Acquisition Setup. Also, charge compensation in exacting situations is helped greatly by using the "scatter" raster. Insure that this is the default raster mode when using charge compensation.

Select the species to be imaged. The list can be set and stored beforehand. It will automatically be implemented upon acquisition start. In Acquisition Setup, go to the Profile Settings page. In Analyze Phase, enter 5 seconds for the Settle Time and 60 seconds for the Analyze Time. Under Charge Comp. Phase, enter 5 seconds for the Settle Time and 5 seconds for Comp. Time. Depending on how quickly the sample charges, the Analyze Time may need to be adjusted.
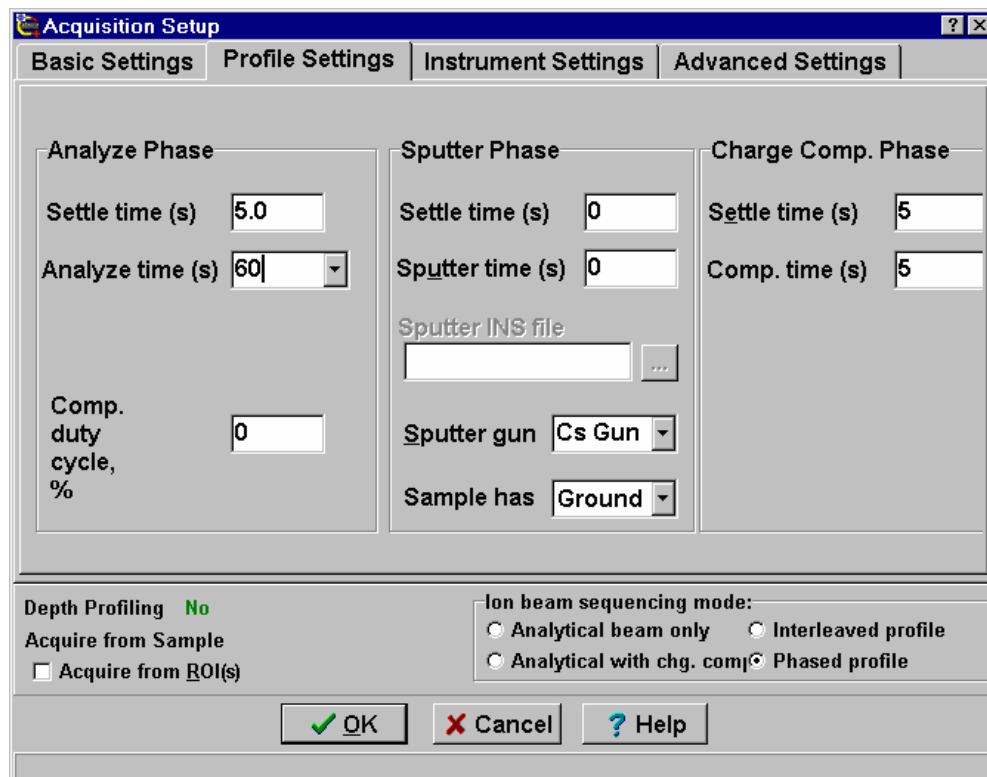
Figure 7-8.   *An example of the Acquisition Setup window with Profile Settings tabbed page selected.*

Upon starting the next acquisition, 'normal' charge compensation will occur during the acquisition phase. The acquisition will then be paused after the set acquisition time. A settle time will allow the sample volts to reach zero. During this charge compensation phase, the surface potential will be stabilized by a D.C. beam of low energy electrons. After a further settle time, the acquisition will continue.

In the example above, the time *between* analysis phases is 15 seconds (5settle + 5ch.comp + 5settle), and thus the total experiment time increases by 25%. This is a small price to pay if the technique allows for achieving results that would be otherwise impossible to obtain.

Depth Profiling of Insulators

The challenge in performing depth profiles on an insulating material such as a ceramic or a glass sample is to ensure that the sputter crater does not drift away from the analytical beam position during the sputter phase in a depth profile.

There are three different methods for attempting to perform profiles on insulators as follows.

- **Coat the sample with a thin (50A) conducting film.**
  This is the simplest, but potentially dirtiest, solution. After the sputter phase has eroded through the coating layer, the sputter crater is pinned in place by the conducting film surrounding it. Any charge that would otherwise build up is immediately drained to the conducting layer. The disadvantage of this method is that unless an extremely clean coating unit exists dedicated to surface analytical techniques, the coating will inevitably contain the usual contaminants such as Na, K, Ca, etc. These species are extremely mobile and will move in advance of the sputter crater. Avoid using this method if any chance exists that the coating could provide a spurious source of the very elements that are being sought in the profile.

- **Using a D.C. electron beam and sputter ion beam simultaneously.**
  Another method for assisting with the profiling of insulators involves the use of a D.C. electron beam concurrent with the sputter ion beam. If the extraction field is zero during the sputter phase (sample potential to zero), it is possible to neutralize the effect of the incoming ion beam by simultaneously using a D.C. electron beam. If the sample potential is not zero, then either the electrons will never reach the sample (negative SIMS), or will be accelerated into the sample, causing further charge build-up (positive SIMS).

  The D.C. electron beam can be set on, concurrent with the sputter beam in the Phased Mode of depth profiling. In Acquisition Setup, in the Profile Settings tab for Phased profile, select "Sample has CComp" in the Sputter Phase. This will result in the D.C. electron beam and the sputter ion beam being on with the sample at ground. If a separate and additional charge compensation phase is desired, a value should be added in the Charge Comp Phase, in the Comp time box. If charge compensation is desired during data acquisition in the Analytical Phase, a value should be entered in the box for Comp. duty cycle% (typically 63%). If the Interleaved Mode of profiling is used, the sample is automatically set to ground between the LMIG pulses. To turn the electron beam on during sputtering, enter a value of up to 71 in the Duty Cycle, % field under Charge Comp. Pulse.

- **Use of metallic grids.**
  Another method for assisting with the profiling of insulators involves the use
  of one of the standard metallic grids placed over the sample in order to stop
  the sputter crater from moving during the D.C. sputter process. Rather than
  attempting to profile in the center of the grid mesh, it helps to be close to one
  of the corners, since the sputter crater will move little before being pinned in
  place by the conducting mesh. If the coarse openings prove to be too large,
  smaller grids such as those used for TEM sample preparation may be used.
  Typically, the electron beam is still used for charge compensation, even with
  the use of metallic grids

Retrospective Analysis: 'Raw' Data Files

During each cycle, of which there can be as many as 4×10^4 per second, every
secondary ion which arrives at the detector has a time-of-arrival, an x and y co-
ordinate, and also an elapsed time with reference to the total acquisition time. A
TOF-SIMS acquisition can be thought of as a multi-dimensional data set. At
every 256 x 256 pixel point, there is a full mass spectrum available and at every
secondary ion mass, there is an image available. Additionally, these data may
also change as a function of elapsed experiment time, for instance in a depth
profile. Such a wealth of data can be overwhelming. Therefore, one of the most
important questions to ask at the start of analysis is "Do I know what information
I need?" Many analyses consist of either mass spectral characterization of the
surface, or of mapping the lateral distributions of specific species x, y and z.

There are, however, many cases where the analyst cannot know beforehand what
are the data of interest. This would be the case, for instance, in a "good" vs.
"bad" failure analysis. In such cases, a facility is provided to record the time-of-
flight (mass), x and y position and appearance time for *every* secondary ion
which is detected. Data reduction of such files can occur off-line, when the data
can be replayed any number of times, experimenting with different mass images,
region-of-interest mass spectra and so on. Another example of the appropriate
use of this facility is when there is a very limited amount of sample, and the act
of acquiring the data essentially uses up the sample. This would be the case for a
single, small organic defect for instance.

Such files, which are known as 'Raw' data files, can occupy 40 Mb of data or
more, and so this facility must be used with care or else the hard disk will be
filled at an alarming rate. The software requires actively turning this feature 'on'
in the Acquisition, Setup acquisition menu for the 'save Raw data to disk'
function to be activated during an analysis. Once the acquisition has finished, the
'save raw file to disk' function turns itself off, to prevent from accidentally
acquiring vast amounts of unwanted data.

Data replay is achieved by turning on the function 'acquire from raw file' in the same 'advanced settings' page of the Setup acquisition page. The data can be replayed at either a 'fast' or 'real-time' rate. Since the data is a bit stream, samples with low ion yields (< 1 secondary per primary ion pulse) produce a bit stream where the replay time is far less than the elapsed time. Such files, when replayed at a 'fast' rate can be accelerated by up to a factor of ten. This is helpful for data experimentation (retrospective analysis). For teaching and demonstration purposes, a 'real time' facility is provided which exactly mimics the way in which data appeared in real time during the data acquisition. Acquiring from disk does not turn itself off once it has been turned on, and thus if the data is being replayed on a TRIFT instrument computer, remember to turn this facility off otherwise the same 'raw' datafile will be continually replayed over and over.

Region-of-Interest (ROI) Analysis

Because the primary ion beams are under computer control, and the raster generator is highly flexible, it is possible to conduct what is known as "region-of-interest" (ROI) analysis either during 'live' data acquisition or, as mentioned above, during retrospective analysis of 'raw' data files. A region-of-interest is defined by using simple drawing tools to define areas in an image from which data will be acquired. For example, if an image contains a defect area and a control area nearby, it is often desirable to:

- Produce a full raster image which clearly shows some secondary ion contrast defining the feature of interest, and
- Produce mass spectra from the two areas in order to compare and contrast the two regions.

The regions-of interest must be defined first, and then data may be acquired subsequently from those pre-defined regions. The region-of-interest facility is accessed in the Advanced settings page of the Acquisition, Setup acquisition page.

Depth profiles may also be stored as 'raw' files to allow reconstruction using species that were not identified as important during the 'live' data acquisition.

Section 8:
Charge Neutralization of Insulators

Sample Charging in SIMS with Positive Ion Beams

Whether the sample is a crystalline ceramic, a glass, or a polymer, the surface of the sample always charges positively when bombarded with a positive primary ion beam (in the absence of any electron neutralization).

In positive SIMS, the rate of arrival of positive charge (from the primary beam) is greater than the loss of positive charge due to positive secondary ion emission, owing to low secondary ion yields from most materials. This results in a net build-up of positive charge.

In negative SIMS, the charging occurs more rapidly. As well as gaining positive charge from the primary beam, negative charge is lost through the emission of secondary ions, and, more significantly, through the emission of copious amounts of secondary electrons. The net result is a build-up of positive charge once again, although this time it occurs at a higher rate.

When charging occurs, secondary ions continue to be emitted, but their energies can are shifted to levels which are outside the mass spectrometer acceptance 'window'. This 'window' is also referred to as the 'band-pass' of the spectrometer, and represents the energy band which can be successfully transferred through the spectrometer to the detector. The band-pass of the TRIFT spectrometer is large, approximately 240 eV, and this means that for a sample potential of 3,000 V, secondary ions with energies ranging from 3,000 - 3,200 eV can be passed around the spectrometer. For insulating materials, a compensating beam of electrons is required to prevent the analyzed area from charging up to a level where secondary ions are emitted with energies outside the spectrometer energy 'window'.

Most molecular ions are emitted with maximum energies of only a few eV above the sample potential, but atomic species can have very large kinetic energy spreads, often out to several hundred eV. This fact accounts for the high transmission of atomic species in the TRIFT spectrometer (often misconstrued from the resulting mass spectrum as a low transmission of molecular species). The significance of these observations will become evident in the following section "Recognizing charging effects: system diagnosis".

Recognizing Charging Effects: System Diagnosis

The appearance of the mass spectrum can reveal significant information concerning sample charging effects, if the neutralization scheme is under suspicion.

As stated earlier, under positive primary ion beam bombardment, the sample will <u>always</u> charge positively, no matter what SIMS mode you have selected. However, this positive charge build-up has a different effect on the appearance of the mass spectrum, depending upon the SIMS mode.

Positive SIMS

Refer to the Figures 8-1 through 8-4. Note the following:

1. Correct neutralization: This results in the kinetic energy distributions of all secondary ions being positioned correctly within the spectrometer energy 'window'. The resulting mass spectrum displays intensities of atomic and molecular species that accurately represents the ratio of emission of these species from the sample surface.

2. Beginnings of positive charge build-up: Now the kinetic energy distributions of the secondary ions are shifted to a higher value. This results in some 'trimming' of the higher energy atomic ions, but the effect is hardly discernible in the mass spectrum.

3. Continued positive charge build-up. The kinetic energy distributions are shifted further to higher energies. Since the maximum of the distribution for atomic species occurs at higher values, the 'trimming' of higher energy species can result in a mass spectrum which appears to have lower intensities of atomic species.

4. Severe positive charge build-up. As the kinetic energy distributions are shifted out of the spectrometer energy 'window', the total secondary ion count rate falls off dramatically. A mass spectrum acquired just before this occurs would have low signal intensities, and would be heavily biased towards molecular species.

Figure 8-1. *Recognizing Charging Effects: The Appearance of the Positive Mass Spectrum (1).*

Figure 8-2. *Recognizing Charging Effects: The Appearance of the Positive Mass Spectrum (2).*

Legend: | | represents the Energy Window.

Figure 8-3.   *Recognizing Charging Effects: The Appearance of the Positive Mass Spectrum (3).*
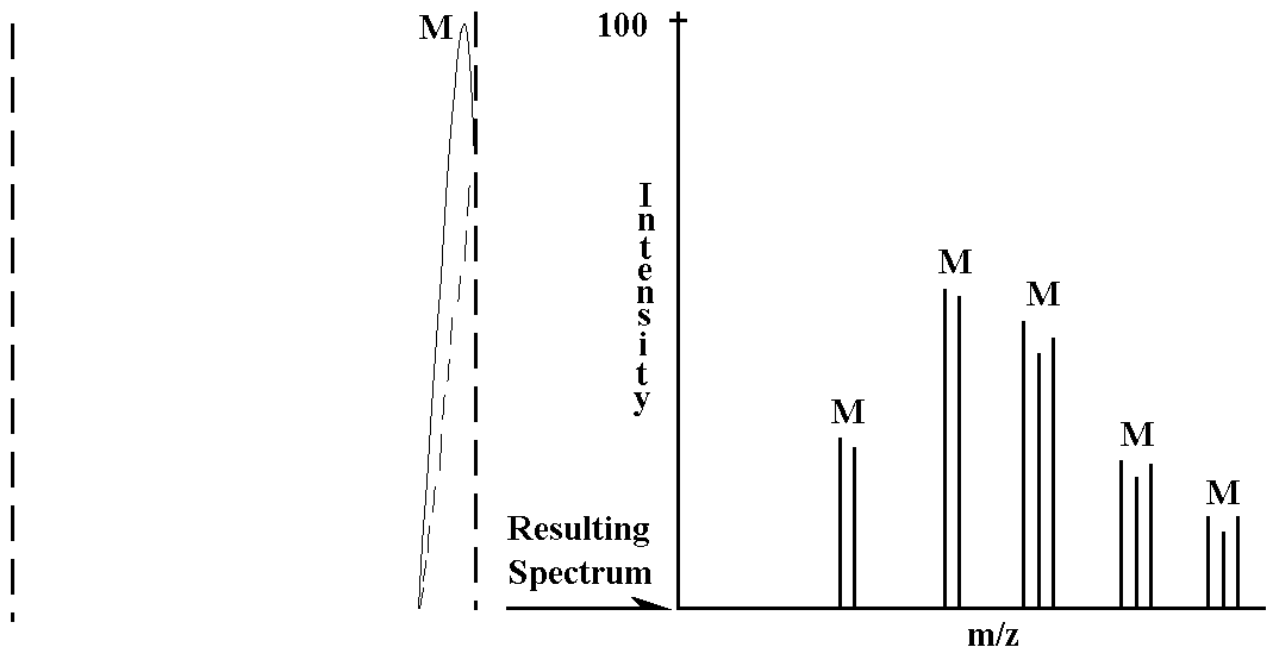
Figure 8-4.   *Recognizing Charging Effects: The Appearance of the Positive Mass Spectrum (4).*

Negative SIMS

Refer to the Figures 8-5 through 8-8. Note the following:

1. Correct neutralization: This results in the kinetic energy distributions of all secondary ions being positioned correctly within the spectrometer energy 'window'. The resulting mass spectrum displays intensities of atomic and molecular species that accurately represents the ratio of emission of these species from the sample surface.

2. Beginnings of positive charge build-up: Now the kinetic energy distributions of the secondary ions are shifted to a <u>lower</u> value (a positive charge build up on a negative surface potential results in a less negative surface potential). This results in a 'trimming' of the low energy ions, and consequently the molecular ions first of all. The resulting mass spectrum appears to have a reduced yield of molecular species.

3. Continued positive charge build-up. The kinetic energy distributions are shifted further to lower potentials. Since the maximum of the distribution for molecular species occurs at lower values, the 'trimming' of low energy species can result in a mass spectrum which appears to contain <u>only</u> atomic species.

4. Severe positive charge build-up. As the kinetic energy distributions are shifted out of the spectrometer energy 'window', the total secondary ion count rate falls slowly with potential changes, owing to the high energy tail of the atomic species. A mass spectrum acquired just before this occurs would have low signal intensities, and would be heavily biased towards atomic species.

In the absence of an electron beam, these effects will always be observed with insulators, at rates that are determined by the dielectric properties of the materials. It is instructive to turn off the electron gun and take a series of mass spectra, sequentially, as this charging process occurs, so that a 'feel' can be developed for the effects of charging, as manifested in a mass spectrum.

These effects are reproduced more controllably by taking TOF-SIMS spectra from a conducting sample, altering the sample voltage to simulate the effects of charging on the emission energies of the secondary ions (increase the sample voltage progressively for positive SIMS, and decrease it progressively for negative SIMS).
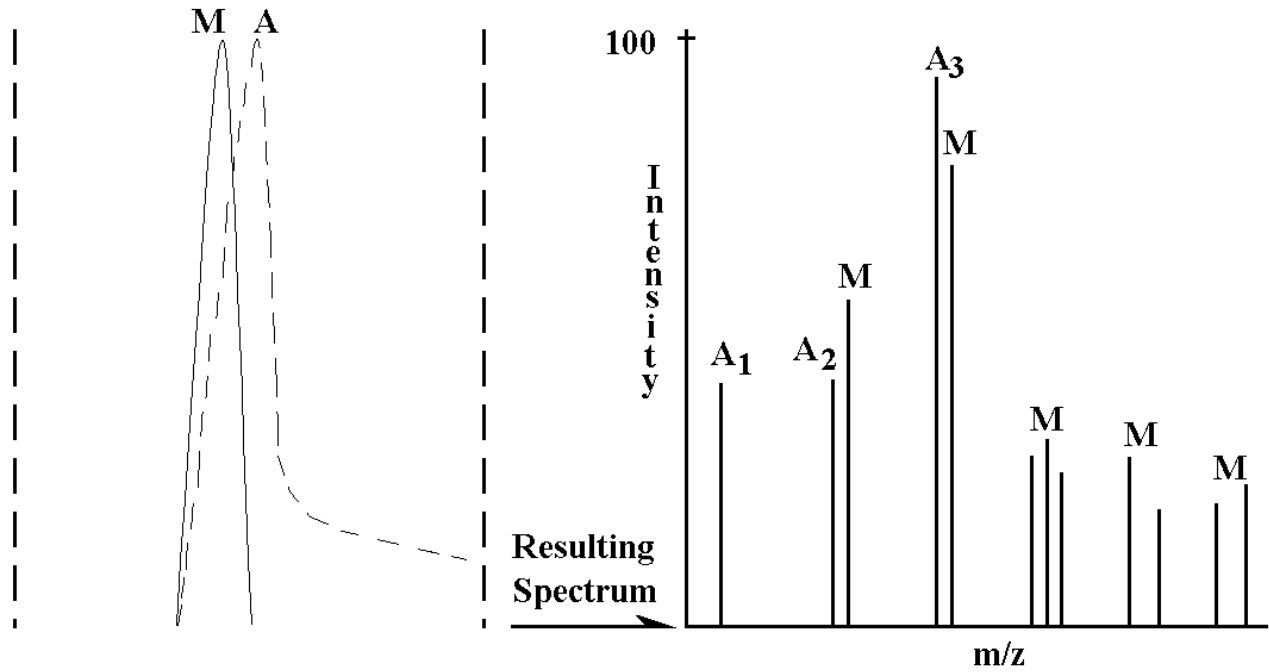
Figure 8-5.   Recognizing Charging Effects: The Appearance of the Negative Mass Spectrum (1).
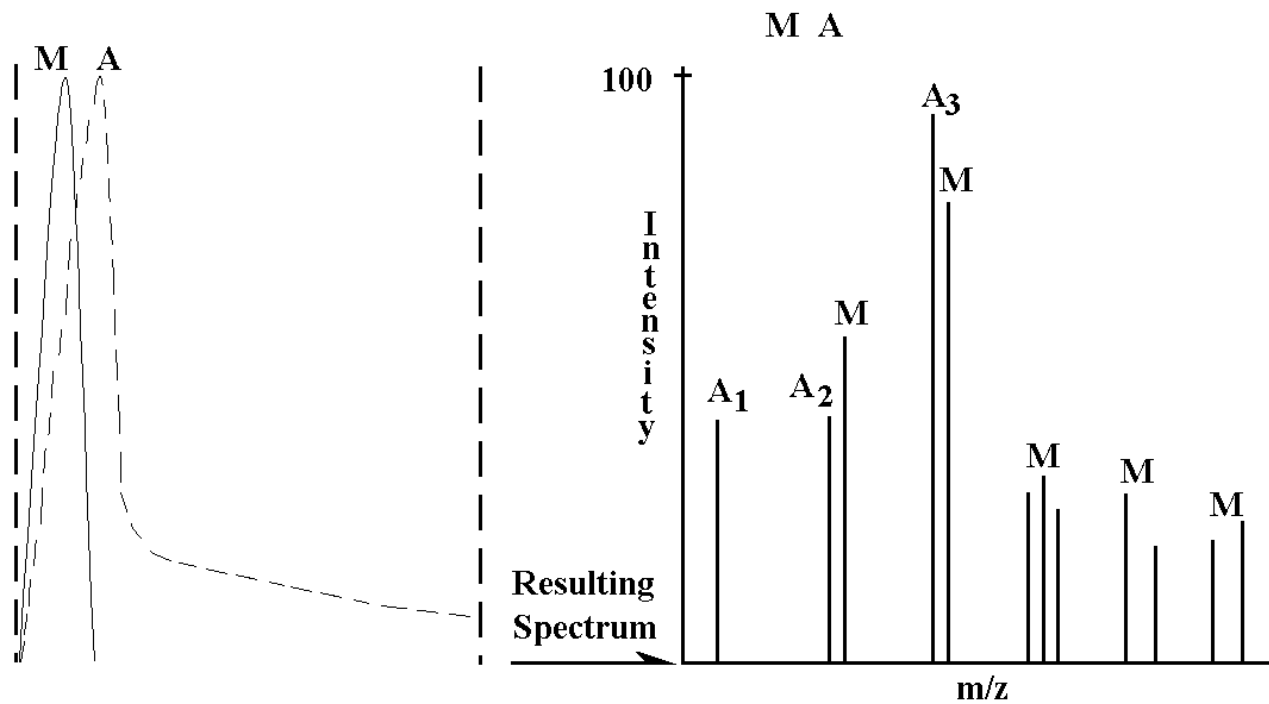
Figure 8-6.   Recognizing Charging Effects: The Appearance of the Negative Mass Spectrum (2).

Legend: | | represents the Energy Window.
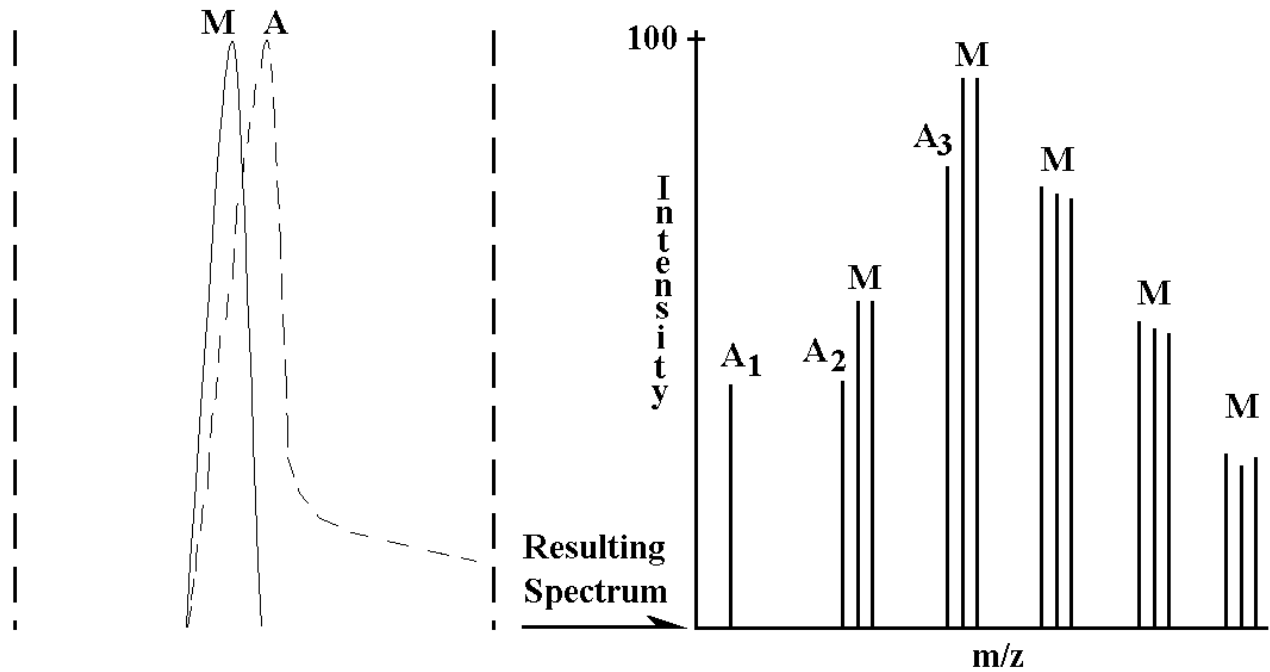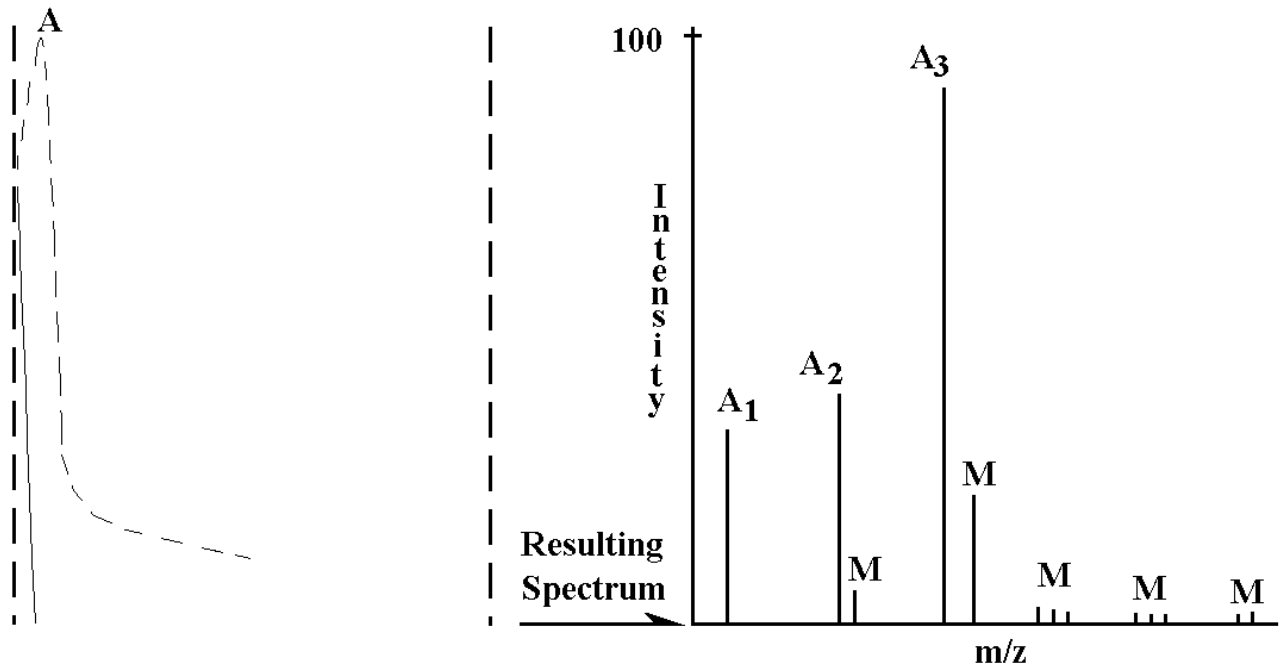
Figure 8-7.   Recognizing Charging Effects: The Appearance of the Negative Mass Spectrum (3).
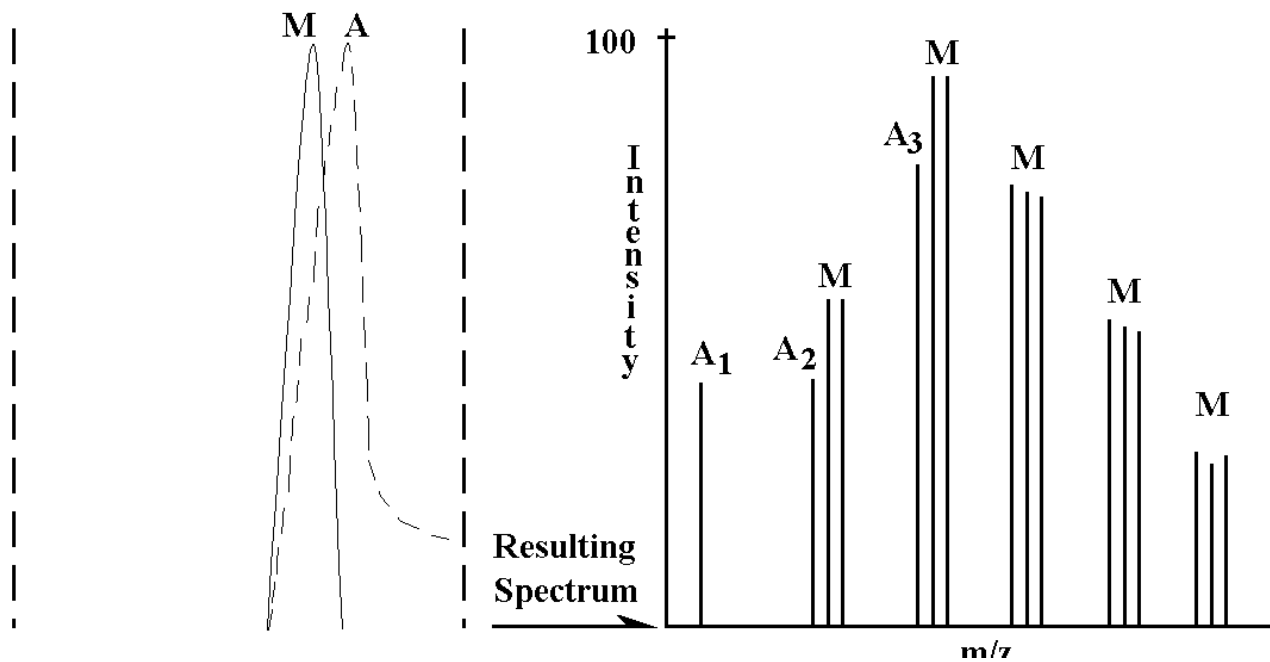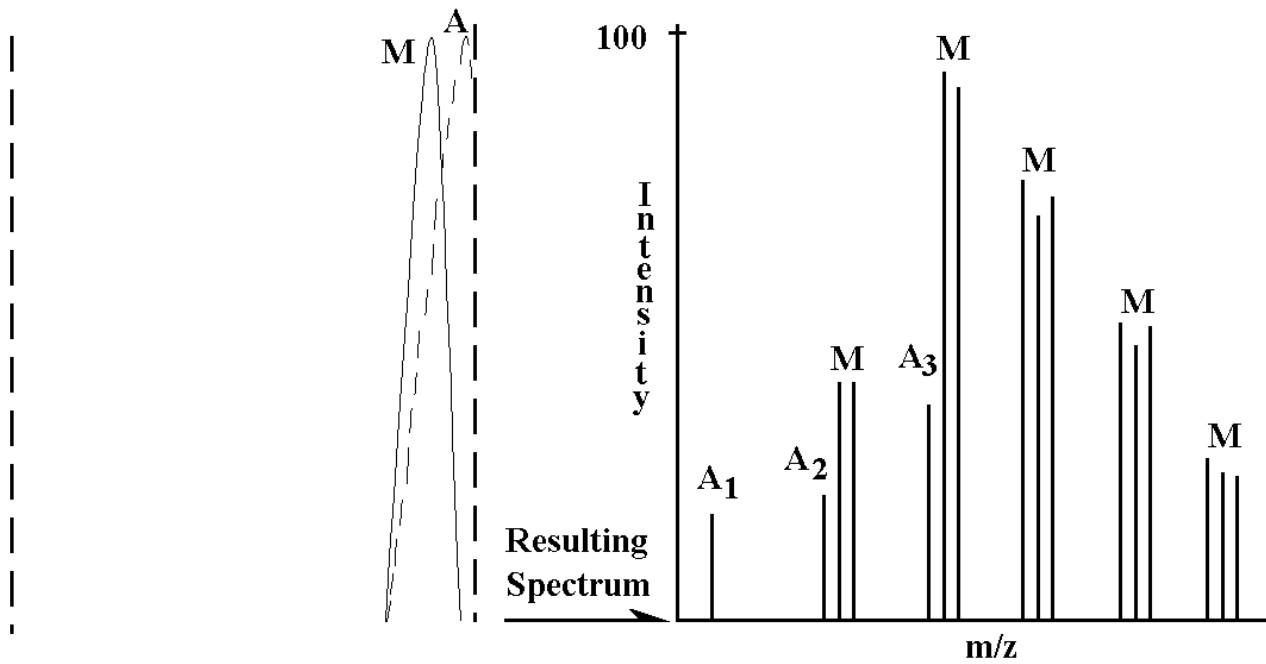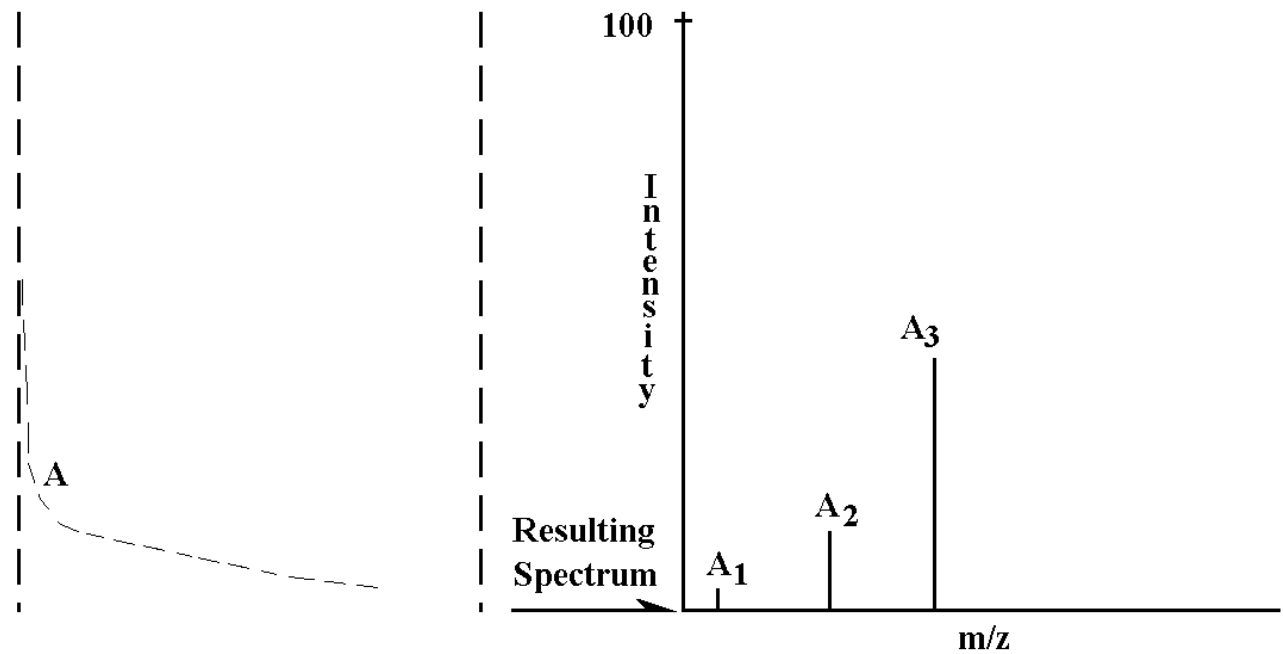
Figure 8-8.   Recognizing Charging Effects: The Appearance of the Negative Mass Spectrum (4).

Legend: | | represents the Energy Window.

Extraction Field Penetration: Techniques for Obtaining Data

It is possible to have correct electron neutralization and yet still obtain poor results from large area, thick insulators, because the extraction field is too low in the area under analysis. This is due to variable field penetration at the insulator surface.

One simple technique which can be used to overcome this effect is to increase the sample potential to a level where, in the area under analysis, the extraction field attains the correct value of 3,000 V (or whatever). Secondary ions are emitted with energies that are 'matched' to the spectrometer energy 'window'. Adjusting the sample potential for maximum secondary ion yield displayed on the rate meter is the normal method.

One drawback with this method, however, is that the required setting of the sample potential tends to be area-specific, i.e., moving to another area on the sample, will typically result in the need to re-tune the sample potential. This is because the field penetration can vary significantly from one area to another.

Use of Metallic Grids

An alternative method for obtaining even field penetration across the sample surface is the use of conducting grids. A grid prevents large variations in the extraction field over a large area insulator. This means that increasing the sample potential is virtually never necessary, since the correct extraction field is obtained all over the sample surface. It is possible, therefore, to move from one grid 'window' to any of the other 'windows' without re-tuning.

It is important to note that placing a grid or mesh over an insulating sample has nothing to do with electron charge neutralization. The grid merely 'pins' the extraction field on the sample surface and allows the user to use the default voltage settings. If using a grid, but without an electron beam, the sample will (eventually) charge up and resulting in the inability to acquire data. If an electron beam is present but without a grid, it is still possible to obtain good, neutralized data, provided the sample potential is varied to the optimum value every time.

The grids provided with the instrument have a thickness of 125 microns, and are fabricated from a non-magnetic stainless steel. The grids are designed to be rigid, and it is possible to re-use them many times, provided reasonable care is exercised.

When mounting a sample with a grid, simply lay the grid inside the inverted sample holder and then place the sample surface to be analyzed against the grid. When inserting the backing plate and Cu-Be sample clip, use the minimum compressive force to hold the sample in place to prevent deformation of the grid. If the grid mesh does not lie flat on the sample surface, but is raised up off the surface, this will cause the sample to be shielded from the field, leading to ion extraction problems. Refer to the portion of Section 9 entitled "Difficult Geometry Samples".

Practical Guide to Insulating Samples

NOTE: When performing analysis on insulating materials, the goal is to pass secondary ions with an optimum energy into the mass spectrometer.

Two simple principles need to be discussed when analyzing insulators: a) the mass spectrometer energy 'window'; and b) field penetration of insulating materials. Once these principles are understood, insulator analysis can be approached much more rationally and viewed much less as a 'black art'.

First, consider the analysis of a 'standard' conductor to introduce the idea of the mass spectrometer acceptance window.

1. **Why** SIMS **signals expected without performing a tuning when a conducting sample is loaded and optimized.**

One of the procedures carried out during instrument checkout is to tune the mass spectrometer for optimum performance. This requires laser-assisted alignment of the spectrometer housing to the analytical chamber, as well as setting the correct values of the various ion optical elements: the immersion lens; the transfer lens; the ESA's and the Matsuda plates. Once set correctly, the TRIFT mass spectrometer can almost be regarded as a 'black box': in other words, no adjustment to any parameters on a regular basis is necessary, nor is anyone required to understand how it functions to be able to acquire good data.

The mass spectrometer, when 'fully open' (i.e., no energy filter) has an energy acceptance window of ±120 eV. This indicates that an ion with ±120 eV about the optimum value will be passed around the spectrometer to the detector. The closer the ions energies to that optimum value, the better the energy and time focusing of the spectrometer, resulting in optimum mass resolution. Conversely, as the secondary ions' mean energies approach the outer limits of this energy window, the energy focusing is not as efficient, and the mass resolution degrades.

An "optimum" value has been mentioned twice, but what is this value? It's basically the energy that an ion must have for it to travel down the centerline of the mass spectrometer. The mass spectrometer has been set to pass ions with a mean energy of -3000 eV around the centerline. Provided that secondary ions enter the spectrometer with this mean energy, they are transported and optimally

energy focused at the detector. They acquire such energy by traveling through the extraction (acceleration) space.

The following analogy may help to reinforce what is happening within the system. Imagine yourself at the top of a steep slope sitting on a 4-wheel sled. The sled has no pedals or engines to propel you, no steering wheel, and no brakes! An accomplice positions you to the very brink of the slope and gently tips you over its edge. The sled begins to accelerate, and velocity increases until you reach the end of the slope. The sled begins to glide with a uniform velocity. After some distance, a building with a doorway leading to the inside of a chamber is encountered. The chamber walls are not vertical, but slope so that the top of the chamber is more open than the bottom, like an inverted cone. If your kinetic energy is correct, the sled stays on a centerline of the chamber wall and passes around 270 degrees to a conveniently located exit door which exits out to safety. If your kinetic energy is severely mismatched to the chamber dimensions, you will either fall to the bottom of the inverted cone, or be spun out at the top. If your kinetic energy is close to but not at the optimum value, you may escape from the chamber provided that the exit door is wide enough. If the chamber dimensions are not to be changed, the height of the initial slope must be adjusted instead to make sure that you pass safely around the centerline and out through the exit door.

The kinetic energy of the secondary ions is determined primarily by the sample potential (equivalent to the height of the slope in our analogy). On a conducting sample, the default instrument settings apply a sample potential that correctly matches the secondary ion kinetic energies to the optimum acceptance energy of the mass spectrometer. No additional tuning is required for a conductor because the sample surface potential is the same as the applied potential. Secondary ions created at the sample surface are launched at a potential which results in kinetic energies that are matched to the mass spectrometer.

The method for determining this correct potential for a conductor is described in the mass spectrometer section within this manual.

2.   What happens when thin, insulating samples are loaded?

(a)   Let us suppose that we now load a sample which consists of a 2 μm thick polymer film on a metal substrate. The film's dielectric properties are such that it is an insulator, and thus we need to invoke the TRIFT's electron charge compensation system to balance the incoming positive charges from the primary ion beam.

Since the polymer film is a dielectric material, the field potential lines from the nearby sample holder permeate the film and terminate at the underlying (conducting) substrate. Since the extraction space is 2 mm (2000 μm), the field line which coincides with the polymer film surface is not the 3000 volt contour, but

$$3000 - (3000 \times (2 / 2000)) = 2997 \text{ volts}$$

In this simple example, the sample potential would need to be raised -3 volts to ensure that the field potential at the sample surface is 3000 volts. In such a case, this can be ignored, and the most critical aspect of the analysis is the effectiveness of the electron neutralizer.

(b) Suppose that the film thickness is 100 μs, rather than 2 μm. The *actual* potential at the sample surface (resulting from the default file) would be:

$$3000 - (3000 \times (100 / 2000)) = 2850 \text{ volts}$$

If the applied potential were <u>not</u> raised -150 volts, the secondary ions would be gain a total of 2850 eV kinetic energy and would enter the mass spectrometer with a mean energy that was 150 eV lower than the optimum. The ions would <u>not</u> make it around the mass spectrometer (energy acceptance ±120 eV). In this case it is critical to raise the applied sample potential by ~150 volts in order to achieve the correct field potential of 3000 volts at the sample surface. Note that it would be very easy to misdiagnose the lack of secondary ion signals as being due to poor electron neutralization.

If you know the approximate thickness of an insulating film, you can estimate by how much you will need to raise the sample potential to achieve a -3000 volt potential at the sample surface, even before attempting the analysis. In practice, even on an extremely thin insulating film, it is necessary to raise the sample potential approximately 40 volts, in part to compensate for the fact that the sample potential is not applied to a zero volt surface, but rather to a surface which has floated down to -20 to -25 eV under the influence of the low energy electron beam from the charge neutralizer.

3.   Practical method for determining the correct sample potential on insulating samples.

It should be clear from the foregoing that the moment you load an insulator, and especially a thick insulator, you will need to determine the appropriate applied potential that will result in a 'real' potential of ~-3000 volts at the sample surface. This at first sounds daunting, especially if you have almost no knowledge of the dimensions or properties of the insulator. In fact, it is very simple to determine.

Make sure that the electron charge compensation is on (filament on and 'on' in the software), and then begin an analysis, preferably over a large area (~400 μm). If no secondary signals are detected, begin raising the sample potential until some counts are detected. Raise the sample potential until you appear to have passed through a-maximum in the count rate, and note that count rate. Now lower the sample potential until the count rate falls off to ~10 to 20% of its maximum value. Note down this sample potential. Now begin raising the sample potential once again, go through the maximum count rate, and then note the sample potential on the <u>high side</u> at which the secondary count rate again falls to ~10 to 20% of the maximum value. The difference in the two sample potentials should be ~240 eV, mapping the energy 'window' of the mass spectrometer. If you set the sample potential half way between these two values, i.e. ~120 volts up from the low potential, you should have a good approximation of the correct applied potential required to match secondary ions to the center of the mass spectrometers energy window. For optimum performance, you may need to vary the sample potential by 10 volts either side of this value to determine if the mass resolution can be optimized further.

Enthusiastic students can perform this experiment on a simple, metallic conductor and see the mapping of the energy 'window' under ideal conditions. Naturally, the correct setting should be at the default potential, but the experiment is instructive because on the low side of the correct setting you should observe that the counts fall off more slowly than on the high side. This is due to the shape of the kinetic energy distribution for atomic species. As you lower the applied potential, the kinetic energy distribution of the secondaries are sliding to the low energy side of the spectrometers energy window. Eventually, only atomic tails appear in the spectrometer energy window, and a mass spectrum acquired at this point would be dominated by atomic species.

Conversely, on the high side of the correct applied sample potential, you are effectively sliding the kinetic energy distribution to the high energy side of the spectrometer's energy window. The count rate remains high (although the energies are no longer correctly ideally matched to the spectrometer), until over the course of a few volts the main peaks of the kinetic energy distributions for both atomic and molecular ions disappear completely from the spectrometer's energy window. At this point the secondary ion count rate falls dramatically. A mass spectrum acquired just before this occurs may even be dominated by molecular species, since it is possible to push the peak of atomic energy distributions out of the energy window and yet still keep the molecular ions within the energy window (just). The correlation of the observed mass spectrum and the position of the kinetic energy distributions within the mass spectrometer's energy window are shown in the schematics which are used to demonstrate what happens when the neutralizer is not functioning correctly and the sample begins to charge up.

4. Practical procedure for analysis in 'worst case' scenario: a thick insulator-mounted at the wrong height.

Having considered points 1, 2, and 3 above, we can now go on to consider a worst case' scenario in which a thick insulator is mounted at the incorrect height. In this case there are now three aspects to consider: a) correct neutralization (taken for granted for the moment); b) finding the correct applied potential; and finally, c) the steering of the primary beam to the spectrometer acceptance area. Raising the sample potential alone may not have the desired result of finding the secondary ion beam if the primary beam is not illuminating the acceptance area. If you can estimate the insulator thickness, raise the potential accordingly and then sweep the x beam position to try to find the secondary beam. If you cannot estimate the change in sample potential, it will be necessary to raise the sample potential in ~100 volt increments, and then sweep the x beam position at each sample potential until you find the beam.

General Description of the Electron Neutralization Scheme

The electron neutralization scheme used in the TRIFT III TOF-SIMS system is unlike most electron neutralization schemes employed with most other D.C. primary ion beam SIMS systems. After initial settings are performed, it is self-regulating and rarely requires any additional tuning.

The electron neutralization scheme consists of a broad (~3 mm), low energy (<25 eV) beam which is concentric with the secondary ion optical gate, as outlined in Section 4.

When charge compensation is turned on in the Acquisition/Acquisition Set-up menu, the default sequencer enables the following sequence of events:

1. After a set number of primary beam pulses with the sample potential at HV, the sample potential is forced to zero by the high voltage pulsing unit.

2. Once the sample potential is at zero (0), the electron gun is fired, producing a stream of low energy electrons aimed at the sample surface.

3. Electrons continue to arrive at the sample until the surface potential approaches -25V, at which point electrons are deflected away.

As previously mentioned, the system is self-regulating and rarely requires tuning. The only exception would be the certainty that the beam is being steered to the correct place on the sample. This requires resetting only if the electron gun is removed from the system, for instance, to change the tungsten filament Refer to the portion of Section 4 entitled "Electron Neutralization Gun".

Changing Default Electron Gun Parameters

The default electron gun parameters have been optimized in the sequencer settings of the instrument files and should not need adjustment. The sequencer files have been set to permit electron charge neutralization at acquisition mass ranges of 1850m/z and higher. Therefore, when "Analytical with chg. comp" is selected in the Acquisition Setup menu, the mass range is automatically increased to 1850m/z if it was previously set lower. This allows an electron pulse to occur after *every* primary ion pulse. If the mass range is changed to less than 1850m/z with charge compensation, the electron gun will only pulse every other ion beam pulse.

The electron pulse duration is typically set at 63% of the time that the sample bias is at zero. This parameter can be varied in Acquisition Setup. With "Analytical with chg. comp" selected, view the Profile Settings tab, and vary the Charge Comp Pulse duty cycle. The should always be set at the maximum allowed value for optimum charge neutralization.

Techniques for Optimum Charge Dissipation

When using a standard 'linescan' raster with a focused LMIG probe on a 'high' insulator, it is sometimes possible to see the effect of surface potential changes on the secondary ion image displayed on the DMCP. It may appear that the linescan width increases and that the line of displayed secondary ions becomes more diffuse as the frame sweep progresses. This is probably due to changing surface potentials as the sweep progresses across the insulating surface.

The software is capable of rastering the primary beam in a 'scatter' raster to address each pixel point such that the next beam pulse is as far away from the previous one within the analyzed area. Although this raster is referred to as a 'scatter raster', it is not truly random in nature, since the separation between each pixel point is constant, and the sequence of pixel points follows a well-defined geometrical pattern.

Using this raster, the charge resulting from each frame of acquisition is distributed evenly across the analyzed area, facilitating charge neutralization. This raster should be used as the default mode for analysis when the charge compensation is used. A variety of raster modes are available and can be set as defaults under different analytical conditions in the "hardware" section of the software. For further details, refer to the portion of Section 7 entitled "SIMS Images".

Section 9:
Sample Preparation Techniques

This entire section of the Operator's Guide pertains to sample preparation. Because samples come in different shapes and sizes, sample mounting and preparation can be difficult. A number of different techniques are addressed including used in preparing different types of samples including powders, slurries, fine suspensions, cross sections, and fracturing samples.

Powder Samples

Typically, a user may think that a vertical stage must, by force of gravity, present many more problems than a horizontal stage when considering powder samples. In fact, the greatest threat to the integrity of powder samples for surface analysis comes not from the stage orientation, but from the initial pump down phase inside the load lock, due to the rapid removal of air from between the powders. Thus, the *most important problem* to overcome is the powders' tendency to blow apart during initial evacuation. This is the same challenge irrespective of stage orientation.

A number of techniques can be used to overcome this problem, and generally speaking, if the powder samples can be kept intact and adhering to a substrate during pump down, they will remain firmly in place no matter whether the sample is held vertically or horizontally in the analysis chamber.

Loose Powders

Loose powders require careful handling, because the moment such samples are placed in a load lock and evacuated, the powders can fly everywhere, contaminating the vacuum system. Generally, the most expedient ways to prepare such powders include the following options:

Trapping the powders on double-sided adhesive tape.
This is by far the most convenient and therefore widely used method. A small sample of tape (5mm x 5mm) is placed onto a metal substrate or sample holder, and the loose powder is sprinkled onto the upper surface of the tape. An excess of powder is normally used to guarantee full coverage of the tape. Then the excess powder is removed by inverting the sample and lightly tapping the back of the substrate. If necessary, a low pressure compressed air line or inert gas dust remover may be used. The remaining powders are well attached to the surface. The sample may then be inserted into a load lock and the load lock pumped down.

The only warning concerning adhesive tape is the quality of the tape used. Some tape products are contaminated by PDMS (polydimethyl siloxane), and this liquid can flow and contaminate powders prepared in this way. The solution is to analyze a number of tape products from the local stores to determine which brand is siloxane-free. Once a good brand is found, contamination of the powders is no longer a concern.

1	2	3	4
Small piece of double-sided tape on flat surface.	Excess of powder spinkled onto double-sided tape.	Substrate inverted to remove excess powder.	Powder sample prepared.
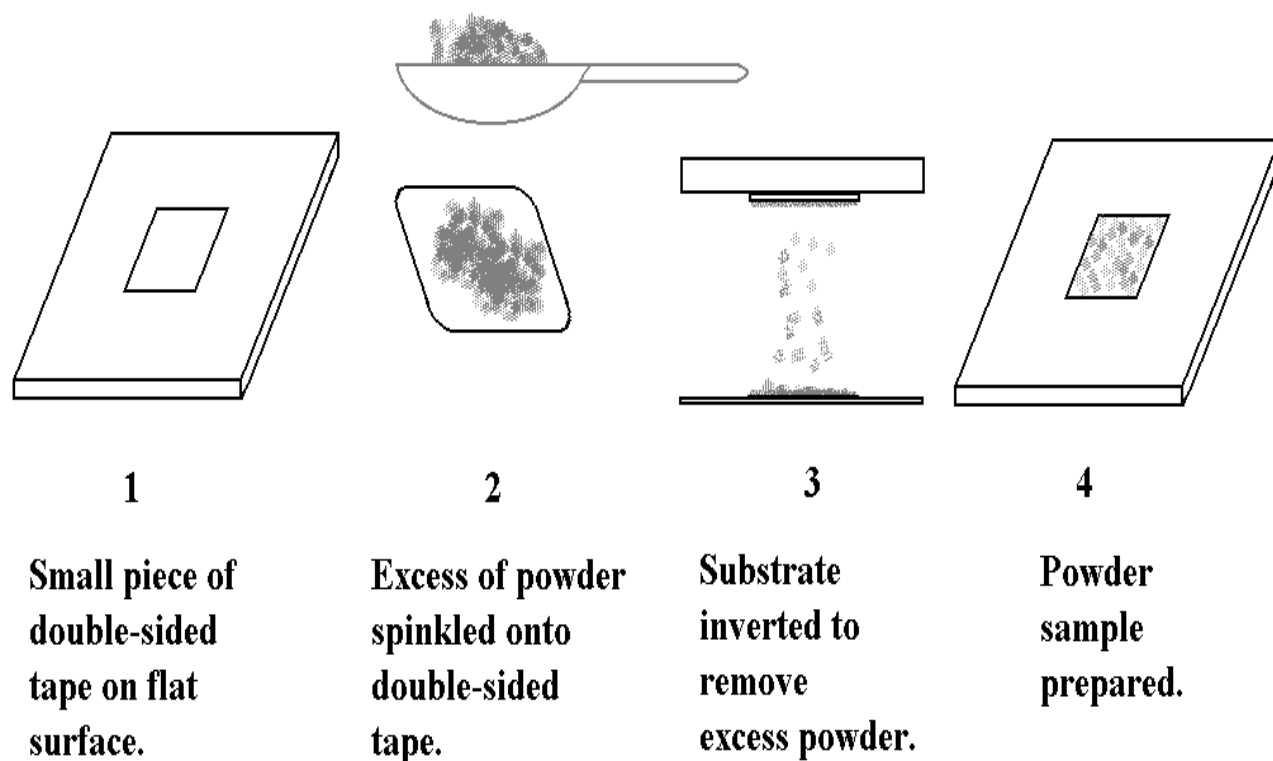
Figure 9-1.   *Diagram of Trapping Loose Powders on Double-Sided Adhesive Tape.*

b. **Preparing an indium/powder "sandwich".**
   Despite the success and ease of preparation described in (a) above, some may worry that the adhesive will contaminate the powders. In such cases, the loose powders may be prepared in the following manner. Cut two small squares from a thin sheet of soft, indium metal (approximately 0.5mm thick); sprinkle powder on one of the squares and then place the second piece of indium on top of the powder. Compress the "sandwich" with a suitable flat tool. Peel the two pieces of indium apart and remove any excess loose powder. The powder that remains is held firm in the soft metal surface. The beauty of this method is that the powders press against themselves and do not pick up indium on their outer surfaces. Again, the forces holding the powders in place are high, and such samples can be readily inserted into the load lock to be pumped down.

1	2	3	4	5
Indium foil with loose powder.	Second indium foil placed over the first foil.	Two layers compressed together.	Peel foils apart.	Two powder samples prepared.
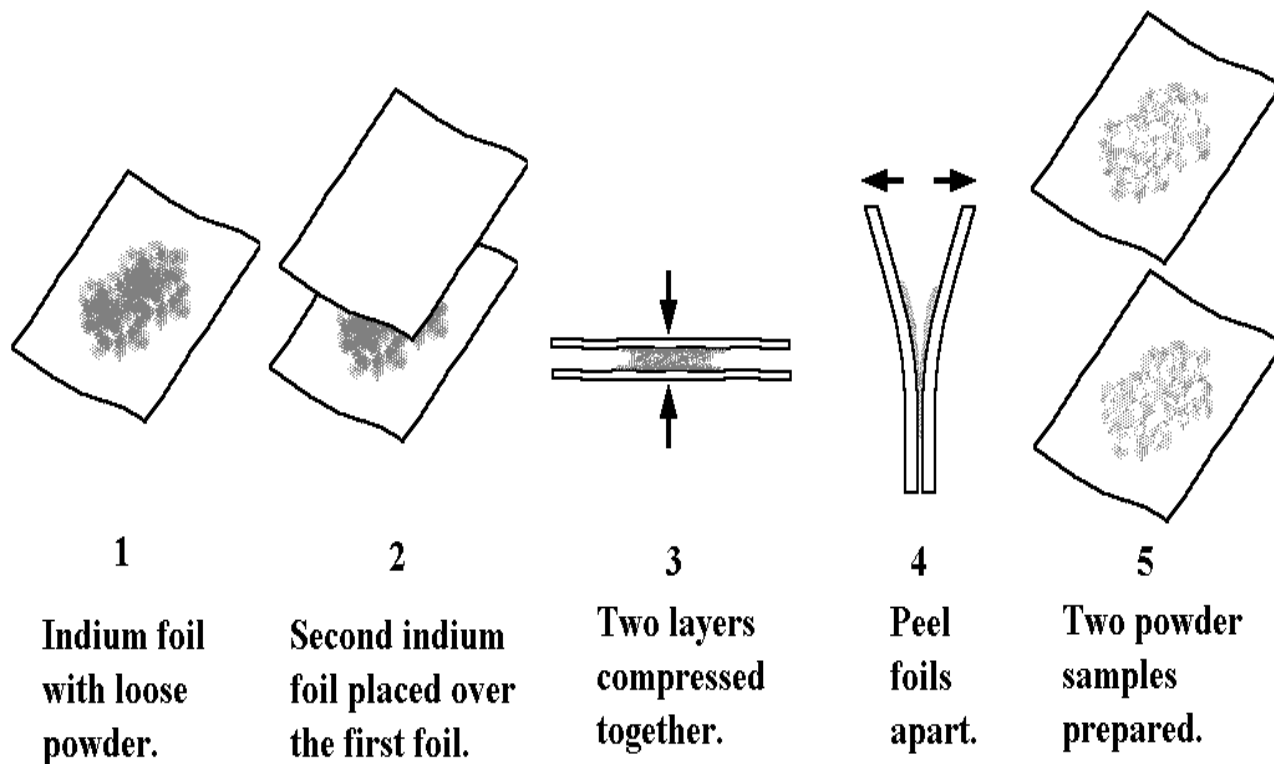
Figure 9-2.   *Diagram of an Indium Powder "Sandwich".*

c. **Preparation of dry powder compacts.**
In this method, a suitable die and press is used to form a pellet from the dry powder. The compacting of powders is due only to mechanical keying of the material to itself, yet the bond is quite strong. The pellet is then fractured to reveal a fresh powder surface which has never been in contact with the die materials. The user can load the pellet into the load lock and rest assured that it will not fracture apart into the powder material it.

1	2	3	4	5	6
Three-piece die.	Loose powder in the die.	Die compressed: powder compacted.	Pellet sample.	Pellet sample fractured.	Two samples prepared.
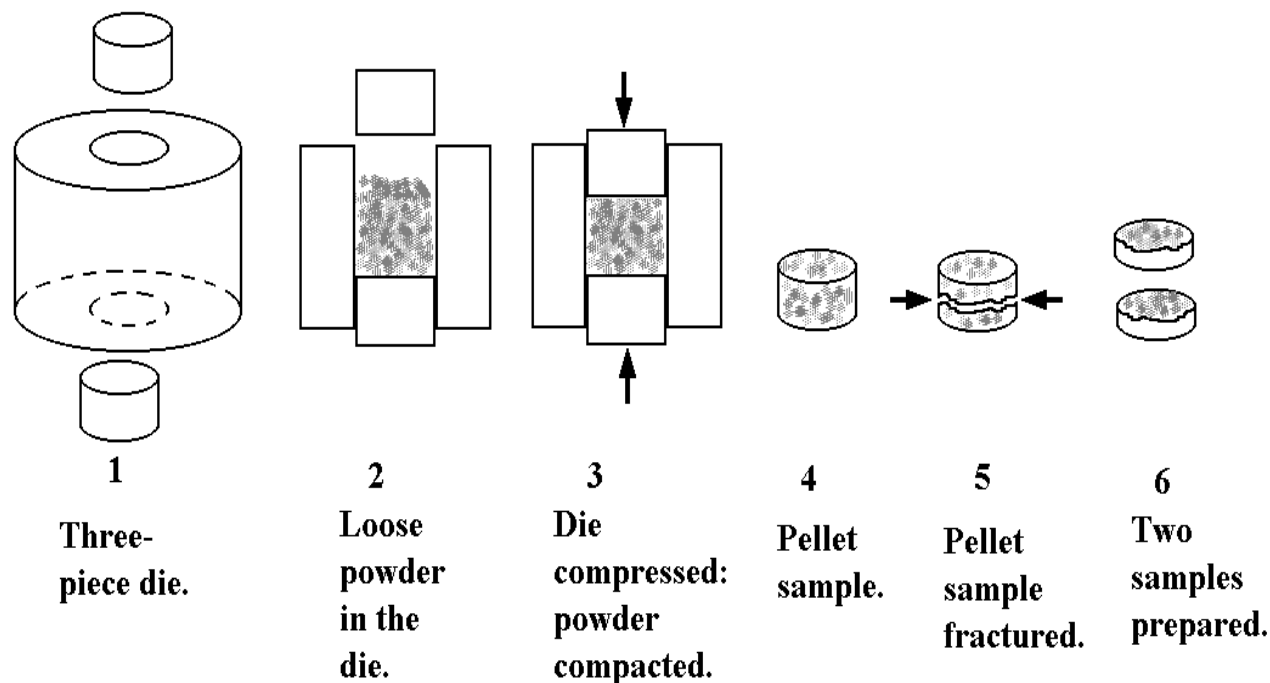
Figure 9-3.   *Diagram of Dry Powder Compacts.*

(d) Settling from fine suspensions.

Sometimes analysis must be carried out on powders where it was important to have a very low surface coverage of the powder, so that individual particles can be studied. In such cases, and where a suitable inert liquid can be identified, it is very easy to simply pipette a droplet of a suspension onto a suitable substrate (such as a carbon planchet). Powders prepared by this method are remarkably tenacious (i.e., great adhesion to a surface), but powders exhibiting poor adhesion may be removed by inverting the planchet and tapping the backside. Note that this method is not good when the powders become large, namely ~>100um, because the bond to the substrate is not as strong as in the above methods. Adhesion can be improved by lightly abrading the substrate beforehand with an abrasive which has a similar dimension to the particles under investigation, greatly improving the "mechanical keying" of the particles to the substrate.

1

Lightly abraded carbon substrate.

2

Droplet of powder suspension in suitable liquid.

3

Low surface coverage of powder on carbon substrate.
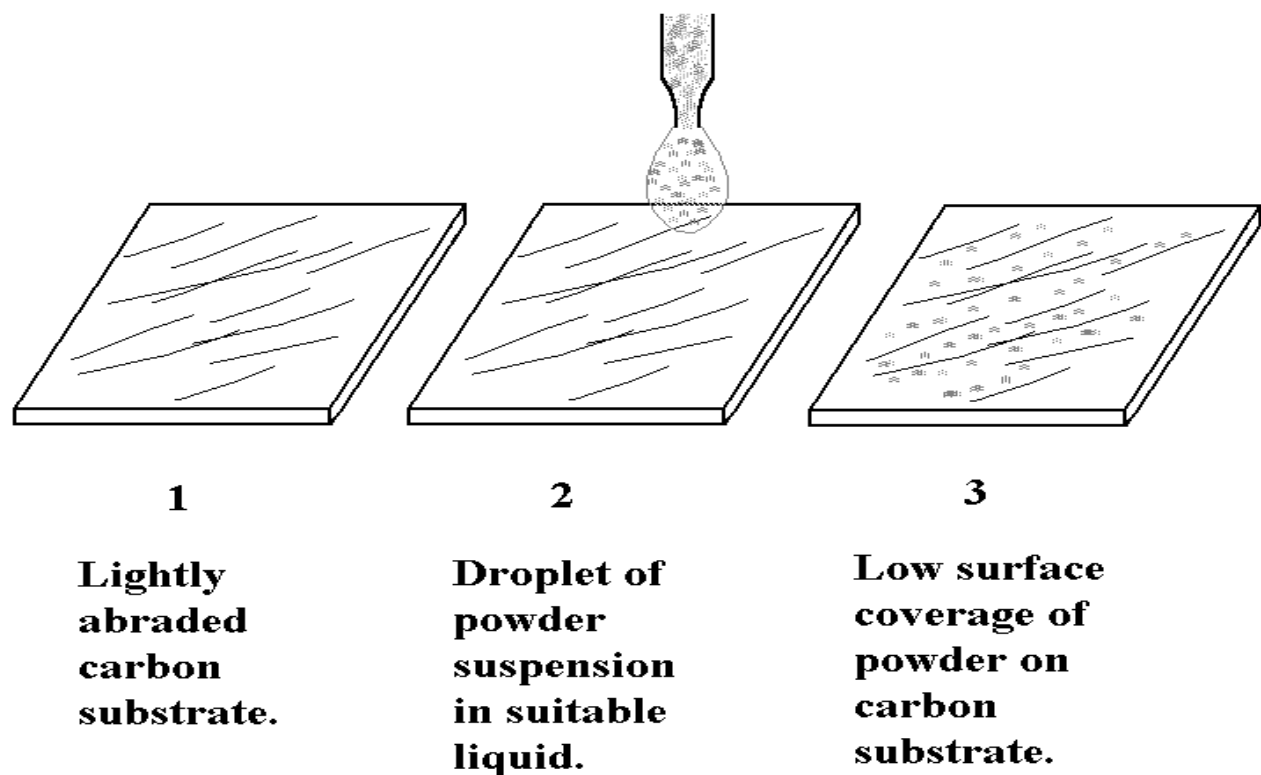
Figure 9-4.   *Diagram of Settling from Fine Suspension.*

Preparation from slurries

Preparation of samples from slurries is relatively simple, since slurries dry with a significant 'green strength' compared to loose powders, even dry powder compacts. This is due to the large range of particle sizes which allows the slurry to dry with a high degree of mechanical keying to both itself and the substrate. Several different methods are discussed below:

a. The simplest method is to pipette a droplet of the slurry onto a suitable substrate, such as a carbon planchet as mentioned above. Carbon is preferred for two reasons. First, carbon has a low sputter and SIMS yield, and therefore, particles tend to "light up" against a dark background. This allows the user to locate particles even at low coverage. Secondly, it is possible to increase the tenacity of the dried slurry to the substrate by prior abrasion, as mentioned in (1c) above. If the dried slurry is rather thick, it can experience cohesive shear failure from the substrate (separates as an intact body). This can be avoided either by controlling the amount of slurry used, diluting the slurry, or increasing the mechanical keying of the slurry particles to the substrate, or all three of the above. Because of the tenacity of thin slurry films, and the ability to verify the tenacity before entry into the vacuum system, such samples present few problems inside the analytical chamber.

For this reason, after preparation of dry powders onto double-sided adhesive

tape, this is the second most convenient way to prepare powder samples for analysis, provided of course, that it is legitimate to use a liquid for suspension purposes.

b. Alternatively, the separation of the dried slurry from the substrate can be exploited by removing the dried layer and placing it onto double-sided adhesive tape as outlined in (1a). This ensures that the dried layer is held firmly in place for subsequent analysis.

c. Slurries may also be allowed to dry in suitable molds to produce thick pellets or cakes which may be fractured to produce fresh surfaces, and then used as described in (1c) above.
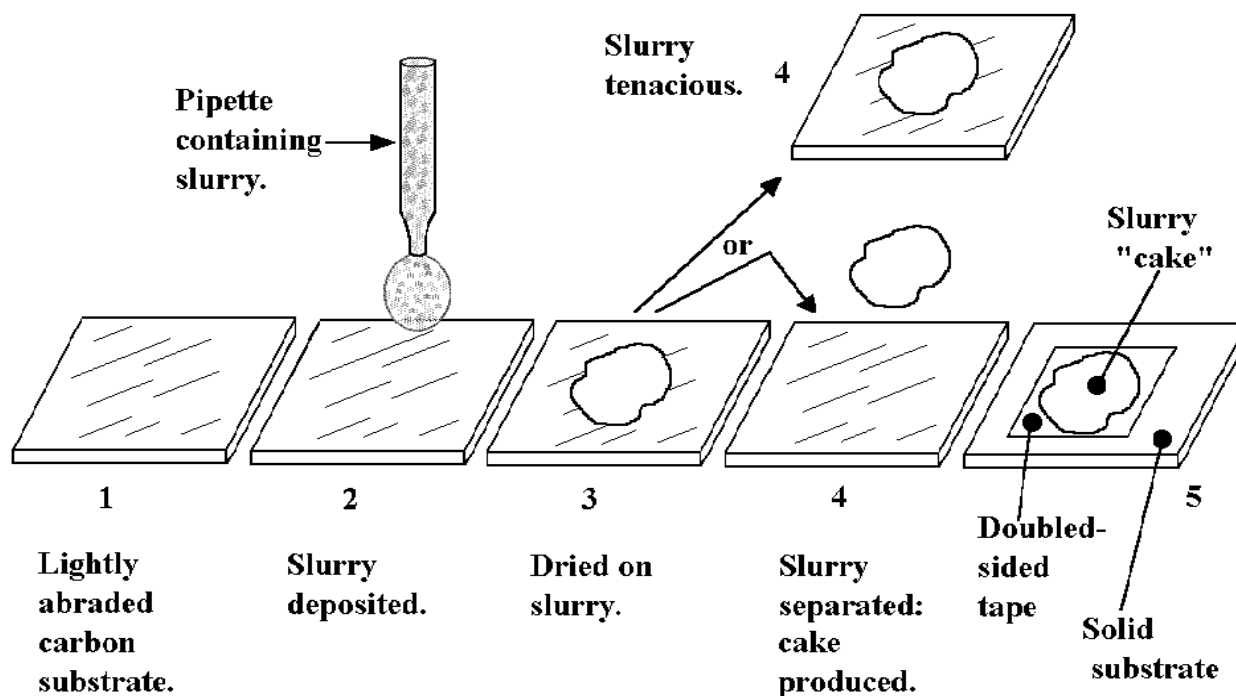
Figure 9-5.   *Diagram of Preparation from Slurries.*

Preparation and Mounting of Cross-Section Samples

Although TOF-SIMS is unique in its *surface* analytical capabilities, there are often times when the elemental sensitivity and organic detection capability of TOF-SIMS is invaluable in the analysis of sample cross-sections. In a sense, a cross-section analysis represents the deepest possible SIMS depth profile, providing information extending several hundred micrometers into a sample. When considering analysis of a cross-section, the most critical aspect is the cleanliness of the preparation. Standards of cleanliness that are acceptable for the preparation of conventional SEM cross-sections are *not* acceptable for TOF-SIMS. The following methods of preparation are recommended:

a. **Preparation of cross-sections**
   Mechanically polished cross-sections. It is possible to follow conventional metallographic polishing techniques, provided tap water is NOT used as a lubricant for the coarse carbide wheels. Use a supply of de-ionized or distilled water instead. When it is time to move on to fine diamond polishing, do not use oil-based diamond pastes, since the organic component of the paste will badly contaminate the sample. Instead, use diamond impregnated polymer films, and use a distilled water or distilled water-methanol mixture for lubrication. As a final, ultra-fine finish, use a γ-alumina (typically 0.05μm) and distilled water mix on a cloth wheel, or a 0.05μm diameter silica suspension such as "syton", also on a cloth wheel. In the latter case, use a final, liberal distilled water rinse to remove the potassium hydroxide solution that is a part of the syton suspension (to prevent aggregation of the silica particles). A final ultrasonic clean in methanol or distilled water is then essential to remove any fine polishing particles. This method works surprisingly well if strictly using either distilled water and high purity methanol. This method has even been used successfully to prepare flat polymer cross-sections.

b. **Microtomed and cryo-microtomed cross-section samples**
   Microtoming is only useful for relatively soft samples such as polymers. The blades and sample holder must be cleaned thoroughly beforehand, beginning with a distilled water wash to remove alkali metals, and also a hexane wash to remove the ubiquitous siloxanes. There are two approaches to preparing samples for TOF-SIMS by microtoming: one is to make a conventional thin slice from the sample; the other is to remove a thin slice (which is thrown away) to reveal a fresh face on the sample cross-section ("facing off" the sample).

The advantage of the first method is that a thin slice can be easy to mount by removing it carefully onto a suitable substrate, such as a small piece of adhesive tape (siloxane-free of course) mounted onto a rigid substrate. The disadvantage is the tendency of thin polymer films to curl up when cut in this manner. These latter problems are avoided by "facing off" a sample, and then the only remaining task is to mount the cross-section correctly.

Microtoming of sample produces both a slice and a fresh surface on the bulk solid.

Fresh face revealed by microtome.

Microtomed thin slice

Double-sided adhesive tape on substrate.

Sample mounted in vice holder.

Vice holder mounted inside standard sample holder.
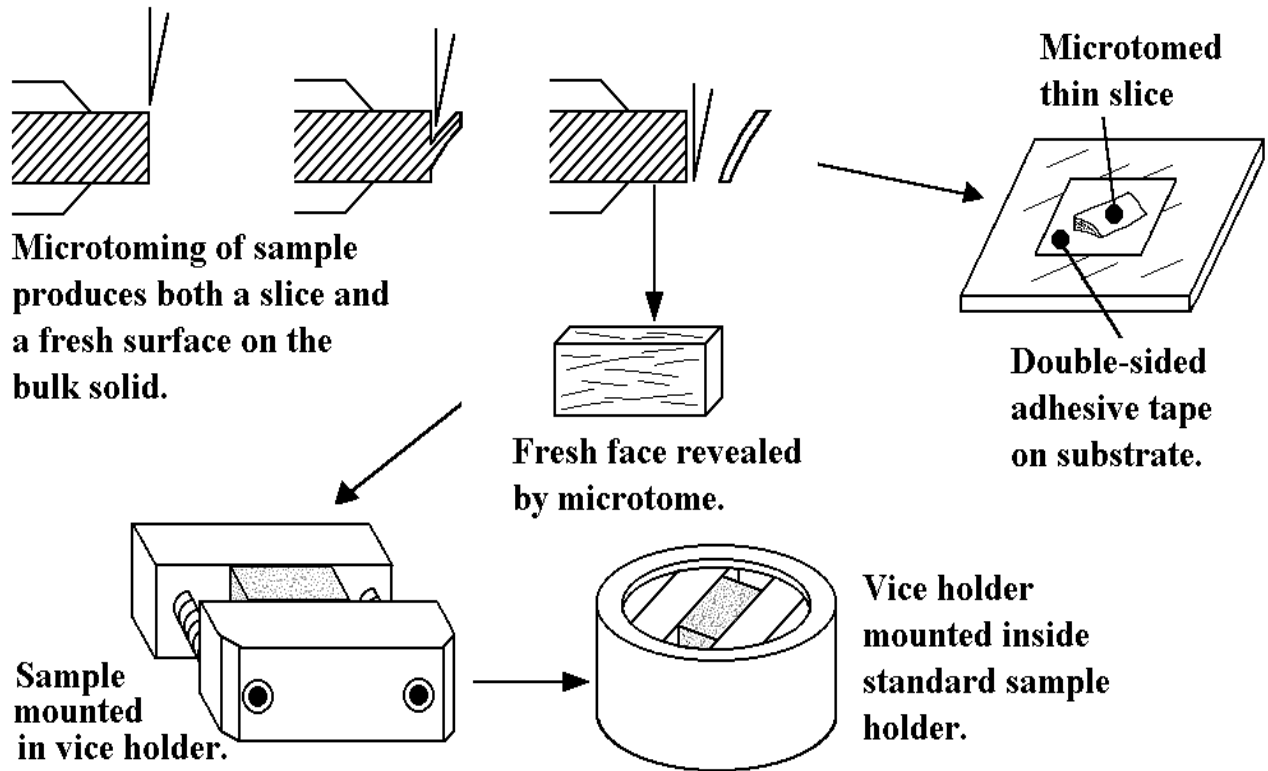
Figure 9-6.   *Diagram of Cross-Section Samples: Microtoming.*

Fracture cross-sections

Fracturing is by far the cleanest way to prepare cross-sections, even if the sample is cryo-fractured in liquid nitrogen. The problem with fracture cross-sections is the tendency of fractures to propagate randomly rather than where directed to go. For brittle samples (glass, silicon wafers), scribe the sample circumferentially beforehand with a diamond scribe to concentrate the stress and define the plane of fracture propagation. For polymer samples, it is still possible to define the fracture plane by making a suitably sized sample and cutting four "v" notches circumferentially. This will dramatically reduce the cross-sectional area in this plane (stress concentration) so that cryo-fracturing the sample greatly increases the chance of producing a flat faced cross-section. Preparing flat fracture cross sections from tough ceramic samples is extremely difficult. Normally, the best that can be hoped for is that a length of the cross section will be flat enough to mount and subsequently analyze.

Stick of polymer cut from bulk sample.

Circumferential "V" notch cut around stick.

Immersed into liquid nitrogen; gripped with two sets of pliers.

Removed from liquid nitrogen and fractured while still brittle; two samples produced.

Fractured sample mounted inside vice holder.

Vice holder mounted inside standard sample holder.
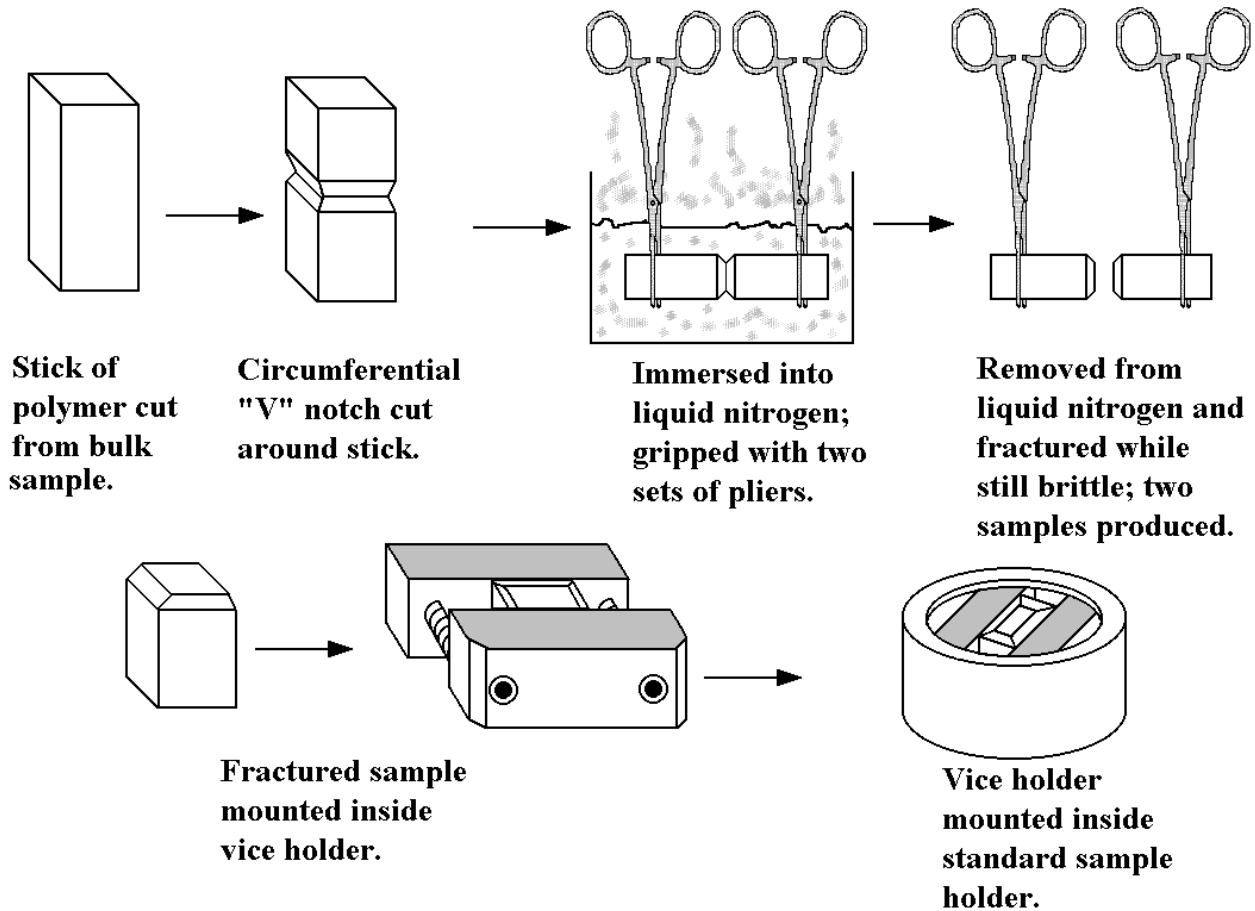
Figure 9-7.   *Diagram of Fracture Cross-Section.*

Mounting of cross-sectioned samples

A simple cross-section sample holder, which fits as an insert into the circular "standard" 25mm sample holders, is provided as part of the sample holder kit. This sample holder restricts the user to samples which have a width that will fit in between the two compression screws (~8mm), and has a maximum thickness of ~4mm.

When mounting the prepared cross-section, remember that an important aspect of mounting is to keep the cross-sectioned sample face at the same height (or very slightly low) as the vice holder. This is particularly important if the cross section is very thin, because the metal of the vice can be used to align the primary beam to the correct height before going to search for an area of interest on the cross-section. When the sample has been fixed in place in the vice holder, invert and lower the holder into the circular "standard" sample holder. If the sample is slightly higher that the vice surface, take care to raise the sample (quadrant) carrier off the mounting bench, otherwise, the clean and carefully prepared sample surface may accidentally be contaminated. Finally, try to align the length of the cross section with the horizontal axis of the system. If the sample is mounted slightly low, shadowing effects of the vice holder are avoided by doing so.

Analysis of Fiber Samples

*NOTE: A critical reminder to perform the analysis of SHORT,CONDUCTING FIBERS such as fine carbon fibers with **<u>great care</u>**. Any fibers which are fine and conducting can leave the sample surface and migrate into the system where they can create shorts between high voltage components and ground if not properly secured to the sample holder. To avoid this problem, use one of the methods outlined below for mounting THESE fibers.*

Analysis of single fibers along the axial direction

The problem of analysis of single fibers along the axial direction is not so much in the analysis but how to mount such samples without accidentally rolling the fiber along the mounting substrate, and thereby risking contamination of the fiber surface.

Method 1

Using a flat, conducting substrate such as a carbon planchet or a silicon wafer that will fit into a circular sample holder, cut two short strips of clean, double sided adhesive and set them on the substrate approximately 5mm apart in the center of the substrate. With clean tweezers or forceps, pick up a fiber and place one end onto one of the strips of adhesive. Using gentle tension to keep the fiber straight, guide the other end to the other adhesive strip and lay it down on the tape. If necessary, use two sets of forceps: one to grip and guide the fiber, the other to apply some force to press the fiber into the adhesive strips. Provided care is used, the upper part of the fiber will not roll on the substrate, and the surface will be unaffected by the mounting method. Multiple fibers can be mounted side by side using this method without too much difficulty.

1

Two strips of double-sided adhesive on a suitable substrate.

2

One end of fiber attached to one of the adhesive strips. Tweezers maintain tension.

3

Other end laid down under tension to keep fiber straight.
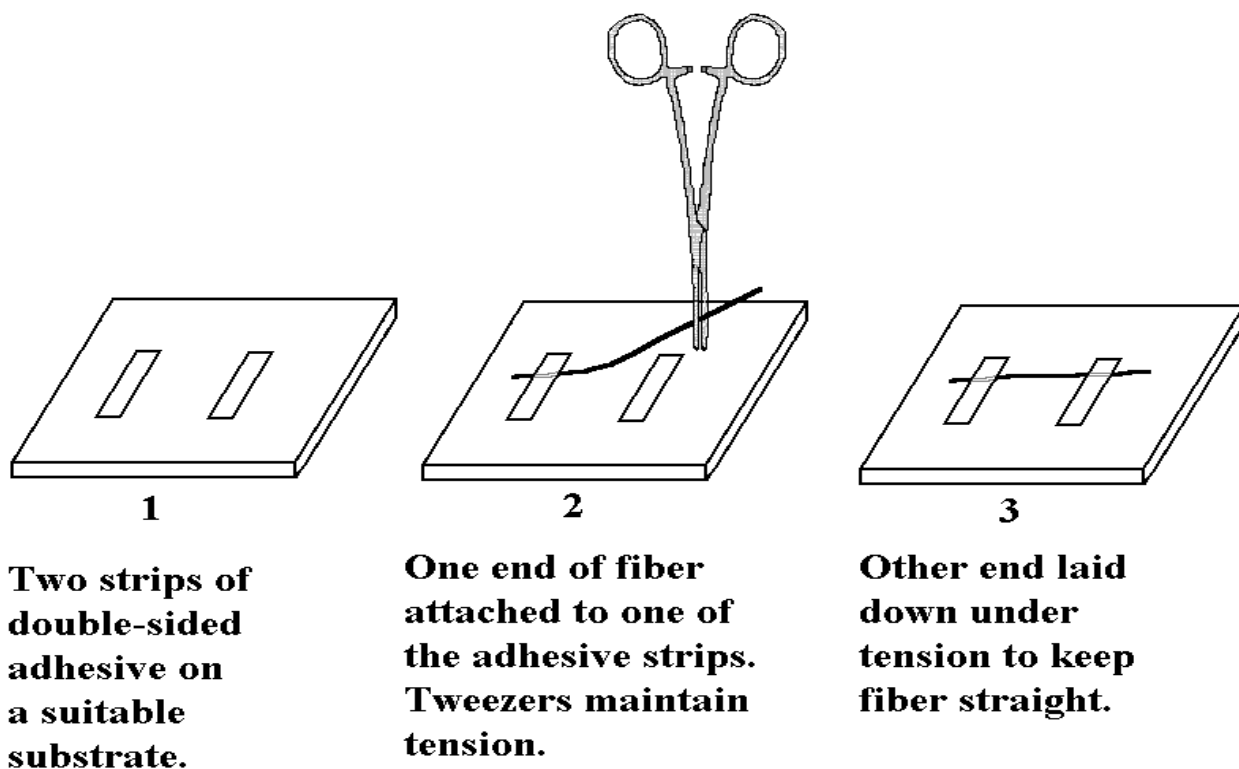
Figure 9-8.   Diagram of Analysis of Fibers: Axial Direction.

This method is also useful because it is easy to identify the mass spectrometer acceptance area on the substrate, and to steer the beam to that position. If the fibers to be analyzed are organic, a carbon substrate is preferable because carbon has a low SIMS yield, and the substrate SIMS signals do not swamp the signals coming from the fiber. For those unfamiliar with SIMS, it is difficult to interpret images where the yields from the fiber are low (and in keeping with the material's ionization yields) but the signals from the substrate are intense.

Method 2
If the fibers are required to not be in contact with the substrate in the area of analysis, try the following method. Lay a stainless steel grid inside a sample holder, and then place several fibers onto the grid. Take a second grid and lay it on top of the fibers and the first grid which produces a type of "grid-fiber-grid sandwich". Finally, take a backing plate and a sample clip and compress the assembly lightly to hold it in place within the holder. The fibers will be raised up from the backing plate by 125μm because the grids are 125μm thick. Although the two grids will obscure portions of the fibers, there will be relatively large areas of exposed fiber lengths for analysis. A variation of this method is to use an annular backing plate (not supplied as standard). A good quality 20mm diameter clean stainless steel (non-magnetic) washer works well. The stainless steel grids define the extraction field potential. The grids also allow for identification of the SIMS acceptance area on the sample holder and the fibers located over the center of the washer will have no substrate underneath.

1
Stainless steel mesh.

2
Fibers laid on top of the mesh.

3
Second mesh laid on top to trap the fibers.

4
Fibers and two meshes mounted inside standard sample holder.
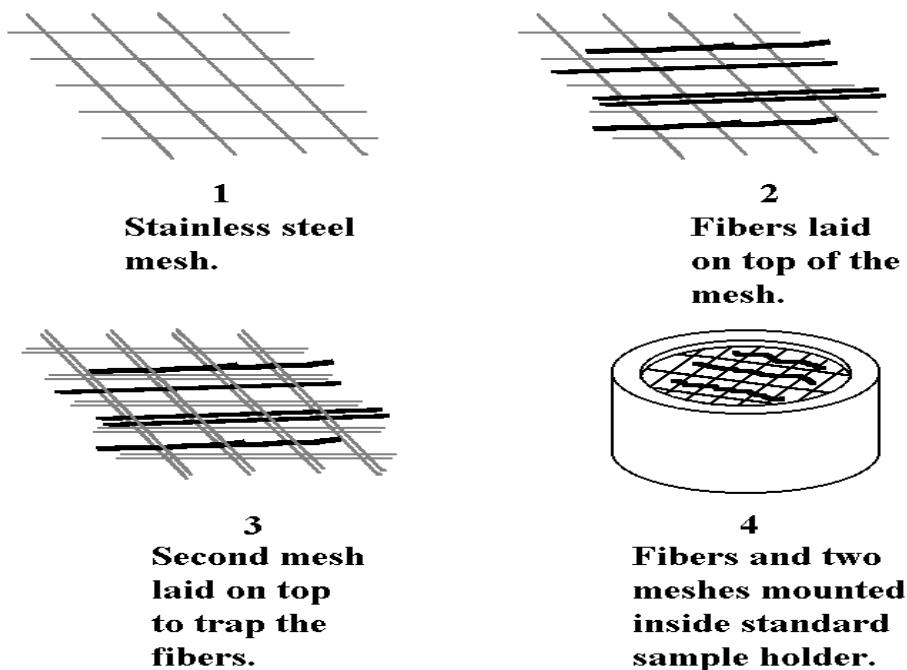
Figure 9-9.   *Diagram of Mounting without a Backing Plate or Adhesive Tape.*

Analysis of fiber mats

The mat to be analyzed should first be cut to size and then placed on a stainless steel grid within an inverted sample holder. Using a metal backing plate and sample clip, lightly compress the matte so that it is held firmly against the grid. It is strongly advised to use compressed air (or an inert gas at raised pressure) to remove any loose fibers in the mat prior to loading the sample holder into the load lock. If the mat to be analyzed has a very low density and is very loose, and *especially* if the fibers are conducting, the mat should be pressed into double-sided adhesive tape and then *all* loose fibers removed using a compressed air line.

1
**Sample cut
from fiber mat.**

2
**Stainless steel mesh
inside the inverted
sample holder.**

3
**Fiber mat placed
onto the mesh.**

4
**Aluminum backing
plate placed over
the fiber mat.**

5
**Sample clip
lightly compresses
fiber mat.**

6
**Compressed gas line
used to remove loose
fibers from the mat.**
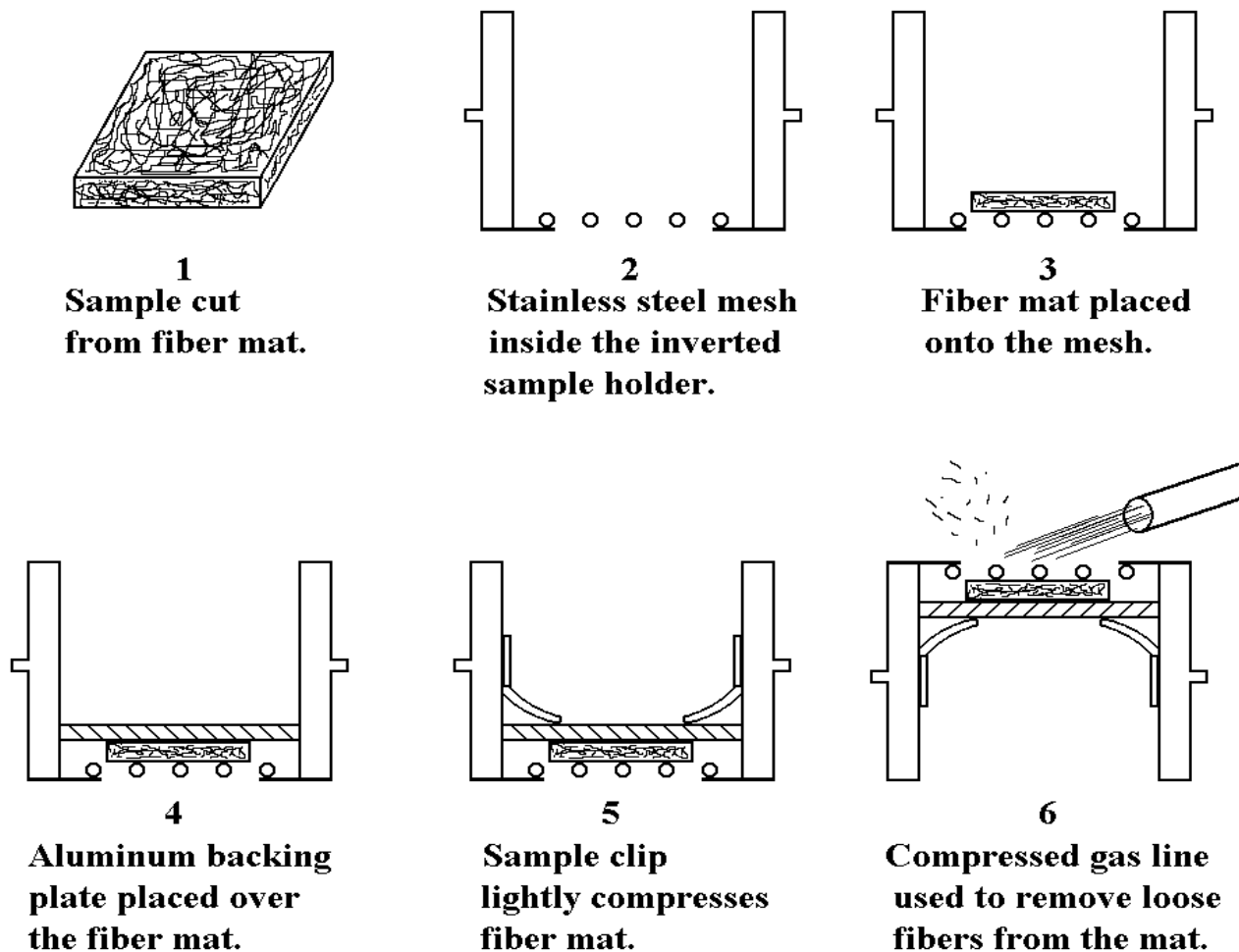
Figure 9-10.   Diagram of Mounting Fiber Mats.

Analysis of fiber cross-sections

One very convenient way to analyze fibers in cross-section is to embed the fibers in an epoxy resin which can then be microtomed to produce a flat face containing multiple fiber cross-sections. There are many commercially available molds which can be used for making such epoxy pieces, although the epoxy block will typically have to ground to fit in the cross-section holder before making the final "facing off" cut with the microtome.

1	2	3	4	5
Fibers stacked in small epoxy mold with low	**Epoxy solid removed from mold.**	**Sample cut so as to fit in vice**	**"Facing off" sample with microtome.**	**Sample mounted in vice**
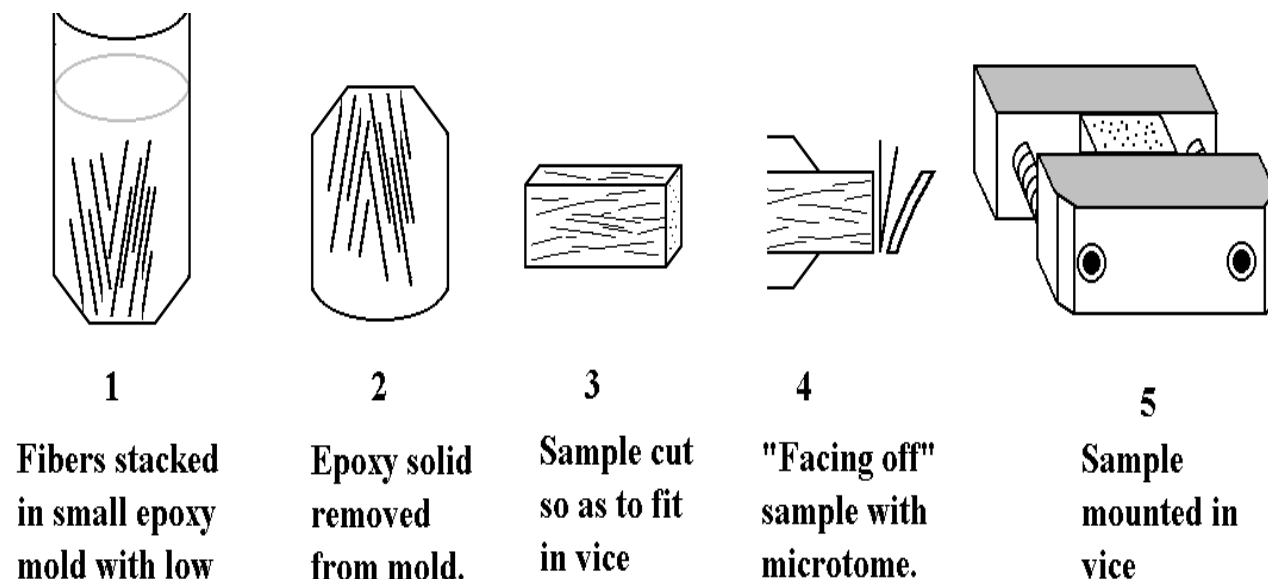
Figure 9-11.   *Diagram of Epoxy Mounting.*

If reluctant to embed the fibers for fear of contamination, the main problem the user faces is how to prepare a clean, flat cross-section. Microtoming single, freestanding fibers can be extremely difficult. A method which has been used successfully for preparing a cross-section from freestanding hairs for optical microscopy was outlined in "Microscopy of Mammalian Hair" by A. Syred, Microscopy and Analysis July 1991, p.23-25. This method was used in preparation of samples for TOF-SIMS analysis in "Molecular Ion Imaging of Human Hair Cross-sections", by D.F. Reich et al., Proc. 40th ASMS Conf. on Mass Spec. and Allied Topics, 1992, p. 558 and 559. The method is outlined below (refer to Figures 9-12a and 9-12b).

1. Make a small (~1mm diameter) hole in the center of a flat stainless steel plate approximately 50mm x 50mm and (up to) 2mm thick.

2. Prepare a bundle of 'sacrificial' fibers from a suitably soft material such as cotton, or a synthetic fiber such as polyester and insert a number of the fibers to be cross-sectioned into the *center* of the fiber bundle, making sure that there is a ring of sacrificial fibers surrounding them.

3. Draw the bundle through the hole in the metal plate, adding additional fibers to make sure that the bundle fits tightly through the plate. Mount the plate in a vice and arrange a collection dish under the vice.

4. Using a new, one-sided, razor blade, carefully sweep the blade at as low an angle as possible across the plate and through the bundle in the center of the plate. As the blade strikes the outer part of the bundle, more force must be applied to begin cutting the fibers. By the time the blade reaches the bundle center, it should be near a constant velocity, and should cut the center fibers smoothly. Keep both sides of the fiber cuts. This will double the chances of finding a suitable face for analysis.

5. Take the fibers to an optical microscope and inspect the cross-sections for the flattest, least deformed cuts. Note that most razor blades are supplied with a light coating of oil, and blades should be de-greased thoroughly before using it.

Stainless steel plate with 1 - 2 mm hole.

Bundle of 'sacrificial' fibers threaded through hole.

Fibers of interest inserted in center of sacrificial bundle.

Razor Blade

Fresh, degreased razor blade slides across surface of the plate.

Razor slicing into sacrificial bundle and fibers of interest.

Inspect the cut fibers under optical microscope: choose best cuts.
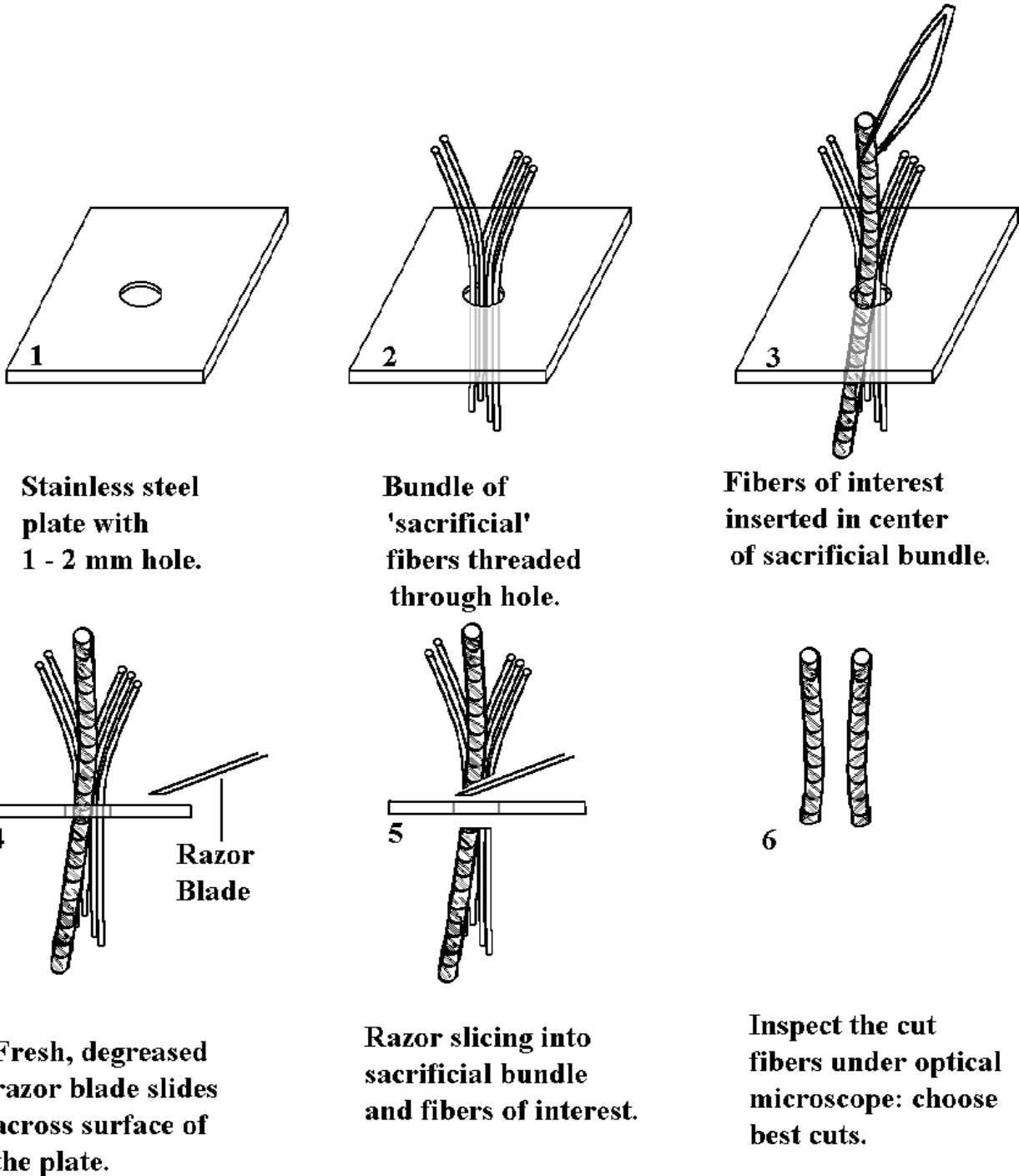
Figure 9-12a.   Diagram of Cross-Sectioning of Free-Standing Fibers.
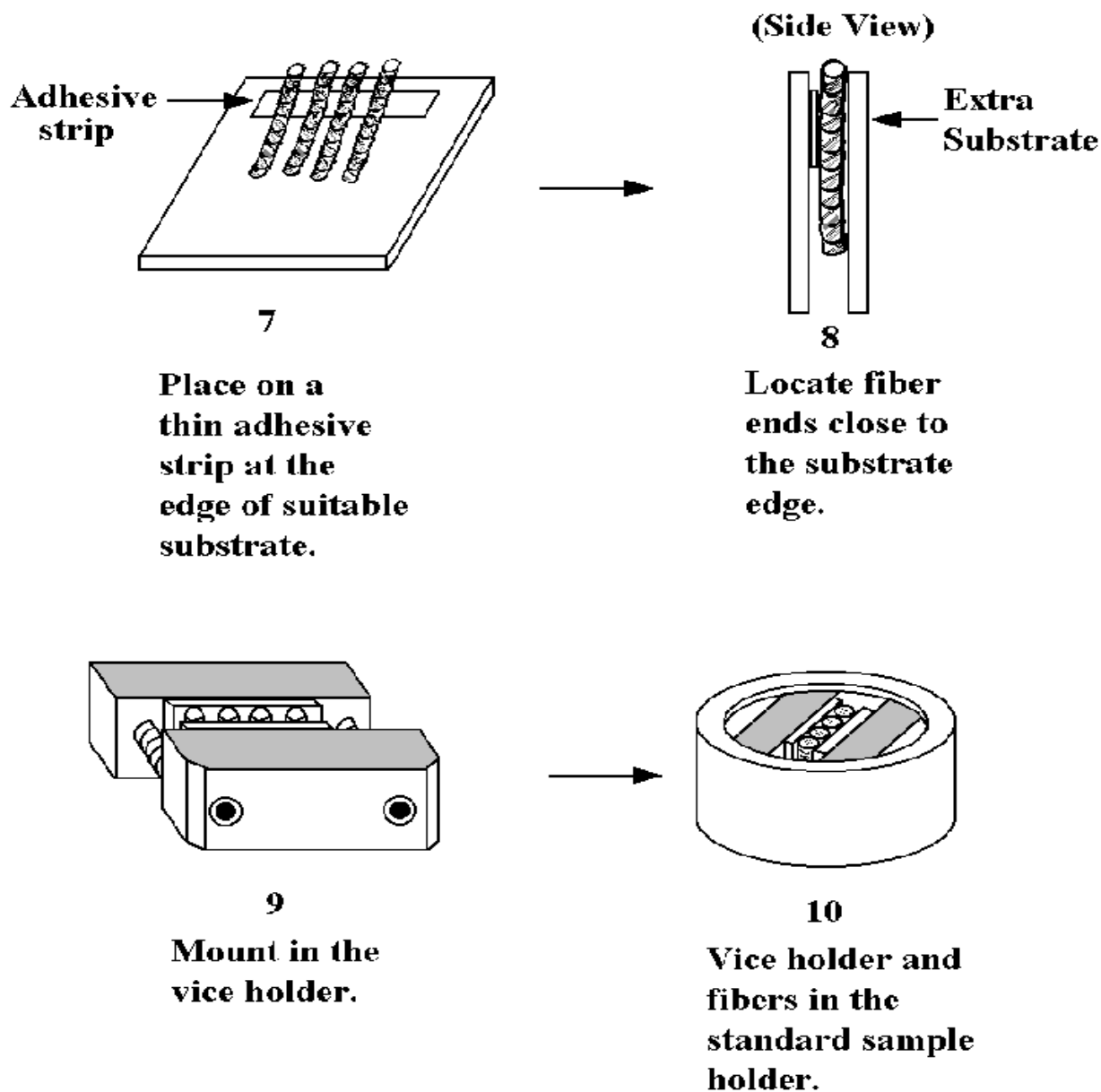
Figure 9-12b.   Diagram of Cross-Sectioning of Free-Standing Fibers.

To mount the fibers, try the following method.

1. Make a substrate ~5mm (x) 5mm (y) 1mm (z). Cut a strip of double-sided adhesive tape ~2mm wide and place it along one of the 5mm edges of the substrate. Carefully pick up the cross-sectioned fibers with tweezers and place a fiber onto the adhesive tape so that the cross-sectioned end is one or two millimeters away from the substrate edge. Mount several fibers in this manner.

2. Mount the assembly within the standard cross-section holder so that the fiber ends are approximately level, or very slightly below, the faces of the cross-section holder.

3. Once within the analysis chamber, use the faces of the cross-section holder to center the analyzing beam to the spectrometer axis, and then search for the fibers in the optical image. By placing the fiber ends at or near the same height as the cross-section holder, locating the fiber ends in the SIMS image should not be difficult. Tests have shown that elaborately-designed holders which grip a single fiber and present it at the correct sample height offered little improvement on the simple method outlined here.

Rough Samples

Introduction

Time-of-Flight SIMS systems require high extraction fields in order to provide subsequent high mass resolution performance. Variations in the sample to extraction distance result in varying total times-of-flight. Take the case of a recessed sample: a secondary ion ejected from a recessed sample spends more time in the (now greater) extraction region. It seems that such small changes should have a minimum effect on the total flight times until one considers that the initial velocities of the secondary ions are very low, and it is only at the end of the extraction (acceleration) space that they acquire their full velocity. Therefore, small changes in the extraction space distance have a disproportionate effect on the total flight times. These shifts are normally detected in practice by the need to adjust the calibration of the mass spectra slightly as one moves from sample to sample. In such cases, and following re-calibration, the mass resolution is preserved, because every peak in the mass spectrum is shifted by a predictable amount, and can be corrected by the simple act of re-calibration.

Recovery of Mass Resolution from Rough Surfaces

For rough samples however, the time shifts can occur within a sample and *within* a single field of view, and the resulting mass spectrum will have broader mass peaks. Since this broadening is due to the summation of mass signals from areas at different heights, it is possible to recover the mass resolution by taking data from specific areas within a field of view which are approximately at the same height. This can either be done during a live acquisition, or later in retrospective analysis mode.

Consider the following simple case. The user is analyzing an area which contains a step (refer to Figure 9-13 below). The left half of the field of view is higher than the right half. In such a case, there are only two discrete heights, and the total mass spectrum may show peaks which are doublets ("peak splitting"), depending upon the step height. Using an image in which the step boundary is observable, define a "region of interest" on one side of the step, and proceed to take data from that area which is at one height. The peaks in the mass spectrum will now not show peak splitting, and the mass resolution of the peaks will be as normal.
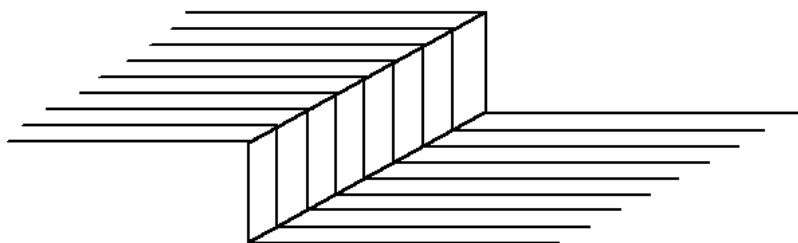
Sample Geometry: Stepped Sample with Two Discrete Heights.
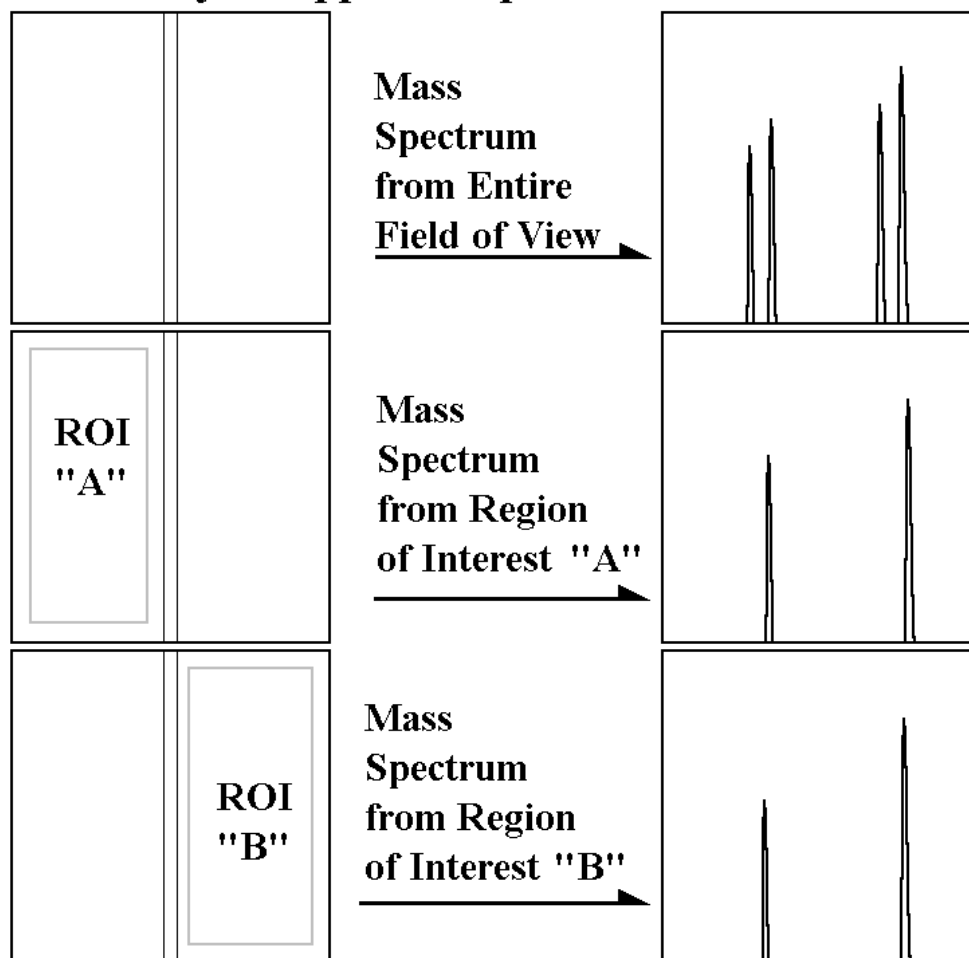
Figure 9-13.   *The Effect of Topography on Mass Spectra.*

Now consider a more complex example, the use of fiber mats (refer to Figure 9-14 below). The peaks in the mass spectrum are all broadened, but now, because the surface heights are changing continuously, there is no obvious peak splitting to warn the user that topographic effects are responsible for the broadening. Since there are no discrete heights, it is not obvious how to proceed, unlike the previous example of the stepped sample. The best that can be done is to acquire data from areas in the image which appear to have the least variations in height; for instance the tops of the fibers, as opposed to the sides. The narrower the band from which mass spectral data is acquired, the better the resulting mass resolution.
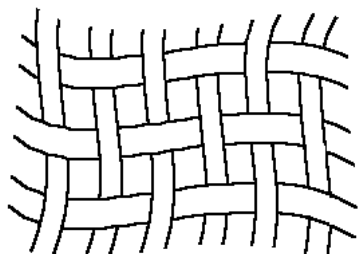
**Sample Geometry:
Woven Fiber Mat -
Continuously Variable Heights**
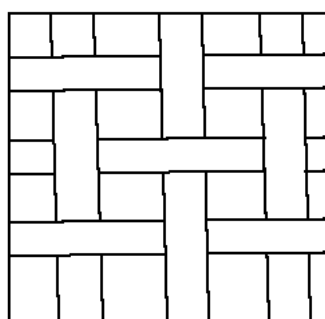
**Mass
Spectrum
from Entire
Field of View**
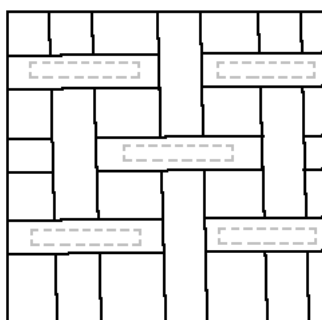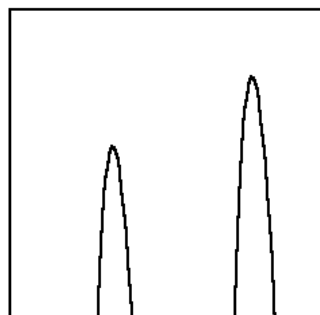
**Mass
Spectrum
from Multiple
Regions &
Interest at
same Height**
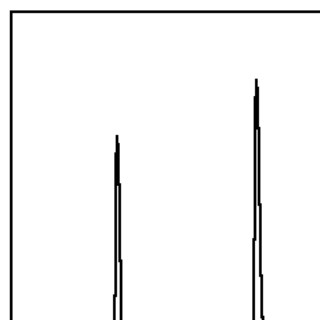
**Analytical
Image**

**Mass
Spectrum**

Figure 9-14.   *Sample Geometry for a Woven Fiber Mat with Continuously Variable Heights.*

Difficult Geometry Samples

Powders or fibers may be thought of as difficult geometry samples, but they have been discussed in previous sections, and are not the subject of this section. "Difficult geometry" refers to the types of samples where it is not at all obvious how to proceed. There are often two aspects to the problem: (1) preparing a suitable sized section of the sample, and (2) sample mounting. Once these two problems have been resolved, the analysis normally proceeds without significant problems.

Ball Bearing	**Protruding Section**	**Section of Tube**	**Highly Irregular Piece: Mineral Grains**
Shaft of Bolt	**Recessed Area**	**Cylinder End**	**Thick Slices; Small Diameter**
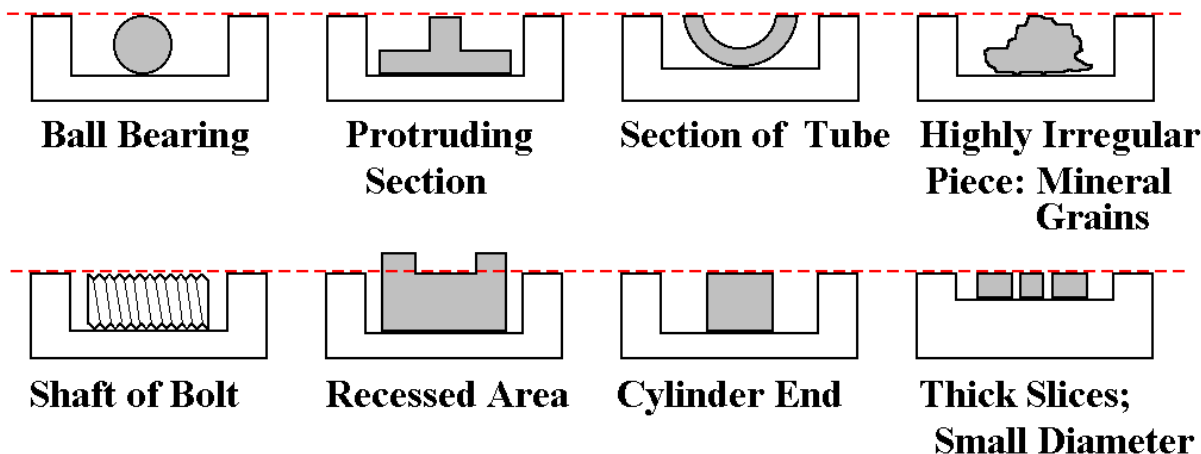
Figure 9-15.   *A Variety of Samples mounted in the Sample Holders for the TRIFT III.*

One of the most important aspects of dealing with difficult geometry samples is their mounting. A general approach is shown in Figure 9-15 above, where a range of difficult geometry samples are shown attached to recessed substrates, so that the area of interest is approximately at the optimum z position. A range of general substrates (refer to Figure 9-16 below), recessed at 1, 2, 3, 4, and 5mm permits the mounting of most samples. Samples can be attached either by double-sided adhesive, or a conducting paste such as silver or carbon paste. If double-sided tape is used, the sample will be insulated from the rest of the sample holder and may charge up, even if it is not insulating. Charge compensation will be required in such a case. If conducting pastes are used, sufficient time is required to allow the paste to dry. The substrates shown will permit a large range of difficult geometry samples to be mounted. In some cases, however, a recessed substrate may need to be fashioned that is specifically designed to accommodate your sample.
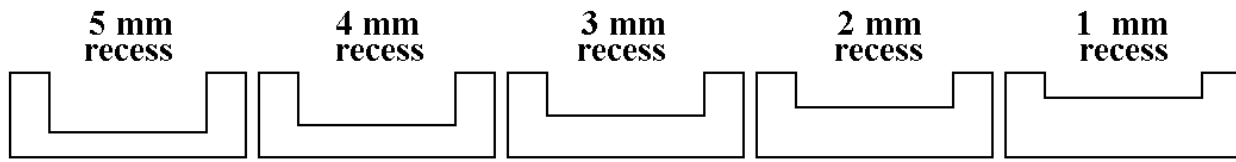
Figure 9-16.   *Cross Section View of the Various Sample Holders for the TRIFT III.*

Having shown the general mounting procedure, consider the requirement to prepare a sample which can be mounted as described above. For most cases, the preparation is simple; in other cases, more preparation is required. The following list of sample types is by no means exhaustive, but should provide a number of ideas as to how to analyze a sample with a specific geometry.
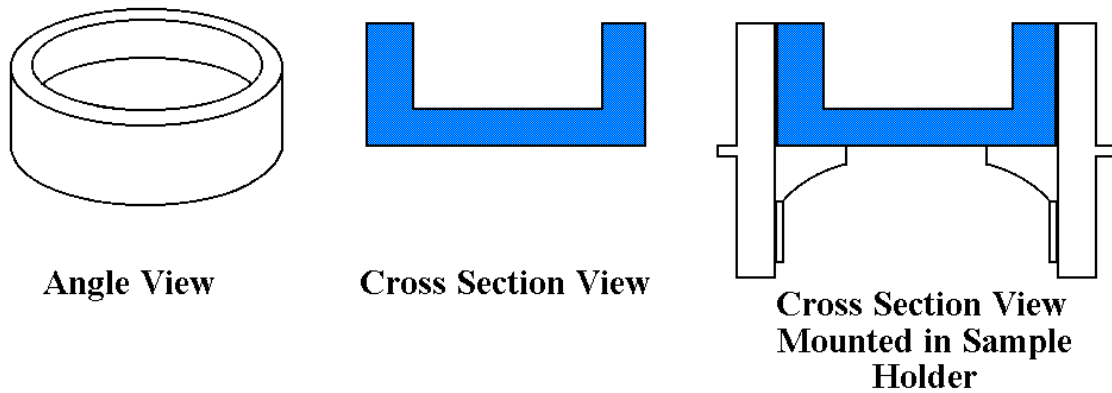
Figure 9-17.   *Cross Section View of the Various Sample Holders for the TRIFT III.*

1. **Small Diameter (£5mm), Thick (³ 1mm) Samples**
   When a sample has a small diameter or width, mounting the sample in a small
   hole sample holder may not be possible. Even if the diameter is large enough
   to back-mount inside a standard sample holder, perhaps too much of the
   precious surface will be obscured in doing so. In such cases, it is best to
   adopt the approach outlined above, using a recessed substrate.

2. **Non-Planar Geological Samples**
   Although many geological samples are prepared as cross-sections, sometimes
   analysis is required of mineral grain surfaces. Fine mineral grains may be
   treated as described in Section 9 on Powders, but as these samples grow
   larger, consider mounting them on recessed substrates instead. Use the
   attachment methods suggested in Section 9, but select a recessed substrate that
   will best accommodate the coarser grain sizes.

3. **Spheres**
   Most powders are spherical, but as the size increases much past a few hundred
   microns, it may be better to attach larger spheres to recessed substrates. An
   obvious extreme case would be ball bearings, where the diameter of the ball
   would dictate the choice of recessed substrate.

4. **Protruding Pieces on Otherwise Planar Samples**
   When the area to be analyzed is protruding from the rest of the sample by
   nearly a millimeter or more, it is better to recess the rest of the sample so that
   the area of analysis is close to the optimum z position. Choose a recessed
   substrate that best matches the height of the feature to be analyzed.

5. **Recessed Areas on Otherwise Planar Samples**
   When the area to be analyzed is recessed with respect to the surrounding
   sample, and the area is not large enough to prepare a sample from that area,
   use a recessed substrate and *raise* the surrounding sample higher than the
   optimum z to bring the recessed area to the correct height. The maximum
   height the rest of the sample should protrude is approximately 1.5mm,
   otherwise risk is involved in striking the extraction plate and damaging the
   sample as it is inserted into the sample chamber.

6. **Analysis on the inner surfaces of tubes**
   First, a section of the tube must be prepared. For tubes with large inner diameters, this is no problem, but more care must be taken as the tube's i.d. (inner diameter) becomes smaller. Aim for a final sample where the distance from the inner (concave) face to the highest part of the cut sample is less than 2mm (refer to Figure 9-18).

   If the tube is metallic, cut the section with a saw. On some samples, it may not matter if debris from the cutting process touches the area to be analyzed. If that is the case, cut with a fresh, degreased saw (no cutting fluids, of course), and try to cut *slowly*, to keep the sample from becoming hot due to the cutting process. Organic detection may be crucial, and the goal is to not remove the all-important organic in the act of preparation.

   After cutting has been completed, remove all debris from the sample by using a compressed air jet. If the cutting debris cannot touch the area of analysis, proceed as follows: cut a strip of fresh Al foil (or some other clean material that is siloxane-free) that is larger than the area of analysis. Slide the strip inside the tube, and use adhesive tape to stick all four of the sides of the protective strip down to the inside of the tube. Leave 2-3mm of sticky tape touching the tube. Cut through the tube so that the saw blade cuts through the sticky tape, leaving a small strip sealing the protective layer to the tube (refer to Figure 9-18).

   Once the section has been cut out from the tube, blow away the cutting debris as mentioned above. Remove the sticky tape and the protective layer, knowing that the surface to be analyzed has been protected during the preparation process. This procedure is equally useful when preparing debris-free sections from flat samples.

Preparation of Samples from Metallic Tube

(1) Plain Tube

(2) Protective Strip
Protective Strip Inserted

(3) Tape
Tape Fixes Protective Strip to Tube

(4)

(5)

(6)
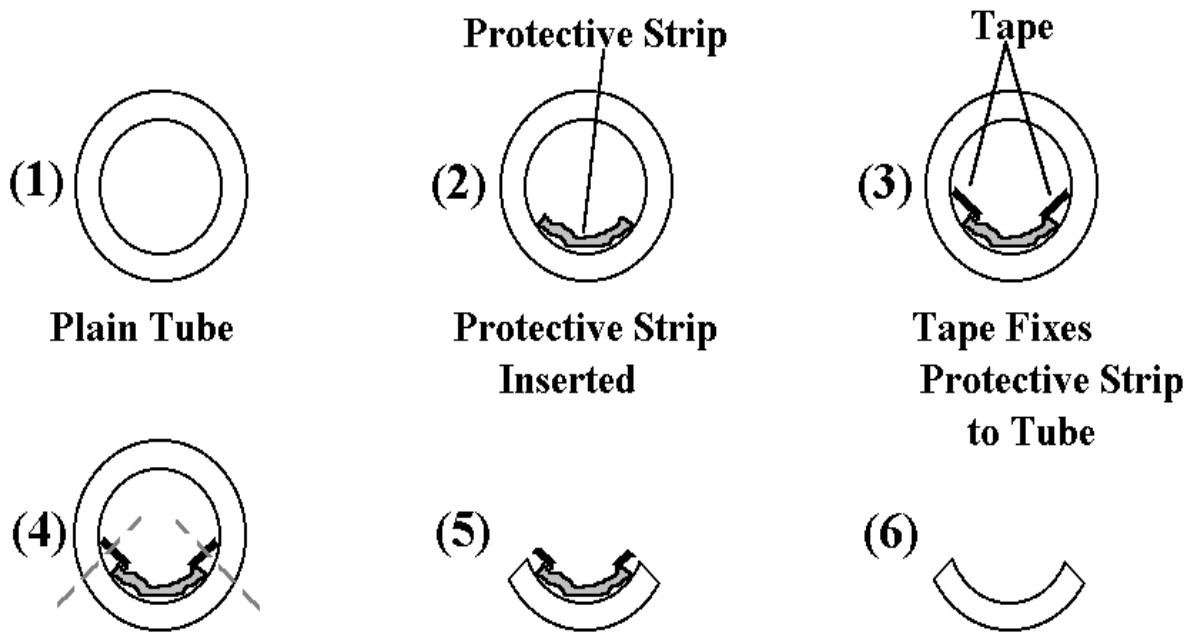
Figure 9-18.   *Illustrations of the Process of Sample Preparation from a Metallic Tube.*

If the tube to be analyzed is a glass, produce pieces for analysis by the inelegant (but effective) method of crushing the tube in a vice while insuring that a clean collection tray catches the fractured pieces. Select a suitable sized sample from among the pieces. Remember to wear protective glasses and to degrease the contact surfaces of the vice beforehand to reduce the chance of contaminating the outside of the tube with unwanted organic material.

If the tube to be analyzed is a tough ceramic, fracture pieces can still be prepared, but a grinding wheel may be necessary to grind cuts into the tube that will confine the fracture to a particular place and to reduce the amount of forces required to fracture the tube. This is a good way to ensure that a sample can be prepared from a glass sample in a specific area, for instance, when the sample must be taken from a very specific area, such as one containing a defect of interest. (Refer to Figure 9-19 below).

Preparation of Samples from
Glass or Ceramic Tube
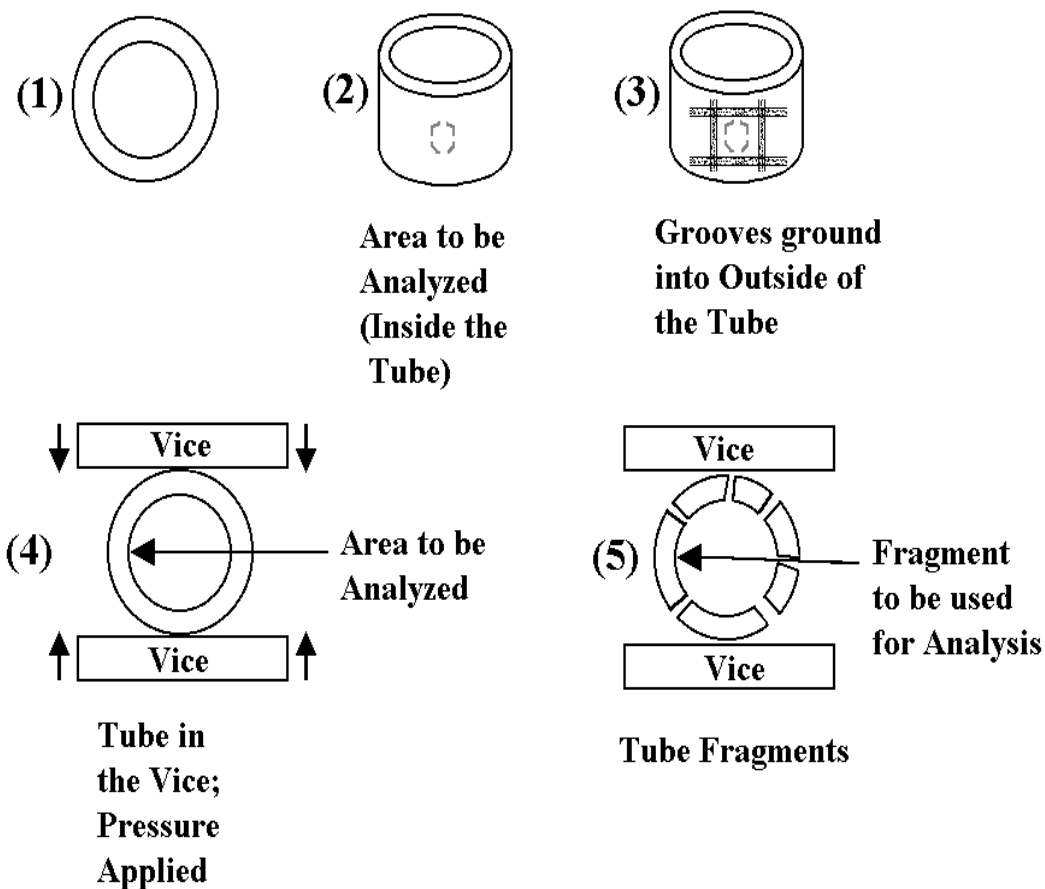
Figure 9-19.   Illustrations of the Process of Sample Preparation from a Metallic Tube.

Sample Mounting

For mounting the sample, refer to Figure 9-15. The objective is to locate the surface of interest at approximately the optimum height for analysis by a simple mounting method.

Be careful to make sure that the sample does not protrude more than ~1.5mm, otherwise the sample will strike the extraction plate as the sample is loaded into the sample chamber. If the sample is insulating, it does not matter if the tube protrudes a little further, as long as it does not exceed 2 mm. The extraction fields permeate the insulator, and so it has little effect on the field shape. If the tube is metallic, it is highly advisable to reduce the amount of the tube that is protruding to a minimum (<1mm), to reduce the distortion of the extraction field, and also to minimize the chance of an arc between the sample and the extraction plate.

Cold Stage Option [For 50mm (2-Inch) Introduction Systems]

Many liquids and solids, which are stable at room temperature and pressure, are rapidly pumped away under UHV conditions. In order to analyze such materials, it is necessary to introduce and maintain them at temperatures where their equilibrium vapor pressures are less than the vacuum inside the chamber. A large number of organic compounds, which would normally pump away at room temperature, may be stabilized at modest temperatures of -50°C and lower in vacuo. To this end, a cold stage option is available for the TRIFT system.

The cold stage option is comprised of the following components:

1. A customized, gold-coated copper sample holder and carrier;

2. A cold transfer vessel and dewar to allow the sample to be cooled using liquid nitrogen and then transferred into the load lock without being exposed to air;

3. A digital thermocouple probe to monitor the sample carrier temperature during initial cooling at atmospheric pressure;

4. A special sample stage which allows delivery of liquid nitrogen to the stage to cool the sample while it is inside the analysis chamber; and

5. A cold stage control unit which controls the rate of delivery of liquid nitrogen; monitors the sample temperature, and permits active heating of the stage to bring it back to room temperature.

Operation of the 50mm Cold Stage

1. Fill the stage cryo dewar (large stainless steel cylindrical) with liquid nitrogen and fit the dewar onto the bottom of the collar or "gooseneck" assembly which protrudes from the front of the stage. The plastic tube hanging from the gooseneck should go down into the liquid nitrogen (refer to Figure 9-20 below). The flask should make a gas-tight seal with the base of the gooseneck. Gently tighten the four screws together finger tight for even compression around the collar. Use an Allen wrench to tighten the screws.
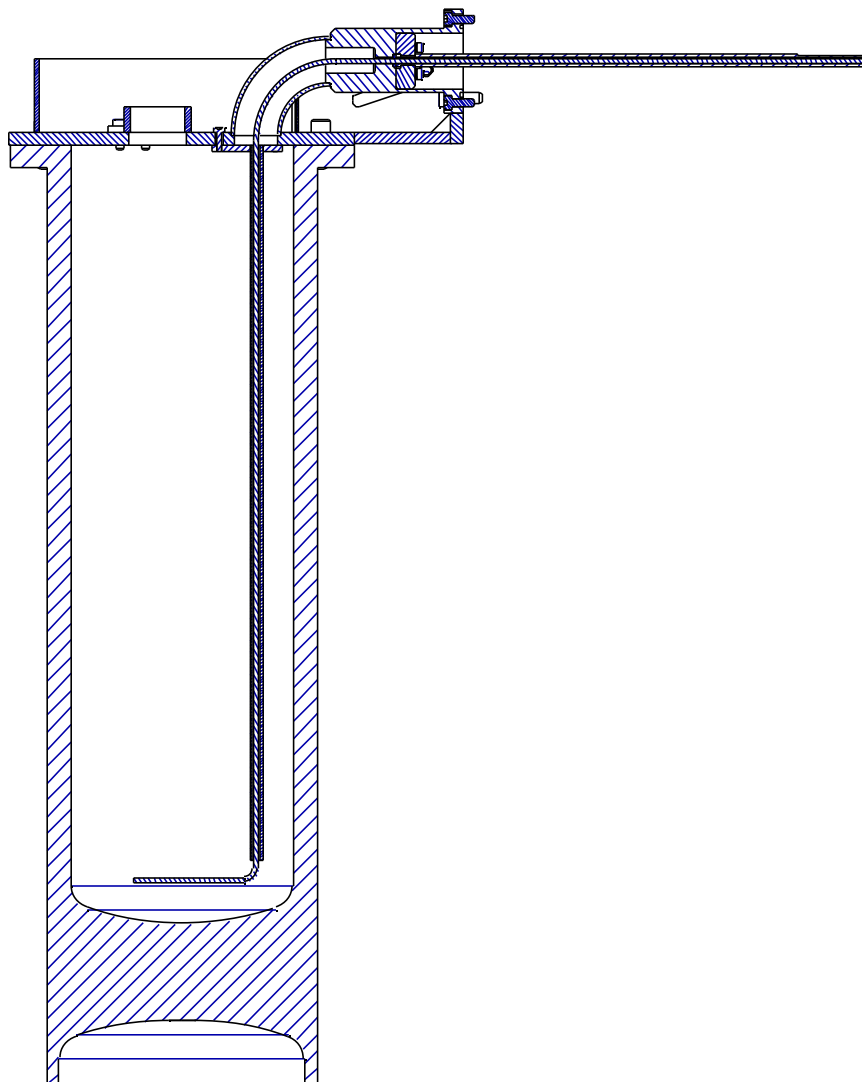
Figure 9-20.   *Cutaway Diagram of the Cold Stage Dewar and "Gooseneck" Assembly.*

2. Connect all relevant pipes and leads to the stage if they are not already in place. Thermocouple connectors will allow for monitoring the sample temperature in-situ, and the heater leads will allow for accelerated warming of the stage once analysis has been finished. The small band heater fitted to the gooseneck will prevent ice from forming around it. The nylon gas pipe from the cold stage controller should be fitted onto the 0.25 inch o.d. stainless tubing which emerges from the gooseneck.

3. The Dewar, the gooseneck, the pipes inside the gooseneck, and the controller all comprise a sealed system when assembled correctly. A pump inside the controller creates a partial vacuum inside the assembly. Liquid nitrogen (LN2) is drawn up from the Dewar and travels down a pipe inside the gooseneck to emerge on the atmosphere side of the stage, just behind where the sample is mounted. The copper-based sample carrier is in good thermal contact with the stage, and heat is conducted away from the sample through the solid wall of the vacuum chamber into the liquid nitrogen. The liquid nitrogen then passes back down towards the front of the stage, where it boils and is pumped away as a gas by the controller pump. Liquid nitrogen should not be seen appearing past the emergent stainless pipe on the gooseneck. The unit has been designed so that the nitrogen will be a gas at this point. Allow the stage to cool well before introducing a sample. This is particularly important if imaging at high magnification is desired. If the stage is not thermally stable, drift in the initial ion images will be observed.

Take precautions against frostbite and liquid nitrogen burns when using LN2. Insulated gloves and goggles should be worn when pouring LN2 from any insulated Dewar.

4. Mount the sample to be analyzed in the sample holder.

5. Next, place the L-shaped thermal mass plate so that the short part of the plate is resting on the top of the carrier, and the long part is in front of the sample carrier.

6. Insert the long sample carrier handle into the top of the transfer vessel through the rubber seal. Open the guillotine at the base of the sample transfer vessel, position the sample carrier and thermal mass plate so that the sample carrier handle can be screwed into the top of the sample carrier, holding the thermal mass plate loosely in place. Pull the sample carrier upwards with the carrier handle so that the guillotine may be closed.

7. Slide open the guillotine on the sample transfer vessel Dewar and fill it with liquid nitrogen. Slide the guillotine shut until ready for sample cooling.

8. Place the sample transfer vessel on top of the sample transfer vessel Dewar, and open both guillotines so that cool nitrogen gas can begin to purge the air from the transfer vessel. Allow approximately 5 minutes for full purging.

9. Lower the sample carrier into the liquid nitrogen using the carrier handle, but do not allow the sample itself to contact the liquid nitrogen. It is sufficient to immerse only the lower cut away section of the sample carrier into the liquid nitrogen. Heat will be conducted away from the main body of the carrier.

10. Raise the sample carrier periodically back up into the transfer vessel, and insert the probe from the digital thermocouple probe through the rubber seal at the back of the transfer vessel. Make contact between the probe and the sample carrier and monitor the temperature. When the sample carrier has reached approximately -70C, raise the carrier into the transfer vessel and slide the transfer vessel guillotine shut.

11. Remove the transfer vessel from the Dewar beneath, and place it on top of the sample introduction chamber.

12. Vent the introduction chamber. Slide both the intro chamber guillotine and the transfer vessel guillotine open. Carefully lower the sample carrier and thermal mass plate so that the thermal mass plate locates on the two pins provided inside the intro chamber. This takes some practice, and may appear to be very difficult at first. The best approach is to practice this maneuver with an empty carrier at room temperature. This will help when the time comes to work with a cold sample carrier.

13. When the thermal mass plate is in position, bring the horizontal sample introduction rod forward and engage the thread on the end of the sample intro rod with the screw thread on the side of the sample carrier. Rotate the sample intro rod at least three turns (but do not tighten fully). Unscrew the sample carrier handle from the sample carrier and withdraw upwards into (but not out of) the transfer vessel. Close the intro chamber guillotine and begin pumping the intro chamber.

14. Inside the intro chamber the sample carrier is not actively cooled, but the mass of the carrier plus the thermal mass plate have been designed to maintain the temperature of the sample for the initial pump down period. Also, since warming of the sample carrier occurs mainly by conduction, and since the carrier is only in contact with the intro rod, the rate of warming is also slowed by being in vacuo. Realistically however, do not allow the sample to remain in the introduction chamber for more than approximately 15 minutes. Once the introduction turbo pump has reached full speed, allow a further 3-5 minutes of pumping before opening the intro gate valve to observe if the pressure is acceptable for sample introduction.

15. Once the sample is inside the chamber, the sample and carrier temperature can be regulated by adjusting the amount of gas flow through the flow controller (determining the rate at which liquid nitrogen is drawn up to keep the sample cool).

16. If initial analysis indicates that the sample surface is covered with ice (characteristic clusters 18 mass units apart), wait a little while for the ice to sublime. Try to exclude this moist air during the next sample preparation.

17. When analysis is complete, remove the sample carrier as normal. Once the sample and carrier are exposed to lab air, they will inevitably ice up quickly. Use a hair dryer to warm the carrier and dry it if further analysis will be performed at low temperatures.

18. If the sample stage must be warmed up to room temperature as quickly as possible, a heater is provided inside the stage to allow for this. Turn the heater on at the controller.

Section 10: Routine System Evaluation \ Maintenance (Non-Service Issues)

Introduction

There are a few procedures which the system operator should follow as a matter of good laboratory practice if the TRIFT III system is to be maintained in optimum condition while possessing full knowledge of that instrument condition.

The following procedures are suggested.

System Bakeout

The following procedure describes baking the TRIFT system. A system bake is typically performed only when:
- necessary to remove contaminants within the vacuum chamber(s); or
- after the system has been up-to-air for servicing.

A system bakeout should result in returning the level within the chamber(s) to ultra-high vacuum. If the system's vacuum level is lower than 5×10^{-10} Torr, system bakeout is not necessary.

The TRIFT III system is essentially "bake-ready" which means there is minimal intervention to set up for a system bake. There are no requirements to remove cables or electronics of any type from the system prior to the bakeout sequence.

Bakeout Prerequisites

Several system conditions must be met prior to starting a bake:

- vacuum levels must be greater than 5×10^{-6} Torr (as displayed on the Vacuum Gauge Control labeled MAIN CHAMBER ION GAUGE);

- the introduction rod must be fully retracted; and

- the Introduction Gate Valve (V1) must be in the closed position.

Bakeout Preparations

1. Remove any wafer or samples from the vacuum chamber.

WARNING: Some samples when heated produce highly toxic vapors that could contaminate the interior of the chamber and the optics. Remove any wafer/sample from the vacuum chamber before baking.

2. Using a clean cloth and a cleaner such as isopropyl alcohol, remove any finger prints or oils on all stainless steel portions of the TRIFT system, especially the spectrometer and analytical chambers.

   ATTENTION: Any oils on surfaces subjected to bakeout temperatures can pit and stain the stainless steel. Oils will turn a brown color and will remain after bake.

3. Within the WinCadence software on the system computer, load a zero.ins file. This instrument file will load all zero values into critical voltage parameters to insure all portions of the system are in an idled state (no high voltage potential applied.)

4. Exit the WinCadence software.

5. On the Electronics Console, press the ELECTRONICS POWER button once to turn it OFF. In the OFF position, the ELECTRONICS POWER button is not illuminated and it will stay OFF throughout the bake.

6. On the vacuum console, insure that no cables are resting against any of the external heater bands including the main turbo heater.

7. On the System Vacuum Control located in the Electronics Console, the BAKE INTERLOCK key switch must be turned to the ON position.

   ATTENTION: Cover the end of the three (3) analytical chamber view ports with aluminum foil prior to starting the bakeout on the analytical chamber. The internal quartz heater lamps produce an intense light and also heat the view port glass.

8. Place the large yellow bakeout warning sign on the vacuum console or in a clearly visible location on the vacuum console or on the top of the electronics console.

WARNING: All metal surfaces exposed to bakeout temperatures will be very hot. To prevent injury, allow sufficient cooling time before attempting to touch any portion of the vacuum console.

Starting the Bake

When the hardware has been set up for bake, the bake is enabled and started.

REMINDER: Insure that the BAKE INTERLOCK key located on the front panel of the System Vacuum Control is turned to the ON position.

1. The Watcher software is used to start and control the bakeout. All bakeout entries should be made within the Watcher software from this point on.

2. The Bakeout Timer Control is located within the Watcher software on the system computer. Watcher must be in the SERVICE MODE in order to access the bakeout settings and to initiate the bake sequence.

3. Insure that the spectrometer gate valve (V5) is in the closed position. This is the gate valve attached to the transfer coupler located between the spectrometer chamber and the analytical chamber.

4. Within Watcher, insure that the CD (Contrast Diaphragm) is in the 2mm (Large) position.

5. Remove the Cesium source housing and close the cesium ampoule vavle on the cesium ion gun, if applicable. Open the cesium isolation valve on the ion gun, if applicable.

NOTE: For TRIFT systems with a 200mm (8-inch) sample stage, install the band heater on the stage bellows assembly prior to starting the bake.

6. With the Watcher user interface open, select the Options pull down. Select Service Mode. When prompted to Enter Password: type in phiphi. This will change the Watcher software from the User Mode to the Service Mode.

7. Under the "Valve Control and Overrides" section, Sample, HV: change from Enabled to Disabled.

8. Under the Vacuum Tasks section, select Bakeout.

9. Under the Comments section, select the Execute Tasks button. A Task Message window entitled Enter Time will appear which prompts the user to set the length of time for the bakeout in hour and minutes.

10. Enter the desired amount of time for the bakeout by setting the Watcher bakeout timer for number of Hours, Minutes, and Seconds. Then choose the OK button.

11. Watcher will prompt the user with another Task Message: "Install and connect the Bake warning sign".

12. Plug the two-pin connector and cable into the BAKEOUT INTERLOCK connection on the utility panel located on the back of the vacuum console. Place the large yellow bake warning sign in a conspicuous place - either on the vacuum console table top or on top of the electronics console.

    WARNING: Insure that the non-bakeable interlock cable does not touch the vacuum chambers or any other area being baked on the system.

Figure 10-1.   *Installation of the Bakeout Warning Sign is required prior to starting the bake.*

13. Once the warning sign is plugged in, click the OK button within Watcher. Watcher will then prompt the user with the following Task Message: "What bakeout zones?"

14. There are three options to select from: **Sample, Spectrometer, Both**. Select the button that represents the portion of the TRIFT system that requires baking.

    -If **Sample** is selected, the analytical chamber and heat clamps associated with that portion of the system will be baked,

    -If **Spectrometer** is selected, only the quartz heater lamps within the spectrometer chamber and the external heat clamp on the detector flange will be used for baking that portion of the system;

-If **Both** is selected, the entire vacuum system will be baked including the spectrometer chamber(internal quartz lamp), analytical chamber (internal quartz heater lamp), and all external heater clamps (ion guns, turbo pump, transfer coupler, and sample stage).

NOTE: A GFI circuit breaker fuse trip error message will occur if the Bake Interlock key switch is not set to the ON position prior to starting the bakeout.

NOTE: Bakeout power is interlocked to the MAIN CHAMBER ION GAUGE using set point #4, which is set to 5 x 10^{-6} Torr. During bake, power will be disabled if vacuum increases to more than 5 x 10^{-6} Torr and will not be re-enabled until the vacuum drops below 5 x 10^{-6} Torr.

15. When the appropriate option has been selected in Watcher, the bakeout process begins and power is applied to all of the corresponding heaters.

16. After the bakeout commences, a Timer display and an "Abort Task" button both appear on the right side of the Watcher user interface.

    ATTENTION: In the unlikely event that the bakeout must be shut down prematurely, the Abort Task button MUST be selected. This will remove power to all of the heaters immediately.

17. Watcher will control the bake process until the timer counts down to 0 Hours, 0 Minutes, 0 Seconds.

After the Bakeout

1. When the bakeout is done, Watcher displays the following Task Message: **"Please disconnect and remove the Bake Warning Sign"**.

    NOTE: There are no voltages involved during removal of the bakeout cable and bakeout sign. The cable and sign only provide an interlock to enable the bakeout to begin.

2. Disconnect the two-pin BAKE INTERLOCK connector from the utility panel on the back of the vacuum console. Return the warning sign and cable to a safe location for usage on the next bakeout.

3. Select the OK button on the Watcher prompt.

WARNING: All metal surfaces exposed to bakeout temperatures will be very hot. To prevent injury, allow sufficient cooling time before attempting to touch any portion of the system.

4. Turn Off the BAKE INTERLOCK key switch on the front panel of the System Vacuum Control (SVC).

5. Remove and discard the aluminum foil that was placed over the three view ports on the analytical chamber.

6. If baking a TRIFT system equipped with a 200mm (8-inch) stage, remove the heater band from the sample stage bellows when it has cooled sufficiently.

7. On the Emergency Off (EMO) panel, push the ELECTRONICS POWER button in to apply power for the main electronics console.

   *In the event that ELECTRONICS POWER was turned Off **before** exiting the WinCadence software at the start of the bakeout procedure, or if WinCadence was started without the ELECTRONICS POWER On, the system computer will need to be rebooted.*

8. Restart the WinCadence software.

9. The bakeout procedure is complete.

Statistical Process Control (SPC)

Purpose: to evaluate the overall performance of the instrument on a regular basis by means of a single analytical measurement which incorporates several important instrumental parameters. The measurement yields a "figure of merit" by which the general instrument condition may be judged. Significant changes in any one of these important instrumental aspects is detected by a figure of merit value outside the normal 2σ deviation of those values, and alerts the user to a possible change of instrument performance which may require further attention.

Nature of measurement: a determination of the yield of a specific secondary ion species from a standard sample with a standard surface condition, bombarded by a known number of primary ions of a given species and energy. The final figure of merit is expressed as counts per nanocoulomb.

Time required to conduct measurement: the measurement can be reduced to a short pre-cleaning of the sample surface (to eliminate varying surface adsorption effects), followed by a 1 or 2 minute acquisition. From a prior calculation of the number of nanocoulombs of primary beam delivered in the standard time for a given instrument state file, the peak integral of the relevant species can be multiplied by a pre-determined constant to generate the counts per nanocoulomb "figure of merit". After positioning the sample, the total time required to obtain the figure of merit is approximately 5 minutes.

Example of SPC Measurements

Figure of merit: counts Al^+ / nanocoulomb of Ga^+, data acquired from Reynolds (or equivalent) Al foil. This is the standard Al foil product available in most common stores. It is extremely clean and readily available, hence its applicability for this measurement.

Method:

1. Sample: mat surface of Al foil

2. Ga LMIG set at 15kV (could be 25kV), 600pA current (third primary aperture), using standard instrument file

3. LMIG emission current adjusted to 1-1.5µA

4. Positive ion spectroscopy mode selected

5. Buncher set to zero (0)

6. Charge compensation set to off

7. Mass range 0.5-1000 m/z (=10kHz rep.rate)

8. Set TDC time/channel to 0.138ns

9. No angular filtering, no energy filtering

10. Post acceleration 10kV (could be 0-10kV, but should not vary once decided)

11. SIMS acceptance area centered (manually or automatically)

12. Sputter clean sample for 40 seconds at raster size of 120µm x 120µm

13. Reduce raster to 20µm x 20µm

14. Begin a 2 minute (countdown) acquisition, and go to LMIG control page

15. Vary primary pulse width to give a secondary count rate of $\sim 5 \times 10^3$/sec

16. Stop and restart acquisition, allow it to perform full 2 minute acquisition

17. Measure number of time channels in hydrogen peak at full width half maximum (FWHM), using the mass spectral data you have just acquired.

    Figure of Merit = (Al peak integral) / (H channels) x (9.94 x 10-5 nC)

In the example described above, Al is measured using readily obtainable Al foil. The operator may decide to conduct the SPC measurement on a material that is more typical of the materials analyzed in their own laboratory, such as a clean silicon surface, or whatever. If a different material is chosen, remember to find a way to guarantee that the sample surface is always in the same, stable condition when making the measurement.

In the example above, a 40 second pre-sputter at a 120µm x 120µm raster removes all surface adsorbed hydrocarbons, and the measurement is made in the native Al_2O_3 oxide. This greatly reduces variations in the final value which would otherwise occur due to differing amounts of surface contamination on the sample from week to week and from month to month. The amount of pre-sputtering required on an inorganic sample will vary from substrate to substrate, and should be investigated by the user. In contrast, an SPC measurement carried out on an organic material should have no pre-sputter treatment (of course), and the operator must then have confidence that the surface condition remains the same from analysis to analysis.

Immersion Lens Conditioning

There are two situations which can contribute to continuous electron emission within the system:

1. **After a system vent.** If the immersion lens voltage (in positive SIMS) and the Cesium gun lens 3 are applied too early, i.e., <u>before</u> a short bake or an overnight pump (or both), it is possible to have voltage breakdown which can result in electron emission from these elements.

2. **After prolonged use of the system with volatile samples.** In practice, systems that are used principally to analyze 'wet' or 'dirty' samples, or simply organic materials with high vapor pressures, appear to develop carbonaceous layers on lens elements. This can eventually lead to formation of electron emission sites, especially where field gradients are highest, and where the element in question is at a negative potential. The elements most prone to this are the immersion lens and lens 3 in the Cesium ion gun.

In either case, unwanted electron emission can occur. <u>This situation is easily diagnosed from a positive ion mass spectrum of an insulator.</u> As noted in Section 8, charging of a sample in positive SIMS (in the absence of a neutralizing electron beam) usually occurs to produce a mass spectrum which displays a bias towards molecular ions just before complete loss of the secondary ion signal.

In the case of continuous electron emission, however, the following phenomenon occurs with insulating samples: electrons are emitted at high negative potential (immersion lens at -7,500V, Cs lens 3 at -12,000V) <u>while</u> the sample potential is at + potential. This can also occur if the electron pulse is not correctly nested within the sample bias 'on' time (refer to Section 8). The net result is that electrons impact the sample with extremely high energies (10,500 and 15,000 keV), and cause rapid negative charging and almost immediate loss of secondary ion signal.

The mass spectrum produced during this process is heavily biased towards atomic species, and typically the only species observed will be H+ and C+, with perhaps a few other metallic atomic species.

Thus, if a mass spectrum as described above is observed when acquiring data from an insulating sample in positive SIMS, first check the 'nesting' of the electron gun pulse in the sequencer menu as described in Section 8. If this is correct, then continuous electron emission <u>must</u> be occurring from one of the lens elements.

If continuous electron emission is suspected , refer to "Remedial Action for Persistent, Continuous Electron Emission" included later in this section.

Refer to the following two sections entitled "Regular Lens Conditioning" and "Conditioning Lens Elements After a System Vent" below. These sections outline the steps necessary to condition the high voltage lens elements in the cesium ion gun.

Regular Lens Conditioning as a Routine Practice

If the system is regularly used for analysis of volatile or 'wet' samples, it is good practice to condition the immersion lens and lens 3 of the Cs column on a routine basis, as outlined above. Doing so approximately once a month appears to prevent periodic electron emission from these lens elements that would otherwise occur. To do this, raise the immersion lens voltage to ~12kV over the course of several minutes (i.e., 0.5kV / minute), and allow the lens to 'soak' for a period of approximately one hour (the lunch break is very convenient for this!)

Note: If any sharp snaps are heard as the lens voltage is raised, do not continue, but 'soak' at a voltage just below the value at which the snapping was heard, and then proceed slowly to a higher voltage. The same procedure applies to the Cs gun lens 3 also.

Conditioning Lens Elements After a System Vent

Always allow sufficient pumpdown time after a system vent before applying system voltages. The recommended procedure after a system bake is as follows:

1. Once the system has been pumping for sufficient time to allow the ion pumps to be switched on, program a bake of at least one hour to desorb water vapor that may have entered the system.

2. If possible, leave the system pumping overnight with a bake going through to the early hours of the morning.

3. Once the system has cooled sufficiently and before the power supplies are switched back on, enter the TRIFT software program and load the 'zero' or 'null' voltage file in positive SIMS mode.

4. Exit the program, turn all the supplies on, and enter the program once again. Defaulting to the 'zero' or 'null' voltage file ensures that no voltages are applied anywhere within the system.

5. Raise the immersion lens voltage in units of 1000 volts slowly up to 5000 volts, and thereafter, in 500 volt increments to 12,000 volts. If possible, connect a picoammeter to the sample bias feedthrough and monitor the current to the sample holder. Leave the lens 'soaking' at each voltage setting for one minute before advancing on to the next setting.

6. If any sudden increases in negative current are measured on a one nA full scale range, immediately decrease the lens voltage. Slowly re-approach that voltage setting, allowing time for the measured current to fall to its baseline value. Note that as the lens voltage increases past 6 or 7kV, it is typical to measure some picoamps of 'leakage' current across the insulators. 'Leakage' current is characterized by a linear increase with applied potential, and is no cause for concern. The magnitude of the leakage current decreases with increasing pumpdown time.

7. Raise the immersion lens voltage to 12kV without measuring any sudden increases in negative current. Leave the lens 'soaking' at this voltage for ~30 minutes or so. Upon completion of this operation, the immersion lens is considered fully conditioned. Set the immersion lens back to zero (0).

Remedial Action for (Persistent, Continuous Electron Emission) an Electron Emitting Lens

If it has been established beyond all reasonable doubt that the immersion lens is emitting electrons continuously at its normal operating voltage, it will be necessary to vent the system and remove that lens element for cleaning and polishing. The vent, removal, cleaning, polishing and re-assembly process requires approximately six hours time.

Before considering undertaking this task, contact one of the PHI service engineers for more detailed advice and further information.

Logging Operating Values and Performance

Whether the system is used by a single operator or shared by many operators, all operators are encouraged to use a dedicated logbook in which the system, LMIG, Cs, and e-gun operating voltages and the performance values achieved are noted on a regular basis (i.e., daily). For example, the LMIG can be operated in a number of different ways, according to the following analytical needs:

1. At 25kV for the best lateral resolution in imaging SIMS.

2. At 15kV to obtain a 'bunched' probe for high mass resolution work.

3. At 5 or 10kV to minimize sputter mixing during depth profiling.

If a log is kept of all the relevant parameters for each of the various system conditions, including primary beam parameters, all users will find it is much faster to consult the logbook than it is to 'rediscover' the relevant settings each time. A regularly updated logbook is particularly helpful in allowing multiple users to develop a common 'language' when describing the system state.

Leaving/Inheriting the System in a 'Standard State'

There are two simple reasons why the system should be left in in a 'standard state' at the end of each day:

1. Ease with which a user can begin analysis on the next day.

2. Ease with which the system can be shared by multiple users.

The system settings which are considered as 'standard' will depend upon what form of TOF-SIMS analysis is performed most frequently, and varies from system site to system site. At the time of installation and training, PHI personnel will assist in setting up default files that are tailored to users specific requests. With subsequent experience, users may wish to change these accordingly.

If a user does not require the system to be in the 'standard state', then at least they know exactly what to change in order to achieve the desired state as quickly as possible. There is nothing more frustrating than struggling to obtain 'normal' secondary ion yields, only to find after an hour that the last user forgot to set the contrast diaphragm, etc. back to its 'standard state'. User protocol should dictate

that the user who changes the system to a 'non-standard state' be responsible for returning the system back to a specific 'standard state' prior to leaving the system for the next user.

A Typical 'Standard State'

Sample and sample holders

1. Sample carrier removed from the instrument.

2. Samples removed from holders. Holders in suitable condition for next user (cleaned if samples are likely to have left any organic material or residue on the sample holders).

3. Load lock placed under vacuum.

Overnight settings of the instrument

1. Load the zero.ins file which will set all instrument voltages to zero.

2. Spectrometer gate valve is closed.

Apertures, electronics and software

1. Exit the Cadence control program.

2. Remove or compress large quantities of data that would otherwise restrict the memory space available to the next user. Remove all 'raw' files to a suitable off-line storage media.

Daily log

1. Make a brief report in the logbook. Minimum comments include: operating mode (e.g. 25kV Ga imaging) and relevant lens settings.

2. If you have time, include notes on the SIMS performance in spectral and imaging modes, including examples of data.

Routine System Back-up on External Media

The reader is gently reminded of the value of routine back-up of both instrument-related files and data files onto an external medium.

General system back-up

You are encouraged to back-up your system routinely. The applications program that is installed on the computer has a variety of options to make this an easy process. It is simple, for instance, to select only certain subdirectories for back-up, such as C:\TFS (instrument-related files), C:\ANALYST1,2,3, etc. The TRIFT III computer is able to support a variety of devices through a SCSI interface, and many users have their own preferred medium, such as CD-RW drives, magneto-optical drives, "Zip" drives, network drives and so on.

Instrument file back-up

Always keep a copy of you master instrument files on a floppy disk (C:\TFS*.INS). The files are small enough that most instrument file libraries should fit onto a single 1.4Mb floppy disk. The PHI service engineer or scientist who visits you laboratory will also take away an updated copy of these files for storage in your customer file at PHI.

TOF-SIMS data back-up

It is possible to generate vast quantities of data on the TRIFT III, even in a single day, and you are encouraged to make plans from the outset as to how you plan to manage the data in your laboratory. The TRIFT III computer is able to support a variety of devices through a SCSI interface, and many users have their own preferred medium, such as CD-ROM writers, magneto-optical drives, "Zip" drives, network drives and so on. Whatever the medium of choice, you will find that it is important to remove data from the instrument computer on a regular basis, particularly if there are a number of analysts who work on the instrument.

In a multi-user laboratory, analysts should be encouraged to remove their data on a regular basis, so as to keep the instrument computer from becoming full. If your laboratory does not already have a standard procedure for data archiving, it is recommended that you appoint someone who will at least perform a system back-up onto DAT tape, including all data files, on a regular basis, say at least once a month. When there are multiple users, it is recommended that you follow the following simple rules:

- No data in the root directory C:\

- No data in the system directory C:\TFS (where instrument-related files are stored).

- Each analyst has their own, easily recognised, subdirectory.

- Appoint a person whose responsibility is to perform routine system back-ups and to gently remind colleagues (a) to (c) above.

Section 11:
Preventative Maintenance Schedule

Introduction

Preventative maintenance (PM) at regular intervals insures that the TRIFT III TOF-SIMS system will operate at maximum performance and minimizes the need for down time and unnecessary repairs. All PM procedures are performed by qualified PHI Customer Service personnel. This section of the Operator's Guide is meant to inform the system operator as what preparations may be required in anticipation of a PM visit.

By mail:

Physical Electronics, Inc.

PHI Customer Service, M/S G11

6509 Flying Cloud Drive

Eden Prairie, MN 55344

USA

By e-mail:

service@phi.com

By telephone or fax:

Region	Telephone	Fax
U.S.	1-800-922-4744	1-952-828-6325
Outside U.S.	1-952-828-5831	1-952-828-6325
Japan	81-46-785-6522	81-46-785-4411
Europe	49-89-96275-0	49-89-96275-50

WARNING: Performing any service tasks other than those described in this section without the assistance of PHI Customer Service could result in serious injury, could damage equipment, and may nullify applicable equipment warranties.

WARNING: Service procedures are for use by qualified and authorized personnel who have experience working with voltages greater than 50 volts. To avoid personal injury, do not perform any installation or service procedures unless qualified to do so.

Preventative Maintenance (PM)

The following maintenance should be performed on a routine basis, if necessary:

- Replace LMIG source

- Turbo and rough pump maintenance

- Replace electron gun filaments*

- Replace ion gauge filaments*

- Replace SEM electron multipliers*

- Replace detector channel plates*

- Cleaning electronic air filters

- MBIG maintenance

- Duoplasmatron maintenance including
  Replace cesium source ion gun ionizer*

- Magnetic intro transfer rod maintenance

*NOTE: *All parts listed above are considered consumables and are replaced at the system user's or operator's expense. It is the operator's responsibility to have these parts on hand prior to the start of the PM visit.*

NOTE: Major repairs to the system and maintenance that is not considered to be of a preventative nature will not be performed during a scheduled PM visit. Examples include (but not limited to) the following:
   - Ion pump rebuilds;
   - Performance checks on samples that are non-standard;
   - Major repairs of non-functional electronics or optics components.

PM Checklist

Preliminary Responsibilities

Review the PM parts checklist on a regular basis.

PM visits are scheduled in advance. The system user should contact Customer Care Center in Eden Prairie, Minnesota (USA) to schedule a PM visit by a customer service engineer.

The user is responsible for purchasing consumable parts and should have the parts on hand prior to the start of the PM visit.

Maintenance

Requested parts to be replaced or repaired inside the vacuum chamber should be scheduled early in the PM visit. This will make best use of pump down and bake time to take care of any other issues.

Electrical calibrations and mechanical alignments should be performed as necessary to optimize the system performance.

The results should be documented and a course of action will be recommended if performance concerns are identified.

<u>*NOTE*</u>*: Unless stated otherwise in writing, PHI **does not** guarantee the system will meet or exceed original performance specifications.*

The following items should also be addressed during a preventative maintenance visit:

- Changing oil in all rough pumps;

- Lubrication of all wicks in all turbo pumps;

- Cleaning all electronics console and electronics chassis filters; and

- Alignment of the introduction probe to the stage.

PM Schedule

A complete PM schedule is listed in Table 11-1 below for the TRIFT III system.
The intervals listed are marked from the time of the initial installation.

Table 11-1.   *TRIFT III System Preventative Maintenance Schedule (based on 8-hour day operation).*

Preventive Maintenance Schedule:	6 Months	12 months	18 months	24 months
Replace Turbo Wicks	X	X	X	X
Replace Rough Pump oil	X	X	X	X
Bakeout Vacuum Chamber	X	X	X	X
Clean Air filters	X	X	X	X
System performance:				
Mass resolution	X	X	X	X
Imaging	X	X	X	X
Blanker calibrations	X	X	X	X
Ion Gun currents				
O2: All apertures	X	X	X	X
LMIG: All apertures	X	X	X	X
Cs: All apertures	X	X	X	X
Charge compensation performance	X	X	X	X
Check Alignment of sample viewing camera	X	X	X	X
Check Operation of automated Stage	X	X	X	X
Recheck and save Instrument files	X	X	X	X
Replace Battery in SVC				X
Replace LMIG In emitter		X		
Clean/rebuild Duoplasmatron O2 source		X		
Replace LMIG Ga emitter			X	
Replace Ion Detector Channel Plates and Phosphor screen				X
Replace Cs Ionizer				X
Replace Cs Column Condenser #1 assembly				X
Replace electron gun filament				X

TRIFT PM Parts Checklist

For an upcoming preventative maintenance visit, use this list to determine which items will be required for your instrument. This will allow us to provide better service to you by insuring that all requirements are fulfilled during the PM visit.

For an upcoming preventative maintenance visit, use this list to determine which items will be required for your instrument. This will allow us to provide better service to you by insuring that all requirements are fulfilled during the PM visit.

- These parts are considered consumables and are replaced at the customer's expense. It is the customer's responsibility to have the parts on hand prior to the start of the PM visit.

Table 11-2.   *TRIFT III Preventative Maintenance Parts List.*

Part Number:	06-656 MICROBEAM
634742	APERTURE DIFFERENTIAL PUMPING, THICK
611881	APERTURE DIFFERENTIAL PUMPING, THIN/MOLY
611880	SPACER DIFFERENTIAL PUMPING
C76054	WASHER CURVED
641778	APERTURE-EXTRACTION TRAP, .100
	CONDENSER APERTURE STACKUP CONSISTS OF
	THE FOLLOWING ARE THREE (3) SEPARATE APERTURES
627540	APERTURE CONDENSER
627549	APERTURE SHIELD
627542	APERTURE CONDENSER BLANKING
1000128	RING-RTNG, INT, .500
621183	CONDENSER CERAMICS (2 PER SECTION)
548724	TEFLON TUBING (UNIT OF MEASURE IS INCHES)
609636	SAPPHIRE BALLS
B77027	SHOULDER CERAMIC
577111	O-RING .424 ID ISOLATION VALVE
622213	APERTURE PLATE (obj.)
508000	BEAD-CER, INSUL,.090X.040X1/8
543802	LUG-RING,26-22GA, #2,UN,.150W
603119	CONTACT-.062 SKT LW TEMP VAV
627001	WIRE-MAGNET, 24GA,ML,QUAD BUILD

FILAMENTS

627285	EMITTER-LMI, TOF GA-69, 1500 HR
642819	EMITTER-LMIG, InPUR MONO
633380	E-GUN FILAMENTS
131-00010	FIL-GAUGE,ION ELECT DEGASS,G.P. 274025

LMIG COLUMN

FEI 16387	EXTRACTOR CAP
FEI 18181	SUPPRESSOR
FEI 20037	STEM APERTURE ASSEMBLY; "20 nA COLUMN"
FEI 168484	MVA ASSEMBLY

Cs COLUMN

612458	FRIT ASSEMBLY
639387	CESIUM AMPULE- 1 GRAM

O2 SOURCE

608892	ASSY-GAS CYLINDER, OXYGEN
613265	KIT-REPAIR,INDIRECT GAS, 06-660A

ION DETECTOR

570-00005	MCP, MATCHED SET, 25MM, HOT
636047	PHOSPHOR SCREEN

SPECTROMETER CHAMBER

638298	GATE VALVE (HVA 11220-0150X), SPECTROMETER
639695	KIT-UPDATE, POSITION MANIPULATOR

SAMPLE CHAMBER

123-00002	MULTIPLIER-ELECTRON, SEM DETECTOR

2" INTRO / STAGE SECTION

248-00001	GATE VALVE, 3 " I.D., MDC
639388	ADPTR-SCREW, LOADLOCK, TRIFT, MAG INTRO
02-03145	SHAFT, SAMPLE INTRO, 2" SLIDING SEAL INTRO
352-00011	O-RING, ELASTOMER, 0.239ID; INTRO ROD
02-03140	BSHG-WILSON SEAL; INTRO ROD
352-00013	O-RING-ELASTOMER, 2.975ID; INTRO COVER

8" STAGE SAMPLE CARRIERS

02-05555	ASSY-SAMPLE CARRIER, SM HLDR, 8"
02-05647	ASSY-SAMPLE HOLDER, 8", TFS

2" STAGE SAMPLE CARRIERS

02-03465	SAMPLE CARRIER, 4-POSITION, 2" STAGE
02-03464	ASSY- SAMPLE CARRIER, LARGE SAMPLES, 3 MASKS

UNIVERSAL SAMPLE HOLDERS / ACCESSORIES

02-03466-03	HOLDER- SAMPLE, .500" ID
02-03467	HOLDER- MULTI SAMPLE
636853	MASK-SMALL HLDR,10MM, TRIFT II
636854	MASK-SMALL HLDR, 5MM, TRIFT II
642207	MASK-SMALL HLDR, 2MM, 4X, TRIFT II
636856	MOUNT-RECESSED BKLD, 1MM, TRIFT II
636857	MOUNT-RECESSED BKLD, 2MM, TRIFT 2
636858	MOUNT-RECESSED BKLD, 3MM, TRIFT 2
636859	MOUNT-RECESSED BKLD, 4MM, TRIFT 2
636860	MOUNT-RECESSED BKLD, 5MM, TRIFT 2
642259	SAMPLE-FARADAY CUP, BOROSILICATE
642260	PLATE-BACKING,FARADAY CUP
02-05695	WAFER MESH, 3/4" DIA, CHARGE COMP GRID
640756	PLATE-BACKING,SAMPLE, .82 OD
640755	ASSY-CLAMP, SAMPLE CROSS SECTION VICE
246-00008	SPRG-CPRSN, CNCL, .225 - .11OD, .01
00-05133	SCR-BUTTON, 4-40X .250"
896-00002	SAMPLE, AL/CU GRID (CAMECA)
02-04677	SPRING- SAMPLE HOLDER, FORMED CU
638031	SAMPLE HOLDER SHIM KIT

TURBO PUMP ACCESSORIES

897-00001	BALZERS, 60 L/S TURBO WICKS
897-00002	BALZERS, 240 L/S TURBO WICKS
619393	OIL, PUMP, ALCATEL, 1 LTR.

MISC ACCESSORIES

A79106	GAUGE TUBE-GRAN PHILLIP 275071, CONVECTRON
638274	LAMP T1.75 28V .04 A (EMO BOX)
131-00002	LAMP, HALOGEN, 250W, 120V
131-00003	LAMP, HALOGEN, 150W, 120V
622301	LAMP LIGHT SOURCE
639187	BATTERY, 3 V LITHIUM SIZE C, FOR SVC
637045	PHOSPHOR SCREEN
636047	TRIFT III STAGE BELLOWS 50 MM

GASKETS

2680750	KIT-GASKET, 1.33 OD FLG, (10)
633382	GASKET-CU, 2.125 FLG, CLN, LMIG MVA
2681500	KIT-GASKET, 2.75 OD FLG, (10)
2682000	KIT-GASKET, 3.37 OD FLG, (10)
2682500	KIT-GASKET, 4.50 OD FLG, (10)
1001887	KIT-GASKET, 4.62 OD FLG, (10), LMIG SOURCE
2684000	KIT-GASKET, 6.00 OD FLG, (10)
633381	GSKT-SPECIAL,TRIFT,6.0 FLG CLN, LMIG THICK
2688000	KIT-GASKET, 8.00 OD FLG, (10)
633357	GASKET-CU, 14.00 FLG, CLN, 8" STAGE
352-00014	GASKET-W/S, 18.00 FLG, SPECTROMETER LID
632197	GASKET-VATSEAL, 46X236, SEAT SEAL, 8" STAGE
632198	GASKET-VATSEAL, 46X236, BONNET SEAL, 8" STAGE

Routine Maintenance

TRIFT parts or components that require maintenance on a routine basis are described below. Manuals for components from original equipment manufacturers (OEM) other than Physical Electronics are supplied with the system for reference.

NOTE: Routine monitoring of certain system performance parameters and maintenance events, as described later in this section, is recommended to reveal trends in the system performance and help uncover potential or apparent problems.

Cesium Ion Gun Source

Service or maintenance of the cesium ion gun source should be nominal, however, if the source becomes unstable and does not operate properly, it may indicate that the reservoir has run out of cesium material or the cesium frit has become plugged.

Cesium Ion Gun Column

Regular service or maintenance of the cesium ion gun column should also be minimal, however, if excessive steering values within the WinCadence software are required to operate the column, excessive cesium material may have built up on the column's steering plates.

Console Panels

Service or maintenance of the system often involves opening or removing one or more panels from the consoles. Most of the panels on the electronics console are hinged for opening and are held closed with captive screws. All of the panels on the vacuum console are held closed with captive screws.

Panels that are not hinged are replaced by (1) lowing the openings on the inside bottom of the panel into the console's receiving brackets, (2) pushing the top of the panel in until it snaps into position, and (3) tightening the captive panel screws to hold the panel in place.

Cooling Filters

The TRIFT TOF-SIMS system only has one 10 inch x 24 inch filter located inside the right-hand door of the back of the electronics console. No tools are required.

To remove the filter, open the door and lift up on the filter frame, which is attached to the console door with L-shaped tabs. Lift the filter out of the frame. The filter should be removed as needed and cleaned with a vacuum cleaner. IF cleaning the filter with a vacuum does not remove the trapped particles, wash the filter with plain water and dry it completely before replacing it in its holder in the console.

NOTE: Replacement filters may be ordered through PHI Customer Service under P/N 636840.

Several electronics units also contain filters mounted over the cooling fans attached to the back of the chassis. These electronics filters also require periodic cleaning.

The filters contained within the units cannot be easily removed, therefore, the preferred method of cleaning should be externally with a vacuum cleaner. If this method does not work sufficiently, the unit must be removed from the electronics console. Take the unit away from the system and use compressed dry air from the inside of the unit to blow the filter clean. The top of the chassis may require removal in order to accomplish this task. Reinstall the top of the chassis and return the unit to the electronics console.

Convectron Gauges

A complete *Installation, Operation and Maintenance Instructions* manual published by Granville-Phillips is shipped with the TRIFT system and contains all of the necessary service procedures and precautions. The instructions listed below contain just a brief overview of the cleaning and maintenance steps.

The TRIFT system contains two different types of Convectron gauges: an analog version and a digital version. Both types attach to the roughing manifold in the vacuum console.

Analog Convectron Gauges: A gauge may become contaminated with oil or other films over time, but can be cleaned. When the small internal sensor wire is contaminated, its emissivity or its diameter may be altered and a change of calibration will result. Cleaning with trichloroethylene, perchloroethyylene, toluene, or acetone is possible, but it must be done very carefully so as not to damage the sensor. Consult the Granville-Phillips service manual for the proper cleaning procedure.

If required, the Convectron gauge may be baked to 150°C non-operating while under vacuum with the connector/cable removed.

A small diameter internal sensor wire can be damaged by small voltages. Do not perform electrical continuity tests with instruments applying in excess of 1 volt when the gauge is at vacuum or 5 volts when at atmospheric pressure. The gauge should show the following resistance measurements (pin numbers are embossed on the gauge base).

Table 11-3.   *Resistance Measurements on a Model 275 Convectron Gauge.*

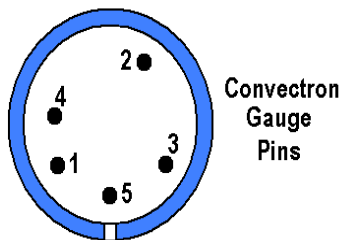| Pin to Pin Connection:	Resistance Measurement:	Notes:
Pins 1 to 2	20 to 25 ohms	If Pin 1 to Pin 2 resistance = 800 ohms, the sensor wire is open.
Pins 2 to 3	50 to 60 ohms	
Pins 1 to 5	175 to 190 ohms	

Figure 11-1.   *Diagram of the Convectron Gauge Pin Layout (analog version).*

Detector (DMCP)

The TRIFT detector consists of a set of Dual MicroChannel Plates (DMCP) separated by a spacer ring. A voltage is applied to the pair of channel plates. The entire DMCP floats between 0 and 10,000 volts. Next is a phosphor screen which faces the DMCP. The front of the phosphor screen is biased by a voltage. Both the DMCP and the phosphor screen are in vacuum and located on the back of the TRIFT spectrometer. A CCD camera, attached to the outside of the vacuum chamber, is directed at the phosphor screen. Electrons impinging on the phosphor screen are viewed by the CCD camera. An image is then transmitted and displayed on a SONY image monitor.

The voltage on the DMCP must be gradually increased as the system ages. At some point in time, it will no longer be possible to reach the voltage at the knee of the counts versus DMCP voltage curve. When this occurs, it will be necessary for Field Service to replace the DMCP detector.

Emergency Manual Off (EMO) Box Lamps

The Emergency Manual Off (EMO) box (P/N 641352) uses lighted switches to indicate whether power is ON or OFF. Pressing the AC MAINS button on the EMO box illuminates all the bulbs for a lamp test. Any of the lamps that do not light when AC MAINS is pressed need to have the bulb replaced. All of the bulbs are identical and are rated at 28 Volts, .04 A.

Use the following procedure to replace any of the bulbs (P\N 638274).

ATTENTION: This bulb replacement procedure involves activating the switch associated with the burned-out bulb. Prepare the system conditions for the activation or deactivation of any switch on the EMO box to replace the bulb(s).

1. Prepare the system for activation of the switch that has the bad bulb.

2. Pull the colored button plate off.

3. Pull the bad bulb out of its seating, and push in the new bulb.

4. Push the colored button plate all the way back on to the box, which will also activate its switch. Push the switch a second time to deactivate the switch.

Ions Gauges

The TRIFT system has a single (1) ion gauge and two (2) Convectron gauges. See also "Vacuum Gauge Controls" later in this section. The Convectron gauges are Granville-Phillips gauges. Refer to the manufacturer's manual for replacement procedures.

The MAIN CHAMBER ION GAUGE is nude ion gauge assembly. It is mounted on a vacuum flange containing a twist-lock connector feedthrough that mates with a corresponding connector on the ion gauge cable. The nude ion gauge is equipped with two filaments, as shown in Figure 11-2 below. Only one ion gauge filament is used at a time, depending on which way the cable is installed on the ion gauge flange.
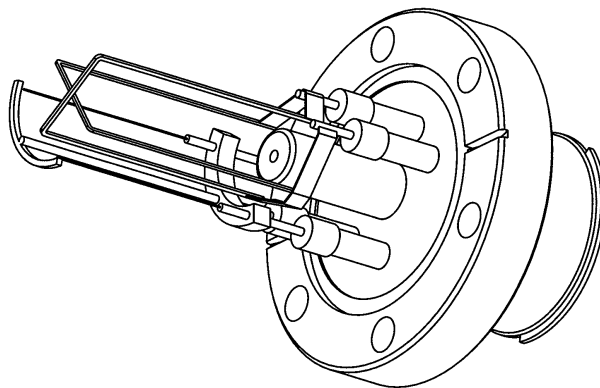
Figure 11-2.   Side View of the Ion Gauge Filament Assembly.

When one filament burns out, the cable connector may be switched to the second filament without breaking vacuum as follows.

NOTE: If both filaments in a nude ion gauge are burned out, the system must be vented to remove the gauge. This is a PHI Customer Service procedure. Filament replacement kits (P/N 2141400) may be ordered from PHI Customer Service. One kit is sufficient for one ion gauge filament change.

Light Source

The light-source control is typically located within the electronics console and includes an extended fiber optic cable to direct the high intensity light to the sample. One end of this fiber optic cable is attached to the power supply and the other end is connected to a mount attached to the vacuum chamber. This light source is used for illuminating the sample area for viewing by the sample camera.

The replacement halogen bulb is a type EKE lamp, rated at 150 Watts and can be ordered from PHI Customer Service (P/N 622301). Replacement of the lamps is relatively easy and is done by turning off power to the unit, removing the burned out lamp, placing the new lamp in the unit and replacing the cover. No tools are required for replacement of the bulbs.

Liquid Metal Ion Gun (LMIG)

Maintenance on the LMI gun may involve replacement of the emitter, the Moveable Variable Aperture (MVA), and/or the Liquid Metal Ion Source (LMIS). Repair and replacement of these items is considered extensive. Therefore, procedures covering the major components within the LMIG are included later in this section of the manual.

The controller for the LMIG does not require routine maintenance of any type.

Rough Pump (Alcatel)

The rough pump does require normal maintenance at six-month intervals including replacement of the wick and the pump oil. Follow the maintenance procedures included in the Alcatel manual shipped with the TRIFT system.

If the rough pump is unusually loud, maintenance may be required sooner than every 6 months.

System Vacuum Control (SVC)

Maintenance and service of the SVC is described in the System Vacuum Control component manual. The SVC does require maintenance on a routine basis.

However, the System Vacuum Control does contain a 3 Volt Lithium backup battery (P/N 639187) which is used to retain the Watcher software tasks. This battery must be changed every 24 months. The battery is located on the main SVC printed circuit board. To replace the battery, remove the SVC from the electronics console and open the chassis. Locate BT1 on the board, cut the plastic tie wrap holding the battery in place and remove the battery. Replace the battery while observing the correct polarity and install a new tie wrap. Reinstall the chassis top and reinstall the SVC unit into the electronics console.
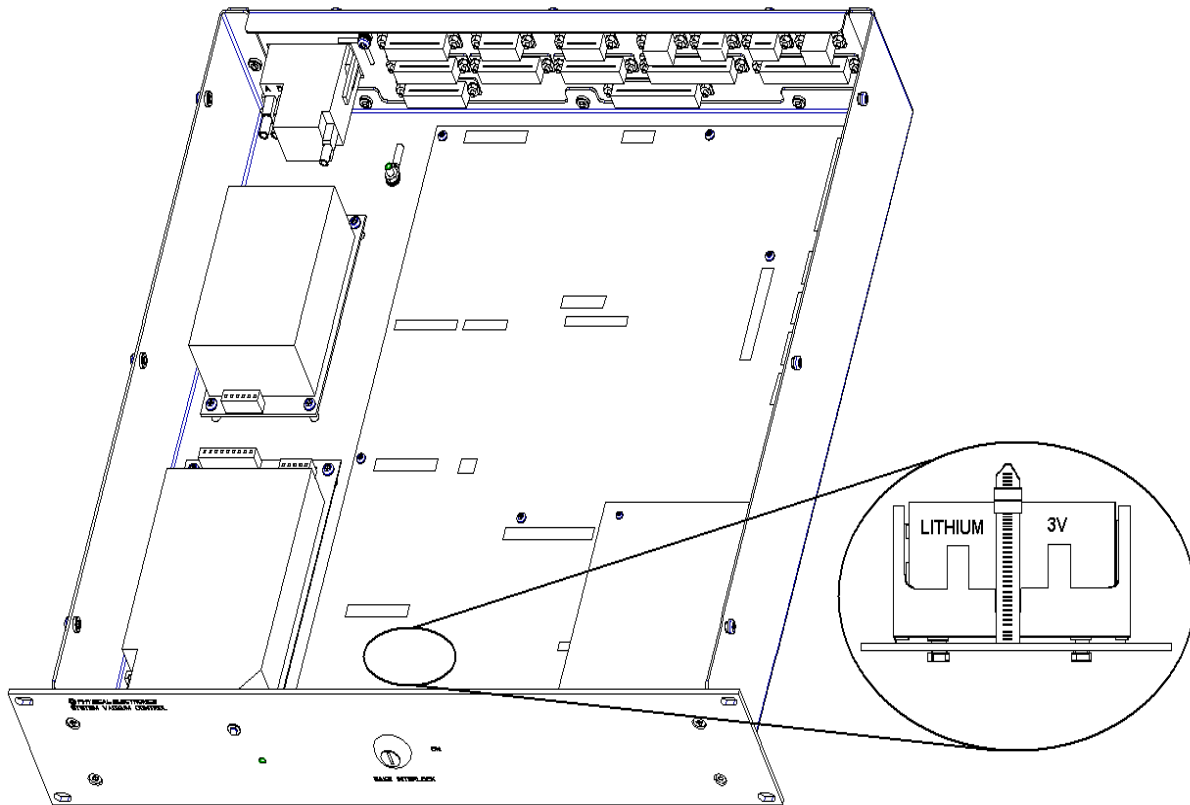
Figure 11-3.   *Location of the backup battery within the System Vacuum Control chassis.*

Sample Bias Feedthrough / Connector

The TRIFT system contains a high voltage bias feedthrough located in the center of the sample stage on the vacuum console. This sample high voltage bias feedthrough should be inspected for corrosion.

If corrosion does exist, the high voltage feedthrough must be replaced (P/N C79141) and also the Sample Bias High Voltage cable (P/N 633797).

Turbo, Rough Pump Maintenance (Wicks)

The TRIFT system uses two (2) turbo pumps: a 160 l/s turbo pump is mounted to the introduction chamber and a 240 l/s turbo pump is mounted to the analytical chamber. It is recommended that the turbo wicks be replaced annually and that rough pump oil be changed every 6 months. Refer to the turbo maintenance section later in this section.

Vacuum Gauge Controls

The Vacuum Gauge Controls must be calibrated on-site as follows for proper operation of the routines built into the System Vacuum Control software:

- All system Convectron gauges must be calibrated on-site to read 760 torr at atmospheric pressure; and

- Ion gauges must be calibrated to 0 torr at full ultra-high vacuum at the site.

Calibration is adjusted by turning the set screw associated with the gauge to be adjusted. The set screw is accessed by opening the "door" on the front of the Vacuum Gauge Control located in the electronics console. Refer also to the OEM manual provided with the TRIFT system.

Appendix A:
Service Procedures

Appendix A contains a set of three service procedures which address the following topics:

- Rigorous heating to start the LMIG Source;
- Mechanical alignment of the Cesium column to the analysis region; and
- Low energy beam alignment.

ATTENTION: The following instructions and procedures are for use by qualified service personnel only. To avoid personal injury, do not perform any installation or servicing procedures unless qualified to do so and having received training from PHI personnel.

Rigorous Heating to Start the LMIG Source

If the source will not fire after performing the steps described in Section 4 ("Heating the Source") of this operator's guide, it may be necessary to heat the source until the gallium boils, thereby removing whatever is causing the source not to fire. This extreme heating will inevitably reduce the lifetime of the source because there is a limited amount of gallium in the source. This lifetime can be reduced dramatically IF the heating process is not carried out extremely carefully. It is recommended that a PHI Service Engineer perform the steps outlined below.
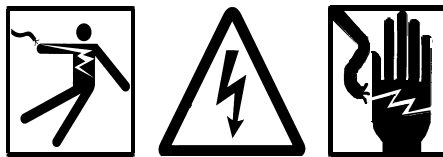
CAUTION: Only use a high voltage probe for the following step.

1. Before the final decision is made to heat the source, check that the extractor voltage supply is functioning properly. First, set the extraction voltage to zero, then remove the extractor lead from the gun. Test the supply by setting it to 1 kV, and verify this potential at the end of the lead <u>with a high voltage probe</u>. If the source will not fire after heating as described above with maximum extractor and suppressor voltages applied, <u>and</u> after the extractor supply has been checked, the following additional steps are recommended.

2. Set the extractor voltage to <u>below</u> the typical value, e.g. 5 kV with the suppressor set to a value of 1 kV.

3. For 500-hour sources, raise the filament voltage to a value of 3.5 - 3.8 amps (PN 11604), and begin timing. After 30 seconds, turn the filament current down to zero (0 Amps) as quickly as possible. For the 1500-hour sources (PN 16830), use LESS current than the 500-hour sources, typically use a range between 3.1 - 3.2 amps. [Do NOT apply 3.5 - 3.8 amps to a 1500-hour source!]

4. Raise the extractor voltage until the source fires. If the source fires at the 'normal' voltage, everything is fine. If the source fires, but at a high extractor voltage, the source tip is probably near the end of its life. Preparations should be made to replace the source when it will be most convenient.

5. If the source fails to fire, even at maximum extractor and suppressor voltages, the source is probably 'dry' and preparations should be made to replace the source immediately.

6. It is possible to conduct step 2 above with the extractor voltage set <u>above</u> the 'normal' operating voltage (e.g., 12 kV). When the source fires, it may do so with a very high emission current. Both the extraction voltage and the filament current should be reduced as quickly as possible. When this occurs, some gallium will have boiled away, and a significant volume of gallium will probably have been extracted in the form of microdroplets, which further reduces the gallium remaining for source emission. For this reason, extreme heating without extraction is preferred at least initially.

Mechanical Alignment of Cs Column to Analysis Region

In the event of not measuring any sample current (given that current can be measured at the Faraday cup), a simple, mechanical misalignment of the column may be the cause. Mechanical alignment of the column to the sample (and more precisely, to the analysis region) can be performed with the ring of knurled knobs located around the circumference of the column, however, this procedure must be referred a qualified technician or service engineer.

Lower the sample potential to zero (0) volts and connect a picoammeter to the stage. Maximize the sample current by adjusting the alignment knobs. Once a sample current of 1-2 nA for a 10kV beam (using the 5ml slit and 5ml aperture) has been achieved, remove the picoammeter, reconnect the MHV stage cable and proceed to fine tuning of the column with the aid of imaging at the DMCP detector.

Appendix B:
Mass Resolution Tuning Grid

Appendix B contains a Mass resolution Tuning Grid template for recording
keeping on the TRIFT III system. The system operator may use this template to
make additional copies to keep with the TRIFT system records.

APPENDIX B:
TRIFT MASS RESOLUTION TUNING GRID

Sample Potential:											
Raster Size:											
Matsuda Voltage:											
Detector Voltage:											
Detector Position:											
Post Accel.:											
C.D. No.:											
LMIG Emission:											
Buncher Voltage:											
Buncher Delay:											
Blanker Voltage:											
Pulser Voltage:											
Mass Atomic:											
Resol. Organic:											

Appendix C:
Indium Liquid Metal Ion Source (LMIS)

Appendix C contains a set of procedures which address the following LMIS topics:

- Daily operation of the source;
- Periodic revitalization of the source; and
- Parameters affected by the Indium source.

LMIS Installation into the TRIFT

Before installing the indium LMIS, check the performance of the instrument (i.e., is the LMIG 'wobble free') and also log the best mass resolution on $^{28}Si^+$.

The indium LMIS installs into the FEI LMIG in exactly the same manner as the gallium LMIS. Following the installation of the emitter, the following procedure should be followed:

Vacuum

The LMIS typically requires installation by a field engineer from Physical Electronics. Evacuation and bakeout of the LMIG is performed overnight (6 hours minimum). Once the gun pressure has fallen into the 10^{-9} torr region (or <2µA ion pump current), it can then be operated.

Conditioning the Source

High Voltage Conditioning

Before attempting to fire the source, the LMIG should go through a high voltage conditioning:

1. Load a 'zero volts' instrument file and gradually increase the beam energy from 0 to 25 keV in 5 keV steps (up to 15 keV) and then 1 keV steps (up to 25 keV).

2. With the filament current still set to 0 Amps, increase the extractor potential in 1 kV steps up to 12 kV (or at least 10 kV, if the LMIG is old and has a recognized voltage limitation). There should be zero emission current.

Outgassing the LMIS

1. Re-load the 'zero volt' instrument file and increase the heater current to 2.0 Amps. Check the ion pump current (i.e. should not rise). Increase the current in 0.2 Amp steps, up to 2.8 Amps. If the ion pump current rises to greater than 5µA, allow the source to sit at 2.8 Amps for approx. 20 minutes while it outgasses. (Note that it can be fatal to raise the extraction potential to 9 or 10 kV while the source is outgassing heavily).

2. Once the ion pump pressure has recovered to less than 5 µA (with the heater current at 2.8 Amps), reduce the heater current to 1.8 Amps and wait 5 minutes for pressure to recover further.

Operation

Igniting the Source

1. Load a 15GA3.INS (gallium file) or a 15IN3.INS (indium file, if one exists) instrument file. If you load a 15IN3.INS file, quickly set the filament current to zero once the file is loaded.

2. Gradually raise the extraction potential to 10 kV (<u>Note</u> For TRIFT I's, where an extractor voltage of 11 kV is required for a gallium LMIS, you should set the extractor potential to 13-14kV) . Check the ion pump current does not rise above 2 µA.

3. Set the filament current to 2.0 Amps. Wait.

4. If there is no emission current, then raise the filament current by 0.05 Amps. Continue to raise the filament current in 0.05 Amp steps, waiting 30 seconds between each increment. **Do not exceed 3.6 Amps.**

5. When the source fires, allow the source to emit at > 20 µA for ~30 seconds and then lower the extraction voltage to give 5 µA of emission current. Let the source emit at 5 µA for a 3-4 minutes.

6. Once the emission current has stabilized, reduce the filament current and extractor voltage quickly until emission terminates.

7. Wait at least one minute. Raise the extractor to 10 kV and then raise the heating current from 1.90 Amps in 0.05 Amp steps (wait 20 seconds between steps), until stable emission is obtained. Lower the extractor voltage, until the emission current is <2 µA. Raise the heater current by an extra 0.1 µA - this will now be the operating filament current.

8. Typically, a source will ignite for the first time with the following parameters:

V_{ext} = 10 kV (TRIFT II) V_{ext} = 13.5 kV (TRIFT I)
I_{heater} = 2.5-3.0 Amps

   And typical operating parameters are :-

V_{ext} = 6.5-7.5 kV (TRIFT II) V_{ext} = 10.0-10.5 kV (TRIFT I)
I_{heater} = 2.1-2.5 Amps

If the source ignites, but requires an excessively large extractor voltage (ie >7.5 kV) to maintain emission, then the source will have to be heated at 3.2-3.4 Amps for approximately 30 seconds with Vext = 9-10kV.

Note down the ignition and subsequent operating parameters for the source in the 'Emitter Record' that came attached to the new source.

Day to Day Operation

1. Raise the extraction potential by 2000 V from the normal operating voltage.

2. Set the filament current to 0.2 Amps above the usual operating value. It will take about 30-60 seconds for the tip to reach operating temperature.

3. When the source fires, allow the source to emit at ~ 20 µA for ~20 seconds and then lower the heater current to the usual operating value and reduce the extraction voltage to give ~1 µA of emission current. The extraction voltage should consistently return to the usual extraction potential and the fine tuning of the emission current should now be done with the suppressor voltage.

4. Typical switch on parameters are:

V_{ext} 8.5 to 9.5 kV
V_{sup} -1.0 kV I_{em} >20.0 µA for 20 seconds
I_{fil} 2.4 Amps

Typical operating parameters are:

V_{ext} 6.5 to 7.5 kV
V_{sup} -0.00 kV I_{em} 1.0 µA
I_{fil} 2.2 Amps I_{targ} 10-20 nA (large MVA, +75 V sample bias)

NOTE: Experience has shown that the extractor potential required for an indium LMIS under normal operation can be as low as 6000 V (as opposed to 7500 V with the gallium LMIS). Since the tip geometry is the same for both gallium and indium sources, the lower extract potential is attributed to the lower surface tension and flow impedance of liquid indium.

Revitalizing the Indium Source (every 1-2 months)

After intervals of 100-200 µA hrs of operation, the liquid flow impedance to the tip often increases to the point where ion emission can not be sustained without raising the extractor potential above the usual value. This is usually accompanied by a very weak 'emission current' v's 'extractor voltage' dependence. In this 'flow restricted' condition it may take 1000-1500 V increase in extractor potential in order to increase the emission current from 5µA to 10µA. After re-vitalizing the source, the emission current should go from 5µA to 10µA with only a 200-500V increase in extractor potential.   In order to revitalize the source, such that it will operate at its usual extractor potential it will be necessary to follow the procedure below.

1. Set the heater current to it's usual value and then increase the extractor voltage by 2500 V in 500 V increments (e.g. from 7000 V to 9500 V). If there are no signs of high voltage break down (e.g. a rapid increase in ion pump current) then you can proceed.

2. Increase the heater current to 2.8 amps and wait 1 minute (or until the emission current increases above ~25 µA).

3. If the emission current hasn't increased significantly (ie by 10 µA or more), then raise the heater current to 3.0 Amps. Wait for 30 seconds. Again, if the emission current hasn't increased significantly, then raise the heater to 3.2 Amps and wait 30 seconds. Usually, an indium source will start to emit strongly once it has been heated at 3.2 Amps for 30 seconds or so. If this is not the case, then increase the current to 3.4 Amps for no longer than 30 seconds.

4. If the source fails to emit all together and the source has been operated for >800 µA hours, then the indium reservoir has probably run dry and will need replacing.

Instrument Parameters Affected by the Indium LMIS

LMIG Alignment

If starting with an old 15 keV gallium *.INS file, the ion source brass knobs should be mechanically aligned, first to measure current on the LMIG Faraday cup and then finally onto the sample. When current is hitting the sample, try to acquire an SEM image of the copper grid.

Operate the LMIG extractor potential at a lower value than that required by the gallium LMIS. Since the extractor electrode has beam focusing properties, the lens 1 potential will need to be tuned again to keep the first crossover in the blanking plates and the second crossover on the sample. Lens 2 should stay the same.

Fine tuning of the source position can be done by aligning the brass knobs while wobbling lens 2, with a view towards minimizing image motion.

Ideally, the new source should be aligned to the existing ion gun deflector voltages, but in reality some fine tuning on the beam deflectors may be required in order to achieve fine alignment of the beam. The procedure for fine tuning the beam is described elsewhere in the User's Guide.

Pulse/Buncher Timing

the LMIG buncher delay will need to be re-optimized for indium because the velocity of ^{115}In is approximately ¾ of ^{69}Ga for a given beam energy. In 15 keV pulse/bunched indium mode, reducing the buncher delay by ~10 ns from that required for gallium is close to the optimum setting. From here, fine tune the delay by 1 or 2 ns (in both directions) to find the optimum delay for best mass resolution.

It should be noted that the energy spread of ions from an indium LMIS is greater than those from a gallium LMIS. The larger energy spread results in a broadening of the primary bunched pulse width, such that the mass resolution for ^{28}Si$^+$ is limited to 7,500 under typical source operating conditions of 1µA emission and a heater current of 2.2 Amps. A shorter primary pulse width can be obtained by running the source at a lower emission current and the lowest possible heater current, but this may result in unstable emission. Mass resolution as a function of emission current is shown in Figure A-1 below, with heater current as a parameter.
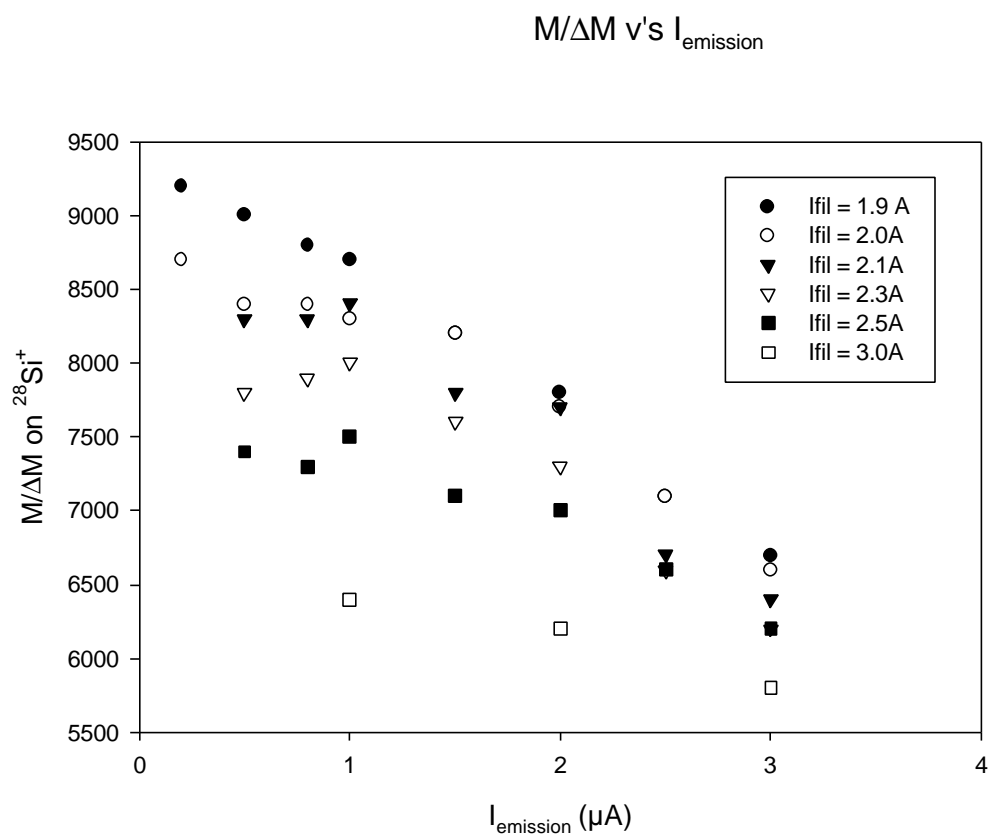
M/ΔM v's I$_{emission}$

Figure C-1.   *Illustration of Mass Resolution as a function of Emission Current.*

The flight time of the indium ions to the sample is slightly longer than that for gallium ions, so the mass spectrum will be slightly shifted up the mass scale if the previous mass calibration was done with a gallium LMIS. The mass spectrum will need to be re-calibrated. Once the instrument file for indium operation has been set up, save the file with a new name.