



ISO/IEC 17025

CALIBRATION LABORATORY

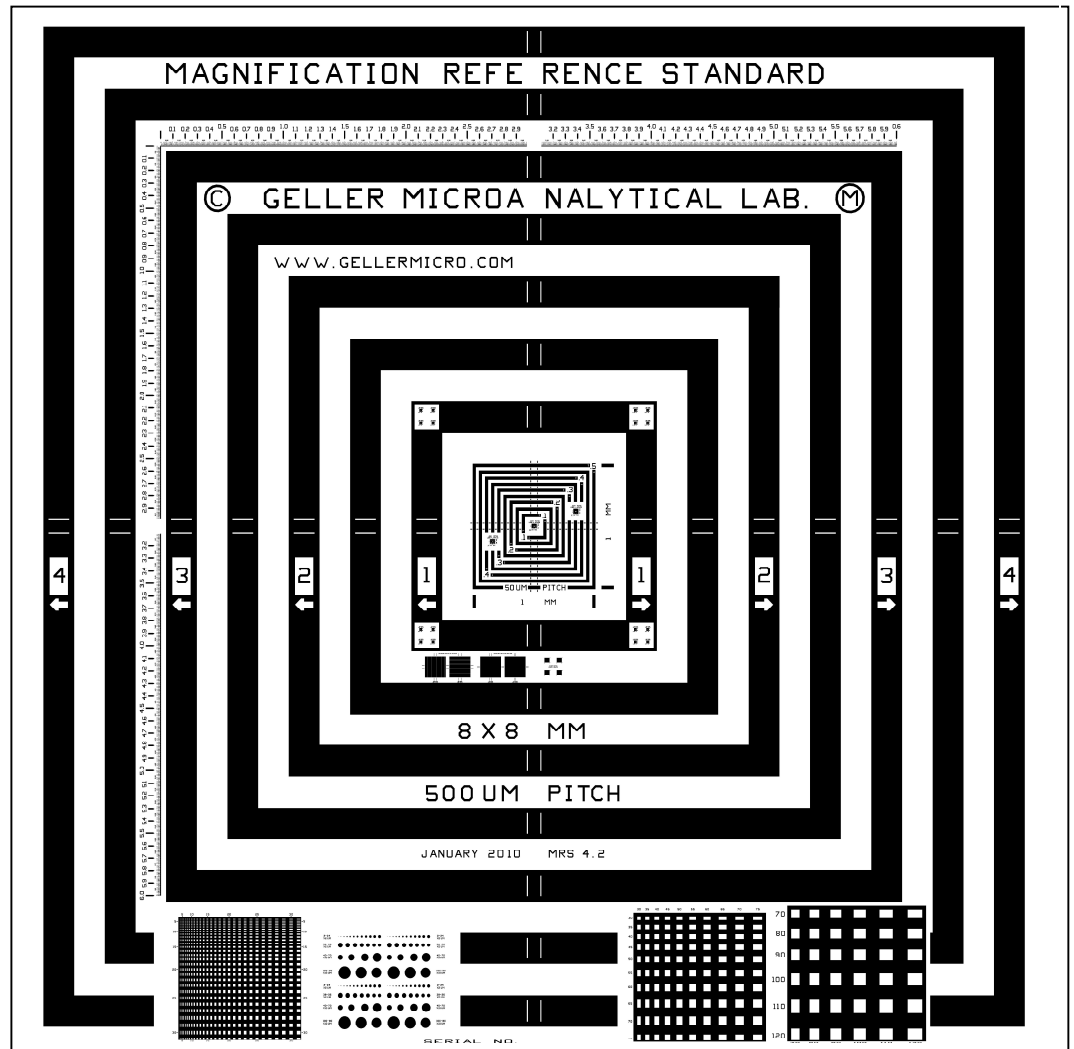


MRS-4.2

NIST and NPL (NIST counterpart in the U.K.) traceable - **Certified Reference Material**. A Magnification Reference Standard designed for Microscopy by Microscopists.

“Do it right... the first time”

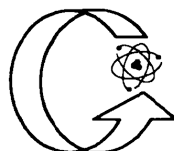
- THE 10X TO 200,000X STANDARD -



for ISO-9001 and ISO-17025 compliance

Resource Guide

- Product Design
- Magnification Measurement & Error Assessment
- Magnification Calibration Procedures for electron, optical, video, Vision Systems and scanning microscopies (following ASTM E766-98 and ISO-16700)



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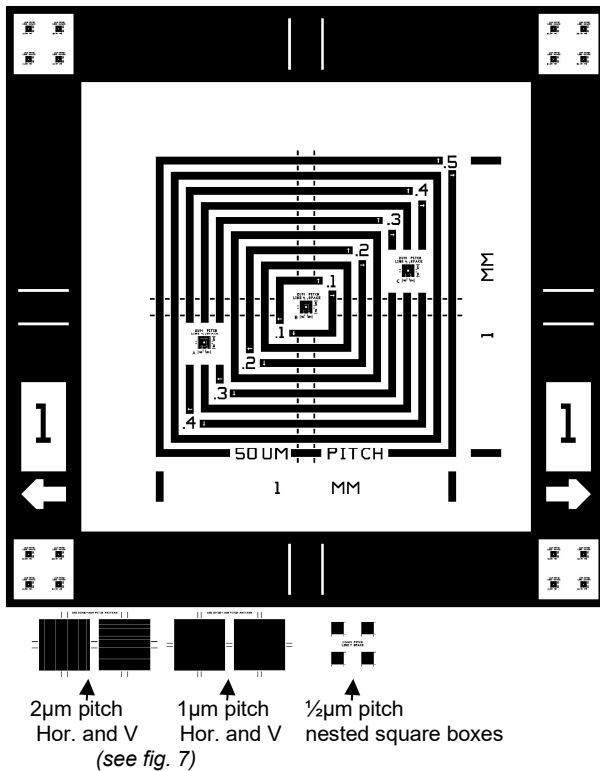
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We are certified to ISO-9001 and accredited to ISO-17025 - March 2011

MRS-4.2

This is our fifth Generation, NIST and NPL (NIST counterpart in the U.K.) Traceable, Magnification Reference Standard & Stage Micrometer. For Instrument Calibration from 10X – 200,000X (1/2 μm minimum pitch).



- **Electron Microscopy:** SEM (secondary (in-lens & conventional and backscattered electrons), TEM (for use with a bulk holder- the MRS-4.2 can be reduced to 3mm \varnothing by ½ mm thick).
- **Scanning Microscopies and Profilometry:** STM, AFM, stylus and optical etc. The pattern height is 70 – 100 nm.
- **Optical Microscopy:** transmitted, reflected, bright/dark field, differential contrast, and confocal.
- **Chemical mapping:** EDS, WDS, micro/macro XRF, XPS, Auger & others. The pattern is fabricated using 70 - 100 nm CrO₂/Cr over quartz.
- **Particle Size Counting:** series of circles, squares & rectangles for calibration confirmation.

INTRODUCTION

Geller MicroAnalytical Laboratory introduces the MRS-4.2, our fifth generation magnification calibration standard (the MRS-3 and 5 (with pitch patterns to 80nm) are still available). These calibration standards are highly accepted pitch standards, with over 1,000 delivered. They are offered as traceable certified reference materials or, optionally, at lower cost without traceability. To support the standard we offer a cleaning service and recertification program, as required by the ISO-9001, ISO-16700 and QS-9000 international quality standards.

PATTERN DESIGN

The MRS-4.2 is fabricated by using the highest accuracy electron direct write semiconductor manufacturing equipment available today. The pattern is anti-reflective chromium (approx. 70 – 100 nm CrO₂ over Cr) over quartz. Imaging contrast in both secondary and backscattered electron mode is very high. The overall size is $\approx 9\text{mm} \times 9\text{mm} \times 2.3\text{ mm}$ thick. The size can be modified for special applications (such as for a TEM bulk holder or mounting on a large wafer) to 3 mm \varnothing by ½ mm thick – or other custom sizes. For applications requiring an electrically conductive sample (SEM at $\approx 1\text{keV}$ and higher), the MRS-4.2 is coated with a proprietary material which allows for image observation at any accelerating voltage. A distinct advantage of this coating is that electron beam tracks from contamination are removable by plasma etching. Applying a fresh coating usually restores the MRS-4.2 to like new condition.

The simple geometric design of the MRS-4.2 has groups of nested squares spanning several orders of magnitude with pitches of ½ μm , 1 μm , 2 μm , 50 μm and 500 μm . Newly incorporated into the standard is a ruler which is 6mm long with 1 μm increments and large 1 and 2 horizontal and vertical pitch patterns for ISO-16700 compliance. We measure and certify pitch (the distance between repeating parallel lines using center-to-center or edge-to-edge spacing. This is the only type of measurement that can be used to relate measurements from different microscopy techniques (see “Submicrometer Linewidth Metrology in Optical Microscopy”, Nyssonen & Larrabee, Journal of the Research of the National Bureau of Standards, Vol. 92, No. 3, 1987). Linewidth measurements (the measurement of a single line or space width) can only be related if the same type of illumination is used as for the calibrating instrument since edge effects lead to uncertainty in the edge locations. Using pitch measurements errors from edge-to-edge locations cancel as long as like positions are measured. Several examples are given on the illustrations in this resource guide.

Square boxes are used for measuring magnification simultaneously in the X and Y directions. This gives a measure of image skew, barrel, pincushion and other non-linearities which can have various origins, such as from stray magnetic fields. With the MRS-4.2 we have provided multiple boxes for the ½, 1, and 2 μm pitches.

The largest pattern has an overall dimension of 8mm square. It contains lines and spaces that are nominally 250 μm wide (totaling 500 μm pitch). This can be used to check magnifications from 10X – 100X. The 50 μm pitch patterns are useful from 100X – 1000X. The 2, 1, and ½ μm pitch patterns will allow calibrations up to 200,000X. The “ruler” has an overall length of 6mm in the X and Y directions. The 1 μm increments have graphics every 10 μm and emphasized lines and graphics every 50 μm and 100 μm . For 0.1 μm pitch patterns see the MRS-6 standard.

Also included are squares and rectangles from 1 – 31 μm in 1 μm increments and 30 – 75 μm in 5 μm increments. Also included is an array of 4 repeating patterns of circles with diameters ranging from 2 – 39 μm in 2 μm increments and 40 – 100 μm in 10 μm increments. These patterns are useful for checking the performance and setup of particle size counting systems. From our experience it is challenging to get the proper size distribution knowing the correct answer. The patterns can also be used for determining imaging and chemical spatial resolution, and chemical mapping. Since the circles and rectangles are not pitch patterns, they cannot be made traceable.

HOW ACCURATE ARE THE PITCH PATTERNS?

Several MRS-4's have been measured by the National Physical Laboratory in the U.K. (the NIST counterpart in the United Kingdom). The results of their measurements combined with our measurement uncertainty follows: 500 $\mu\text{m} \pm 0.25$, 50 $\mu\text{m} \pm 0.104$, 2 $\mu\text{m} \pm 0.031$, 1 $\mu\text{m} \pm 0.03$, 0.5 $\mu\text{m} \pm 0.038$. It goes without saying that these reported results from a national laboratory, such as NPL, are highly conservative. See http://gellermicro.com/mag_standards/MRS-CofA.pdf.

WHAT IS INCLUDED THE CERTIFICATION REPORT?

We follow the ISO guidelines for certification and traceability. Included is the unique serial number engraved on the standard, certification date, recertification due date, operator, instrumentation used, and actual pattern measurements along with a measure of accuracy. This report has satisfied 100% of our customer audits.

Selected MRS-4.2 Patterns

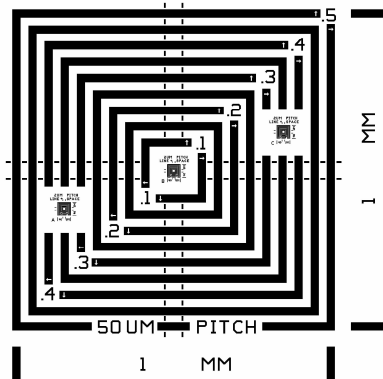


Fig. 1 - 50µm pitch pattern. The overall width is 1mm. Measure magnification using multiple pitches totaling 0.950mm, as above.

$$\leftarrow 43\mu\text{m}/11\mu\text{m}=3,909\text{X} \rightarrow$$

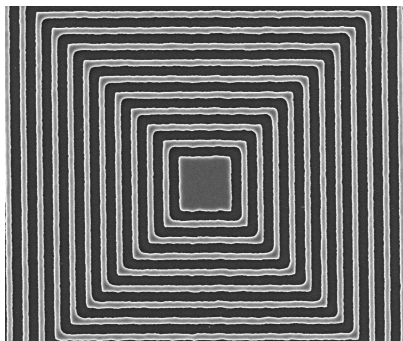


Fig. 4 - 1/2µm pitch pattern. Overall size is 40µm. 4 of these patterns are located 1 mm above the "MM" just below the pattern center.

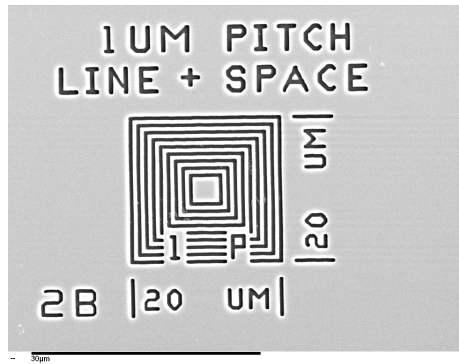


Fig. 2 - 1µm pitch pattern. 16 of these patterns are located at the outside of the 1mm box

$$20,000\mu\text{m}/0.5\mu\text{m} = 40,000\text{X}$$

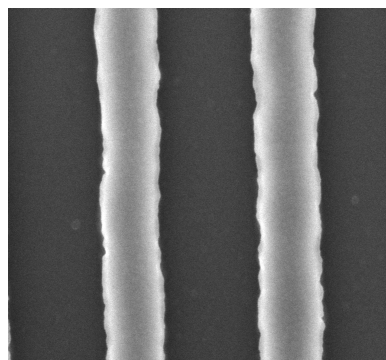


Fig. 5 - 1/2µm pitch pattern. Edge variation is approximately 20nm.

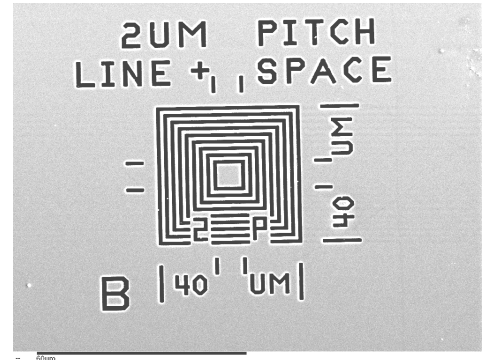


Fig. 3 - 2µm pitch pattern. 3 sets of this pattern (with one in the exact center) can be seen in fig. 1.

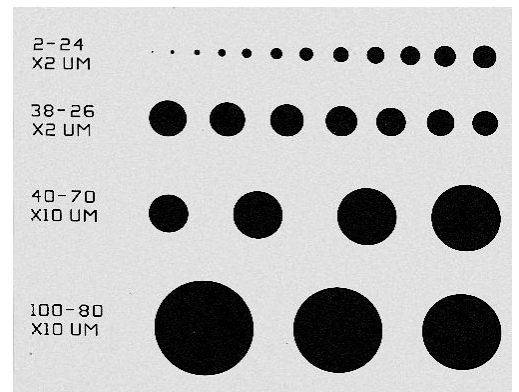


Fig. 6 - Circle pattern from 2 to 100µm. 4 sets are included.

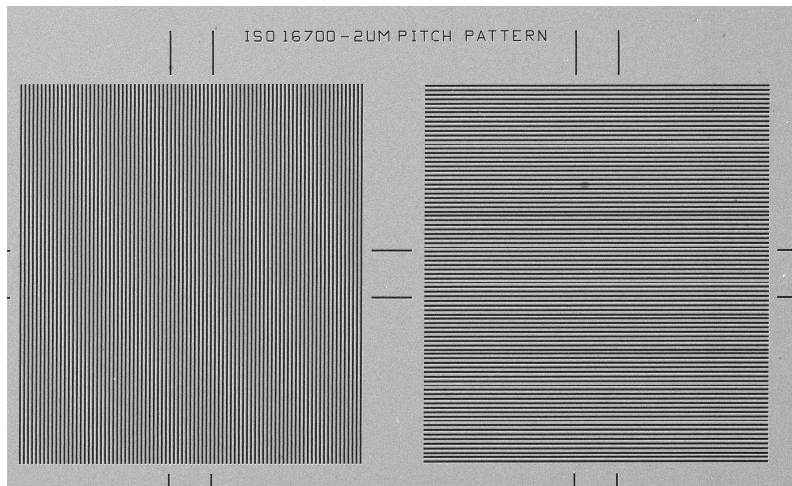


Fig. 7 - Large 1 and 2 µm patterns; horizontal & vertical.

$$\begin{aligned} \text{X axis: } 30 \text{ mm}/0.030\text{mm} &= 1000\text{X} \\ \text{Y axis: } 24 \text{ mm}/0.025\text{mm} &= 960\text{X} \end{aligned}$$

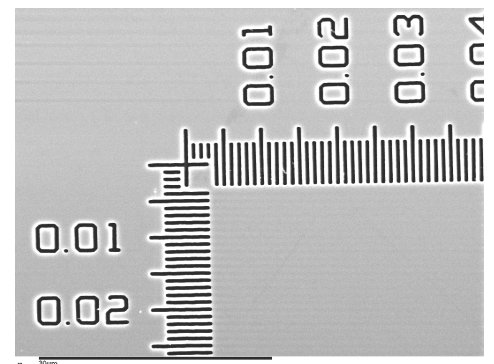


Fig. 8 - The "ruler" has 1µm increments over a 6mm distance in both X and Y axes. This pitch pattern can be used at any magnification.

Magnification Determination

- Magnification should be determined using pitch measurements- the process of measuring repeating structures. Several examples are shown above. Since the circles and squares cannot be measured using "pitch" their sizes cannot be made traceable (see ASTM E766-98).
- Magnification is simply defined as the ratio of the image size to object size (be careful to use the same units - µm, mm, nm, etc.). Please carefully note the locations of the above magnification measurements relative to the patterns in figure 4, 5, and 9.

RETAINERS

The MRS-4.2 pattern sits on highly stressed quartz, which is easily chipped. We strongly suggest ordering the standard with one of our retainers, or letting us do the mounting on your holder to protect the standard. We can also size modify the MRS-4 to meet your needs. The following retainers and holders are available. See http://gellermicro.com/mag_standards/mrs.html (page bottom).

- SEM/R – 25mm ϕ X 3mm thick with a central hole. Can be used for optical transmitted light and SEM applications. This is our most popular retainer. The MRS-4.2 is secured with ultra high vacuum compatible silver epoxy.
- 3mm ϕ – modify the MRS-4.2 to 3mm ϕ X 0.5 mm thick.
- Pin Stub – commonly used SEM stubs have a 1/8" pin and 1/2" top surface.
- Others – Hitachi, VG PHI, etc. Please call for your special needs.

TRACEABILITY

Why should you consider a traceable standard? Beyond the requirements of national and international quality standards, purchasing a CRM (certified reference material) from a national laboratory or a traceable standard from a certifying body (such as Geller MicroAnalytical Laboratory) guarantees dimensions. Most commercially available standards have unknown accuracies. The use of these standards, such as grids and spheres (which may change size under vacuum or are distorted), have called into questions the accuracy of our MRS. In every case it was determined their dimensions were not as represented or there was miss-operation by the user or service engineer calibrating the instrument. The MRS-4.2 is offered with or without traceability. The non-traceable standard differs only in documentation and cost. Traceability in the X and Y dimensions is established from "masters" that have been measured by NPL (National Physics Laboratory), the NIST counterpart in the U.K. The "Z" dimension (100nm) has been established on MRS "masters" by NIST.

RECERTIFICATION PROGRAM

We are often asked why the MRS needs re-certification. Under ISO-17025 guidelines your quality department should determine the re-certification interval. They are most familiar with the company's quality system requirements. Recertification is a common practice for devices such as gage blocks and electronic instruments. Over the years we have found several standards which could not be recertified due to physical damage and excessive contamination. The physical damage is most often caused by optical microscope objectives being drawn across the pattern surface or abuse. In a few cases we have seen electron beam damage and corrosion from storage in a corrosive or hostile environment. Recertification insures your standard will perform its proper task and that you will be meeting your quality system guidelines.

OPERATING PARAMETERS

Optical microscopes can use the MRS-4.2 in all imaging modes. The antireflective coating greatly reduces scattered light enabling high contrast images to be observed and photographed. Magnifications can be measured directly on viewing CRTs, in reticles mounted within the ocular, or directly on photomicrographs. For instruments with verniers or electronic calipers distance measurements can be verified using a pitch pattern of appropriate size. If your application primarily consists of optical microscopy we suggest ordering the MRS-4.2 without the conductive coating since it reduces the reflected optical contrast in the image.

Geller MicroAnalytical Laboratory

Geller MicroAnalytical Laboratory, certified to ISO-9001 and 17025, offers several unique products and services to the technical community. We have developed products out of the need to support our analytical services. Our staff takes pride in performing state-of-the-art analyses on difficult specimens and we do our utmost to go beyond just offering analytical data. We interpret the information, as well. As our satisfied repeat clients and publications reveal, we have developed several unique techniques for problem solving. Rest assured we will maintain your confidentiality with the utmost care as our small, but capable company, concentrates on providing you with answers.

PRODUCTS

- **UHV-EL:** Chemical standards for microanalysis; choose from over 250 pure elements, compounds, minerals, glasses, and alloys. Some are NIST traceable. These standards are used for energy and wavelength dispersive x-ray analysis, surface analysis and macro & micro x-ray fluorescence. We also have a traceable aluminum/copper standard for EDS eV calibration.
- **Ion Sputtering Standards:** for ion sputter rate calibration. Thin films of Si₃N₄ and SiO₂ on Si and Ta₂O₅ on Ta. Various thicknesses.

Scanning electron microscopes can image using the secondary (in-lens or conventional) or backscattered electron signals. For SEM applications we recommend ordering the device with the electrically conductive coating for two reasons. First, it allows for imaging at any accelerating voltage. Second, the coating is removable. When it is removed electron beam tracks from contamination go with it. We provide this cleaning service for a nominal fee. Cleaning is included at no charge with the suggested yearly recertification program. To avoid pattern damage use <10nA electron beam current.

ASTM E766-98: STANDARD PRACTICE FOR CALIBRATING THE MAGNIFICATION OF A SCANNING ELECTRON MICROSCOPE

The American Society for Testing and Materials has published the above standard. The copyrighted text is available from ASTM. A convenient way of obtaining E766-98 (or a later version) is online from the ASTM web site at: <http://www.astm.org>. This excellent standard offers terms, definitions and guidance to calibrate your instrument's magnification. Using the standard you should be able to calibrate to better than 5% precision in the magnification range from 10X to 50,000X. In our opinion, it is reasonable to extend the range to 200,000X using the MRS-4.2 since there was no standard with 1/2 μ m pitch when it was updated in 1998. Another SEM magnification standard is also under preparation by ISO Technical Committee 202 on microbeam analysis. The scope of E766-98 states "This practice covers general procedures necessary for the calibration of magnification of scanning electron microscopes. The relationship between the magnification and indicated magnification is a complicated function of operation conditions. Therefore, this practice must be applied to each set of standard operating conditions to be used." ASTM E766-98 describes several factors for successful calibration. These include what one should consider in a calibration specimen, a procedure for adjusting the SEM settings, how to calculate the magnification accuracy (on the "micron" marker, display CRT, digital image and final print) and how often it should be calculated, measurement of precision and bias, application of magnification to the measurement of sample detail and an annex on parameters that influence the resultant magnification.

E1951-98 STANDARD GUIDE FOR CALIBRATING RETICLES AND LIGHT MICROSCOPE MAGNIFICATIONS

This guide covers methods for calculating and calibrating light microscope magnifications, photographic magnifications, video monitor magnifications, grain size comparison reticles, and other measuring reticles. Reflected light microscopes are used to characterize material microstructures. Many materials engineering decisions may be based on qualitative and quantitative analyses of a microstructure. It is essential that microscope magnifications and reticle dimensions be accurate. The calibration using these methods is only as precise as the measuring devices used. It is recommended that the stage micrometer or scale used in the calibration should be traceable to the National Institute of Standards and Technology (NIST) or a similar organization.

Please note: our measurements are made by a special SEM where each individual pattern is measured. Other CRMs may calibrate using optical diffraction. This technique averages over large patterned areas, it doesn't measure individual pitches- like your microscope.

- **Counter-Rota-Cutter™:** pat. #4949605) for low speed diamond saws (Buehler ISOMET™, LECO VC-50™ and Struer's Minitom™) allowing faster cutting, with smoother and thinner sections and less deformation
- **Vacu-Storr™:** vacuum desiccators with holding times up to 5 years!. Developed to protect our analytical standards for storage and shipping. Various sizes are available.

COMPUTER CONTROL SYSTEMS

- **Dpict7:** PC based digital imaging for most SEMs. Active scan generator. Collect up to 9 images simultaneously. Industry compatible formats.
- **dQuant7:** Quantitative electron probe microanalysis program for WDS and EDS analysis.
- **dSpec7:** Wavelength spectrometer, stage control, and beam current measurements for electron probes.
- **Auger-II:** PC based digital control for Auger electron data collection and reduction as well as Auger imaging.

ANALYTICAL SERVICES

- **Scanning electron microscopy:** JEOL 6300, with SDD light element x-ray detection, digital imaging and particle size counting.
- **Electron Probe microanalysis:** JEOL JXA-8600 (fully automated) with 4 WDS and EDS.
- **Auger electron spectroscopy:** JEOL JAMP-7800 (fully automated).
- **Optical microscopy, profilometry, metallography, microhardness +...**