MRS-3

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 50,000X STANDARD -

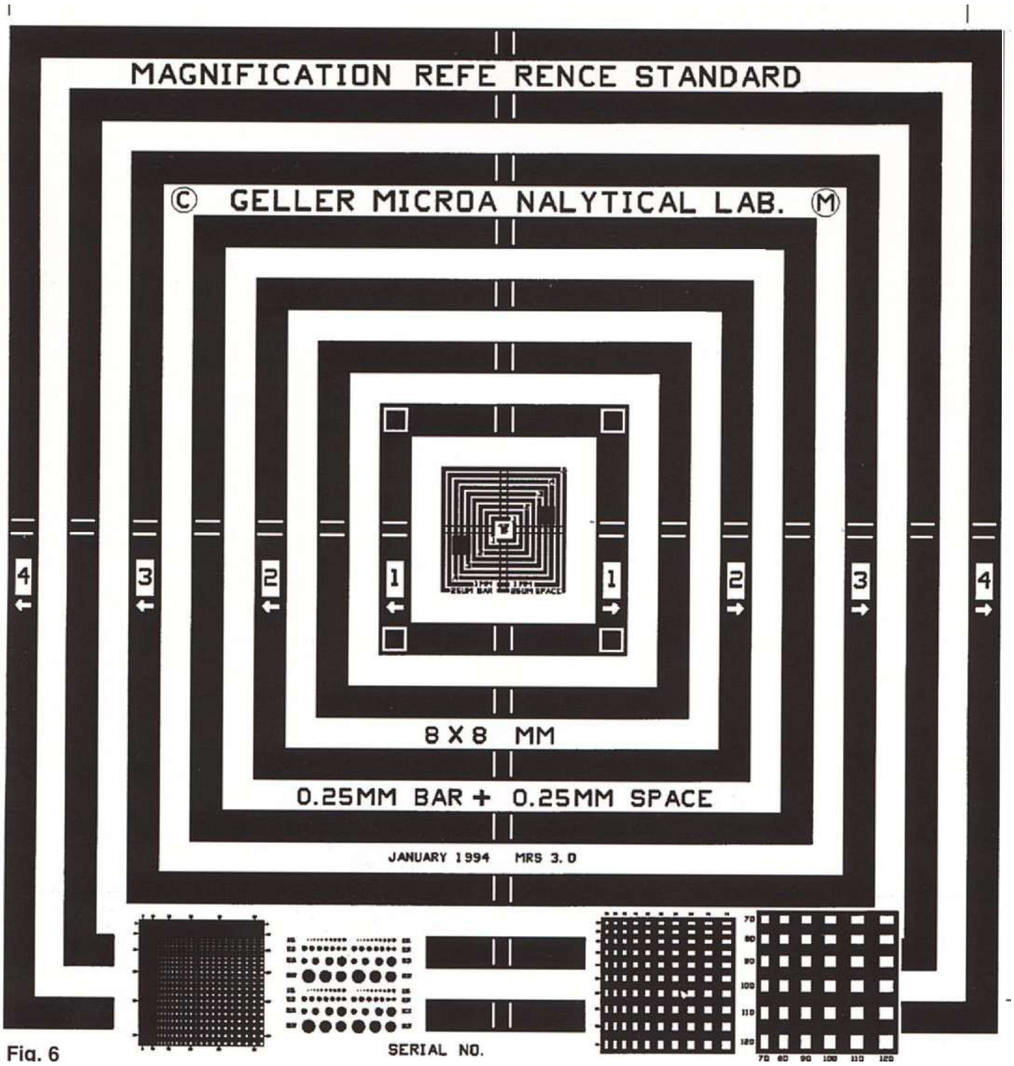


Fig. 6

Resource Guide

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

MRS-3

This is our second generation, NIST and NPL (NIST counterpart in the U.K.) Traceable, Magnification Reference Standard & Stage Micrometer. For Instrument Calibration from 10X – 50,000A (2µm minimum pitch).

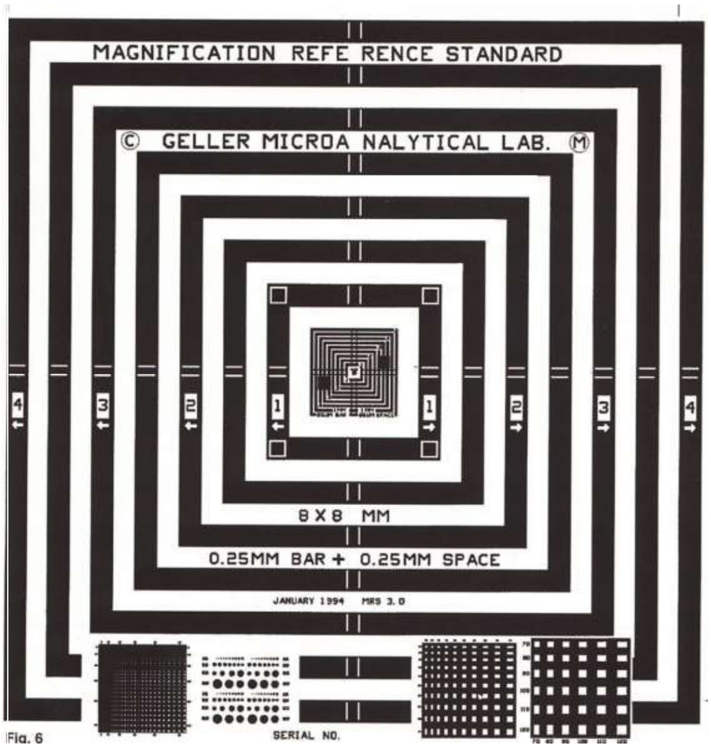


Fig. 6

Applications:

- Electron Microscopy: SEM (secondary & backscattered electrons), TEM (for use with a bulk holder- the MRS-3 can be size altered to 3mm ∅ X ½ mm thick). Call for other sizes.
- Scanning Microscopies and Profilometry: STM, AFM, stylus and optical etc. The pattern height is 0.1µm.
- 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 0.1µm CrO_2 over quartz.
- Particle Size Counting: series of circles, squares & rectangles for calibration confirmation.

INTRODUCTION

Geller MicroÅnalytical Laboratory introduces the MRS-3, our second generation magnification calibration standard (the MRS-4 with a ½µm pitch is also available). Our MRS series of calibration standards are highly accepted pitch standards, with more than 1,000 delivered. We offer them as a certified reference material (a traceable standard) or, optionally, without traceability. We also offer a cleaning service and a recertification program, as required by international quality standards such as the ISO and QS-9000 and ISO 17025.

PATTERN DESIGN

The MRS-3 is fabricated by using the highest accuracy electron direct write semiconductor manufacturing equipment available today. The pattern is anti reflective chromium (30nm of CrO_2 over 70nm of Cr) over quartz. Imaging contrast in both secondary and backscattered electron mode is very high. The overall size is ≅9mm X 9mm X 2.3 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 ∅ X ½ mm thick – or another custom size. For applications requiring an electrically conductive sample (SEM at ≅1keV and higher), the MRS-3 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 restores the MRS-3 to like new condition.

The geometric design of the MRS-3 has groups of nested squares spanning several orders of magnitude with pitches of 2µm, 50µm and 500µm. 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", Nyysonen & 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 and in the figure captions of this resource guide.

Square boxes are used for measuring magnification simultaneously in the X and Y directions. This gives a measure of image skew, barreling, pincushion and other non-linearities which can have various origins, such as from stray magnetic fields.

The largest pattern has an overall dimension of 8mm square. It contains lines and spaces that are nominally 250µm wide (totaling 500µm pitch). This can be used to check magnifications from 10X – 100X. The 50µm pitch patterns are useful from 100X – 1000X. The 2µm pitch patterns will allow calibrations up to 50,000X.

Included are squares and rectangles from 1 – 31µm in 1µm increments, 30 – 75µm in 5µm increments, and 70 to 120µm in 10µm increments. Also included is an array of 4 repeating patterns of circles with diameters ranging from 2 – 39µm in 2µm increments and 40 – 100µm in 10µm increments. These patterns are useful for checking the performance and setup of particle size counting systems. From our experience it is very difficult 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-3's have been measured by the National Physical Laboratory in the U.K. (the NIST counterpart in the United Kingdom). The uncertainties we state for the 500 µm pitch pattern is ±0.25µm, for the 50µm pitch pattern it is 0.104 µm and for the 2µm pitch pattern it is 0.031µm. It goes without saying that these reported results from NPL combine with our uncertainties are highly conservative. Our customers have also sent MRS-3's to NIST. The NIST data shows better accuracy than NPL reports.

With the even numbered pitch patterns on the MRS-3 magnifications can usually be determined without the use of a calculator. For instance, if the length of five 2µm pitches are measured over a distance of 49mm on a micrograph or display screen the magnification would be 49mm ÷ 0.01mm = 4900X. **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, not individual pitches- like your microscope.

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.

ANATOMY OF THE MRS-3

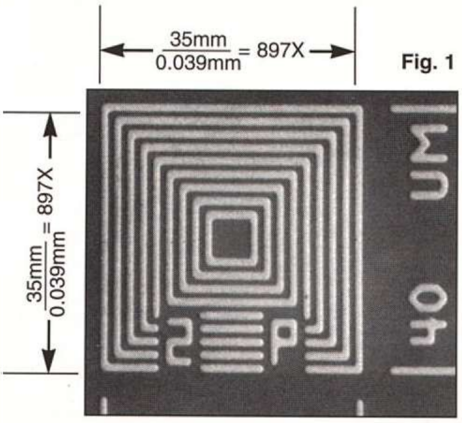


Fig. 1

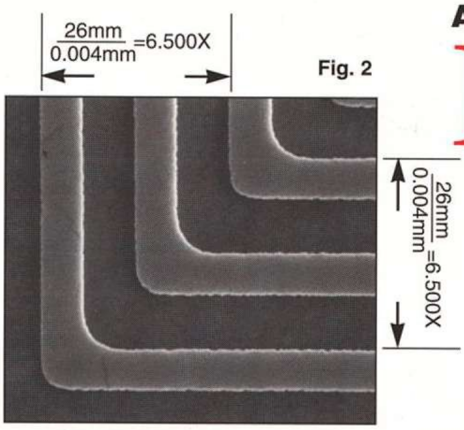


Fig. 2

Fig. 3

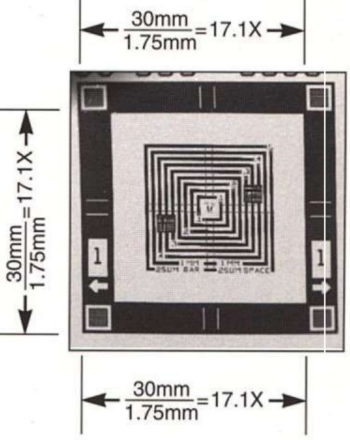


Fig. 4

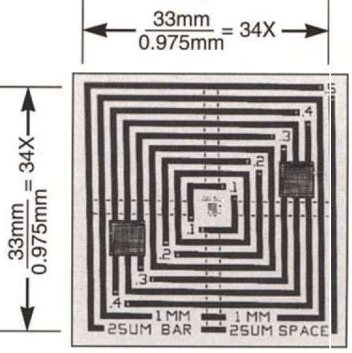
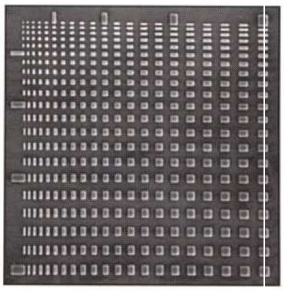


Fig. 5



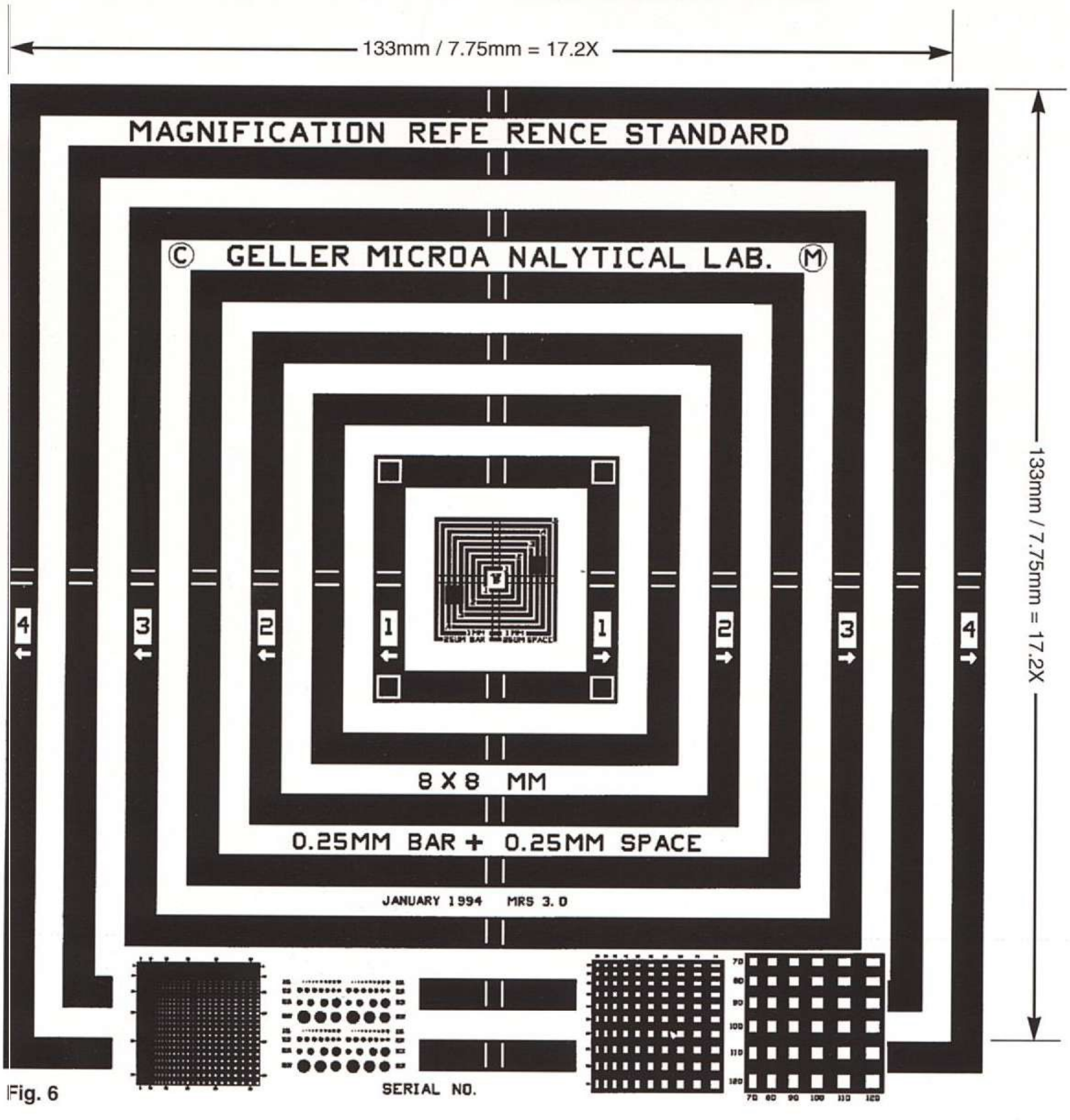


Fig. 6

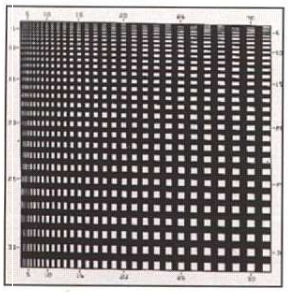


Fig. 7

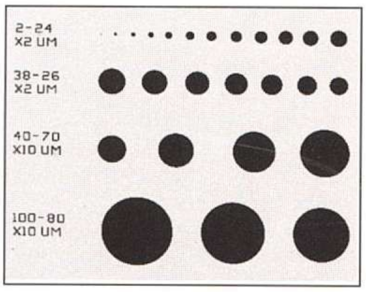


Fig. 8

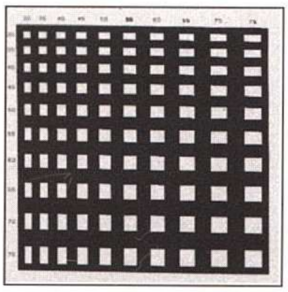


Fig. 9

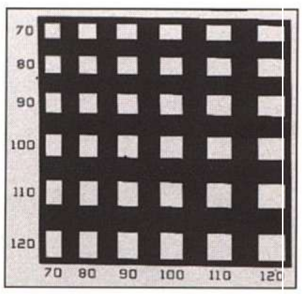


Fig. 10

Magnification Determination

- Magnification shall 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. See examples below.

Fig. 1: The smallest pattern has a 2µm pitch. The pitch dimension of the largest box is 39µm. Starting at the left side with a light-to-dark transition we measure across to the last light-to-dark transition. Our magnification is 35mm ÷ 0.039mm = 897X. (SEM, 1kV).
Fig. 2: At high magnification we image the line and space structure. Two cycles of the horizontal 2µm pitch measures 26mm ÷ 0.004mm = 6,500X. (SEM, 10kV).
Fig. 3: The 6 CRT measurement patterns are visible (see arrows). The magnification of this image is 30mm ÷ 1.75mm = 17.1X.
+**Fig. 4**: Using the 50µm pitch the magnification is 33mm ÷0.975mm = 34X.
Fig. 5: This is one of the 6 CRT measurement patterns. Squares (going from top left to bottom right) rangingfrom 1 to 5µm in 0.2µm steps.
Fig. 6: Magnification of the whole pattern width 133mm ÷ 7.75mm = 17.2X.
Fig. 7: Array of squares and rectangles from 1- 31µm in 1µm steps.
Fig. 8: One of 4 identical circular patterns from 2 - 38µ in 2µm steps, and 40 - 100µm in 10µm steps. A particle size distribution should show a flat distribution of either 1 or 4 particles (depending upon the number of patterns).
Fig. 9: Array of squares and rectangles from 30 - 75µm in 5µm steps.
Fig. 10: Array of squares and rectangles from 70 - 120µm in 10µm steps.

RETAINERS

The MRS-3 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-3 to meet your needs. The following retainers and holders are available:

- 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-3 is secured with ultra high vacuum compatible silver epoxy.
- 3mm ϕ - modify the MRS-3 to 3mm ϕ X 0.5 mm thick.
- Pin Stub – commonly used SEM stubs have a 1/8" pin and ½" top surface.
- Others– Hitachi, VG PHI, special sizes, 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 MicroÅnalytical 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-3 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 directly measured by NPL (National Physics Laboratory), the NIST counterpart in the U.K. and is traceable to NIST. 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 requirements as it relates to ISO-9000, QS-9000 or ISO-17025. Re-certification 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 hostile environment. Re-certification 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-3 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. For **electron microscopes** use any kV @ <10^{-7}A current.

ASTM E766–98: STANDARD PRACTICE FOR CALIBRATING THE MAG. 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 since there was no standard with ½µm 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 be traceable to the National Institute of Standards and Technology (NIST) or a similar organization.