Desktop SEM Calibration Standard

Product No. 601

DESCRIPTION:

Combined standard for resolution, magnification calibration, and EDS performance monitoring. Standards have engraved labels and are mounted in plane for ease of use. The 25mm diameter by 5mm tall aluminum cylinder mount has an M4 threaded base that comes with a removable pin adapter (#**15319**) allowing the standard to be used on systems that utilize pin, cylinder, or M4 threaded mounts.

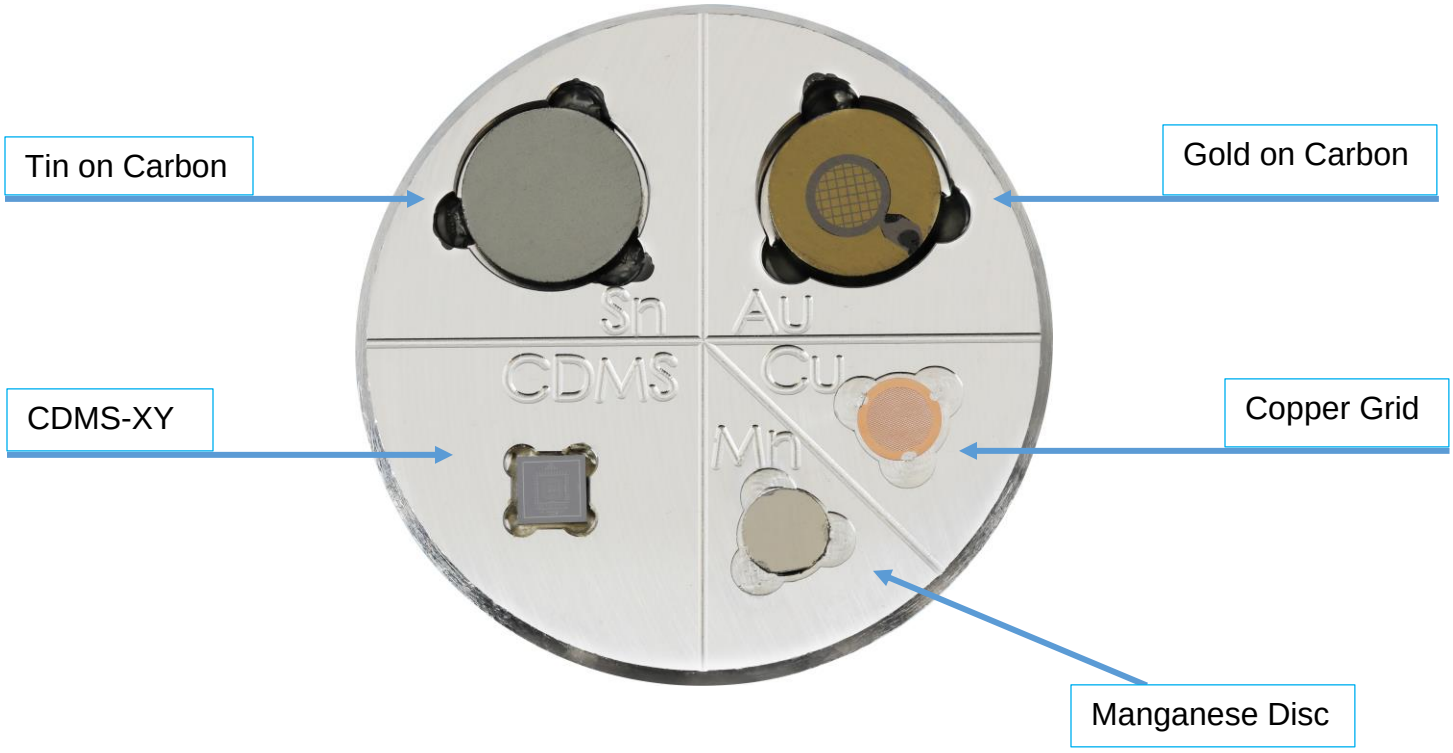

Figure 1: SEM Calibration Standard with Labels

CLEANING AND HANDLING:

WARNING! Avoid mechanical abrasion of the surface such as touching or wiping with a cloth.

Remove dust and large particles with dry filtered nitrogen gas. A photographic lens quality “dust-off” product may be used as an alternative. Avoid gases that leave residues.

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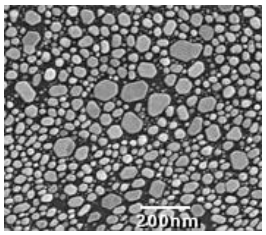
SEM RESOLUTION TEST SPECIMENS:

Gold on Carbon 1



15.0kV x100K 200nm Particle size range: 5-150nm

Figure 2: Resolution Test Specimen, Gold on Carbon 1

Gold on Carbon Test Specimen Product No 617 is excellent for SEM resolution testing. The gold particles, with superior feature details, generate high secondary and/or backscatter electron signals with excellent contrast against the carbon background.

The Resolution Test Specimen, Gold on Carbon 1 has a square grid pattern with large crystals in the center of each square and very fine crystals at the edges of each grid (See *Figure 2*). Thus, medium and high resolution gap tests are performed on the same specimen. The particle sizes range from approximately 5nm to 150nm, with the larger crystals showing facets which allow assessment of the gray level reproduction available at high resolution.

A suggested imaging strategy for using this specimen is to first, start at a low magnification (below 150x), focus on the edges of the dark grid bars and search for a suitable square of gold. Raise the magnification to 500x and focus on a heterogeneous portion of the gold film. Increase the magnification to 40,000x, keeping the specimen in focus. Shift the specimen to image an area well within a grid square (if the stage is tilted, use X-shift for convenience so that you don't lose focus); now focus carefully on the gold particles. Raise the magnification to 80,000x (or above) and make final adjustments to the stigmators and the fine-focus controls. The latter adjustments should be performed quickly if the vacuum is contaminated in your SEM, since a heavy layer of contamination can be deposited even within a minute. Record the image using a very slow probe scanning rate.

See the full PELCO® Technical Notes for Gold on Carbon SEM Resolution Test Specimens for further information.

Tin Spheres on Carbon (Low Magnification)

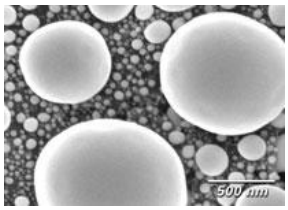


Figure 3: Tin Spheres on Carbon

Tin Spheres on Carbon Product No. 600 are used similarly to the Gold on Carbon standard described above, but are very useful for SEM astigmatism correction due to their nearly perfect round shape. The round tin spheres are also particularly useful for testing image quality, distortion, contrast, brightness, and probe size. Sphere sizes range from 1-10µm, making this standard suitable for desktop SEMs and low magnification (250-5000 X) testing.

CRITICAL DIMENSION MAGNIFICATION STANDARD CDMS-XY:

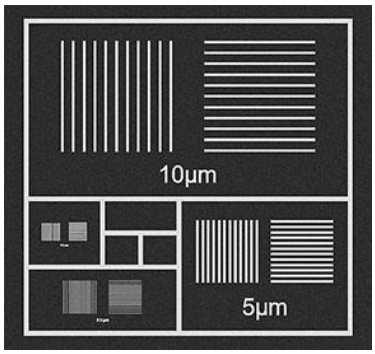


Figure 4: Pelcotec™ CDMS-XY-1T

The Pelcotec™ CDMS-XY-1T is NIST traceable[1] (uses average data measured for each production wafer) with features from 2mm to 1µm for magnification 10x-20,000x, ideal for desktop SEM. The feature sizes included are: 2mm, 1mm, 0.5mm, 0.1mm, 50µm, 10µm, 5µm, 2µm, and 1µm.

To maintain optimum planarity, the patterns are fabricated on ultra-flat silicon wafers. Calibration features between 2.0mm and 5µm were patterned using photolithography and are 50nm thick chrome. Using typical imaging conditions in an SEM, the perimeter of each line produces a brightness that allows for easy edge detection and measurement center-to-center distance.

The 2µm, and 1µm calibration features were patterned using e-beam lithography and consist of a 20nm thick chrome base with 50nm thick gold on top. These features produce a high contrast image against the silicon substrate, again allowing for easy measurement of center-to-center distance.

The zeros in the fiducial identifiers as well as the alignment cross-hairs make excellent features for focus and stigmation. As always, for optimum results ensure that the instrument is aligned with a stable beam current. Use the largest number of pixels for image capture and a slow enough scan rate to achieve a good signal-to-noise ratio. By performing both short- and long-term repeat measurements the user will be able to evaluate instrumental reproducibility and stability.

The design of the standard allows instrument calibration up to three orders of magnitude. Starting with the largest dimension that can be imaged (e.g. 2.0mm) and doubling the magnification brings the next calibration marker into view. Continuing this process until the 10 µm features are imaged, the user can now move in a clockwise spiral to the next smallest calibration feature until a complete magnification calibration curve is obtained over the range of interest.

Less or no use of the calibration lines to focus on the critical dimension (CD) in a SEM will minimize the build-up of contamination on the surface and increase the periods between cleaning. When it becomes necessary to clean the CDMS standard, see *CLEANING AND HANDLING, page 1*.

We do not place an end date on the certification. The CDMS standards are constructed of Si, Cr, and Au and so are considered chemically stable under normal conditions of use. These standards are not intended to be mechanically touched. The only deterioration is caused by contamination. When used under clean conditions, we expect the validity of the certification to last between 5-10 years. The recertification frequency should be determined by the end-user taking into account the frequency of use, handling, storage and the condition of the standard over time or if a catastrophic event occurs like scratching the standard. Recertification can only be effected by purchasing a new certified standard.

[1] For wafer level traceability of the CDMS-1T the measurements for 10 certified standards across each wafer are used to obtain the statistics given on the traceability certificate.

EDS SYSTEM MONITORING – MANGANESE AND COPPER SAMPLES:

The following tests can be performed with the Mn and Cu Standards:

1. Spectrum calibration with aluminum and copper
2. Manganese (in Mn88/Ni12 alloy) full width at half max (FWHM) resolution
3. Low magnification imaging calibration

Spectrum Calibration:

Most EDS systems can be calibrated on the aluminum Kα and copper Kα peaks (1.49 keV and 8.04 keV respectively). To perform a spectrum calibration using the Copper Grid (#1GC400), set the SEM to its lowest magnification and orient the grid under the beam so it fills ¾ of the screen and the remainder with the aluminum (avoiding silver paint as much as possible). The accelerating voltage should be at 20 kV and the scan speed at TV or rapid scan. Adjust the condenser lens (spot size) and final aperture to achieve a count rate of about 1000 X-ray counts per second. On the spectrometer, adjust the ratio of aluminum and copper on the SEM screen, if necessary, by moving the stage or changing the magnification so the aluminum and copper peaks are about the same height.

The copper Lα and Kα lines (0.93 and 8.04 keV) should also provide a good full-scale calibration for general use. The calibration should be performed at 10 kV. Once you have obtained a good spectrum with this procedure, follow the calibration instructions for your EDS system as outlined by the manufacturer.

Resolution:

The resolution of your EDS system should be checked periodically to ensure correct operation of your detector and electronics. This is easily accomplished using the manganese standard by measuring the full width at half max (FWHM) of the manganese Kα peak.

To measure FWHM, orient the manganese standard under the beam and set the magnification to 100X. Set the accelerating voltage to 15 kV for manganese. Adjust the condenser lens (spot size) and final aperture to get a count rate of about 1000 counts per second (count rate for a pure element). If your EDS system has an adjustment to vary the signal processing time constant, select the setting with the longest time constant to provide the best resolution of the spectrometer. Acquire a spectrum until there are at least 2000 counts in the centroid (middle) channel of the manganese peak.

Determine the vertical midpoint of the peak - this is the half max. Measure the width of the peak at half max - this is the full width at half max (see *Figure 5*). Record this value in the *Table 1: Resolution Table*, page 7. Resolution should be checked periodically and this table will provide a handy record of your detector performance over time. It is recommended that you check the resolution of your system every six months.

Many factors can cause a degradation of resolution. Small variations in resolution (10 eV or less) can result from statistical error. A small one-time variation in resolution probably does not indicate a problem, but a steady degradation over time is a sure sign of a detector or system problem. If your measurements show an increase in FWHM over time, you should notify the EDS system manufacturer.

NOTE: It is an accepted practice in the EDS industry to specify the resolution of X-ray detectors using radioactive Fe-55 source, which has a photon emission close to the same energy as the manganese Kα peak. Measurements taken with the manganese sample may vary slightly from those made with an Fe-55 source due to background radiation (Bremsstrahlung) X-rays produced in the bulk sample. Remember that this sample is used to detect changes in resolution overtime and is not a substitute for an Fe-55 source. However, if the background is subtracted from the manganese peak before the FWHM measurement is made, the resolution should be very

close to that measured with an Fe-55 source. This manganese sample contains about 12% nickel, so it is normal to see small nickel peaks in your spectrum.

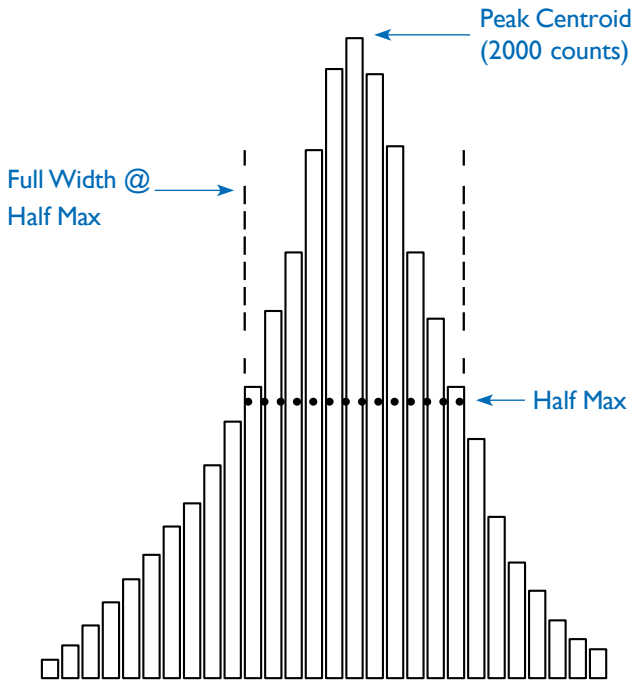


Figure 5: FWHM Peak Determination

Sensitivity:

Detector windows can become coated with vacuum pump oil or other contaminants that can attenuate the incoming X-ray signal. This is especially serious when analyzing low energy X-rays. Unless you monitor your system's sensitivity on a regular basis, you may not be aware that your window is dirty and you are losing some of your signal. There are several ways to monitor system sensitivity but the simplest is to perform a peak ratio measurement using a high and low energy peak.

The most common test is performed using the manganese standard. Since this standard contains about 12% nickel, you can ratio the manganese Kα to the nickel Lα peak. As the detector window gets dirty, the manganese K peak will appear larger relative to the nickel Lα peak because the nickel L X-rays will become increasingly absorbed by the contamination on the window. If you have a windowless or thin window detector, you may notice a small oxygen peak; this is normal.

Set the accelerating voltage to 10 kV and put the beam in the center of the manganese standard. Adjust the condenser lens (spot size) to achieve a count rate of about 1000 counts per second (count rate for a pure element) and acquire a spectrum for 100 seconds. If you need to tilt the sample, make sure to record the tilt angle since this test must be performed at the same tilt angle each time. Using your system software or by manually measuring the peak heights, calculate the peak ratio of the manganese Kα peak to the nickel Lα peak. Record this value in the *Table 2: Sensitivity Table*, page *7*.

NOTE: For beryllium window detectors, the peak ratio will be about 2:1. For ultra-thin window detectors, the ratio will be closer to 1:1. It is critical that you determine the sensitivity of your detector and use that value as a benchmark for your system. The values quoted here are only examples and your ratio value may vary considerably. All detectors and windows are slightly different and sensitivity values in the form of peak ratios

can vary from one system to another. It is best to perform the benchmark measurement when the detector is new or right after the window has been cleaned. This way you will be able to measure future performance against an optimum condition. Detectors already in service may already have some contamination on the window, usually vacuum pump oil.

DO NOT ATTEMPT TO CLEAN YOUR DETECTOR WINDOW WITHOUT FIRST CONTACTING THE MANUFACTURER.

This test is not designed to measure absolute sensitivity of your detector, but rather a degradation of sensitivity over time. For this reason, it is important to check sensitivity at least every six months. This test should be performed whenever you check the FWHM resolution since both tests are designed to show changes in system performance over time.

Image Calibration:

Many EDS systems come with image analysis software to measure particle size distributions and perform other size measurements. These software packages should be checked to ensure they are reporting accurate data.

Figure 6: 400 mesh grid
Grid opening = 38 x 38µm. Average grid area = 1, 444µm^2+/-5% (2σ)

The Desktop SEM Calibration Standard comes with a Copper Grid (#1GC400) grid that can be used to check the length and magnification calibration of your image analysis software. This is a 400 mesh grid for low magnification calibration and provides a pattern shown in *Figure 6*.

To perform a size check, place the grid under the beam, and acquire an image in your EDS software. Then use your image analysis software to measure the grid pattern and calculate the average grid opening area. You can do this at various magnifications, but it is a good idea to perform this test at or near the magnification with which you intend to work. Perform measurements in both X and Y directions. Any discrepancy with the micron marker on your SEM should be checked with a standard recommended by the SEM manufacturer. Some grids may have an arrow in the center of the grid; this arrow should not be used in your measurements. As there is some variation in grid opening size, make sure you include at least 30 grid openings in your measurement.

NOTE: This test is designed to see if your image processing software is working properly. It is not intended as an absolute test of the accuracy of the measurement displayed on the SEM. Your SEM manufacturer can recommend a high precision standard you can use to verify the performance of your microscope.

REFERENCES:

RESOLUTION

Date	eV

Table 1: Resolution Table

SENSITIVITY

Date	Mn-K / Ni-L	Tilt

Table 2: Sensitivity Table

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