SPI NiOX™ Test Specimen

Use Instructions

The SPI Supplies NiOX™ Test Specimen consists of a uniform thin film of nickel oxide on a molybdenum support grid. It can be used for the following six test protocols. Please let us know if you think of more!

1. To test for stray electrons and x-rays in a TEM column, measure the Ni $_{K}\alpha$/Mo$_{K}$ α count ratio with the electron beam near the center of the test specimen and in the center of a grid square. Typical values for this ratio are in the range of 3 to 7 for modern TEMs. *This test should not be performed if you use a molybdenum condenser aperture.*

2. To test for the total contribution of the instrument to the EDS spectrum background, measure the total integrated counts, P (above background), in the Ni$_{K}\alpha$ peak and the flat background B_{500} integrated over 500 eV (the average of the two regions on either side of the peak). The peak/background ratio is P/B = 50 x P/B_{500}. For 100 to 200 kV accelerating voltage and probe diameters in the range 20 to 200 nm, P/B should be at least 1000, and more modern instruments may give a value closer to 3000.

3. Measurement of the full width at half maximum of the Ni$_{K}\alpha$ peak {FWHM (Ni)} allows the energy resolution of the EDS detector using Mn$_{K}\alpha$ radiation to be estimated as FWHM(Mn) ≈ 0.926 x FWHM(Ni). For a light-element detector, FWHM(O) should be lower by a factor of 1.6. Thereference for the assumption of the 0.926 factor is Bennett and Egerton, J. Microsc. Soc. Amer. 1 (1995) 143-150. It is based on O$_{K}$ and Ni$_{K}$FWHM's measured on both Si(Li) and Ge detectors.

4. The collection solid angle of the EDS detector (in sterad) is given by omega = 4.05 x P/(t x τ x I), where P is the characteristic Ni$_{K}\alpha$ counts recorded in a live time of τ seconds with a probe current of I picoamperes, measured using a picoammeter connected to the TEM screen, to a Faraday cup or if to a spectrometer made by Gatan, Inc., to the flight tube and t (≈ 50) is the thickness in nm of the NiO film.

5. For windowless and thin-window EDS detectors, the O$_{K}\alpha$ /Ni$_{K}\alpha$ count ratio is typically 0.2. The theoretical value is 0.73. If the measured ratio decreases with time, check for a buildup of ice or hydrocarbon on the detector or on the window.

6. For E-axis calibration in electron energy loss spectroscopy, the $O_{K\ edge}$ should appear at 533 eV and the Ni_{Ledge} at 854 eV if they are measured at the point of maximum slope. A measurement of the areas under these two edges provides a test of the EELS quantification procedure.