TECHNICAL IMPLEMENTING PROCEDURE

TIP-CM-2

Operator Calibration of Scanning Transmission Electron Microscopes

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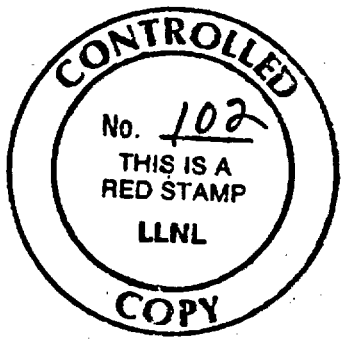

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OPERATOR CALIBRATION OF SCANNING TRANSMISSION ELECTRON MICROSCOPES.

1.0 PURPOSE

This procedure describes requirements necessary for operator calibration of Scanning Transmission Electron Microscopes (STEM's or TEM's) in support of the Yucca Mountain Project (YMP). These requirements are established to assure that TEM calibrations are documented, and that measurements made on TEM images and diffraction patterns are accurate.

2.0 SCOPE

This procedure applies to operator calibration of transmission electron microscopes for scientific investigations for the Yucca Mountain Project . It applies to measurements made using photographic images obtained using a Scanning Transmission Electron Microscope. Measurements are made on photographic positive prints made from negatives which are produced by the electron microscope. Calibrations are made in accordance with well established practices, descriptions of which appear in most practical texts on transmission electron microscopy.

Magnification and diffraction standards are available commercially and may be used provided that they are traceable to the National Institute for Standards and Technology (formerly the National Bureau of Standards) or other nationally recognized standards.

3.0 RESPONSIBILITIES AND AUTHORITIES

The Task Leader (TL) whose activities warrant the use of this procedure is responsible for implementing the requirements of this procedure. The Task Leader may delegate this responsibility and authority to a Principal Investigator.

The individual calibrating the STEM and making the measurements is responsible for following the requirements of this procedure, documenting calibrations and assuring that the latest revision of this document is followed.

The YMP Quality Assurance Manager (QA Manager) is responsible for monitoring work to assure proper implementation of this procedure and for assuring its continued effectiveness.

4.0 CONTROLS

Controls are established by written procedures or instructions prepared in accordance with procedure 033-YMP-QP 5.0, "Technical Implementing Procedures." Procedures are issued in accordance with procedure 033-YMP-QP 6.0, "Document Control."

4.1 Selection

The Task Leader, or his designate, will ensure that the STEM's used are of the proper type, design, range, accuracy, and tolerance to accomplish their required function.

4.2 Identification

The STEM's are identified by using unique identification numbers (DOE Nos.). The identification number is recorded on data sheets, laboratory note books, logs, etc., along with the measurements taken to assure traceability to the electron microscope used to make the measurement.

4.3 Calibration

The standards used for calibration of the STEM are traceable to the National Institute for Standards and Technology (formally the National Bureau of Standards) or other nationally recognized standards. Traceability requires the ability to relate individual measurement results through an unbroken chain back to NIST or other nationally recognized standard. The chain of calibration must be documented and auditable.

5.0 PROCEDURE

This procedure utilizes calibration with shadowcast carbon or silicon monoxide replicas of diffraction line gratings as well as negatively stained catalase crystals and graphitized carbon black. The diffraction grating replicas provide parallel line gratings with 2160 lines/mm (54,864 lines/inch). The line spacing for such a grating is 463 nm allowing magnifications to be

established in the range 1000X to 100,000X. Catalase crystals contain lattice plane spacings of 8.75 nm and 6.85 nm and are useful for establishing magnifications between 40,000X and 300,000X. Carbon black contains the 0.34 nm lattice spacing and can be used to calibrate magnifications greater than 300,000X. Diffraction standards of evaporated films of very fine grained polycrystalline aluminum, gold or thallous chloride allow determination of the camera constant of the electron microscope and so enable identifications of unknown crystalline materials to be made.

5.1 Calibration

Bring the electron microscope into operation in accordance with the procedures specified by the manufacturer. Select the appropiate magnification or diffraction standard, place it into the specimen holder and insert it into the microscope column. Saturate the filament and select an area for imaging.

Note 1 -- For the JEM 200CX scanning transmission electron microscope, DOE No. 3660933, the operating procedures recommended by the manufacturer are located in the room with the instrument. The document is entitled "Instructions JEOL, JEM-200CX Electron Microscope, No. 1EM 200CX (EM132001), JEOL Ltd., Tokyo, Japan" and Chapter 5, pages 5-1 to 5-76, defines the operating procedures.

5.1.1

Microscope calibrations will normally be done using an untilted specimen at the eucentric position of the column. This will insure that a common reference point and reference orientation are used during calibration events and any inconsistencies or changes will be evident and meaningful. Since the eucentric position is fixed, the current in the objective lens needed to focus upon an object placed at the eucentric position will not vary unless the electron microscope is malfunctioning or exhibiting instability. Significant variations can then be related to loss or change of calibration.

In accordance with the manufacturer's instructions for tilt axis alignment, using the tilt control(s) and the Z-axis control, adjust the vertical location of the thin specimen so that the tilt axes or axis lie(s) in the specimen plane. When this is accomplished, tilting the specimen will produce minimum image shift for an arbitrary chosen object at the center of the final image screen -- only foreshortening of features at the edges of the screen will occur. This is the eucentric position. Set the tilt angle to 0 (zero).

Note 2 -- In a situation where calibration documentation is required for other specimen configurations, the relevant microscope operating parameters (tilt settings, objective and intermediate lens currents) should be recorded for the unknown and duplicated with the calibration standard in place.

5.1.2

If magnification calibration is being performed, select various magnification settings in the range for which the standard is suitable. At each magnification, focus the image and expose the photographic film or plate. Record the objective lens reading for each of the exposures.

5.1.3

If camera length calibration is being performed with a diffraction standard in place, follow the manufacturer's procedure for selected area electron diffraction. Select the camera length to be calibrated, focus the selected area diffraction pattern, defocus the condenser lens, insert the beam stop to mask the direct beam and expose the photographic film or plate. Record the objective lens reading.

Note 3 -- While the single diffraction pattern taken from the standard specimen at the eucentric position is sufficient to calibrate the microscope, it is good practice to obtain additional selected area diffraction patterns, while the opportunity presents itself, at the upper and lower extremes of the vertical movement of the specimen that the Z-axis control allows. This provides infomation, very often helpful during diffraction pattern analyses of unknowns, as to what variation of camera length exists.

5.1.4

Develop the photographic negatives and make prints on which measurements can be made. In most cases contact prints will provide suitable magnification that fringes, lattice planes or diffraction rings can easily be measured and no additional magnification step is involved in the calculations. If enlargements are used then another image (of a calibration standard for which contact printing has been successfully used) must be printed at the same enlargement in order to provide the darkroom magnification factor.

Note 4 – Although it is possible to take the measurements directly from the glass or film negatives, it is not advisable to do so because of the fragile natures of these media. Prints on the other hand can easily be replaced as long as the original negatives remain intact.

5.1.5

For magnification calibration, measure the grid or lattice spacings on each of the images and determine the image magnifications from these spacings. Tabulate the results, showing the nominal magnification which is printed on the negative at the time of exposure, the calibrated magnification and the objective lens meter reading noted during image exposure.

5.1.6

For calibration of the camera constant follow the established procedure given in textbooks on electron microscopy. Measure the diffraction rings along a line which passes through the center of the pattern. Using the interplanar spacings supplied by the manufacturer of the diffraction standard, convert these measurements into camera constants through the relationship

$\lambda L = rd$ (the product of the measured ring radius and the corresponding interplanar spacing of the diffracting planes).

Tabulate the results, showing the Miller indices of the diffraction rings and the camera constants determined for each ring. Since the camera constant normally varies slightly with distance from the center of a diffraction pattern and also with the azimuthal angle, it is probably best to leave the calibrated data in the tabular form, thereby demonstrating the spread of results. A single number camera constant can be obtained, if necessary, by averaging the data or performing a least-squares analysis.

5.2 Establishing Traceability of Standards

5.2.1

Carefully place a grating replica TEM magnification standard on top of an NBS Scanning Electron Microscope Magnification Standard and insert into an SEM, using an edge of cellophane tape to keep the grating from moving. Select an operating voltage of 5 KeV to ensure sufficient contrast of the replica features. At an image magnification of 10,000X

select lines on the NBS standard which have separations of 1, 2, 3, 4, and 5 micrometers (μm), then center and align these features and obtain a micrograph. Under the same conditions, move to the TEM replica standard and obtain a representative image. The spacing of the lines on the replica can now be calibrated to within 5% accuracy or better as allowed by the NBS standard.

Note 5 -- The specific NBS standard in the possession of the electron microscopy group (Materials Characterization Section, Condensed Matter and Analytical Science Division, Chemistry & Materials Science Department, Lawrence Livermore National Laboratory) is of 1978 vintage and hence is identified as an NBS, rather than an NIST, standard. It is identified as Standard Reference Material 484a, Scanning Electron Microscope Standard with Specimen identification number JY-55-LL, issued by the U.S. Department of Commerce, National Bereau of Standards, Washington, D.C. 20234.

5.2.2

Compare any other TEM magnification standard to the replica, which has been calibrated and is traceable, by obtaining TEM images of the two standards taken under the same imaging conditions, thereby obtaining traceability of the second magnification standard.

5.2.3

Having obtained traceability of a catalase crytal TEM magnification standard, perform selected area electron diffraction to produce a diffraction pattern from this standard. Measurements and calculations of interplanar spacings which are derived from this catalase pattern will then be traceable to the NBS standard. Diffraction patterns obtained from other diffraction standards taken under similar operating conditions can then be calibrated and traced to the NBS standard.

6.0 HANDLING AND STORAGE

Electron microscopy standards shall be handled and stored so as to minimize their exposure to dusty or corrosive atmospheres and mechanical shock. Normally, isolating the standard inside a gelatin capsule which is then placed in an outer container that identifies the contents is sufficient to satisfy those requirements.

7.0 RECORDS

The laboratory note book will identify the calibration status and the calibration procedure (including revisions) used to perform each calibration.

STEM calibrations will be recorded in the laboratory note book in accordance with procedure 033-YMP-QP 3.4, "Scientific Notebooks."

Documentation of NIST traceability of the standards will be maintained in a record file kept in the laboratory, with copies sent to the QA Local Records Center. STEM's are identified by make, model, serial number, and LLNL property number.