TECHNICAL IMPLEMENTING PROCEDURE

TIP-CM-4

Operator Calibration of The Optical Metallograph

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# OPERATOR CALIBRATION OF THE OPTICAL METALLOGRAPH.

## 1.0 PURPOSE

This procedure describes requirements necessary for operator calibration of an optical metallograph in support of the Yucca Mountain Project (YMP). These requirements are established to assure that metallograph calibrations are documented, and that measurements made on metallographic images are accurate.

## 2.0 SCOPE

This procedure applies to operator calibrations of optical metallographs for scientific investigations for the Yucca Mountain Project. It applies to measurements made using photographic images obtained using an optical metallograph. Measurements are made on photographic positive prints either produced directly or made from negatives produced by the metallograph. Calibrations are made in accordance with well established practices.

Standards for optical metallography 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 metallograph 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 optical metallographs used are of the proper type, design, range, accuracy, and tolerance to accomplish their required function.

### 4.2 Identification

The metallographs 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 optical metallograph used to make the measurement.

### 4.3 Calibration

The standards used for calibration of the optical metallograph are traceable to the National Institute for Standards and Technology (formally the National Bureau of 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 includes calibration with an SEM and Light Microscope Calibration Specimen made of single crystal silicon with overall dimensions of 5 x 5 mm and marked with clearly visible squares of periodicity 9.9 μm. The lines are 1.9 μm wide, formed by electron beam lithography, and a broader marking line is written every 500 μm.

### 5.1 Calibration

Place the calibration standard on the specimen stage of the microscope.

5.1.1

For a magnification in the range 10X to 1500X, select the magnichanger, the eyepiece and the objective lens to be used to obtain this magnification. At each magnification so selected, expose a photographic film.

5.1.2

For magnification calibration, use a commercial scale to measure the line spacings on each of the images and determine the image magnifications from these spacings.

$$\text{Magnification} = \frac{\text{Distance measured between image lines on the micrograph}}{\text{Calibrated distance between same number of lines on the standard}}$$

5.2 Establishing Traceability of Standards

5.2.1

Place an NBS magnification standard (Standard Reference Material 484a) in a specimen holder and insert it into a scanning electron microscope column. Select an operating voltage and bring the electron microscope into operation in accordance with the procedures specified by the manufacturer. Saturate the filament and select the area of the SRM containing the Knoop indentation and gold-filled scribed lines for imaging. Adjust apertures and stigmators for optimum resolution.

Note 1 -- For the Hitachi S-800 field-emission scanning electron microscope, DOE No. 4616427, the operating procedures recommended by the manufacturer are located in the room with the instrument. The document is entitled "Instruction Manual for Model S-800 Field Emission Scanning Electron Microscope, Part No. 580-8600-2, YR-G (FT-LT)" and Chapter 3, pages 3-1 to 3-37, defines the operating procedures.

5.2.2

Select a working distance and set the lens current for focussing at this distance. Using the Z-axis control, move the SRM into focus. Record the microscope identification number, the SRM identification number and the working distance.

5.2.3

Select magnification settings of 10X, 100X, 500X, 1,000X and 2,000X. At each magnification, focus the image (by Z-axis control) and expose the photographic film.

5.2.4

Remove the SRM calibration standard and mount the SEM and Light Microscope calibration specimen in the SEM specimen holder and insert it into the column of the electron microscope. Select the same working distance used for the SRM calibration standard and set the lens current for focussing at this distance. Using the Z-axis control, move the SEM and Light Microscope calibration specimen into focus.

5.2.5

Select the same magnification settings in the range 10 to 2,000X used for the SRM calibration standard. At each magnification, focus the image of the SEM and Light Microscope calibration specimen (by Z-axis control) and expose the photographic film.

5.2.6

A traceable path now exists, for the SEM and Light Microscope calibration specimen through the SRM calibration standard, to the National Institute for Standards and Technology.

## 6.0 HANDLING AND STORAGE

Optical microscope 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 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.

Metallograph 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 Local Records Center. Optical metallographs are identified by make, model, serial number, and LLNL property number.