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Scanning Electron Microscopy (SEM) Procedure for HE Powders on a Zeiss Sigma HD VP SEM

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Disclaimer

This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

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1.0 SCOPE & APPLICATION

This method describes the characterization of inert and HE materials by the Zeiss Sigma HD VP field emission Scanning Electron Microscope (SEM). The SEM uses an accelerated electron beam to generate high-magnification images of explosives and other materials. It is fitted with five detectors (SE, Inlens, STEM, VPSE, HDBSD) to enable imaging of the sample via different secondary electron signatures, angles, and energies.

In addition to imaging through electron detection, the microscope is also fitted with two Oxford Instrument Energy Dispersive Spectrometer (EDS) 80 mm detectors to generate elemental constituent spectra and two-dimensional maps of the material being scanned.

An IBSS plasma cleaner GV10x DS is connected to the SEM to clean the chamber periodically.

SE = secondary electron
Inlens = in-lens secondary electron
STEM = scanning transmission electron microscope
VPSE = variable pressure secondary electron
HDBSD = high definition backscattered electron detector

2.0 SUMMARY OF METHOD

HE particles are dispersed onto an aluminum standard SEM specimen mount. Electron micrographs are collected at various magnifications depending on HE particle size.

3.0 APPLICABLE SPECIFICATIONS, OTHER PUBLICATIONS

3.1 Other Publications:

3.1.1 Ted Pella Cressington Magnitron Sputter Coater, model 208HR operator's guide.

3.1.2 Zeiss Sigma HD VP SEM operator's manual.

4.0 APPARATUS AND MATERIALS

The following equipment or equivalent equipment is required to analyze samples in accordance with this method:

4.1 SEM specimen mounts: standard is 1/2 inch diameter x 1/2 inch tall, aluminum, P/N 16202 (Ted Pella).

4.2 Cressington Magnitron Sputter Coater (model 208HR) with gold, Pt, Ir source, Ted Pella.

4.3 Specimen Box, and carbon double sided adhesive tape, Ted Pella Figures 1&2.

4.4 Sigma HD VP field emission Scanning Electron Microscope (SEM), Zeiss.

4.5 MRS-3XY grid, magnification standard from Geller MicroAnalytical.

4.6 Nitrogen gas for the SEM and argon gas for the sputter coater. Both are of ultra-high purity grade.

5.0 SAMPLE DISTRIBUTION & HANDLING

Sample distribution, handling and transportation will be in accordance to the LLNL explosives safety procedures.

6.0. PROCEDURE

6.1. Sample Preparation

6.1.1. Obtain the SEM Logbook, B191/R1443 and record sample (ID) numbers and any other information about the sample and the state of the SEM in the logbook.

6.1.2. Disperse a small quantity of HE particles, <5 mg, on a standard ½-in diameter by ½-in tall aluminum SEM specimen mount. Repeat for each sample to be imaged.

6.1.3. Place the samples in the sputter coater, Ted Pella Cressington Magnitron sputter coater 208HR.

6.1.4. Put enough argon in the chamber so that the vacuum reads 0.08 mbar. Coat the sample with gold, Pt or Ir as appropriate for a specified period of time and current to obtain an acceptable coating. Recoat as necessary to achieve a good image after analyzing on the SEM. Rotating and tilting of the stage during coating may be necessary to coat the edges of the samples.

6.1.5. Transfer the samples back to the specimen box after coating.

6.2. SEM Characterization

6.2.1. A sample can be placed in the SEM chamber either by the load lock or the chamber door.

6.2.2. MRS-3XY grid, magnification standard from Geller MicroAnalytical, can be used from time to time to insure SEM calibration's validity. One can measure the calibration gridlines at various magnifications, 100-10KX, Figure 3.

6.2.3. Coated samples are placed in the Sigma HD VP Field Emission Scanning Electron Microscope (FESEM). High-resolution imaging is performed via appropriate adjustment of 1) accelerating voltage (1-5 kV), 2) probe current, 3) working distance, and 4) tilt angles until satisfactory electron micrographs are obtained. The standard operating tilt angle is 0. One may modify/manipulate these values to achieve better resolution and high quality images while minimizing damage to the sample.

6.2.4. Electron micrographs will be taken at various magnifications as appropriate and according to the needs of the sample. Care shall be taken to insure that the electron beam does not damage the sample. Slowly adjust voltage, current and working distance to minimize charging and damage to the particles.

6.2.5. Besides voltage and current, the aperture and detector type can be changed to achieve a high-resolution sample.

6.2.6. Variable pressure (VP) mode, using the VPSE detector, is an alternative to high vacuum mode where samples can be imaged without or very minimal coating. Nitrogen gas is used in the VP mode and it should be introduced to the chamber in small increments. Increase nitrogen pressure to the chamber as needed to achieve a good micrograph.

6.2.7. If elemental composition or identification of impurities is desired for a sample, the Oxford EDS detector(s) is used to either generate an elemental map the sample over a scanned area and/or obtain an EDS spectrum on at a specific position on the sample.

6.2.8. General morphology of HE powder shall be noted while examining several areas of the samples. If particle dispersion is uniform, examine several random areas. Visually assess the typical largest and smallest particle sizes. Take electron micrographs of at least several areas exhibiting a good sampling of particles, at appropriate magnifications showing numerous particles to individual particles.

6.2.9. Note the corresponding HE parent serial number on each electron micrograph.

6.3. Sample Archival and disposal

6.3.1. Return any excess HE to the Sample Custodian and dispose of the SEM samples according to the HEAF waste management guidelines.

6.4. Data Recording and Storage

6.4.1. Record samples file IDs in the notebook. All the image files are saved in the image directory.

6.4.2. Each image will be stored using SEM software (SmartSEM) on the computer.

6.4.3. Copies of the images are transferred using Ironkey or any other medium. The images can be manipulated with Photoshop for further enhancements.

7.0 QUALITY CONTROL AND METHOD PERFORMANCE

The quality of measurements or qualitative judgments made from SEM electron micrographs is subject to operator training and expertise.

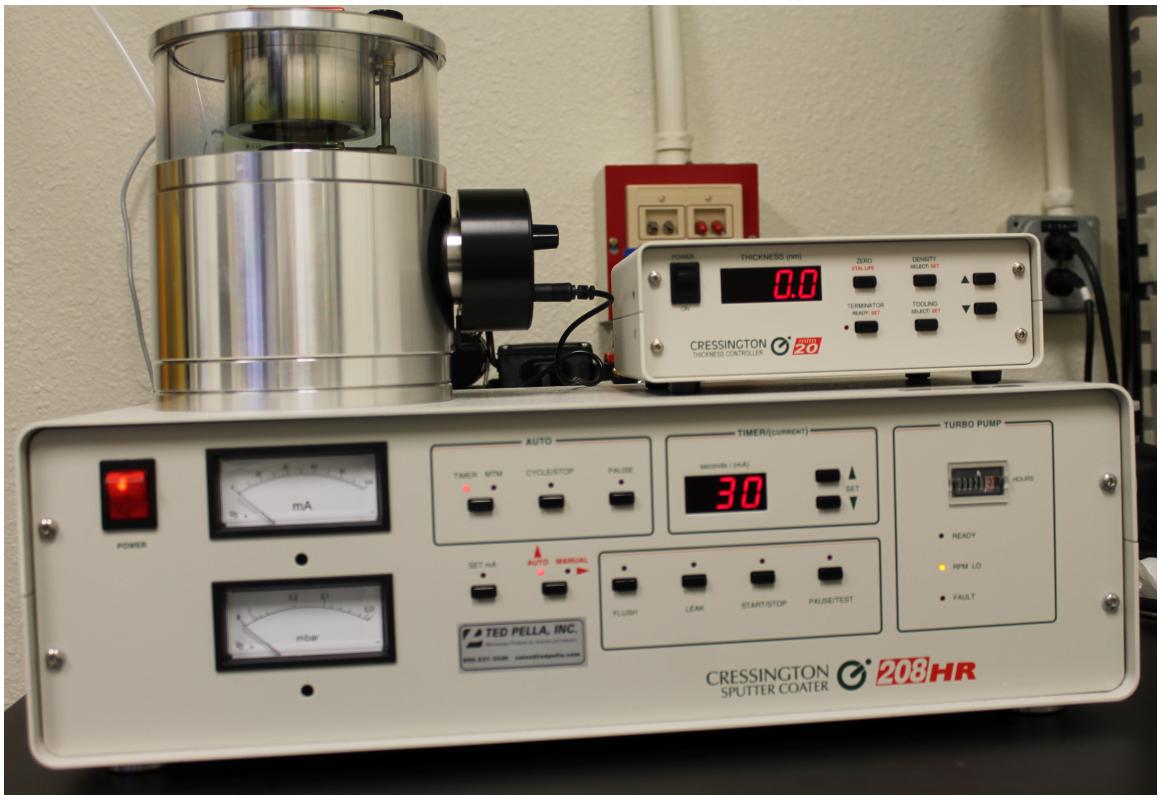

Figure 1: Cressington Magnitron Sputter Coater 208HR

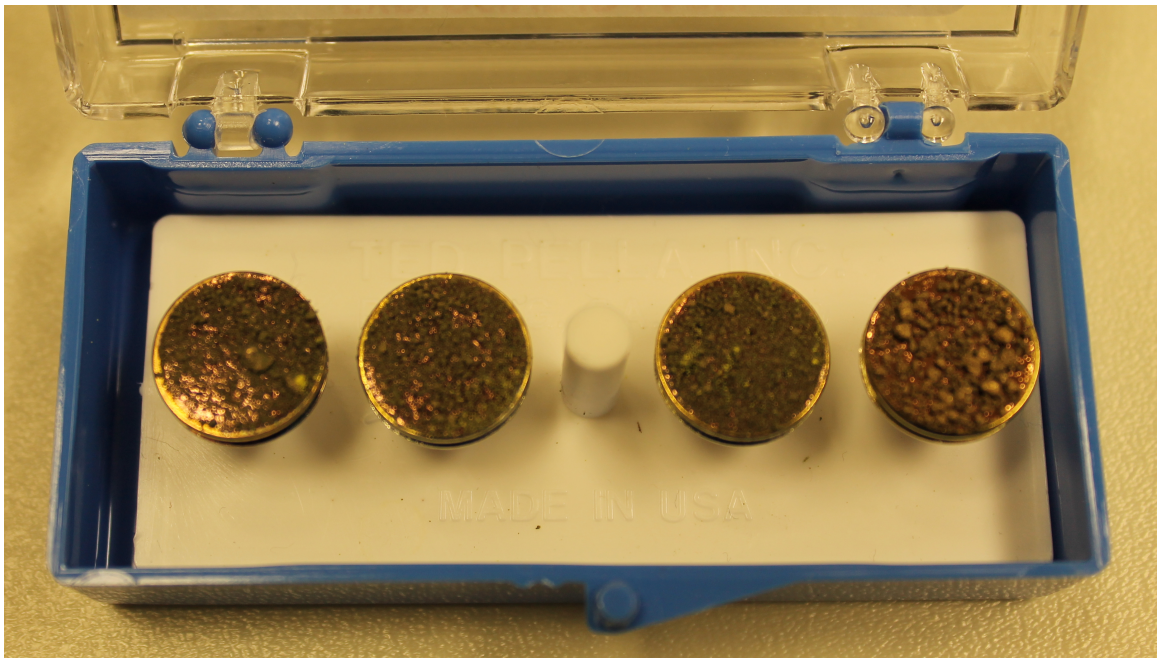

Figure 2: Gold coated samples

Figure 3: Carbon Double Sided Adhesive Tape, Ted Pella

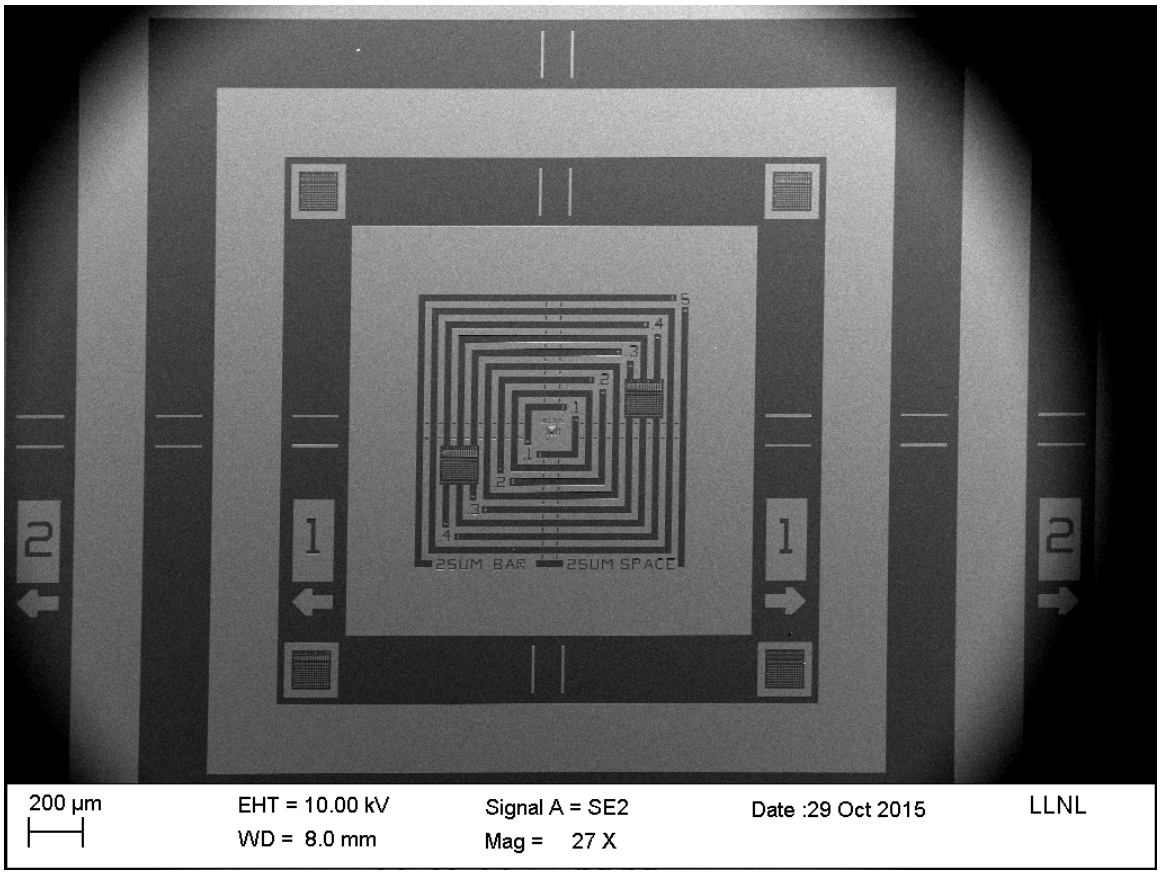

Figure 4: MRS-3XY grid image

Figure 5: Zeiss SEM sample carousel

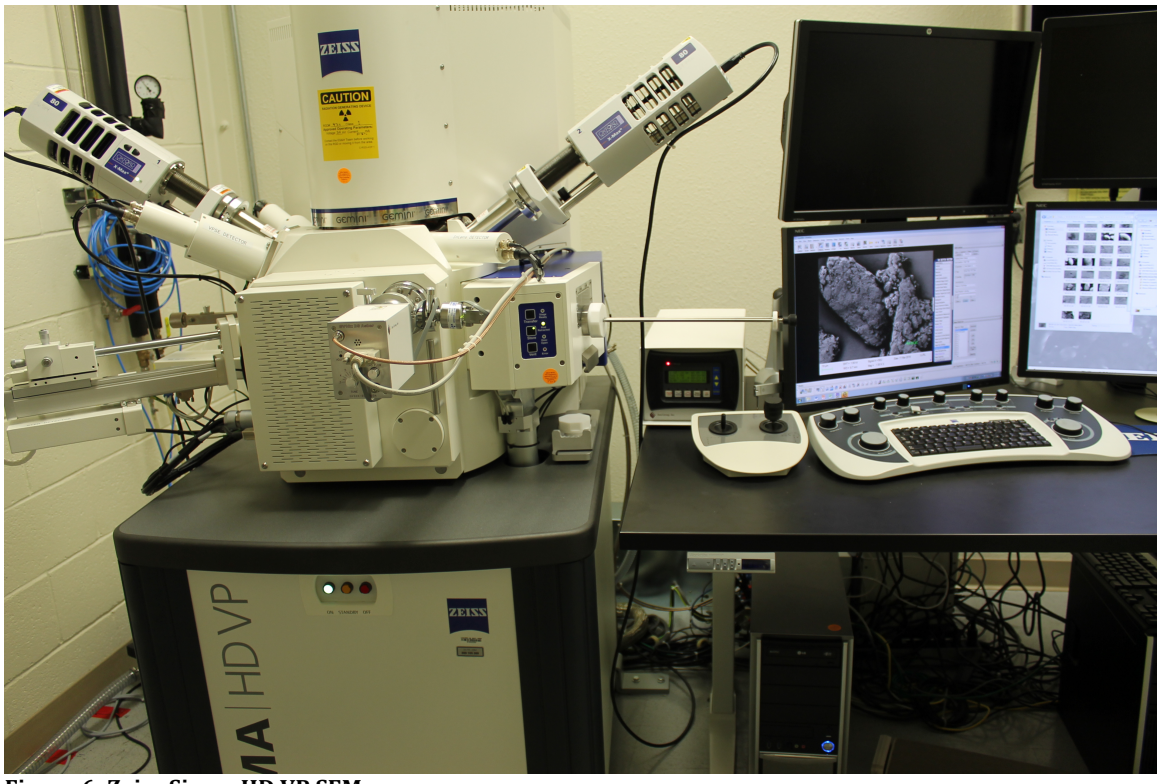

Figure 6: Zeiss Sigma HD VP SEM