Particle Image Analysis Process

Revision: 0
Effective Date is 3 days after the date of approval

Prepared By:  
Signature on File  03/28/17
Robert Hasson  
Ash Fall Project QA Lead

Approved By:  
Signature on File  03/28/17
John April  
ORP Ash Fall Project Engineer

Concurrence:  
Signature on File  04/03/17
Ken Armstrong  
EMCBC Associate Deputy Director
1.0 PURPOSE

This procedure establishes the responsibilities and process for characterizing the shape of volcanic ash particles using the Particle Image Analysis methodology for the Ash Fall Project.

2.0 SCOPE

The scope of this procedure includes identifying the process for the Mount Saint Helens and the Valley of 10,000 Smokes collected samples to be characterized utilizing the Scanning Electron Microscope (SEM) located at the United States Geological Survey (USGS) location in Vancouver, WA in support of the Department of Energy Environmental Management Consolidated Business Center (EMCBC) Ash Fall Project supporting the Office of River Protection Program.

3.0 APPLICABILITY

This procedure applies to EMCBC Ash Fall Project personnel who use collected samples that are required to be characterized for the modeling activities in support of the Ash Fall Project.

4.0 REQUIREMENTS and REFERENCES

4.1 Requirements

4.1.1 EM-QA-001, EM Quality Assurance Program (QAP)

4.1.2 ASME NQA-1-2008/2009a, Quality Assurance Requirements for Nuclear Facility Applications

4.2 References

4.2.1 AFP-QAPP-01, Quality Assurance Project Plan (QAPP)

4.2.2 AFP-AP-05, Control of Electronic Management of Information

4.2.3 AFP-AP-18, Sample Control

4.2.4 AFP-AP-20, Quality Assurance Records

4.2.5 AFP-AP-21, Laboratory Notebook Control
5.0 DEFINITIONS and ACRONYMS

None.

6.0 RESPONSIBILITIES

6.1 Ash Fall Project Staff (User)

6.1.1 Responsible for maintaining the samples to be used, preparation of the equipment to perform the characterization of the samples, using Particle Image Analysis to collect information, and for processing the results to the Desert Research Institute (DRI) supporting the Ash Fall Project.

6.1.2 Responsible for collection and submittal of the Particle Image Analysis equipment records, including any annual reviews by the Manufacturer.

6.1.3 Responsible for the being trained on the use of the Particle Image Analysis methodology, including SEM equipment and software.

6.2 Ash Fall QA Lead

6.2.1 Responsible for the oversight of the sample controls and process utilizing the Particle Image Analysis methodology.

7.0 GENERAL INFORMATION

None.

8.0 PROCEDURE

8.1 SEM Set-up for the Collected Samples

8.1.1 The samples to be used shall be provided by the Ash Fall Project Staff who collected the samples either from Mount Saint Helens or the Valley of 10,000 Smokes using the Chain of Custody form, AFP-AP-16, Sample Control.

8.1.2 Once the appropriate Ash Fall Project Staff and selected samples are at the USGS Vancouver, WA facility, the samples designated for characterization will be prepared for SEM analysis.
8.1.3 The selected volcanic ash particles (typically dry-sieved between 45–63 microns diameter) will be mounted on pin-type SEM stubs by lightly dusting the particles onto double-sided carbon tape.

8.1.4 The mounted particles will be coated with carbon using a Denton Vacuum Desk Sputter Coater.

8.1.4.1 Insert the pin mounts in a sample holder, and place them into the Denton Vacuum chamber on top of clean white paper.

8.1.4.2 The chamber needs to be evacuated to approximately 20 mtorr (typically 5–10 minutes).

8.1.4.3 Start Argon Gas flow into the chamber.

8.1.4.4 Apply high voltage to the target by manually increasing the power to level 10 or 20 until the elements start to glow. Then increase the dial at a moderate pace to 40 or 50 until the rod just starts to sputter, followed by quickly turning the dial back down to zero.

8.1.4.5 Power off the coater, remove the vacuum, and ensure that the samples have been evenly coated by inspecting the white paper underneath. The paper should indicate that the coat extends beyond the edge of the samples.

8.1.5 Prepare the set-up and use of the SEM in accordance with Attachment A, Instructions for SEM Set-Up and Use.

Note: The SEM is not required to be calibrated in accordance with the Ash Fall Project procedure AFP-AP-17, Control of Measuring and Test Equipment. To obtain information on particle shapes for this procedure, the SEM will be used as a digital imaging device and not used for chemical mapping or chemical identification. Therefore, the key function to verify is the magnification of the image, which will be performed prior to each batch of analyses, as described below.

8.2 SEM Verification

8.2.1 Prior to each batch of analyses, the magnification of the SEM will be verified using a NIST-traceable LMS-20T Low Mag Calibration Standard. The standard is placed in the SEM by the user in accordance with the
Particle Image Analysis Process

suggested conditions in Attachment A, Instructions for SEM Set-Up and Use.

8.2.2 Using the SEM software, a measured line will be drawn across two lines a known distance apart on the standard at 2500x magnification. The measured line must agree with the standard within 2.5 microns to meet verification requirements.

8.2.3 Verification images of the standard will be recorded and maintained as a record.

8.3 Using the SEM to Obtain Images of the Samples

8.3.1 The samples are placed in the SEM by the user in accordance with the suggested conditions in Attachment A, Instructions for SEM Set-Up and Use. These can be adjusted as needed based on the user’s needs.

8.3.2 The user will navigate the sample for areas of interest to represent the sample characteristics.

8.3.3 Once satisfied with the live scan, an image will be recorded and maintained as a record.

8.4 Using Photoshop Software to Process Images

8.4.1 To prepare images of ash particles for shape characterization, the images must be pre-processed using Photoshop software, as specified in Attachment B, Instructions for Image Processing with Photoshop.

Note: Photoshop is used for image preparation only, and not used to quantify any values used in the analysis.

8.5 Using ImageJ Software to Analyze Particle Characteristics

8.5.1 In the ImageJ software, open both the original SEM image and image that was processed using Photoshop.

8.5.2 Use the scale bar on the original image to set the scale by zooming in on it, drawing a straight line on it (shift+drag), and hitting measurements → set scale → actual size (insert scale here μm) and check ‘global’.
Particle Image Analysis Process

8.5.3 Return to the processed image and click process → binary → make binary and binary → fill holes. Save.

8.5.4 Set Measurements to include perimeter, area, bounding rectangle, and feret diameter.

8.5.5 To measure grains, go to process → analyze particles → include 1–infinity pixels, 0–infinity roundness, draw outlines, show results and proceed. Verify that the grain outlines look realistic.

8.5.6 Copy the results into spreadsheet format using edit → select all → ctrl+C.

8.5.7 Images of the outlined, numbered grains, and the spreadsheet output will be recorded and maintained as records for the Ash Fall Project.

8.6 Documentation of the SEM Characterization Information

8.6.1 The user will save the original and processed SEM images to an external drive or flash drive. The images will be maintained as the records in accordance with AFP-AP-20, Quality Assurance Records.

8.6.2 SEM process information regarding the various samples selected for investigation, sample control, images collected, and equipment used shall be obtained by the user and recorded in the user’s laboratory notebook in accordance with AFP-AP-21, Laboratory Notebook Control.

8.6.3 The collected images of the samples will be transmitted to DRI meeting the criteria specified in AFP-AP-05, Control of Electronic Management of Information.

8.6.4 The samples removed from the SEM shall be maintained by the user at a designated storage area and properly controlled until turnover of the SEM images are provided to the ORP at the conclusion of this project.

8.6.5 The images of the collected samples at the Vancouver, WA facility shall be maintained on the SEM database until turnover of the SEM images are provided to the ORP at the conclusion of this project. The SEM images maintained on the database can then be deleted after proper notification by either the QA Lead or the Ash3d modeling staff.
Environmental Management Consolidated Business Center
Ash Fall Project

Particle Image Analysis Process
Procedure: AFP-PIA-01
Revision 0, 04/06/17

9.0 RECORDS

9.1 The approved document in its entirety shall be submitted by the EMCBC Coordinator to records in accordance with AFP-AP-20, Quality Assurance Records.

9.2 The following are considered Lifetime QA Records:
- SEM Images of Particles (Original and Processed)
- SEM Images of Scale Verification
- SEM Equipment Records (Initial Set-up/Acceptance)
- Spreadsheet Values from Output of Particle Shape Characterization

10.0 FORMS USED

None.

11.0 ATTACHMENTS

Attachment A – Instructions for SEM Set-Up and Use
Attachment B – Instructions for Image Processing with Photoshop
Attachment A – Instructions for SEM Set-Up and Use

1. Select “Vent” to bring the SEM to ambient pressure.
2. Slide open the sample chamber and load the appropriate sample holder.
3. Position sample holder and sample mounts at an optimal working distance, approximately 15-17 mm from the detector.
4. Draw the orientation of each sample mount in log book (may also take a photograph).
5. Use a gloved finger to wipe the sample chamber gasket and ensure the chamber is free of dust.
6. Caution: Obey the ruler rule. After positioning the samples, run a plastic ruler across the top of the chamber to ensure that nothing protrudes above the sides (otherwise, the detector can be damaged).
7. Gently close the sample chamber by sliding it in and lifting the chamber door until it clicks.
8. Press green check to engage the vacuum. [Note: cannot power up machine until vacuum is ready].
9. Open the SEM software (Aspex Perception Software console).
10. Once the software indicates the vacuum is ready, click ‘Power’ in upper left-hand corner of software to power up the SEM.
11. Select the beam energy (recommend 20 kV under vacuum).
12. Navigate to sample in the stage window and ensure low magnification (500x), fast image scan, and low image size (256x256).
13. Caution: Spot size should be set to the high end of the recommended (highlighted) range at all times, which changes with magnification. Using a larger spot size can burn out the filament.
14. Saturate the filament by driving up the ‘Filament Drive’ dial. Increase until something becomes visible in the scan window viewfinder. A good guide is to use the last-used saturation in the log (e.g., 62.8%).
15. Manually adjust the brightness and contrast. A good starting point for volcanic ash -4% brightness, 96% contrast, but needs to be manually adjusted to optimize sample visibility.
16. Find a target in the sample to focus on, and perform a rough focus by click-dragging your cursor in the focus window.
17. To fine-tune the focus, use the second computer monitor (right side) and click on the square icon below the live image. This enlarges the view for focusing purposes. Zoom in to >500x and click-drag in the focus window until target appears sharp.
18. Ctrl+left-click on the live image (monitor on right) will center the view on the clicked object.
19. Continue to saturate the filament (step #14) as necessary as the instrument warms up. This can be performed by selecting F4 to enter line scan mode.
20. Set-up is now complete.
21. To shut down the SEM, drive down the filament to zero, power off, click ‘vent’ on the SEM, remove samples, and return the SEM to vacuum by clicking ‘vent’.
Environmental Management Consolidated Business Center
Ash Fall Project

Particle Image Analysis Process

Procedure: AFP-PIA-01
Revision 0, 04/06/17

Attachment B – Instructions for Image Processing with Photoshop

1. Open an SEM image in Photoshop.
2. Click image ➔ mode ➔ greyscale. Then save as a copy.
3. Increase brightness and contrast, and filter out noise using filter ➔ blur ➔ smart blur.
4. Use the magic wand tool to highlight the grains (turn off anti-alias option, non-contiguous, and put threshold around 100). This should highlight most grains, but there will be grain edges that need to be included. Lower the threshold to 30–50 and hold shift while using the magic wand to add grain facets to the selection until they are all reasonably represented.
5. While this is still highlighted, hit Ctrl+C, make a new layer, and Ctrl+V the selected grains into the new layer. Now the new layer can be clicked on and off to compare with the original image. In this new layer, select the background using magic wand, then click image ➔ select inverse to activate the grains again, and hit edit ➔ fill ➔ white to change their color to white. Save.
6. The grains need to be cleaned up and separated (if touching) using the pencil tool (size ~4 pixels wide). If grains cannot be separated without sacrificing the integrity of one of them (i.e., < 90–95% intact), save the one on top and erase remnants of the other by drawing over it in black. Also, in the analysis process any grains touching the edge of the frame won’t be counted, so if there are grains >90–95% visible that should be counted, separate them from the frame edge with a black pencil edge (at least 1 pixel wide).
7. Once all the grains all accounted for, SAVE, then select a white patch in the image using magic wand, click select ➔ inverse, and fill in the background with black. Make sure the processed image compares well with the original image, making sure there are not any tiny white artifacts from crushed grains or image noise. Then crop off the scale bar and SAVE.
8. Both the original SEM image and the processed image will be saved as records for the project.
9. Image processing is now complete.
Form 12-1 – Record of Revision

**DOCUMENT:** AFP-PIA-01, *Particle Image Analysis Process*

<table>
<thead>
<tr>
<th>Revision Number</th>
<th>Description of Changes</th>
<th>Revision on Pages</th>
<th>Effective Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Initial Issue</td>
<td>All</td>
<td>04/06/2017</td>
</tr>
</tbody>
</table>