Submission ID #: 61636
Scriptwriter Name: Bridget Colvin
Project Page Link: https://www.jove.com/account/file-uploader?src=18799028

Title: Three-Dimensional Particle Shape Analysis Using X-Ray Computed Tomography: Experimental Procedure and Analysis Algorithms for Metal Powders

Authors and Affiliations: Edward J. Garboczi and Nik Hrabe

Applied Chemicals and Materials Division, MS647, National Institute of Standards and Technology

Corresponding Authors:
Edward J. Garboczi
edward.garboczi@nist.gov

Nik Hrabe
nik.hrabe@nist.gov

Author Questionnaire

1. **Microscopy**: Does your protocol require the use of a dissecting or stereomicroscope for performing a complex dissection, microinjection technique, or similar? **N**

2. **Software:** Does the part of your protocol being filmed demonstrate software usage? **Y**

Videographer: All screen captures provided, do not film

3. **Interview statements:** Considering the Covid-19-imposed mask-wearing and social distancing recommendations, which interview statement filming option is the most appropriate for your group? **Please select one**.

☒ Interviewees wear masks until the videographer steps away (≥6 ft/2 m) and begins filming. The interviewee then removes the mask for line delivery only. When the shot is acquired, the interviewee puts the mask back on. Statements can be filmed outside if weather permits. **We would like the outside option – nice background of mountains at NIST-Boulder. If weather permits.**

4. **Filming location:** Will the filming need to take place in multiple locations (greater than walking distance)? **N**

Protocol Length
Number of Shots: **40**

Introduction

1. Introductory Interview Statements

REQUIRED:

1.1. **Edward Garboczi**: This protocol characterizes particle size and shape in three dimensions, with no assumptions. This is important, because particle size and shape are not independent quantities **[1]**.

1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

REQUIRED:

1.2. **Nik Hrabe**: This technique can be used to quantitatively distinguish between single, near-spherical particles, which are best for additive manufacturing, and non-spherical and multi-particles, which often occur in metallic additive manufacturing powders **[1]**.

1.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

OPTIONAL:

1.3. **Edward Garboczi**: Any particles of millimeter-size and smaller can be analyzed by this method. Larger particles can also be characterized, but it is harder to scan enough particles for adequate statistics **[1]**.

1.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

Introduction of Demonstrator on Camera

1.4. **Edward Garboczi**: Demonstrating the procedure will be Newell Moser, a National Research Council Post-Doctoral Fellow from my laboratory **[1][2]**.

1.4.1. INTERVIEW: Author saying the above
1.4.2. The named demonstrator(s) looks up from workbench or desk or microscope and acknowledges the camera

Protocol

NOTE: Author mentioned that some shots may have been combined, but didn't say which ones

2. Epoxy Powder Preparation

2.1. Begin by placing about 25 grams of quick-curing epoxy in a small, disposable dish **[1]**.

2.1.1. WIDE: Talent emptying two packs of epoxy into boat *Videographer: Important step*

2.2. Use the equation to determine the number of grams of metal powder needed to give a volume fraction of about 10% after the powder has been mixed into the epoxy **[1-TXT]**.

2.2.1. BLACK TEXT WHITE BACKGROUND:

$$0.1 = \frac{M/\rho}{25\ g/\rho_e}$$

2.3. Use a disposable stirring rod to vigorously mix the metal powder into the epoxy for about 30 seconds to adequately disperse the powder throughout the epoxy **[1]**.

2.3.1. Powder and epoxy being mixed *Videographer: Important/difficult step*

2.4. After mixing, scrape the viscoplastic mixture into a compact clump with as much vertical extent as possible **[1]** and quickly insert the nozzle of a 0.5-meter-long plastic hose connected to a small vacuum pump into one end of a 3-millimeter-diameter, 150-millimeter-long polymer straw **[2]**.

2.4.1. Mixture being scaped into clump *Videographer: Important/difficult step*

2.4.2. Talent inserting nozzle into straw *Videographer: Important step*

2.5. Holding the nozzle and the straw end firmly pinched together, insert the free end of the straw into the compact epoxy-powder clump **[1]** and turn on the vacuum pump **[2]**.

2.5.1. Shot of nozzle and straw pinched together, then straw being inserted into clump *Videographer: Important step*

2.5.2. Talent turning on pump

2.6. When the straw is filled to within 10 millimeters of the top **[1]**, wipe the epoxy mixture from the filling end of the straw **[2]** and use a small lump of clay to plug both ends of the straw **[3]**.

2.6.1. Straw being filled/shot of filled straw *Videographer: Important step*

2.6.2. Epoxy being wiped *Videographer: Important step*

2.6.3. Straw being inserted into clay *Videographer: Important step*

2.7. After filling a second straw in the same manner, allow the epoxy to cure before using a razor to cut off the clay ends **[1]** and cutting each straw in half to obtain four samples **[2]**.

2.7.1. Shot of cured epoxy, then end(s) being cut *Videographer: Important step*

2.7.2. Straw being cut in half *Videographer: Important step*

3. X-Ray Computed Tomography (XCT)

3.1. For XCT (X-C-T) particle imaging, mount one straw sample vertically to allow the X-rays to penetrate across the circular cross-section of the straw **[1]**.

3.1.1. WIDE: Talent mounting straw

3.2. Set the instrument to about a 120-kilovolt voltage **[1]** and select a full 360-degree scan **[2]**.

3.2.1. SCREEN: 3.2.1-3.3.1: 00:44-00:48 *Video Editor: please emphasize Voltage in bottom center of frame*

3.2.2. SCREEN: 3.2.1-3.3.1: 00:49-00:54 *Video Editor: please emphasize Angle Full 36 in center right*

3.3. Duplicate this field-of-view at different parts of the sample to acquire 2-8 fields of view to obtain a minimum of 1000 particles for analysis. After reconstruction, save the image information in about 1000 cross-sectional slices per field-of-view in 8-bit format **[2]**.

3.3.1. SCREEN: 3.3.2: 00:19-00:44 *Video Editor: please speed up* **TEXT: Image >1 sample as necessary**

3.4. Note the pixel size of each image set, which is the same as the voxel size in micrometers, and the pixel size of the reconstructed slices **[1]**.

3.4.1. SCREEN: 3.4.1: 00:09-00:17

4. Slice Assembly and Geometrical Information Generation

4.1. For slice assembly, open terminal **[1]** and enter the command as indicated **[2]**.

4.1.1. WIDE: Talent opening terminal

4.1.2. SCREEN: 4.1.1-4.1.2: 00:00-00:22

4.2. Use the command to process the particle files **[1-TXT]**.

4.2.1. SCREEN: 4.2.1: 00:18-00:29 **TEXT: Processing can take several hours**

4.3. After processing, view the **OriA-0500.tiff** image to visualize the k = 500 slice of the first field of view in the **particle-class-sysconfig.dat** file without any image processing. View the second image file to visualize the same image but after segmenting and thresholding **[1]**.

4.3.1. SCREEN: 4.3.1: 00:00-00:08

4.4. For each processed spherical harmonic particle, the 3D VRML (V-R-M-L) image file will contain two images side-by-side, a voxel image of the original particle, and a smoother rendered image using the spherical harmonic coefficients **[1]**. For each non-spherical harmonic particle, only the voxel image will be stored **[2]**.

4.4.1. SCREEN: 4.4.1-4.4.2-4.4.3: 00:00-00:10 **TEXT: VRMUL: virtual reality modeling language**

4.4.2. SCREEN: 4.4.1-4.4.2-4.4.3: 00:11-00:26

4.5. Make a list of the spherical harmonic **Particle-class-name-anm-particle-number.dat** file names and a list of the non-spherical harmonic **Particle-class-name-part-particle-number.dat** particles **[1]**.

4.5.1. SCREEN: 4.5.1 *Video Editor: please speed up*

4.6. Then use the small program **number.f** to change the spherical harmonic **anm.lis** file so that it has the number of the particle and the filename on each line of the list file **[1]**.

4.6.1. SCREEN: 4.6.1: 00:09-00:33 *Video Editor: please speed up*

5. SH and nonSH Particle Subset Selection

5.1. To determine the single, near-spherical and non-spherical length and thickness cutoffs, run the **VRML-select-multi-single.f** program **[1]** to select 10 particles in each length by a thickness interval of 0.1 to store up to 100 spherical and non-spherical particles with length by thickness ratios between 1 and 2 **[2]**.

5.1.1. WIDE: Talent running program, with monitor visible in frame

5.1.2. SCREEN: 5.1.2: 00:26-01:29 *Video Editor: please speed up*

5.2. Two text files will be generated listing the length by thickness values and the identified VRML image files. Transfer these data into a spreadsheet ordered according to the length by thickness value **[1]**.

5.2.1. SCREEN: 5.2.1: 00:00-00:25 *Video Editor: please speed up*

5.3. Visually examine the 3D images of each of these particles to determine the overall range of morphologies, starting from the lowest length by thickness value particles **[1]**.

5.3.1. Talent at computer, visually examining images

5.4. The particles are assessed in terms of whether they are broken **[1]** … double **[2]** … multiple **[3]** … or irregular **[4]** and whether they have satellites attached to the main particle **[5-TXT]**.

5.4.1. LAB MEDIA: 5.4.1: 00:18-00:34

5.4.2. LAB MEDIA: 5.4.1: 01:02-01:08

5.4.3. LAB MEDIA: 5.4.1: 01:32-01:42

5.4.4. LAB MEDIA: 5.4.1: 02:05-02:22 *Video Editor: please speed up*

5.4.5. LAB MEDIA: 5.4.1: 02:40-02:51 **TEXT: Satellite = protuberance with <1/5 diameter of main particle**

5.5. An approximate length by thickness cutoff value is used to separate single, near-spherical particles **[1]** from multiple and very non-spherical particles **[2]**.

5.5.1. SCREEN: 5.5.1: 00:14-00:18

5.5.2. SCREEN: 5.5.1: 00:44-00:48

5.6. The first double or multiple particle found determines the cutoff value for both spherical harmonic **[1]** and non-spherical harmonic particles **[1]**.

5.6.1. SCREEN: 5.6.1: 00:15-00:19

5.6.2. SCREEN: 5.6.1: 00:47-00:52

6. 2D Projection Data Generation

6.1. For the generation of 2D projection data from the 3D particle information, use the **proj-mpi-SH-LWT.f** and **proj2D-nonSH-LWT.f** programs **[1]** to generate three orthogonal projections for each particle along the direction of the length, width, and thickness unit vectors **[2]**.

6.1.1. WIDE: Talent running program(s), with monitor visible in frame

6.1.2. SCREEN: 6.1.2: 00:23-00:52 *Video Editor: please speed up*

6.2. These programs generate a series of x-y points that are stored for each projection. Enter these data into any graphing program that accepts this input, making sure that the axis limits of x and y are the same and that each axis is the same physical length **[2]**.

6.2.1. SCREEN: 6.3.2: 00:25-00:59 *Video Editor: please speed up*

Protocol Script Questions

A. Which steps from the protocol are the most important for viewers to see? Please list 4 to 6 individual steps.

2.1., 2.3.-2.7.

B. What is the single most difficult aspect of this procedure and what do you do to ensure success?
2.1.-2.7. preparing the sample so that the metal powders are sufficiently dispersed. Ensure success by keeping the volume fraction at around 10 %.
4.2. using pp-Otsu.f to find and analyze the particles. Success is ensured by being careful with the input parameters in the *sysconfig.dat file, and many years of optimizing the software to make it "foolproof."
5.4., classifying the particles in regard to single, double, multiple, irregular, broken, and satellites. Success is ensured only through practice and looking at the VRML images for many particles.

Results

7. Results: Representative 3D Particle Shape Analysis

7.1. In this representative analysis **[1]**, a total of 16,970 particles were analyzed **[2]**, of which 14,580 were spherical harmonic particles **[3]** and 2390 were non-spherical harmonic particles **[4]**.

7.1.1. LAB MEDIA: Table 1
7.1.2. LAB MEDIA: Table 1 *Video Editor: please emphasize All data row*
7.1.3. LAB MEDIA: Table 1 *Video Editor: please emphasize SH data row*
7.1.4. LAB MEDIA: Table 1 *Video Editor: please emphasize nonSH row*

7.2. The limiting value of length by thickness for the spherical harmonic particles was 1.17 **[1]** and 1.1 for the non-spherical harmonic particles **[2]**.

7.2.1. LAB MEDIA: Table 1 *Video Editor: please emphasize 1.17 data cell*
7.2.2. LAB MEDIA: Table 1 *Video Editor: please emphasize 1.10 data cell*

7.3. Here a raw gray-scale reconstructed X-ray CT scan image **[1]** and an image segmented by Otsu routine for the 500th slice of one of the microstructures can be observed **[2]**.

7.3.1. LAB MEDIA: Figure 1 *Video Editor: please emphasize Figure 1a*
7.3.2. LAB MEDIA: Figure 1 *Video Editor: please emphasize Figure 1b*

7.4. The particle size distribution **[1]** can be plotted for the single, non-spherical and non-spherical particles separately **[2]** or combined **[3]**.

7.4.1. LAB MEDIA: Figure 2
7.4.2. LAB MEDIA: Figure 2 *Video Editor: please emphasize Figure 2a*
7.4.3. LAB MEDIA: Figure 2 *Video Editor: please emphasize Figure 2b*

7.5. Three 2D projections can be made **[1]**, allowing the equivalent circular diameter to be used as a measure of particle "size" **[2]**.

7.5.1. LAB MEDIA: Figure 3
7.5.2. LAB MEDIA: FIgur3 3 *Video Editor: please emphasize data bars*

7.6. In this volume-fraction based distribution of the 3D length by thickness parameter of all of the particles **[1]**, the long tail is mainly composed of particles consisting of two or more particles stuck together and some irregular single particles **[2]**.

7.6.1. LAB MEDIA: Figure 4
7.6.2. LAB MEDIA: Figure 4 *Video Editor: please emphasize end of graph from about 2.25 to 3.5*

7.7. For the 2D projections, the aspect ratio can be defined as the ratio of the maximum to the minimum Feret diameters **[1]**, allowing plotting of the area fraction-based distribution **[2]**.

7.7.1. LAB MEDIA: Figure 5
7.7.2. LAB MEDIA: Figure 5 *Video Editor: please emphasize data bars*

7.8. In this Table **[1]**, a summary of the porosity values in terms of the number of particles having internal pores **[2]**, the average porosity per particle having pores **[3]**, and the maximum value of porosity found can be observed **[4]**.

7.8.1. LAB MEDIA: Table 2
7.8.2. LAB MEDIA: Table 2 *Video Editor: please emphasize # or particles with internal pores column*
7.8.3. LAB MEDIA: Table 2 *Video Editor: please emphasize Average porosity per porous particle column*
7.8.4. LAB MEDIA: Table 2 *Video Editor: please emphasize Maximum porosity found*

7.9. In this analysis **[1]**, a slight trend for smaller particles having higher porosities was observed **[2]**.

7.9.1. LAB MEDIA: Figure 6
7.9.2. LAB MEDIA: Figure 6 *Video Editor: please emphasize dots on left side of graphs*

Conclusion

2. Conclusion Interview Statements

8.1. **Edward Garboczi**: The accuracy of the software and particle analysis depends on the preparation of epoxy-powder samples with sufficient particle dispersions. Keeping to the 10% volume fraction guidelines and using a fast-setting epoxy are crucial **[1]**.

8.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

8.2. **Nik Hrabe**: The whole question of how particle shape affects powder-based additive manufacturing processes and how these processes affect recycled powder particle shape in turn can now be addressed in 3D **[1]**.

8.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera