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Title:

Study of the microstructure of samples “57.3” pre- (M1) and post- (M2) injection by XCT.

Summary:

Two samples named “57.3” were measured by XCT (pre- and post- CO_2 injection). The acquired volumes have been delivered to CIEMAT for their evaluation. The different phases are visible in XCT experiments and the differences from the two states are going to be evaluated by CIEMAT.

Keywords:

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1 Introduction

This report summarizes the results of X-ray computed tomography for microstructure characterization.

1.1 Samples

The two received samples were named as:

1) 57.3_M1_PRE

   “57.3.M1 PRE” annotation on one face of the sample. Cuboid-like shape, approx. dimensions: 15x15x20 mm.

2) 57.3_M2_POST

   “57.3.M2 POST” annotation on one face of the sample. Cuboid-like shape, approx. dimensions: 18x18x20 mm.

1.2 Resolution evaluation & reshaping

In order to avoid artifacts and improve the achievable resolution, a cylindrical sample is ideal. To evaluate if the achieved resolution was sufficient, a first scan was performed on the as-received biggest sample, i.e. 57.3_M2_POST. Figure 1 shows sample's orientation in this scan.

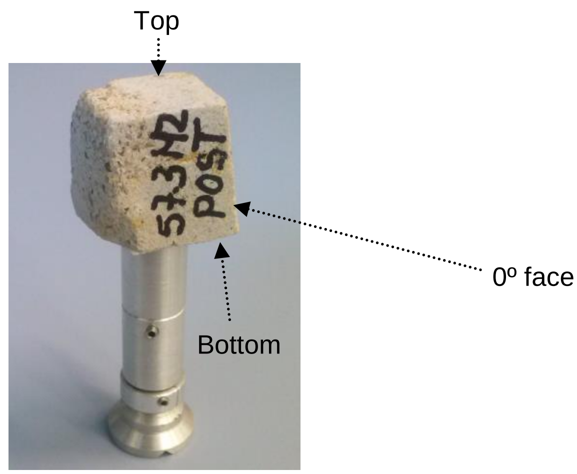
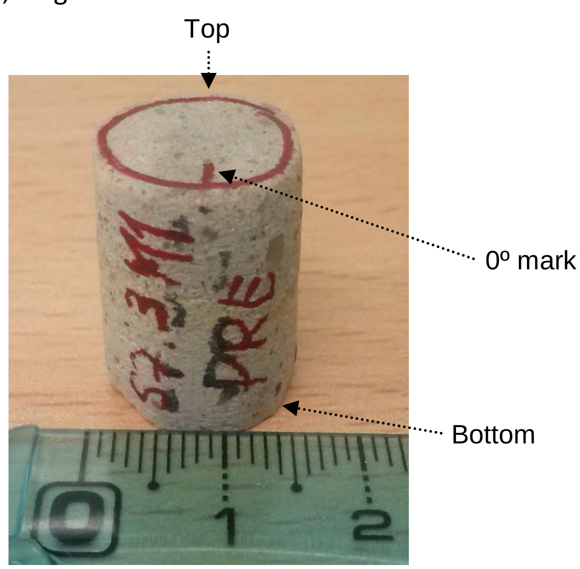


Figure 1. 57.3_M1_PRE first scan orientation.

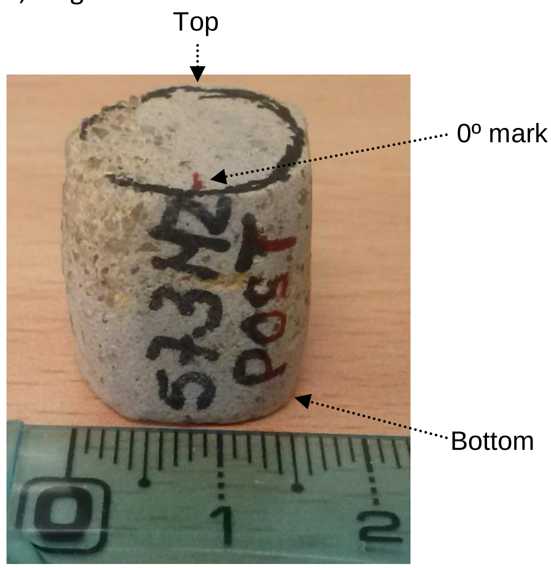
With the as-received shape, a voxel size of 12.28 µm was achieved, and although most of the phases could be distinguished the existence of small grains required higher resolution. A local tomography was then performed on the same sample (same orientation), with a voxel size of 6.00 µm. With local tomography we were able to increase the resolution, but this method produces some artifacts in the volume which were eliminated by cropping the volume. As it was unclear if the resulted scanned volume was representative enough, both samples were grinded down to a cylindrical-like shape. The on-sample annotations were kept at the same position.

Resulting shape, approx. dimensions and measurement orientations were:

1) 57.3_M1_PRE. Diameter: 15 mm; height: 20 mm.



2) 57.3_M2_POST. Diameter: 20 mm; height: 20 mm.



Thus, a voxel size of 9.28 µm was attained.

2 Results

2.1 X-Ray Computed Tomography measurements

Tomographic inspection was performed on samples M1_PRE_C02_62-45 and M2_POST_C02_62-45. The tomograms were acquired & reconstructed with following settings:

Scan	Voltage (kV)	Intensity (µA)	Filter	Images	Exposure time (ms)	Voxel size (µm)	Beam hardening correction
57.3_M1_PRE_scan_9.2um	100	90	No	2000	500	9.28	9.2
57.3_M2_POST_scan_12.2um	100	98	No	2000	500	12.28	7.9
57.3_M2_POST_scan_9.2um	100	98	No	2000	500	9.28	9.2
57.3_M2_POST_scan_6um	95	97	No	2000	500	6.00	7.0

The full reconstructed dataset for each scan is provided in a raw data file. These reconstructed volumes can be visualized using different software, like ImageJ (free software, see section 4.1) or VGStudio Max (commercial software).

For instance, Figure 2 shows a cross section of sample 57.3_M1_PRE, while Figure 3 shows cross sections of sample 57.3_M2_POST from the three different scans performed.

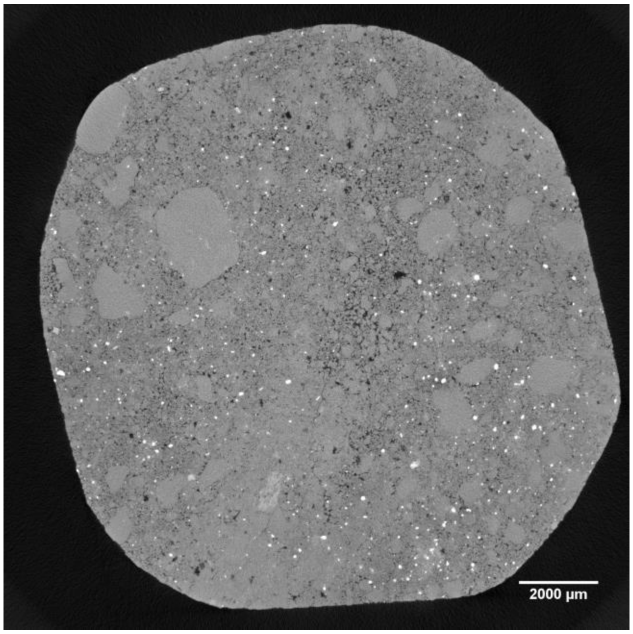


Figure 2. Cross section of 57.3_M1_PRE. 9.28 µm voxel size.

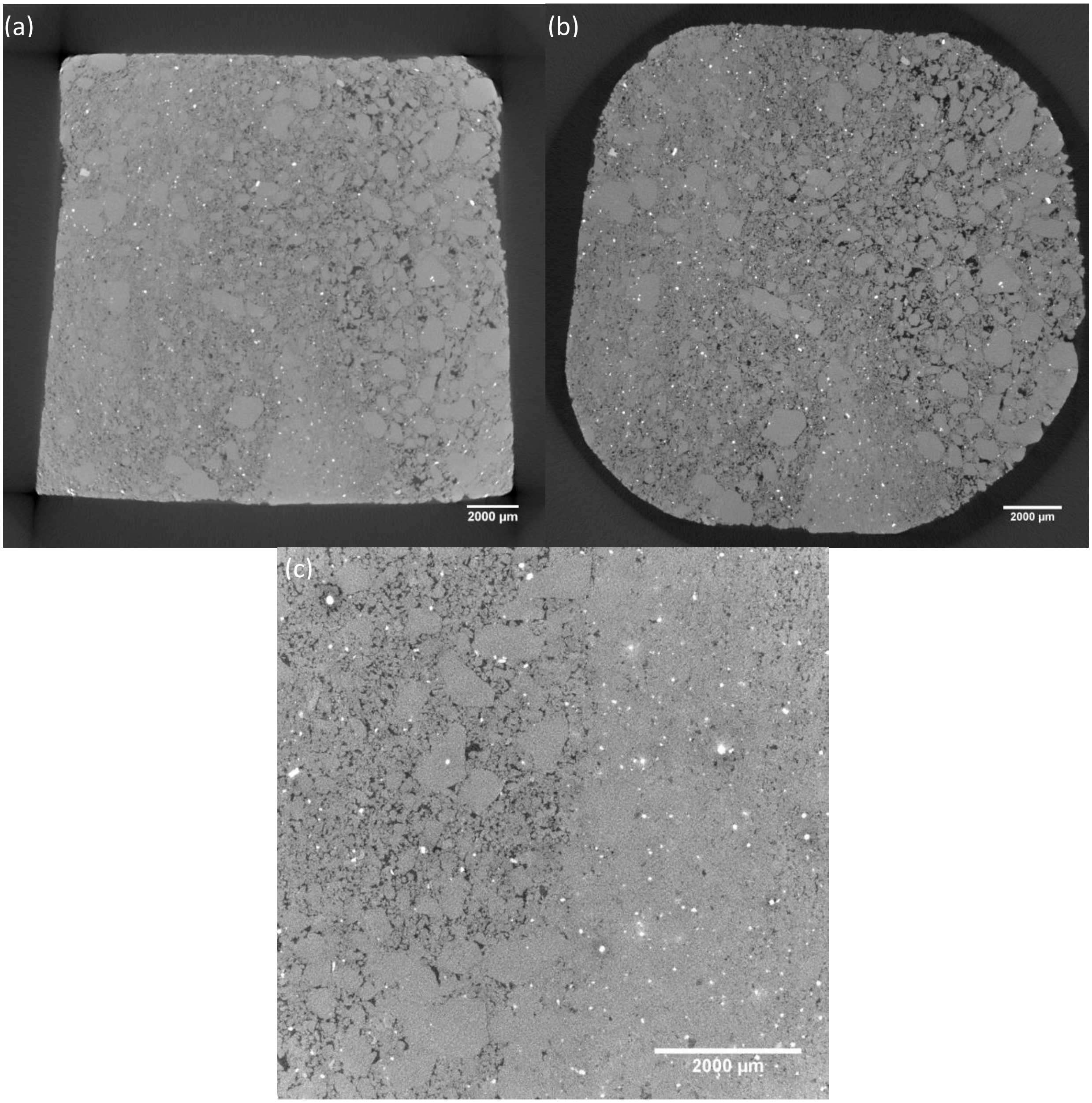


Figure 3. Cross sections of 57.3_M2_POST. (a) 12.28 µm voxel size. (b) 9.28 µm voxel size (c) 6.00 µm voxel size.

Figure 4-Figure 7 show three-dimensional renderings created in VGStudio of samples 57.3_M1_PRE and 57.3_M2_POST in varying voxel sizes.

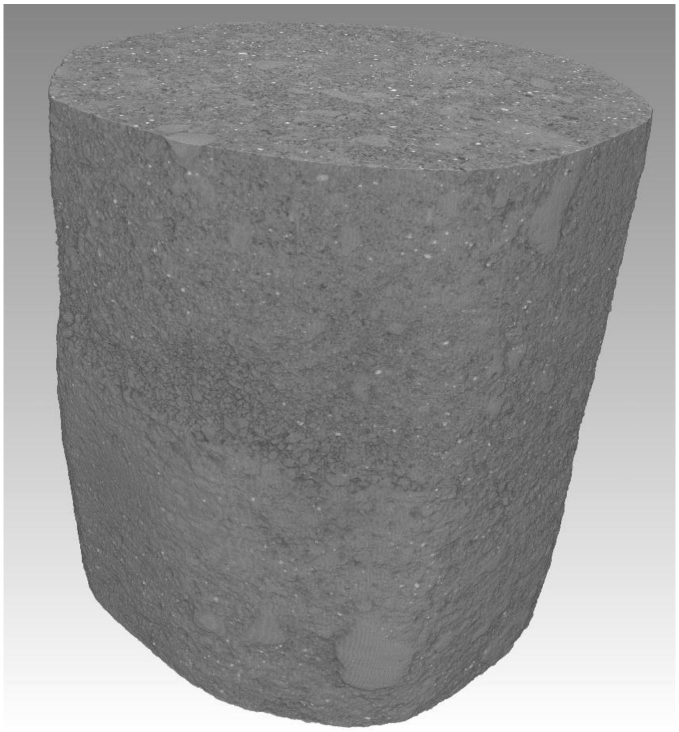


Figure 4. 3D view of 57.3_M1_PRE. 9.28 µm voxel size.

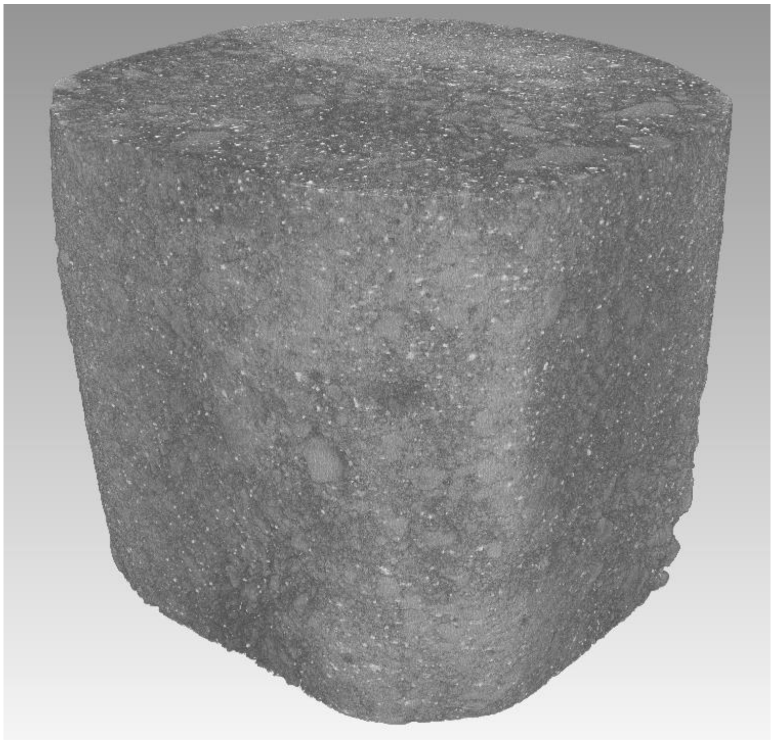


Figure 5. 3D view of 57.3_M2_POST. 9.28 µm voxel size.

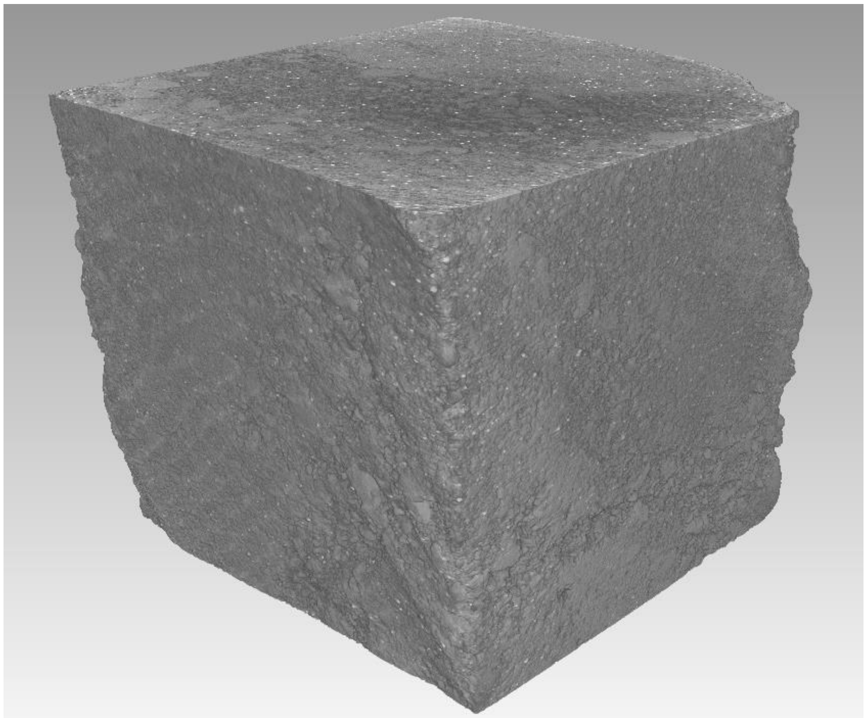


Figure 6. 3D view of 57.3_M2_POST. 12.28 µm voxel size.

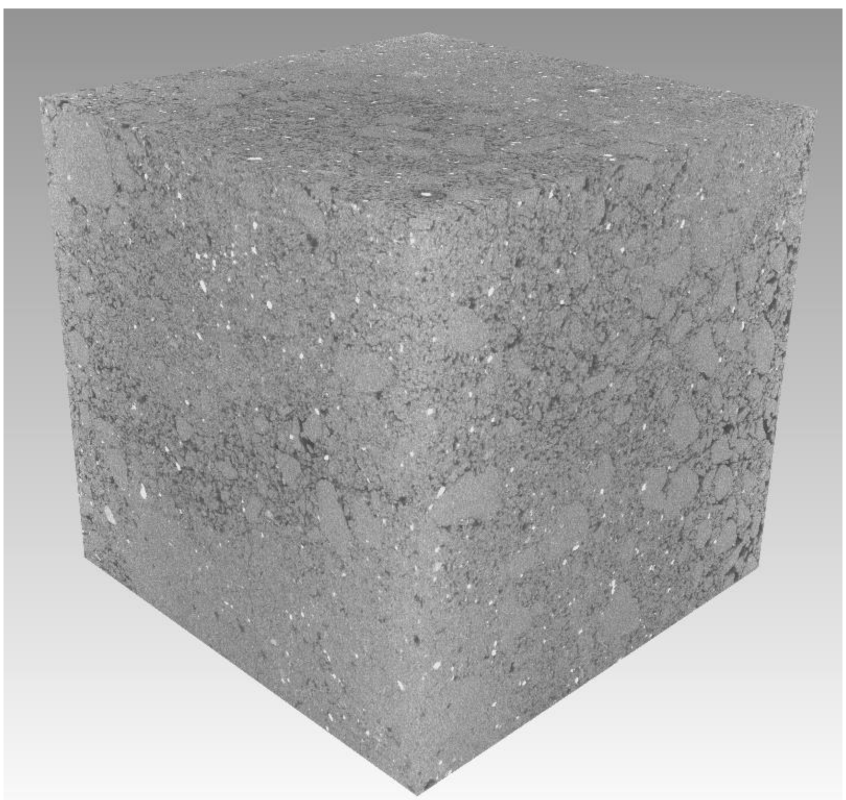


Figure 7. 3D view of 57.3_M2_POST. 6.00 µm voxel size.

3 Conclusions

- The microstructure of both samples could be revealed by X-ray tomography. The quantitative analysis and the usefulness of this measurements will be determined by CIEMAT.

- Higher resolution (up to approximately 2 μm) can be achieved, however in smaller sample volumes (diameter of 4 mm maximum). These measurements could provide a better insight of the smaller features in the specimens. In this case it is important to evaluate if such a small volume is representative enough.

4 Deliverables

All samples were returned to CIEMAT. Together with this report ("Report_Tomography_57.3_M1_and_M2_v01.pdf") the following tomographic volumes were delivered to CIEMAT:

Filename	Format	Dimensions (pixels)	Type*	Voxel size (μm)	Size (bytes)
57.3_M1_PRE_scan_9.2um_1700x1700x1721_16b.raw	raw	1700x1700x1721	16 bit unsigned	9.28	9,947,380,000
57.3_M1_PRE_scan_9.2um_1700x1700x1721_8b.raw	raw	1700x1700x1721	8 bit	9.28	4,973,690,000
57.3_M2_POST_scan_12.2um_1700x1700x1300_16b.raw	raw	1700x1700x1300	16 bit unsigned	12.28	7,514,000,000
57.3_M2_POST_scan_12.2um_1700x1700x1300_8b.raw	raw	1700x1700x1300	8 bit	12.28	3,757,000,000
57.3_M2_POST_scan_9.2um_2000x2000x1721_16b.raw	raw	2000x2000x1721	16 bit unsigned	9.28	13,768,000,000
57.3_M2_POST_scan_9.2um_2000x2000x1721_8b.raw	raw	2000x2000x1721	8 bit	9.28	6,884,000,000
57.3_M2_POST_scan_6um_1250x1250x1222_16b.raw	raw	1250x1250x1222	16 bit unsigned	6.00	3,818,750,000
57.3_M2_POST_scan_6um_1250x1250x1222_8b.raw	raw	1250x1250x1222	8 bit	6.00	1,909,375,000

*All files are big endian.

Note: The acquisition tomography data (projections, scan parameter files, etc.) of both measurements were not required by CIEMAT. However, they will be stored at IMDEA for two months before deletion.

4.1 Visualization of delivered files

The raw data files can be opened using the ImageJ free software (http://rsbweb.nih.gov/ij/). All the information required to load the reconstructed volumes is included in the filename. For example, the name of one of the files that correspond to sample 57.3_M1_PRE is:

57.3_M1_PRE_scan_9.2um_1700x1700x1721_8b.raw

Where:

- The numbers "1700x1700x1721" indicate the dimensions in "x" (width), "y" (height) and "z" (slices) respectively.
- "8b" means the file was saved in an 8 bit format.

Figure 8 shows a screenshot of the ImageJ software:

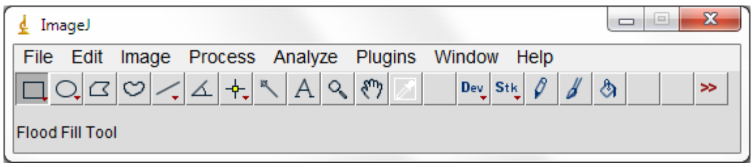


Figure 8. Screenshot of the ImageJ software.

Please note that due to the large size of the files, you will probably need to check the memory & threads settings of ImageJ under Edit→Options. Please refer to the ImageJ webpage for more information.

In order to open the file, go to File→Import→Raw… The following window will pop up, where the aforementioned parameters shall be entered:

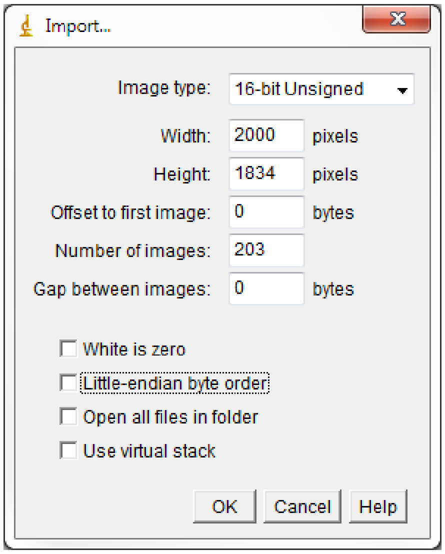


Figure 9. Screenshot of the ImageJ software (values showed in the Import… window are only examples).

4.1.1 Volumes orientation-physical orientation correlation

In the raw files, as opened in ImageJ, the last slice of the volume (i.e. 1721 for the 57.3_M1_PRE_scan_9.2um_1700x1700x1721_8b.raw) is close to the bottom part of the sample that was named as “bottom”.

The 0º orientation corresponds to the upper middle zone of the slices as showed in Figure 10. This holds for all reconstructed volumes.

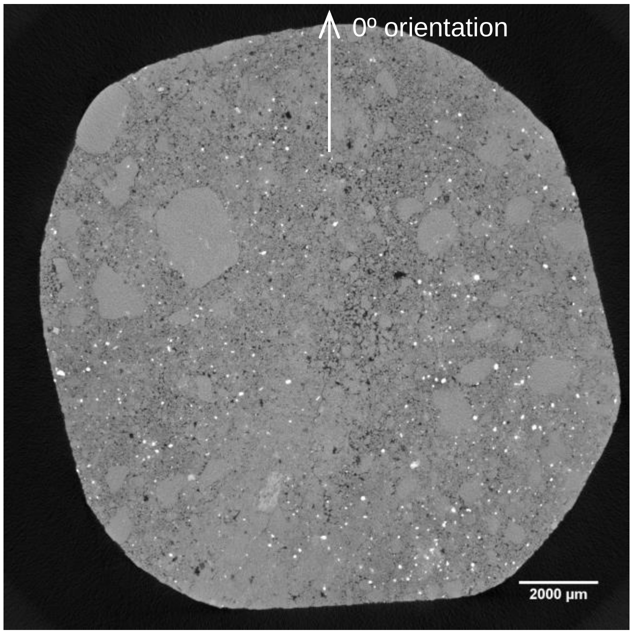


Figure 10. 0º orientation.