X-ray computed tomography vs Archimedes method: a head-to-head comparison

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Abstract. Metal additive manufacturing (AM) is growing rapidly towards industrial adoption in various industries, but porosity remains a concern because it creates areas of stress that affects the mechanical properties and reduces reliability. Porosity can be quantified by X-ray computed tomography (XCT), but the method is relatively slow and expensive. The Archimedes density measurement method is widely used due to the low cost and ease of operation; however, it is limited in its precision for low porosity levels. In this work, a series of additively manufactured aluminium 6061-Ti6 alloy samples with different types and quantities of porosities are subjected to Archimedes, gas pycnometer and X-ray methods. This work clarifies the application range and limitations of each of these methods, using 10 mm cubes of Al 6061-Ti6 manufactured by laser powder bed fusion.

1 Introduction

Additive Manufacturing (AM) is a promising manufacturing technology being adopted in various industries, its application is highly useful in manufacturing complex and custom geometries, as well as combining different parts of a particular component into one [1,2], but the presence of porosity is a concern that requires special attention. A decade ago, the state of the art was such that 1% porosity was not uncommon in AM [3], but as the technology evolved rapidly over the last few years this number has dropped to below 0.1% for ideal process parameters [4]. Nevertheless, even under ideal process parameters, many other influences can lead to porosity formation in small quantities, which cannot always be predicted. For this reason, process optimization and regular process quality control is required, for ensuring the best possible manufacturing with a minimal value of porosity.

To this end, the Archimedes method is a low-cost, simple, and widely adopted method in the AM field already [5,6]. With this method, the density is determined using equation (1) below which is based on a sample mass weighed in air as well as mass weighed in liquid such as distilled water or acetone, among others [7]. The variables in this equation are in such that ρ

represents the density being measured, w_a is the weight of the sample in air, w_l is the weight of the sample in liquid, and ρ_l is the density of the liquid which the sample is submerged in.

$$\rho = \frac{w_a}{(w_a - w_l)} \times \rho_l \dots (1)$$

X-ray computed tomography (XCT) is used more often in recent times especially for samples with porosity at levels below the detection limit of the Archimedes [8]. The Archimedes method is affected by permeability, and that will affect the results if the samples have pores that are accessible from the surface and if the pore size and surface tension allow these surface pores to be filled with liquid. XCT directly calculates the porosity volume or volume fractions based on images.
In the XCT method, in addition to the volume fraction, the size range and distribution of pores in 3D space can be determined which provides unique and useful information on the cause of porosity and its severity in the intended application [9].

The percentage porosity ($R_{porosity}$) is directly determined in the analysis of XCT results by quantifying the total volume of the pores and the total volume of the sample.

Gas pycnometry is also a non-destructive testing method which has been applied in the past for the purpose of porosity analysis on additively manufactured samples. This type of density measurement method is applied in such a way that the mass and volume of a sample are determined separately, where the volume is determined through displacement of inert gas such as Nitrogen or Helium. Similar to the Archimedes density method, gas pycnometry is an easily applied method [7]. It also penetrates surface pores or cracks and provides no information on such pores, evaluating only those pores not penetrated from the surface by the gas (i.e., closed pores).

The shortfall of the Archimedes density method is its limit to only determining bulk density relative to the liquid used during measurement. The percentage porosity is determined by comparing the measured density to that of the solid material, which limits accuracy and makes it impossible to individually assess localized pores [7]. In the gas pycnometry method, the gas fills the pores that are connected to the surface, excluding these pores from the measured volume. When comparing the measured density to that of the solid material, only the porosity percentage related to closed subsurface pores will be obtained [7].

Looking at a series of additively manufactured aluminium alloy samples with different types and quantities of intentionally seeded porosities, this study compares the three methods quantitatively, shedding light on their limitations, possible errors, and the benefits thereof.

2 Materials and methods

This study used Aluminium 6061-T6 cube samples of 10x10x10 mm that were printed with the same AM manufacturing technique and the process parameters for the samples were selected as shown in table 1 below, these samples were printed in two sets of seven cubes per set. Two samples in one set were not available for this study, making the total number of samples equal to twelve. The samples are labelled 1-7 and ·3-·7 (the second batch of samples has dots next to the numbering). The samples and XCT data used in this work were obtained from the PhD work in progress as reported in [10], where the effect of different process parameters on porosity reduction was studied.

Table 1. Process parameters used to print Al 6061-T6 samples, reproduced from a draft. PhD dissertation [10].

Set 1					
	Hatch Track		Contour Track		Additional Changes
Sample	**Laser Power (W)**	**Laser Scanning Speed (mm/s)**	**Laser Power (W)**	**Laser Scanning Speed (mm/s)**	
1	370	1300	370	300	
2	370	1170	370	270	
3	410	1170	410	270	
4	370	1300	370	300	Three contour tracks (default two)
5	370	1300	370	300	Four contour tracks (default two)
6	370	1300	370	300	Total remelt of each layer
7	370	1300	370	300	Reduced corridor value (reduced to 0.001 mm)
Set 2					
	Hatch Track		Contour Track		Additional Changes
Sample	**Laser Power (W)**	**Laser Scanning Speed (mm/s)**	**Laser Power (W)**	**Laser Scanning Speed (mm/s)**	
.1	370	1300	260	167	
.2	370	1170	260	150,3	
.3	410	1170	286	150,3	
.4	370	1300	260	167	Three contour tracks (default two)
.5	370	1300	260	167	Four contour tracks (default two)
.6	370	1300	260	167	Total remelt of each layer
.7	370	1300	260	167	Reduced corridor value (reduced to 0.001 mm)

2.1 Density measurements

The three methods that were used to determine the density of the samples in this study were the Archimedes method, XCT and gas pycnometry.

The gas pycnometer method was performed using an UltraPyc gas pycnometer 1200e, which is a true volume and density analyser system with Nitrogen gas, and it was operating at 5 Psi

(34,474 kPa). In each sample, three volume measurements were recorded and averaged for the purpose of determining density.
For Archimedes density measurements, a KERN ABT 120-5DM scale was used to determine the weight in air and weight in liquid per sample. This scale was calibrated before and during the process of measurements to ensure the accuracy of measurements. Several measurements were taken on different days, distilled water was used as the liquid during measurements. The temperature of the liquid was determined for the purpose of using accurate liquid density, and the densities of the samples were calculated at corresponding water temperatures. These density values were then used to compute the percentage of porosity. The values of the weights obtained during measurements were rounded off to four decimal places. Equation (1) was then used to determine the density of each sample.
For better statistics and confidence in the results, the Archimedes and gas pycnometer measurements were performed on different days under different conditions. This was in such a way that the temperature measured in the gas pycnometer system was from a minimum of 23,1 °C, while for the second time of performing the measurements the temperature in the gas pycnometer system was from a minimum of 21 °C, and the third set of measurements was performed at temperatures ranging from 22,9°C to 24,3°C Similarly for the Archimedes measurement method, the first set of measurements was performed with liquid at 17°C, while the second set of measurements was performed with liquid at 14°C, and the third day of measurements was carried out with liquid temperature of 13°C.
The XCT-based density method was applied in such a way that the samples were scanned with a micro-XCT system following the detailed procedure in [9]. Porosity analysis was achieved using Dragonfly software, using automatic Otsu thresholding and volume calculation.

3 Results and discussion

Table 2 shows the results of density for each sample obtained through the Archimedes method. Porosity was calculated using $R_{porosity} = \frac{\rho_0 - \rho}{\rho_0} \times 100\%$ with the theoretical density of the solid material chosen as $\rho_0 = 2.733005$ g/cc. Variation in density of the solid material may be caused by variation in the alloy composition used during the manufacturing process, or may vary due to the manufacturing process itself, i.e. some elements can be selectively vaporized.

Table 2. Summary of porosity results measured with the Archimedes and XCT methods.

Sample number	Number of runs measurement with Archimedes method per sample	Average density (g/cc) measured with Archimedes method	Average Archimedes porosity (%)	XCT porosity (%)
.1	7	2,723601	0,3441	0,03
.2	5	2,725452	0,2764	0,03
.3	5	2,726605	0,2342	0,19
.4	11	2,731567	0,0526	0,02
.5	5	2,729763	0,1186	0,03
.6	5	2,719758	0,4847	0,35
.7	5	2,723017	0,3655	0,03
3	7	2,721919	0,4057	0,14
4	7	2,723122	0,3616	0,12

5	7	2,727909	0,1865	0,13
6	5	2,729505	0,1281	0,06
7	5	2,721233	0,4307	0,11

Figure 1 below represents the density for the first three runs that were performed for each sample. All the runs were found to have density values that are above the mostly cited theoretical density (2,70 g/cc[11]), hence the highest density (sample .4) in this study was assumed to be the density of the solid material, and the percentage of porosity for each sample in Archimedes method was calculated relative to this density. Approximately 41% of the samples have less density in the first run as compared to the other runs. The differences between the second and the third run in each sample are very small, and this could be due to more precautions taken in the second and third runs, such as using a tweezer to hold the sample and using an Optica paper instead of the normal tissue paper to place the samples. These results indicate some variation is possible and special care should be taken during measurements to ensure repeatability.

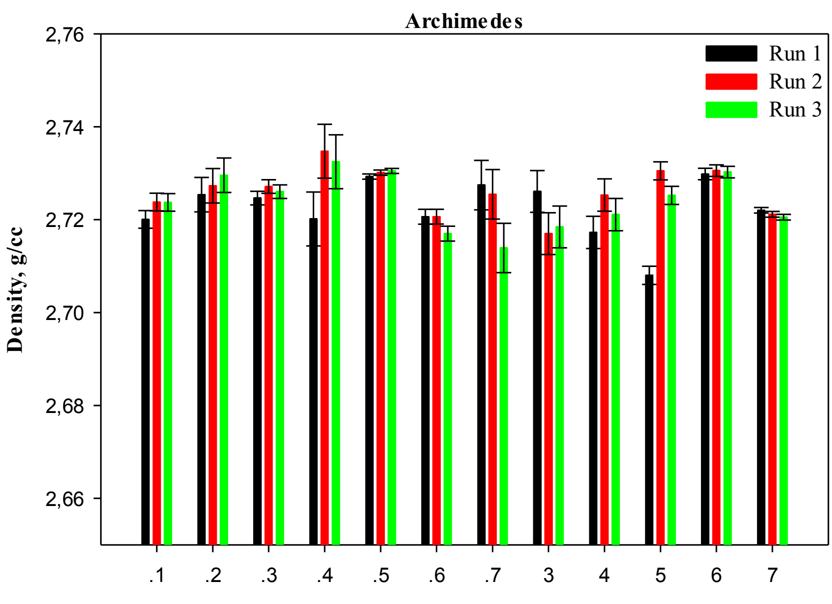

Fig. 1. Bar chart of 3 sets of density measurements obtained from the Archimedes method for 12 different samples of Al 6061-Ti6, with error bars.

Figure 2 represents the data obtained using the ULTRAPYC 1200e pycnometer that measures density by gas displacement. In the plot, * represents a value that was very different from the rest of the data and was thus not included. The used samples were cleaned and dried before the measurements, except for run 2. The sample chamber was calibrated with a traceable calibration sphere. For each result, the instrument took an average of 4 measurements and provided a standard deviation value. In run 2, for one sample (.4), we did multiple measurements in one experiment run and got a wide variation. The small error bars on runs 1 and 3 are what the machine provides for the average of 3 and 4 measurements respectively. The standard deviation of averages from 4 separate measurements of sample .4 was considered a measurement error during the plot of run 2. These results show that error bars given by the instrument underestimate variation in actual measured values. Additionally, in this work the pycnometer results give density values ranging from 2.15 g/cc to 2.75 g/cc which are equivalent to porosity values from 9%-18%. This is not reasonable for the given samples, considering the values obtained from XCT and Archimedes, and also considering pycnometry should provide values lower than Archimedes and XCT due to penetration of surface porosities, these values are considered erroneous. Gas pycnometer measurements with this instrument are not suitably accurate to compare with XCT measurements in this study and shows a further potential for experimental error in what is considered a simple and standard density measurement method.

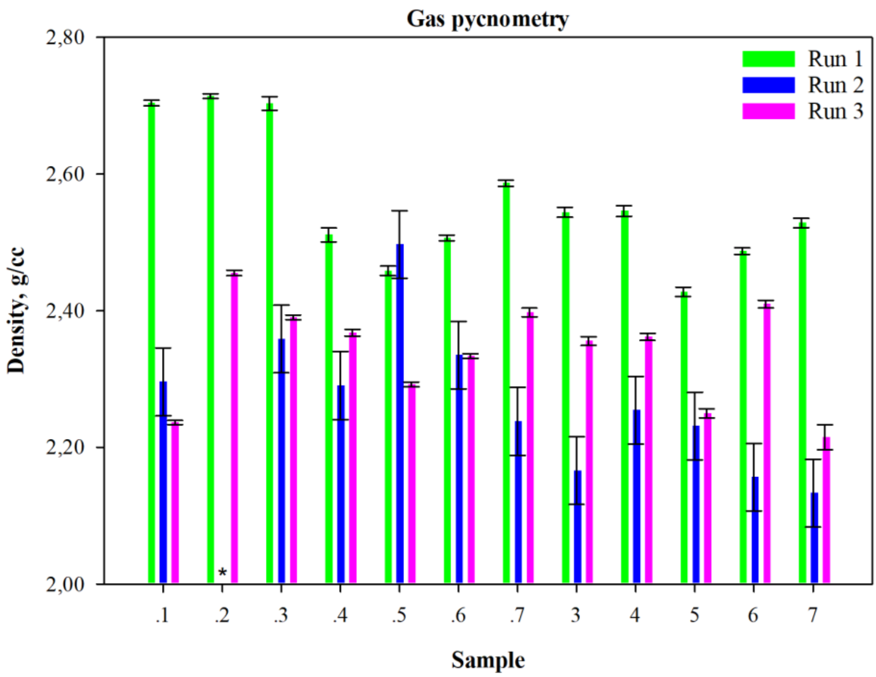

Fig. 2. Bar chart of 3 density measurements obtained from 6061-Ti6 gas pycnometer for 12 different samples of Al 6061-Ti6.

Figure 3 shows the porosity of samples obtained through the Archimedes method compared to the porosity percentage per sample that was obtained through the XCT method. The XCT measured porosity values are overall lower than Archimedes measurements, which is sensible considering the method uses images to quantify pores and the method is limited in resolution – some pores could potentially be missed in the analysis. However, the Archimedes density measurement produced unreliable values with some density values obtained being higher than the expected fully dense material. In order to resolve this, the maximum density obtained was used as a new reference for zero porosity and this resulted in the porosity values reported in Figure 3. With the assumption of density as described, the resulting porosity values in Fig 3 have a poor relative correlation with the XCT results, despite the highest porosity and lowest porosity values correlating to the same samples. In some cases, the Archimedes would show a high porosity while XCT shows low values, e.g., sample (.7). In other cases, a high XCT value would show a relatively low Archimedes value, e.g., sample 5. The poor correlation between the two methods indicates potential error on the Archimedes approach for such values of porosity less than 1%, which is typical for AM metals. Due to the limitation of the Archimedes method, it was unlikely that the Archimedes method would have given accurate absolute results for these low porosities below 1%. We have shown that the Archimedes method also does not produce relative values that agrees qualitatively with the trend in porosity as determined by XCT.

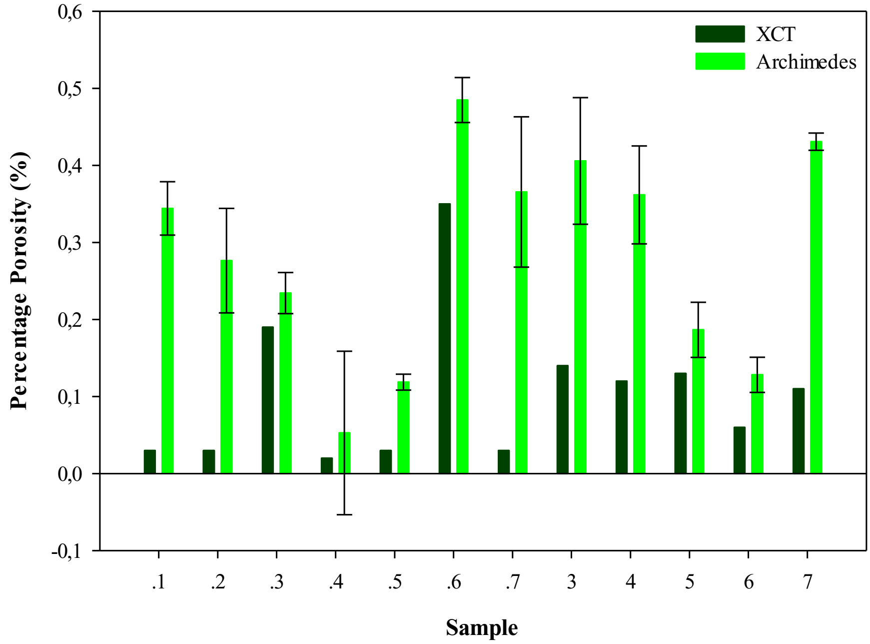

Fig. 3. Archimedes measured percentage porosity obtained from the average over all recorded experimental runs as shown in table 2 compared to XCT percentage porosity.

Some XCT images of samples with low porosity (0,03%), medium porosity (0,14%) and high porosity (0,35%) are shown in figures 4, 5 and 6 respectively. XCT data gives meaningful results, and the added advantage is the ability to observe the internal structure of the sample without having to cut through the sample. From this data one can observe also the size and distribution of the pores, pores that are localized under surface for example. Figure 4 shows moderate contour porosity and low bulk porosity with some few large pores. Most of the pores in figure 5 are gathered at the edges of the sample (known as contour porosity) with very low bulk porosity. Similarly, figure 6 shows high contour porosity, large pores and a higher total percentage porosity.

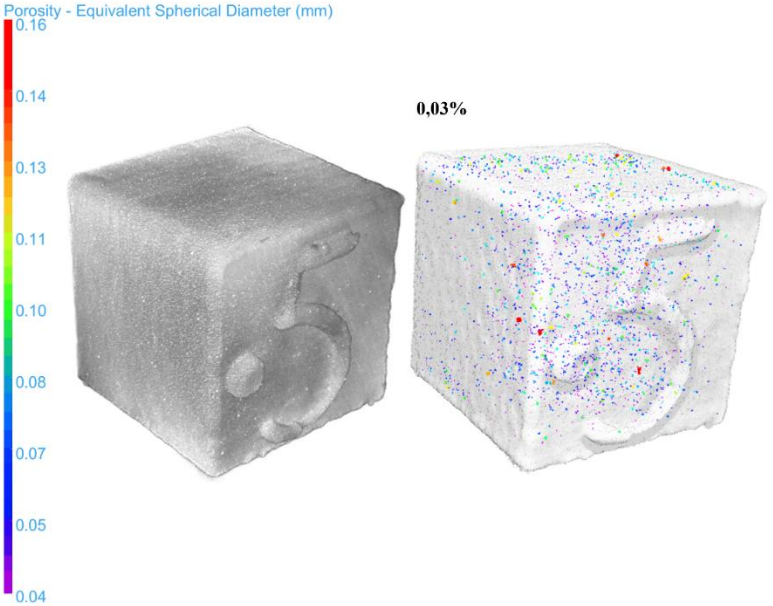

Fig. 4. XCT of LPBF Al 6061-Ti6 sample with low porosity of 0,03%. The image on the left shows the surface view and the image on the right shows a transparent view revealing the quantitative analysis (colour coding by equivalent spherical diameter) of porosity.

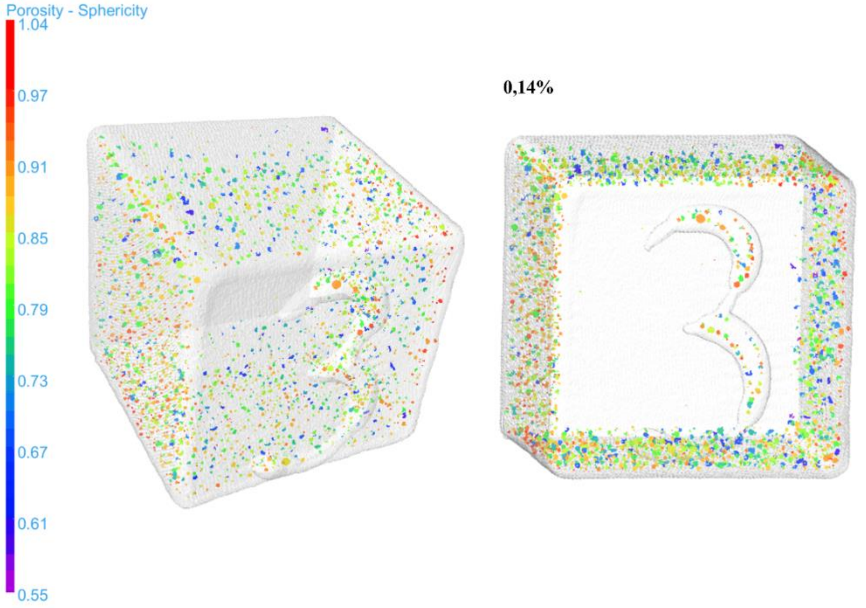

Fig. 5. Example of porosity analysis in an Al 6061-Ti6 cube produced by LPBF with total porosity of 0,14%. The porosity is visualized and quantified in this case according to sphericity from XCT data.

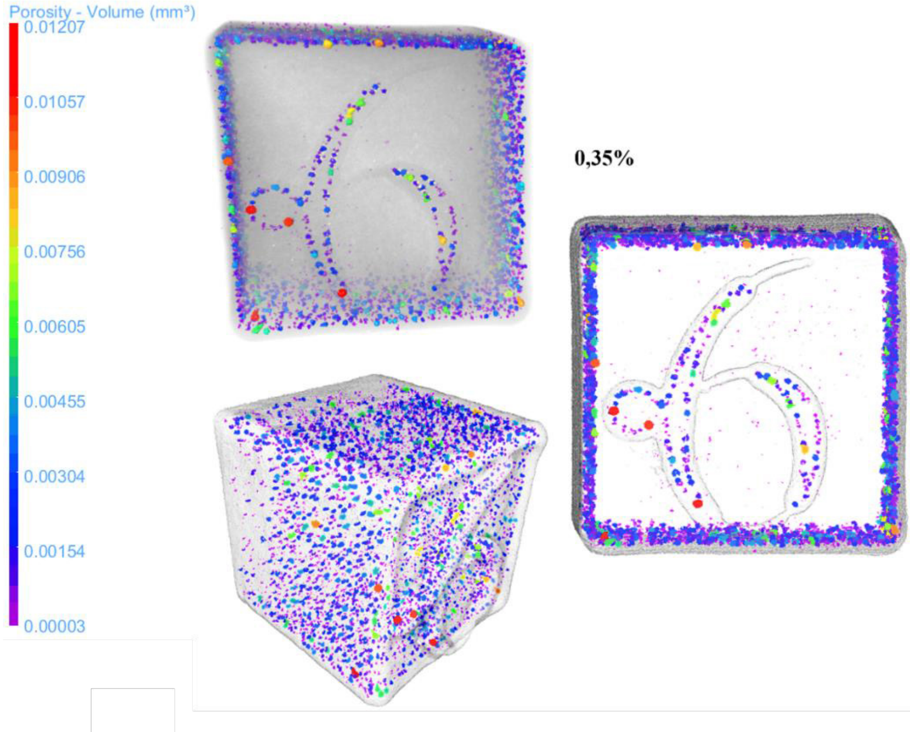

Fig. 6. Multiple views of a LPBF Al 6061-Ti6 cube with high porosity of 0,35%, shown with colour coding according to volume.

4 Conclusion

The direct comparison of Archimedes, XCT and gas pycnometry was performed for a series of additively manufactured Al6061-Ti6 coupon samples. The results can be summarized as follows:

This work helps to clarify the application range and limitations of each of these methods, using 10mm cubes of Al 6061-Ti6 manufactured by laser powder bed fusion.

- Density values determined using the Archimedes method were higher than the theoretical density cited in literature for this alloy.
- The Archimedes method was repeated on three different days with different results each time, and we have identified some sources of error that could be reduced by utilizing good sample handling and careful operation of the equipment.
- A modified Archimedes "maximum density" was necessary to calculate porosity fraction, to compare with XCT.
- Both Archimedes and XCT porosity values could be calculated, all below 0.5% but showing a poor correlation between the methods. Despite poor correlation, the sample with lowest porosity and the one with highest porosity is identified by both methods.

Differences between the methods in terms of correlation could not be explained in this work but could be speculated to be attributed to differences in alloy density due to process parameter changes (while an assumption is made of a fixed value in Archimedes). Further work could assist in better understanding these differences.

- Gas pycnometry was attempted but results were erroneous with values >8%.
- XCT porosity measurements were all lower than those obtained by Archimedes, which could be partly explained by lack of resolution and segmentation error due to the small pore sizes in images.
- XCT produced information of porosity distribution and pore sizes which was not possible by other methods.

Results indicate that for the range of porosity in this work (below 0.5%), XCT was more reliable and produced visual information on porosity that can be useful for process optimization. In the case of the Archimedes method, some sources of possible error have been identified. Future work should investigate wider ranges of porosity above 0.5% to further evaluate the suitable ranges for each method, and their specific limits to be considered.

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