On the uncertainty of porosity measurements of additively manufactured metal parts

Abstract

Equations and a methodology are presented for calculating the uncertainty of porosity measurements of additively manufactured (AM) metal samples. The bulk porosity of six metal AM samples are measured using Archimedes' principle; the density of two metal powders are measured using a gas pycnometer. The AM sample geometries include a 10×10×10 mm (H×W×D) cube, a 10×10 mm (Ø×H) cylinder and a bracket with a complex freeform geometry. Each geometry is fabricated in both titanium and cobalt chrome. The AM fabrication settings are modified to print samples with bulk porosities of less than 2%. The expanded uncertainties of the porosity measurements are evaluated and found to range from ±0.14% to ±0.18% (with 95% confidence). The dominant uncertainty contributor is found to be the measurement repeatability. The equations presented in this work can be used by researchers and technicians to identify the key uncertainty contributors in their own porosity measurements.

Keywords: porosity, measurement uncertainty, additive manufacturing, pycnometer, Archimedes' method, density, ASTM B311 – 17.

1 Introduction

The porosity of additively manufactured (AM) parts is a basic physical quantity that correlates strongly with other mechanical properties, such as: fatigue life, ultimate tensile strength, ductility and yield strength [1, 2, 3]. Pores act as stress concentrators that not only degrade the mechanical strength and fatigue life of a component, but also introduce variations in mechanical test results; the latter is due to variations in pore sizes and pore spatial distributions [2]. This variation in mechanical testing results hinders the qualification of AM components for critical applications. Additive manufacturing processes normally strive to achieve the minimum possible porosity in any alloy, hence the measurement of porosity of AM parts is of great importance.

The porosity of a part can be measured using a range of methods, such as: optical imaging of a polished cross-section [4, 5, 6], using Archimedes' principle [7, 8], and by X-ray computed tomography (XCT) [9, 10, 11]. This work is concerned with the use of Archimedes' principle for bulk porosity measurement, as per ASTM B311-17 [12]. Additive manufacturing techniques are now being optimised and reported to produce parts at 0.5%, 0.1%, or even 0.02% porosities [8, 13, 14]; these are very low levels of porosity, and without undertaking a rigorous uncertainty analysis it is questionable if these values can be trusted. Note that according to the Guide to the Expression of Uncertainty in Measurement [15], a measurement result is incomplete unless accompanied by a statement of uncertainty, where the uncertainty quantifies the doubt associated with a measurement result. To illustrate this point, consider the repeatability and reproducibility intervals of density measurements stated in ASTM B311-17, these are given as ±0.025 g/cm³ and ±0.03 g/cm³ respectively, propagating these uncertainties through a porosity calculation gives porosity uncertainties of ±0.30% to ±0.36% respectively for a dense AM alloy like Inconel (ρ=8.44

g/cm³), this is without considering the uncertainty of the powder density. Clearly a more detailed investigation is required as the uncertainty of the porosity values far exceeds some of the aforementioned levels of porosity claimed in the literature.

To the authors' best knowledge there are no exhaustive examples demonstrating how the uncertainty of porosity measurements can be evaluated for metal AM parts. In a very comprehensive piece of work, Slotwinski et al. [16] evaluated the repeatability of bulk AM porosity measurements, however, measurement bias, thermal effects and other uncertainty components were neglected. Another critical factor neglected in the work of Slotwinski et al. was the uncertainty of the AM powder density. An in-depth study on the uncertainty of porosity measurement for cast components was conducted by Taylor et al. [17], however, the work does not cover experimental evaluation of the uncertainty of the "porosity-free" material (equivalent to the AM powder density in this work); instead, these values were obtained from reference sources. Relying on reference data may be the only option available in some cases, but for the sake of completeness the uncertainty of the AM powder density is considered empirically in this work.

Equations and a method for evaluating the uncertainty of porosity measurements of AM parts are presented in this work. We expect the equations to be useful to lab technicians and researchers who want to know how uncertain their AM part porosity measurements are. This analysis can be used to make meaningful comparisons between porosity measurements conducted using different measurement methods and between different laboratories.

2 Methodology

The porosity of six AM samples are measured, three different sample geometries are considered and two different materials. Two simple geometries are considered, a 10×10×10 mm cube and a 10×10 mm cylinder (Ø×H). One complex geometry is considered, this being a structural bracket. The samples are shown in Figure 1. Each sample is fabricated from two powder feedstock, these being EOS MP1 (cobalt chrome) and EOS Ti64 (titanium). The fabrication parameters are modified to generate porosities of approximately 2%; the AM process parameters are given in Table 1.

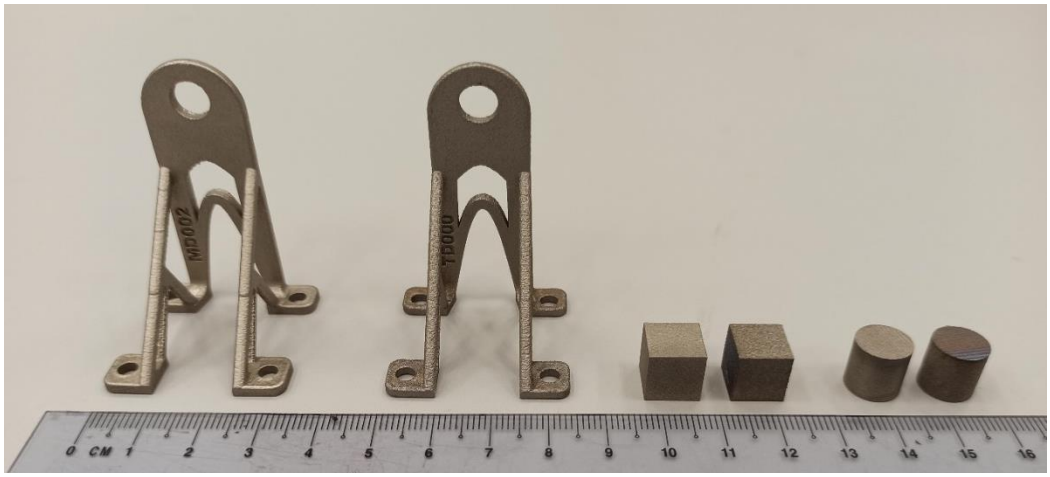

Figure 1: Photograph of the six AM samples that are measured in this work. From left to right: cobalt chrome bracket, titanium bracket, cobalt chrome cube, titanium cube, cobalt chrome cylinder, titanium cylinder.

Table 1: AM process parameters for each of the samples shown in Figure 1.

AM process parameter name (units)	Sample material and geometry	
	Cobalt chrome bracket, cube, cylinder	Titanium bracket, cube, cylinder
Print layer thickness (µm)	40	30
Laser power (W)	200	205
Scan speed (mm/s)	735.29	1000
Hatch distance (mm)	0.17	0.23
Volumetric energy density (J/mm^3)	40	29.71

To measure the porosity of an AM part requires the measurement of the part's bulk density, ρ_{bulk}, and the density of the powder material from which the part is fabricated, ρ_{powder}. The part's bulk density is its mass divided by its volume, where the volume includes space occupied by material and space occupied by voids. The percentage porosity of an AM part is calculated as:

$$P = 1 - \frac{\rho_{bulk}}{\rho_{powder}}. \tag{1}$$

Using equation 1, we can take partial derivatives to calculate the standard uncertainty of the porosity, u_P:

$$u_P = \sqrt{\left|\frac{\partial P}{\partial \rho_{bulk}}\right|^2 u_{\rho_{bulk}}^2 + \left|\frac{\partial P}{\partial \rho_{powder}}\right|^2 u_{\rho_{powder}}^2} \tag{2}$$

where:

$$\frac{\partial P}{\partial \rho_{bulk}} = -\frac{1}{\rho_{powder}} \tag{3}$$

$$\frac{\partial P}{\partial \rho_{powder}} = \frac{\rho_{bulk}}{\rho_{powder}^2}. \tag{4}$$

The term $u_{\rho_{powder}}$ is the standard uncertainty of the powder density and, $u_{\rho_{bulk}}$ is the standard uncertainty of the bulk density. Therefore, to evaluate the standard uncertainty of a porosity measurement we need four pieces of information: ρ_{bulk}, ρ_{powder}, $u_{\rho_{powder}}$, and $u_{\rho_{bulk}}$. In the sections that follow we will see how each of these terms can be estimated. We will first consider evaluating ρ_{powder} and $u_{\rho_{powder}}$, this requires measuring the volume and mass of the powder using a gas pycnometer and an analytical balance respectively. We will then consider evaluating ρ_{bulk} and $u_{\rho_{bulk}}$, this requires an analytical balance and using Archimedes' principle.

2.1 Measuring powder density

The following analysis assumes that the individual AM powder particles used do not contain closed pores. The literature [18] and our own in-house work show that closed pores can form in powder particles, but it is beyond the scope of this work to include this influence factor in the uncertainty analysis.

Powder density is calculated as:

$$\rho_{powder} = m_{powder}/V_{powder} \tag{5}$$

where m_{powder} is the mass of the powder and V_{powder} is the volume of the powder. The standard uncertainty of the powder density is calculated by taking partial derivatives of equation 5:

$$u^2_{\rho_{powder}} = \left|\frac{\partial \rho_{powder}}{\partial m_{powder}}\right|^2 u^2_{m_{powder}} + \left|\frac{\partial \rho_{powder}}{\partial V_{powder}}\right|^2 u^2_{V_{powder}} \tag{6}$$

where:

$$\frac{\partial \rho_{powder}}{\partial m_{powder}} = \frac{1}{V_{powder}} \tag{7}$$

$$\frac{\partial \rho_{powder}}{\partial V_{powder}} = -\frac{m_{powder}}{V^2_{powder}} \tag{8}$$

and the terms $u_{m_{powder}}$ and $u_{V_{powder}}$ are the standard uncertainty of the mass of the powder and the standard uncertainty of the volume of the powder respectively. The volume of a metal powder can be measured using a gas pycnometer. The mass of a metal powder can be measured using an analytical balance; we will first consider the volume measurement.

2.1.1 Measuring powder volume

In this work a gas (helium) pycnometer from Anton Paar GmbH, Austria, model Ultrapyc 5000 is used to measure the powder volume according to ASTM B923-21 [19].

The main uncertainty components in the powder volume measurement are: measurement bias, u_b, the measurement repeatability, u_{RE}, thermal expansion of the powder, u_{TE}, the uncertainty due to the error of the thermometer, u_T, the uncertainty due to the thermal expansion coefficient of the powder, u_{EC}, and the uncertainty of the material standards used to calibrate the instrument, u_{cal}. Hence the uncertainty of the powder volume measurements is calculated as:

$$u_{V_{powder}} = \sqrt{u^2_b + u^2_{RE} + u^2_{TE} + u^2_T + u^2_{EC} + u^2_{cal}}. \tag{9}$$

The measurement bias is estimated by measuring the volume of a calibrated sphere 5 times and calculating the mean error, b_V. The bias is assumed to have a uniform distribution hence it is divided by $\sqrt{3}$ [15], the standard uncertainty due to the measurement bias is therefore written:

$$u_b = \frac{b_V}{\sqrt{3}}. \quad (10)$$

Note that it is through the use of this calibrated sphere that the powder volume measurements can be claimed to be traceable to international standards.

The measurement repeatability is estimated as the standard deviation of five repeated measurements of the metal powder.

The measurement uncertainty due to thermal expansion of the powder is calculated as:

$$u_{TE} = \frac{\alpha V \Delta T}{\sqrt{3}} \quad (11)$$

where α is the volumetric thermal expansion coefficient of the powder, V is the nominal volume of the powder, and ΔT is the change in temperature during the measurement.

The uncertainty due to the thermometer error, u_T, is estimated to be:

$$u_T = \frac{\alpha V u_t}{\sqrt{3}} \quad (12)$$

Where u_t is the thermometer error according to the manufacturer's specification.

The uncertainty due to the thermal expansion coefficient, u_α, is estimated to be:

$$u_{EC} = \frac{u_\alpha V \Delta T}{\sqrt{3}} \quad (13)$$

The standard uncertainty due to the calibrated sphere used to evaluate the measurement bias of the pycnometer, u_{cal}, is evaluated as the expanded uncertainty from the calibration certificate, U_{cal}, divided by $k = 2$, where k is a coverage factor:

$$u_{cal} = \frac{U_{cal}}{2} \quad (14)$$

Thus all the terms in equation 9 can be evaluated giving us $u_{V_{powder}}$, the standard uncertainty of the powder volume.

2.1.2 Measuring powder mass

The powder mass is measured with an analytical balance from DKSH, Switzerland, model LabPRO-DT224C. The following uncertainty components are considered in the analysis: measurement bias, u_b, repeatability, u_{RE}, rounding error, u_{RA}, the uncertainty of the material standards used to calibrate the instrument, u_{cal}, thermal drift, u_{TD}, and thermometer error, u_T.

The measurement bias is estimated by measuring the mass of a calibrated weight 5 times and calculating the mean error, b_m. The bias is assumed to have a uniform distribution, the standard uncertainty due to the measurement bias is therefore written:

$$u_b = \frac{b_m}{\sqrt{3}}. \tag{15}$$

Through the use of this calibrated weight, the powder mass measurements can be claimed to be traceable to international standards.

The standard uncertainty due to the repeatability u_{RE} is evaluated as the standard deviation of three repeated measurements.

The standard uncertainty due to the resolution of the instrument u_{RA} is evaluated as the resolution of the instrument d divided by 2 and is converted from a limit to a standard uncertainty by dividing by $\sqrt{3}$:

$$u_{RA} = \frac{d}{2\sqrt{3}}. \tag{16}$$

The standard uncertainty due to the calibration weight u_{cal} is evaluated as the expanded uncertainty from the calibration certificate, U_{cal}, divided by $k = 2$, where k is a coverage factor:

$$u_{cal} = \frac{U_{cal}}{2} \tag{17}$$

The standard uncertainty due to the thermal drift of the balance u_{TD} is evaluated as the thermal sensitivity of the balance α_T multiplied by the temperature variation in the lab ΔT multiplied by the mass of the powder, m_{powder}. A rectangular distribution is assumed, hence the uncertainty due to thermal drift is:

$$u_{TD} = \frac{\alpha_T \Delta T m_{powder}}{\sqrt{3}} \tag{18}$$

The uncertainty due to the thermometer error, u_T, is estimated to be:

$$u_T = \frac{\alpha_T u_t m_{powder}}{\sqrt{3}} \tag{19}$$

Where u_t is the thermometer error according to the manufacturer's specification.

The standard uncertainty of the powder mass is therefore calculated as:

$$u_{m_{Powder}} = \sqrt{u_b^2 + u_{RE}^2 + u_{RA}^2 + u_{cal}^2 + u_{TD}^2 + u_T^2} \tag{20}$$

With reference to equation 6 we now have the all the required information to evaluate the uncertainty of the powder density, $u_{\rho_{powder}}$.

2.2 Measuring bulk density

The bulk densities of the AM parts are measured with ethanol as the weighing liquid and using Archimedes' principle described in ASTM B311-93 [12]:

$$\rho_{bulk} = \frac{m_a \rho_e}{m_a - m_e} \tag{21}$$

where m_a is the mass of the part in air, ρ_e is the density of ethanol and m_e is the mass of the part in ethanol.

The mass measurements are performed using an analytical balance from Sartorius Lab Instruments GmbH & Co., Germany, model KGSECURA224-1S.

The standard uncertainty of the bulk density is calculated by taking partial derivatives of equation 21:

$$u_{\rho_{bulk}}^2 = \left|\frac{\partial \rho_{bulk}}{\partial m_a}\right|^2 u_{m_a}^2 + \left|\frac{\partial \rho_{bulk}}{\partial m_e}\right|^2 u_{m_e}^2 + \left|\frac{\partial \rho_{bulk}}{\partial \rho_e}\right|^2 u_{\rho_e}^2 \tag{22}$$

where:

$$\frac{\partial \rho_{bulk}}{\partial m_a} = -\frac{\rho_e m_e}{(m_a - m_e)^2} \tag{23}$$

$$\frac{\partial \rho_{bulk}}{\partial m_e} = \frac{m_a \rho_e}{(m_a - m_e)^2} \tag{24}$$

$$\frac{\partial \rho_{bulk}}{\partial \rho_e} = \frac{m_a}{m_a - m_e} \tag{25}$$

and the terms u_{m_a}, u_{m_e} and u_{ρ_e} are the standard uncertainty of the mass of the part in air, the standard uncertainty of the mass of the part in ethanol and the standard uncertainty of the density of ethanol, respectively.

The uncertainty components u_{m_a} and u_{m_e} can be evaluated using the equations described in Section 2.1.2; the repeatability terms will correspond to the repeatability of the mass measurements in air and in ethanol.

The density of ethanol is influenced by temperature. To minimize this influence the temperature of the ethanol is tracked throughout the AM part bulk density measurements and the density value used in equation 21 is adjusted accordingly. There is still however an uncertainty component due to the accuracy of the thermometer. The accuracy of the thermometer is, u_t, which corresponds to a change in ethanol density of $\Delta\rho$. The uncertainty due to the temperature measurement of the ethanol is therefore:

$$u_{\rho_e} = \frac{\Delta \rho}{\sqrt{3}} \tag{26}$$

All the terms required to evaluate equation 22 have now been evaluated, hence the standard uncertainty of the part's bulk density can be calculated. This in turn completes the uncertainty analysis and allows the standard uncertainty of the porosity of the part, u_P, to be evaluated as per equation 2. The expanded uncertainty U is calculated as $U = k.u_P$, where K is a coverage factor where $k = 2$ for a confidence probability of 95%.

3 Results

The results of the uncertainty analysis are given in Tables 2 to 6. The standard uncertainty of the powder volume, powder mass and powder density are given in Table 2, 3 and 4 respectively. The uncertainty of the samples' bulk densities are given in Table 5. The uncertainty of the samples' porosities are given in Table 6.

Comparing Table 4 and 5 shows that the powder density measurements and the sample bulk density measurements have similar uncertainties; we suspect that this is because the dominant sources of uncertainty in both measurement procedures is the measurement repeatability. The repeatability of the sample bulk density measurements could be improved by removing the rough outer surface of the samples by means of surface post-processing such as machining, this will prevent surface undulations from trapping microscopic air pockets as the samples are immersed in ethanol.

Looking at Table 4, the powder density values specified by the vendor are given in brackets; note that the values agree with our independent measurements up to the stated resolution. Not all labs have access to a pycnometer for independent powder density measurements, we can therefore suggest that if practitioners wish to estimate the standard uncertainty of the powder density, they can do so by using the resolution of the powder density as specified by the vendor. For example, in the case of cobalt chrome the standard uncertainty of the powder density would be $\pm 0.05/\sqrt{3}$ g/cm^3 assuming a rectangular distribution, giving a value of ± 0.03 g/cm^3.

Table 2: Powder volume measurements and uncertainty.

Powder material	Volume (cm^3)	Standard uncertainty (cm^3)
Cobalt chrome	58.623	0.037
Titanium	57.175	0.035

Table 3: Powder mass measurements and uncertainty.

Powder material	Mass (g)	Standard uncertainty (g)
Cobalt chrome	485.9426	0.0041
Titanium	252.2285	0.0026

Table 4: Powder density measurements and uncertainty.

Powder material	Density (g/cm^3) (specified by vendor)	Standard uncertainty (g/cm^3)
Cobalt chrome	8.289 (8.3)	0.005
Titanium	4.412 (4.41)	0.003

Table 5: Sample bulk density measurements and uncertainty.

Material	Sample geometry	Density (g/cm^3)	Uncertainty (g/cm^3)
Cobalt chrome	Cube	8.128	0.003
	Cylinder	8.204	0.003
	Bracket	8.166	0.006
Titanium	Cube	4.375	0.002
	Cylinder	4.347	0.003
	Bracket	4.367	0.001

Table 6: Part porosity measurements and uncertainty.

Material	Sample geometry	Porosity (%)	Standard uncertainty (%)	Expanded uncertainty (%) $k = 2$
Cobalt chrome	Cube	1.94	0.07	0.14
	Cylinder	1.03	0.07	0.14
	Bracket	1.48	0.1	0.2
Titanium	Cube	0.82	0.08	0.16
	Cylinder	1.46	0.09	0.18
	Bracket	1.01	0.07	0.14

4 Discussion and conclusions

Using standard lab equipment, we have calculated the expanded uncertainty of porosity measurements of a set of small additively manufactured parts made from cobalt chrome and titanium to be in the range of ±0.14% to ±0.18% ($k = 2$). Many publications present porosity values lower than these uncertainties, and with no statement of measurement uncertainty; given that no measurement is complete unless accompanied by a statement of uncertainty, we would treat such measurements with caution and encourage AM researchers to conduct a basic uncertainty analysis as a bare minimum.

We have built upon the work of Slotwinski et al. [16] who evaluated the repeatability of bulk density measurements made using Archimedes' principle. In our work a much more comprehensive uncertainty budget has been provided for both bulk density measurements and powder density measurements. The standard uncertainty of bulk density measurements presented by Slotwinski et al. range from ±0.003 to ±0.194 g/cm^3 for samples with a nominal density of 8 g/cm^3, these values can be compared to the cobalt chrome density measurements presented in this work (Table 5) which range from ±0.003 to ±0.006 g/cm^3. Clearly the lower uncertainties provided by Slotwinski et al. are comparable to those presented here, whereas the larger uncertainties given by Slotwinski et al. are much larger than those presented here.

Looking back to the introduction of this work, reference was made to the repeatability and reproducibility intervals of density measurements stated in ASTM B311-17, comparing these values to those in Table 5 shows that the values stated in ASTM B311-17 are somewhat conservative (note that the values in Table 5 are standard uncertainties and must be multiplied by 2 in order to be comparable to the expanded uncertainties given in the standard). We can therefore suggest that if care is taken during the sample bulk density measurements, and similar equipment is used to that used in this work, then the lower levels of measurement uncertainty can be achieved than those stated in ASTM B311-17.

The methodology and equations presented in this work should be useful to researchers and technicians who wish to quantify how good (uncertain) their porosity measurements are; only then can informed engineering decisions be made, and only then can meaningful comparisons be made between measurements from other instruments.

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