Joint Special Interest Group meeting between euspen and ASPE
Advancing Precision in Additive Manufacturing
Inspire AG, St. Gallen, Switzerland 2021
www.euspen.eu

Analysis of metal powder geometrical characteristics influencing the quality of additively manufactured parts

Filippo Zanini[1], Nicola Buzzacchera[1], Simone Carmignato[1]

[1]*Department of Management and Engineering, University of Padova, Vicenza, Italy*

filippo.zanini@unipd.it, simone.carmignato@unipd.it

Abstract
Laser powder bed fusion (LPBF) is increasingly used to produce metal industrial components for high value-added sectors, such as aerospace, automotive and biomedical. However, mechanical and structural properties of LPBF parts are often hindered by the large quality variability, poor geometrical and dimensional accuracy, complex surface texture and low density. The quality of the feedstock material is an important aspect to be taken into account, as it significantly influences such possible issues. In particular, metal powder used in LPBF should have shape and size distribution designed to facilitate good flowability and packing behaviour, so that the final fabricated parts have acceptable density, surface finish and mechanical properties. The powder size and shape can be simultaneously measured from three-dimensional reconstructions obtained by X-ray computed tomography (CT). This work investigates the accuracy of these CT measurements of powder characteristics. X-ray CT is compared with other methods such as laser diffraction and scanning electron microscopy. The influence of powder characteristics evaluated by X-ray CT on the surface texture and density of LPBF parts is also discussed.

Laser powder bed fusion, X-ray computed tomography, metal powder, metrology, quality

1. Introduction

Among additive manufacturing (AM) technologies, laser powder bed fusion (LPBF) is increasingly used to produce metal industrial components for high value-added sectors, such as aerospace, automotive and biomedical [1]. However, mechanical and structural properties of AM parts are often hindered by the large quality variability, poor geometrical and dimensional accuracy, complex surface texture and low density [2]. In order to overcome these quality and performance issues, the major efforts are commonly oriented to the optimization of LPBF process parameters [3]. Another important aspect that is currently receiving increasing attention is the quality of the feedstock material [4,5]. For example, metal powder used in LPBF should have shape and size distribution designed to facilitate good flowability and packing behaviour, so that the final fabricated parts have acceptable density, surface finish and mechanical properties [6]. Two techniques that are used for the evaluation of such powder characteristics are scanning electron microscopy (SEM) and laser diffraction (LD) [7,8], the first being limited to the analysis of bi-dimensional (2D) images and the second limited to evaluate only the size distribution. X-ray computed tomography (CT) is a valid alternative that is currently under investigation for the powder characterization as it enables the measurement of both powder size and shape based on a three-dimensional (3D) reconstruction of powder particles [9]. This paper is part of a wider work aimed at enhancing the accuracy of CT metal powder measurements. In particular, X-ray CT measurements are compared here with characterizations obtained by the other methods. The possibility of using CT measurements of powder size and shape to effectively study the relationship of such powder characteristics with density and surface texture of LPBF fabricated parts is also discussed as future development of this research.

2. Materials and methods

This section presents the metal powders investigated in this work (Section 2.1) and the instruments and methodologies used to characterise them (Section 2.2).

2.1. Metal powder

Three powder batches of different materials were investigated in this work: Ti6Al4V, CuCrZr, and AlSi10. All batches are composed of powders recycled after being used, but not processed, in laser powder bed fusion process. The reasons behind the choice of analysing recovery materials are: (i) the importance of recycling for sustainable manufacturing [10], (ii) recycled powder allows reducing process-related costs and (iii) recycled powder is typically characterized by particles with more complex morphology with respect to newly-produced powder [11], with possible effects on the microstructure and mechanical behaviour of products [12]. This latter aspect is relevant for this work, because the measurement of particles with complex morphology is not trivial. For example, laser diffraction technique is limited to the evaluation of particle size distribution but does not give outcomes on particles shapes, and image analyses (e.g. using SEM) are commonly limited to a single side of powder particles, which might lead to results not representative of the actual particle morphology. On the contrary, X-ray CT measurements are potentially capable of obtaining complete geometrical information of powder particles including size and shape, but the measurement accuracy has not been thoroughly investigated so far.

2.2. Powder characterization

The powder batches described in Section 2.1 were characterized using three different methods: X-ray computed tomography, SEM image analysis and laser diffraction.

X-ray CT scans were conducted using a metrological X-ray CT system (Nikon Metrology MCT225; X-Tek Nikon Metrology, UK), equipped with micro-focus X-ray tube (minimum focal spot size equal to 3 µm) and 16-bit flat panel detector composed by 2000×2000 pixels. SEM analyses were performed with a FEI Quanta 400 scanning electron microscope (FEI Company, USA). Laser diffraction was executed with a Malvern Mastersizer 2000 instrument (Malvern Panalytical Ltd, UK).

To compare X-ray CT with SEM image analysis, a number of particles were attached to the top surface of a polymeric pillar, as illustrated in Figure 1a. The diameter of the top circular surface of the pillar was equal to 4 mm. The contained dimensions were designed to enable CT scans of the powder with the maximum achievable resolution [13]. In particular, the upper part of the pillar was CT scanned as schematized in Figure 1b. The pillar was then mounted on the SEM rotary table and powder particles were imaged in two different configurations. In the first configuration (Figure 1c), the table was kept horizontal and the powder was imaged from above; while in the second configuration (Figure 1d), the table was tilted by 60° to image the powder particles from a lateral view.

In order to compare X-ray CT with laser diffraction, a higher number of powder particles were inserted into a thin polymeric cylindrical pipe with external diameter of 4 mm. Also in this case, CT scans could be conducted with the highest achievable resolution.

The CT reconstructions were elaborated through the following steps: (i) local-adaptive surface determination using the analysis and visualization software VGStudio MAX 3.2 (Volume Graphics GmbH, Germany), (ii) binarization based on the determined surface, (iii) watershed algorithm to separate individual particles and (iv) size and shape computation based on volume and surface measurements. The last three steps were performed using the open-source software ImageJ (National Institutes of Health, USA).

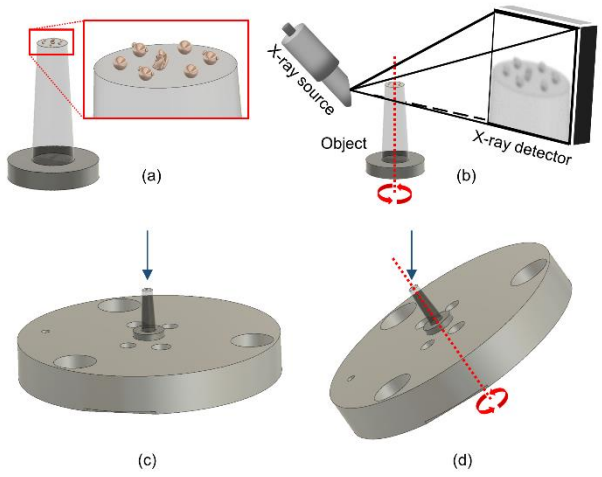

Figure 1. Schematic representations of: (a) powder particles disposed above a polymeric pillar for CT and SEM analyses, (b) CT scanning of powder particles and (c, d) configurations used for SEM analyses. Blue arrows represent the observation direction for both configurations. The top circular surface of the pillar has a diameter of 4 mm.

3. Comparison of characterization methods

This section presents the results obtained by comparing X-ray CT powder characterisation with SEM image analysis (Section 3.1) and with laser diffraction (Section 3.2).

3.1. X-ray CT - SEM

While X-ray CT enables the 3D reconstruction of powder geometry, the comparison with SEM images was performed in 2D, due to the 2D nature of SEM data. In particular, the CT reconstructed powder particles were firstly aligned on the screen according to the SEM point of view and then 2D images were taken. Figure 2 shows the comparison between CT and SEM concerning three particles taken as examples, one for each investigated material. The first one made of Ti6Al4V is spherical, the second one made of CuCrZr is more elongated, and the third one made of AlSi10 has a more intricate shape.

The overall shape of each particle is well represented by CT, even if the resolution is lower than SEM. The smallest details (e.g. the small particles attached to the powder surface) are instead not all visible.

SEM and CT images of a single particle were compared from different points of view, according to the configurations illustrated in Figure 1c and 1d.

Concerning the measurement of the equivalent diameter, percentage deviations below 5 % were found for Ti6AlV and AlSi10, and below 20 % for CuCrZr.

From these preliminary results, it was observed that dimensional deviations between CT and SEM were in all cases negative. This suggests a possible bias, probably due to the surface determination procedure. Further investigations are ongoing to better understand the cause of the bias and if it can be corrected to improve the accuracy of CT measurement results.

As far as the circularity (chosen here as morphological index) is concerned, deviations below 14 % were found for Ti6AlV and AlSi10, and below 30 % for CuCrZr. The higher deviations determined for CuCrZr can be due to the higher X-ray attenuation coefficient of this alloy, which leads to more intense image artefacts (e.g. beam hardening and metal artefacts [13]).

Figure 3 shows an example of three different SEM images of the same CuCrZr powder particle taken at three different angles: the image in Figure 3a is acquired from the top (as seen in Figure 1c), while images in Figure 3b and 3c are acquired laterally by tilting the SEM table (similarly to the scheme shown in Figure 1d). It can be observed that, for non-spherical particles, the SEM results are highly dependent from how the particle is positioned on the plane. The computed areas are, in fact, respectively equal to $3.8 \cdot 10^{-4}$ mm^2, $2.9 \cdot 10^{-4}$ mm^2 and $2.5 \cdot 10^{-4}$ mm^2. This confirms that a 3D analysis is to be preferred to better evaluate the actual dimension and shape of powder particles.

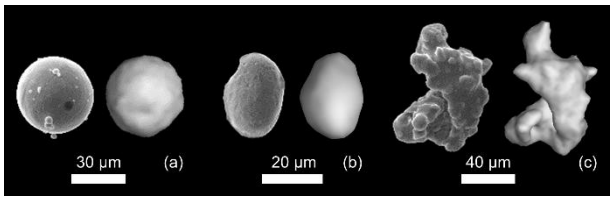

Figure 2. Comparison of SEM images (left) and CT images (right) for three different materials: Ti6Al4V (a), CuCrZr (b) and AlSi10 (c).

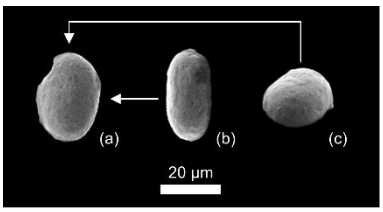

Figure 3. Comparison of SEM images of the same particle taken at different angles: from above (a) and laterally with directions shown by the white arrows (b and c).

3.2. X-ray CT – laser diffraction

Figure 4 shows the cumulative size distribution curves obtained in the case of CuCrZr powders by using both X-ray CT and laser diffraction. In addition, Table 1 reports D10, D50 and D90 values derived from Figure 4. Deviations up to 2.2 µm were found.

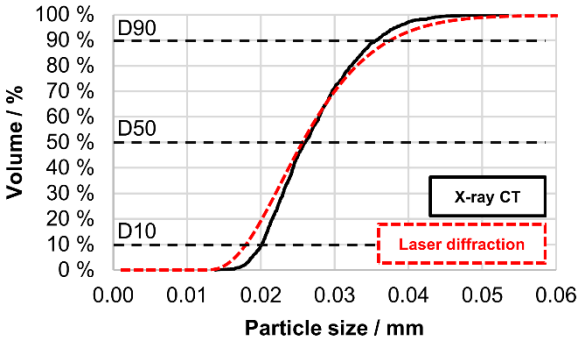

Figure 4. Cumulative size distribution curves obtained with X-ray CT and laser diffraction in the case of CuCrZr powder.

Table 1 D10, D50 and D90 values derived from the cumulative size distribution curves related to X-ray CT and laser diffraction.

	X-ray CT	Laser diffraction
D10 / µm	20.2	18.0
D50 / µm	26.0	25.6
D90 / µm	35.7	37.6

4. Conclusions and future works

This paper was focused on the comparison of CT powder measurements with powder analyses performed by scanning electron microscopy and laser diffraction. Results confirmed that X-ray CT is a promising solution, especially thanks to the possibility of conducting 3D measurements of both size and shape of powder particles, overcoming the limitations of the other investigated methods.

Even if the spatial resolution was not as good as in the SEM images, CT was proved to be capable of obtaining a good representation of the powder morphology. In addition, dimensional deviations were determined to be below 5 % for Ti6AlV and AlSi10, and below 20 % for CuCrZr. Since a bias was found (mean deviations had negative sign), additional investigations are planned to evaluate if the correction of systematic errors is appropriate to reduce deviations and improve measurement accuracy. The comparison with laser diffraction showed a maximum deviation of 2.2 µm, with regard to D10, D50 and D90 values.

Future works will further address the evaluation and improvement of the accuracy of CT metal powder characterisation. In addition, the improved results will be used to map the actual relation between powder geometrical characteristics and the quality of LPBF parts, particularly in terms of surface texture and density. In fact, as observable in the examples reported in Figure 5, the surface texture as well as the internal porosity – which can be measured non-destructively by X-ray CT – are strongly related to the powder characteristics. For example, the surface profile shown in Figure 5a is characterized by protrusions that have similar shapes and dimensions in the same order of magnitude than metal powder. Moreover, Figure 5b shows typical examples of lack-of-fusion voids, with non-totally melted powder entrapped inside.

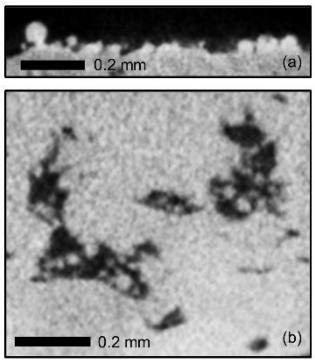

Figure 5. Examples of surface profile (a) and lack-of-fusion pores (b) of a sample fabricated by laser powder bed fusion.

References

[1] Leach, R., Carmignato, S. (Eds.). (2020). Precision Metal Additive Manufacturing. CRC Press.

[2] Gibson, I., Rosen, D., Stucker, B., Khorasani, M. (2021). Additive manufacturing technologies. New York: Springer.

[3] Oliveira, J. P., LaLonde, A. D., Ma, J. (2020). Processing parameters in laser powder bed fusion metal additive manufacturing. Materials Design, 193, 108762.

[4] Balbaa, M. A., Ghasemi, A., Fereiduni, E., Elbestawi, M. A., Jadhav, S. D., Kruth, J. P. (2021). Role of powder particle size on laser powder bed fusion processability of AlSi10mg alloy. Additive Manufacturing, 37, 101630.

[5] Brika, S. E., Letenneur, M., Dion, C. A., Brailovski, V. (2020). Influence of particle morphology and size distribution on the powder flowability and laser powder bed fusion manufacturability of Ti-6Al-4V alloy. Additive Manufacturing, 31, 100929.

[6] Vock, S., Klöden, B., Kirchner, A., Weißgärber, T., Kieback, B. (2019). Powders for powder bed fusion: a review. Progress in Additive Manufacturing, 1-15.

[7] Slotwinski, J. A., Garboczi, E. J., Stutzman, P. E., Ferraris, C. F., Watson, S. S., Peltz, M. A. (2014). Characterization of metal powders used for additive manufacturing. Journal of research of the National Institute of Standards and Technology, 119, 460.

[8] Grubbs, J., Tsaknopoulos, K., Massar, C., Young, B., O'Connell, A., Walde, C., ... Cote, D. (2021). Comparison of laser diffraction and image analysis techniques for particle size-shape characterization in additive manufacturing applications. Powder Technology.

[9] Du Plessis, A., Sperling, P., Beerlink, A., Du Preez, W. B., Le Roux, S. G. (2018). Standard method for microCT-based additive manufacturing quality control 4: Metal powder analysis. MethodsX, 5, 1336-1345.

[10] Ford, S., Despeisse, M. (2016). Additive manufacturing and sustainability: an exploratory study of the advantages and challenges. Journal of cleaner Production, 137, 1573-1587.

[11] Mohd Yusuf, S., Choo, E., Gao, N. (2020). Comparison between Virgin and Recycled 316L SS and AlSi10Mg Powders Used for Laser Powder Bed Fusion Additive Manufacturing. Metals, 10(12), 1625.

[12] Cordova L., Bor T., de Smit M., Carmignato S., Campos M., Tinga T. (2020), Effects of powder reuse on the microstructure and mechanical behaviour of Al–Mg–Sc–Zr alloy processed by laser powder bed fusion (LPBF). Additive Manufacturing, 36, 101625.

[13] Zanini, F., Carmignato, S. (2017). Two-spheres method for evaluating the metrological structural resolution in dimensional computed tomography. Measurement Science and Technology, 28(11), 114002.

[14] Carmignato, S., Dewulf, W., Leach, R. (Eds.). (2018). Industrial X-ray computed tomography. Cham: Springer International Publishing.