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Uncertainty of particle size measurements using dynamic image analysis $\stackrel{\scriptscriptstyle \ensuremath{\boxtimes}}{\sim}$



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A R T I C L E I N F O A B S T R A C T Metal powder particle size distribution (PSD) is a critical factor affecting powder layer density and uniformity in additive manufacturing processes. Among various existing measurement methods, dynamic image analysis (DIA) instruments are very appealing for measuring PSD. However, the 'black box' nature and complex measurement process inherent to DIA make quantification of uncertainty challenging. A method to establish DIA-based measurement uncertainty based on calibrated powder samples via a scanning electron microscope is described. Uncertainty analysis was performed taking into

calibrated sample and the measured sample.

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1. Introduction

Metal-based additive manufacturing (AM) has great industrial potential in a wide range of industries including aerospace, automotive, and biomedicine. However, to realize this potential, it must demonstrate the capability to consistently fabricate high quality industrial parts. Metal-based AM is sensitive to metal powder quality [1,2]. Poor powder quality can produce defects in the end part including pores, cracks, inclusions, residual stresses. and suboptimal surface roughness. Particle size and particle size distribution (PSD) of the metal powder used in additive manufacturing are among the most influential factors. For example, proper flowability of metal powder during the AM process depends on particle size, shape, and PSD among various other environmental conditions (e.g., moisture) and AM machine properties (e.g., dynamic characteristic of recoating mechanism). In general, smooth, spherical particles tend to flow more easily and therefore produce uniform, high-density layers. Additionally, maximum packing density of particles in a formed layer is achieved with a specific PSD that includes both coarse and fine particles. While finer particles increase density by filling the gaps

https://doi.org/10.1016/j.cirp.2019.04.075 0007-8506/© 2019 Published by Elsevier Ltd on behalf of CIRP. left by larger ones, they are more susceptible to Van der Waals forces and create a more cohesive powder which can lead to poorly spread layers [3].

account uncertainties associated with the calibration of the sample as well as non-similarities of the

While a variety of industrially common particle size measurement methods exist such as sieve analysis, laser light scattering, static image analysis, and dynamic image analysis (DIA), there is a lack of information detailing the uncertainty of these methods, particularly when they are applied to sizing gas-atomized metal powders like those used in AM. Furthermore, when common sizing methods were compared, they produced significantly different results [4]. The DIA method is attractive for its relative low cost, measurement speed, and ability to provide morphological information. In its most basic form, the DIA measurement principle, involves establishing contours of moving particles by capturing their silhouettes (shadows) using a high-speed camera following the computational theory developed in the 1990s [5]. Using either a liquid or gaseous dispersion, the DIA instrument conveys a stream of sample particles past a light source, and images are taken of the projected shadows of the particles. The images undergo processing and eventually are converted to grey-scale images so the two-dimensional (2D) morphology and various size metrics of the particle population can be extracted.

Although the basic operating principle is well established and standardized [5,6], as shown in Fig. 1, the particular means employed can be complex and can vary depending on the instrument manufacturer. For example, since the powder is in a

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Fig. 1. (a) Flow diagram for typical DIA method: 1. dispersed particles 2. device for control of particle motion 3. measurement volume 4. light source 5. optical system 6. depth of field 7. image capture device 8. image analyzer and 9. PSD display. (b) DIA Schematic.: 1. light source 2. camera and 3. measurement volume. (adapted from Ref. [6]).

dynamic state, both the illumination and the exposure duration will impact what the image sensor transduces into a signal. This is further complicated by the fact that the powder is entrained by a gas or liquid, and the drag force will produce a gradient of particle speeds dependent on the size and shape of the particles in the flow.

While this potential source of measurement error is, to some extent, alleviated by a compensating algorithm, the proprietary nature of commercially available DIA instruments prevents the user from knowing the means or extent of the compensation. This velocity gradient is just one of the sources of uncertainty.

The sources of uncertainty in a DIA-based measurement [6] include optical distortion error (pincushion, barrel, etc.), depth of field of camera focus/defocus algorithm, motion blur (particle velocity and integration time), particle velocity as a function of size, contaminations, overlapping of particles, incomplete imaging of particles (particles at edge of image), particle orientation, and insufficient number of particles measured. Furthermore, during image analysis the sources of uncertainty can be from pixel size calibration error, background correction (for non-uniformity of illumination), particle edge determination (thresholding, edge detection), and particle size determination along different orientations. Consequently, there is an urgent need to carry out complete and rigorous uncertainty analysis of the particle size and PSD measurements using the DIA method. However, due to the proprietary nature of image acquisition, processing, and analysis used by commercial DIA instruments, quantification of uncertainty is challenging. In such cases, calibrated artifacts are used to assess measurement uncertainty [7]. But uncertainty associated with the differences between the calibrated artifacts (e.g., spherical glass beads) and metal powders used in AM (with complex shapes and material optical properties) makes this approach impractical. Therefore, we used a variation of this approach to establish DIAbased PSD measurement uncertainty based on AM metal powder samples calibrated via a scanning electron microscope (SEM).

2. Materials and methods

In this study, a commercially available 17-4 stainless steel argon-atomized powder was used. A commercial DIA system and an SEM were used for PSD measurements with the measurand of equivalent circular area diameter (ECAD). The typical PSD specification (reported in AM powder certification sheets) is given by the cumulative frequency distribution as a function of the size of the particles. However, PSDs are generally reported as a volume fraction, rather than as a number fraction, of the particles. To generate such PSD functions, ECAD values for particles were first converted to volume assuming a sphere with the given diameter. Particle volumes were binned with respect to their ECAD values. Volume fraction-based PSDs were then calculated as the sum of the volumes for each size bin divided by the sum of all particle volumes. The cumulative frequency distribution function generated from these bins is used to determine sizes corresponding to the 10th, 50th, and 90th percentiles, which are labelled as D10, D50, and D90, respectively. Therefore, the final uncertainty estimates in

DIA-based ECAD measurements at these three levels were calculated from the derived uncertainties in corresponding frequencies by Monte Carlo simulations.

Multiple powder samples with nominally identical PSDs and morphologies representative of the bulk metal AM powder were prepared using a rotary riffler [8].



Fig. 2. (a) Illustration of SEM sample preparation apparatus (not to scale) and, (b) a typical SEM micrograph of the stainless-steel metal particles. Horizontal full width of the SEM image is 1.06 mm.

2.1. SEM sample preparation

A prerequisite for a proper PSD analysis by an SEM is a welldispersed powder sample. Each dispersion should have a sufficient number of particles to be statistically significant while minimizing contact between particles. The SEM sample preparation apparatus used for the metal powder in this work is illustrated in Fig. 2(a). It includes a carbon tape adhered on a metal substrate covered with a glass beaker, a curved-tip glass eye-dropper holding the powder sample, and a manually operated air bulb to blow the powder sample through the eye-dropper onto the carbon tape. All components were sealed with rubber joint compound.

The manually operated air bulb fitted with a check valve to prevent backflow was forcefully squeezed, and then the pressure on the air bulb was released slowly (over about 20 s) while using one hand to stabilize the beaker. This air bulb dispersion procedure was repeated three to five times to ensure no powder was left in the curved tip and all powder was dispersed onto the SEM substrate. Following this procedure, two SEM samples of dimensions (50 mm \times 50 mm) were prepared for SEM imaging. Fig. 2(b) is an example of a typical SEM micrograph showing uniformly dispersed metal particles.

2.2. SEM data acquisition

SEM images were acquired in an automated mode, without overlap, and using the SEM's annular backscattered electron detector. Optimum imaging parameters were determined by trial and error as a beam landing energy of 15 keV, an electron beam current of 8.9 nA, and a dwell time of 2 μ s per pixel. To increase the depth of field and minimize defocus problems, the working distance was set at 9.8 mm. The horizontal full width, set at 1.06 mm and resulting in pixel size of 0.518 μ m, was considered sufficient for imaging powder particles with sizes of tens of micrometers. The choice of dwell time and pixel size was also a trade-off between the number of SEM images and the time required to acquire them.

Eqs. (1-4) from the reference [9] were used to determine the minimum number of particles necessary for achieving at least a 95% probability level (*P*) with no more than 3% relative error.

$$n = \omega \delta^{-2},\tag{1}$$

$$\omega = p^2 \alpha^2 s^2 (2c^2 s^2 + 1), \tag{2}$$

$$\phi(-|p|) = (1-P)/2, \tag{3}$$

$$c = \beta + \alpha/2, \tag{4}$$

where *n* is the minimum number of particles and δ is relative error. In Eqs. 2–4 *p*, an intermediate parameter, is derived from a cumulative distribution function, ϕ , of the standard normal

distribution. α and β are constants defined from the desired metric (e.g., mass median or Sauter diameter) and *s* is the standard deviation of the population, which can be derived from the 84th and 50th percentile assuming log-normal distribution by number (i.e., $s = x_{84,0}/x_{50,0}$).

We found that 59 866 particles were sufficient for achieving a 95% probability level and 3% relative error in measuring the mass median diameter of this argon atomized metal powder. In this study, two SEM samples, each 50 mm \times 50 mm, were imaged, and in total 4575 SEM images were acquired; this acquisition strategy yielded SEM images of more than 69 000 particles.

2.3. SEM image processing

Each of the acquired SEM images underwent the same processing. First, the image was converted to an 8-bit grey-scale format which lowers the computational cost without sacrificing image information content. A Kuwahara filter [10] with window size of 5 pixels was then used to smooth the intra-particle features while maintaining sharp contrast at the particle's edges. The image was then binarized with a lower threshold value of 13, which was empirically found to maximize particle detection while not including the SEM tape's surface features. An open-source software application [11] was used to analyze each of the particles, extracting various metrics for each individual particle such as area, Feret diameter, and roundness. To avoid uncertainty induced by stitching, non-overlapping images were taken and any particle touching the edge of a frame was not considered. Since larger particles have a higher probability of being vetoed by the edgeexclusion algorithm, the measured PSD may be significantly skewed. This bias was accounted for via a correction as shown in Eq. (5), where P_i is the probability of a particle *i* having a horizontal Feret diameter of x_{F1} and a vertical Feret diameter of x_{F2} in a rectangular measurement frame of size Z_1 by Z_2 [9]. In this application, there was no preferential orientation found, and Eq. (5) was therefore simplified to use the same metric for x_{F1} and x_{F2} . The population of each size category is divided by its calculated probability, P_i , where

$$P_i = \frac{(Z_1 - x_{F1})(Z_2 - x_{F2})}{Z_1 Z_2}.$$
(5)

2.4. SEM calibration uncertainty

To eliminate systematic error in the SEM image processing, we used a reference material sample for pixel size (magnification) calibration [12]. Further, we considered a few relevant sources of uncertainty in our SEM measurements such as those related to (a) choice of pixel size, (b) focus and, (c) unevenness of the sample. To quantify these uncertainties, the same region of dispersed powder (5350 particles) was analyzed using two different values of these parameters, chosen over a range that exaggerates the practical limits for SEM imaging of these particles. Specifically, we acquired SEM images with pixel sizes of 0.518 μ m and 0.259 μ m and focus lengths of 9.8 mm and 10.6 mm. To estimate the effect of unevenness of the sample, we acquired SEM images at stage height Z = 9.8 mm and Z = 8.8 mm. We compared the two frequency distributions resulting from each set of parameters. The differences



Fig. 3. Standard uncertainty from each of the considered three sources in SEM measurements.

in bin frequencies (% by volume) for each parameter were considered as a full range of variation. Standard uncertainties were calculated from these range values for each size and each parameter assuming variations have uniform distribution (Fig. 3).

In addition to the above-mentioned sources of uncertainty, the most important image processing uncertainty to address is random variation in pixel segmentation, i.e., determination of the location in the image of the boundary between the particle foreground and the substrate background. The SEM image acquisition strategy was designed specifically to minimize this uncertainty by selecting the backscattered electron detector operated in composition mode (not topographic mode) instead of the more commonly used Everhart-Thornley detector, thus relying on a contrast mechanism based on the large difference in atomic number of the metal particles compared to the low-Z carbon substrate. Further, the gain and offset of the detector amplifier were adjusted to deliberately lower the dynamic range in the image, thus acquiring images in hardware close to the ideal binarized data needed for input into the particle shape and size analysis software. While this approach sacrifices fine spatial detail in the image of the particle surface (not needed in this study), it has the advantage of greatly reducing the sensitivity of the segmentation result to random variations and the choice of particular parameters and hyperparameters in the image processing chain. Therefore, uncertainty associated with image processing is considered negligible compared to the uncertainties shown in Fig. 3. Finally, the calibration uncertainty (u_{cal}) of SEM measurements in terms of frequency (% by volume) with respect to each bin was calculated by summation of all uncertainty components (Fig. 3) in quadrature.

3. Results and discussion

SEM results were considered as the calibrated artifact (i.e., reference) to determine error (bias) and combined standard uncertainty associated with DIA measurement results for each bin.

3.1. Results from a DIA instrument

Powder samples riffled from the same batch were measured in the DIA instrument taking over 275 images per second with 20 mm field of view and $1 \,\mu$ m resolution, while the particles were dispersed within the measurement volume. Two types of powder dispersion methods were used in the DIA instrument: dry dispersion, using a vibratory feeding system passing the particles through the viewport by a combination of gravity and pressurized



Fig. 4. PSD results from a DIA instrument using samples from the same batch. (a) average results from the dry and wet module and, (b) standard uncertainty in frequency (% by volume) results from both modules.

air, and wet dispersion, using recirculating liquid suspension of particles in the viewport. Fig. 4(a) shows the PSD, which is the average of three repetitions each of dry and wet measurements. Fig. 4(b) shows standard uncertainties (u_{meas}), in frequency (% by volume), associated with the measurement procedure (repeatability) of DIA with respect to each bin [7].

DIA results were further analyzed by calculating the difference between each dispersion type (wet and dry) for each ECAD bin of 1 μ m width. The results are shown in Fig. 4(a). While both dispersion methods produced reasonable repeatability (an average standard uncertainty of frequencies across all size bins was 0.02% for wet dispersion and 0.06% for dry dispersion), the wet module clearly provided a more repeatable measurement, as evident from Fig. 4(b), especially for the 25 μ m to 35 μ m and 42 μ m to 50 μ m size ranges, where the dry module's repeatability was worst. Investigation of the causes of this increase in variability at these sizes (e.g., drag induced velocity variability, agglomeration of certain sizes) is a subject of a future study.

3.2. Uncertainty analysis of DIA measurements

The SEM data with calibration uncertainty u_{cal} , wet-DIA data, and the calculated error with respect to SEM are shown, for example, in Fig. 5(a). Fig. 5(b) shows the SEM and both DIA data



Fig. 5. Error analysis of DIA-dry and DIA-wet module. (a) error in frequency of particles with respect to particle sizes using wet module and, (b) cumulative frequency (% by volume) of results from all three methods.

sets in a cumulative plot with common percentiles noted. As illustrated earlier (see Fig. 4(a)), both dry and wet dispersion modules have produced very similar measurement results, and therefore their errors trend similarly. Fig. 5(b) shows the smallest deviation in the cumulative plot from the calibrated SEM data was at or below the D10 size, with this error increasing through the D50 and reaching a maximum around 45 μ m (below the D90 size).

The combined standard uncertainty, u_{DIA} , of DIA-based measurements was calculated based on guidelines provided in Ref. [7] and using Eq. (6).

$$u_{\rm DIA} = \sqrt{(u_{\rm cal})^2 + (u_{\rm meas})^2 + (u_{\rm bias})^2 + (u_{\rm sample})^2},$$
 (6)

where u_{cal} is the uncertainty associated with the SEM calibrated sample, $u_{\rm meas}$ is the uncertainty associated with the DIA-based measurement procedure (see Fig. 4b), u_{bias} is the uncertainty associated with the systematic error between SEM and DIA measurement results, and u_{sample} is the uncertainty associated with the differences between calibrated sample and the actual measured sample. Since SEM measurements are used as the calibration of the artifact, u_{bias} is calculated as the standard deviation of the mean of DIA measurements. Furthermore, we considered u_{sample} as negligible since we used the same batch of powder for all measurements and sampling was done via a riffling operation. To calculate the combined standard uncertainty of DIA (u_{DIA}) of each of the conventionally reported percentiles (i.e., D10, D50, and D90), a Monte Carlo simulation with 500 000 iterations was used. First, we calculated the u_{DIA} in terms of frequency for each bin. Then, a random frequency value was selected between the upper and lower limits of each uncertainty band. This was done for each bin that was used in calculating the respective percentile. After each iteration, the bin's frequency values were normalized to ensure they sum to unity.

Finally, the percentiles were calculated using the unique combination of randomly selected frequency values. Since it was assumed that the uncertainty is uniformly distributed, the range between the nominal percentile value and the highest percentile, which includes the randomly generated frequencies, is recorded over all iterations. This is the uncertainty of the respective percentiles as shown in Table 1.

Table 1

Summary of expanded (k = 2) uncertainty and systematic error in volume fraction percentiles of ECAD for both DIA dispersion methods.

	D10 (µm)	D50 (µm)	D90 (µm)
SEM Sys. error $\pm 2u_{DIA}$ [Wet] Sys. error $\pm 2u_{DIA}$ [Dry]	$\begin{array}{c} 21.77 \\ 0.31 \pm 0.58 \\ 0.06 \pm 0.58 \end{array}$	$\begin{array}{c} 33.51 \\ 1.93 \pm 0.42 \\ 2.13 \pm 0.50 \end{array}$	$\begin{array}{c} 50.86 \\ 5.45 \pm 0.76 \\ 5.04 \pm 0.82 \end{array}$

4. Summary

In this study, we showed a novel methodology of using PSD data from SEM as a calibrated artifact to investigate uncertainty in DIAbased measurements. This methodology involves converting uncertainty of size measurements into uncertainty in frequencies and then back to uncertainty of size corresponding to specific percentiles. A novel method of sample preparation was developed for SEM imaging of metal powder. To eliminate the uncertainty associated with the differences of calibrated artifact and the measured sample, samples with identical characteristics representing the bulk powder properties were obtained via well-established riffling methods. Both error and uncertainty of DIA-based ECAD measurements were calculated using a Monte Carlo simulation and calibrated SEM data. Despite the proprietary nature of commercially available DIA systems, we have demonstrated a useful methodology of PSD measurement uncertainty evaluation based on AM metal powder samples calibrated via a SEM. Future study will involve reducing uncertainty in SEM measurements (u_{cal}).

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