Contents lists available at ScienceDirect

# Powder Technology

journal homepage: www.elsevier.com/locate/powtec

# A comparison of particle size distribution and morphology data acquired using lab-based and commercially available techniques: Application to stainless steel powder

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# ARTICLE INFO

Article history: Received 21 July 2021 Received in revised form 29 October 2021 Accepted 30 October 2021 Available online 17 November 2021

Keywords: Particle size distribution Morphology Gas atomized metal powder Metal Powder Additive Manufacturing

## ABSTRACT

The particle size distribution (PSD) and particle morphology of metal powders undoubtedly affects the quality of parts produced by additive manufacturing (AM). It is, therefore, crucial to accurately know the PSD and morphology of these powders. There exist several measurement techniques for these quantities, but since each method is based on different physical phenomena, which are sensitive to different aspects of a particle's shape and size, it is unclear how the measured PSDs and morphology compare to one another. In this study, five different techniques are used: sieve analysis, dynamic imaging analysis, laser diffraction analysis, X-ray computed tomography (XCT), and scanning electron microscopy. The first three are commonly used in the powder metallurgy field while the last two are laboratory-based tools capable of providing robust size and shape data. Nominally identical samples of stainless-steel powders were produced via riffling, and each technique was employed to measure effectively the same PSD and in some cases the morphology. In this paper, the differences among these measurement techniques are explored by a comparison of the measured results. Besides the random variations of the various measurement processes, the difference in the results is partly due to the fact that the particles are not perfectly spherical and that there are many multi-particles present. Each of these affect the principle of each method differently. Three-dimensional particle morphology and size data collected via XCT is used to provide insight regarding the discrepancies among other sizing and morphology measurement techniques. (Official contribution of the National Institute of Standards and Technology; not subject to copyright in the United

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1. Introduction

Complex three-dimensional (3D) parts made via the laser powder bed fusion (LPBF) additive manufacturing (AM) process have tremendous potential in different industries ranging from aerospace to biomedicine [1]. However, the full benefits of the LPBF-AM process to consistently fabricate high-quality parts are not yet completely realized across the manufacturing industry. It has been understood that the size and morphology of the powders used in additive manufacturing play crucial roles in the performance of the process and in turn the quality

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of the final part [2], [3]. However, there are no AM-specific standard methods for characterizing the raw powders [4]. Non-sphericity, internal porosity, incorrect alloying, or other defects in the AM powder can in turn produce defects in the end part including pores, cracks, residual stresses, and undesired surface roughness [5], [6]. Particle size and particle size distribution (PSD) of the AM powder are among the most influential factors. Smooth, spherical particles can flow more easily and therefore produce higher apparent densities, a bulk material property correlated to the creation of high-density layers [7]. However, AM powders are generally not perfectly spherical because they are usually produced by the method of gas or plasma atomization of molten metal, in which some particles become welded together and create elongated shapes. Finer particles (< 10  $\mu$ m) increase density by filling the gaps left by larger ones, but they are more susceptible to Van der Waals forces and can create a cohesive powder resulting in poorly spread







layers [8], [9]. Moreover, the theoretical maximum density of a granular material is comprised of a combination of both coarse and fine particles that create a specific, optimal PSD [8], [10].

There exist several techniques for analyzing particle size, shape, shape distribution, and PSD of an AM powder. Some of these techniques are commonly used in the additive manufacturing and powder metallurgy (AMPM) industries whereas others are primarily used in research laboratory settings due to the instrument cost and/or the extra effort required. Each of these techniques uses different physical phenomena to measure the size of particles, and therefore it is unclear how the PSD results from one technique are more or less trustworthy than from other techniques. To highlight this critical issue, in this study we have evaluated powder samples with effectively the same PSDs and morphology using the following five sizing methods. Sieve analysis, dynamic imaging analysis (DIA), and laser diffraction (LD) analysis are commercially available and commonly used in the AMPM industries. X-ray computed tomography (XCT) and scanning electron microscopy (SEM) are commercially available instruments, but not commonly used as sizing techniques, especially not in industrial settings. They each are capable of providing low uncertainty and robust particle size and morphology data. A brief introduction to these five techniques is given as follows.

## 1.1. Sieve analysis

During sieve analysis, powder is passed through a series of sieves, each with progressively smaller mesh sizes, while the stack of sieves is vibrated. The mass of powder collected by each sieve as a fraction of the whole mass is reported along with the respective sieve size. The particles that pass one sieve and fail to pass the next smaller sieve are considered to have particle size between the two sieve sizes. Sieve analysis is a standard test method described by both ASTM and ISO standards organizations [11], [12].

# 1.2. DIA

The DIA method is an image-based analysis method capable of measuring the size and shape of powder particle projections as they pass a light source by capturing individual particle silhouettes using a highspeed digital camera. The powder is conveyed using a liquid or gas and software is used to calculate the 2D size and shape of the particles. The size of the particle is estimated using various mathematical analyses of the particle image parameters. The typical pixel size of the collected images is  $\approx 1 \mu$ m. DIA is a standard test method described by ISO 13322-2:2006 [13].

## 1.3. LD

In LD analysis, the diffraction patterns produced by laser light scattering from clouds of particles are used to generate a PSD. Similar to DIA, the powder is conveyed and often dispersed using liquid or gas. Particle shape is assumed to be spherical and algorithms, often proprietary, are used to extract particle sizes from the diffraction patterns in terms of the volume fraction of particles having a diameter equal to the light scattering of an equivalent sphere. Therefore, LD does not measure powder morphology at all. LD is a standard test method described by both ASTM and ISO standards organizations [14], [15].

## 1.4. SEM (static image analysis)

The SEM method can generate high contrast, highly resolved images (resolution  $\approx$  a few nm) of the AM powder particles. Similarly to DIA, 2D images are analyzed to find particle size and shape, but with better resolution than what is possible using optical sizing methods. Particles must be dispersed and mounted so that particles are not touching one another and on a substrate that produces a high contrast (e.g., SEM

carbon tape). While not specific to SEM, static 2D image analysis is a standard test method described in ISO 13322-1:2014 [16].

#### 1.5. XCT

The XCT method is an image-based method where X-rays are used to image particles from different perspectives, mathematically compiling a full 3D geometry (i.e., computed tomography). It is the only method among all of these five methods that gives full 3D particle size and shape information of the particles, including the internal pores, within the resolution of the scan, of each particle. Therefore, it can be used to help understand the 2D shapes measured by DIA and SEM. The voxel size of the XCT 3D images used for AM powders is typically  $\approx 1 \,\mu\text{m}$ . More about the particular XCT method used herein is available [17].

In this study, two 17–4 stainless-steel powders with unique PSDs are analyzed. It should be noted that the PSD of AM powders, as measured by LD, is most often log-normally distributed, and depending on the specific AM process, the PSD has a median diameter in the range of tens of micrometers [18], [19]. For the LBPF, one of the more common metal AM technologies, the volume-weighted 10th percentile range is about 20 µm and the volume-weighted 90th percentile range is about 45 µm, as measured by sieve analysis. This is the approximate size range that is investigated in this work.

There have been some studies describing the comparison of particle sizing methods [20]-[23], but no comprehensive study exists that compares the PSDs of these AM powders as measured by different methods. This is important because each particle size measurement technique is affected by the particular morphologies common in gas and plasma atomized metal powder. While some studies have used Standard Reference Materials (SRMs) [24], which are often nearly perfect spheres and don't sufficiently test the relationship between morphology and the measurement principle employed, others have used atomized metal powders, but either only employ a minimal number of techniques [21] or are outdated and novel techniques (e.g., DIA, SEM, and XCT) were not available [22], [23]. Here, the results are presented from these techniques without showing a bias towards a particular one. Each technique has inherent advantages and disadvantages. The average particle size is reported for each of the different techniques, the full PSD from each method is compared from SEM and XCT perspectives, and a general comparison of 2D and 3D data and the mechanisms that may drive the differences seen between the techniques are presented here

Finally, we discuss the need for AM-specific standard methods for characterizing metal feedstock powders and the possibility of creating a reference material of an AM powder sample that can be used to calibrate various sizing methods.

## 2. Materials and methods

A total of five sizing techniques were employed with two being laboratory-based methods and three commercially available sizing methods. While for brevity's sake the phrase 'laboratory-based' is used, it is noted that the equipment used for these two methods (SEM and XCT) is commercially available, but the protocols employed are not.

Each of the techniques was used to evaluate two distinct and commonly used AM 17–4 stainless-steel alloy powders. While the two powders have been produced using the same 17–4 (i.e., SAE type 630 or UNS 17400) stainless steel alloy guidelines and atomized in an inert argon atmosphere, the PSDs are unique. Each powder consists of the same nominal chemical composition and should have nominally the same metallographic grain structure. Therefore, only the size and morphology should significantly vary between the powders, which are the independent variables in this study. It is noted that the morphology and surface characteristics cannot be said to be the same a priori. These both largely depend on the atomization process (e.g., fluid dynamics of high-pressure cooling gas, speed, and chemical composition of the feedstock, chamber geometry), which can vary between batches (colloquially known as heats) and atomizer type, and therefore, there is the potential for these two powders to differ in terms of their surfaces and morphology.

#### 2.1. Preparing powder samples with effectively identical PSDs

Each of the techniques uses a different sample size to measure the PSD, and the mass differences can sometimes be expressed in orders of magnitude. For example, the SEM sample preparation needed  $\approx$ 15 mg/sample of the AM powder whereas sieve analysis evaluated 100 g/sample. Starting with the 10 kg as-received container of powder, aliquots were produced using a rotary riffler (i.e., sample divider) as prescribed in [26]. While previous work has shown minimal variability in PSD as a function of sampling location in the as-received container [19], sampling using a rotating riffler ensures each sample is representative of the initial powder [25]. Further, sample sizes were chosen so that the entire sample was evaluated in each respective method to limit uncertainty introduced via sampling. This is the case for all methods except XCT due to the dispersion technique. Because of the large range in sample sizes, a larger standard rotary riffler was used in combination with a micro rotary riffler, which is shown in Fig. 1. The micro rotary riffler used in this work was capable of dividing up to 80 g of powder into eight portions (i.e., eight samples 10 g each) and could create samples smaller than 10 mg. Care must be taken when creating the smaller samples, due to their increased sensitivity to deviations from the representative PSD. Subsequent riffling operations were used as needed to create the smaller samples. While standards are available for determining the optimal sample size for sieve analysis, LD and DIA rely on user experience. As is discussed in Section 3.2, SEM and XCT sample sizes require more attention and are not controlled by any standard.

# 2.2. XCT

This technique has been described in detail elsewhere [4], [17], [27]. Briefly, a powder is dispersed by hand in a quick-setting epoxy, such that the volume fraction of powder in the powder-epoxy composite is about 10% or less. This assures that the powder particles are on the whole not touching each other so that no separation via image analysis is generally necessary. The epoxy-powder mixture is drawn into a 3 mm diameter straw using a small vacuum pump. After the epoxy has



Fig. 1. Schematic of a spinning riffler (i.e., rotating sample divider).

hardened, a 50 mm long section is placed upright into a 3D x-ray microscope and scanned with a voxel size of about 1 µm for this powder size range. Four or five fields of view (FOV) were taken inside the straw for each particle class, with about one thousand 1000-pixel x 1000-pixel images in each FOV, to assure that enough particles were scanned so that various averages and distributions would be independent of total particle number.

NIST-written software was run on each set of images, automatically segmenting them into binary images (white powder, all else black) using a version of Otsu's method for three phases [28]. Other software then identified particles and analyzed them. 3D geometric information was stored for each particle, including volume, length L, width W, and thickness T. L is the longest length across the particle, W is the longest length across the particle that is perpendicular to L, and T is the longest length across the particle that is perpendicular to both L and W. By this construction,  $T \perp W \perp L$ ,  $T \leq W \leq L$  and a rectangular box of dimensions L, W, and T is the smallest volume box that just contains the particle [29]. The three aspect ratios, L/T, W/T, and L/W were useful for approximately classifying particles, in particular L/T. Note that only two of these ratios are independent. Only particles that had a volume of at least 512 voxels (approximately 8 voxels across the particle) were analyzed since volumes smaller than this meant that particle shape could not be accurately analyzed. Therefore, the minimum size of the particle studied was  $\approx$  8 µm. Each particle that was analyzed was stored in a database, and a 3D image was also stored for each particle.

Analyzing around 1000 or more particles can give an adequate statistic [30]. Here, it should be noted that 2D projection results can be computationally generated from the inherently 3D XCT results [31], [32]. Since the 3D results are not used in the rest of the paper, in order for better comparison with the other methods, it is important for the reader to know where the 2D XCT results came from. Two samples were evaluated for each powder, and each sample contained about 0.3 mg.

# 2.3. SEM

This technique has been described in detail elsewhere [18]. SEM imaging-based PSD analysis requires a well-dispersed powder sample. The SEM sample preparation apparatus based on the aerosol dispersion method was used for the AM powder in this work. Briefly, the apparatus includes a carbon tape adhered on a metal substrate (i.e., SEM 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 AM powder through the eye-dropper onto the carbon tape. In this way, the particles of the AM powder ( $\approx 15 \text{ mg}$ ) were deposited on the SEM substrate (50 mm  $\times$  50 mm). This sample preparation procedure ensures that there were a sufficient number of particles present on the substrate while minimizing contact between particles. Two samples were evaluated for each powder.

SEM images (non-overlapping) were acquired in an automated mode using the SEM's annular backscattered electron detector. The beam landing energy was 15 keV, the electron beam current was 8.9 nA, a dwell time of 2  $\mu$ s per pixel was used, the working distance was set to 9.8 mm, and the horizontal full width of the acquired images was set to 1.06 mm (i.e., a pixel pitch of 0.518  $\mu$ m). To eliminate systematic error in the SEM scale, a reference material sample [33] for pixel pitch (magnification) calibration was used. SEM images of more than 69,000 particles were acquired.

The image processing was done according to the guidance in ISO 13322-1-2014 [16]. First, the SEM images were converted to an 8-bit grey-scale format. A Kuwahara filter with a window size of 5 pixels was then used to smooth the intra-particle features while maintaining sharp contrast at the particle's edges [34]. An open-source software called FIJI was used to analyze various size and morphology metrics of each of the particles [35].

## 2.4. DIA

DIA measurement was performed according to ISO 13322-2-2006 [13]. A sample size of 18 g of powder was used after a series of tests were conducted, in which incrementally larger samples were run through the device until no appreciable difference could be visually detected in the PSD. The PSD was found to not significantly change beyond pprox 15 g, and therefore 18 g was used to include a factor of safety. A total of five samples were evaluated for each unique powder using DIA. The powder was poured through the vibratory feeding system, which conveys the particles towards the viewport. Prior to being imaged, the powder was dispersed with air pressurized to 50 kPa to separate any nonpermanently attached agglomerated particles. A focused, strobing light-emitting diode was used to back-illuminate the particles creating silhouettes that were imaged with the charge-coupled device camera. The instrument captures about 275 images per second with a 20 mm field of view and a pixel size of 1 µm. The device's software used the 2D silhouettes to calculate size and morphology metrics.

# 2.5. LD

Laser diffraction sizing was performed according to ISO 13320:2020 [14] and ASTM B822–20 [15] using a commercially available LD device. Using the vendor's guidance, a sample of 10 g was used for each test and five unique samples were evaluated for each powder (i.e., 50 g of each powder is tested). The dispersion pressure was set to 0 kPa and a suction pressure differential of 20.7 kPa was used to aid in conveying the powder through the measurement cell. The particles being sized must be opaque and substantially larger than the wavelength of the interrogating laser (780 nm); therefore, Fraunhofer's theory was used to analyze the angles of the scattered light and no index of refraction was required [36].

#### 2.6. Sieve

As prescribed in ASTM B214 [12], 100 g aliquots were used for sieve analysis. A total of six samples were tested, three of each powder class. Eight sieves were used with the following sizes: 76  $\mu$ m, 64  $\mu$ m, 53  $\mu$ m, 48  $\mu$ m, 39  $\mu$ m, 32  $\mu$ m, 25  $\mu$ m, and 20  $\mu$ m. The powder that passed through all of the sieves (i.e., pan) was also weighed. Since sieve analysis is only reporting the size of an opening that allowed a certain mass fraction of particles to pass, it can be considered a two-dimensional (2D) measurement, but with sufficient time and vibration, particles can be oriented so that their smallest dimensions in 3D (e.g., W and T) are parallel with the sieve's surface, allowing it to pass through the sieve opening if W or T is smaller than the size of the sieve opening. This will be further discussed below. Sieve analysis cannot be used for determining the shape of particles. It essentially gives a two-dimensional (2D) measurement of inherently 3D particles.

#### 3. Data analysis

Since each of the techniques is evaluating a different physical property (e.g., diffraction patterns of laser light, silhouettes produced via back illumination, ability to pass through a sieve), the data must be processed to ensure all data is comparable. First, a common metric of size must be chosen. While some methods are more robust in terms of measured features, others are inherently limited. For instance, laser diffraction sizing produces a single metric, the equivalent circular area diameter (ECAD) that corresponds to the produced diffraction patterns, while techniques like SEM and DIA produce full 2D profiles from which many metrics can be extracted. Even more robust, XCT measures the complete 3D geometry of each particle, allowing both 2D and 3D metrics to be extracted. Therefore, using LD as a constraint, when comparing PSD's from all techniques, the ECAD is used. It is noted that for this comparison an assumption must be made that the 2D techniques are evaluating the particles without any preferred orientation (the validity of this assumption is evaluated and discussed later). All methods except sieve analysis are capable of measuring the ECAD of the particles.

There are specific metrics that are appropriate for comparing certain sizing techniques. A prime example of this is the minimum chord diameter (XcMin), which is the smallest value from a measured set of maximum chords, applied to the particle across every angle. This metric can be evaluated in both 2D and 3D. Fig. 2 helps illustrate this concept. Imagine the 2D outline of the particle is rotated and the maximum horizontal chord is measured for each rotation. The smallest value of this set of measurements is XcMin. This metric is often very similar to what sieve analysis is measuring, although it depends on the particle's morphology. Since sieve analysis is evaluating how particles pass through square openings there are two caveats to the XcMin similarity. First, since the opening is square there is the potential for particles to orient themselves so that they are aligned with the larger diagonal dimension of the opening. Secondly, since XcMin is inherently a 1D metric and sieves have 2D openings, it is effectively evaluating the particle in two dimensions that are perpendicular to one another. For particles that have similar widths and thicknesses (i.e., W and T), which is generally the case for gas atomized metal powders due to the fact that surface tension during solidification creates a generally symmetric, rounded morphology, both of these caveats can be disregarded. If sufficient time and vibration are used during sieve analysis, the particles can



Fig. 2. The concept of the minimum chord diameter (XcMin), the equivalent circular area diameter (ECAD), and the maximum Feret diameter (FeMax); the data presented is only for illustration purposes.

orient themselves so that their XcMin dimension is orthogonal to the sieve's opening, allowing each to pass through. Therefore, the data produced by sieve analysis could be considered a 3D version of XcMin. Along with ECAD and XcMin, also illustrated in Fig. 2, the maximum Feret diameter (FeMax) will be used for comparison. FeMax can be thought of as the maximum length that a caliper would measure if the particle was rotated infinite times and measured at each orientation. The concepts of XcMin and FeMax can be generalized, in principle, to 3D. In 3D, ECAD would no longer be in terms of an area equivalent, but a volume equivalent spherical diameter (VESD). For this work, to meet statistical requirements, three orthogonal projections from each particle measured using XCT will be used for PSDs, taken along the directions of L, W, and T computed for each particle from the 3D data.

Another decision that must be made when reporting PSDs is whether the distribution is based on number, length, area, or volume fraction. For this work, unless otherwise noted, the data is presented in terms of volume fraction, defined in the following way. For LD, the volume of the sphere with the same diameter as the measured diameter is used to define particle volume. For sieve analysis, when only one kind of powder is sieved at a time (i.e., constant material density), the fraction of the total mass passing one sieve and being retained on the next smaller sieve is the same as the volume fraction. For DIA, SEM, and the 2D projections made from the 3D XCT results, the volume of a particle projection is defined to be the volume of the sphere with diameter equal to the ECAD of the particle.

While a number basis may reveal interesting details of the distribution, the uncertainty of reporting number-based fraction is inordinately high for powders in this size range. Calculating a PSD in terms of number fraction inherently shifts the distribution towards smaller sizes; thus, any uncertainty in measuring these finer particles is more prevalent in the overall measurement uncertainty. In general, fine particles are much more difficult to measure since they are more prone to Van der Waals forces and static charging, which can create issues during sampling and dispersion. Likely the most apparent contributor to the uncertainty of measuring these fine particles is the resolution of the device. The resolution's contribution to the standard uncertainty is

$$u = \delta x / \sqrt{12} \tag{1}$$

where  $\delta x$  is the resolution [37]. For example, an instrument with a 1  $\mu$ m (i.e., the pixel size of the DIA device used in this study) resolution would have a corresponding resolution standard uncertainty equal to 0.29  $\mu$ m. It is apparent that this is of relatively little concern when measuring a large particle, say 1 mm, as compared to the measurement of particles around 1  $\mu$ m.

Additionally, sieve analysis and LD are the most common sizing techniques used currently in AM and use volume fraction, defined in



		Volume Weighted Mean Size		Arithmetic Mean Size	
	Sizing Technique	Mean (µm)	SD (µm)	Mean (µm)	SD (µm)
Powder 01	LD	33.98	0.04	26.25	0.12
	DIA	32.90	0.00	26.95	0.16
	SEM	34.55	0.07	27.45	0.49
	XCT	33.75	1.20	25.35	0.49
	Sieve	28.60	0.13	14.74	0.02
Powder 02	LD	42.17	0.04	31.55	0.55
	DIA	39.70	0.07	32.57	0.12
	SEM	43.00	0.57	31.80	1.70
	XCT	41.20	0.28	30.80	0.42
	Sieve	36.77	0.05	20.60	0.20

the manner previously discussed. The widespread acceptance of these two methods has made volume fraction the most familiar representation and most accepted format for representing PSD. Consequently, this work will focus on volume fractions and any particles with ECAD's (VESD for XCT) less than 8 µm measured in any way have not been included in the analysis.

## 3.1. Volume weighted mean and arithmetic mean sizes

The moment notation [38], based on volume-weighting, is used to simplify both the representation and calculation of mean sizes.

Eq. 2 is the form of the moment equation that assumes a continuous PSD.

$$M_{k,r} = \int_{x_{\min}}^{x_{\max}} x^k q_r(x) \mathrm{d}x \tag{2}$$

where *M* is the moment, *k* is the order of the moment, and *r* is the type of quantity of the distribution with r = 0 and r = 3 referring to  $q_r(x)$  (i.e., PSD) being a number distribution density and volume distribution density, respectively. The variable *x* is the particle size (e.g., ECAD) with  $x_{\min}$  being the minimum and  $x_{\max}$  the maximum particle size in the distribution. This allows the mean, variance, and kurtosis to be calculated using the first, second, and third moments.

From here, it becomes evident there is a need for a means to directly compare the moments of different types of quantities of the distribution densities. For example, sieve results (e.g., distribution density or  $q_r(x)$ ) are inherently in terms of mass, (or equivalently volume, assuming constant particle density) while SEM sizing produces data in terms of numbers of certain sizes. It is apparent that the average measurement from each technique cannot be directly compared. The moment-ratio



Fig. 3. (a-b): (a) Volume weighted mean (b) arithmetic mean sizes of particles as determined by various techniques. Error bars represent +/- standard deviation.

notation provides a convenient way to represent various types of mean sizes. First, the moment is defined in a discrete nature as shown in Eq. 3, where  $M_p$  is the *p*-th moment, *N* is the total number of particles measured,  $D_i$  is the midpoint of the *i*-th bin, and  $n_i$  is the number of particles in the *i*-th bin. Eq. 4 is the general formula for calculating mean sizes,  $\overline{D}_{p,q}$ , using the moment-ratio notation, whereas Eq. 5 allows one to calculate mean sizes using any size distribution,  $q_r(D)$ , using Eqs. 2–4, where *m* is the number of bins or classes [39].

$$M_p = N^{-1} \sum_i n_i D_i^p \tag{3}$$

$$\overline{D_{p,q}} = \left[\frac{M_p}{M_q}\right]^{1/(p-q)} \tag{4}$$

$$\overline{D_{p,q}} = \begin{bmatrix} \sum_{i}^{m} q_r(D_i) D_i^{p-r} \\ \frac{D_i}{\sum_{i}^{m}} q_r(D_i) D_i^{q-r} \end{bmatrix}^{\frac{1}{p-q}}$$
(5)

#### 3.2. Number of particles measured

Due to the substantial level of effort required for SEM and XCT, care must be taken in choosing the sample size and thus number of particles measured. The other techniques are specialized for sizing and are capable of measuring hundreds of thousands or millions of particles relatively quickly and without substantial user input. It is noted that, partially due to the proprietary, black-box nature of LD and DIA devices, the exact number of particles measured is unknown. It can be estimated using the sample's mass, but the fraction of unmeasured particles would also need to be estimated leading to substantial errors in this kind of guesswork.

In order to ensure a sufficient number of particles are measured in terms of statistical significance, Eqs. (6–9) are used.

$$n = \omega \delta^{-2},\tag{6}$$

$$\omega = p^2 \alpha^2 s^2 (2c^2 s^2 + 1), \tag{7}$$

$$\phi(-|p|) = (1-P)/2, \tag{8}$$

$$c = \beta + \alpha/2, \tag{9}$$

where *n* is the minimum number of particles and  $\delta$  is relative error [16, p. 133]. In eqs. 5, 6, and 7, *p*, an intermediate parameter, is derived from a cumulative distribution function,  $\phi$ , of the standard normal distribution.  $\alpha$  and  $\beta$  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.,  $s = x_{84,0}/x_{50,0}$ ). The minimum number of particles required for achieving a 95% probability level (*P*) with no more than 5% relative error in estimating the mass



Fig. 4. (a-d): Comparison of ECAD and Xcmin plots for Powder 1 (PS-01): (a) ECAD plot comparing Sieve and SEM data, (b) XcMin plot comparing Sieve and SEM data, (c) ECAD plot comparing Sieve and XCT data, and (d) XcMin plot comparing Sieve and XCT data.

median diameter is calculated using the average distribution widths, s, produced using SEM and XCT data. The number of particles required is estimated to be 9313 for Powder 1 and 9925 for Powder 2; therefore a threshold of 10,000 particles is used. XCT was used to evaluate 17,451 and 12,450 projections for Powder 1 and Powder 2, respectively (5817 particles for Powder 1 and 4150 particles for Powder 2, each projected from three orientations), and SEM was used to evaluate 41,406 and 22,588 particles for Powder 01 and Powder 02, respectively. Each of the techniques evaluated more than the required 10,000 particles. Assuming a volume-weighted mean size of 36 µm and material density of 7700 kg/m<sup>3</sup> [40], 1.9 mg of powder would contain more than the calculated 10,000 particles. As noted earlier, for this work the nominal and much larger sample sizes for the other techniques were 18 g for DIA, 10 g for LD, and 100 g for sieve analysis.

#### 4. Results and discussion

With so many metrics, there are many options in terms of how the data can be plotted and analyzed. An appropriate starting point is to generalize the results by computing the commonly reported metrics (e.g., percentile values and average sizes). For the average particle sizes, the volume-weighted mean sizes and the arithmetic mean sizes are reported. The percentile values will be reported only in terms of volume fraction. A more in-depth analysis is conducted by comparing the full PSDs of each technique. Since SEM and XCT are much more robust techniques, a further analysis comparing each is made. The existence of preferential orientation will be explored using the full 3D morphologies characterized by XCT. Finally, aspect ratios will be analyzed for each of the techniques with such capability.

# 4.1. Average size of particles

The volume-weighted mean particle size and arithmetic mean particle size from each technique, calculated using Eq. 5, are presented in Fig. 3 and also in Table 1. It is immediately apparent that sieve results are much lower than the other techniques and especially so for the arithmetic mean sizes. It should be noted that since the bin resolution of the histogram is determined by the sieve sizes used, and since it is impractical to use sieves spaced as fine as 1 µm apart, many of the bin values must be interpolated for comparison purposes. While there is knowledge a priori from the other techniques that could be used to interpolate the distribution, this isn't always the case; therefore, the authors have used a linear interpolation. This results in an overestimation of the sizes of the powder that falls into the pan (i.e., the smallest particles that pass the finest sieve). While there is potential for this misrepresentation to also take place on the large end of the distribution, it is much less pronounced for several reasons. First, the log-normal shape of the PSDs creates a lower slope on the larger end of the distribution, which translates to less volume or number fraction error per size. Because the sieve sizes used to encompass the largest XcMin, the point at which the distribution approaches 0% has been better defined on the large end. This is best visually represented and is shown in Fig. 4 with the sieve sizes marked with vertical hashed lines on Fig. 4a. Finally, considering the arithmetic mean sizes, the mean sieve size is pushed even lower, due to the fact that a number based PSD inherently has higher fractions at lower sizes (i.e., many small particles equal several large particles in terms of volume). While this is true for each of the sizing methods, the lack of sieve resolution at the smaller end of the distribution exaggerates this effect.



Fig. 5. (a-d): Comparison of XCT PSD results with the mean PSD results with error bars representing the standard deviation of other techniques. (a-b) including the data from sieve analysis, and (c-d) without including the data from sieve analysis.

As described in the introduction to Section 3, XcMin is a more appropriate metric for comparing sieve data with other techniques. As illustrated in Fig. 4, it is obvious sieve data more closely matches the XcMin (see Fig. 4b and Fig. 4d) metric for both SEM and XCT than does the ECAD metric (see Fig. 4a and Fig. 4c). Note 'PS-01' is interchangeable with 'Powder 01' and is used for the sake of brevity. The root mean square (RMS) of the difference of each comparison is lowered for the XcMin comparison versus the ECAD comparison. For SEM, the RMS of the difference is reduced from 0.98% to 0.75% and for XCT it is reduced from 0.71% to 0.54%. It is obvious that both SEM's and XCT's XcMin PSD shift towards the sieve's PSD matching the median closely.

The general trend from the other methods places SEM's volume weighted mean size as the highest for both powders and SEM's arithmetic mean size as highest for Powder 01 and as a close second for Powder 02 with DIA's arithmetic mean size being larger by only 0.77 µm. This is illustrated in Table 1 and Fig. 3. LD and XCT are within 1 µm of each other for both powders, which is interesting considering they are both 3D techniques. Besides sieve analysis, DIA produces the smallest volume-weighted mean for both powders. Evaluating the variance from each test method, nearly all methods have a coefficient of variation (COV) well under 5% with the largest variances coming from XCT's maximum standard deviation 1.2 µm (i.e., COV equal to 3.6%) for the volume-weighted mean of Powder 1 and SEM's maximum of 1.7 µm (i.e., COV equal to 5.3%) for Powder 2's arithmetic mean. This is mainly attributed to the lower number of particles measured for these two techniques. In juxtaposition to this, both LD and DIA produce very low standard deviations across their respective measured samples (n = 5). Each is below COV of 0.25%, which is likely due to the large number of particles measured.

Further investigating the mean sizes, the full PSDs of SEM and XCT are compared to the mean PSD of all other techniques. Fig. 5 compares XCT to the other sizing techniques, while Fig. 6 compares SEM to the other methods. The sieve data can be removed to better illustrate the spread of all techniques, especially considering the earlier discussion of sieve data's similarity to an XcMin metric. This is done for both Fig. 5 and Fig. 6. All of the aforementioned plots have error bars representing the standard deviation. Not considering sieve data, for PS-01 the largest deviation takes place near the peak and between 30 µm and 45 µm when comparing both XCT and SEM to the other methods. PS-02 has much less variability near the peak of the distribution. For XCT it is about equally spread between the rise and fall of the PSD, but for SEM it is primarily for the larger particles (i.e., 45 µm to 65 µm).

## 4.2. Volume fraction percentiles

There are three PSD percentiles of the volume-weighted size data that are commonly reported: the size at which 10% of the particles are smaller (D10), the size at which 50% of the particles are smaller (D50, i.e., the median), and the size at which 90% of the particles are smaller (D90). As shown in Fig. 7, it is apparent that while most of the techniques are in close agreement, sieve analysis is again an outlier. On the cumulative PSD curves, Figs. 7a and 7c, the horizontal dashed lines show the values of D10, D50, and D90, which are then displayed in bar charts in Figs. 7b and 7d. The cumulative PSD curves are well-suited to displaying the D10, D50, and D90 values in a clear manner. As discussed previously, this is primarily due to the fact that the data produced via sieve analysis is much more comparable to the XcMin metric, not ECAD or VESD, and therefore sieve analysis results will be



Fig. 6. (a-d): Comparison of SEM PSD results with the mean PSD results and standard deviation of other techniques. (a-b) including the data from sieve analysis, and (c-d) without including the data from sieve analysis.



Fig. 7. (a-d): Percentile comparison of all techniques for PS-01 shown in (a) and (b) and PS-02 shown in (c) and (d).

ignored for the remainder of this volume fraction percentiles discussion. Another notable feature in this comparison is that LD has the largest D90 value and DIA the lowest D90 value for both powders. As is seen in Figs. 7a and 7c, the LD results approach 100% volume fraction much slower than the other cumulative volume fraction curves. There is a difference between D90 values for DIA and LD of over 4 µm for both powders, which is the largest difference of any of the percentile-technique combinations (other than those involving sieve analysis). LD and XCT are in close agreement for the 10th and 50th percentiles with the D90 values having the worst agreement due to the relatively large LD value. In general the four methods produce similar D50 values with an average of 32.2 µm and a COV of 2.34% for PS-01, while PS-02s average D50 is 39.96 µm and produced a COV of 4.14%. This translates to a range of 1.7 µm and 3.97 µm for PS-01 and PS-02, respectively.

# 4.3. Particle size distribution comparisons

To further evaluate the discrepancies between the percentile and mean values, the full PSD differential curves are plotted and shown in Fig. 8 and Fig. 9 with the calculated difference of volume fraction for each bin colored red and plotted on a second axis. Similar to Fig. 4, the plots have been divided into comparisons with XCT and with SEM for simplification and for ease of visualization. There is a great deal that can be extracted in terms of analysis, but an attempt is made to generalize the findings in the following subsections.

# 4.3.1. Laser diffraction

LD data produces a smooth curve, with tails extending further on the large end than the other sizing techniques. The smoothness is mostly due to the large sample size and the fact that the laser light diffracts off of many particles at once effectively averaging the produced diffraction patterns. It seems LD is overestimating the amount of larger particles for both powders, which has also been reflected in the D90 comparisons (Section 4.2). Previous work comparing LD and XCT PSD results has shown that LD couples more strongly to the L value of each particle, which will lead to an apparent overestimate of particle size [41]. More specifically, since diffraction takes place at all surfaces impinged by light, there exists the potential for the various surface features and satellite particles, ubiquitous in gas atomized particles and which tend to cause higher values of L, to distort the PSDs produced with LD. Since LD assumes a spherical particle shape, this coupling to larger lengths on the particle surface becomes averaged into the effective particle size, increasing its value. This distortion has been previously documented in [22], where Kaye dubs these "ghost particles". Most commercially-available sizing techniques are effectively black boxes where powder goes in and a PSD is produced. There are parameters that can be changed, but it is difficult to quantify how each can change the measurement. An algorithm must be used to extract sizes from the diffraction patterns. Some of the features of the PSDs (as measured by other methods) are not evident on the LD curves in Figs. 8 and 9. This is apparent for both powders, and particularly so near the median size. LD produces a significantly higher peak at the median sizes. For



Fig. 8. (a-d): Comparison of XCT PSD results of Powder 01 and 02 with DIA and LD. (a-b) with DIA, (c-d) with LD.

Powder 2, XCT and SEM show an almost flat top distribution but LD maintains the same smooth, more sharply-peaked shape as measured in Powder 1. Even with these differences, overall LD produces PSDs that generally match those measured using XCT, especially so for Powder 1. Note that using ECAD for the XCT 2D projections tends to mix L, W, and T for the particles, so that the XCT results also have a large-ECAD tail as the LD results do, and for similar reasons.

## 4.3.2. Dynamic image analysis

The SEM and DIA results shown in Fig. 9 closely agree, especially so for Powder 1, which may reflect their similar sizing methods (i.e., 2D imaging of silhouettes). The shape of the DIA PSD is quite close to XCT but has a 2 µm to 3 µm shift towards larger particles on the left, rising side of the PSD curve. For unknown reasons, DIA produces higher volume percentages at similar ranges (about 30 µm to 45 µm) for both powders. The flat-top PSD shape seen in both SEM and XCT data for Powder 2 is present, but with a slight peak at the median size for DIA. Similar to LD, commercial DIA devices have a black box nature. In DIA, the powder is moving past the optics using a flowing gas or liquid. The variable drag force that is dependent on a particle's size and shape creates a varying particle velocity. This in turn alters the likelihood of particles being imaged and leads to a potential for skewing the PSD. While there is a feature on the specific device used in this work to compensate for this change in velocity, it is difficult to quantify the effect and to what extent the compensation nullifies this effect. There also exists the potential for preferential orientation, which is especially likely for laminar, non-turbulent flows. The particular nature of the entraining fluid (pressurized air is used for this work) is unknown, but since a dispersion pressure of 40 kPa is used, it is assumed the fluid is sufficiently turbulent. Additionally, the finite depth of field present in the optics system causes particles to become blurry as they exit this region. This can be seen when individual images are saved and analyzed. This instrument includes a means to remove this effect, but due to the proprietary nature, it is difficult to quantify the effect without knowing what is being done. It is this depth of field that is attributed to being the cause of the shift upwards in the volume fractions between 30  $\mu$ m and 45  $\mu$ m. In summary, because the goal of this work is to provide a comparison of what is common to the AMPM industries in terms of sizing, it is best to treat these as black boxes, only adjusting parameters that would commonly be changed. Therefore, the data presented here is what would commonly be expected when an operator in the AM or PM industry used DIA to measure the PSD of his/her powder.

As stated in Section 3, a 3D equivalent of the minimum chord diameter (XcMin) is what sieve analysis probably measures, but since methods like LD are incapable of producing this metric, the ECAD from each method is compared with sieve results and shown in Fig. 10. The small-particle tail is readily apparent for the sieve analysis results as compared to the other techniques.

# 4.4. Comparing the lab-based techniques

Fig. 11 shows a comparison of the SEM and XCT differential PSD results. SEM and XCT are in overall close agreement and their differences seem similar for both powders. For both powders, SEM has a higher volume fraction of particles over 30  $\mu$ m with the difference in volume fraction hitting a maximum of 0.94% by volume at the 36.5  $\mu$ m bin for PS-01 and 0.76% by volume at the 54.5  $\mu$ m for PS-02. The XCT data has the largest positive difference at effectively the same bin for both powders with the magnitude of difference slightly larger for PS-02 compared to PS-01 (i.e., 0.92% at 21.5  $\mu$ m for PS-01 versus 1.22% at 22.5  $\mu$ m for PS-02. It is



Fig. 9. (a-d): Comparison of SEM PSD results of Powder 1 and 2 with the DIA and LD (a-b) with DIA, (c-d) with LD.

plausible the variability induced via sampling causes these differences. Fig. 12 presents a comparison of the subsequent samples tested, and while the differences are much more random (i.e., no apparent shifting of PSD curves), the magnitude of the sample differences is similar to the difference between the methods (i.e., SEM and XCT). The RMS of each of the differences for each powder and for each method are included in Fig. 12. As shown in Fig. 11, the RMS of the difference between SEM and XCT PSDs for PS-01 is 0.31% while the same metric for PS-02 is 0.4%. This is comparable with what is found when comparing the RMS of the difference between samples, which ranges from a low of 0.13% by volume for PS-01 between the SEM samples and a high of 0.32% by volume for XCT's PS-02 sample difference.

Since SEM is a 2D sizing technique, there is the potential for preferential orientation. This may be induced during dispersion (e.g., orientation via laminar air flow) or when the particles fall onto the SEM tape. To evaluate whether this condition is present, one can start by looking at the aspect ratios of XCT and SEM data. If the particles' orientation is the source of the discrepancy between SEM and XCT PSDs, there should be a noticeable difference in the aspect ratios. As shown in Fig. 13, the Feret aspect ratio ( $Fe_{min}/Fe_{max}$ ) is calculated for all particles



Fig. 10. (a-b): Comparison of the differential PSD for (a), the results of PS-01 and (b), the results of PS-02.



Fig. 11. (a-b): Comparison of XCT and SEM PSDs of powder 01 and powder 02.

and then binned according to the respective particles' ECAD. The average Feret aspect ratio is plotted for each ECAD bin. It is important to note that each bin contains a range of Feret aspect ratios, which is reflected by the blue oval in Fig. 13a. This variability is better represented by a box and whisker plot of the particles in each bin as shown in Fig. 13c. The blue box represents the 25th and 75th percentiles, the red crosses represent outliers, which have been determined to be outside  $\pm 2.7\sigma$ , where  $\sigma$  is

the standard deviation, the dashed vertical line is the range and the red horizontal line is the median. The impressive stochasticity of these powders becomes immediately apparent. While one can attempt to present representative images of certain particle types, the wide breadth of the Feret aspect ratio helps illustrate the inadequacies in doing so. Nevertheless, several images from each powder and representing various Feret aspect ratios are shown in Fig. 13 (a-b). Particles made from agglomeration



Fig. 12. (a-d): Comparing the variability induced via sampling: RMS and differences are calculated for each of the duplicate measurements and plotted with the PSD for a) PS-01 XCT, b) PS-02 XCT, c) PS-01 SEM, and d) PS-02 SEM.



**Fig. 13.** (a-c): Comparison of ratios of Fe<sub>min</sub>/Fe<sub>max</sub> calculated by XCT and SEM. (a) for powder 1, (b) for powder 2, and (c) a box and whisker plot is used of the particles in each bin and is shown (for powder 1) to better represent this variability. It is important to note that each bin contains a range of Feret aspect ratios, which is reflected by the blue oval in Fig. 13a. This variability is better represented by a box and whisker plot of the particles in each bin as shown in Fig. 13c.

and/or partial fusion with other particles are represented at a high concentration at the lower Feret aspect ratios with most of the Feret aspect ratios near unity being single, generally spherical particles. This is by no means a generalizable categorization of gas atomized particles, but it provides a basic understanding of the ranges of aspect ratios as well particle morphologies that are common for certain sizes and Feret aspect ratios. The trends of the Feret aspect ratios per ECAD agree surprisingly well between SEM and XCT, and especially when the range of Feret aspect ratios present in each bin are considered. In summary, comparing the Feret aspect ratios plotted using ECAD binning does not reveal any significant difference between the two laboratory-based methods. Using XCT's full 3D characterization of each particle may shed light on this difference.

XCT's full 3D characterization allows for the virtual orientation of particles. The virtual particles can be rotated so that they mimic those that may fall in a certain direction (i.e., preferential orientation). As explained earlier, the L, W, and T metrics from XCT are found using the largest Feret diameter, L, and the next largest that is also perpendicular to W, and finally T, the largest Feret diameter that is perpendicular to both L and W. Assuming SEM particles are dispersed onto the SEM tape in a fashion that would preferentially avoid orienting the smallest dimension (T for XCT) parallel to the substrate (i.e., increasing the measured size), there should be better agreement between SEM data and the L and W XCT metrics than the random orientations. Fig. 14 contains the comparison of SEM and XCT's four projection orientations (i.e., random, L, W, T). Note that an L orientation corresponds to L being along the interrogation direction and therefore would produce a 2D particle outline with the two smallest dimensions (W and T). As seen in Fig. 14(a-b) SEM's ECAD PSD doesn't seem to be closer to any of the XCT projections for either PS-01 or PS-02. In fact, the projections from XCT all seem to be effectively the same in terms of ECAD, which may be due to the fact that the area is used and may hide some of the extreme values. Basically, in terms of ECAD the particles appear quite similar no matter the rotation. FeMax may reveal discrepancies between the two data sets. Fig. 14c shows SEM most closely resembles XCT's random orientation, but does have several large particles that



Fig. 14. (a-d): Comparison of SEM and XCT's four projection orientations (i.e., random, L, W, T). (a) ECAD of PS-01, (b) ECAD of PS-02, (c) FeMax of PS-01, and (d) FeMax of PS-01 with a low pass filter at 100 µm.

may contribute to a shift in the PSD towards the larger end, which is what has been seen previously. Black arrows have been included to denote the hard-to-see SEM bins that represent individual particles with large FeMax values and Fig. 14d contains the same data presented in Fig. 14c, but with a low pass filter at 100 µm. While the majority of the particles are generally spherical and seem to have no preferential orientation when falling onto the carbon taped substrate, the particles with extremely large aspect ratios have near zero chance of falling with their largest dimension orthogonal to the substrate (imagine a bowling pin tossed into the air landing in a stable manner on either end). Therefore, SEM data will always show the largest FeMax particles, plausibly causing this slight shift in the PSDs.

## 5. Summary

Five different sizing techniques were compared: sieve analysis, DIA, LD analysis, X-ray CT, and SEM using nominally identical samples of two different stainless-steel powders produced via riffling. The powder volume requirements for these techniques were not the same and in fact differed by up to three orders of magnitude. For example, SEM needed a powder sample of 15 mg whereas DIA needed a powder sample of 18 g. Well-controlled sampling is accomplished via multiple riffling steps. Since these sizing methods are not based on the same physical phenomena, it becomes challenging to compare each technique's PSDs to the others. Certain specific metrics can be more appropriate for comparing with certain sizing techniques. For example, the minimum chord diameter XcMin is a much more appropriate metric for comparing sieve data with other techniques.

Overall, the differences in sizes measured were not large with the exception of sieve data. Sieve analysis is inherently measuring a single metric, XcMin, that is not measurable by all techniques (i.e., LD). Therefore, when considering statistics of the PSD data from all five methods, sieve analysis is not considered and must be only compared when using the XcMin metric.

Summary of conclusions:

- LD and DIA produce low variance across samples due to their large number of measured particles;
- LD is unable to provide PSDs with detailed features; LD data repeatedly produces a smooth PSD curve compared to DIA, XCT, and SEM;
- the range of D50s measured by all methods excluding sieve analysis is 1.7 μm and 3.97 μm for PS-01 and PS-02, respectively;
- the COV of all methods except sieve analysis is 2.34% for PS-01 and 4.14% for PS-02.

The lab-based techniques (SEM and XCT) are more robust than the commonly-used sizing techniques. XCT is the only true 3D technique used in this work. It can produce the L, W, and T metrics (i.e., using XCT projections) of the particles and produce 2D projections that are oriented along the L, W, or T directions for each particle. However, SEM's ECAD PSD doesn't seem to be closer to any of the XCT virtual projections and instead closely matches results calculated from random orientation of the XCT 3D data, which is an indication that the SEM data does not suffer from preferential particle orientation in its sample preparation step. While this does not disprove the theory that preferential orientation is the cause for the difference between PSD data between

XCT and SEM, it bolsters a rejection of it. A comparison of the maximum Feret diameter measured by both techniques reveals that it is perhaps the most extreme (in terms of FeMax) particles that cause the shift in ECAD PSDs, especially when considering that the shift in the PSD curves agree with this hypothesis. The variance between samples tested with SEM and XCT is similar in magnitude to the difference between the average PSD's but this does not explain the reproducible trend seen in this work.

This work should serve as a picture of the expected variance between these PSD sizing methods, of course assuming proper protocol and sampling are employed. It should be clear that this work is not a quantification of uncertainty. Future work shall include a quantification of uncertainty for each of these methods done similarly to the work in [18]. Additionally, the creation of an SRM using gas atomized AM metal powders should be considered. There would be substantial work to ensure each sample is of the same nominal PSD and morphology, but it would allow laboratories to validate their PSD measurements. Currently, for dimensional metrology of metal AM powder particles, there are no SRMs available.

# **Declaration of Competing Interest**

None.

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