Importance of Particle Size in Powders Used for Additive Manufacturing

AccuSizer® SPOS system

Additive manufacturing (AM), or three-dimensional (3D) printing can be performed using various materials including liquid resins, polymers, and powder metals (Figure 1). When using polymers and metal powders, the particle size distribution is important for processability and final part quality. The Entegris AccuSizer is the most accurate and high-resolution particle sizing technique used to characterize powders for AM.

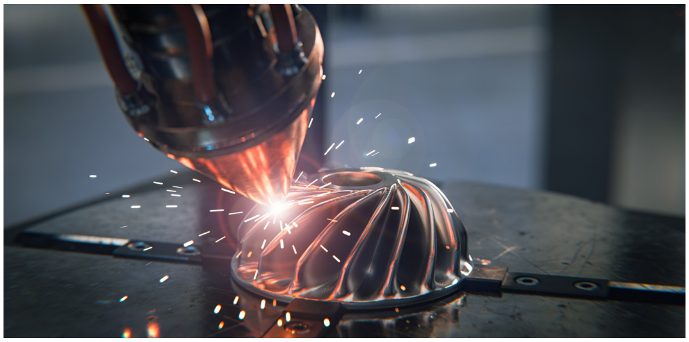

Figure 1. 3D printer printing a metal turbine.

INTRODUCTION

AM is the construction of a 3D part from a computer aided design (CAD) or digital 3D model. It can be performed using a variety of processes in which material is deposited, joined, or solidified according to the CAD data. The raw material is typically added together layer by layer as the part is created from the bottom up. After the printing process is complete, post processing is typically required to achieve the desired part dimension.

The particle size of powder used in AM plays a crucial role in determining the quality of the final product.[1, 2, 3] The powder particles act as building blocks, and their size affects the packing density, surface area, and flowability of the powder bed.

The importance of particle size can be summarized as follows:

1. **Powder packing density:** Optimized particle size distributions improve packing density. Large particles with suitable percentage of finer particles occupying the interstitial space can achieve high packing density (Figure 2).[4] A high packing density is associated with the production of high quality and minimally flawed components.

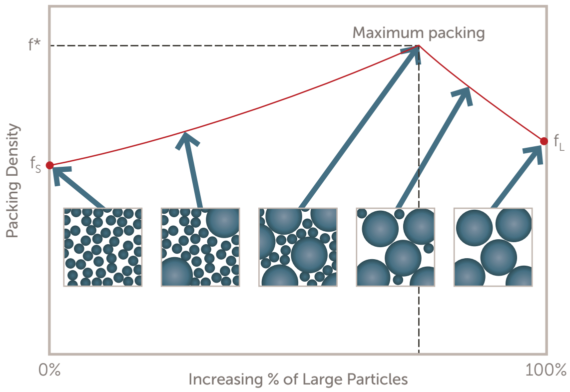

Figure 2. Effects of particle size distribution on powder packing density.

2. **Surface area:** Smaller particles have a larger surface area per unit volume than larger particles. This may make the particle more susceptible to oxidation. During laser melting, the powder oxide is entrained into the molten pool, altering the Marangoni convection from an inward centrifugal to an outward centripetal flow, resulting in more pores. In addition, the oxides may also decrease the toughness of the parts printed by AM.

3. **Flowability:** The flowability of powder is affected by its particle size and shape distribution. Although fine powder is beneficial to achieving high resolution of AM-printed parts, excessive amounts of fines can reduce powder flow rates due to increased cohesion force. Besides, fine particles become increasingly sensitive to moisture which should be avoided whenever possible.

4. **Melting behavior:** Smaller particle sizes have a higher surface area to volume ratio, leading to faster melting. Nevertheless, higher laser absorption occurs for particles with a higher area-to-volume ratio. Therefore, the melt pool may become unstable with excessive energy input, leading to more defects of the printed parts.

   Therefore, controlling the particle size distribution of the powder is key to achieving high-quality parts in AM.

PARTICLE SIZING TECHNIQUES

Laser diffraction is a common technique used to analyze the particle size distribution of powders. Although this is a quick and easy analytical technique, the resolution of laser diffraction is inherently limited since it is based on ensemble light scattering. Single particle optical sizing (SPOS) is a high-resolution particle sizing method where individual particles are sized and counted, so results are more accurate, and the distribution width is reported more accurately. The Entegris AccuSizer is the most automated and easy to use SPOS system available.

EXPERIMENTAL

Different types of powders that could be used in AM were analyzed using the Entegris AccuSizer system. The AccuSizer with the LE400 sensor (dynamic range of 0.5 – 400 µm) was used for all analyses. In the first part of this study powder samples were first dispersed into a liquid and then analyzed using the AccuSizer SIS. The following samples were analyzed by the AccuSizer SIS: SiO_2, zeolite, In 718, Mat 21, SS 316, PMMA, and SiC.

Sample preparation: Five hundred microliters of Triton X-100 (1%) was added to 300 mL of DI water. An amount of sample was weighed out relative to the volume of diluent in order to achieve an appropriate count rate in the AccuSizer SIS. Most samples required 2 – 100 mg of powder dispersed into 300 mL of diluent. Once the suspension was made, it was mixed using a manual mixer at medium speeds to make sure that the particles were both well dispersed and mixed. Then a measurement was made with 3 replicates in rapid succession.

Results: The results in Appendix I show tabulated results of the D10, D50, and D90[5] for the number and volume-based particle size distributions. The coefficient of variation (standard deviation/mean expressed as a percentage) is calculated to quantify the precision. The graph shown in Figure 3 shows the repeatability for the volume distributions for three of the samples.

The tables in Appendix I and plots in Figure 3 provide quantitative proof that the AccuSizer results are highly repeatable and can be used for powder characterization for these samples.

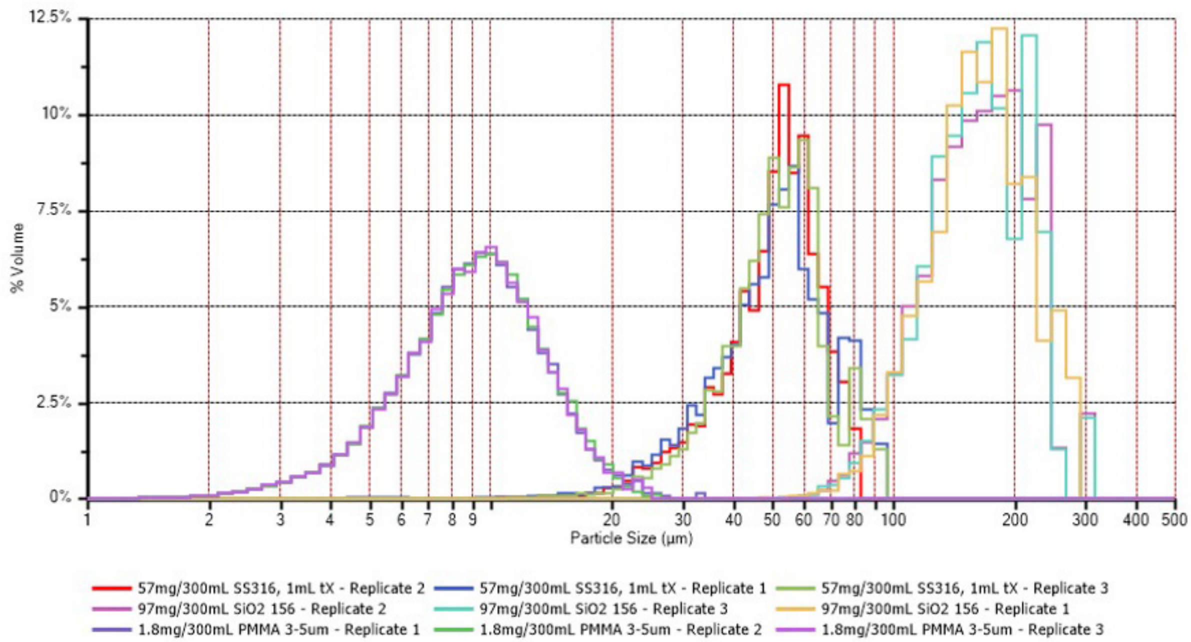

Figure 3. Graphical AccuSizer volume % results.

PARTICLE SIZING TECHNIQUES: LASER DIFFRACTION VS. SPOS

SPOS is a common technique for measuring both the size and concentration of particles suspended in liquid. In the SPOS technique, particles in liquid suspension flow through a photo-zone where they interact with a laser light source via extinction and/or scattering. The extinction/scattering by the particle is related to particle size and concentration through the use of a pulse height analyzer and a calibration curve. The result generated is the concentration and particle size distribution of the particles in suspension.

The SPOS method is in stark contrast to the laser diffraction technique where the latter measures all particles in the measurement zone at the same time. Instruments that perform particle size analysis using ensemble techniques, such as laser diffraction, are inherently limited in accuracy and resolution since the raw detected signal is "inverted" mathematically to estimate the particle size distribution. After the scattered light is collected on the multiple detectors in a laser diffraction system, an algorithm is used to convert the scattered light to particle size. The calculated result is influenced by inter–connected factors including:

- Optical design
- Algorithm: Fraunhofer or Mie theory
- Refractive index of the sample/dispersing medium

LASER DIFFRACTION VS. SPOS RESULTS

The following samples were analyzed on both the AccuSizer SPOS system and a laser diffraction analyzer (not manufactured by Entegris): AlSiMg, PMMA, and SS420. The figures below show the volume and number distributions for these measurements. The x-axis size scale is similar in all plots.

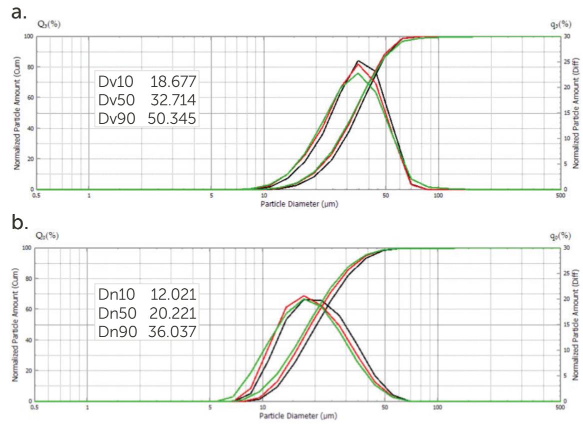

Figure 4. Laser diffraction AlSiMg results, a. volume above, b. number below.

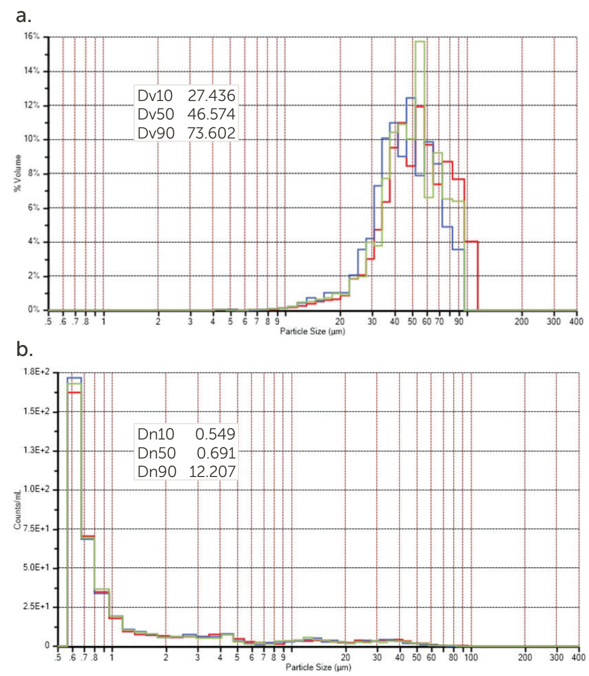

Figure 5. AccuSizer AlSiMg results, a. volume above, b. number below.

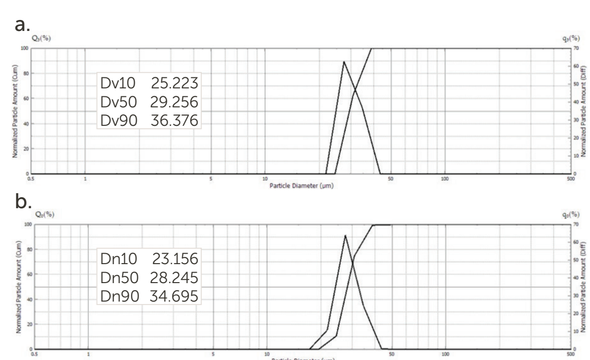

Figure 6. Laser diffraction PMMA results, a. volume above, b. number below.

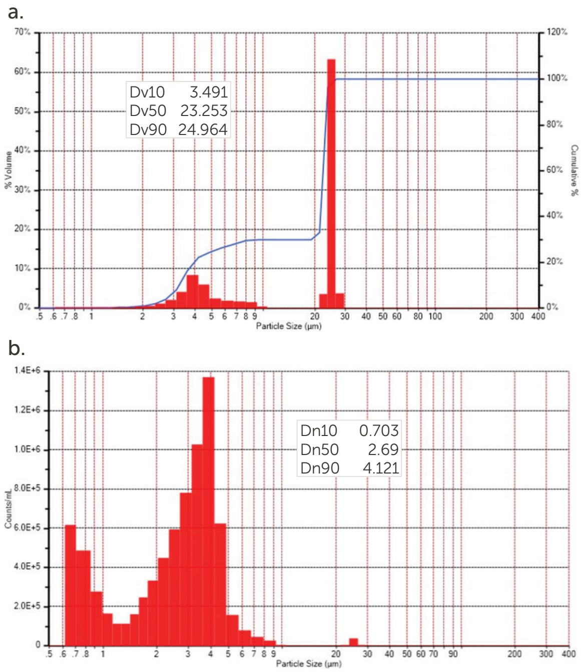

Figure 7. AccuSizer PMMA results, a. volume above, b. number below.

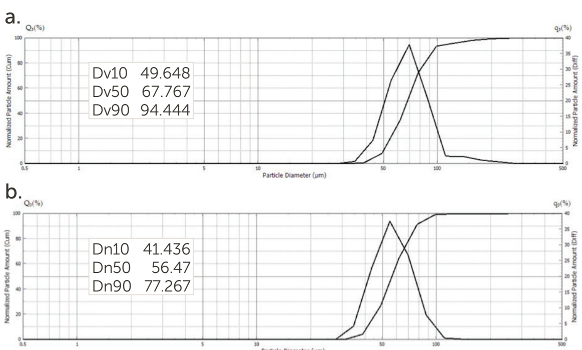

Figure 8. Laser diffraction SS420 results, a. volume above, b. number below.

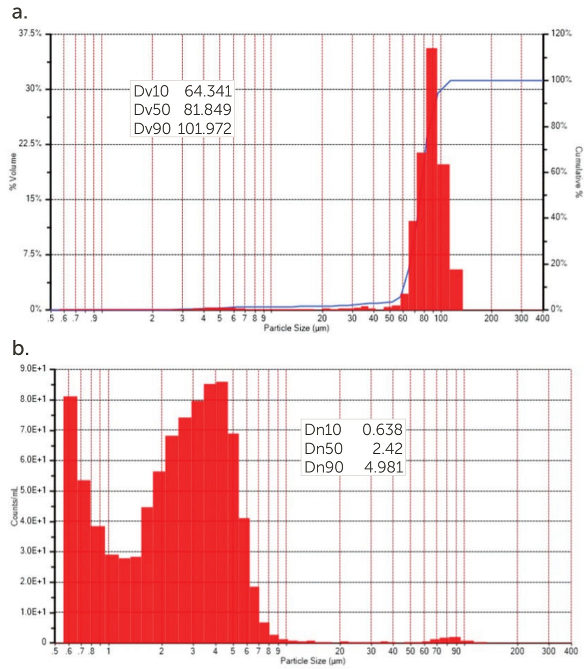

Figure 9. AccuSizer SS420 results, a. volume above, b. number below.

VOLUME FRACTION AND ABSOLUTE VOLUME

Additional quantitative information from the AccuSizer includes the calculation of absolute volume – the volume in µm³ that a particle occupies in suspension. This calculation requires knowledge of the weight density, and specific gravity of the powder and the volume of water the powder is dispersed into. The two tables below show absolute volume calculations for the PMMA sample. The upper table shows size distribution and absolute volume results. The lower table shows particles/gram ≥ diameter.

Channel	Diameter	Counts	Cumulative	Number	Area	Volume	Number	Volume	Abs. volume
9	0.674 – 0.964 µm	485/mL	27057/mL≥Dia.	1.8%	0.0%	0.0%	1.8%≤Dia.	0.0%≤Dia.	1396.8 µm³
10	0.964 – 1.336 µm	653/mL	26572/mL≥Dia.	2.4%	0.0%	0.0%	4.2%≤Dia.	0.0%≤Dia.	5201.6 µm³
11	1.336 – 1.793 µm	902/mL	25919/mL≥Dia.	3.3%	0.1%	0.0%	7.5%≤Dia.	0.0%≤Dia.	18092.7 µm³
12	1.793 – 2.483 µm	1151/mL	25017/mL≥Dia.	4.3%	0.2%	0.0%	11.8%≤Dia.	0.0%≤Dia.	58869.9 µm³
13	2.483 – 3.468 µm	1519/mL	23866/mL≥Dia.	5.6%	0.4%	0.1%	17.4%≤Dia.	0.1%≤Dia.	209483.7 µm³
14	3.468 – 4.74 µm	1866/mL	22346/mL≥Dia.	6.9%	1.0%	0.3%	24.3%≤Dia.	0.4%≤Dia.	675494.8 µm³
15	4.74 – 6.237 µm	2399/mL	20480/mL≥Dia.	8.9%	2.2%	0.8%	33.2%≤Dia.	1.2%≤Dia.	2076673.5 µm³
16	6.237 – 8.073 µm	3009/mL	18081/mL≥Dia.	11.1%	4.8%	2.3%	44.3%≤Dia.	3.5%≤Dia.	5771184.8 µm³
17	8.073 – 10.329 µm	3744/mL	15073/mL≥Dia.	13.8%	9.8%	6.1%	58.1%≤Dia.	9.6%≤Dia.	15268731.8 µm³
18	10.329 – 13.097 µm	4268/mL	11329/mL≥Dia.	15.8%	18.2%	14.3%	73.9%≤Dia.	23.9%≤Dia.	35905413.9 µm³
19	13.097 – 16.332 µm	4024/mL	7062/mL≥Dia.	14.9%	270%	26.7%	88.8%≤Dia.	50.6%≤Dia.	67131023.8 µm³
20	16.332 – 20.098 µm	2288/mL	3037/mL≥Dia.	8.5%	23.5%	28.8%	97.2%≤Dia.	79.5%≤Dia.	72411693.7 µm³
21	20.098 – 25.091 µm	666/mL	749/mL≥Dia.	2.5%	10.5%	16.0%	99.7%≤Dia.	95.5%≤Dia.	40200280.5 µm³
22	25.091 – 30.753 µm	72/mL	83/mL≥Dia.	0.3%	1.7%	3.3%	100.0%≤Dia.	98.7%≤Dia.	8195631.8 µm³

Size	Parts/gram ≥ diameter
0.611 micron	908,239,192
1.031 micron	618,733,736
5.682 micron	485,794,789
8.923 micron	481,501,687
10.254 micron	480,300,548
11.708 micron	479,405,506
23.602 micron	446,769,401
26.704 micron	286,657,595
29.85 micron	52,722,246
36.832 micron	18,648,649
50.627 micron	1,104,273

Figure 10. Absolute volume calculations for PMMA.

The AccuSizer software can also calculate the volume fraction within any chosen size range. This calculation requires knowledge of the concentration and density of the powder and the volume of water the powder is dispersed into. Figure 11 shows two volume fraction calculations for the PMMA sample.

Percent solids (vol/vol)	Range	Volume fraction
0.001%	1.5-27.8 µm	15.82096%

Percent solids (vol/vol)	Range	Volume fraction
0.001%	10.0-100.0 µm	79.28612%

Figure 11. Volume fraction results.

DISCUSSION AND CONCLUSIONS

While the volume distribution results for these samples are quite similar using both SPOS and laser diffraction, the number distribution results are obviously completely different. The reason for this is that laser diffraction does not measure individual particles, but the ensemble light scattering and the results are calculated from models. The laser diffraction technique is inherently limited in resolution. The transformation from volume to number distribution is not expected to be accurate. This conversion is just offered for comparing results with other techniques. As shown in Figures 4 – 9, the conversion is merely a shift to the left for the calculated distribution. This provides no accurate information concerning the relative quantity of fines to coarse particles in the distribution.

In contrast, the AccuSizer accurately provides count and size for each individual particle passing through the sensor. The particle size distribution is equally quantitative for both the number (particles/mL) and volume % distribution. For applications like AM where the quantity of fine particles impacts both powder flow and part quality, the SPOS technique provides far more valuable information.

APPENDIX I: TABULATED RESULTS

	NUMBER			VOLUME		
			SiO_2			
Run	D10	D50	D90	D10	D50	D90
1	0.545	0.874	83.525	105.151	160.302	228.483
2	0.539	0.835	76.625	103.48	160.317	226.816
3	0.541	0.836	78.91	103.608	158.434	221.836
Mean	0.542	0.848	79.687	104.08	159.684	225.712
STD Dev	0.003	0.022	3.515	0.93	1.083	3.458
COV %	0.56%	2.62%	4.41%	0.89%	0.68%	1.53%
			Zeolite			
1	0.582	2.118	7.73	5.735	11.666	18.63
2	0.58	2.092	7.379	5.45	11.081	17.212
3	0.574	2.029	7.361	5.53	11.449	17.72
Mean	0.579	2.08	7.49	5.572	11.399	17.854
STD Dev	0.004	0.046	0.208	0.147	0.296	0.718
COV %	0.72%	2.20%	2.78%	2.64%	2.59%	4.02%
			In 718			
1	0.521	0.649	2.716	34.483	52.992	71.967
2	0.521	0.653	3.18	35.903	55.315	71.39
3	0.523	0.656	3.491	35.184	52.598	73.481
Mean	0.522	0.653	3.129	35.19	53.635	72.279
STD Dev	0.001	0.004	0.39	0.71	1.468	1.08
COV %	0.22%	0.54%	12.46%	2.02%	2.74%	1.49%
			Mat 21			
1	0.519	0.724	4.019	28.936	43.864	55.654
2	0.521	0.738	4.056	29.981	43.593	55.801
3	0.522	0.732	4.264	31.892	47.356	60.964
Mean	0.521	0.731	4.113	30.27	44.938	57.473
STD Dev	0.002	0.007	0.132	1.499	2.099	3.024
COV % 0.521	0.29%	0.96%	3.21%	4.95%	4.67%	5.26%

APPENDIX I: TABULATED RESULTS (CONTINUED)

		NUMBER			VOLUME	
			SS 316			
1	0.514	0.676	18.61	30.166	50.178	74.69
2	0.514	0.675	24.239	31.58	50.954	66.152
3	0.513	0.656	24.142	33.241	50.535	68.436
Mean	0.514	0.669	22.33	31.662	50.556	69.759
STD Dev	0.001	0.011	3.222	1.539	0.388	4.42
COV %	0.11%	1.68%	14.43%	4.86%	0.77%	6.34%
			PMMA			
1	0.542	1.485	4.845	3.645	5.972	9.774
2	0.54	1.417	4.86	3.663	6.139	10.21
3	0.542	1.41	4.839	3.699	6.323	11.109
Mean	0.541	1.437	4.848	3.669	6.145	10.364
STD Dev	0.001	0.041	0.011	0.027	0.176	0.681
COV %	0.21%	2.88%	0.22%	0.75%	2.86%	6.57%
			SiC			
1	1.152	3.139	6.101	3.663	6.073	9.017
2	1.115	3.068	5.997	3.585	5.976	8.9
3	1.139	3.073	5.969	3.565	5.925	8.736
Mean	1.135	3.093	6.022	3.604	5.991	8.884
STD Dev	0.019	0.04	0.07	0.052	0.075	0.141
COV %	1.65%	1.28%	1.15%	1.44%	1.25%	1.59%

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[5] Entegris white paper 7127-10785ENT-1019, *Particle Size Analysis Overview*

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