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The Effect of Metal Additive Manufacturing Powder Recycling on Part Characteristics and Powder Reusability

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Direct metal laser sintering (DMLS) is a powder bed fusion (PBF) process commonly used within the medical device and aerospace industries to fabricate high value, complex components. Powder material used in the DMLS process can be costly and it is rare for a single build to require a full batch of powder. The un-melted powder, which differs in particle size and morphology from virgin powder, is often recycled for further builds. This work presents a study of the effects that recycling a stainless steel metal powder used in the DMLS process has on finished parts. Hence in this paper, powder material characteristics, such as particle size, particle morphology and bulk chemical composition have been monitored throughout the recycling process. An analysis of parts manufactured via DMLS on an EOS M280 demonstrate the negative effect of powder recycling on part quality in terms of surface roughness, part density, hardness and dimensional accuracy. Results from this research provides an insight to the effect that recycling AM powders has on the powder characteristics and on the quality of the parts produced.

Keywords: Additive Manufacturing, Direct Metal Laser Sintering, Powder Bed Fusion, Metal Powder Recycling, Powder Characteristics

Introduction

Additive manufacturing (AM) allows for complex, high value parts to be produced for a range of industries including the aerospace, medical and automotive industries [1]. EOS 316L stainless steel is a commonly used stainless steel powder within AM for these applications allowing for the production of highly corrosion resistant parts [2]. The additive manufacturing process in this research is Direct Metal Laser Sintering (DMLS), which selectively melts a metallic powder material layer-by-layer to build the final metal part. This powder material consists of spherical particles with a mean particle size distribution typically between 10 and 80 µm in diameter. The metal powders used within the PBF processes can be costly and to fully utilise a batch of metal powder, recycling the un-melted powder is beneficial. Recycling of powder consists of sieving the un-melted powder and reusing this sieved powder for subsequent builds. However, this recycling process leads to changes in the characteristics of the powder material due to exposure to the building process multiple times.

It is well understood that the characteristics of the powder material affects the resulting qualities of the parts produced [1]; however, the impact of powder recycling is an issue that has received little attention to date [3]. The methodology presented here allows the powder characteristics to be monitored throughout the powder recycling process as well as monitoring the resulting part qualities. At present this practice of recycling metal powders for AM varies in industry, as corresponding standards for powder material in AM are currently only at development stage [4]. The chemical composition of the powder affects the resulting microstructure of the part; therefore, changes in the chemical composition due to the recycling process will lead to change in the part microstructure produced [5]. Previous research [6] has found that with increased powder recycling, the powder particles become larger in size and less spherical in shape. These characteristics affect layer homogeneity as the powder is distributed across the build plate. This in turn affects the melting process [7] and the resulting quality of the parts built [3]. The distribution of particles has been found to affect the as built density and surface roughness of the parts the presence of more coarse particles in the powder leads to a rougher surface and parts with a lower density [8].

The results presented in this paper provide an insight into the effect of powder recycling on the powder characteristics and the resulting part qualities, thus allowing for a greater utilisation of powder material for the AM process.

Methods and Materials

The powder material, EOS 316L Stainless Steel, was assessed after each build, with a sample taken after the sieving process. The characteristics of the sampled powder as outlined in Table 1 were then assessed by the corresponding test methods. The characteristics of the powder sampled after sieving have been assessed for this study, as this is the material used for subsequent builds.

The un-melted powder particles were sieved after each build using a 63 µm meshed sieve to ensure that any contaminants, in particular, any oversized or partially-melted particles are not introduced into the powder batch. This sieved powder was then used for the next build. This cycle was repeated for a total of seven builds resulting in a total of 88 hours of accumulated build time on the powder.

As shown in Table 1, the chemical composition of the powder was determined through the use of SEM and EDX analysis using a Hitachi TM3030Plus Tabletop Microscope. To carry out this analysis, a sample of powder was mounted in epoxy resin and then micro-sectioned and polished before being placed in the SEM and an EDX map was then conducted to determine the composition of the powder. The recorded composition was then compared to the specified composition as per the powder manufacturers datasheet [2].

The particle size was assessed and monitored at each recycling stage of the process using a Microtrac Series 3500 laser diffraction particle size analyser. This process disperses a sample of powder in front of a tri-laser light source, and the resulting diffraction pattern measures the particle size of each powder particle. The mean particle size and particle size distribution was recorded for three samples of powder and averaged.

The morphology of the powder particles was also assessed at each recycling stage through SEM imaging (x500 magnification) and image analysis using the ImageJ software. The circularity, C, of the particles was assessed using Equation (1).

$$C = 4\pi(\frac{A}{P^2}) \quad (1)$$

Where A is the area of the particle and P is the perimeter of the particle. A circularity value of 1 depicts a perfectly circular particle [9].

To assess the impact of the powder characteristics on the resulting as built part qualities a series of parts were designed and printed on an EOS M280 DMLS machine using the default EOS parameters, in 20 µm layers. The parts, included three density cubes, an overhang part and a geometry block, as shown in Figure 1; these allowed for the tests outlined in Table 2 to be carried out.

The dimensional accuracy of all the printed parts was determined using a CNC coordinate measuring machine, OGP Smartscope CNC 500, with a dimensional resolution of 0.5 µm. Built parts were measured prior to removal from the build plate. Measurements were repeated five times and averaged; the averaged maximum difference from the designed dimensions was recorded.

Hardness of the built parts was tested using a 1/16” ball indenter with a 100kgf applied on a Rockwell Hardness tester on the HRB scale. Part hardness was measured on the top surface as well as the side surface of the geometry block. Measurements were repeated five times and averaged for each surface.

Surface roughness for the overhanging surfaces of the overhang part was measured using Bruker Contour GT-K (vertical resolution ~ 0.1 nm) white light interferometry (WLI). This method allows for a non-contact measurement of the arithmetic mean, *Ra*, of the surface profile. Five measurements of each overhanging surface was taken and averaged, allowing for a greater representation of the overall surface roughness.

Part density of the as built components was monitored using micro-sectioning of the three density cubes, the three cubes were sectioned in different orientations.

Micrographs, using a Keyence digital microscope VH-Z 100R at x500 magnification, of the sections of the cubes allowed for the internal pores to be identified and thus, the resulting part density calculated through image manipulation using the ImageJ software.

Results and Discussion

Effect of Recycling on Powder Characteristics

The bulk powder chemical composition primarily consists of Iron (Fe), Chromium (Cr), Nickel (Ni) and Molybdenum (Mo) with smaller quantities of other elements present [2]. Throughout the multiple recycling stages the EDX map results showed no significant change in the powder composition with the repeated use of the powder.

Powder particle size affects how the powder material is layered and melted. In general, a distribution of larger and smaller particles is required to maintain consistent layering and melting during the build process. The effect of powder recycling, in terms of accumulated build time, on the mean powder particle size is shown in Figure 2 (each data point on the graph represented a recycling stage). An increase in the mean particle size is observed as the build time increases. This is due to the ease that smaller particles are melted in the build process, often being melted first causing the larger particles to be recycled for future builds.

Powder morphology was measured using Equation (1) as described above. The morphology of individual particles affects the way in which the powder flows. The less circular the particle, the more difficult it will be to flow, thus, affecting the consistency and repeatability of the layering process. The effect of powder recycling, in terms of accumulated build time, on powder morphology, i.e. circularity, is presented in Figure 3. A decrease in the powder circularity is observed as powder is recycled and the accumulated build time increases. Particles are less circular compared to the virgin powder due to the inclusion of particles which contain agglomerates as a result of partial melting of adjacent particles in the build process. Figure 4 provides examples of particles with agglomerates and partially melted particles observed at various powder recycling stages.

Effect of Powder Recycling on Manufactured Part Quality

Analysis of the parts, shown in Figure 1, was carried out after each build as per the methods detailed in Table 2. Dimensional accuracy of the parts produced was assessed and the maximum difference, in mm, from the designed dimension was observed. The effect of the accumulated build time on the dimensional accuracy, i.e. the maximum difference from the designed dimensions, is show in Figure 5. The result shows that as the accumulated build hours increase due to powder recycling the maximum difference from the designed dimensions also increases.

The part hardness for 316L built components is specified in the manufacturers data sheet as 89 HRB [2]. The measured part hardness remained within ± 5% of this specified hardness from the data sheet regardless of the recycling stage.

Surface roughness for as built parts using 316L powder is 13 ± 5 µm, as stated in the manufacturers data sheet [2]. The effect of accumulated build time due to powder recycling on the surface roughness of the manufactured overhang part is presented in Figure 6. The surface roughness, *Ra*, increases with the increase in the number of powder recycling stages. The last 3 data points are located outside the manufacturer’s stated surface roughness.

Part density for as built 316L parts is 100%, as stated in the powder manufacturer data sheet [2]. The effect of accumulated build time (powder recycling) on as built part density is presented in Figure 7. The results show as the accumulated build time increases, due to multiple powder reuses, the as built part density decreases.

Influence of powder characteristics on the part qualities

Relating the changes in the powder characteristics to the resulting part qualities allows for a greater understanding of how the powder affects the built parts. The analysis presented here investigates the effect of the increasing particle size on the part density as well as showing the effect of the decreasing circularity of particles on the as built surface roughness, as shown in Figure 8.

Figure 8 shows that the change in the part qualities, specifically, part density and surface roughness can be attributed to changes in the powder characteristics, such as powder size and morphology. Often, part density and surface roughness are critical part qualities required by designers, particularly in industries such as medical device and aerospace. Therefore, the influence of raw material properties on the resultant part characteristics are important for the manufacturing of high quality parts. Figure 8(a) shows that as the mean particle size increases the part density decreases. This is attributed to the change in the powder layering process due to differences in the mean particle sizes. Figure 9 depicts the layering process of powder in the build plate for (a) virgin material with more fine particles present and (b) recycled powder showing that with the larger mean particle size and fewer small particles, voids and pores are presented in the powder layer due to the unevenness of the layering process; this in turn results in voids and powder in the as built part.

Surface roughness is often a critical feature of a manufactured part due to the relationship between surface roughness and component performance and also due to difficulty in post-processing some features to produce the desired surface finish [10]. Figure 8(b) shows that the decreasing circularity of the particles leads to an increase in the surface roughness of the overhanging surface. This is a combination of both the larger and less circular particles being present for the melting of the surface of the overhang part. The larger and less circular particles remain partially melted to the surface producing the rougher surface. Figure 10 shows the changing powder characteristics (SEM images at top in Figure 10 showing particle size and morphology) and the increasing surface roughness (WLI images at bottom of Figure 10) at various stages (0, 16.75 and 88 accumulated build hours) in the recycling process.

Conclusions

The results from this research show that the continued recycling of powder material has an effect on the powder characteristics and the resulting part qualities. The mean particle size of the powder increases with the number of powder reuses, indicating that smaller particles are being melted by the process leaving larger particles for subsequent builds. The particles also become less circular, suggesting a change in morphology due to the partial melting of smaller powder particles to larger particles causing agglomerates to form. The results show that there was no significant change found in the powder chemical composition through the recycling process.

The analysis of part qualities showed that the maximum difference of part dimensions from the specifications increases with the increased powder use. The surface roughness of the overhanging feature increased with the number of powder reuses, indicating that the presence of larger particles in the powder affects the surface roughness. The hardness of the parts remains consistent with the expected value. Part density decreases with the number of powder reuses, indicating that an increase in mean particle size produces increased porosity in the built parts.

These results allow for a greater understanding of the effect that powder recycling has on the powder and the parts built. This will enable a more controlled use of recycled powder materials as well as allowing for a higher utilisation of powder batches.

Acknowledgements

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Table 1: Test methods for powder characterisation

Table 2: Test methods for part qualities assessment

Figure 1: Designed and printed test parts

Figure 2: Effect of powder recycling (accumulated build time) on mean particle size

Figure 3: Effect of powder recycling (accumulated build time) on powder morphology (circularity)

Figure 4: Examples of powder particles with agglomerates and partially melted particles attached

Figure 5: Effect of accumulated build time (powder recycling) on maximum difference in part dimensions

Figure 6: Effect of accumulated build time (powder recycling) on part surface roughness

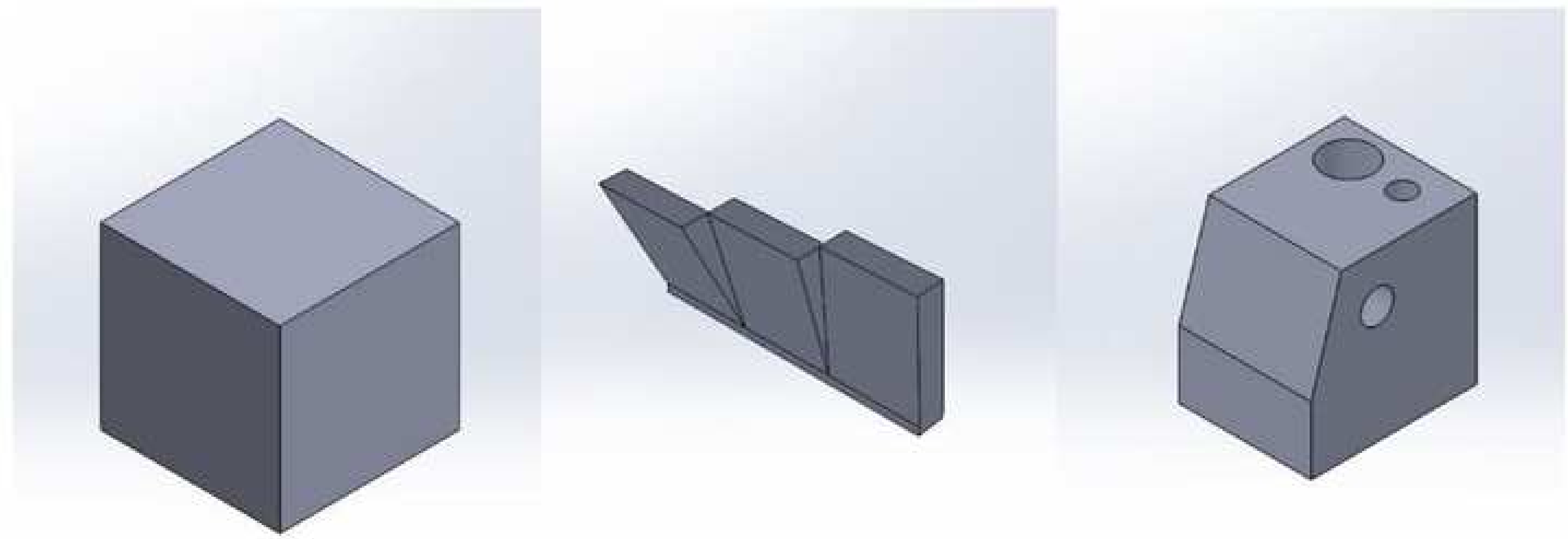
Figure 7: Effect of accumulated build time (powder recycling) on as-built part density

Figure 8: Relating powder characteristics to the part qualities (a) Particle size and part density (b) Powder morphology and part surface roughness

Figure 9: Powder layering process for (a) virgin powder and (b) recycled powder

Figure 10: SEM images (top) and surface roughness plots (bottom) for (a) Virgin Powder, (b) Powder used for 16.75 hours and (c) Powder used for 88 hours

Figure 1



Density Cube

Overhang

Geometry Block

Figure 2

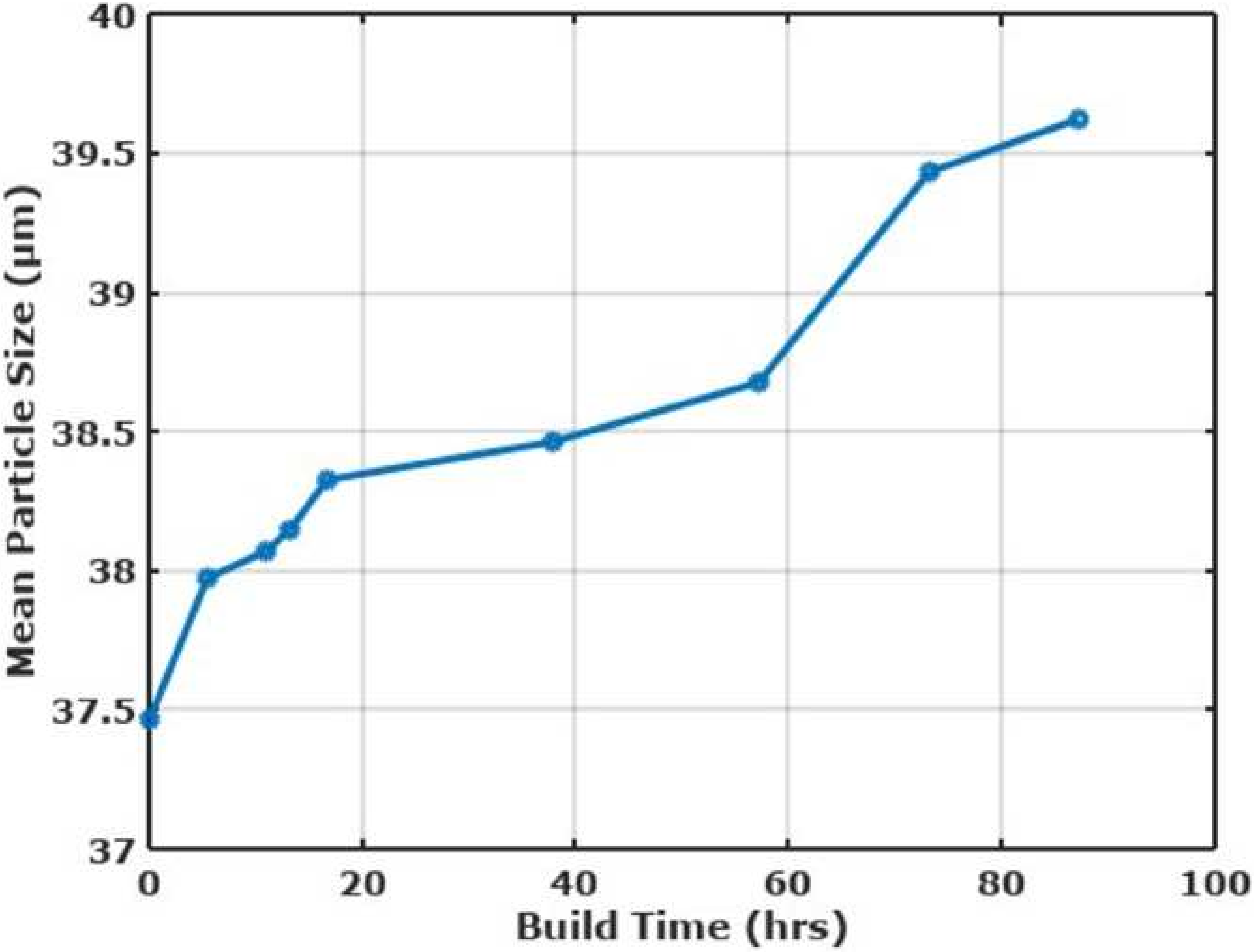
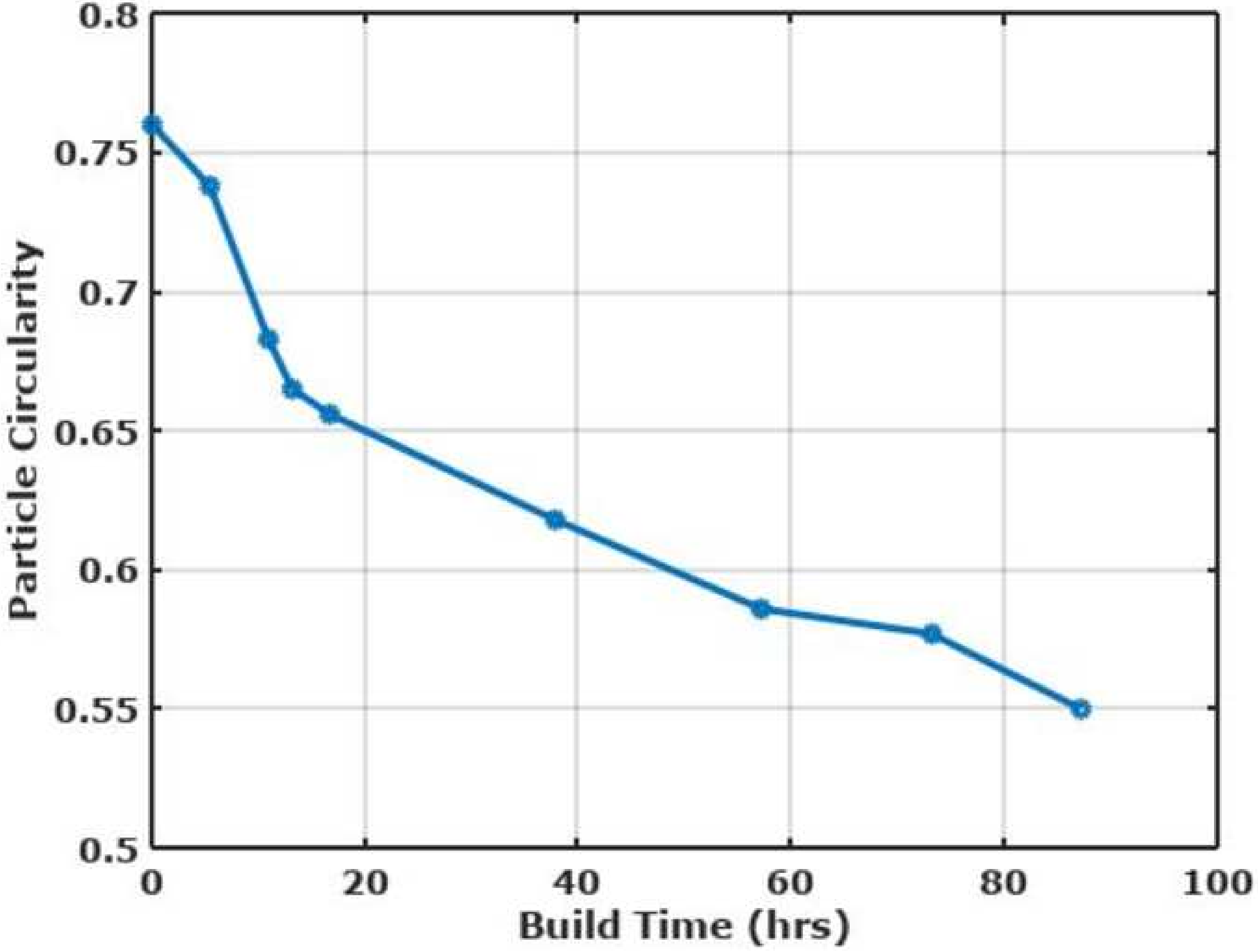

Mean Particle Size (µm)
40
39.5
39
38.5
38
37.5
37
0
20
40
60
80
100
Build Time (hrs)

Figure 3



Particle Circularity
0.8
0.75
0.7
0.65
0.6
0.55
0.5
0
20
40
60
80
100
Build Time (hrs)

Figure 4

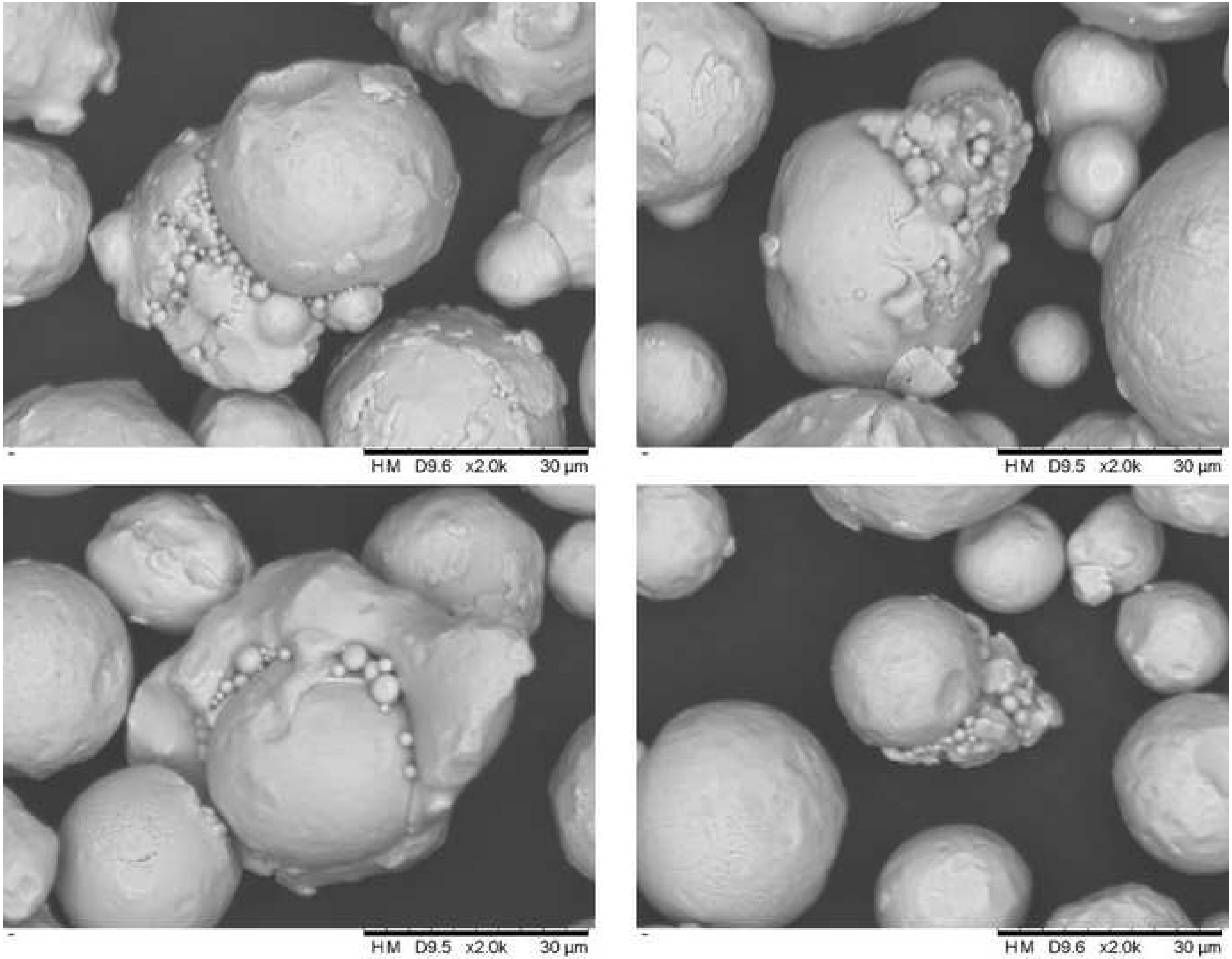
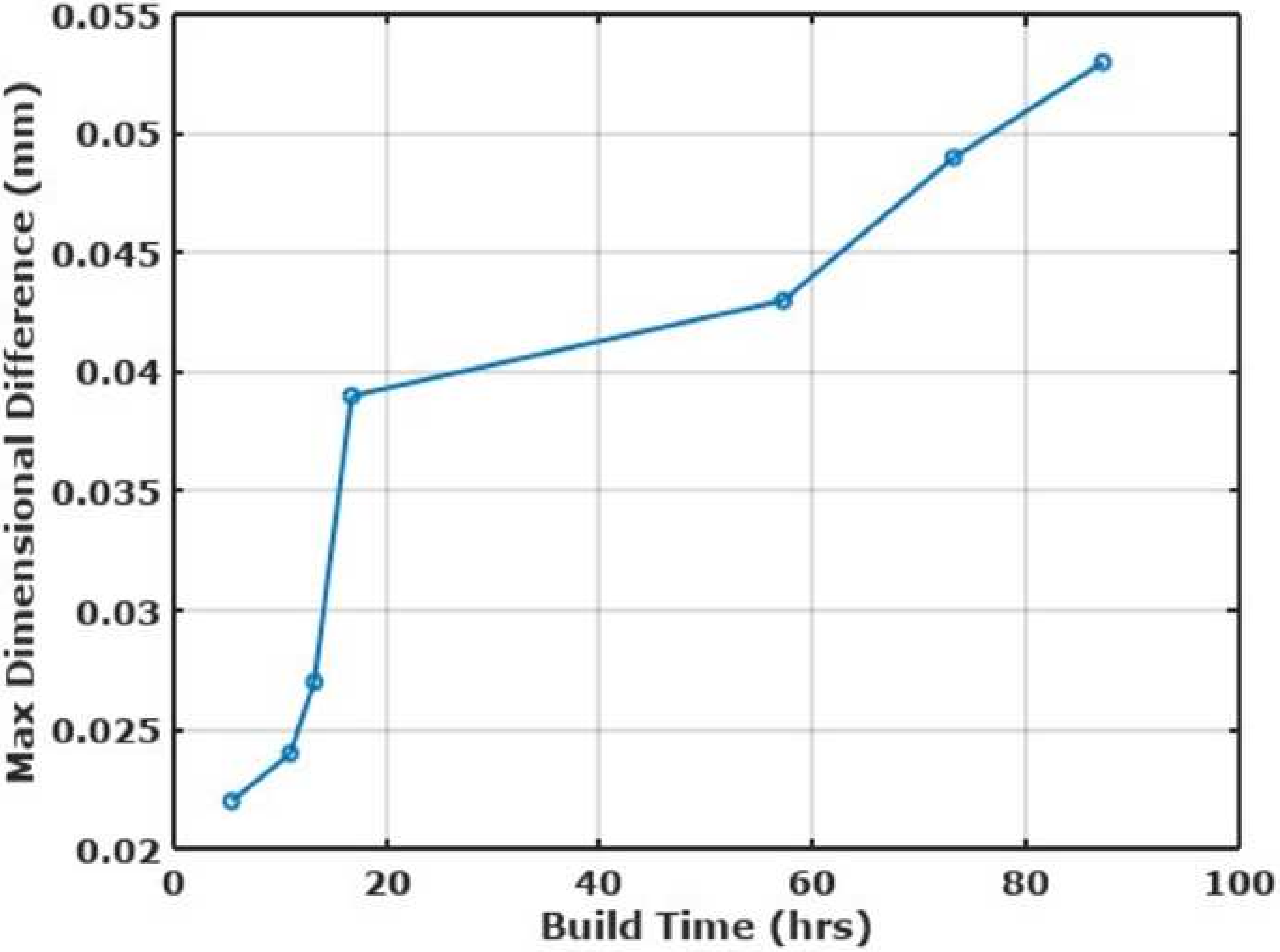

HM D9.6 x2.0k 30 µm
HM D9.5 x2.0k 30 µm
HM D9.5 x2.0k 30 µm
HM D9.6 x2.0k 30 µm

Figure 5



0.055
0.05
0.045
0.04
0.035
0.03
0.025
0.02
0
20
40
60
80
100
Max Dimensional Difference (mm)
Build Time (hrs)

Figure 6

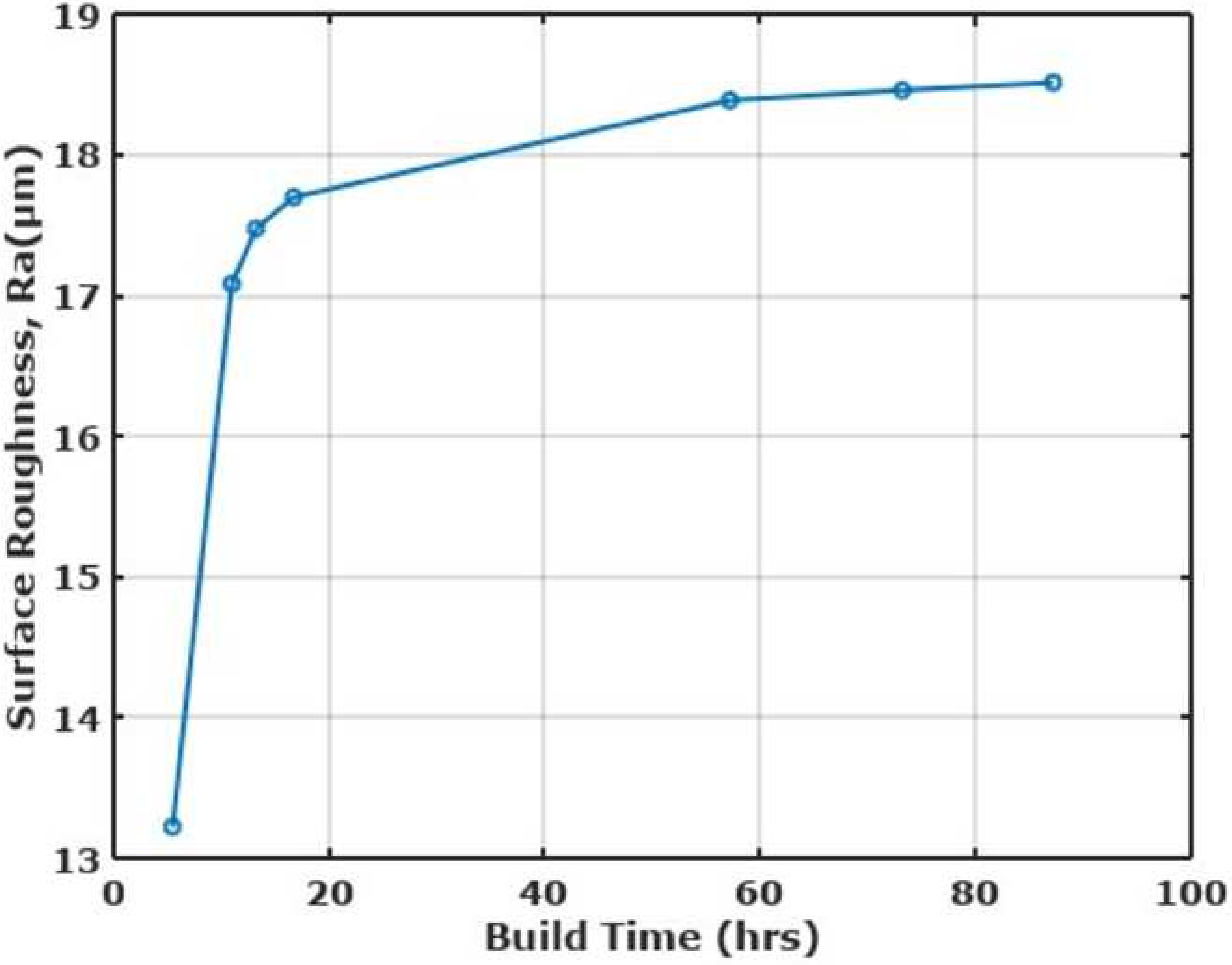
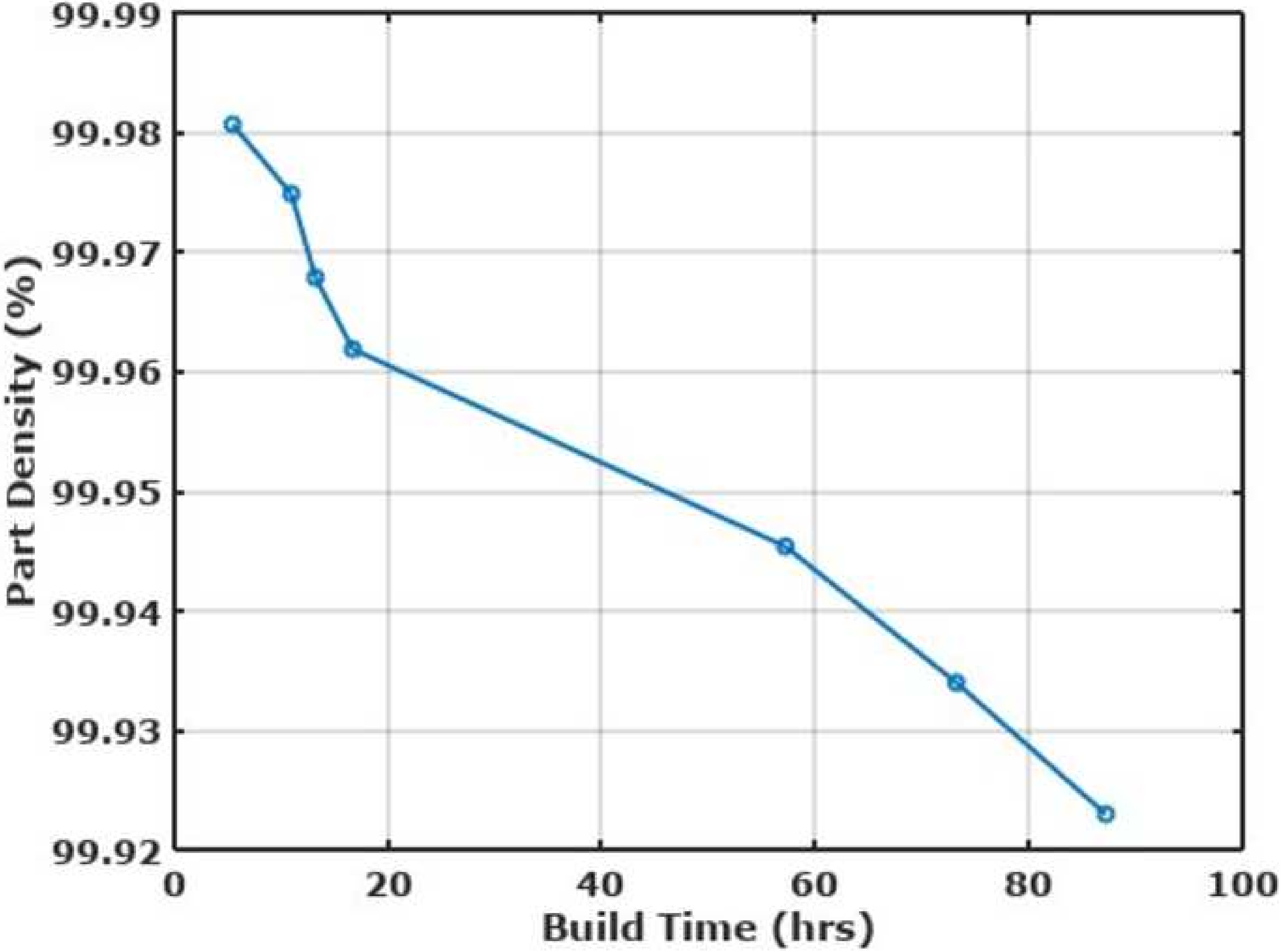
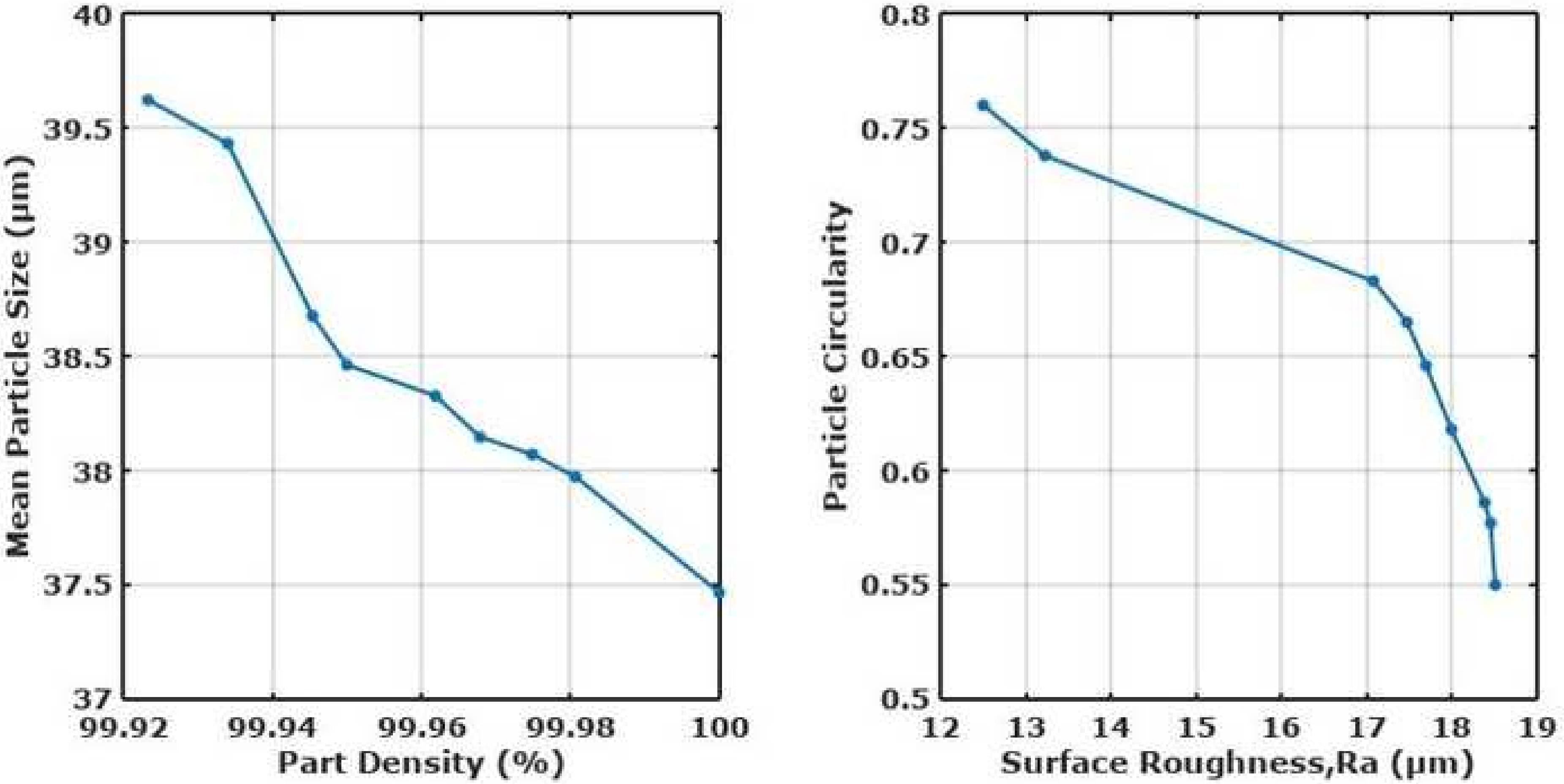

19
18
17
16
15
14
13
0
20
40
60
80
100
Surface Roughness, Ra(μm)
Build Time (hrs)

Figure 7



Part Density (%)
99.99
99.98
99.97
99.96
99.95
99.94
99.93
99.92
0
20
40
60
80
100
Build Time (hrs)

Figure 8

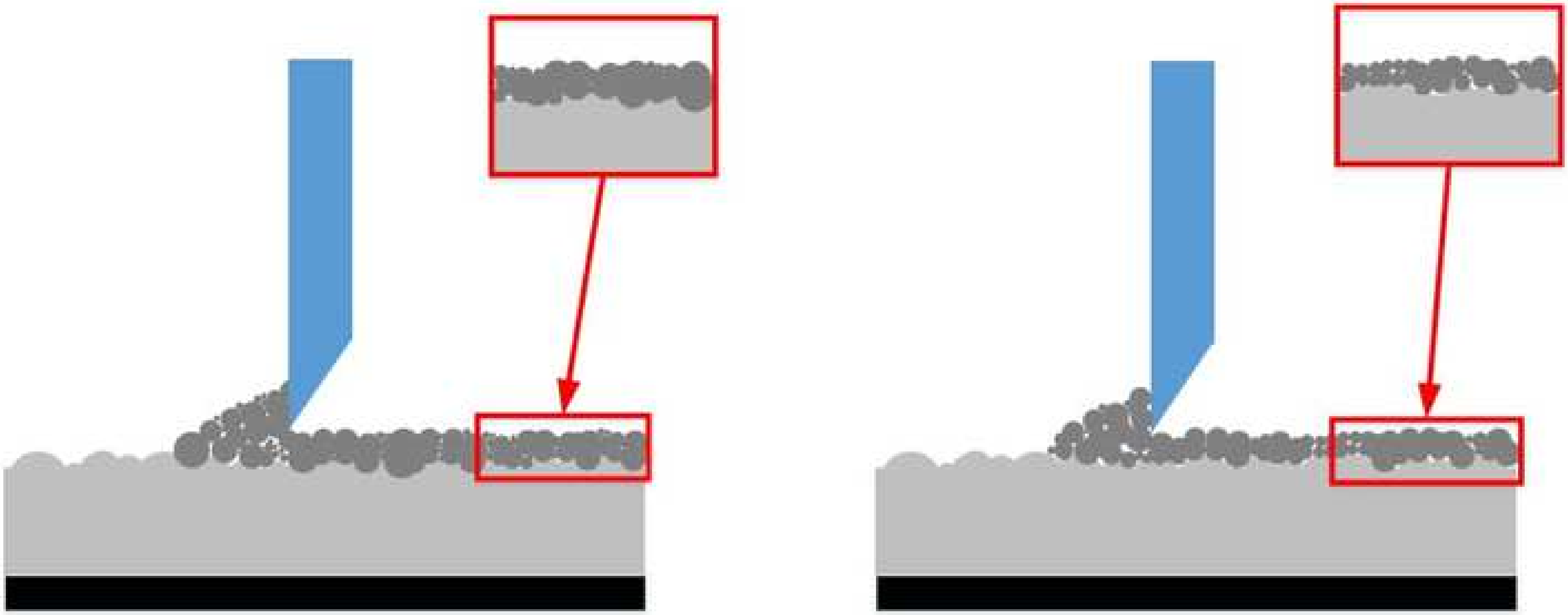


40
39.5
39
38.5
38
37.5
37
Mean Particle Size (µm)
99.92
99.94
99.96
99.98
100
Part Density (%)
0.8
0.75
0.7
0.65
0.6
0.55
0.5
Particle Circularity
12
13
14
15
16
17
18
19
Surface Roughness,Ra (µm)

(a)

(b)

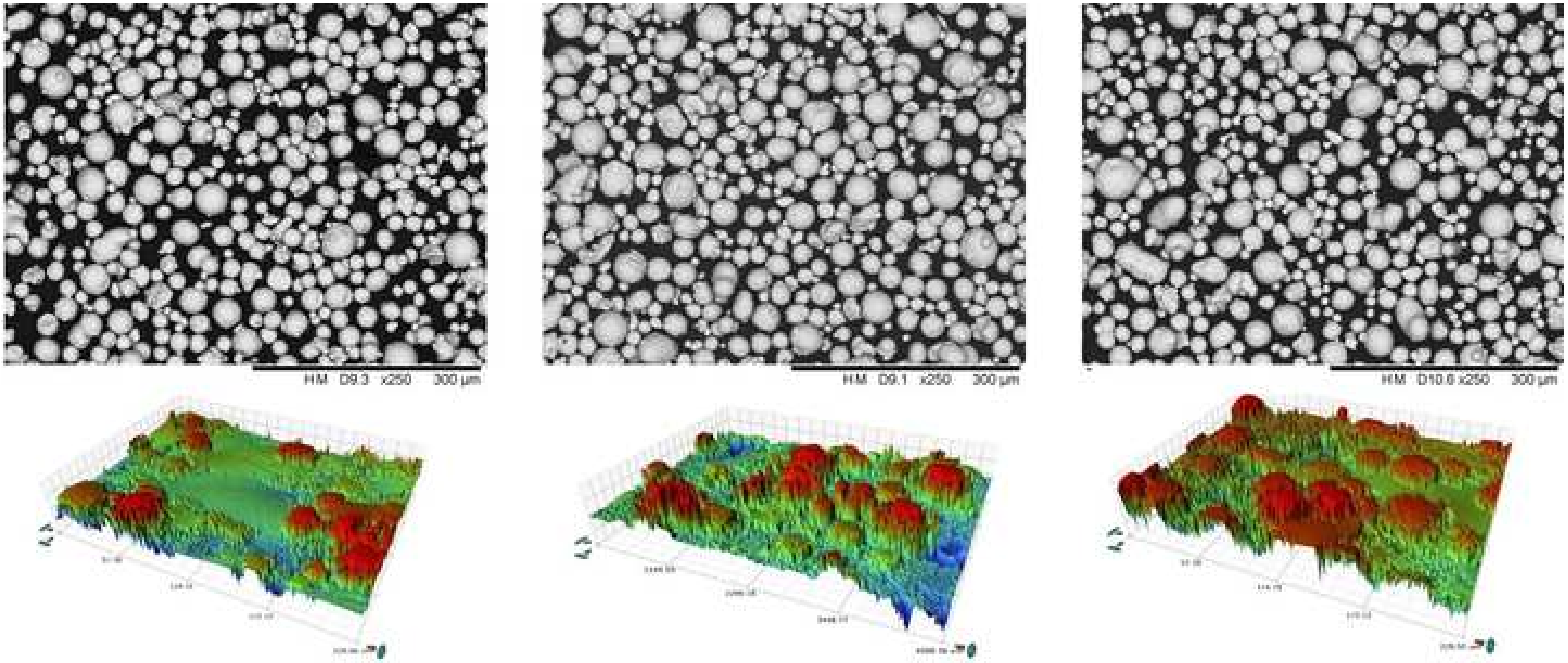
Figure 9



(a) Virgin Powder

(b) Recycled Powder

Figure 10



Mean Particle Size = 37.47 µm
Circularity = 0.76
Ra Value = 13.23 µm

(a) Virgin Powder

Mean Particle Size = 38.32 µm
Circularity = 0.64
Ra Value = 17.73 µm

(b) After 16.75 hours of use

Mean Particle Size = 39.62 µm
Circularity = 0.55
Ra Value = 18.57 µm

(c) After 88 hours of use

Table 1

Powder Characteristics	Test Method
Powder Chemical Composition	Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray (EDX)
Particle Size	Dry Dispersion Laser Diffraction
Particle Morphology	SEM Imaging and Morphology analysis

Table 2

Part Quality	Test Method	Test Part
Dimensional Accuracy	CNC Coordinate Measuring Machine	All Parts
Hardness	Rockwell Hardness	Geometry Block
Surface Roughness	White Light Interferometry	Overhang
Part Density	Micro-sectioning and Image Analysis	3 x Density Cubes