

# QUALITY OF SN-AG-POWDERS PRODUCED BY DROP-ON-DEMAND TECHNIQUE

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## ABSTRACT

Narrow particle size distributions are achievable with single fluid atomization techniques. The droplet generation involves a balance between the flow characteristics and the break-up mechanism of the fluid at the orifice exit. An experimental setup was constructed to explore the drop-on-demand mode by varying orifice diameter, plunger frequency and melt temperature. Particularly, the drop-on-demand mode is favourable to achieve small particle size distributions with geometric standard deviations down to 1.03. Sn-Ag particles were produced with this experimental setup varying the diameter of the orifices between 250  $\mu\text{m}$  and 350  $\mu\text{m}$ . Light-microscopic methods, laser diffraction and image analysis methods were used to characterize the particles. Finally, the powder was investigated with respect to mass median diameter, geometric standard deviation, and shape.

## INTRODUCTION

Metal Powders are commonly used in techniques such as powder metallurgy, metal injection molding or soldering pastes [1, 2]. Many of these techniques have certain requirements regarding the powder properties. Mostly only a certain powder size range can be used for a specific process, and when good flowability of the powder is needed, spherical powder shape without satellites is a requirement. Hence, a narrow particle size distribution and the particle sphericity are two major quality criteria for metal powders.

A common commercial technique for the production of metal powders is the gas atomization of a melt such as the close-coupled atomizer [3] or the free-fall atomizer [4]. These techniques are known to achieve high throughputs. However, gas atomizers produce powders with a comparatively high geometric standard deviation in particle size which limits the yield if only a certain fraction is usable for a process. Furthermore, the high load in the spray of these atomizers yields to an increased probability of deformed particles and satellites due to collisions.

Single fluid atomization techniques involve the simple droplet breakup first described by Rayleigh [5]. His model predicted a very uniform droplet diameter that is exactly 1.87 times the orifice diameter. An alternative technique – drop on demand – will be presented in this paper. This single fluid atomization technique involves an oscillating plunger that pushes out the volume of a single drop through an orifice (**Fig. 1**). Powders produced by drop-on-demand technique stand out by their very narrow particle size distribution (if not monodispersity) as well as by their sphericity [6,7]. The influence of major process parameters on these particle properties will be shown and evaluated in this paper.

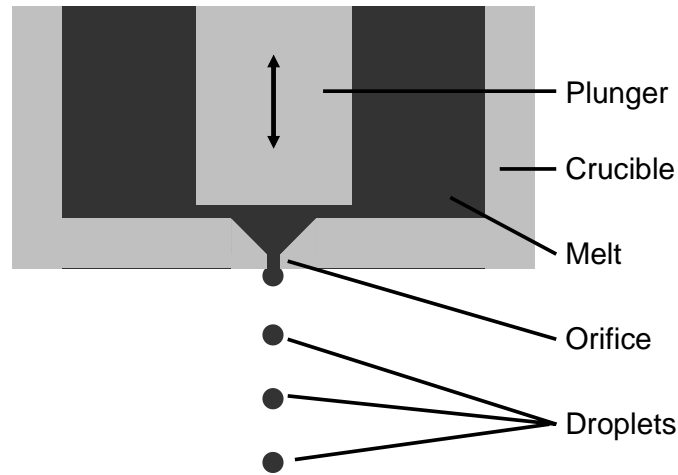


Figure 1: Drop-on-Demand (schematic)

## EXPERIMENTS

Experiments were performed with the parameters shown in table 1. The eutectic alloy SnAg3.5 (weight basis) was melted to three different melt temperatures 250, 275 and 300 °C (the eutectic temperature is 221 °C) under a nitrogen atmosphere to prevent oxidation of the melt. Orifice diameters of 250 μm and 350 μm were chosen with a ratio of orifice length to diameter  $l_o/d_o$  of unity as proposed by Sohn et al. [6]. Nozzles were machined from graphite. The vibrating frequency of the plunger was varied in a range between 40 and 110 Hz.

Table 1: experimental parameters

Parameter	Variations
Melt material	SnAg 3.5 (eutectic, melting point: 221°C)
Melt Temperature [°C]	250; 275; 300
Orifice diameter [μm]	250; 350
Ratio $l_o/d_o$ [ - ]	1
Number of orifices [ - ]	1
Plunger frequency [Hz]	40; 70; 90; 110

The droplet ejection was recorded using a high-speed camera (Kodak Ektapro HS Motion Analyzer 4540) at a frame rate of 4500 images per second (Figure 2). As the recording was performed under an angle from the bottom, droplet distances and diameters might appear to increase misleadingly with increased distance to the nozzle. In Fig. 2a and 2b the droplet ejection is shown for a 250 μm orifice and a plunger frequency of 40 Hz and 110 Hz, respectively. As expected, the increased plunger frequency leads to a decreased distance between the droplets. A stable droplet chain could be achieved.

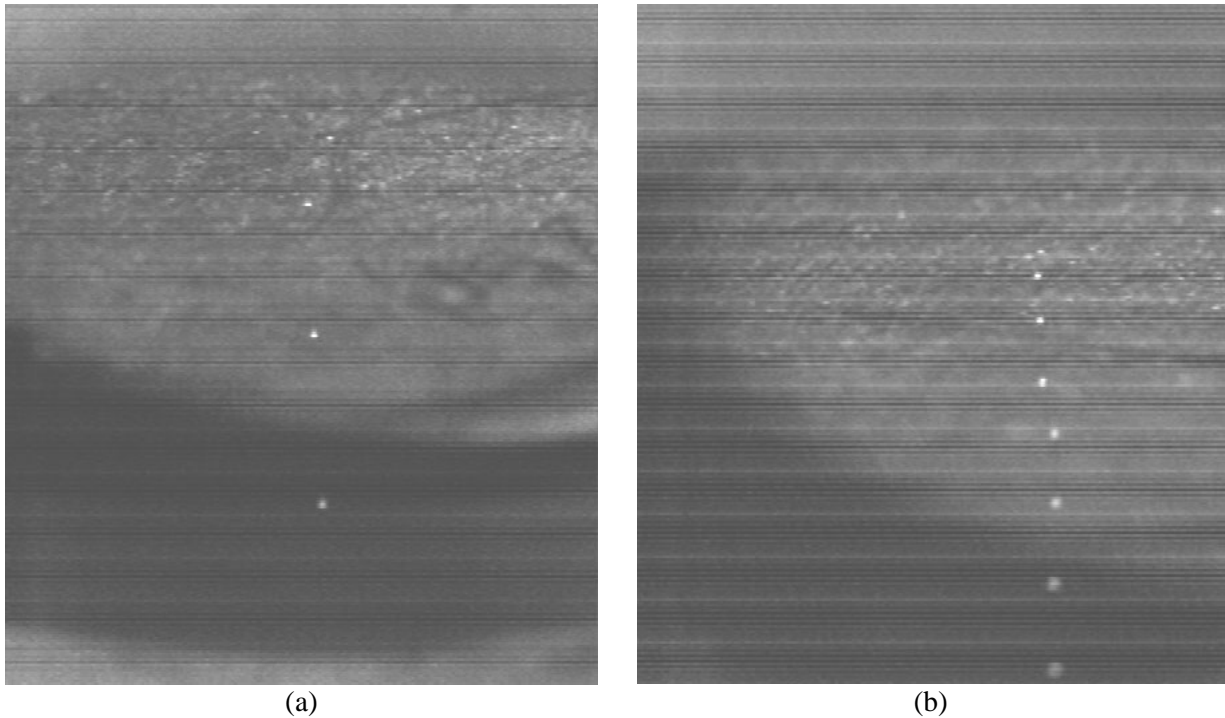


Figure 2: Process photos taken with highspeed-camera, orifice diameter: 250  $\mu\text{m}$ , plunger frequency (a) 40 Hz, (b) 110Hz

After 1 m of falling distance, the droplets were collected in water and dried for further characterization. The spray time was measured and used to calculate the average melt flow rate using the collected powder mass.

### POWDER CHARACTERIZATION

Images of the dried powder were taken using a light-optical microscope. Figure 3 (a) and (b) show images of generated powders for two different orifice sizes 250 $\mu\text{m}$  and 350  $\mu\text{m}$ , respectively. Both powders stand out due to their uniform particle size and satellite-free high sphericity.

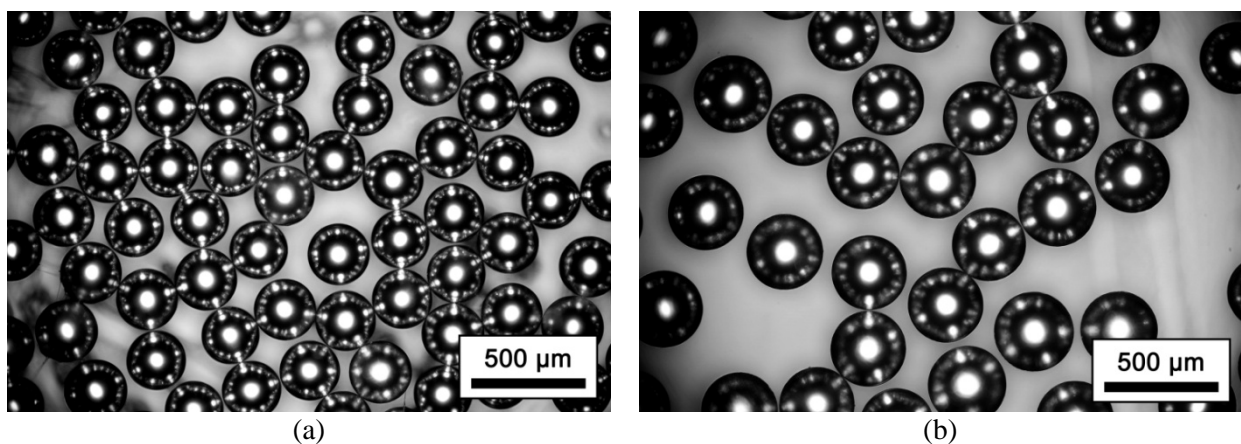


Figure 3: Light-optical microscope images of generated powders  
 (a) orifice diameter: 250  $\mu\text{m}$ ,  $f=90$  Hz,  $T=275$   $^{\circ}\text{C}$ ,  $d_{50,3}=258$   $\mu\text{m}$ ,  $\sigma_g=1.03$   
 (b) orifice diameter: 350  $\mu\text{m}$ ,  $f=70$  Hz,  $T=275$   $^{\circ}\text{C}$ ,  $d_{50,3}=326$   $\mu\text{m}$ ,  $\sigma_g=1.05$

Besides this qualitative optical characterization, the powders were characterized using laser diffractometry (Malvern Mastersizer 2000) and using image analysis (Retsch Technology Camsizer [8]) regarding their particle size distribution. The Camsizer takes images at two different magnifications of powder trickling through a measurement volume. Image analysis is then applied to

determine particle diameters and shape. Finally, a conversion of the projection area distribution to a volume distribution is applied.

Figure 4 compares mass median diameters determined with the Camsizer and laser diffraction. It can be seen that applying both techniques results in similar mass median diameters. The diameters measured with the Camsizer are systematically approximately 12  $\mu\text{m}$  below those measured by laser diffraction in the shown size range (240 $\mu\text{m}$  – 380  $\mu\text{m}$ ).

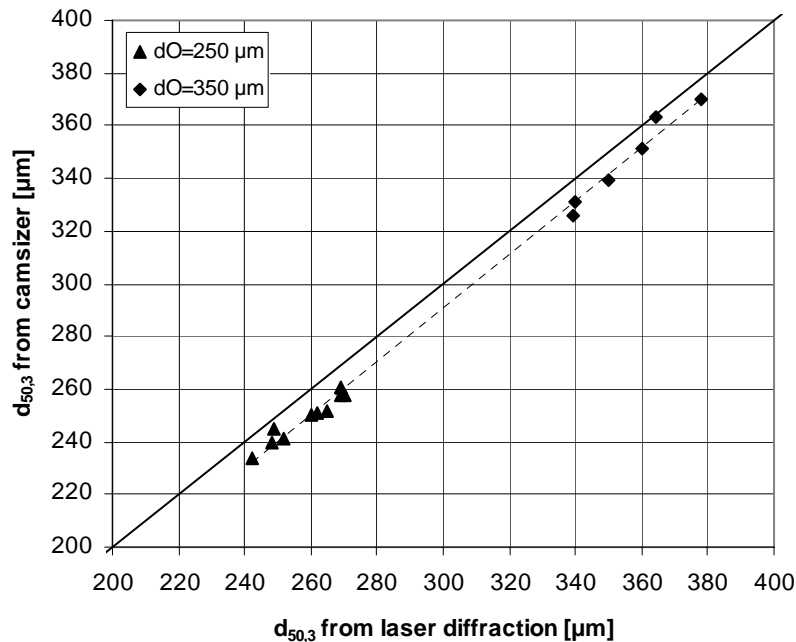


Figure 4: Comparison of mass median diameters measured with laser diffraction (Malvern Mastersizer 2000) and image analysis (Camsizer, Retsch Technology GmbH)

It was decided to use data from the Camsizer for further characterization, because it also measures shape factors for particles and mixing of particle size data from different measurement techniques is not appropriate.

Figure 5 shows the dependency of the mass median diameter on orifice diameter, melt temperature and plunger frequency. It can be seen that there is almost no influence of these process parameters on the mass median for the 250  $\mu\text{m}$  orifice: mass median diameters achieved under these conditions are  $250 \mu\text{m} \pm 5 \mu\text{m}$ . For the 350  $\mu\text{m}$  orifice, an increase of mass median diameter of 50  $\mu\text{m}$  could be noted when the plunger frequency was increased from 70 to 110 Hz.

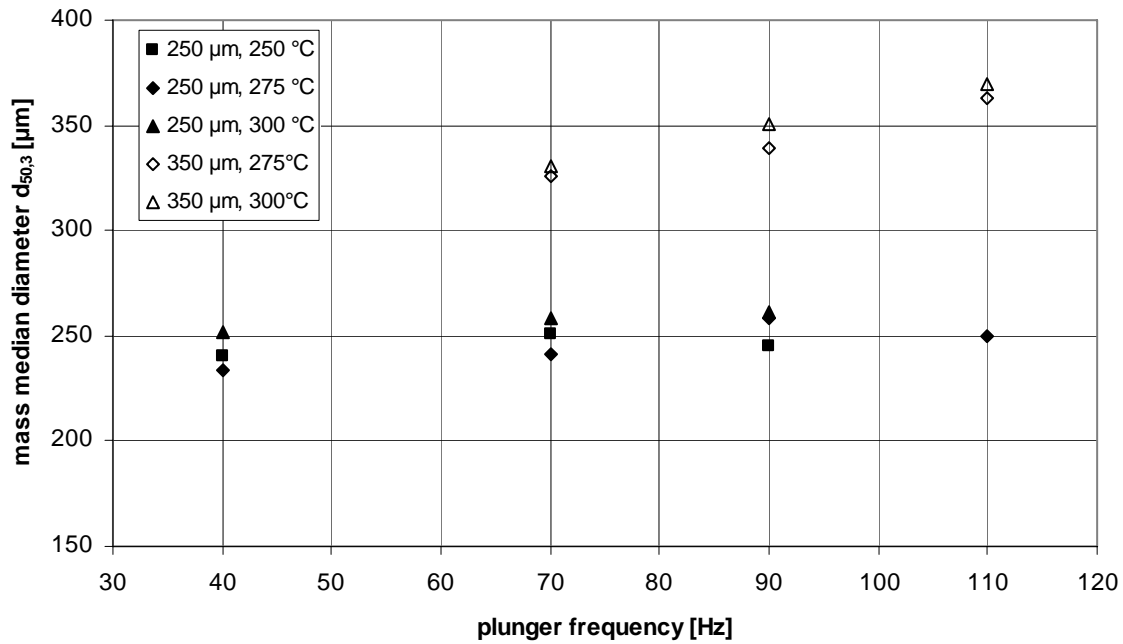


Figure 5: Dependency of mass median diameter on orifice diameter, melt temperature and plunger frequency.

One quality criterion for a powder is its geometric standard deviation as defined above. Previous experiments had shown that especially for very narrow size distributions the laser diffraction technique is not suited to resolve fine differences in the geometric standard deviation.

$$\sigma_g = \frac{d_{84,3}}{d_{50,3}}$$

The minimum geometric standard deviation that could be measured by laser diffraction was 1.25, powders that were more uniform resulted in the same standard deviation. Hence, standard deviation values as well as the mass median diameter (as shown to be nearly the same as that measured by laser diffraction) were taken from the Camsizer for further characterization.

Figure 6 shows the geometric standard deviations achieved with this process as a function of plunger frequency for different orifice diameters and melt temperatures. For the 250 μm orifice, the geometric standard deviation is almost constant in a range of 1.03 to 1.06. No trend is visible in this narrow range. For the 350 μm orifice, the geometric standard deviations are slightly higher in the range of 1.04 to 1.12. The highest value of the geometric standard deviation at 70 Hz for the 350 μm nozzle plate can be explained by the fact that not perfect drop on demand (one drop per pulse) could be achieved for these experiments. It can be said that the geometric standard deviation of the powder is almost constant for all process parameters.

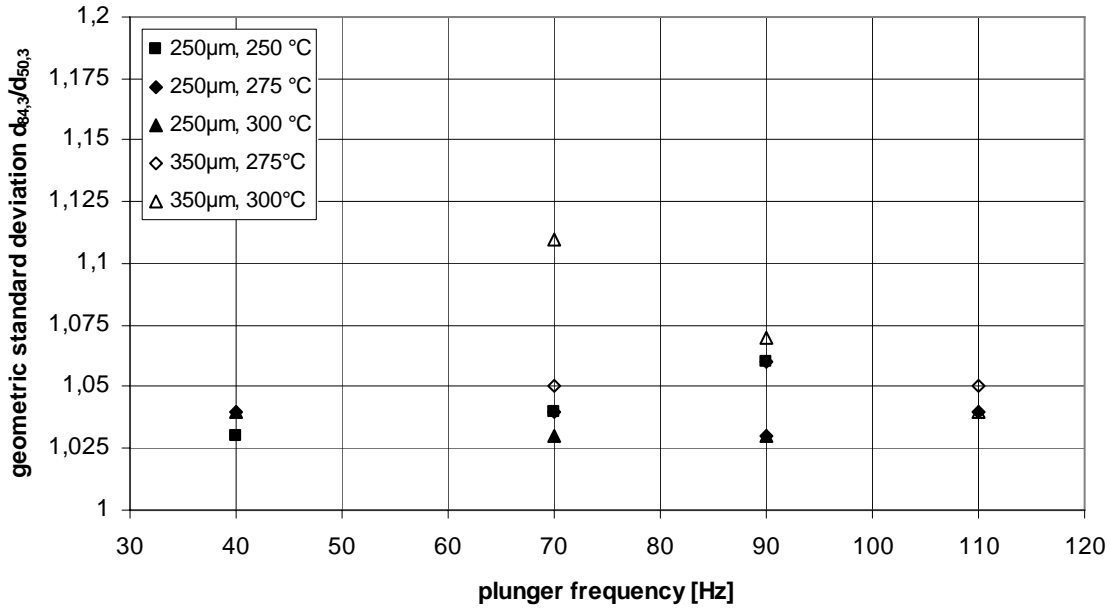


Figure 6: Geometric standard deviation as a function of orifice diameter, melt temperature and plunger frequency

The achieved mass flows with a single orifice are shown in Fig. 7 depending on plunger frequency for the two different orifice diameters and a melt temperature of 300 °C. Measured mass flows are compared to mass flows calculated from the average droplet volume and droplet frequency as

$$\dot{M}_{l,theo} = f \cdot \rho \cdot \frac{\pi}{6} d_{50,3}^3 \cdot$$

This is valid if the droplet size distribution is very narrow and stable drop-on-demand maintained. For the 250 μm orifice, very good agreement between calculated and measured mass flows is achieved, whereas there is a difference between calculated and measured values for the 350 μm orifice. For 70 Hz plunger frequency, the measured mass flow is higher than the calculated one whereas it is the other way round for 90 and 110 Hz plunger frequency. For 70 Hz plunger frequency sometimes more than one droplet was pushed out the orifice; for the other two frequencies the amplitude was sometimes not high enough to push out a droplet. Pushing out more than one droplet per plunger movements usually leads to a bigger and a smaller droplet, which affects the size distribution more than pushing out no droplet. This explains the higher geometric standard deviation at 70 Hz shown in Fig. 7.

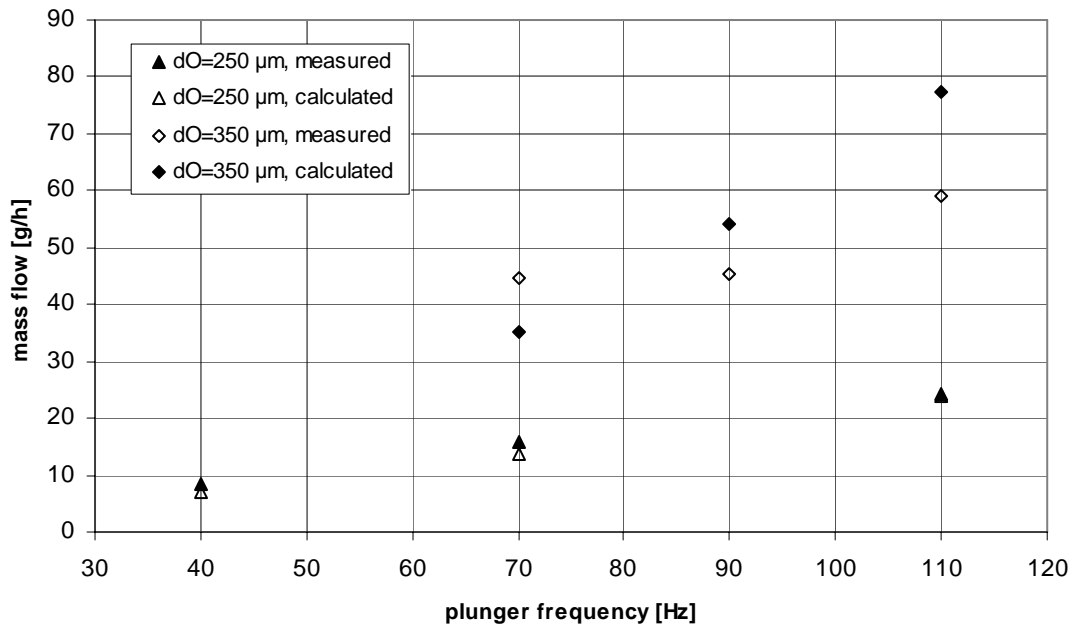


Figure 7: Mass flow achieved with a single orifice as a function of plunger frequency for two different orifice diameters, melt temperature: 300°C

Finally, the particle sphericity  $f_c$  was determined using image analysis and the images taken with the Camsizer device (Fig. 9). The sphericity is defined as the squared ratio of the equivalent diameters ( $d_p$  and  $d_A$ ) obtained from particle perimeter  $P$  and particle area  $A$ . A sphericity ( $f_c$ ) of unity refers to a perfect sphere and decreases as the particle shape is more deformed. An average sphericity of the particles was calculated for each experiment. The average standard deviation of these average sphericities was calculated to be 0.014.

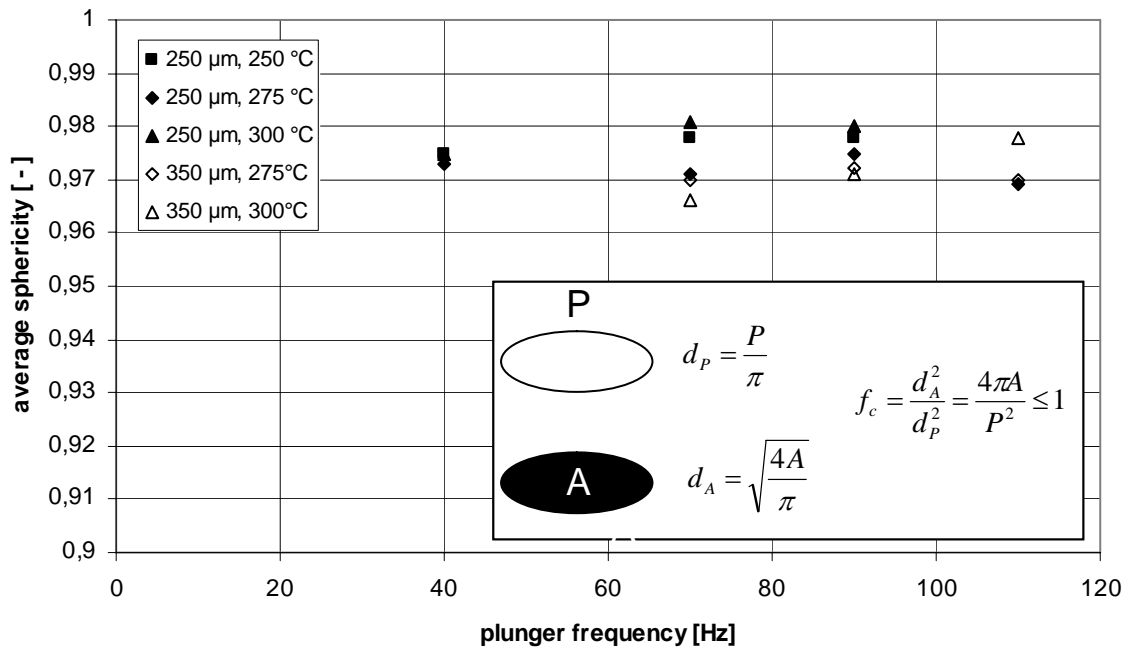


Figure 8: Average sphericity of particles as a function of plunger frequency for two different orifice diameters, melt temperature

It can be seen that sphericity values fall in a very small range of 0.965 to 0.985. Solidification shrinkage might be part of the reason for the slight deviation from perfect sphericity. There seems to be no dependency on the varied process parameters, so that over the range of variables explored highly spherical particles could be achieved.

## CONCLUSION

Powder produced with a drop-on-demand process was analysed for size distribution and its shape. Highly spherical powder with a narrow particle size distribution with geometric standard deviations in the range of 1.02 to 1.08 could be achieved. Mass median diameters were in the range of the orifice diameter (250µm and 350 µm). The achieved properties were almost independent of the major process parameters such as melt temperature and plunger frequency.

Future work will include increasing the plunger frequency as well as designs with multiple orifices to increase the throughput of the process.

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