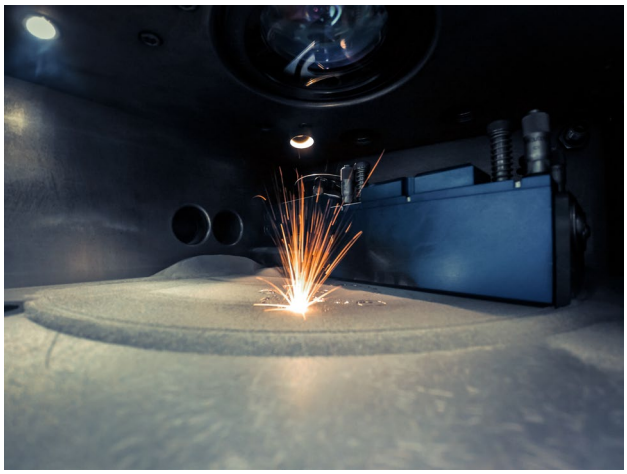
Metal powders for additive manufacturing: characterization of ultrasonically atomized magnesium

Relevant for: Dynamic image analysis, metal powders, additive manufacturing

Particle size and shape characterization were used to conduct a quality inspection of ultrasonically atomized, metallic powders for additive manufacturing. Quick quality control helped to identify errors in the used process atmosphere and greatly improve the powder quality of the next atomization process.



1 Introduction

Metal additive manufacturing is a rapidly growing industry, which offers a solution in areas where the production of complex parts from high-value materials in unique shapes is required. A few examples are aerospace, medical, and the automotive industry. Most metal-based 3D printing technologies rely on metallic powders as a feedstock material. Since the quality of the used powder directly influences the quality of printed parts and printing speed, it is crucial to monitor its properties. The influence of the powder quality can be the most prominent in powder bed techniques such as Laser Powder Bed Fusion (LPBF), Electron Beam Melting (EBM), and Binder Jetting (BJT). In these techniques, the powder is required to characterize with a high packing density and to form smooth layers, usually in the range of 25-100 µm.

1.1 Influence of the feedstock quality

The particle size distribution (PSD) of the metallic powder is a critical factor that directly affects the packing density of the powder bed, and through that the build quality and properties of the final product. A powder bed with a narrow PSD will have a more uniform energy absorption, a consistent layer-by-layer melting and an improved surface finish. On the other hand, a feedstock with a wide PSD results in a higher packing density but an inconsistent energy absorption, leading to defects such as porosity, improper fusion, and reduced strength of the final component. In conclusion, a well-controlled particle size distribution is crucial in powder bed fusion technology for achieving consistent, high-quality final products with desired mechanical and physical properties.

Another important aspect is the cleanness of the surface of metallic powders. Due to their very high specific surface, powders can readily oxidize – especially at higher temperatures. Furthermore, metallic particles can easily absorb moisture from the air deteriorating their properties. That is why the composition of the processing and storage atmosphere must be strictly controlled during the atomization processes, and powders – especially highly reactive like magnesium and its alloys - should be stored in sealed containers in protective atmospheres.

1.2 Ultrasonic atomization

Ultrasonic atomization is a liquid-to-solid process for the production of metallic powder. In contrast to the gas atomization techniques that rely on high-velocity gas, as the name suggests, ultrasonic vibrations are used to create the powders. In this approach, the velocity of sprayed particles is much lower than in gas driven processes. Therefore, ultrasonic atomizers are much smaller and do not require the construction of large atomization towers. The main principle of ultrasonic atomization is based on both vibration amplitude and surface wettability of the sonotrode. Standing capillary waves are generated when the threshold vibration amplitude in the liquid layer wetted to the sonotrode is exceeded. [1] Further increasing of the amplitude breaks the intermolecular forces of the

liquid, thus forming small droplets that are ejected from the melt and ultrasonic atomization begins.

Depending on the processed material, it is either melted in a crucible and poured into the vibrating element (i.e., induction melting) or melted directly at the sonotrode (i.e., arc or plasma melting). The lack of pressurized gas allows system miniaturization, making the process easier, faster, inexpensive, and more energy-efficient. As a result, new materials can be developed for various manufacturing processes that require powder material as a feedstock, e.g., additive manufacturing (AM) or coating technologies. The induction method is preferred for volatile materials because it prevents the evaporation of such elements (e.g. Zn-,Al-,Cu-based alloys). In contrast, the plasma melting approach is designed for materials with medium to high melting temperatures (e.g. Fe-, Ti, Nb, W-based alloys).

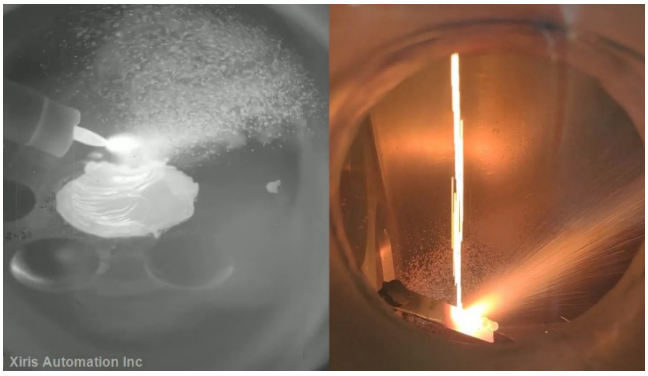


Figure 1: Metal ultrasonication with plasma (left) and induction (right) modules for different materials

Unlike conventional gas atomization, the narrow PSD of ultrasonic atomization allows the use of up to 80% of the manufactured powder for dedicated technology. The ultrasonic frequency and amplitude are the main factors influencing the PSD of the obtained powder. Considering these relationships, Table 1 summarizes the relation between the choice of ultrasonic frequency and the resulting particle size distribution.

Frequency	Particle size / Q_{50}	Technologies
20 kHz	80 – 100 µm	EBM, Directed energy deposition (DED)
40 kHz	40 – 50 µm	LPFB
60 kHz	30 – 40 µm	Binder jetting, LPBF

Table 1: Examples of optimal ultrasonic frequency for preparing powder feedstock for different AM technologies

Ultrasonic atomization can accelerate the development of new materials. Material changeover is quick, due to the small size of the required processing chambers and allows many research teams to work with a single device. Thus, different teams working with distinct technologies and materials can easily switch from manufacturing powders via LPBF to direct metal deposition or EBM or even incorporate thermal spraying steps for coating technologies.

In this application report we demonstrate the importance of parameter optimization during ultrasonic atomization, such as atmosphere and pressure on the example of magnesium.

2 Experimental

Magnesium (99.8 wt.%) was atomized using an induction module of AMAZEMET´s rePowder device. Melting of the material was performed in a graphite crucible, and atomization was carried out on a refractory plate with a frequency of 40 kHz.

Two atomization processes were done: the first one with standard chamber purging and flow ratio for Ar shield gas (Batch A). The second process with optimized process parameters for reactive materials – with additional argon purging cycles and a higher flow of Ar gas in the chamber to remove any metal vapors that could deposit onto the atomized powder particles (Batch B).

The particle size and shape were analyzed by Anton Paar Litesizer DIA 500 dynamic image analyzer in free fall mode. Additional scanning electron microscope images were taken by Hitachi S-3500N.

3 Results and Discussion

Dynamic image analysis provides a unique opportunity to analyze the same sample based on multiple size parameter concepts, which allows highlighting various properties of the sample.

The minimum Feret diameter (xFmin) – or the shortest dimension of each particle – highlights the diameter of spherical particles, while neglecting deformities, and imperfections (e.g. satellites, fusion). Figure 2 displays the PSD comparison of the two batches, based on the xFmin in volume weighting, Table 2 summarizes the corresponding Q-values.

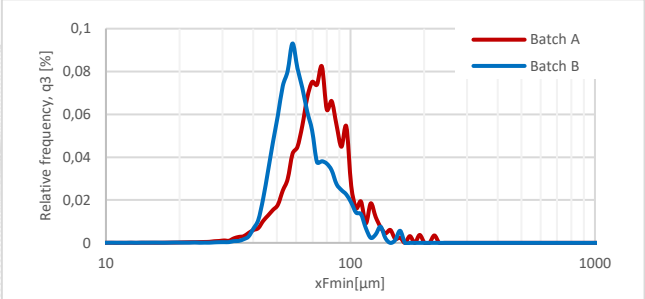


Figure 2: xFmin volume-weighted particle size distribution of magnesium manufactured in two different atmospheres

As seen on the PSD and reflected in the Q-values as well, optimizing the atmosphere of the atomization process leads to an increased percentage of particles

ejected in the expected size range (compare with Table 1).

xFmin	Q_3 = 10% [µm]	Q_3 = 50% [µm]	Q_3 = 90% [µm]
Batch A	54.1	76.3	107.8
Batch B	49.2	63.8	96.8

Table 2: Volume weighted xFmin Q-values of two batches of magnesium powder

Besides the primary particle size, the shape of the resulting particles is also of utmost importance. Metallic additive manufacturing expects a feedstock consisting of perfectly spherical particles. Elongated particles are difficult to separate after production and; therefore, can introduce irregularities into the powder bed. Dynamic image analysis offers a possibility to analyze the shape of the particles as well, based on several parameters. Aspect ratio (AR) is the ratio of the minimum and maximum Feret diameter. The closer a particle is to a perfect sphere, the closer the aspect ratio is to 1. Figure 3 displays the density distribution of the aspect ratio, weighted by volume of the two magnesium samples.

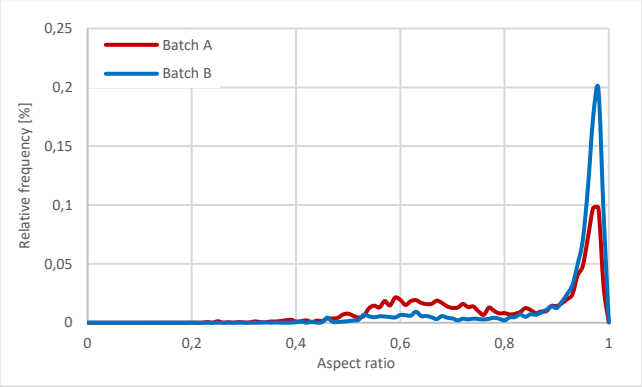


Figure 3: Density distribution of aspect ratio, weighted by volume of two magnesium samples

The amount of strongly non-spherical particles is significantly larger in case of an unoptimized atmosphere inside the atomization chamber. This is also reflected in the cumulated distribution of the aspect ratio. While less than half of the particles are close to spherical in case of Batch A, in a controlled atmosphere this ratio is increased to over 75% (Table 3)

	Aspect ratio > 0.9
Batch A	45%
Batch B	77%

Table 3: Cumulated percentage of particles with an aspect ratio larger than 0.9 of two magnesium samples

Another important aspect of the final product is the quality of the resulting surface. As discussed in the introduction, magnesium is prone to oxidation in an uncontrolled environment, which can be recognized in a characteristic flaky surface. Normally, a sensitive investigation of the surface is performed by scanning electron microscopy (SEM), which is an expensive and time-intensive approach.

Figure 4 compares single particle images from the two batches, captured by the Litesizer DIA 500.

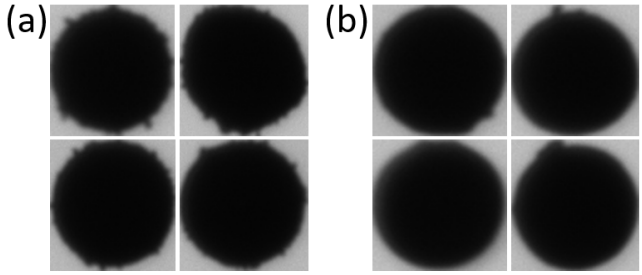


Figure 4: Single particle images taken by Litesizer DIA 500 of magnesium Batch A (a) and Batch B (b)

It can be clearly seen how the unoptimized atmosphere in the first test led to the characteristic flaky oxidation of the freshly produced magnesium (Figure 4a), while in the optimized run the resulting surface is significantly cleaner (Figure 4b).

Figure 5 displays scanning electron microscope images taken of the same samples, confirming the observations made based on the DIA measurement analysis.

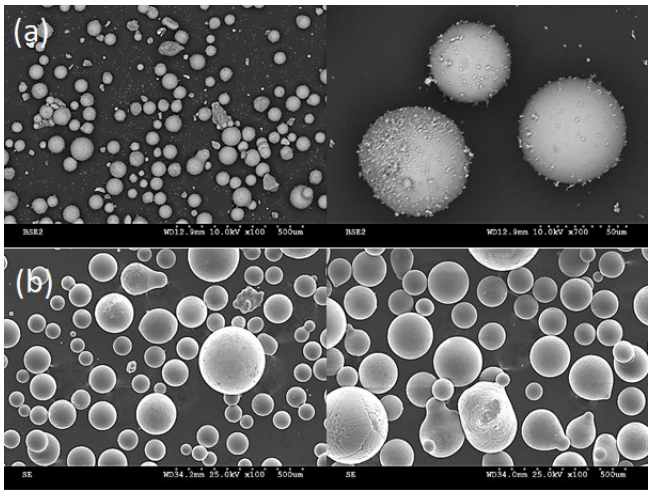


Figure 5: Scanning electron microscope images of Batch A (a), and Batch B (b)

4 Summary

Two batches of magnesium were manufactured using the rePowder ultrasonic atomizer from AMAZEMET. The first batch was done with a standard procedure for induction atomization, the second batch was produced after process optimization for reactive materials like Magnesium. The particle size and shape parameters were measured and analyzed using the Litesizer DIA 500 from Anton Paar. The analysis has revealed, that the product produced in a standard

process showed a generally broader particle size distribution, as well as a larger median particle size. Comparing the aspect ratio distribution has also shown that by careful adjustment of the atomization parameters, a 50 % increase of optimally shaped particles by volume can be achieved. The individual particle images were used to identify oxidation of the surface, which could have a detrimental effect on the final manufactured product, if it goes undetected. The findings were confirmed by scanning electron microscope images.

In summary, dynamic image analysis is an excellent tool for quick assessment of the production quality of metallic powders manufactured as a feedstock for additive manufacturing processes, as well as finding the optimal production parameters.

5 References

1. Lierke, E. G., and G. Griesshammer. "The formation of metal powders by ultrasonic atomization of molten metals." Ultrasonics 5.4 (1967): 224-228.2.

Contact Anton Paar GmbH

Tel: +43 316 257-0

example@anton-paar.com | www.anton-paar.com