CHAPTER 8

Double Slit Nozzle Design and Production of Metal Powders by Gas Atomization

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Introduction

In this section, Grade AA1070 (Etial-7) pure aluminum metal was atomized using the gas atomization method. A dual-slit nozzle with both sonic and supersonic zones was designed to increase the gas atomization efficiency and to obtain a narrower powder distribution. Our original design of the dual-slit nozzle in this study was expected to solve three main problems. The first is to control the liquid metal viscosity of the gas released from the sonic zone with low pressure and speed, thereby facilitating its transfer to the atomization zone and assisting the formation of smaller spherical particles. The second one is to eliminate the solidification problem at the end of the metal flow tube, which is a major problem with close-coupling systems, and the formation of positive pressure. The third one is to prevent the shutting of the gas expansion zone, also called the closed transition zone, by keeping the pressure fixed using supersonic nozzle geometry. Using a dual-slit nozzle integrated into the gas atomization system, production parameters (gas pressure, fusion temperature, flow tube diameter, etc.) were optimized and aluminum metal powders with high added value were produced.

Next to steel, the second most commonly used material in the world is aluminum alloys (Gökçe et. al., 2017). The “Powder Metallurgy (PM)” method, an advanced procedure for manufacturing technological material, is a significant method regarding small-piece production and cost reduction. With technological developments, aluminum and its powders have enabled the production of high-durability parts thanks to its low density and corrosion resistance. Aluminum powders in particular find much use in defense, aviation, and automotive industries. Aluminum is used in various electronic circuit parts because it remains unaffected by magnetic field. The gas atomization

technique was first used in the development of aluminum alloys around the mid-18th century (Akkaş et. al., 2019). Metal powders, being rather economical, are used in the production of parts that are difficult and costly to produce, particularly in machining. The necessity of fine grains in the field of industry has gradually increased; moreover, special requirements are present for the production of spherical fine powders (Urionabarrenetxea et. al., 2021). Today, close-coupling nozzle systems are used in atomization systems due to high production rate, powder quality, and production ratio. In the atomization of fused metals, atomized grains complete their solidification during their freefall inside the tower (Arkhipov 2016). High-pressure gas atomization systems are used to satisfy the need for spherical grains. Keeping the atomization conditions under control is critically important to attain a fine and spherical product with high performance (Liu et. al., 2018). The grain size of powders produced by the close-coupling gas atomization technique varies between 1µm and 1000µm (Zhang 2020). Among the gas atomization parameters, fusion temperature, nozzle geometry, gas pressure, and inner diameter of the flow tube greatly affect the particle size and distribution. Ünal et al. described gas atomization as the process of pulverizing a bundle of liquid metal using pressurized gas and claimed that grain size and distribution can be controlled by optimizing variables such as nozzle geometry, gas pressure and flow, and inner diameter of the flow tube and that the cooling speed of grains directly affects powder shape (Ünal et. al., 2008). Berndt et al. stated that the gas atomization technique is the most effective method of manufacturing fine and spherical metal powders (Berndt et. al., 2008). Sarı, on the other hand, describes gas atomization as the process of cutting and breaking metal drops into pieces by transferring kinetic energy from gas expanding at high speed to liquid metal (Sarı 2010). Tıng et al. claimed that liquid metal attains its spherical shape while freefalling inside the tower and cooling down after pulverization and that the resulting grains would range from 1µm to 1000µm in size; they concluded that the tower should be filled with a noble gas to protect the excessively-heated liquid metal from surface oxides during atomization (Ting et. al, 2002). Antipas stated that the properties of the gas and the liquid metal are the major factors in determining the powder shape (Antipas 2009). In their study, Miller et al. found that the gas exhausted from the nozzle at a higher speed yields smaller sizes (Miller et. al., 1997). German described the usability of nitrogen, argon, carbon dioxide, and helium as atomization gases German 2007). In a study, Yıldız emphasized the importance of using noble gases for metals with high oxygen affinity (Yıldız 2007). Literature includes several studies on the optimization of the atomization process. Mates et al. studied four different nozzles and reported that droplet pulverization is carried out by dynamic gas pressure and takes place at the tip of the nozzle. They have determined that using a supersonic nozzle and starting the atomization in the molten metal outlet, combined with increased dynamic gas pressure, can yield finer grains (Mates et. al., 2000). Mates and Settles reported high dynamic pressure and higher efficiency compared to close-coupling systems in their circular-slit supersonic nozzle design (Mates & Settles 2005). In their study, Aksoy and Ünal showed smaller grain sizes with increased atomization gas pressure. The gas/liquid mass ratio increases with shorter flow tube outlet length, and the decrease in molten metal flow rate is due to the effect of gas circulation area under constant pressure (Aksoy & Ünal 2023). In a study, Özbilen described sterilization as a structure formed by smaller grains with shorter cooling time sticking to larger grains with longer cooling time while grains of differing temperatures and sizes are drifting inside the tower. In addition, they suggested the possibility of the amount of oxygen and gas pressure in the environment controlling coarse grain formation when the optimized metal has high oxygen affinity, and the direct influence of a lack of homogeneity in the thickness of powder surface oxides on surface roughness (Özbilen 1999).

Despite the bulk of literature in the opposite direction, an original close-coupling dual-slit nozzle system design was used to produce AA 1070 (Etial-7) pure aluminum powder. In particular, the effects of parameters such as dual-slit nozzle performance, fused metal temperature, and gas pressure, and the inner diameter of the flow tube on

grain shape and morphology were studied in the production process.

Material and Method

Using our original design of a dual-slit nozzle, Grade AA 1070 (Etial-7) pure aluminum metal was atomized via the gas atomization method. By using a dual-slit nozzle of an original design, parameters directly affecting grain size and distribution in powder production such as gas pressure, inner flow tube diameter, and molten metal temperature were modified to allow the production of pure aluminum powder with highly economical value, small size, and narrow size distribution. High-purity argon (Ar) gas was used in our studies. The gas pressure of 15 to 40 bars was used, and according to these gas pressure values, the fusion temperature was adjusted to remain between 810ºC and 830ºC and the inner diameter of the molten metal flow tube between 3mm and 5mm. All these specifications were combined to achieve optimal conditions, making it possible to reach the targeted powder size and distribution. About 80.82 percent of the metal used at the start of the atomization was turned into grains of spherical morphology. The loss is 10.18 percent, which is made of dust stuck on the inner walls of the tower and escaped the vacuum pump filter. Grains obtained by atomization were individually gathered from dust collectors and cyclone dust collectors and grain sizes were measured. Average grain size was measured as 25.4µm. Gathered grains were properly packed and stored. Analyses of grain size, XRD, SEM, and BET were conducted on the obtained samples and the results were shared at the end of the study.

Table 1.
Chemical composition of AA 1070 (Etial-7) pure aluminum

Elements	Al	Fe	Si	Ti	Mn	Zn	Cu	Mg
Ratio (%)	99,7	0,25	0,20	0,03	0,03	0,04	0,04	0,03

Grade AA 1070 (Etial-7E) pure aluminum used in this study was obtained from Eti Aluminum Inc., Foundry Management. Pure aluminum powder was produced by using a close-coupling gas atomization unit with a vertical design. Tables 1 and 2 illustrate the chemical composition and physical properties of Grade AA 1070 pure aluminum ingot.

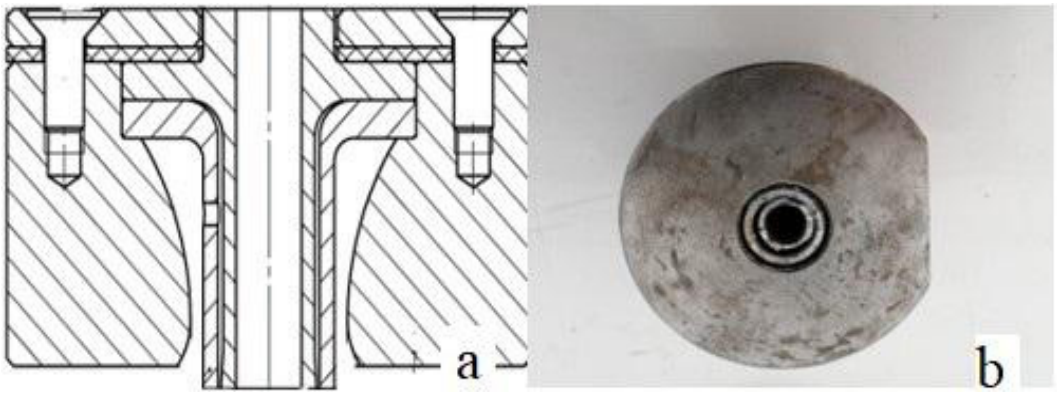
Table 2.
Physical properties of AA 1070 (Etial-7) pure aluminum

Physical Properties	
State of matter	Solid
Density	2.7 g/cm^3
Fusion point	660 °C
Boiling point	2519 °C
Fusion heat	10.71 kJ/mol
Vaporization heat	294 kJ/mol
Heat conductivity	237w/mK
Brinell hardness	245

Our dual-slit nozzle of the original design is made of stainless steel. As seen

in Figure 1, it has two areas for gas transfer and geometry. In this original work, we combine two different geometries, known in the literature as Laval and Mannesmann types. This nozzle can be installed and operated on both close-coupling and freefall systems. Situations that would interrupt the atomization process, such as solidification inside or at the end of the metal flow tube, positive pressure formation, or gas expansion zone closure, while the trials were conducted on our double-slit nozzle design. The nozzle was installed on the top center of the atomization tower. While atomization gas was connected and transferred, different values for influx pressure were studied and the optimal range was determined by using a manometer.

Figure 1.
Cross-section of the dual-slit nozzle (a) an image of dual-slit nozzle (b)



The outlet length of the flow tube was specified as 2.2mm by the nozzle design. This design choice allows the graphite to act like a flow tube and keep the system operational in case the flow tube breaks due to thermal shock. The flow tube and graphite pot are heated before each atomization process to eliminate thermal shock.

The grain size of the powders obtained from Grade AA 1070 pure aluminum was measured via Malvern Mastersizer 2000 in Eti Aluminum Inc., Q/C, and R&D Laboratory. Phase analyses of the powders obtained from Grade AA 1070 pure aluminum were carried out with an XRD device of GNR-EXPLORER brand in Eti Aluminum Inc., Q/C, and R&D Laboratory. This device has a θ/θ vertical goniometer design. The X-ray source is copper. Analysis of the pure aluminum powder product was carried out with a scanning angle of 5 to 70 degrees. Characterization of the powders produced via atomization for shape and morphology was conducted using a scanning electron microscope (SEM). Surface area inspections on the powders produced after optimal conditions were achieved were carried out using NOVA 4200e BET in Eti Aluminum Inc., Q/C, and R&D Laboratory. Powder samples went through a degasser at 105ºC before analysis.

Results and Discussion

Grade AA 1070 pure aluminum was atomized using a dual-slit nozzle of our original design, and optimal conditions were determined. Raw material obtained as ingots was reduced to a size that fits in the furnace. Atomization parameters including gas pressure, inner flow tube diameter, and fusion temperature were optimized.

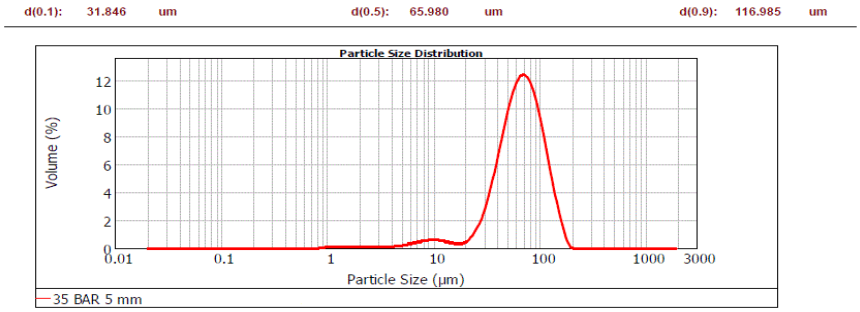
Table 3.
Powder production trials

Trial No.	Inner Flow Tube Diameter (mm)	Temperature (°C)	Gas Pressure (bar)	Average Grain Size (d50) (µm)
1	5	810	15	99.5
2	5	810	25	80.3
3	5	810	35	71.8
4	5	820	15	90.9
5	5	820	25	82.6
6	5	820	35	68.6
7	5	830	15	88.6
8	5	830	25	74.3
9	5	830	35	66.0
10	4	810	15	90.3
11	4	810	25	74.3
12	4	810	35	63.2
13	4	820	15	87.6
14	4	820	25	73
15	4	820	35	61.6
16	4	830	15	83.9
17	4	830	25	70.5
18	4	830	35	58.6
19	3	810	15	81.2
20	3	810	25	63.1
21	3	810	35	50.8
22	3	820	15	78.2
23	3	820	25	53.1
24	3	820	35	41.15
25	3	830	15	63.5
26	3	830	25	37
27	3	830	35	25.4

Optimized parameters are as follows: 35 bars for gas pressure, 3mm for inner flow tube diameter, and 830ºC for fusion temperature. Powder yield underwent sieve analysis at first. As seen in the trials shown in Table 3, the smallest grain size was 25.4µ, obtained with a temperature of 830ºC, gas pressure of 35 bars, and an inner flow tube diameter of 3mm. Grain collected from the tower and cyclones were mixed to homogeneity and grain size analysis was carried out (Figures 2-4). Figure 4 illustrates the device data. A smaller

inner flow tube diameter as opposed to increased gas pressure and fusion temperature reduces grain size. Gas pressure of 40 bars and above is demonstrated to negatively affect powder morphology and grain size. The resulting powders have a leaflike appearance.

Figure 2.
Results of the grain size analysis for Trial 9.

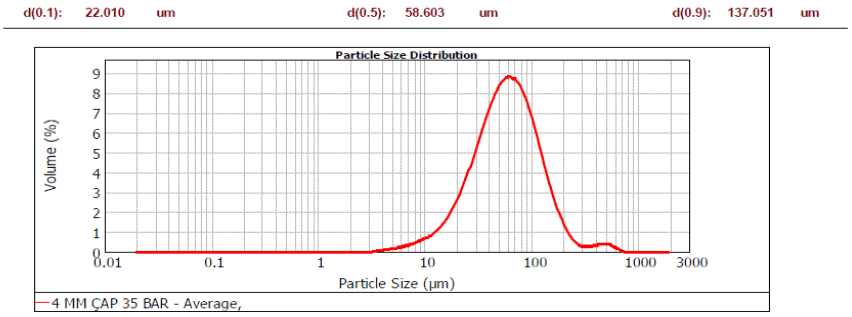


Size (µm)	Volume In %
0.010	5.41
20.000	16.50
44.000	

Size (µm)	Volume In %
44.000	37.41
74.000	38.41
149.000	

Size (µm)	Volume In %
149.000	2.28
2500.000	

Figure 3.
Results of the grain size analysis for Trial 18.

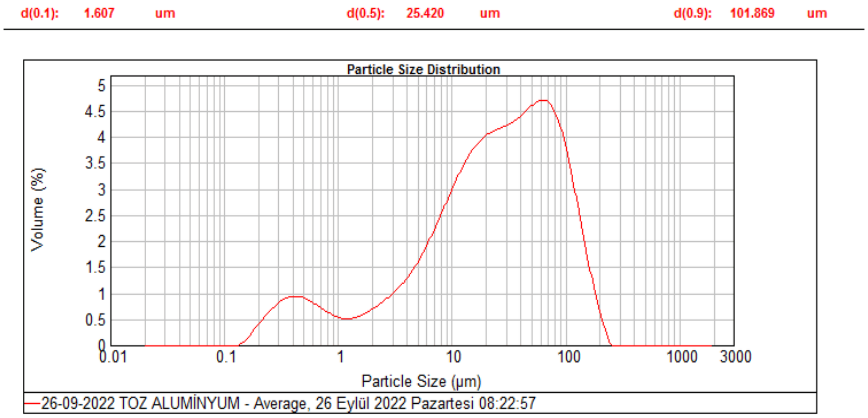


Size (µm)	Volume In %
0.010	8.25
20.000	26.10
44.000	

Size (µm)	Volume In %
44.000	28.97
74.000	28.74
149.000	

Size (µm)	Volume In %
149.000	7.94
2500.000	

Figure 4.
Results of the grain size analysis for Trial 27.



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010		44.000		149.000	
20.000	43.59	74.000	15.74	2500.000	2.86
44.000	21.73	149.000	16.07		

Table 4.
Device data on grain size

Trial No	D_{10}	D_{50}	D_{90}
9	31.85	65.98	115.99
18	22.01	58.6	137.05
27	1.6	25.4	101.9

Results of the grain size analysis suggest smaller grain sizes with constant temperature and gas pressure and smaller inner flow tube diameter. Narrowing down the flow tube reduces the amount of liquid metal transferred to the atomization zone at a given time while increasing the kinetic energy transferred by the gas at the same rate, which results in smaller grains.

Powder samples pressed into the XRD sample tray under 20 kN of pressure were analyzed in the range $2\theta = 5 - 70^{o}$. X-ray diffraction is a non-destructive testing technique that does not damage the crystalline structure and phases of the material. XRD analysis results for coarse and fine grains are shown in Figures 5 and 6.

Figure 5.
XRD chart for Trial 18.

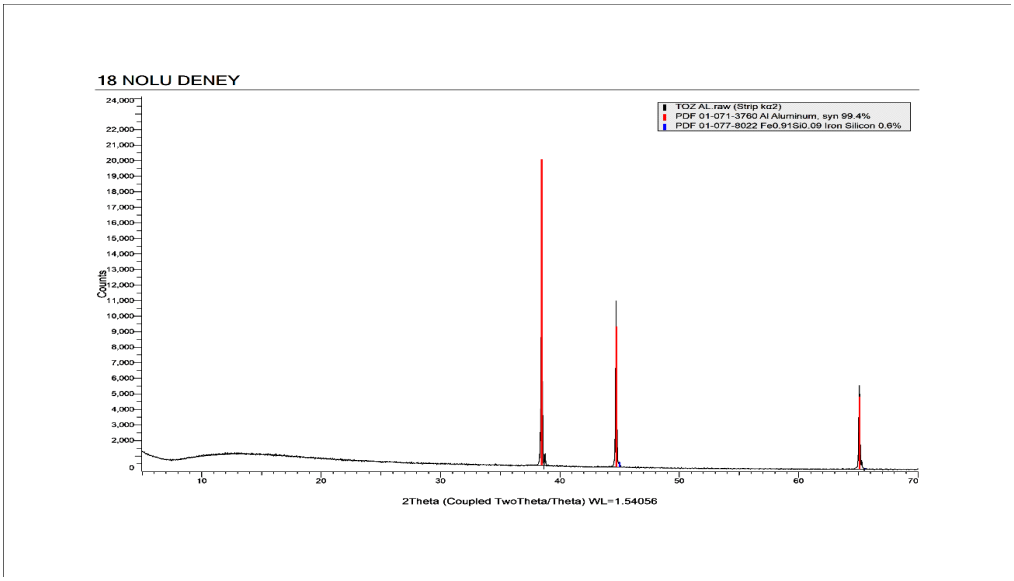
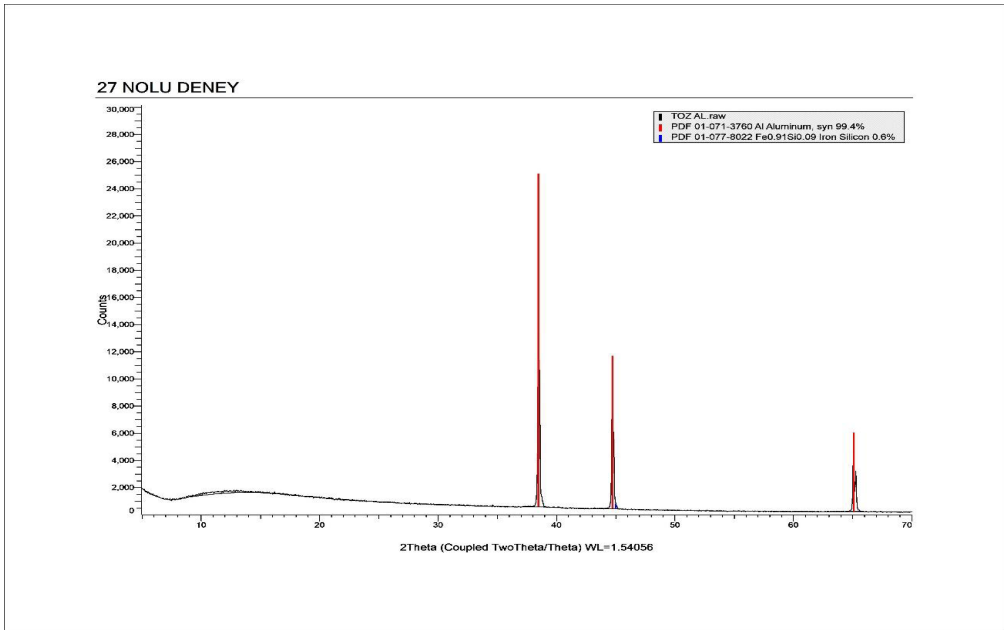
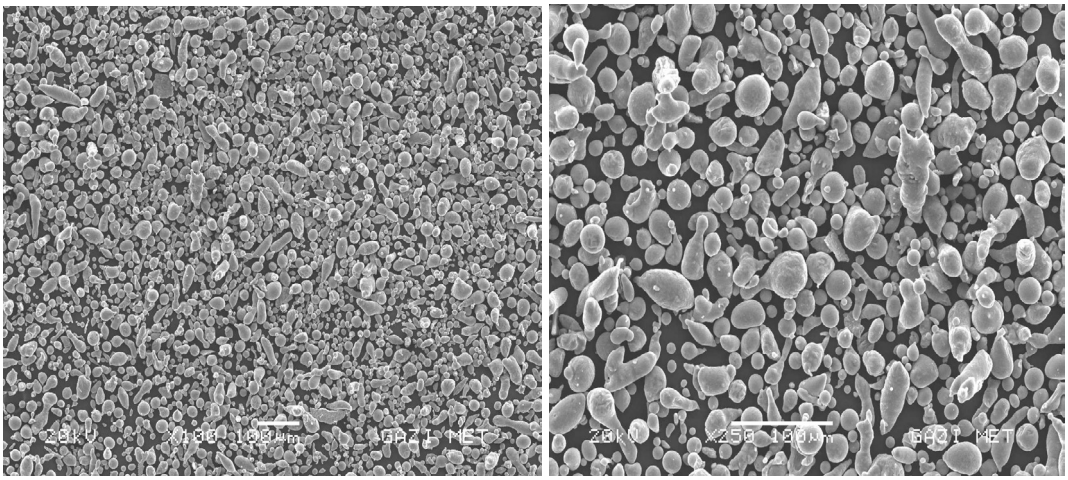


Figure 6.
XRD chart for Trial 6

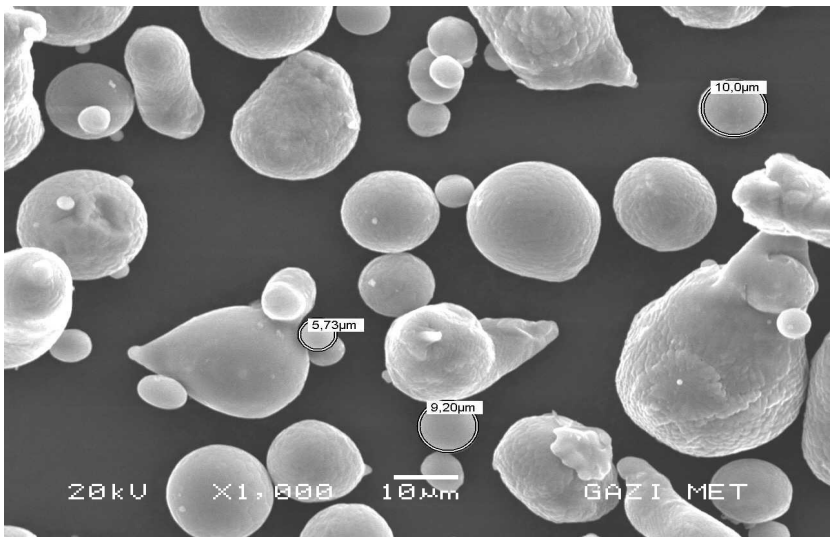


Powders obtained in Trial 27 were pressed and analyzed in the XRD device in the range 2θ = 5 – 70º. Inspection of the diffractogram shows metallic aluminum as the main phase structure. Data from trials 9, 18, and 27 in particular were included in SEM analyses, and the device results are detailed below. Specifically, the effects of temperature, gas pressure, and inner flow tube diameter optimization in this study were verified by SEM images as well (Figure 7).

Figure 7.
SEM images for Trial 9



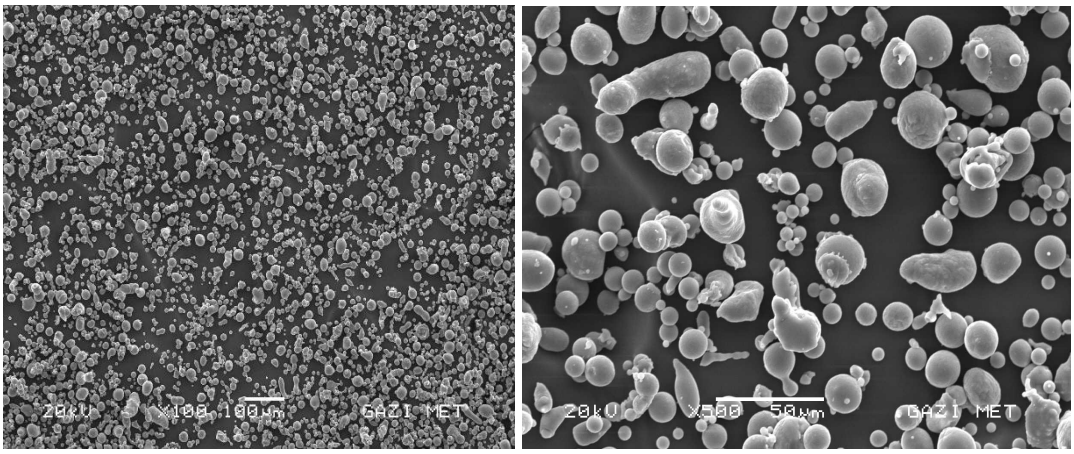
(a) (b)



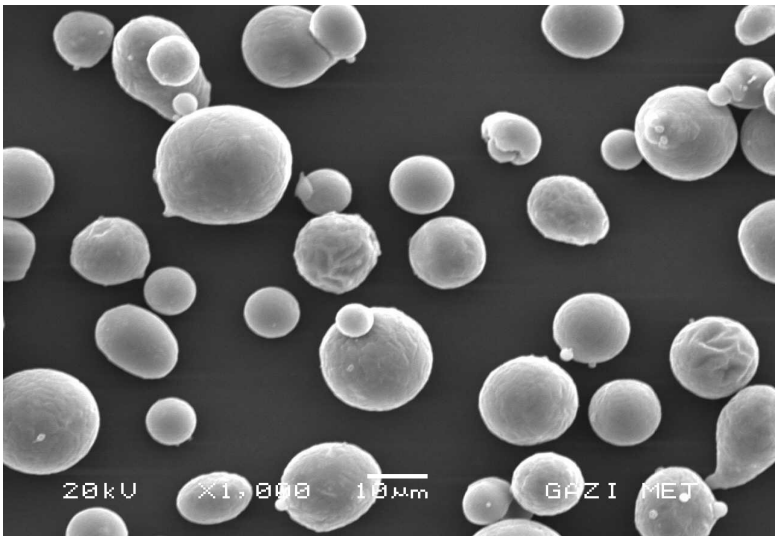
(c)

SEM images seen in Figure 7 (a), (b) and (c) show powders of d50 = 66µm from Trial 9, magnified by 100, 250, and 1000, respectively. Inspection of SEM images shows no regular morphology for the powder yield, with smaller grains being spherical and larger ones forming teardrops and other shapes. SEM images in Figure 8 (a), (b) and (c) show powders of d_{50} = 58.6µm from Trial 18, magnified by 100, 250, and 1000, respectively. More spherical formation and occasional satellization is observed compared to Trial 9.

Figure 8.
SEM images for Trial 18

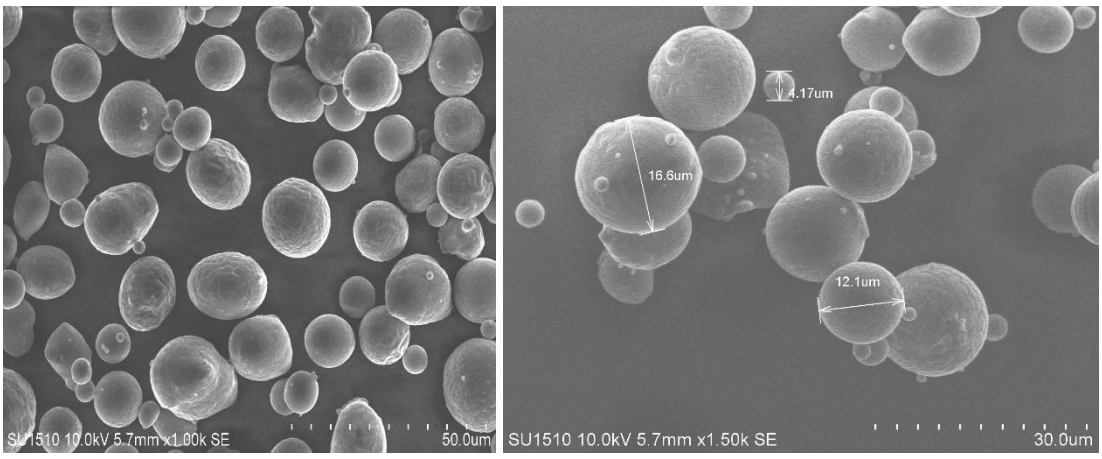


(a) (b)



(c)

Figure 9.
SEM images for Trial 27



Examination of the SEM images of the powders produced in the close-coupling gas atomization system with a dual-slitt nozzle of our original design demonstrated powder morphology including bar, teardrop, and sphere types, as also mentioned in the literature. Inspection of the SEM images from Trial 9 showed a general prevalence of bar and teardrop shapes. Low temperature and larger inner flow tube diameter compared to optimal conditions is found to result in a lower ratio of sphericity. Particularly, cases with a large inner flow tube diameter had lower gas/metal flow rates, which is the major factor influencing powder size and morphology. A reduced flow rate increased the amount of liquid metal transferred to the gas expansion zone, preventing the energy transferred to the liquid metal bundle from carrying out its task of pulverization effectively. Bar-shaped grains formed at the first stage turned into teardrops at the second stage of fission due to surface tension. Finer grains which solidify more quickly during the freefall inside the tower displayed an inclination for satellization, namely clinging to coarser grains. Inspection of SEM images for Trial 18 revealed an increased ratio of spherical grains compared to other forms. This is a result of a smaller inner flow tube size and increased fusion temperature. Almost all of the grains produced under optimal conditions in Trial 27 (Figure 9) are found to have a spherical form. Reduced grain size and a high ratio of spherical grains were observed with a higher liquid metal temperature and a narrower flow tube. Satellization was seen more clearly, especially in higher image enhancements. The satellization rate is found to be lower under optimal conditions compared to other trial conditions. Smaller grain size may be considered the major cause of this since smaller grains cool down and solidify faster than larger ones. BET analysis results for powders obtained in Trial 18 and optimal conditions are shared below.

The surface area of the powder produced under optimal conditions was measured to be 0.843 m2/gr, while the surface area of the larger powder produced in Trial 18 was 0.380 m2/gr (Figures 10 and 11). These results suggest that the surface area of the powder increases as its grain size decreases.

Figure 10.
BET analysis results for the powder produced in Trial 18

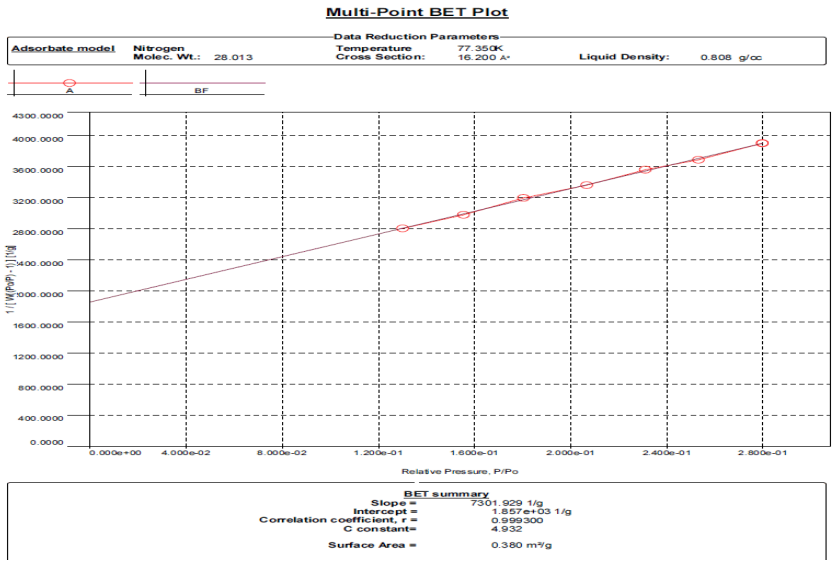
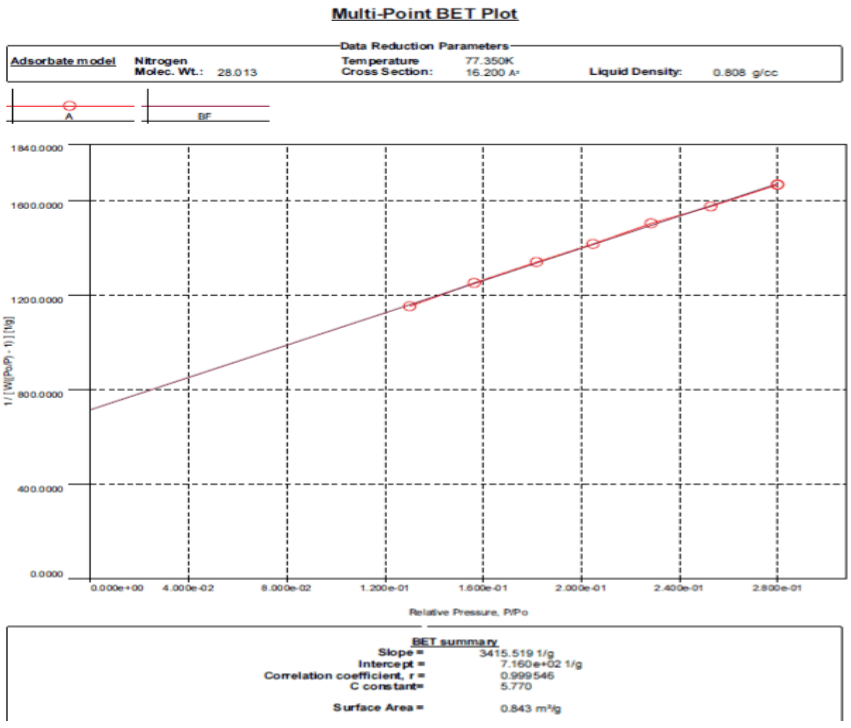


Figure 11

BET analysis results for the powder produced under optimal conditions (Trial 27)



Conclusion

The nozzle, which is a main parameter governing powder size and morphology in the gas atomization method, was constant in this study. Optimal conditions were achieved by altering gas pressure, fusion temperature, and flow tube diameter. They were identified as 35 bars for gas pressure, 3mm for inner flow tube diameter, and 830ºC for fusion temperature and taken as points of reference in all evaluations that followed. Grain size

tests demonstrated that the finest grains and the narrowest range of grain distribution are obtained under optimal conditions. Rates of sphericity were found to increase as grain sizes decreased. Additionally, a narrower grain distribution resulted in reduced satellization. Database scanning via diffractogram during the XRD phase study verified metallic aluminum as the main phase. XRD results are semi-quantitative. The reference peak point of metallic aluminum has a face-centered cubic lattice structure. SEM studies demonstrated a direct influence of inner flow tube diameter and gas pressure over grain size. A narrower flow tube was shown to cause a lower rate of liquid metal drifting to the atomization zone, increasing the cutting power of the atomization gas. In this context, higher gas pressure was associated with increased gas cutting power, letting the transferred kinetic energy pulverize the liquid metal and creating a spherical product with a narrower grain distribution. Another result of increased gas pressure is reduced satellization. Increased gas pressure yields smaller grains which cool down and solidify more quickly, reducing the satellization rate. Analysis results for powders produced via the X-ray diffraction method identified metallic aluminum as the main phase. Separate analyses of coarse and fine grains demonstrated narrower peaks for coarse structures and wider peaks for fine grains. Peak width is associated with crystal size. XRD analysis displayed higher severity of finer samples (Trial 27) compared to coarser samples (Trial 18). Smaller grain size was shown to be associated with larger surface areas. Trial 18 yielded an average grain size of 59.8µm and surface area of 0.380 m^2/gr, whereas the values from Trial 27 were an average grain size of 25.4µm and surface area of 0.483 m^2/gr.

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