Investigation of the Effect of Nozzle diameter on AZ31 Powder Production by Gas Atomization Method

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In this study, a series of experiments was conducted to investigate the effect of nozzle diameter on the size and shape of AZ31 magnesium alloy powder produced by gas atomization method. The experiments were conducted at a constant temperature of 97°C with a gas pressure of 53 bar and using four nozzle diameters of 3, 5, 5.5, and 3.5 mm. The melt was atomized using argon gas, while the shape and size of the produced AZ31 powder were determined using a scanning electron microscope (SEM). XRD and XRF were used to determine the phases and the weight fractions of the phases, respectively. Also, a guage was used to measure the size of the produced powders. The general aspect of the produced AZ31 powder was observed under a microscope. It was concluded that the best powder was obtained at a nozzle diameter of 3 mm.
Abstract

In this study, the effect of nozzle diameter is investigated experimentally on the shape and size of the AZ31 alloy powder produced via the gas atomization method. To do this, tests are carried out at a constant temperature of 790 °C, with a gas pressure of 35 bar and by applying nozzle diameter at four different values: 2, 3, 4, 5 mm. The atomization of the melt is done with argon gas, while scanning electron microscopy (SEM) is used to determine the shape of the AZ31 powder produced, XRD and XRF analyses are applied to determine the phases generated in the internal structures of the powders produced as well as the percentages of each phase. Also, a laser-assisted measurement device is used for powder size analysis. The general appearance of the AZ31 alloy powders produced is in the form of ligament, rod, droplet, flake and spherical, and most of the powders got into flake and spherical forms depending on the increase in gas pressure. It is determined that the finest powder is obtained at a temperature of 790 °C and a gas pressure of 35 bar at a diameter of 2 mm, and that the overall powder get in form of both droplet and spherical.

Key words: Gas atomization, AZ31 alloy powder, nozzle diameter.

1. Introduction

Magnesium is known as the lightest engineering metal having a density of 1.74 g/cm³, that is 35% less than aluminum and 75% less compared to primary metals used today [1,2]. In comparison, magnesium has a remarkable hardness and brings
many benefits such as better specific strength and increased absorption capacity [1,3]. The AZ31 series alloys are one of the most common magnesium alloys thanks to their lower cost, better resistance to corrosion as well as mechanical strength obtained through adding aluminum, zinc and manganese [4,5]. In this family of alloys, the AZ31 alloy has additional structural strength since it precipitates from the magnesium matrix and forms dual precipitates with aluminum and manganese [6,7]. Furthermore, as it adds to the strength of these materials with fine microstructures, producing them with low formability such as that of magnesium’s and their alloys with powder metallurgy today has turned into a necessity in the industry. For this reason, powder metallurgy has been proved to be an option in manufacturing methods like casting, hot and cold pressing, and machining. Whereas coarse-structured microstructures can be formed via casting, powder metallurgy is regarded as a useful method so as to come up with finer microstructures [8,9]. Making composites with the help of the powder metallurgy method, characteristics like increased surface wear resistance, surface friction and surface tensions can be achieved at high temperature [10,11]. Powder production technique using this method can be carried out in four different ways: mechanical, chemical, electrolysis, and atomization. Among the other methods, gas atomization is the most common for the purpose of obtaining fine and spherical powders. The primary reason for demand behind spherical powder material is that powder-powder contact in pressing and sintering stages has to be both homogeneous and multi-directional [12,13]. In this respect, atomization can be described as the degradation and solidification of molten metal into tiny droplets using either water, air and gas pressures or mechanical pressure. In return, the atomization process has 4 different forms: water atomization, gas atomization,
centrifugal atomization, and vacuum atomization. Producing over 60% of the metal and nonmetallic powders with gas atomization makes has made this specific method an advantageous one over the other forms. Gases including air, nitrogen, argon and helium can be applied as pressurized fluid so as to decompose the liquid metal bundle in gas atomization [14]. In other words, one can produce any form of metal and alloy powder which can be melted using the gas atomization method. In doing so, there are major factors at play, mainly the type of gas, its pressure, nozzle diameter and melting temperature. As the gas pressure is added, the temperature and viscosity of the molten material tend to drop, thereby allowing the formation of smaller powders [15].

2. Experimental Work

Experimental studies were carried out at the Gas Atomization Unit at Karabük University Faculty Manufacturing Engineering Department of Technology Faculty. The Gas Atomization Unit shown in Figure 1 consists of seven basic parts:

1. Melting furnace,
2. Atomization tower
3. Nozzle and nozzle holder
4. Powder collection unit
5. Gas system
6. Cyclones
7. Control panel
The melting furnace is designed and manufactured to operate continuously at approximately 1200 °C. During the melting, gas inlet and outlet units are placed on the side of the melting furnace in order to form a protective gas in order to prevent the formation of oxide as soon as and after atomization.

In order to control the flow of the melted metal in the ladle Figure 2b, the worm screw system and the graphite stopper were used. The melting furnace is shown in Figure 2a.
Magnesium alloy AZ31 has a melting temperature of 620 °C. The leakage temperature of the materials ranges between 150 and 200 °C of melting temperature. For this reason, a minimum of 790 °C, a maximum of 850 °C and an average temperature of 820 °C. The liquid metal was heated for one hour after reaching the required temperature, and the graphite plug with the helical screw system was manually turned up and the metal flow was ensured. During the metal melting process, argon gas was released at low pressure (about 2 bar) in the furnace to protect the dissolved metal against the onset of oxidation and combustion reaction.

The atomization tower of the Gas Atomization Unit is shown in Figure 3. The produced powders are collected in the powder collector located at the bottom of the atomization tower.
In order to atomize the liquid metal, a closely coupled supersonic nozzle holder with a circular hole is used in Figure 4a. Nozzle holder and nozzles are made of stainless steel.

The nozzle (Figures 4, a and b) is located on the nozzle holder in the furnace. Four different nozzles with diameters 2, 3, 4 and 5 mm were used. In closely matched systems, the heating of the nozzle is extremely important.

Figure 3. Atomization tower.

Figure 4. (a) Nozzle holder and Nozzle (b) Front view (c) Top view
If the nozzle is not heated sufficiently, the temperature of the melt metal at the nozzle end will decrease and the liquid metal flow will decrease. Uslan and Küçükarslan [20], in their study on the experimental investigation of gas atomized tin powder production parameters, the effect of back pressure at high pressures caused by molten metal. Therefore, the nozzle holder and nozzle used in this study were placed in the interior of the melting furnace to prevent the solidification of liquid metal in the nozzle. The test was carried out at nozzle diameter of 2, 3, 4 and 5 millimeters.

The produced powders are collected in the lower part of the atomization tower and in the lower part of the two cyclones connected in parallel to the powder collection unit Figure 5.

Figure 5. Powder collection unit.
The powder collector and cyclone are made of stainless steel. The powder collector is cylindrical, 400 mm high and 300 mm in diameter. Cyclone separator; it is made of a cylindrical diameter of 800 mm diameter and a diameter of 400 mm. After each experiment, the inside of the atomizing device and the cyclone were cleaned. The resulting powder was stored in desiccators to prevent oxidation. In the experiment, an argon pressure tube having an operating pressure of 200 bar was used as the atomizing gas. Three argon tubes are connected to the gas ramp to prevent gas pressure fluctuations and fluctuations when the gas pressure in the tube drops during the atomization process Figure 6. After the atomization process begins, the pressure reading in the gauge is considered to be the gas pressure. Figure 7 shows the pressure gauge used during the atomization process. Argon gas is used as the atomizing gas. The test was carried out at pressures of 35 bar.

Figure 6. Argon gas system.

Two cyclones connected in parallel with each other were used to evacuate the gas used in the atomization tower and to maintain fine
powder. The image of the cyclone is shown in Figure 8. In addition, a powder removal fan is used to remove fine powder. The power of the powder removal fan is 2,500 rpm. The powder we originally produced and what we call the roughest powders fall into the powder collector. Second, the average size of powder in the middle of the powder falls to the center. Finally, the finest powders fall through the powder fan to the last cyclone.

Figure 7. Manometer

Figure 8. Cyclones.
3. Atomization work

Gas-atomized AZ31 powder was produced using a closely matched circular perforated supersonic nozzle system during operation in a gas atomization device. During the atomization work, the liquid was superheated at AZ31 melting temperature (620 °C). The operations performed during the atomization work are listed below.

1. First, install the nozzle holder for the test at the bottom of the furnace.
2. Place the nozzle used in the experiment in the nozzle holder to provide flow between the pot and the nozzle holder.
3. Place the stainless steel pot on the nozzle in the oven.
4. The oven top cover is closed. A graphite plug having a stainless steel annular thread system that controls the flow of molten metal when the lid is closed is mounted in a manner to provide a seal for the center of the pot in the furnace.
5. During the metal melting process, argon gas is introduced into the furnace at a low pressure (about 2 bar) to protect the molten metal from the onset of oxidation and combustion reactions.
6. But about 50 g of AZ31 material in the ingot for each test, the AZ31 material was heated to 790, for one hour. The temperature of the melted material was measured by means of two thermocouples which were immersed into the stainless pot and were located outside the pot.
7. Argon gas was given at low pressures within the atomization tower 15 minutes before the atomization process to prevent the produced powders from reacting with air.
8. The atomization gas pressure was adjusted to the desired pressure with manometer and gas was sent to the nozzle.
9. It is ensured that the molten AZ31 alloy is flowing and atomized by argon gas by turning up the graphite plug with worm screw system. After the metal flow was completed, the gas flow was stopped and the atomization process was completed.

10. Powder collected in powder collector and cyclones are collected in desiccators. The powder collector, cyclones and atomization tower were cleaned for the next test. The installation of the unit was repeated and the above listed procedures were repeated for a new test. The basic principle of gas atomization and powder production is given in Figure 9.

![Gas Atomization Flow Chart](image)

Figure 9. Gas atomization flow chart.

The atomization parameters of the AZ91 powders produced by the gas atomization method are given in Table 1.
Table 1. Powder production parameters

<table>
<thead>
<tr>
<th>No</th>
<th>Melting metal temperature $^\circ$C</th>
<th>Gas pressure bar</th>
<th>Nozzle diameter mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td></td>
<td></td>
<td>2</td>
</tr>
<tr>
<td>S2</td>
<td>790</td>
<td>35</td>
<td>3</td>
</tr>
<tr>
<td>S3</td>
<td></td>
<td></td>
<td>4</td>
</tr>
<tr>
<td>S4</td>
<td></td>
<td></td>
<td>5</td>
</tr>
</tbody>
</table>

3. Results and Discussion

The experiments were carried out at a constant temperature of 790 $^\circ$C, 35bar and 4 different nozzle diameter (2,3,4,5 bar). Argon gas is used as the atomizing gas, and powder size analyses is performed with the Mastersizer 3000 model device. The working principle of the device is to throw red and blue laser lights on the sample. Then, the reflected and broken laser beams are examined using light detectors. The angle and intensity of the scattered light determines the particle size distribution of the sample. The dimensional values of the AZ31 alloy powders produced appear in Table 2 as $D_v$ (10), $D_v$ (50) and $D_v$ (90). In addition, the influence of the gas pressure on the powder size is clearly seen in Figure 10. Nozzle is known to have an important influence on the powder size and shape in powder production using the atomization method. As shown in Table 2, the smallest nozzle diameter value in this study is 2mm.
Table 2. Particle size of AZ31 powders

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Gas Pressure (bar)</th>
<th>Nozzle Diameter (mm)</th>
<th>Dv (10) μm</th>
<th>Dv (50) μm</th>
<th>Dv (90) μm</th>
<th>Specific Surface Area (m²/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>790</td>
<td>35</td>
<td></td>
<td>2</td>
<td>18.1</td>
<td>46</td>
<td>99.2</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>3</td>
<td>28.1</td>
<td>66.1</td>
<td>146</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>4</td>
<td>35.4</td>
<td>92.2</td>
<td>235</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>5</td>
<td>41.3</td>
<td>119</td>
<td>296</td>
</tr>
</tbody>
</table>

When Table 2 and Figure 10 are examined, it can be seen that the powders produced are in the range of 18.1 to 203 μm, and that the smallest average powder size is obtained at 2mm diameter of nozzle which is 46 μm. A 10% portion of the powder produced under 2mm nozzle diameter is below 18.1 μm, while the remaining 90% consists of powder below 99.2 μm. At least 10% of the powders produced were found to be composed of powders sizes smaller than 10 μm, but these powders sizes were not measurable because they were plunged into the atomization towers and cyclones, or even to the containers in which the powders were stored. When the Specific Surface Area known as the area per kg in the literature is examined, it is seen that the area expands as the nozzle diameter decrease. It is known that the powders produced increase the press ability and sinter ability properties. As a result of experiments in the production of the AZ31 powder by gas atomization method, the effect of nozzle diameter is clearly seen.
The SEM images given in Figure 10 reveal that the grain size of the powders produced in the vessel is increase.

![Graph showing dimensions of AZ31 powders produced at different nozzle diameters.](image)

Figure 10. Dimensions of AZ31 powders produced at different nozzle diameter.

When examining Figure 10, it can be seen that due to the increase in nozzle diameter, the average powder size increases. The average powder size (Dv50) of the powder produced at 5mm diameter was 80.2μm, and the average powder size (Dv50) was reduced to 46 μm when the nozzle diameter was decreased to 2mm. In this study,
an SEM image (100X) of powders produced from different nozzle diameter is given in Figure 11.

Figure 11. SEM images of powders (100X) at different nozzle diameter (a) 2mm (b) 3mm (c) 4mm (d) 5mm.

As can be seen in Figure 11, the powder size increase with the increase of nozzle diameter. The reason for the increase in powder size due to the nozzle diameter can be explained as an increase in the average powder size value in powder production because of the lower energy transfer to the molten metal at higher nozzle diameter. It is understood from Figure 12 that the powders prepared are generally spherical, droplet, ligament, complex and flank
Figure 12. SEM images of the powders produced (1000X) at different nozzle diameter (a) 2mm (b) 3mm (c) 4mm (d) 5mm

As can be seen from the SEM images shown in fig. 12, the powder shaped spheres and droplets become ligaments and flanks, due to the increased nozzle diameter. In particular, the powder produced with 2mm of diameter and giving the SEM image in Figure 12a and b is significantly reduced, droplet and spherical. Examining Figures 12a and b, it can be seen that the powder has spherical and droplet shapes.

Table 3. Chemical (XRF) analysis results of the produced AZ31 alloy powder

<table>
<thead>
<tr>
<th>Elements</th>
<th>Mg</th>
<th>Al</th>
<th>Zn</th>
<th>Mn</th>
<th>Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>Content (%)</td>
<td>94,71</td>
<td>2,75</td>
<td>1,62</td>
<td>0,61</td>
<td>0,22</td>
</tr>
</tbody>
</table>
In the XRD pattern of the AZ31 alloy, Mg which is the $\alpha$ phase and the $\beta$ phase were determined Figure 13. The AZ31 alloy, whose chemical analysis is presented in Table 3, contained 94.71 wt.% Mg, 2.75 wt.% Al, 1.62 wt.% Zn, 0.61 wt.% Mn, and 0.22 wt.% Si. It is seen in the XRD results given in Figure 13, that Mg and Al combine to form Al$_{12}$Mg$_{17}$ precipitate during the solidification.

3. Conclusion

The following results were obtained in this study conducted on the characterization of AZ31 powder produced using the gas atomization method with different parameters:

1. Powders in different shapes and sizes were obtained with this method. The smallest powder size occurs at 790$^\circ$C, with a 2mm nozzle diameter and at 35 bar pressure.
2. The powder size decreased as nozzle diameter decreased.
3. With decreasing nozzle diameter, the powder shape changed from ligament, flake, and complex to droplet and spherical.

4. According to the XRD and XRF results, the produced AZ31 alloy powder was composed of α and β (Al_{12} Mg_{17}) phases in the structure.

References


[15]. Oğuz, Ş; Öztürk, Z; Uzun, E; Kurt, A; Boz, M. Gaz atomizasyonu yöntemi ile kalay tozu üretiminde gaz basıncının toz boyutu ve şekline etkisi. 6th International Advanced Technologies Symposium (IATS’11), 2011, 565-568.