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Study Effect of Temperature on AZ31 Power Production by Gas Atomization Method

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الملخص
في هذه الدراسة، تم دراسة تأثير دراجات الحرارة بشكل تجريبي على شكل وحجم مسحوق سبيكة ماغنيسيوم AZ31 المنتجة بواسطة طريقة الانحلال الغازية. للقيام بذلك، يتم إجراء الاختبارات مع ضغط غاز 35 بارًا وبتطبيق قطر الفوهة 2 ملم. يتم تفتيت المصهور باستخدام غاز الأرغون ، في حين يتم استخدام المسح المجهري الإلكتروني (SEM) لتحديد شكل مسحوق AZ31 المنتج، يتم تطبيق تحليلات XRD و XRF لتحديد المراحل المتولدة في الهياكل الداخلية للمساحيق المنتجة وكذلك النسب المئوية لكل مرحلة. أيضا ، يتم استخدام جهاز قياس بمساعدة الليزر لتحليل حجم المسحوق. المظهر العام لمساحيق السبيكة AZ31 المنتجة على شكل رباط، قضيب، قطرة، تقشر وكروية، ومعظم المساحيق حصلت على تقشر وأشكال كروية اعتمادا على التغير في درجة الحرارة. يتم تحديد أفضل مسحوق يتم الحصول عليه عند درجة حرارة 790 درجة مئوية عند ضغط غاز 35 بار بقطر 2 مم، وأن المسحوق يظهر على شكل قطرات واشكال كروية.

Abstract

This study experimentally investigates the effect of temperature on the size and shape of the AZ31 alloy powder made by the gas atomization method. A constant nozzle diameter of 2mm was used during the tests at a gas pressure of 35 bar and three different temperatures of 790, 820, and 850°C. Argon gas was used for the atomization of the melt while the shape of the powder produced was determined by scanning electron microscopy (SEM). In addition, XRD and XRF analyses were adopted to determine the phases of the powders' internal structure as well as the percentages of each phase. Furthermore, a laser-assisted measurement device was utilized for powder size analysis. The results revealed that most of the AZ31 alloy powders got into flake and spherical forms and few in the form of ligaments, rods or droplets depending on the temperature. Moreover, the finest powder was obtained at a temperature of 790 °C with powder shape of both droplet and spherical.

Keywords: *Gas atomization, AZ31 alloy powder, temperature.*

1. Introduction

Magnesium is commonly referred to as the lightest engineering metal with a density of 1.74 g/cm^3, which is 35% less than aluminium and 75% less than most primary metals used today [1,2]. In comparison, magnesium has a better specific strength and increased absorption capacity with a remarkable hardness [1,3]. The most common magnesium alloys used recently are the AZ31 series alloys because of their lower cost, better resistance to corrosion as well as mechanical strength resulting from adding aluminium, zinc and manganese [4,5]. The additional structural strength of the AZ31 alloy is attributed to that it precipitates from the magnesium matrix and forms dual precipitates with aluminium and manganese [6,7]. Furthermore, besides the strength and the fine microstructures of these materials, the low formability of magnesium and its alloys has made it attractive for the industry. Powder metallurgy has been proven to be an option in manufacturing fine microstructures as compared to other manufacturing methods such as casting, hot and cold pressing, and machining [8,9]. Additionally, making composites with the powder metallurgy method leads to achieving desirable characteristics such as increased surface wear resistance, surface friction and surface tensions, especially at high temperatures [10,11]. There are four different ways for powder production which include: mechanical, chemical, electrolysis, and atomization. Among these methods, gas atomization is the most common for obtaining fine and spherical powders. The powder-powder contact in the pressing and sintering stages must be homogeneous and multi-directional which explains the reason why spherical powder material is desired [12,13]. Atomization is the degradation and solidification of molten metal into tiny droplets using either mechanical or hydromechanical pressures using water, air, or gas. In response, atomization process can be divided into four different forms: water atomization, gas atomization, centrifugal atomization, and vacuum atomization. Gas atomization has an advantage over the other forms regarding producing over 60% of the common metallic and nonmetallic powders. Air, nitrogen, argon and helium can be used as working fluids in the gas atomization method [14]. In addition, there are major factors at play in the gas atomization method, mainly the type of gas, its pressure, nozzle diameter and melting temperature. However, the gas pressure is playing a major role that is when the pressure increases the temperature and viscosity of the molten material tend to drop, allowing the formation of smaller powders [15].

2. Experimental Work

The experimental work was carried out using a locally produced Gas Atomization Unit at Karabuk University. The Gas Atomization Unit is shown in Figure 1 and can be divided into the following seven basic parts:

(1) Melting furnace, (2) Atomization tower, (3) Nozzle and nozzle holder, (4) Powder collection unit, (5) Gas system, (6) Cyclones, and (7) Control panel.

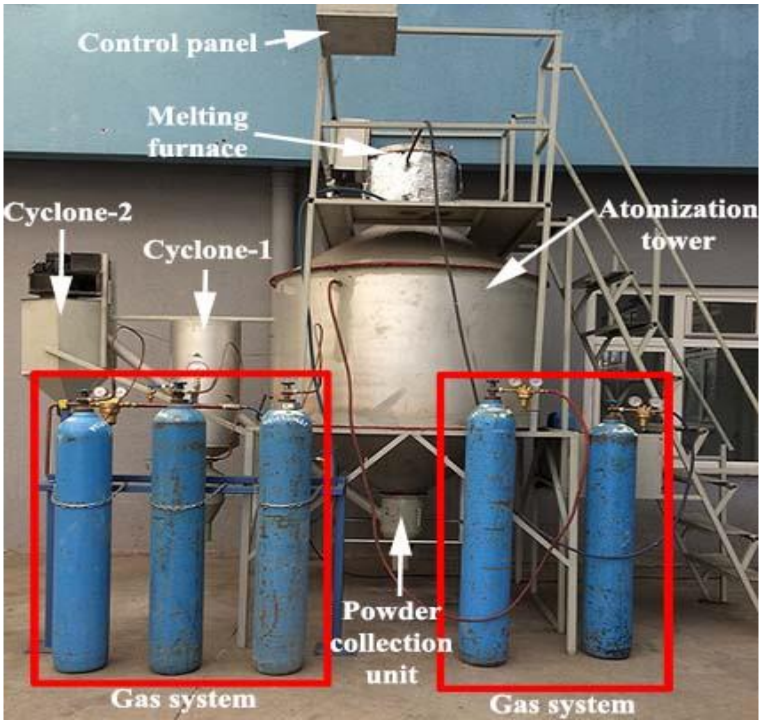

Figure 1. Gas atomization unit

The melting furnace is designed and manufactured to work constantly at around 1200 °C. The gas inlet and outlet units are located on the melting furnace sides in order to prevent the formation of oxide after the atomization of the melted metal. To maintain the flow of the melted metal under control inside the ladle, a system of worm screw and graphite stoppers were utilized as shown in Figure 2b.

Figure 2. (a) Image of the interior of the melting furnace (b) Melting pot

Magnesium alloy AZ31 has a melting temperature of 620 °C. So, the temperature of the melted alloy was maintained in the range between 790 °C and 850 °C (average of 820 °C). The liquid metal was manually stirred, using the helical screw system, for one hour after reaching the required temperature in order to ensure constant flow of the liquid metal. During the whole process, the oxidation of the dissolved metal and the combustion reactions were prevented by the means of Argon gas released at low pressure of 2 bar in the furnace.

Figure 3 shows the Gas Atomization Unit where the produced powders are collected in the powder collector located at the bottom of the atomization tower.

Figure 3. Atomization tower.

With the purpose of atomizing the liquid metal, a supersonic nozzle and holder with a circular hole are used as shown in Figure 4. The nozzles and the holder were made of stainless steel.

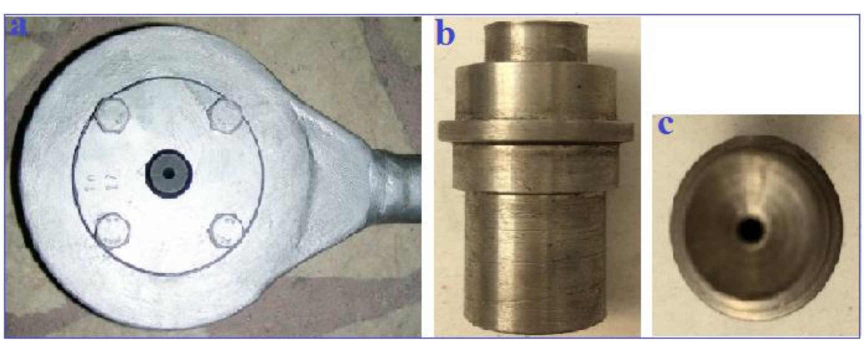

Figure 4. (a) Nozzle holder and Nozzle (b) Front view (c) Top view

A nozzle with a diameter of 2 mm was used. In addition to that, the nozzle was heated to maintain the temperature of the melted metal at the nozzle tip at a sufficient temperature which is important for the flow of the liquid metal. It is recommended by Uslan and Küçükarslan [20] to place the nozzle holder and the nozzle in the interior of the melting furnace to prevent the solidification of liquid metal in the nozzle. The experiment was performed at different temperatures of 790, 820, and 850C^{o}.
The powder produced is collected using a powder collection unit located in the lower part of the atomization tower as shown in Figure 5. The powder collector has dimensions of 400 mm in height and a diameter of 300 mm. While the two Cyclone separators have diameters of 800 mm and 400 mm.

Figure 5. Powder collection unit.

The atomizing device and the cyclone have to be cleaned after each experiment. Also, desiccators were used to store the resulting powder in order to prevent oxidation. Argon gas at a pressure of 200 bar was used as atomizing gas. In addition, and as shown in Figure 6, the gas was connected to the atomizing unit via three tubes to prevent gas pressure fluctuations. Figure 7 shows the pressure gauge used during the atomization process.

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Figure 6. Argon gas system.

Figure 7. Manometer

During the atomization process, two cyclones connected in parallel were used to evacuate the gas. One of the two cyclones is shown in Figure 8. Moreover, the fine powder was removed by means of a dedicated, 2,500 rpm, fan. In contrast, the roughest and heavier powder falls into the powder collector. On the other hand, the average-size powder falls into the center of the collector.

Figure 8. Cyclones.

3. Atomization work

A circular perforated supersonic nozzle system was used to produce gas-atomized AZ31 powder. The operation temperature during the atomization work was equal to the melting temperature of the superheated AZ31 (620 C°). The sequence performed during the atomization work is as below:

1. First, the nozzle holder is installed at the bottom of the furnace.
2. Fit the nozzle to the nozzle holder in such a way that it provides flow between the pot and the nozzle holder.
3. put the stainless steel pot on the nozzle inside the oven.
4. close the oven top cover. The cover is a graphite plug with stainless steel annular thread that controls the flow of molten metal.
5. A 2-bar flow of Argon gas is introduced into the furnace to prevent oxidation and combustion reactions.
6. A 50 g of AZ31 material was placed in the pot for each test. Then, the pot was heated to different temperatures for one hour and the temperature of the melted material was monitored through two thermocouples immersed into the pot's wall.
7. The atomization gas was sent to the nozzle at a desired pressure.
8. The flow of the molten AZ31 alloy and the atomization process is checked by opening up the graphite plug using the worm screw system. Then, the atomization gas is stopped just after the molten metal flow is completed.
9. The produced powder gathered from the powder collector and the cyclones are saved in desiccators. The whole unit is cleaned for the next test. Figure 9 illustrates the basic principle of gas atomization and powder production.

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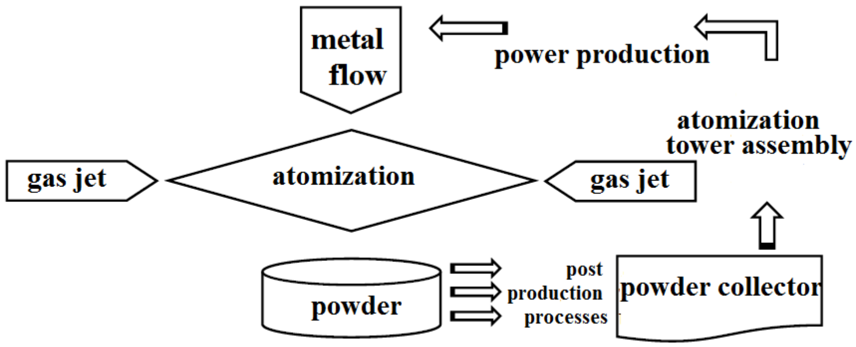

Figure 9. Gas atomization flow chart.

Table 1 lists the atomization parameters of the AZ31 powders produced.

Table 1. Powder production parameters

No	Melting metal temperature C°	Gas pressure bar	Nozzle diameter mm
S1	790		
S2	820	35	2
S3	850		

3. Results and Discussion

The results presented in the following sections were conducted at a constant gas pressure of 35 bar, nozzle diameter of 2mm, and at three different temperatures of 790, 820, and 850 °C. The size of the produced powders was measured and analyzed with a Mastersizer 3000 model device. The device irradiates red and blue laser lights on the sample and then examines the reflected and broken laser beams to determine the particle size and distribution of the sample. Table 2 presents the dimensional values of the produced powders in three groups Dv (10), Dv (50) and Dv (90). On the other hand, Figure 10 shows the influence of temperature on the powder size. It can be seen that temperature has a significant impact on the powder size and shape in the atomization method.

Table 2. Particle size of AZ31 powders

Gas pressure (Bar)	Nozzle Diameter (mm)	Temperature (C°)	Dv (10) µm	Dv (50) µm	Dv (90) µm	Specific Surface Area(m²/kg)
35	2	790	18.1	46	99.2	186.7
		820	31.2	79.1	201	101.6
		850	21.6	84.6	252	143.7

The results concluded from Table 2 and Figure 10 suggested that the powder size is in the range of 18.1 to 252 µm and the smallest size of 46 µm was obtained at 790 **°C**. Also, at this temperature**,** 90% of powders are below 99.2 µm while 10% are below 18.1 µm. In contrast, there are sizes, smaller than 10 µm, that cannot be measured because they were plunged into the cyclones, atomization towers, and even the storage containers. Moreover, it is clear that the Specific Surface Area per kg expands as the temperature increases. Figure 11 reveals this fact where the grain size of the powders is increased with the increase of the temperature. For example, the average powder size produced at 850 °C was 84.6 µm, and it is reduced to 46 µm when the temperature decreased to 790°C.

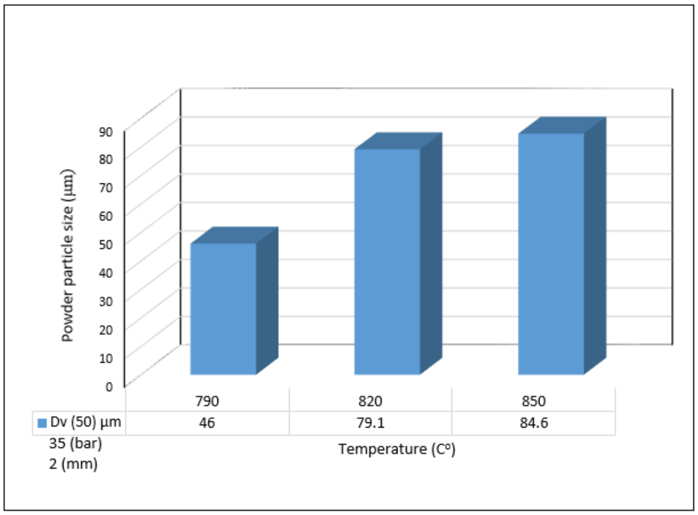

Figure 10. Dimensions of AZ31 powders produced at different nozzle diameters and temperatures.

Figure 11 presents an SEM image (100X) of the produced powders for different temperatures. Figure 11 reveals that the powder size increases as the temperature increases. The reason for that is that lower energy is transferred to the molten metal at higher temperatures.

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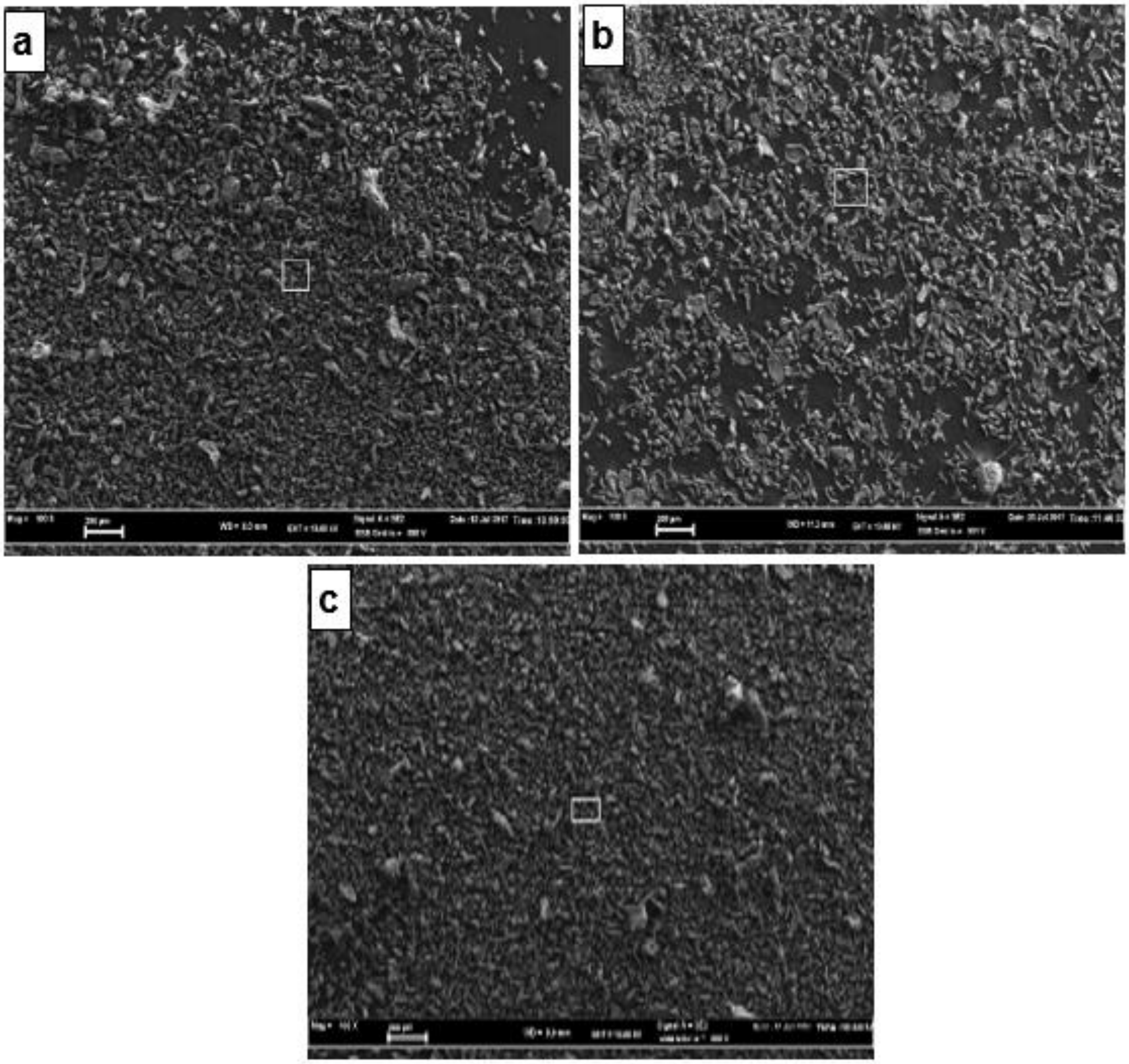

Figure 11. SEM images of powders (100X) at different temperatures (a) 790 C°, (b) 820 C°, and (c) 850 C°.

Regarding the shape of the powder particles, Figure 12 shows that the powders are in general spherical, droplet, ligament, complex and flank.

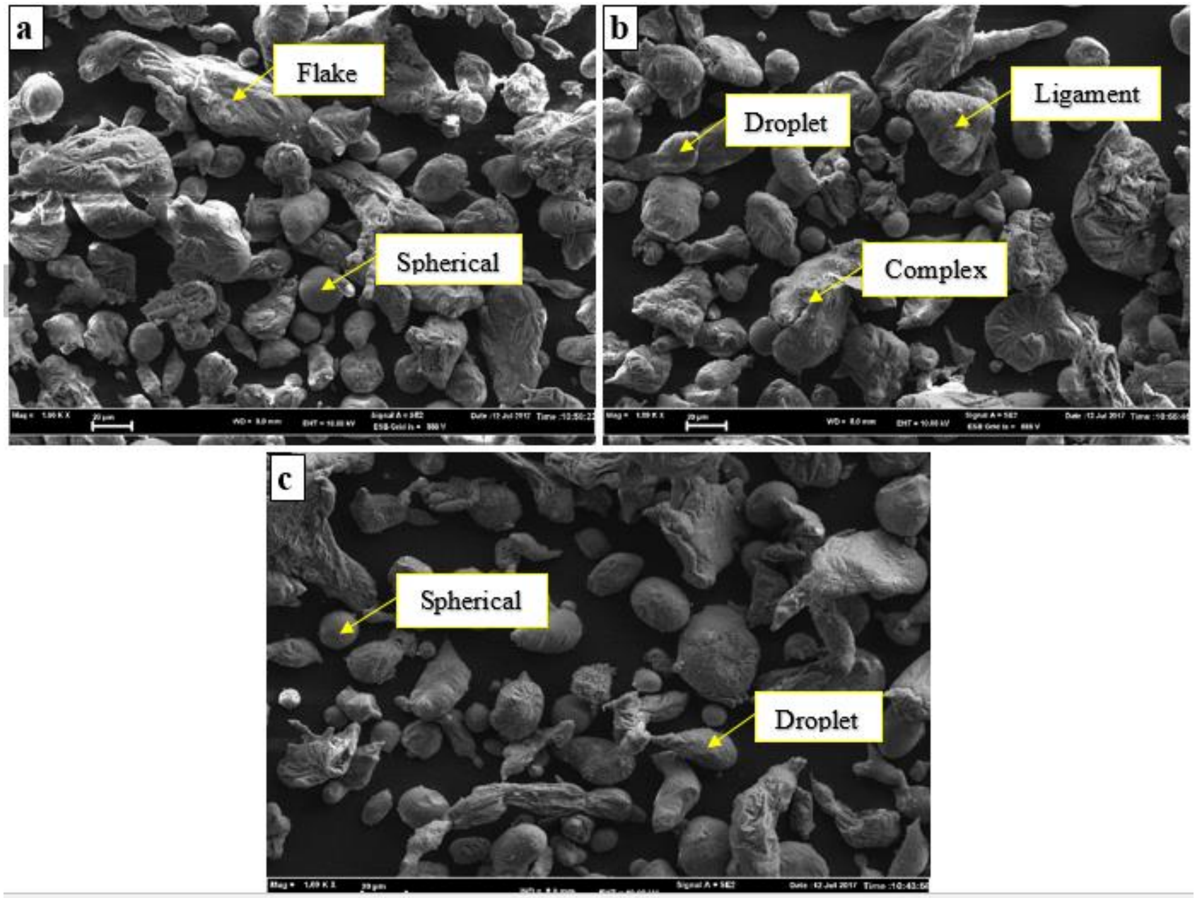

Figure 12. SEM images of the powders produced (1000X) at different temperatures (a) 790 C° (b) 820 C° (c) 850 C°

Figure 13 shows the XRD pattern of the AZ31 alloy which provides information about the chemical structure of the AZ31 alloy. Table 3 presents the chemical compositions of the alloy, specifically, there are 94.71 % Mg, 2.75 % Al, 1.62 % Zn, 0.61 % Mn, and 0.22 % Si.

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

Elements	Mg	Al	Zn	Mn	Si
Content (%)	94.71	2.75	1.62	0.61	0.22

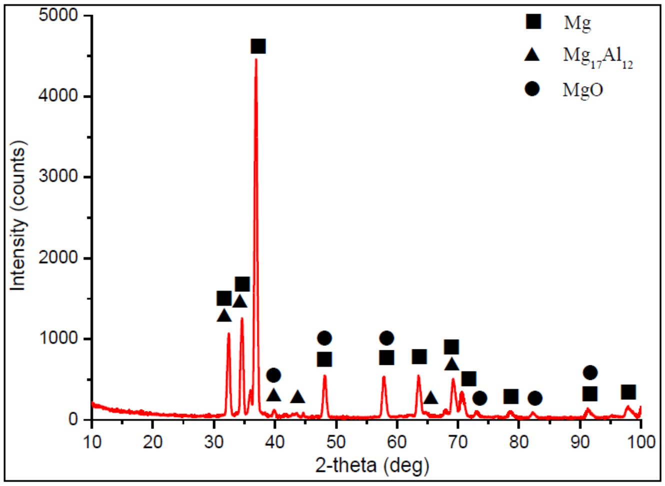

Figure 13. XRD pattern of AZ31

4. Conclusion

An experimental investigation has been conducted on the effect of temperature on the size and shape of the AZ31 alloy powder made by the gas atomization method. The following results were obtained:

1. Different shapes and sizes of AZ31 alloy powder can be made by this method. The smallest powder size was obtained at 790°C, with a nozzle diameter of 2mm and at 35 bar pressure.
2. As the temperature increased, the powder size decreased.
3. The powder shape changes with the temperature that is shapes such as ligament, flake, and complex occur at high temperatures while shapes such as droplet and spherical occur at lower temperatures.

4. The XRD and XRF results suggested that the structure of the produced AZ31 alloy powder was mainly composed of α and β (Al_{12} - Mg_{17}) phases.

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