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The Effect of Sintering and Compaction Conditions on the Microstructure and Properties of AZ31 Magnesium Alloy

Amin Saghafi[1, *], Seyed Ehsan Eftekhari Shahri[2], Hossein Jamshidi[3], Mohammad Kazem Salari[4]
Department of Mechanical Engineering,
Birjand University of Technology, Birjand, Iran
E-mail: a.saghafi@ birjandut.ac.ir, e.eftekhari@birjandut.ac.ir, ai.hjamshidi@gmail.com, mk.salari@gmail.com
*Corresponding author

Razieh Khoshhal[5]
Department of Materials and Metallurgical Engineering,
Birjand University of Technology, Birjand, Iran
E-mail: rkhoshhal@birjandut.ac.ir

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Abstract: Magnesium and its alloys are attractive materials in industrial applications due to the low density and high strength. The properties of AZ31 magnesium alloy can be much improved by choosing proper sintering conditions. In this study, the microstructure and mechanical properties of AZ31 prepared by mechanical alloying, compaction, and sintering of elemental powder, were studied. The effect of parameters such as compaction pressure, heating rate, and sintering time were investigated to determine the optimal sintering condition of AZ31 magnesium alloys. Previous researches have focused on the specific conditions of sintering, while in this study, various factors of sintering were examined simultaneously. The results showed that sintering time is one of the major variables that have a considerable effect on the final properties of AZ31. In short sintering times, recrystallization leads to small grain formation inside the powder. However, as the sintering time increases, the growth of new grains slows down and no trace of them can be detected in the microstructure. Furthermore, the conditions for recrystallization were also determined, which can be used to provide small grain size and, consequently, better properties after the initial powder milling and sintering. At optimal sintering conditions, the average grain size, porosity percentage and hardness of the samples AZ31 magnesium alloy was obtained as 104 µm and 2.05%, and 79.5 HV, respectively which is expectable result in comparison to the bulk AZ31.

Keywords: AZ31 Magnesium Alloy, Microstructure, Powder Metallurgy, Sintering

Biographical notes: Amin Saghafi is Associate Professor at the Department of Mechanical Engineering, Birjand University of Technology, Birjand, Iran. He received his PhD in Mechanical Engineering from Ferdowsi University of Mashhad, Mashhad, Iran in 2015. **Seyed Ehsan Eftekhari Shahri** received his PhD in Mechanical Engineering/Manufacturing branch from University of Birjand, Birjand, Iran in 2013. He is Assistant Professor at the Department of Mechanical Engineering, Birjand University of Technology, Birjand, Iran. **Hossein Jamshidi** is MSc student of Mechanical engineering/ Manufacturing branch at the Birjand University of Technology, Birjand, Iran. He received his BSc. in Mechanical engineering from Islamic Azad University, Eqlid Branch, Eqlid, Iran in 2014. **Mohammad Kazem Salari** is MSc student of Mechanical engineering/Manufacturing branch at the Birjand University of Technology, Birjand, Iran. He received his BSc. in Mechanical engineering from Islamic Azad University/ Mashhad Branch, Mashhad, Iran in 2006. **Razieh Khoshhal** received her PhD in Metallurgy and Materials Engineering from Iran University of Science and Technology, Tehran, Iran in 2014. She is Assistant Professor at the Department of Materials and Metallurgical Engineering, Birjand University of Technology, Birjand, Iran.

Research paper

1 INTRODUCTION

Magnesium and its alloys are widely used in various applications such as automotive, electronics, aerospace, and medicine because of its lightweight, low density, high strength and biocompatibility properties [1-2]. However, these alloys have some limitations such as low elastic modulus, and poor corrosion resistance [3-5].

The improvement of the mechanical properties of AZ31 magnesium alloys is important. Previous researchers have pursued this goal under various conditions. Reinforcing particles in metal-based composites and powder metallurgy (PM) techniques are commonly used to increase mechanical properties [6-10]. Powder metallurgy refers to the method in which a solid formed product can be achieved through 3 basic steps powder mixing, compression, and sintering. Magnesium and its alloys are often produced by casting. Applying the powder metallurgy on Magnesium requires special preparations. A high tendency to react with oxygen leads to surface oxidation and makes the sintering process difficult. Argon and nitrogen are usually used to solve this problem [11-12]. The microstructure and mechanical properties of alloys formed by powder metallurgy can be affected by the porosity and the contact surface of the particles [13-14]. It also can be improved by choosing the proper sintering conditions. The researchers showed that the properties of materials after sintering depend on various factors such as temperature, time, particle size, and compacting pressure [15-16]. Ram Kim et al. [17] investigated the microstructural evolution and mechanical properties of 6% Al Mg alloy in the spark plasma sintering (SPS) process by degassing before sintering. Due to the type of grafting at the grain boundaries and the improvement of mechanical properties, optimal temperature and compaction pressure were reported as 530°C and 130 MPa, respectively. Mondet et al. [18] performed spark plasma sintering on AZ91 magnesium alloy to improve the compressive strength of the AZ91. The influence of the sintering temperature was investigated. The optimum microstructure was obtained for a sintering temperature of 380ºC. Burke et al. [19] investigated the effect of pressing pressure, and temperature of sintering on AZ31 magnesium alloy produced by traditional sintering. They found that the sintering temperature and time have the greatest effect on the properties of the sample. The maximum tensile strength was observed at 20 min and 500°C. The SPS process to prepare compact samples from gas-atomized and attritor-milled AE42 magnesium powder was studied by Minarik et al. [20]. After the short attritor-milling, the powder was sintered in the range of 400-550°C for 3 minutes. It was found that a short milling time applied on magnesium alloy powder before SPS can be effectively used to improve the mechanical properties. In magnesium-based metal composites, the addition of reinforcing particles improves tensile strength. Similarly, Jayakumar et al. [21] prepared AZ31 alloy composite reinforced with multiwall carbon nanotubes (MWCNTs) by mechanical alloying and powder metallurgy technique. The results showed that an addition of 1 wt% carbon nanotubes leads to an improvement in 0.2% yield strength without any losing ductility. Galindes et al. [22] examined three alloys of magnesium AZ31, AZ61, AZ91 produced by high energy milling and hot sintering. It was found that as milling speed and time increased, the particles were fractured to the size of 10 μm, and the improvement of hardness was also achieved.

The main objective of this research was to improve the properties of AZ31 magnesium alloy by choosing proper sintering conditions. Based on the above literature, the previous researches have focused on specific conditions of sintering, while in this study, various factors of sintering were examined simultaneously. The effect of sintering parameters on the microstructure and mechanical properties of the AZ31 magnesium alloys were evaluated in terms of grain size, porosity, and hardness. Then, the optimal sintering conditions (sintering time, heating rate, and compaction pressure) were determined to achieve improved mechanical properties.

2 EXPERIMENTAL PROCEDURE

Powder metallurgy and mechanical alloying have been used to fabrication of AZ31 magnesium alloy. Elemental powders of Mg, Al, Zn and Mn were selected based on AZ31 stoichiometry ("Table 1").

Table 1 Specifications of powders used for powder metallurgy of AZ31 magnesium alloy

Materials	Mg	Al	Zn	Mn
Purity (%)	>99.9	>99.8	>99.8	>99.8
Particle size (μm)	100	40	10	10
Wt%	Base	3	1	0.35

The mechanical alloying was conducted on mixed powders using a high energy ball mill with RPM of 300, a ball ratio of 20:1 for 3h (in which a break time of 8 min was performed every 20 minutes). Stearic acid (3 wt%) was added to prevent excessive cold welding. Also, argon was utilized to prevent oxidation. The milled powders were compacted under the pressures of

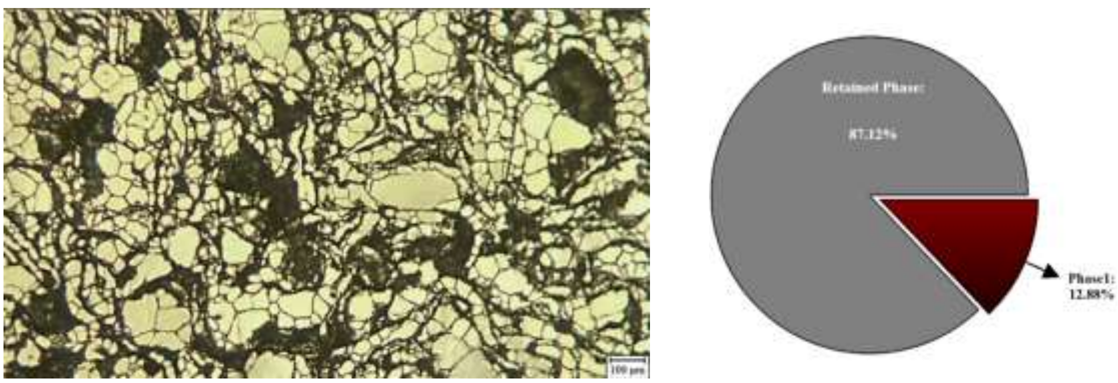
500, 600, 700 MPa for 3 min at room temperature to obtain samples with dimensions of Ø14×21 mm. Subsequently, the samples were sintered at 520°C in a tube furnace. XRD (X-ray diffraction) analysis was used to determine the phase composition and crystallite size in the range of 5-90° by Cu Kα radiation.

In order to examine the effect of the sintering factors, the specimens were prepared for microstructure and hardness tests. Microstructure investigations were carried out using an optical microscope and scanning electron microscope (TESCAN MIRA-3 FE-SEM). The grain size and the porosity percentage of the phases were evaluated using MIP (microstructural image processing) software. The results were obtained based on 3 repetitions for each experiment to minimize the errors. Finally, the hardness of each sample was determined by using a Vickers hardness tester with a load of 1 kg and a holding time of 5 seconds. It was measured at 5 places for each sample and the average value was reported.

3 RESULTS AND DISCUSSION

3.1. Determination of the Optimal Compaction Pressure

A series of recent studies have indicated that compacting pressure plays an important role in the properties of sintered products [23]. This part of the study, therefore, set out to assess the effect of compacting pressure (500, 600, and 700 MPa) on the microstructure, porosity, and hardness of sintered AZ31. Images obtained by optical microscope and porosity percentage are shown in “Fig. 1”. The grain size and Vickers hardness results of AZ31 alloy samples are shown in “Fig. 2”. As shown in these figures, the sampled pressed under 600 MPa has the least porosity while it delivered the most hardness. When the compacting pressure was increased from 500 MPa to 600 MPa, the porosity percentage of the samples decreased significantly from 12.88% to 5.6%. Also, the hardness increased from 44.7 HV to 81.2 HV, and the grain size decreased from 74 µm to 71 µm. The reason could be the weak contact between the raw materials in low pressure (500 MPa), and more work hardening created at the grain boundaries which spread into the grains and causes more porosity as a result of the high pressure in hardened regions (700 MPa). Accordingly, the pressure of 600 MPa was selected as the proper compaction pressure for the subsequent experiments based on the high enough hardness and minimum porosity and grain size.



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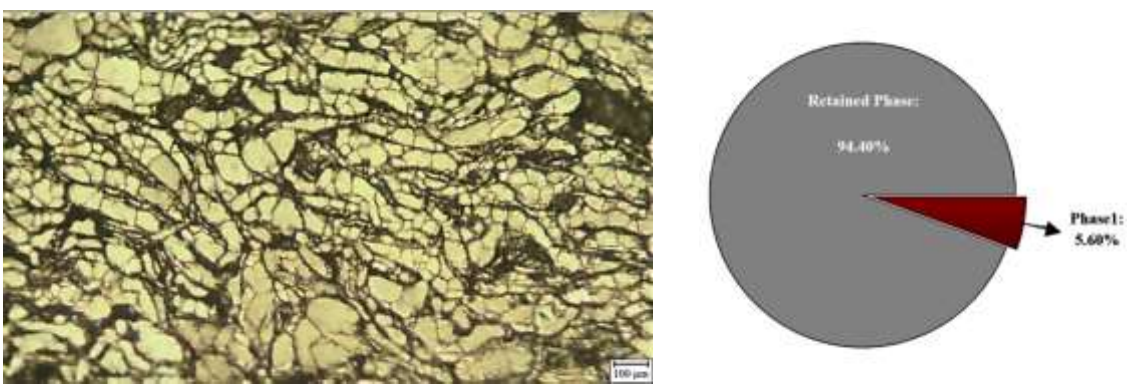

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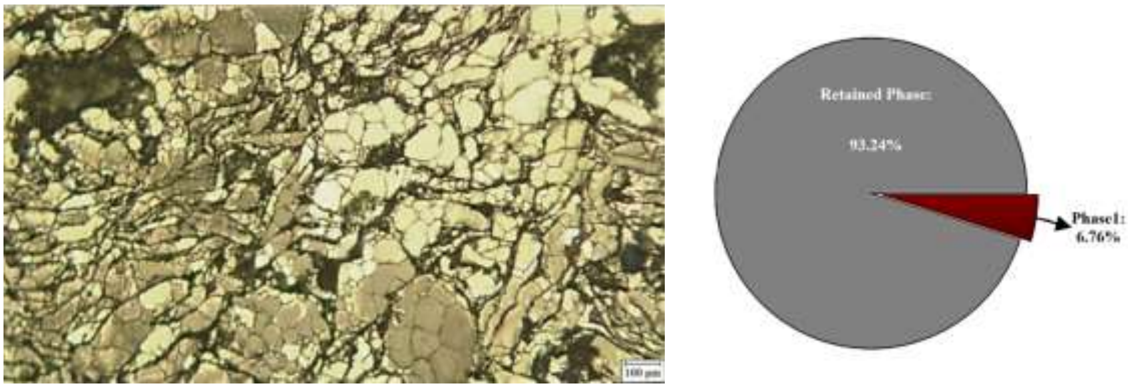

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Fig. 1 The optical microscope images and the porosity percentage of samples pressed under: (a): 500, (b): 600, and (c): 700MPa and sintered at 520°C for 3h.

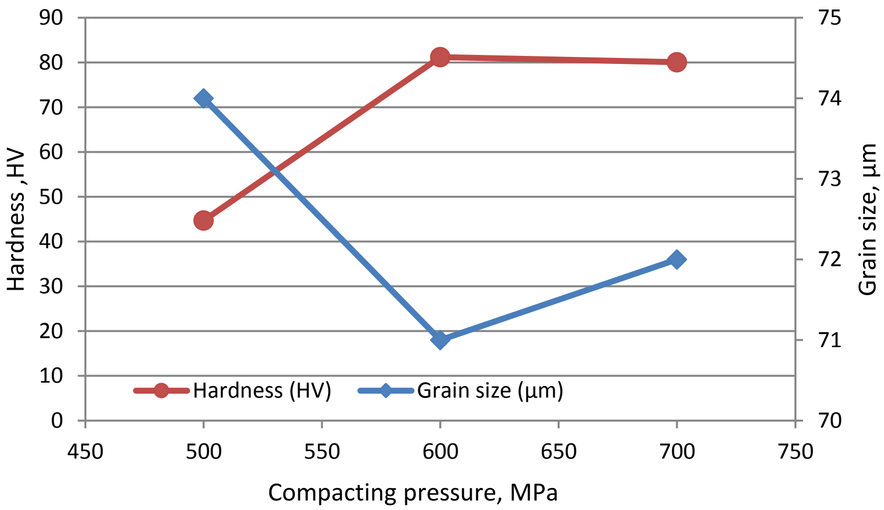
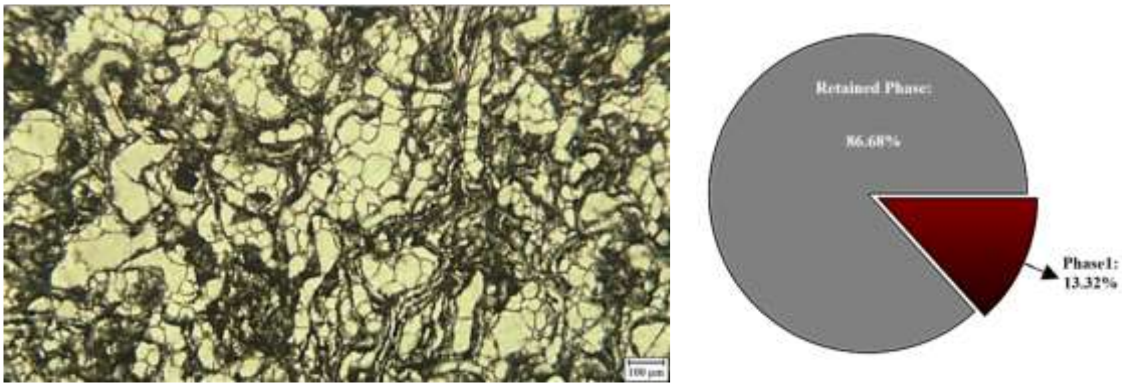


Fig. 2 The hardness and grain size of samples pressed under 500, 600, and 700MPa and sintered at 520°C for 3h.

3.2. The Effect of Sintering Cycles

It has previously been observed that 520°C is the best temperature for sintering of AZ31 [24]. The heating rate and sintering time were considered as the factors that could affect the microstructure and mechanical properties of AZ31. Figure 3 shows the microstructure of samples pressed under 600MPa and sintered with different heating rates. Figure 4 shows the grain size and hardness values of each sample. As illustrated in "Fig. 3(a) and Fig. 4", by interring the pressed sample directly at 520°C without applying a heating rate, the obtained product has low hardness and high porosity, which can be attributed to the thermal shock. To reduce the negative effects of heat shock, the samples were preheated at a specified rate.

As can be seen in "Fig. 3", in short sintering times, small grains are formed inside the powder particles. A possible explanation for this might be the recrystallization process. Since the samples have been milled, a lot of strain is stored in powders, which are released at the temperature of sintering in the form of new strain-free grains. However, as the sintering time increases, these new grains grow slowly and no trace of them can be detected in the microstructure ("Fig. 5"). Another reason that can confirm the recrystallization process in the early times of sintering is the shape of the particles which is stretched and shows the stored strain ("Fig. 3"), while with increasing the sintering time, there is no trace of these distorted particles. Comparing "Fig. 5 b and c", it can be seen that sintering time does not have any effect on the microstructure of the sample while it can reduce the hardness by releasing more strain or the grain growth ("Fig. 6"). The optimum microstructure was obtained for sintering time 16h, for which the average grain size, porosity percentage, and hardness were measured 104 µm, 2.05%, and 79.5 HV, respectively. These conditions are recommended as the proper conditions for sintering.



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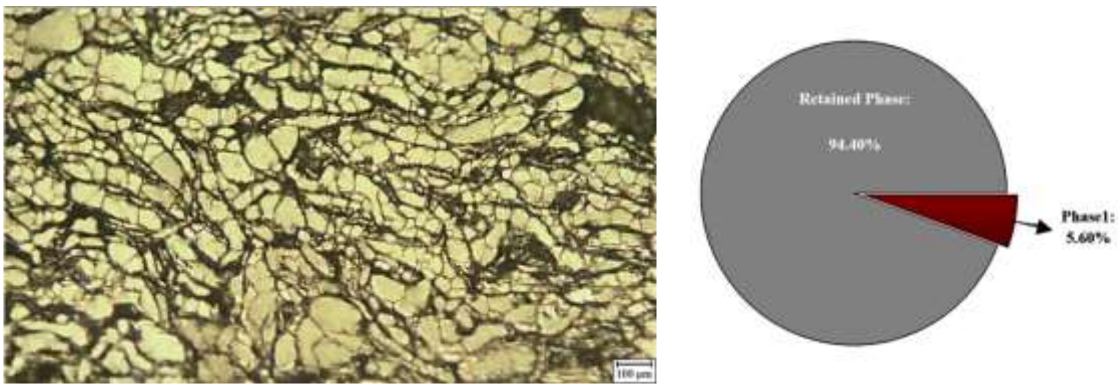

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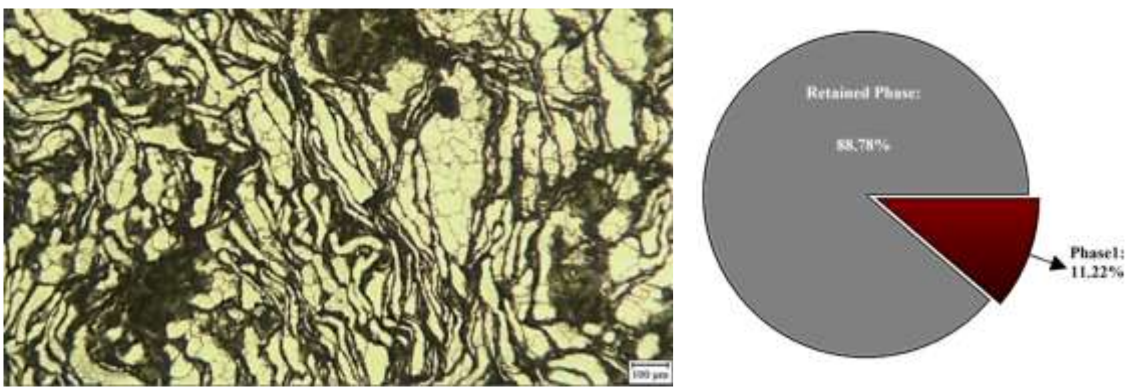


(c)

Fig. 3 Optical micrographs and the porosity percentage of AZ31 magnesium alloys for 3h sintering time with different heating rate: (a): by interring the sample directly in tube furnace, (b): 9 ºC/min heating rate, and (c): 5 ºC/min heating rate.

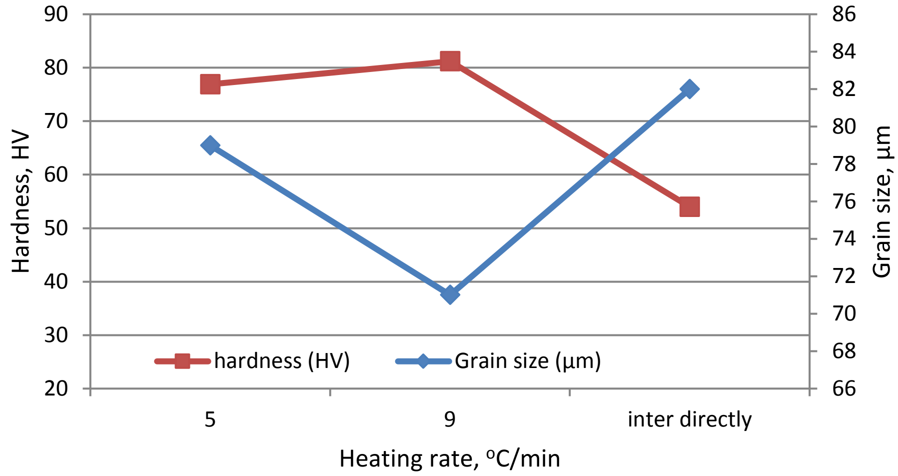
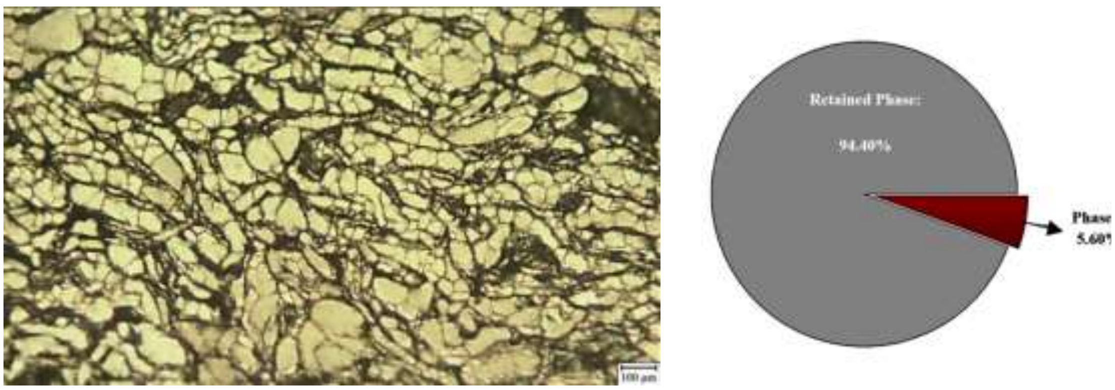


Fig. 4 The hardness and grain size of samples with different heating rate.



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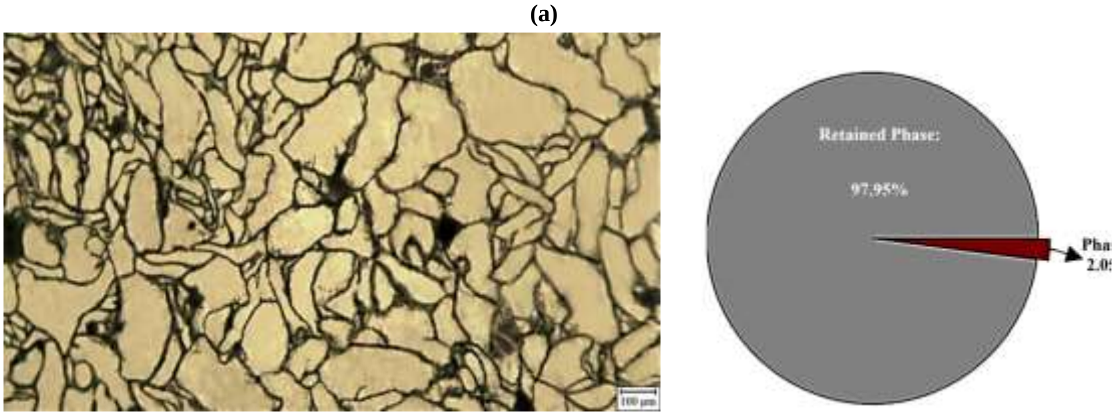

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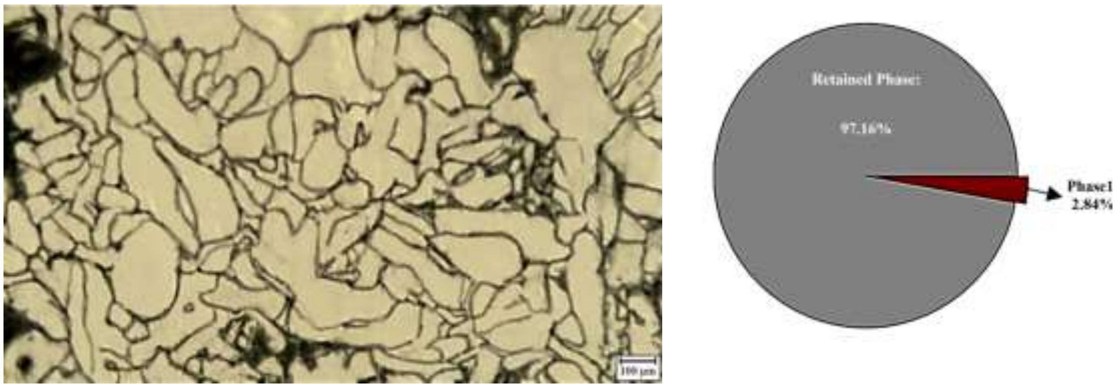


(C)

Fig. 5 Optical micrographs and the porosity percentage of AZ31 magnesium alloys sintered with 9 ºC/min heating rate and different sintering time: (a): 3h, (b): 16 h, and (c): 24 h.

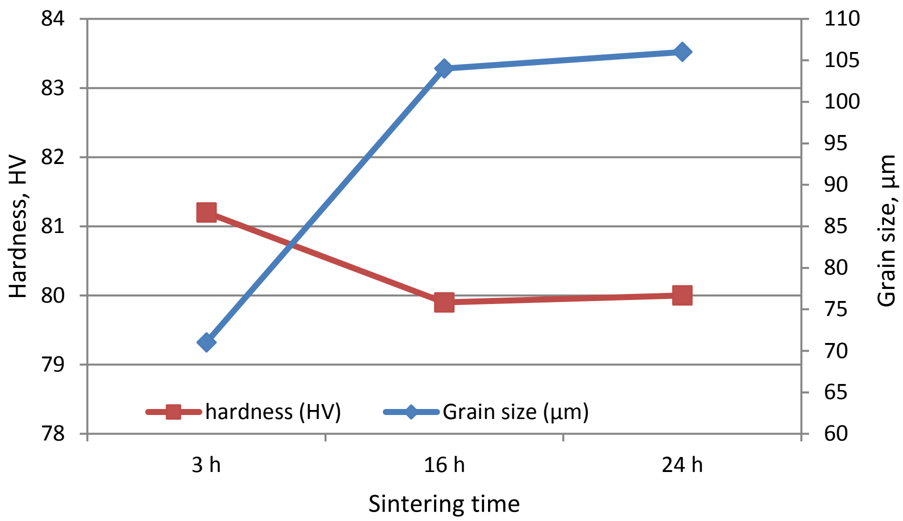


Fig. 6 The hardness and grain size of samples with different sintering time.

3.3. XRD Results

The milling process was applied for the mechanical alloying of raw materials to make them active. It was also conducted to improve the sintering procedure. To study the possible changes, the XRD results for the primary mixed sample and the milled one have been compared as illustrated in "Fig. 7". The crystallite size for both the mixed and milled powder was calculated via the Scherrer method which was equal to 1155 and 701 Å, respectively. These results confirm the association between the milling process and crystallite size. The data can be interpreted with the mechanical alloying energy which leads to lattice distortion and increase in defect quantity and lattice strain, subsequently. These two phenomena lead to the breaking of crystals and making them smaller. The Scherrer method can be described as the following Equation:

$$\tau = \frac{K\lambda}{\beta\cos\theta} \qquad (1)$$

In which, τ is the crystalline mean, K is the shape factor, which is often equal to 0.9, X-ray wavelength is shown by λ; β is equal to full width at half maximum (FWHM), and θ is the Bragg angle.

After the sintering process at 520°C for 3h, the new component of $Al_{12}Mg_{17}$ has appeared in XRD pattern as is shown in "Fig. 8". This is due to the reaction of aluminium and magnesium. This pattern was utilized for the calculation of crystallite size of magnesium after sintering which was equal to 484 Å. The most striking finding is that the magnesium crystallite size decreased during the sintering process. One possible explanation for this may be that the defects in the milling stage lead to recrystallization as the temperature rises, which ultimately causes the newly formed grains to become smaller. α-Mg phase and secondary β-$Al_{12}Mg_{17}$ phase are the two main phases of AZ31 alloy.

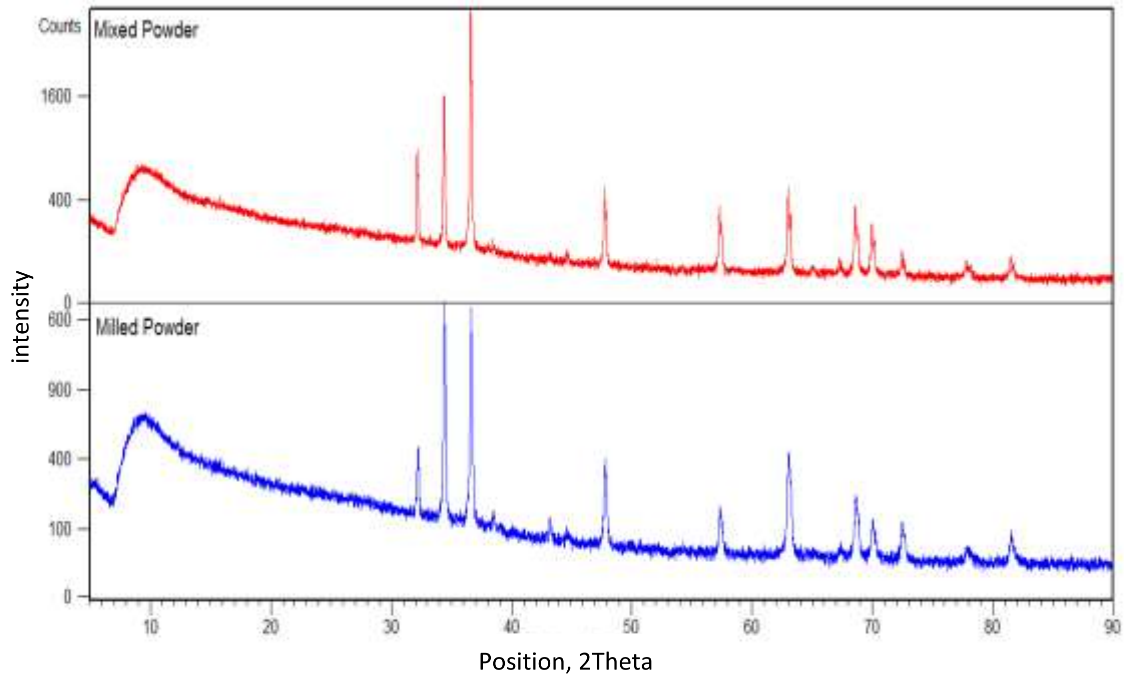


Fig. 7 The XRD results for the mixed and milled powder.

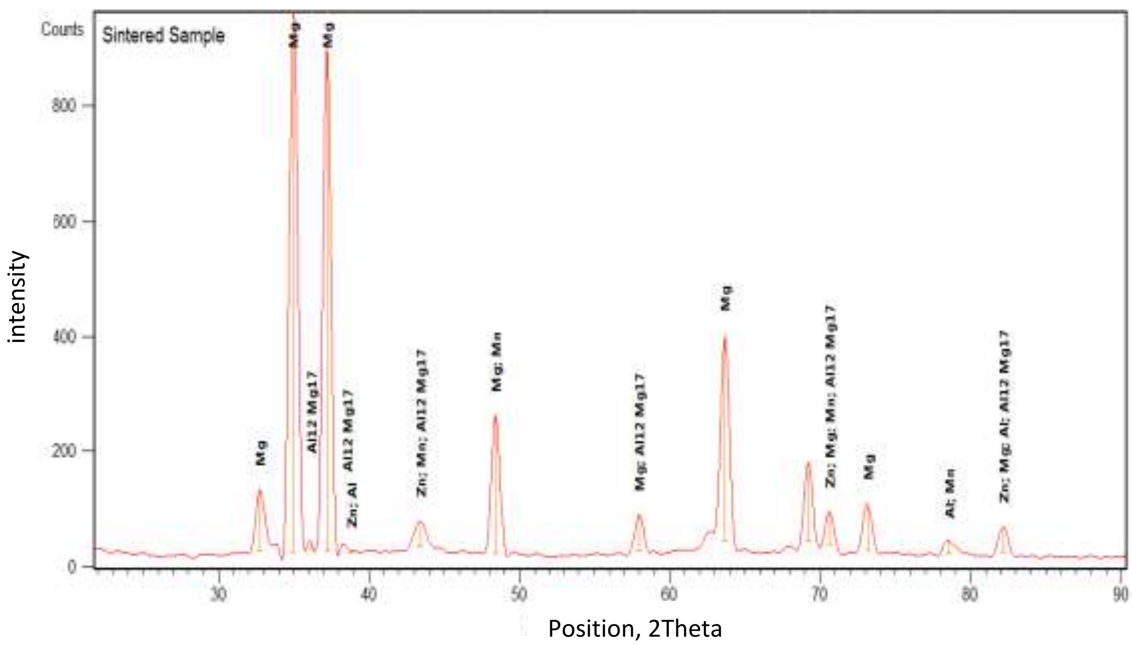
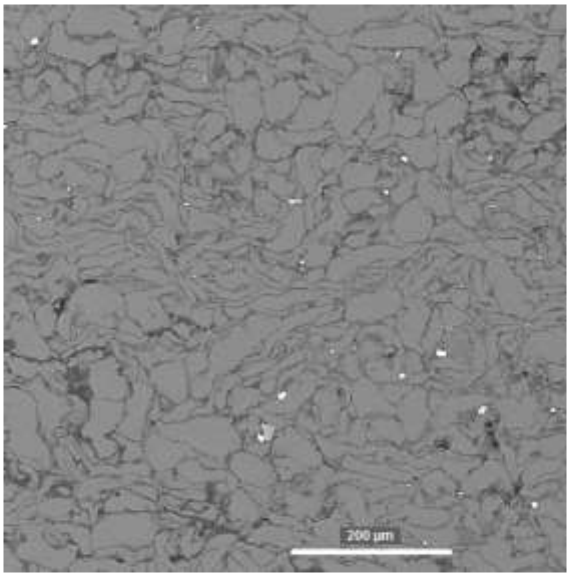
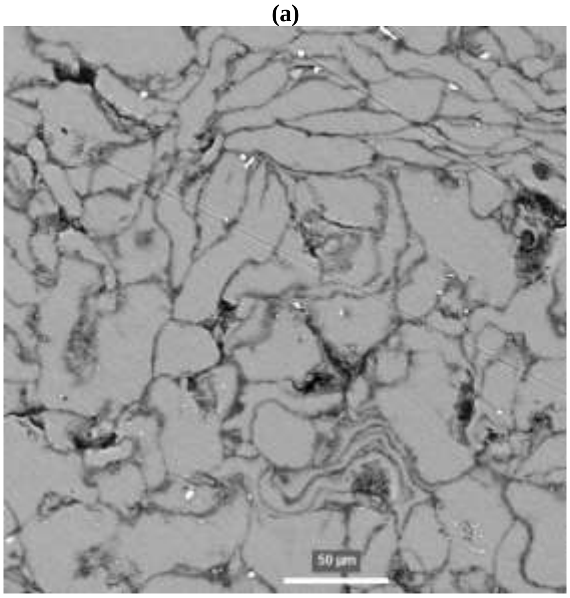


Fig. 8 The XRD results after the sintering process.

The SEM results of the sample sintered at 520°C for 3h, were shown in "Fig. 9". As can be seen, white components are detectable between the magnesium grains ("Fig. 9(a)"). EDX showed that this component is rich in Mg and Al. It means that MgAl was formed at the grain boundaries of Mg. EDX also showed that MgO presents in all grain boundaries of Mg. This is unavoidable due to the high tendency of magnesium to oxidize.



(a)



(b)

Fig. 9 The SEM image of the sintered sample at 520°C for 3h in two different magnifications.

4 CONCLUSIONS

AZ31 magnesium alloy was prepared based on its stoichiometry from elemental powders with ball milled, pressed and sintered in different conditions. The microstructure and the mechanical properties of AZ31 magnesium alloy were investigated to determine the effect of sintering parameters. The results showed that the sintering time is one of the major variables. In short sintering times, recrystallization leads to small grain formation inside the powder. However, as the sintering time increases, these new grains grow slowly and no trace of them can be detected in the microstructure. The high performance of sintered AZ31 magnesium alloy were obtained at sintering temperature of 520 °C, sintering time of 16h, and heating rate 9ºC/min, for which values of grain size, porosity percentage and hardness of the samples were measured as 104 µm, 2.05%, and 79.5 HV, respectively.

CONFLICT OF INTEREST

The author(s) declared no potential conflicts of interest with respect to the research, authorship and publication of this article.

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