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INVESTIGATION OF TRIBOLOGICAL AND MECHANICAL PROPERTIES OF BIODEGRADABLE AZ91 ALLOY PRODUCED BY COLD CHAMBER HIGH PRESSURE CASTING METHOD

In this study, AZ91 Magnesium alloy is produced by cold chamber high pressure die casting (HPDC) method. Different combinations of the cold chamber HPDC process parameters were selected as; in-mold pressure values of 1000 bar and 1200 bar, the gate speed of 30 m/s and 45 m/s, the casting temperatures of 640°C and 680°C. In addition, the test samples were produced by conventional casting method. Tensile test, hardness test, dry sliding wear test and microstructure analysis of samples were performed. The mechanical properties of the samples produced by the cold chamber HPDC and the conventional casting method were compared. Using these parameters; the casting temperature 680°C, in-mold pressure 1000 bar and the gate speed 30 m/s, the highest tensile strength and the hardness value were obtained. Since the cooling rate in the conventional casting method is slower than that of the cold chamber HPDC method, high mechanical properties are obtained by the formation of a fine-grained structure in the cold chamber HPDC method. In dry sliding wear tests, it was observed that there was a decrease in friction coefficient and less material loss with the increase of hardness values of the sample produced by the cold chamber HPDC method.

Keywords: Die casting, AZ91 magnesium alloy, cold chamber high pressure casting method, mechanical properties of AZ91 magnesium alloy, wear of AZ91 alloy, tribological properties of AZ91 alloy

1. Introduction

Magnesium is one of the lightest metallic materials (1.79 g/cm³ density) whose density approximately equals two-third of the aluminum and one-fifth of the steel, makes it desirable alloys in various industrial applications. Magnesium alloys offer many attractive properties such as good strength to weight ratio, low density, machinability, castability. All mentioned properties make that magnesium alloys can be used especially in aircraft industries, automotive industries mobile phones, sporting goods, handheld tools, household equipment, and various wear-resistant applications. Besides, magnesium alloys are biodegradable and it has drawn attention from orthopedic applications. Among the various types of magnesium alloys, the most preferred and successfully used commercial alloy is AZ91 which contains 9.0 wt % Al., 0.9 wt. % Zn and a small quantity of Mn [1-22].

High pressure die casting (HPDC) method is very good manufacturing technique in terms of efficiency and high capacity with a large scale i.e. from a few grams to a hundred kilograms. In 1905, after patenting of die casting method in the United States of America, there is a still great interest because of the method is suitable for fully automated casting lines for production of magnesium, aluminum, zinc and copper alloys into the desired shape. According to the injection systems, HPDC machines are classified into two main groups as cold chamber and hot chamber. The alloys which have low melting temperatures are suitable for hot chamber machines while have high melting temperatures ones are suitable for cold chamber machines. The quality and mechanical properties of products which is produced by HPDC methods are depend on many factors. Generally, these factors depend on part and die design but the process parameters play key role in affecting the mechanical properties of produced parts by HPDC method. The major process parameters are casting and die temperature, volumetric flow rate, filling time, nozzle and plunger velocity, gate speed and injection (specific) pressure [23]. The main advantages of HPDC process are high production amount with single die, low cost for a single product with high surface quality, fine microstructure due to fast cooling and it is not need to secondary machining. The disadvantages of this method can be stated as limited range of alloys, gas pores because of entrained

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air, limited size of casting parts, heat treatment difficulties because of gas pores and high tooling costs [24]. The mentioned problems can be eliminated by changing different process parameters and observing the effect on them. In recent years, there has been a growing number of publications focusing on producing magnesium alloys by HPDC method [25-28]. The researchers have investigated the influence of the different process parameters on mechanical properties of AZ and AM series of Mg alloys.

The main aim of this study is to investigate the effect of casting temperature, in-mold pressure and gate speed parameters on AZ91 Magnesium alloy which is produced by cold chamber HPDC method. In addition, the test samples were produced by conventional casting method. Mechanical properties such as tensile test, hardness test and wear test and microstructure analysis of samples produced by cold chamber HPDC and conventional casting method were compared

2. Experimental Procedure

The alloy selected for this study is AZ91 Mg alloy which is one of the most successfully used commercial alloy among the magnesium alloys. Magnesium ingots were supplied from Esan Eczacıbaşı Endüstri Hammaddeler San. ve Tic. A.Ş. from Turkey. The chemical composition of AZ91 Mg alloy ingots is shown in Table 1.

Chemical composition of AZ91 Mg alloy ingot (in weight %)

TABLE 1

Mg	Al	Cu	Ca	Zn	Mn
88	8.50	Max. 0.025	Max. 0.010	0.45	0.17

Firstly, the experimental samples were prepared using conventional casting method with metal melting furnace which has 200 kg capacity and they were used as control samples. Secondly, the HDPC samples were produced at Yıldız Technical University Die Cast Laboratory using Metal Pres MP100 (1600 kN) die cast cold chamber machine as shown in Figure 1. During casting



Fig. 1. Metal Pres MP100 die cast cold chamber machine

process, the temperature of the fixed die half was set to 175° C and that of the movable die half was set to 225° C (mean value was considered as 200° C), protective gas % 0.25 SF₆-N₂ (Balance) was used as 600 l/h flow rate. In order to investigate the effect of casting temperature, in-mold pressure and gate speed parameters on AZ91 Mg alloy 3 different experimental groups were used as listed in Table 2.

TABLE 2

Casting parameters used in experimental investigation for AZ91 Mg alloy

Test	HPDC Parameters				
Group	Casting Temperature °C	In-mold Pressure (bar)	Gate Speed (m/s)		
1	680°C	1000	30		
2	680°C	1000	45		
3	640°C	1200	45		

The sample preparation for metallographic characterization was performed from injected and casted parts according to standard metallographic procedures, it was carried out by grinding with progressively finer grades of sand papers from 320 grit to 1200 grit. The samples polished with a polishing cloth in 3 µm alumina paste. The grain structure was revealed by etching in solution containing 20 ml acetic acid, 1 ml nitric acid, 60 ml ethylene glycol and 19 ml distilled water. Nikon Eclipse MA100 optical microscope was used to microstructural investigation and the grain size was calculated by the intercept length method according to ASTM E112 for determining average grain size. During grain size measurement Metalim software was used. The tensile tests were performed using ALSO UTM 100 universal testing machine which has 300 kN capacity according to ASTM B557M-2016 with a crosshead speed of 0.2 mm/min at room temperature. Each data represents at least average of three samples in tensile tests. Brinnel hardness measurements were performed with Emcotest DuraVision 30 model under a load of 75 kg 5 mm the spherical indenter was pressed into a sample with a holding time of 30 s. The reported the Brinell hardness (HBW) values represents at least three different measurements. The dry sliding wear behavior of the injected and casted AZ91 Mg samples were assed using a ball-on-disc CSM-Tribometer with WC ball of 3 mm (certified sphericity) as static friction partner. The tribometer and the view of the wear test setup is shown in Figure 2. The wear test was conducted according to ASTM G99 test standard. The wear samples were mounted to hold easily into the holder of the tribometer. Before the wear test, the mounted samples were ground to obtain an average same surface roughness value (R_a) . All wear tests were performed at 20°C, 32% relative humidity, 10 m wear distance, 5 mm/s speed, 2.5 mm the radius of the wear track and 2 N load. At least average of three wear tests results was reported. After completion of the wear tests, the wear tracks were examined using a SEM with and EDS and also 3D optical profilometer (surface roughness and wear measurement) device in order to identify the dominant wear mechanism.

TABLE 4



Fig. 2. CSM tribometer and the view of the wear test setup

3. Results and Discussion

The in-mold pressure values of first group, second group and third group of experimental samples which were produced by cold chamber HPDC were specified as 1000 bar, 1000 bar and 1200 bar, respectively. The gate speed was defined as 30 m/s, 45 m/s and 45 m/s and casting temperature was set to 680°C, 680°C and 640°C respectively for each group of samples. The calculated average grain size and ASTM grain size number (G) of the samples which were produced by cold chamber HPDC and conventional casting methods were given in Table 3. The microstructure of all group specimen was shown in Table 4.

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Average grain size and ASTM grain size number of the experimental samples [22,28]

	HPDC Parameters			A	ASTM
Test Group	Casting Temperature °C	In-mold Pressure (bar)	Gate Speed (m/s)	Average grain size (mm)	grain size number (G)
1	680	1000	30	0.013	10
2	680	1000	45	0.010	10
3	640	1200	45	0.015	9
4	conventional casting methods			0.051	5.5

The average grain size of the first test group was higher than that of second test group but same ASTM grain size number (G) was obtained for both test groups. Comparison of the group first and second tests, these findings suggest that as the gate speed increased from 30 m/s to 45 m/s, the grain refining was observed. As the casting temperature decreased to 640°C and the in-mold temperature increased to 1200 bar, the average grain size became more coarser which was similar to test group obtained by conventional casting methods. When the optimal and appropriate process parameters are specified in cold chamber HPDC method, the fine-grained microstructure is obtained because of the cooling rate is faster than the conventional casting methods. In comparison to cold chamber HPDC method, as the conventional casting method has slower cooling rate, the coarse-grained microstructure is obtained. The microstructure and the screenshot of software which shows the ASTM G number [22,28]



According to previous research which was conducted by Caceres et al. (2005), five different mechanism i.e. solid solution strengthening, grain boundary strengthening, dispersion strengthening, Orowan looping and general dislocation hardening can be carried out on AZ91 magnesium alloys [29]. Several parameters i.e. the casting temperature, the thickness of the casting part, the distance of the part from the gate, the speed of the melt at the gate and in-mold pressure affect the microstructure of the AZ91 Mg alloy which was produced by cold chamber HPDC method. There is a direct proportion between mechanical properties and microstructure. The quality and mechanical behavior of the casted parts can be improved by controlling process parameters. In all crystalline materials, small grain size gives higher mechanical properties according to famous Hall – Petch equation. It is

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also known as grain boundary strengthening mechanism with this formula $\sigma_v = \sigma_0 + kd^{-1/2}$, where σ_v is yield strength, *d* is grain size, σ_0 and k are constants which are reported in several reports and researches. Generally, the grain size of AZ91 Mg alloys produced by HPDC methods varies from 5 µm to 10 µm [30]. But in the literature the reported grain size has very wide range. The reasons of this case are the effect of skin region, externally solidified crystals and grain boundary strengthening. The skin effect is the main characteristic of the HPDC microstructure. As the metallic die is used, rapid cooling is active in casting process and it is resulted a thin surface layer with fine grain size [31]. A thin solid layer is formed at the shot sleeve wall during solidification of the melted metals and it generally formed in equiaxed dendritic crystals in liquid. These solidified primary crystals are defined as presolidified crystals, floating crystals and externally solidified crystals (ESC). The ESCs are undesirable for HPDC magnesium products. As they have larger grain size than the grains formed in the cavity, the mechanical properties are affected in negative way. In conclusion, it is clear that the main strengthening mechanism is fine grain microstructure by choosing optimum process parameters for AZ91 Mg alloy.

At the end of the HPDC process, grain size finer and final microstructure more complex in comparison to other casting process. In Mg-Al casting alloys, aluminum is the main alloying element. Variation in the distribution of Al for the same region, α -Mg regions or β -Mg₁₇Al₁₂ region can be observed. For magnesium AZ91 alloy, zinc is the second alloying element and its concentration varies from 0.45 to 0.90 wt%. It is important for corrosion behavior of magnesium alloys. Mn is the third alloying element with 0.15 to 0.30 wt%. The HPDC Mg alloys exhibit partially or fully divorced morphology. These morphology types depend on several factors such as aluminum content, zinc content and cooling rate [32].

The tensile tests were performed to determine the tensile strength and elongation of the samples which were produced by cold chamber HPDC and conventional casting methods according to ASTM B557M-2016. The same grouping was used for designation, test groups 1, 2 and 3 belong to cold chamber HPDC method and test group 4 represents the conventional casting method. Tensile test and elongation results can be seen in Figure 3. The highest tensile strength and elongation value was obtained from samples of test group 1. For the test group 2, the results showed that the tensile strength and elongation of samples decreased in comparison to test group 1 with the increasing gate speed from 30 m/s to 45 m/s and keeping constant casting temperature and in-mold pressure. This can be attributed to effect of grain size coarsening. For the test group 3, by determining process parameters as casting temperature 640°C, in-mold pressure 1200 bar and gate speed 45 m/s, almost the same tensile strength and elongation were obtained as the conventional casting methods. Figure 4 shows the graphical representation of Brinell hardness measurements of the samples produced by cold chamber HPDC and conventional casting methods. The same trend was also observed for hardness tests for all tested groups. As the average grain size was decreased, the measured hardness values were increased. Overall, these results indicated that the mechanical properties such as tensile strength, elongation and hardness of the cold chamber HPDC parts were a function of grain size as mentioned before by many researchers. The comparison of the obtained tensile properties data and the behavior of the finegrained AZ91 Magnesium alloys are in good agreement with literature [29,30,33-35]. The change of the grain size of the casted samples from coarse grain to fine grain improved the mechanical properties. The dislocations are defects in the lattice structures and the movement of the dislocations are hindered by the grain boundaries. The more grain boundaries mean that there are more difficult for dislocations to move. Therefore, the fine-grained alloys have higher mechanical properties than coarse-grained alloys. Another reason of obtained higher mechanical properties of AZ91 Mg alloy in recent study is the effect of the second phase hardening. A series of phase transformations occur during the solidification of a casting from the liquid state. In simple binary alloys such as Mg-Al which is shown in Figure 5, the primary α -phase nucleates first and as the temperature falls, the α -phase will grow until the eutectic temperature is reached at which point the eutectic nucleates consuming the remaining liquid [36]. On the other hand, the pressure applied during solidification makes the accepted solidification theory inapplicable. The formation of second phase of Al in magnesium alloys affected the mechanical properties of AZ91 alloy in positive way. But the ratio of formed phases is more dominant parameter on behavior of mechanical parameters of the alloy. For specimens of test group 3 and test group 4, the samples have coarse grain structure and the ratio of α -Mg has higher in comparison the other groups. It can be said that the temperature over 650°C, the viscosity has a tendency to decrease and the melted metal can easily fill the die. With choosing appropriate pressure value, it is possible to reducing resistance to flow and porosity. In regard to the grain morphology and the ratio of the phase, the lower gate speed is more suitable for HPDC process.



Fig. 3. Tensile strength and elongation result of the samples produced by cold chamber HPDC and conventional casting methods





Fig. 4. Brinell hardness of the samples produced by cold chamber HPDC and conventional casting methods



Fig. 5. Mg-Al phase diagram [36]

The dry sliding behavior of the samples which were produced by cold chamber HPDC and conventional casting methods were assessed using a ball-on-disc tribometer with WC ball of 3 mm as a static friction partner. For each group, the wear experiments were carried out by triplicate. In the literature, the dry sliding wear tests were performed under three different load levels such 2 N, 5 N and 10 N for AZ91 Magnesium alloys. Generally, the friction coefficient of AZ91 alloys varies in the range of 0.24-0.40 and it is almost independent of different test loads [2,6,7]. Srinivasan et al. reported that under 2 N, 5 N and 10 N wear loads, the mean coefficient of friction were obtained as 0.31, 0.28 and 0.27, respectively [7]. It can be said that the friction coefficient is almost independent of different loads of wear testing. That is the reason in present study, all of the wear tests were performed only under 2 N wear load. The average friction coefficients of the tested all samples were calculated and tabulated in Table 5. Additionally, the coefficient friction as a function of sliding distance were depicted in from Figure 6 to Figure 9 for one of the samples from each tested group.

Summary of the friction coefficient of the tested AZ91 Magnesium alloys

Test Group	HPDC Parameters			E-d-d-	Average
	Casting Temperature °C	In-mold Pressure (Bar)	Gate Speed (m/s)	Friction coefficient (µ)	Friction coefficient (µ)
1-1				0.26	
1-2	680	1000	30	0.21	0.26
1-3				0.30	
2-1				0.29	
2-2	680	1000	45	0.28	0.29
2-3				0.30	
3-1				0.32	
3-2	640	1200	45	0.29	0.31
3-3				0.33	
4-1				0.35	
4-2	Conventional casting methods			0.34	0.33
4-3				0.32	



Fig. 6. Dry sliding wear behavior of test group 1



Fig. 7. Dry sliding wear behavior of test group 2



Fig. 8. Dry sliding wear behavior of test group 3



Fig. 9. Dry sliding wear behavior of test group 4

During the dry sliding wear tests, two different characteristics were observed such as running-in and steady state behaviors. Initially, the friction coefficient increased until it reached the maximum value and it followed by a gradual decreasing to steady state condition. After one tenth of the wear test, all tested samples exhibited stable friction characteristics. Additionally, in the friction plots there was no large fluctuations were observed until the end of the wear test and all the tests were completed in steady state. Nearly all metals are oxidized in the air and this oxide layer prevents the contact of WC ball and surface of AZ91 magnesium alloy at the first stage of the wear test. As the formed oxide layer has decreasing effect on friction, generally lower friction coefficient is observed after starting of the wear test. During the test, if the applied load is enough to break down the thin oxide layer, it is removed from surface and WC ball starts to contact the surface of the tested alloy. This case is responsible for



Fig. 10. 3D surface profile of sample from test group 1



Fig. 11. 3D surface profile of sample from test group 2

observing higher adhesion and friction coefficient after beginning of the test. For the test groups 1, 2, 3 and 4, the average friction coefficient values under at 2 N load were found to be 0.26, 0.29, 0.31 and 0.33, respectively. The highest friction coefficient was obtained the sample from test group 4 which was produced by conventional casting method. According to the wear tests, it is clear that the friction coefficient gradually decreased from test group 3 to test group 1. The findings stated that it is possible to decrease the friction coefficient with choosing optimal HPDC parameters. Srinivasan et al. found that the average friction coefficient of AZ91 alloy was 0.31 under 2 N load. As stated before, the friction coefficient values were fluctuating in the range of 0.24-0.40. The results from earlier studies demonstrated a strong and consistent association between present study. After the wear tests, the surface topography analysis was carried out by 3D surface profilometer and the obtained surface profiles were depicted in from Figure 10 to Figure 13 for each group. For the test groups 1, 2, 3 and 4, the average surface roughness values were found to be 0.026 μ m, 0.027 μ m, 0.033 μ m and 0.0059 μ m, respectively. It was observed that the surface roughness values obtained by cold chamber HPDC method showed parallelism with the friction coefficient. Higher amount of wear was obtained due to the coarse grain structure for the sample which was produced by conventional casting method and it resulted with lower roughness value. Besides, the sample which was prepared from conventional casting method showed lower mechanical properties in both hardness and tensile tests. Therefore, the rate of wear was expected to be high. The grain



Fig. 12. 3D surface profile of sample from test group 3



Fig. 13. 3D surface profile of sample from test group 4

refinement is an important factor and it has direct effect on the mechanical properties.

In order to determine the specific wear rate (mm³/Nm) which was calculated using normal load (2N) and sliding distance (10 m) of all tested sample against WC ball, the wear profile was measured as shown in Figure 14. The specific wear rates of the AZ91 magnesium alloys which were produced by different casting methods was depicted in Figure 15. The specific wear rate were found to be 6.095, 9.715, 10.615 and 12.910 mm³/Nm for the test group 1, 2, 3 and 4, respectively. The highest wear rate was obtained for the test group 4 which was produced by cold chamber HPDC method while the lowest wear rate observed for the test group 1 which was produced by conventional casting method. It is known that the wear resistance is directly proportional the hardness value. The test group 4 had highest value (84.3 HBW) in terms of hardness and this group alloy provided highest wear resistance when compared to other test groups. In addition to that the test group 4 had gained the highest mechanical properties such as tensile strength and elongation because of the fine grain microstructure. These mechanical properties gave higher plastic deformation resistance to test group 1 samples. As expected, the highest specific wear rate was obtained for the test group 4 because it had the lowest value (62 HBW) in terms of hardness. By taking into consideration of the results of test group 1 and 4, the wear resistance of AZ91 magnesium alloy can be doubled by choosing cold chamber HPDC parameters in comparison to the conventional casting method. In summary, it can be said that the specific wear rates and wear resistance were governed by the hardness differences of produced AZ91 magnesium alloy with different casting methods and parameters.

SEM and EDS analysis of the wear track for each group were presented in from Figure 16 to Figure 20. As the substrate material was magnesium and the wear ball was WC, it was expected to result in abrasive wear for all group of the wear tests. Because there was a great difference between WC ball and AZ91 magnesium alloy in terms of hardness value. It was observed that every wear track more or less similar for every group. In the initial stages, because of the a point contact, a high contact stress was obtained. During the sliding of WC ball, groove formation which sometimes called as ploughing was observed. Besides, the micro valley and the hills which were formed as parallel direction to each other as shown in Figure 14 and almost these wear scars were observed for tested all samples. As the temperature and the pressure were high, the wear particles of magnesium substrate transferred to the WC ball by adhesive wear mechanism. It can be said that the wear scar was visible and



Fig. 14. Worn area of test group 1, test group 2, test group 3 and test group 4 samples



Fig. 15. Specific wear rate of test group 1, test group 2, test group 3 and test group 4 samples

the wear debris got smeared on the surface for specimen which had lower hardness value. On the contrary, for the specimen which had higher hardness value, the wear scar was decreased and no smeared debris was observed on the surface. The wear scar was not clear for the test group 1 while the wear debris got smeared on the surface of specimen for the test group 3. It is known that there is an inverse proportion between hardness and wear process. The grain size varied between from 5.5 µm to 10 µm depending on cold chamber HPDC parameters and it had great effect on the mechanical properties. It can be said that the fine-grained structures had higher mechanical properties and therefore the wear surfaces were obtained more smoothly under the same wear test conditions. During the continuous wear test, the material which was transferred to the WC ball surface got work hardened as a result of the extensive plastic deformation. As the temperature of the contact surfaces increased, the transferred material oxidized during the sliding, the deep grooves were observed in the wear track. The EDS results showed that predominantly magnesium, aluminum, carbon and oxygen were found and these findings were proof of the oxidation process. In some region of the worn surfaces showed the signs of the formation of adhesive transfer particle. It can be said that the



Fig. 16. SEM of wear tracks for test group 1



Fig. 17. SEM of wear tracks for test group 2





Fig. 18. EDS analysis of the wear track for test group 2



Fig. 19. SEM of wear tracks for test group 3



Fig. 20. SEM of wear tracks for test group 4

adhesive wear mechanism was not fully dominant but partially observed in the worn surfaces.

4. Conclusions

In this study, the dry sliding wear behavior and mechanical properties of experimental samples which was produced by cold chamber high pressure die casting method using different process parameters and by conventional casting method were assessed and compared. As a result of the mechanical tests, the wear tests and the metallographic examinations; different strength and hardness values, wear and friction behaviors and grain size values were obtained depending on the casting process parameters.

- When suitable cold chamber HPDC process parameters are selected for producing AZ91 magnesium alloys, mechanical properties can be obtained as desired by controlling the grain structure.
- The highest mechanical properties such as the tensile strength and the hardness value were obtained for test group 1 which was produced using these parameters; casting temperature 680°C, in-mold pressure 1000 bar and the gate speed 30 m/s.
- As the cooling rate in the conventional casting method was slower than the cold chamber HPDC method, higher mechanical properties were obtained due to the formation of the fine grain structure in the cold chamber HPDC method. Since grain boundaries act as a barrier to dislocation motion during deformation, the strength of the fine-grain alloys is higher in comparison to coarse grain alloys.
- According to the results of the dry sliding wear tests which was assessed by ball-on-disc configuration, it was observed that there was a decrease in friction coefficient and less material loss with the increase in hardness values of the sample produced by the cold chamber HPDC method. The wear resistance can be increased with the choosing optimum process parameters of the cold chamber HPDC method.

If the high pressure die casting process parameters are not controlled during the production of magnesium alloys, almost the same mechanical properties can be obtained with the conventional casting method. When considered from this point of view, unnecessary investment is made due to the high cost of the high pressure die casting.

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