

Effects of process parameters on mechanical and metallurgical properties in high pressure die casting of AZ91 magnesium alloy

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High pressure die casting (HPDC) of magnesium (Mg) alloys has been the fastest grown up and the most globally developed section in magnesium industry. HPDC of complex shape Mg alloy products have increased considerably in recent years. But low mechanical and metallurgical performance of the casting products could be experienced due to defects in HPDC of Mg alloy parts under heavy working conditions. Process technologies should be modified and process parameters need to be optimized for the use of Mg based alloy products as high performance casting parts. The correct selection of the process parameters means the correct manufacturing of the casting parts. There is a wide range of suggested process parameters for HPDC of different Mg alloys in the literature. This paper specifies optimum process parameters for the required mechanical and metallurgical properties of the die casting parts, experimentally. Experimental tests are performed by using Taguchi experimental procedure to determine the optimum process parameters in cold chamber HPDC of Mg alloy parts. Confirmation and statistical analyzing tests have confirmed the results. The results minimize the available range of process parameters in the literature for high mechanical properties and low porosity content of casting products by conducting the designed experiments in an industrial scale mass production line considering the product quality.

Keywords: Magnesium alloys, High pressure die casting, Optimum process parameters, Mechanical and metallurgical properties, Product quality

Mg alloys have been used and preferred in many sectors, especially in automotive, aerospace and electronics industries with appropriate characteristics such as lightness and strength. In the magnesium industry, HPDC of Mg alloys has been developing significantly on the global scale for many years. Due to lack of experience and knowledge in this area, some industries deliberately stay away from this sector thus they cannot benefit from Mg metal which has been widely using all over the world for many years¹⁻³. Although HDPC is a very precise process, there are many factors effective on product quality and mechanical properties in a negative way. These factors include the product design, die construction and the various process parameters⁴. Some academic researches that represent the positive correlation between the process parameters and the mechanical properties of the casting parts are very valuable⁵⁻¹⁵. Gas protection systems must be integrated to the production line in order to be successful in the melting process of Mg alloys due to the high affinity of Mg to the oxygen. Otherwise, molten Mg reacts with the oxygen in the air exothermally. This reaction could be too hard to control. Moreover, the eutectic phase,

Mg₁₇Al₁₂, oxidizes at 430°C. Because of that, aluminium included die casting Mg alloys must be protected over 400°C, before the melting starts. There is significant number of researches on protective ability of fluorine containing gases on molten metal bath¹⁶⁻¹⁹. There is a wide range of suggested process parameters for die casting of different Mg alloys in the literature⁵⁻¹⁹. This paper specifies optimum process parameters for high mechanical and metallurgical properties of the casting parts experimentally. It is aimed to minimize the available range of process parameters in the literature for high mechanical properties and low porosity content of casting products by conducting the designed experiments in an industrial scale mass production system considering the product quality.

Experimental Procedure

The optimum process parameters should be determined precisely to obtain high mechanical properties with fulfilling the casting part features and low porosity content²⁰. The HPDC production line used in the experimental stage is shown in Fig. 1 as a flow chart. The system includes a dosing furnace, a molten metal transfer system, a gas mixing unit, a die heating-cooling device, and a cold chamber die

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casting machine. In the flow chart, three basic production steps (melting, dosing, and casting) were detailed precisely. Since the process parameters are the most effective factors on product quality, they were optimized by using experimental results.

Bath and die temperatures, gate velocity and intensification pressure with protective gas concentration are considered as the experimental factors of the process parameters. After manufacturing the physical, mechanical, metallurgical and geometrical properties of the samples were determined and compared with available data in the literature. Finally, the optimum process parameters were selected by analyzing test results including mechanical strength and other determined quality properties of the die casting products. In the experimental stage of the work, AZ91 series commercial Mg alloys are preferred due to their widespread usage and favorable physical, mechanical and metallurgical properties. The common mechanical

and physical properties of AZ91 alloy are; 230 MPa in tensile strength, 150 MPa in yield strength, 7% elongation in 50 mm, 63 HB hardness, 1.81 g cm⁻³ density and 595°C melting temperature²¹⁻²³. Chemical composition of the used AZ91 alloy in the experiments is given in Table 1.

The size of the required HPDC machine is basically determined by the shot volume and the projection area of the casting product including the designed gating system considering the maximum pressure during the intensification phase. The selected METAL PRES, MP100 type, Turkey, is a cold chamber type. The injection system has three steps respectively, the slow (the 1st), the fast (the 2nd) and the intensification (the 3rd) phases. Plunger velocity and injection pressure are adjusted electronically by a hydraulic system with a proportional valve. The schematic drawing of the integrated system components is shown in Fig. 2.

Table 1 — Chemical composition of the used AZ91 magnesium alloy

Element	Mg	Al	Zn	Mn	Fe	Cu	Si	Ni	Cr	Ti	V	W
%	89.830	9.077	0.682	0.180	0.026	0.002	0.014	0.004	0.009	0.033	0.018	0.009
±	0.303	0.297	0.013	0.014	0.006	0.001	0.009	0.002	0.003	0.008	0.005	0.003

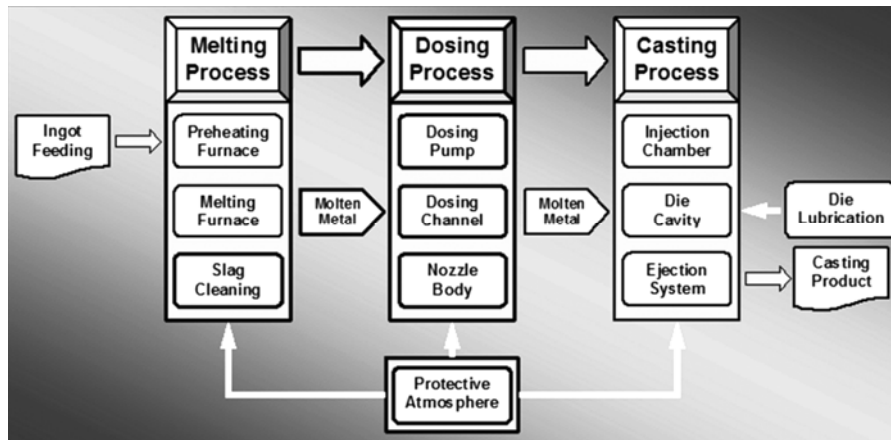


Fig. 1 — The detailed flow chart of the HPDC production line

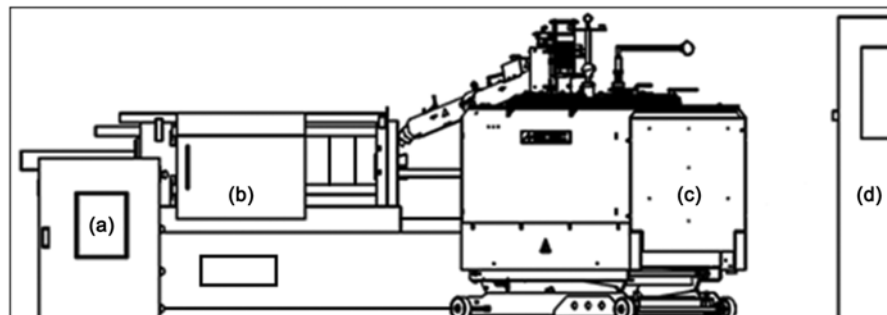


Fig. 2 — Schematic drawing of the integrated system components (a) control panel, (b) HPDC machine, (c) melting furnace and (d) gas mixing unit

The used melting unit is a stable crucible type electrical resistance furnace, MELTEC GmbH, MDF-200C, Austria. Its melting temperature is 700°C in maximum which is high enough for Mg die casting alloys. The resistance furnace with 85 kW heating power, 200 kg.h⁻¹ melting rate, and 520 kg crucible capacity is integrated to the mass production system for the experiments. The protection of molten Mg alloy with the protective gases (fluxless method) is preferred for the HPDC process. One of the most effective protective atmospheres in melting process of the magnesium alloys is the mixture of fluorinated gases. N₂+SF₆ gas mixing is a proper solution to protect the die casting Mg alloys from oxygen during manufacturing²²⁻²⁴.

The die is designed with four cavities. The cast parts manufactured as standardized cylindrical tensile test samples according to ASTM B557M-15²⁵. The projected area is calculated approximately 125 cm², and the total injection weight is 250 g. Die halves are made of AISI H13 hot work tool steel. The technical drawing of the designed die is given in Fig. 3. The die heating and cooling device, ISITAN, CH210-S type, Turkey, is used to have a homogeneous temperature distribution of the fixed and the moveable die halves.

Experimental Procedure

The presented experimental design method in this study is the Taguchi design by which the inherent variability of materials and manufacturing processes has been taken into account at the design stage. The

parameter design stage has been widely using to optimize the manufacturing processes²⁶. The vital goal of this stage is to determine the optimal process conditions that yield the minimum porosity results the maximum density and the maximum strength. Taguchi L27 orthogonal experimental design matrix is given in Table 2. The investigated experimental factors as the process parameters are bath temperature, die temperature, protective gas concentration, intensification pressure and gate velocity. In addition, the obtained test results as the response factors are density, diameter, elongation, yield and tensile strengths. Taguchi experimental design has been analyzed considering these factors related with signal/noise (S/N) ratio for "larger is better" approach by Minitab 16.1.1 statistical software. It means, the optimum process parameters will be the maximum points of the S/N ratio graphics that yield the maximum density, elongation and mechanical strength with the optimum dimensional tolerance. The experimental factors and their levels are given in Table 3. A number of 27 distinctive runs were performed in the production system. Then, samples were tested in order to determine their physical, mechanical and metallurgical characteristics. The mechanical properties of AZ91 cast samples were determined mechanically at Shimadzu, AG-X type, 100 kN, Japan tensile test machine, at 4 mm.min⁻¹ tensile velocity according to ASTM B557M-15²⁵. The obtained results are informative about elongation, yield and tensile strengths of the samples. Additionally, Brinell macro hardness tests were performed repeatedly according to ASTM E10-15a at Zwick Roell, ZHU/2.5 type, Germany, universal hardness testing equipment²⁷.

Moreover, the manufactured tensile bars were subjected to diameter and density measurements. Porosity percentage calculations were made by means of density measurements in accordance with Archimedes' principle. Optical microscope images were captured by Leica, DM 750M type, Germany, lightening microscope at 500X. The captured surfaces were polished by a proper metallographic surface preparation technique and dispensed by 2.5% Nital solution for 15 s^{28,29}. SEM examinations were done at the fractured surfaces of the tensile bars. No sample preparation was made on the fractured surfaces. In addition, SEM and EDS analyses were done at Hitachi, TM1000 type, Japan, scanning electron microscope with an acceleration voltage of 15.0 keV,

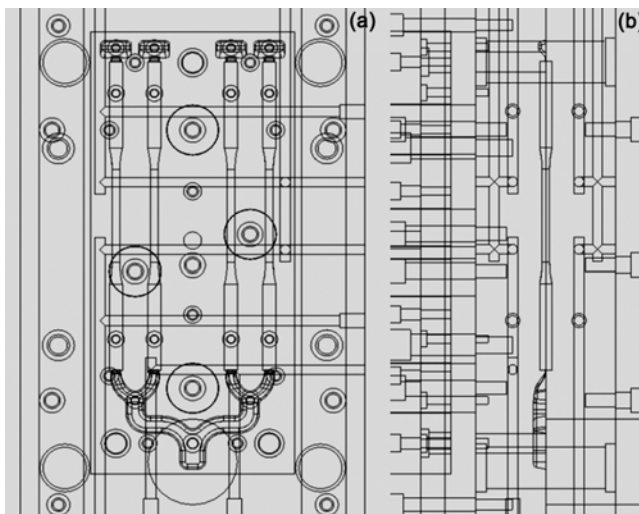


Fig. 3 — Technical drawing of the die casting mold (a) front view and (b) side view

emission current of 140 mA, 30 s for plasma activation and 6.5 mm working length. Elemental dispersions on fractured surfaces were determined in % weight by EDS analysis. Finally, XRD analyses were conducted by GBC, Mini-Materials Analyzer type machine, USA, at 28.5 mA emission current, 35.0 kV acceleration voltage and with 1.0 kW power at 3.19 A filament current to understand metallurgical characteristics of the samples. All given results are the average values of the repeated measurements.

Results and Discussion

Tensile and yield strengths, density and diameter measurement results with elongation values are given in Table 2 as the response factors. ± 0.04 mm dimensional tolerances, 66 HB hardness, 1.78 g.cm^{-3} density and less than 2% porosity values in average and maximum 157 MPa yield and maximum 248 MPa ultimate tensile strengths with maximum 7.67% elongation in 50 mm were achieved. Considering one of the most used "larger is better" approach of the statistical software, the optimum process parameters

Table 2 — The used Taguchi L27 orthogonal matrix

Run	Experimental factors					Response factors				
	Bath temp. °C	Die temp. (°C)	Gas conc. % Vol.	Intensification pressure MPa	Gate velocity m.s^{-1}	Density g.cm^{-3}	Diameter mm	Elongation % (in 50 mm)	Yield strength MPa	Tensile strength MPa
1	1	1	1	1	1	1.78	5.94	4.98	140.53	204.58
2	1	1	1	1	2	1.78	5.95	4.85	154.61	228.75
3	1	1	1	1	3	1.77	5.94	4.38	143.37	219.94
4	1	2	2	2	1	1.77	5.93	5.65	152.09	211.78
5	1	2	2	2	2	1.77	5.94	4.03	125.77	172.50
6	1	2	2	2	3	1.77	5.95	5.54	141.53	229.35
7	1	3	3	3	1	1.78	5.97	7.08	146.87	230.54
8	1	3	3	3	2	1.77	5.94	5.58	135.04	206.02
9	1	3	3	3	3	1.78	5.94	5.40	143.20	169.78
10	2	1	2	3	1	1.78	5.92	7.65	146.42	232.47
11	2	1	2	3	2	1.78	5.95	7.67	157.10	248.09
12	2	1	2	3	3	1.78	5.96	6.92	148.66	230.37
13	2	2	3	1	1	1.79	5.99	6.58	140.21	217.22
14	2	2	3	1	2	1.78	5.97	6.33	135.20	222.20
15	2	2	3	1	3	1.77	5.97	6.07	133.48	229.53
16	2	3	1	2	1	1.78	5.98	5.50	149.00	202.98
17	2	3	1	2	2	1.78	5.97	4.97	126.44	204.34
18	2	3	1	2	3	1.78	5.97	4.83	138.37	216.59
19	3	1	3	2	1	1.78	5.96	6.47	142.13	212.68
20	3	1	3	2	2	1.78	5.95	5.42	143.30	218.24
21	3	1	3	2	3	1.77	5.98	4.83	125.33	221.25
22	3	2	1	3	1	1.78	5.98	7.43	132.19	236.44
23	3	2	1	3	2	1.78	5.98	5.00	129.45	213.07
24	3	2	1	3	3	1.78	5.99	7.08	133.06	239.29
25	3	3	2	1	1	1.78	5.97	6.62	149.83	201.43
26	3	3	2	1	2	1.78	5.98	6.30	137.51	218.63
27	3	3	2	1	3	1.77	5.98	6.08	133.70	218.81

Table 3 — Experimental factors and their levels

Levels	Bath Temperature °C	Die Temperature (°C)		Gas Concentration % Vol.	Intensification Pressure MPa	Gate velocity m.s^{-1}
		Cover Side	Ejector Side			
1	640	150	200	0.20	80	30
2	660	175	225	0.25	100	45
3	680	200	250	0.30	120	60

are appeared as the maximum points of the given five S/N ratio graphics in Fig. 4. With the help of these results, the optimum levels of bath and die temperatures, gate velocity, and intensification pressure with protective gas mixing rate were determined. Respectively, the second level for melt temperature, i.e., 660°C; the third level for die temperature, i.e., 200/250°C; the third level for gas concentration, i.e., 0.30% vol; the third level for intensification pressure, i.e., 120 MPa; and the first level for gate velocity, i.e., 30 m.s⁻¹ are determined as the optimum process parameters. Effectiveness rank of the process parameters on work-piece quality is given in Table 4. The gate velocity is the most effective parameter on mechanical properties according to response factors of S/N ratios in Table 4. Besides, the conducted confirmation test results as seen in Table 5 prove that “the determined optimum process parameters give one of the highest average mechanical strength and the maximum density for the casting products”.

At the suggested optimum melting temperature, i.e., 660°C, that is 65°C higher than the liquids temperature of AZ91 alloy supplies both the required time for intensification pressure before surface solidification and the minimum gas porosity formation. Moreover, the optimum die temperature, i.e., 200/250°C, prevents shrinkage effectively. In order to prevent shrinkage, intensification pressure at the 3rd phase is performed until the surface solidification starts. In general, the surface solidification is completed between 250 and 750 ms depending on size of Mg cast part and die surface temperature. Die cavity must be filled with molten metal by the plunger and then the intensification pressure should be completed at the required time. Hot die surface retards surface solidification of the part for a few milliseconds and provides an effective intensification impact. In addition, the optimum gas concentration, i.e., 0.30 %vol, decreases the oxidation of molten Mg alloy and supplies fluorine contented protective film on the melt surface. With the increase of gas concentration, the protective film would be created quickly and be more

Table 4 — Effectiveness rank of the process parameters (larger is better)

Level	Bath Temperature	Die Temperature	Gas Concentration	Intensification Pressure	Gate Velocity
1	9.380	9.400	9.400	9.396	9.415
2	9.412	9.393	9.389	9.388	9.399
3	9.403	9.401	9.405	9.411	9.380
Delta	0.032	0.009	0.016	0.022	0.035
Rank	2	5	4	3	1

Table 5 — The repeated confirmation test results with the optimum process parameters

Run	Bath temp. °C	Die temp. (°C)		Gas conc. %vol	Int. pres. MPa	Gate velocity m s ⁻¹	Density g cm ⁻³	Dia. mm	Elongation % (in 50 mm)	Yield strength MPa	Tensile strength MPa
		Cover half	Ejector half								
1	2	3	3	3	3	1	1.78	5.97	5.91	140.35	227.73
2	2	3	3	3	3	1	1.77	5.98	6.58	144.86	231.21
3	2	3	3	3	3	1	1.78	5.98	6.93	147.14	235.75
4	2	3	3	3	3	1	1.78	5.99	7.25	154.25	241.10
5	2	3	3	3	3	1	1.77	5.98	5.93	142.36	230.27
Avg	2	3	3	3	3	1	1.78	5.98	6.52	145.79	233.21

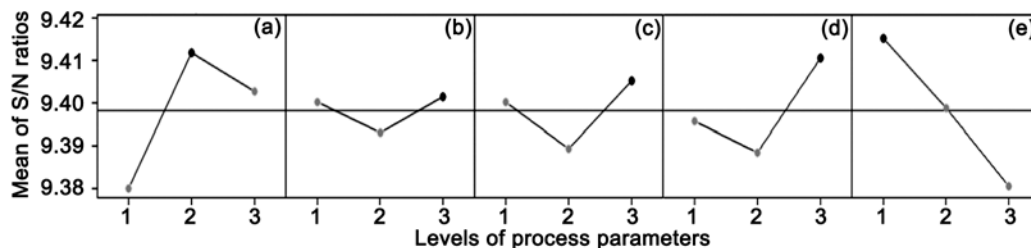


Fig. 4 — Main effect plots for S/N ratios indicating the optimum process parameter levels as the maximum points (a) bath temperature, (b) die temperature, (c) gas concentration, (d) intensification pressure and (e) gate velocity

stable. Under the created protective atmosphere conditions, minimum gas absorption of molten metal would be kept so that the porosity formation can be reduced as possible as it permits. Furthermore, 120 MPa intensification pressure is suggested as another optimum process parameter by the statistical software. Relatively high intensification pressure applied at the 3rd phase supplies a stronger material structure by preventing shrinkage porosity perfectly. Finally, 30 m.s⁻¹ is suggested as the optimum gate velocity by the program. To work with the low gate velocities helps to fill the cavity just before the beginning of the solidification. The suggested gate velocity by the program is the minimum value of our experimental levels. The lower gate velocities could evacuate the air from the die cavity and prevent product from porosity defects. The higher gate velocities could trap the air inside the cavity and cause macro gas porosity in the cast parts^{5,6,8,10}.

In this study, variation due to both the experimental factors and the possible errors were taken into consideration. The ANOVA was established based on the following considerations; the sum of the square (Seq SS), the degree of freedom (DF), and the adjusted mean square (Adj MS). Since the yield strength is one of the most important output for Mg alloy parts usage, ANOVA test has run on this factor. The results are summarized in Table 6. The data given in the table shows the contribution of the five factors; bath

temperatures, die temperatures, protective gas concentration, intensification pressure and gate velocity, respectively. Among these selected factors; gate velocity is the most effective factor on the yield strength.

In this study, all Brinell macro hardness values are measured between 63 and 70 HB. All process parameter combinations in our experiments are in line with macro hardness values in the literature^{22,23,28}. When the results are analyzed it is realized that, increasing of hardness values affect the strength of casting products positively. Tensile strength and Brinell hardness have a positive linear correlation empirically until app. 400 HB hardness. The stronger microstructure causes the higher hardness values of work-pieces. So that, the mechanical properties of the casting parts get better. The optical microscope images of the two smallest (Run 5 and 9), the two medium (Run 18 and 20), and the two highest (Run 11 and 24) tensile strength valued cast samples are given in Fig. 5. Aluminium intensive β -Mg₁₇Al₁₂

Table 6 — ANOVA test results for yield strength

Source	DF	Seq. SS	Adj. MS
Regression	5	464.55	92.91
Bath Temperature	1	177.30	177.30
Die Temperature	1	95.58	95.58
Gas Concentration	1	0.29	0.29
Intensification Pressure	1	0.70	0.70
Gate velocity	1	190.68	190.684

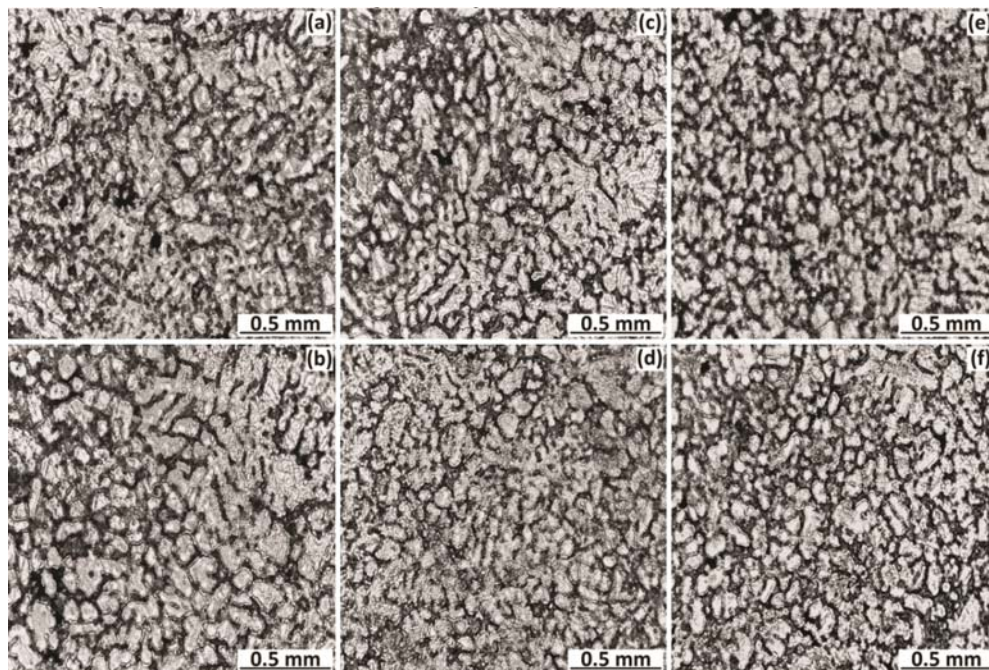


Fig. 5 — Optical microscope images of the die cast parts (500X) (a) Run 5, (b) Run 9, (c) Run 18, (d) Run 20, (e) Run 11 and (f) Run 24

intermetallic phases can be seen very clearly as the dark sections at the grain boundaries. Primary and secondary α -Mg phases can also be seen clearly as the blond sections. The grain size measurement results are; 214 μm for Run 5 and Run 9, 163 μm for Run 18 and Run 20 and 138 μm for Run 11 and Run 24 in average. It means that, grains of Runs 11 and 24 get 35.5% finer than the Runs 5 and 9. Repeated grain size measurements taken from optical microscope images prove that the smaller grain sizes affect the strength of the casting parts positively²⁹⁻³⁴.

The SEM images of the fractured surface of the two smallest (Run 5 and 9), the two medium (Run 18 and 20), and the two highest (Run 11 and 24) tensile strength valued test samples are given in Fig. 6. When the SEM images of tensile bar's fracture surfaces are analyzed, the typical inter-crystalline semi-ductile fracture mechanism of die cast Mg alloy parts can be seen clearly. The observed fibrous-cavity microstructures verify that the material has been plastically deformed before the fracture. The fracture occurred by pulling out of the α -Mg phases from the β - $\text{Mg}_{17}\text{Al}_{12}$ intermetallic phases³²⁻³⁴. The obtained results show that, optimum process parameters which reinforce the interface between α -Mg phase and β - $\text{Mg}_{17}\text{Al}_{12}$ intermetallic phase by refining of α -Mg grains, have improved the mechanical properties of the casting parts^{11,32,33}. In other words, strength increase is positively impacted by refining of the grain structure. Moreover, working with low gate velocities and high concentration of the protective gas provide favorable effects on the mechanical properties and the final product quality by reducing the

formation of the gas porosity^{5,7,8,15}. High intensification pressure values have a direct impact on the reduction of shrinkage originated porosity. In addition, increasing die temperature as much as possible retards the molten metal solidification on die surfaces and increase the duration of the effect of applied intensification pressure at the 3rd phase. At the optimum value of the melt temperature, the desired melt fluidity and the minimum gas absorption of liquid metal were ensured. Besides, the stronger interface decreases the tendency of α -Mg phase pulling out from β -intermetallic phase. As a result, the fracture mechanism works on the brittle β -intermetallic phase through the grain boundaries of the material^{5,8,11,12}.

Although process parameters are not effective on phase formations of the cast parts, they have an intensive impact on interfacial interactions of them. Both α -Mg and intermetallic β - $\text{Mg}_{17}\text{Al}_{12}$ phases existences of the two smallest (Runs 5 and 9), the two medium (Run 18 and 20), and the two highest (Runs 11 and 24) tensile strength valued casting parts are determined by XRD analysis clearly in Fig. 7.

The chemical compositions of the fracture surfaces of the two smallest (Runs 5 and 9), the two medium (Run 18 and 20), and the two highest (Runs 11 and 24) tensile strength parts are determined by EDS analysis. In Table 7, the percentages of Mg, Al, Zn and Mn components are given in wt%. The most remarkable part of this investigation is the higher Al amount of the fractured surfaces of the samples than the general Al amount of the material. It indicates that, the fracture occurs at Al intensive β - $\text{Mg}_{17}\text{Al}_{12}$

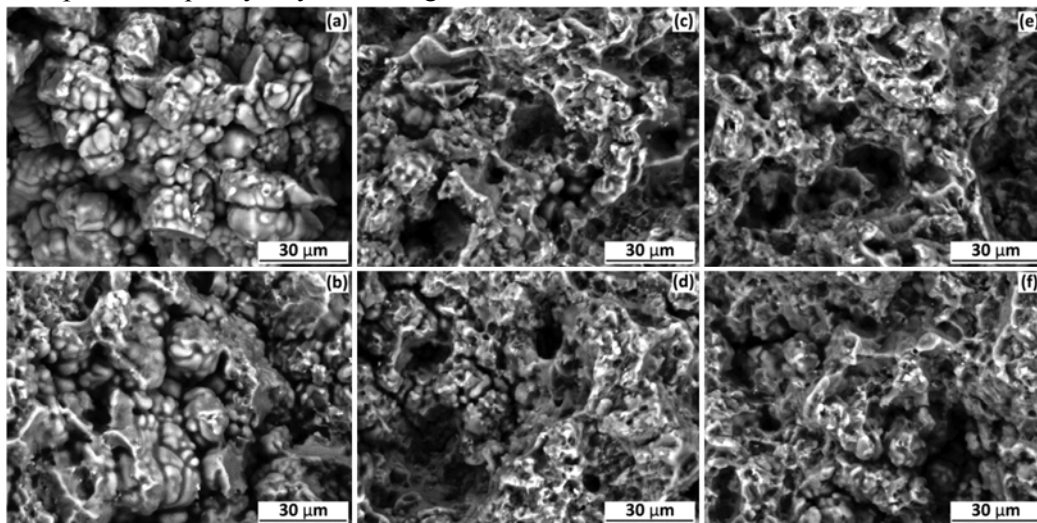


Fig. 6 — SEM images of the fractured surface of the die casting parts (3000X) (a) Run 5, (b) Run 9, (c) Run 18, (d) Run 20, (e) Run 11 and (f) Run 24

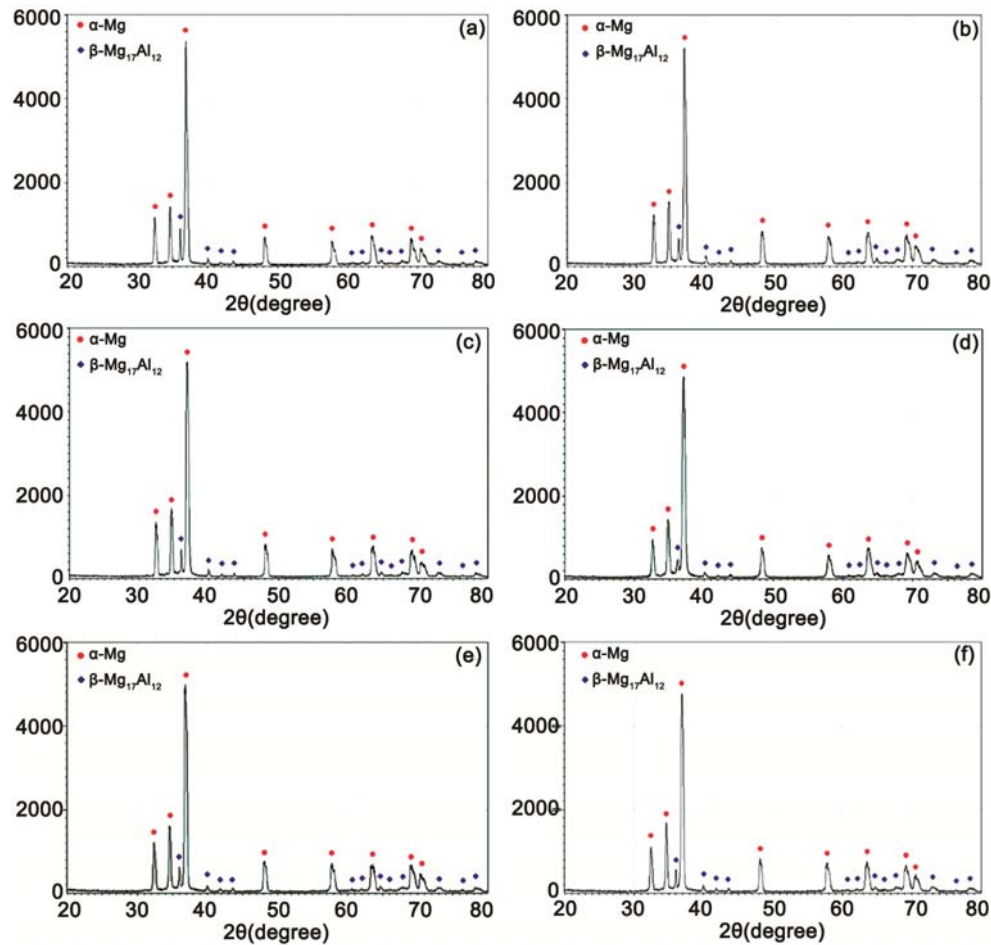


Fig. 7 — XRD graphical analysis of the die casting parts Run 5, (b) Run 9, (c) Run 18, (d) Run 20, (e) Run 11 and (f) Run 24

Table 7 — EDS tests results of the die cast parts

Run	Mg wt%	Al wt%	Zn wt%	Mn wt%	Fe wt%	Cu wt%	Ni wt%	Cr wt%
5	87.0	11.8	0.8	0.3	0.0	0.0	0.0	0.0
9	87.5	11.4	0.9	0.3	0.0	0.0	0.0	0.0
18	83.8	15.3	0.6	0.2	0.0	0.0	0.0	0.0
20	84.2	14.5	1.1	0.3	0.0	0.0	0.0	0.0
11	80.4	18.1	1.0	0.3	0.0	0.0	0.0	0.0
24	81.5	17.0	1.1	0.4	0.0	0.0	0.0	0.0

intermetallic phases at the grain boundaries. β -phase is more brittle than α -Mg phase. It thoroughly explains why the fracture cracks take place throughout the grain boundaries^{11,34}.

Conclusions

Low mechanical and metallurgical performances of the cast parts could be experienced due to the defects in HPDC of Mg alloy parts under heavy working conditions. Process technologies should be modified and the process parameters are needed to be

optimized for the make use of Mg alloys as high performance casting products. In this study, different process parameters including bath and die temperatures, protective gas concentration, intensification pressure and gate velocity, which are effective on the formation of physical, geometrical, mechanical and metallurgical properties of the casting products are evaluated by Taguchi experimental procedure. The optimum process parameters were determined by means of product quality. The obtained test results show that, the optimum parameters are 660°C for bath temperature; 200/250°C for die temperature; 0.30 vol% for protective gas concentration; 120 MPa for intensification pressure and 30 m.s⁻¹ for gate velocity by resulting minimum porosity with high mechanical strength and density. The AZ91 alloy samples manufactured with the optimum process parameters represent ± 0.04 mm dimensional tolerances, 66 HB hardness, 1.78 g.cm⁻³ density and less than 2% porosity values in average

and maximum 157 MPa yield and 248 MPa ultimate tensile strengths with maximum 7.67% elongation value in 50 mm. Additionally, gate velocity is determined as the most effective process parameter on the product quality.

All process parameters that increase the interface between α -Mg phase and β -Mg₁₇Al₁₂ intermetallic phase by refining α -Mg grains have improved the mechanical properties of the cast parts. Strength increase is positively affected by refining of the grain structure. In addition, reduction of the gate velocity decreases the total amount of porosity significantly. Working with low gate velocities and high concentration of the protective gas provides favorable effects both on the mechanical properties and the final product quality by reducing the formation of the porosity. Application of high intensification pressure decreases the total amount of porosity by reducing the shrinkage porosity significantly. Furthermore, an increase of die temperature delays the molten metal solidification on the die cavity surfaces. Besides, it increases the effect and duration of the applied intensification pressure at the 3rd phase. The required levels of the melt fluidity and the minimum levels of the gas absorption were ensured at the optimum values of the bath temperature. A decrease in the bath temperature also reduces the total amount of porosity via reduction in the gas porosity. The results of this study are very valuable and comparable to the data in the literature and very promising for future works of manufacturing high quality Mg alloy products in HPDC industry.

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