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Process Optimization and Surface Modification of Die Casting AZ91 Magnesium Alloy Products

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This work includes optimization of the AZ91 magnesium alloy cold chamber die casting process by experiments conducted according to the Taguchi experimental technique, to find the effective process parameters which determine the mechanical and metallurgical properties of the casting products. Optimum process parameters determined according to the test results 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 ms^{-1} for gate velocity. These parameters result in minimum porosity formation which provides high mechanical strength and density and good metallurgical properties. AZ91 alloy samples manufactured with the optimum process parameters show dimensional tolerances of ±0.04 mm, density of 1.78 gcm^{-3}, less than 2% porosity in average, maximum yield strength of 157 MPa and ultimate tensile strength of 248 MPa and maximum elongation of 7.67% in a 50 mm sample. Finally, surface modification of Mg die casting parts is crucial for meeting the long term usage requirements of the consumers. The electroless/electro nickel-plating with appropriate baths and pre-treatment processes were applied for providing fine surface conditions. The most effective process and modification parameters were analyzed in this study by considering the product quality.

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1. Introduction

Magnesium (Mg) is the lightest of all construction metals [1]. Mg alloys have been used and preferred in many sectors, especially in automotive, aerospace and electronic industries due to their suitable characteristics like lightness and strength [2]. Selection of the proper casting process for a specific part is determined based on designed shape, desired mechanical and surface properties, the number of parts to be produced and castability of the alloy.

Die casting of Mg alloys has shown the fastest growth and became the most globally developed section in magnesium industry. Die casting of complex shape Mg alloy products have increased considerably in recent years. Manufacturing of the Mg alloy parts with die casting process has a huge economic benefit, which cannot be presented by any other production system [1, 2].

Process parameters are the most important factors which are affecting the casting quality, product strength and defect formation. The correct selection of the process parameters means correct manufacturing of the casting product. The process parameters are bath and die temperatures, filling time of the die, plunger and gate velocities, intensification pressure, and the protective atmosphere concentration.

There is a wide range of suggested process parameters for die casting of different Mg alloys in the literature [3–6]. This paper specifies optimum process parameters for better mechanical and metallurgical properties of the casting parts, obtained 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 line, considering the product quality.

After manufacturing of the Mg alloy samples with optimum parameters, dimensional and geometrical tolerances are controlled, then surfaces are plated. It is important to increase the corrosion and wear resistances of Mg alloys via surface treatments. Plating of Mg alloy parts is not widely used yet in the industry. This is why application of surface treatments and plating technologies are in the focus of the research in the present time [7–10].

Among surface treatment technologies, electro/electroless plating with nickel coatings are especially popular due to their good conductivity, electromagnetic shielding, corrosion and wear resistance. Mg alloys are hard to plate because they exhibit electrochemically high activation to oxygen in the air. Thus, a suitable pre-treatment is critical for the quality of the coatings [9, 10].

With this work, the electroless Ni-plating is applied the surface of AZ91 alloy samples considering appropriate bath condition and pre-treatment process. Electroless Ni coating was successfully achieved with the preferred optimum modification conditions. In order to have good surface coating quality, the detailed surface polishing and pre-treatment processes are needed.

2. Experimental work

Size of the required die casting machine is basically determined by the shot volume and the projection area of the casting product including the designed gating sys-

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tem, considering the maximum pressure during the intensification phase. METAL PRES, MP100 type, Turkey, cold chamber die casting machine is preferred for production line. Many types of co-equipment are used in the melting process of Mg alloys. The employed melting resistance furnace unit is a stable crucible type electrical resistance furnace, MELTEC GmbH, MDF-200C, Austria. It is integrated into the mass production line for the experiments.

The protection of molten Mg alloy with the protective gases (fluxless method) is preferred for the casting procedure. The employed protection gas mixing unit PGM-3000 and dosing channel 1400-V3 type were produced by MELTEC GmbH, Austria. One of the most effective protective atmospheres in die casting process of the magnesium alloys is the mixture of fluorinated gases. This is why N_2+SF_6 gas mixture would be a proper solution to protect the magnesium melt from the oxygen of the air during manufacturing [11, 12].

The schematic drawing of the components of the integrated system is shown in Fig. 1. The geometrical and mathematical design of the runner and sprue were calculated for four cavities. The cast parts were manufactured as the standardized cylindrical tensile test samples according to ASTM B557M-15 [13]. Die halves were made of EN X40CrMoV5-1 hot work tool steel and hardened 48 HRC. The die heating and cooling device, ISITAN, CH210-S type, Turkey, was used to achieve a homogeneous temperature distribution of the fixed and the movable die halves.

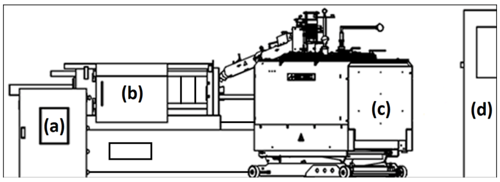

Fig. 1. Schematic drawing of the components of integrated system: (a) control unit, (b) die casting machine, (c) melting furnace, (d) gas mixing unit.

Since the process parameters are the most effective factors for product quality, they were optimized by using experimental results. Taguchi experimental design method was used in this study, by which the inherent variability of materials and manufacturing processes has been taken into account at the design stage. Taguchi L27 orthogonal matrix has been used to determine the optimum values of the process parameters which determine the product quality and defects occurrence in the experimental stage. The details of the obtained test results can be seen in the previous work of the authors [1].

The investigated experimental factors considered as the process parameters are bath temperature, die temperature, protective gas concentration, intensification pressure and gate velocity. In addition, the obtained test results, used as the response factors are density, diameter, elongation, yield and tensile strengths. Taguchi experimental design has been analyzed considering these factors related to signal/noise (S/N) ratio for "larger is better" approach using Minitab 16.1.1 statistical software. 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 using Shimadzu, AG-X type, 100 kN, Japan tensile test machine, at 4 mm$\,$min^{-1} tensile velocity, according to ASTM B557M-15 [13]. 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. Finally, scanning electron microscopy (SEM) examinations were done at the fractured surfaces of the tensile bars using Hitachi, TM1000 type, Japan, scanning electron microscope with an acceleration voltage of 15.0 keV, emission current of 140 mA, 30 s for plasma activation and 6.5 mm in working length.

After manufacturing the samples with the optimal parameters, dual Ni-plating process was applied. The coating helps to increase corrosion resistance of the products. Metallic coating process is applied to a conductive surface, however the MgO layer formed on the magnesium surface has insulating properties. Therefore, pre-treatment process is needed to remove the insulating layer. The applied pre-treatment and coating procedure steps are given in Table I. After electroless nickel coating, the surface of magnesium alloy can be easily coated with suitable metallic materials by electro plating [7, 8].

In the electroless procedure, an unpigmented polypropylene tank was used in the experiments. Tank was filled with 50 dm^3 of deionized water and then 40 dm^3 (equal to 43.6 kg) HESSONIC EN-MG1 part A/KN 301921 (Dr. HESSE GmbH, Germany) were added. Then the tank was filled-up with the deionized water up to the final volume of 100 dm^3. The bath was filtrated and heated up to the initial working temperature of 80 °C. The pH value ranged from 6 to 6.2 and the samples were rinsed in deionized water between each treatment steps [14].

Dimensional parameters of the products were measured and analyzed by conventional measurement techniques. Surface texture plays an important role in determining how a work piece or product will interact with its environment in usage. Rough surfaces usually wear more quickly than the smooth ones. Moreover, they have higher friction coefficients than smooth surfaces in tribological applications. Roughness often gives good predictions of the performance of a component or a workpiece, since irregularities in the surface may form cracks or corrosion or may promote adhesion. The major problems in usage start from minor defects on surfaces. Homogeneous coating needs to be provided and verified [8, 14].

3. Results and discussion

As the test results, average values of dimensional tolerances were ±0.04 mm, density was 1.78 gcm^{-3}, poros-

TABLE I

Pre-treatment and coating procedure steps of the electroless/electro nickel-plating.

Step	Solution	T [°C]	Time	Intention
Hot degreasing	50 g/l NaOH 10 g/l of Na_2HPO_4	80	10 min.	To clean the surface from oil and polishing waste
Pickling erosion	125 g/l CrO_3 110 ml/l HNO_3	25	45 s	To create pores on the surface to accumulate floride by pickling
Surface conditioning	500 ml/l HF	25	10 min.	To accumulate fluoride in the pores formed in the previous step
Electroless plating	10 g/l $NiCO_3$ 5 g/l $C_6H_8O_7$ 7.5 g/l F_2H_5N 10 ml/l HF 20 g/l NO_2P	80	30 min.	To create on the surface nickel coating by electroless plating
Electro plating	55 g/l $NiCO_3$ 240 g/l of $NiSO_4(H_2O)_6$ 45 g/l H_3BO_3	55	15 min.	To create on the surface nickel coating by electro plating process

ity was less than 2%. The maximum yield strength was 157 MPa, ultimate tensile strength was 248 MPa and the maximum elongation in 50 mm was 7.67%. The optimum process parameters have resulted from S/N ratio plots given in Fig. 2. 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. The optimum process parameters were determined as follows, 660 °C the second level for melt temperature; 200/250 °C the third level for die temperature; 0.30 vol.% the third level for gas concentration; 120 MPa the third level for intensification pressure; and 30 ms^{-1} the first level for gate velocity. The gate velocity is the most effective parameter influencing the mechanical properties, according to effectiveness rank given in Table II. Besides, the results of the conducted confirmation test prove that the determined optimum process parameters give the maximum mechanical strength and density of the casting product.

TABLE II

Response table for S/N ratios (larger is better).

Level	(a)	(b)	(c)	(d)	(e)
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

The SEM images of the two smallest (Run 5 and 9), the two medium (Run 18 and 20), and the two highest

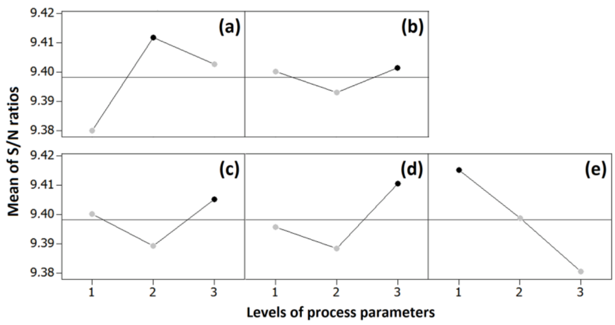

Fig. 2. Main effect plots for S/N ratios indicating the optimum process parameter levels as the maximum points for (a) bath temperature, (b) die temperature, (c) gas concentration, (d) intensification pressure, (e) gate velocity.

(Run 11 and 24) tensile strength valued work pieces are given in Fig. 3. When the SEM images of tensile bar 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 micro structures indicate that the material has been plastically deformed before the fracture. The fracture occurred by pulling out the α-Mg phases from the β-$Mg_{17}Al_{12}$ intermetallic phases.

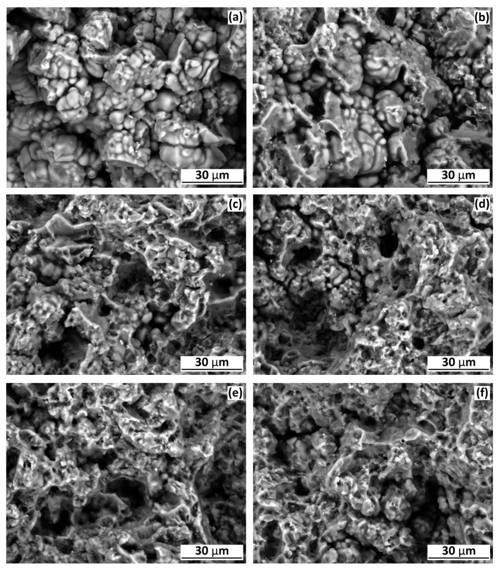

Fig. 3. SEM images of the fractured surface of the die casting parts (3000×) for (a) Run 5, (b) Run 9, (c) Run 18, (d) Run 20, (e) Run 11, (f) Run 24.

The obtained results show that optimum process parameters which reinforce the interface between α-Mg phase and β-$Mg_{17}Al_{12}$ intermetallic phase by refining of α-Mg grains, have improved the mechanical properties of the casting products [1, 2]. In other words, the increase in strength is positively impacted by the refining of the grain structure. Moreover, working with low gate velocities and high concentration of the protective gas has favorable effects on the mechanical properties and on the final product quality by reducing the formation of the gas porosity [3–6].

High intensification pressure values have a direct impact on the reduction of shrinkage originated porosity. In addition, increasing die temperature as much as possible delays the molten metal solidification on die surfaces and increases the duration of the effect of applied intensification pressure. 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.

Electroless/electro Ni-plating processes were applied to the surface of AZ91 alloy samples. An appropriate bath condition and pre-treatment process were chosen for the samples. Electroless/electro Ni coating was successfully achieved and bath formula is indicated here. The detailed surface polishing and pre-treatment procedures are necessary to achieve a good surface coating quality. Without any pre-treatment, cavities and black pitting appear on the coating surface [8]. The bath temperature should be at 80 °C and pH value at 6.1 for 30 min. to have a good coating quality in the electroless plating process. For these conditions, the thickness of nickel coating was found to be 10 μm in average.

The results show that, dense and uniform coatings can be deposited on the AZ91 substrate after hydrofluoric acid pickling pre-treatment and surface polishing. The density of coatings and the adhesion can be improved simultaneously by using diluted phosphoric acid as pickling solution. Thus, the coating provides a good protection to the substrate [7, 14]. During the surface measurements by the tactile instrument, Taylor Hobson, Form Talysurf Intra, UK, the related standards were considered. Surface measurements were made by the tactile instrument in the 1.0 mm range with 16 nm resolution. It is possible to analyze and monitor operations just with one crossing by using the software.

R_a which is the most used parameter for information about a surfaces in the literature is the arithmetical average surface roughness. R_t and R_z are the maximum height of the profile in the assessment length, R_v is the deepest profile height, R_p is the highest profile height and R_q is the root mean square of the profile. These parameters were measured repeatedly and standard deviations of them were calculated.

Other parameters such as R_{sk}, R_{ku} and $R_{mr(c)}$ were also considered [15, 16]. Arithmetical average of the values of un-coated surface roughness were obtained as 1.21 μm using Keyence, Optical Microscope, Japan, as seen in Fig. 4, since it is important to know the surface conditions before the coating. Coated surfaces were repeatedly measured in order to increase the measurement reliability after plating. Measurement results of the determined quality characteristics of the manufactured part are given in Table III.

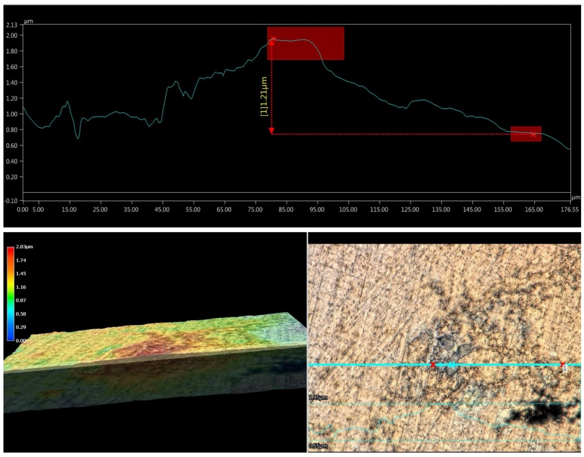

Fig. 4. Roughness profile (up) and an image (down) of the un-coated surface.

Figure 5 shows one of the coated surface profiles of the measured part, pointing an average surface profile type of the product. Valley intensive R_{sk} parameter is negative. Thus surface texture is very suitable for the next possible lacking operations after coating application. Unfortunately, positive R_{sk} results in the measured surfaces means that the surface will usually have high friction and will wear off quickly. In addition, R_{ku} results for the analyzed surfaces were determined to be higher than 3 at all measurements.

TABLE III

Results of the measurements of roughness of the coated surface.

Measurement	R_a [μm]	R_v [μm]	R_t [μm]	R_q [μm]	R_p [μm]	R_z [μm]	$R_{mr(c)}$ [%]
1st	0.2920	0.7732	3.8926	0.4260	0.4269	1.2002	59.98
2nd	0.2268	0.5993	2.5884	0.3225	0.3242	0.9236	61.24
3rd	0.2320	0.5667	4.2806	0.3438	0.3524	0.9192	59.02
4th	0.2626	0.6786	4.4464	0.4148	0.3996	1.0782	59.20
5th	0.2944	0.8023	3.3838	0.4119	0.4129	1.2153	61.24
Mean	0.2616	0.6840	3.7184	0.3838	0.3832	1.0673	60.14
Std.dev.	0.0320	0.1036	0.7525	0.0471	0.0432	0.1432	1.070

When the results are analyzed it can be said that the surface is well coated and a homogeneous structure was achieved. The importance of a detailed surface polishing and pre-treatment processes for acquiring a good surface coating quality has been demonstrated. Not only the determined arithmetical average of surface roughness of 0.2616 μm, but also the maximum surface peak value are acceptable for the usage of the product. As expected, R_q is found to be more sensitive to occasional valleys and peaks, making it a valuable complement for the R_a.

4. Conclusions

Although die casting is a precision manufacturing process, there are lots of factors affecting the product quality and the mechanical properties. In this study differ-

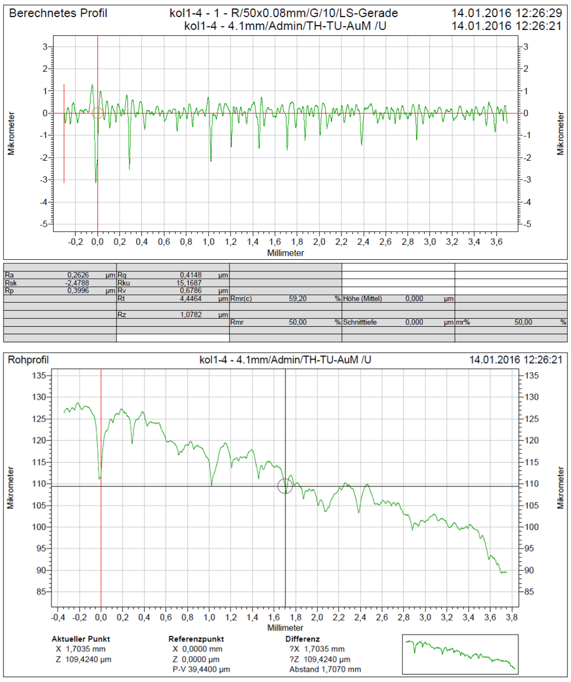

Fig. 5. Measurement parameters (up) and profiles (down) of the coated surface.

ent process parameters including bath and die temperatures, protective atmosphere concentration, intensification pressure and gate velocity, which affect the physical, geometrical, mechanical and metallurgical properties of the casting products were evaluated by Taguchi experimental procedure. The optimum process parameters were determined by considering the product quality. These 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 ms^{-1} for gate velocity. These parameters have resulted in minimum porosity formation, which leads to high mechanical strength, high density and good metallurgical properties. Additionally, gate velocity is determined as the most effective process parameter for the product quality. All process parameters that increase the interface between α-Mg phase and β-$Mg_{17}Al_{12}$ intermetallic phase, by refining the α-Mg grains, have improved the mechanical properties of the work pieces. Strength increase was positively affected by refining the grain structure.

Surface plating plays an important role in increasing the corrosion and wear resistances of Mg alloys. Among surface treatment technologies, electro/electroless nickel coatings are especially popular with their good conductivity, electromagnetic shielding, and corrosion and wear resistance properties. Mg alloys are hard to plate because they exhibit electrochemically high activation to oxygen in the air. Thus, a suitable pre-treatment is critical for the quality of the coatings. With this work, the electroless Ni-plating of AZ91 Mg alloy was applied by considering appropriate bath condition and the pre-treatment process. In order to have good surface coating qualities, the detailed surface polishing and pre-treatment processes needed to be applied. Roughness parameters have given good predictions of the fine visual view which may lead to high usage performance of the workpiece in the future.

After appropriate coating, silver and copper ionized lacking applications can be applied to the product surfaces to meet antibacterial demands in further works. Process technologies should be modified and process parameters need to be optimized to use Mg alloys in the high performance casting products. Results obtained in this study are very comparable to the data in literature and are very promising for future works in manufacturing of high quality Mg alloy parts in die casting industry.

Acknowledgments

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