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OPTIMIZATION OF POWDER METALLURGY PROCESS PARAMETERS TO RECYCLE AZ91 MAGNESIUM ALLOY

Naveen Kumar✉, **Ajaya Bharti**

Applied Mechanics Department, Motilal Nehru National Institute of Technology Allahabad, Prayagraj, India-211004

✉ chaudhary56naveen@gmail.com

Abstract. A global increase in the demand for magnesium and its alloys has put increased pressure on magnesium's natural sources (magnesium ores) due to its high strength-to-weight ratio and environment-friendly nature. Therefore, it has become necessary to look for efficient recycling techniques for magnesium and its alloys. Powder metallurgy proved to be very efficient compared to conventional recycling techniques for the recycling of aluminum, copper, and other alloys. In the present work, an experimental study was done to see the suitability of powder metallurgy for recycling magnesium and its alloy. Optimization of process parameters (compaction pressure, sintering temperature, and sintering time) was done with respect to AZ91 magnesium alloy's sintered density because almost all the properties (i.e., physical, mechanical, electrical, thermal, etc.) of a powder metallurgy product are dependent on the sintered density. Sintered density is dependent on the extent of diffusion and the thermal expansion in the material. And the extent of diffusion and the thermal expansion depends on the compaction pressure, sintering temperature, and sintering time. After experimental and statistical investigations, it was observed that compaction pressure is the most influencing parameter. The optimum set of process parameters was found out to be a combination of compaction pressure of 450MPa, the sintering temperature of 723K, and sintering time of 2h.

Keywords: ANOVA, AZ91 Mg Alloy, powder metallurgy, sintered density, Taguchi

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1. Introduction

Magnesium has the highest strength-to-weight ratio among the structural metals (i.e., aluminum, steel, copper, etc.) [1-3]. Also, magnesium is bioresorbable (can be degraded safely within the body) [4-6]. Due to these extraordinary properties, demand for magnesium and its alloys is increasing exponentially in almost all industries, i.e., automobile, aerospace, bio-medical, electronic devices, etc. [7-9]. With the increasing demand, there is high pressure on the natural sources of magnesium (extraction of magnesium from ores) [10,11]. Therefore, the efficient recycling of magnesium is necessary to reduce primary magnesium (extracted from ores) consumption. But, the conventional recycling technique's efficiency is very low because there is a heavy loss of material during melting, casting, and machining [12].

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Therefore, researchers around the world are working on the development of efficient magnesium recycling techniques.

Powder metallurgy proved to be very efficient for the recycling of aluminum and copper. Fogagnolo et al. recycled the aluminum alloy 6061 (AA6061) and AA6061/Al_2O_3 composite chips successfully through the powder metallurgy route [13]. High strength (being even higher than that of the primary composite material) was obtained for the recycled composite material. Aluminum chips were recycled directly by a powder metallurgy route by Samuel et al. also [14]. It was observed that material utilization is very high in the direct recycling of scraps compared to conventional recycling techniques. Fuziana et al. even recycled AA6061 chips directly by the powder metallurgy route, and Al_2O_3 was used as a reinforcement material [15]. The addition of alumina reduced the compressibility, but strength was increased. Canakci et al. also recycled the AA7075 chips through the powder metallurgy route with pure aluminum and SiC [16]. The addition of pure aluminum improved compressibility. Powder metallurgy was found to be a very efficient technique for recycling materials by Wan et al. also [12]. Magnesium and its alloys can also be recycled by powder metallurgy route, but magnesium's high reactivity and poor sinterability is the main issue. Ercetin et al. used the powder metallurgy hot press method to prepare Mg alloy and very high densification (more than 99%) was achieved [17-19]. Ozgun also used the powder metallurgy method and the problem of poor sinterability was minimized [20]. For efficient recycling of magnesium and its alloys by the powder metallurgy route, there is a need to optimize the powder metallurgy process parameters (i.e., compaction pressure, sintering temperature, and sintering time). In the present work, the optimization of powder metallurgy process parameters for the efficient recycling of AZ91 magnesium alloy is done using Taguchi and ANOVA techniques.

2. Experimentation

Material Used. Mg alloy AZ91 was used as a starting material. Jagada Industries, Virudhunagar supplied the coarse AZ91 magnesium alloy powder, prepared by grinding scrap. The size of the particles of non-uniform powder was less than 300µm.

Design of Experiments. Experiments were designed using the Taguchi Design of Experiment (DOE). There are multiple orthogonal arrays available in the Taguchi DOE. A standard orthogonal array can be selected based on the number of parameters and the number of levels. For the present work, three parameters (i.e., compaction pressure, sintering temperature, and sintering time), each with three levels, were chosen for the design of experiments. Table 1shows the parameters (along with the respective levels) selected for the design of experiments.

Table 1. Parameters selected for the design of experiments

Parameter	Level 1	Level 2	Level 3
Compaction Pressure (MPa)	350	400	450
Sintering Temperature (K)	673	723	773
Sintering Time (h)	1	1.5	2

For a full factorial design with three parameters, each with three levels, the number of experiments to be conducted will be twenty-seven. However, it is not easy to conduct twenty-seven different experiments because it requires a lot of energy, materials, and time. Therefore, by using partial factorial design (half factorial design, one-third factorial design, etc.), the number of experiments can be reduced. Taguchi DOE offers a one-third factorial design (L9 orthogonal array) for three parameters (each with three levels). Only nine experiments (in place of twenty-seven experiments) were required for the optimization process using a one-third factorial design. In this way, the consumption of resources and time can be reduced without significant loss in the necessary information gathered from conducting the experiments. Table 2 shows the designed sets of parameters for conducting experiments.

Table 2. Designed sets of parameters with respective sample codes (L9 orthogonal array)

S. No.	Compaction Pressure (MPa)	Sintering Temperature (K)	Sintering Time (h)	Sample Code
1	350	673	1	A
2	350	723	1.5	B
3	350	773	2	C
4	400	673	1.5	D
5	400	723	2	E
6	400	773	1	F
7	450	673	2	G
8	450	723	1	H
9	450	773	1.5	I

Methodology. In the present work, the powder metallurgy technique was used for recycling AZ91 magnesium alloy. In the powder metallurgy technique, powders are compacted at a specified pressure to produce green compacts [21,22]. Then the green compacts are sintered to make the final products [23-26].

Powder Compaction. Powders were compacted at three different compaction pressure (350MPa, 400MPa, and 450MPa) in a split powder compaction die. A cylindrical mold with an internal diameter of 20mm was used, producing cylindrical green compacts [27]. Powder compaction was done on the Universal Testing Machine (UTM) with a constant strain rate.

Sintering. Green compacts obtained after powder compaction were sintered in a muffle furnace. Sintering was done in a nitrogen gas environment to prevent the oxidation and auto-combustion of samples. After holding the samples at a specified temperature for a specified time, samples were furnace-cooled.

Characterization, Testing, and Statistical Analysis

X-ray Diffraction. X-ray diffraction (XRD) of the purchased powder was done to know the various phases present in it. As the compressibility and sinterability of a material depend on the oxides and different phases present in the material, oxides and other hard phases reduce the compressibility and sinterability of a material.

Green Density. Archimedes' principle was used for measuring the density of the green compacts produced after powder compaction. Green density was evaluated to investigate the effect of compaction pressure on the densification of the material.

Sintered Density. Archimedes' principle was used for measuring the density of samples after sintering. Sintered density has a high significance for a powder metallurgy product because almost all the properties (i.e., physical, mechanical, electrical, thermal, etc.) of a powder metallurgy product are dependent on the sintered density. Sintered density is dependent on the extent of diffusion and the thermal expansion in the material. And the extent of diffusion and the thermal expansion depends on the compaction pressure, sintering temperature, and sintering time. Therefore, by optimizing the sintered density, powder metallurgy process parameters (i.e., compaction pressure, sintering temperature, and sintering time) can be optimized.

Statistical Analysis. Statistical analysis was done using Taguchi and Analysis of Variance (ANOVA). In Taguchi's analysis of design, variation around the optimum value or target value of a performance characteristic is reduced by optimizing the process parameters [28]. The optimum level of process parameters can be found out by analyzing the main effect plots. Quantitative analysis of the effect of various process parameters can be done by the ANOVA technique. The percentage effect of different process parameters can be found out by analyzing the ANOVA table. Performance characteristic always belongs to one of the three categories:

The bigger, the better (Target value = ∞);
The smaller, the better (Target value = 0);
The nominal values are better (Target value = fixed).

In Taguchi's design analysis, the signal-to-noise ratio (SN ratio) is the most important statistical relation. The SN ratio's high value implies the high value of the signal and the low value of noise or variation. As the optimization of the parameter is based on reducing the variability around the target value, the SN ratio's high value is desirable. Therefore, the set of parameters that yield the highest value of the SN ratio is the local-optimum set of parameters (from the sets of parameters available in the experiment's present design). Global-optimum set of parameters (from all possible sets of parameters) may not be present in the present experiment design. Global-optimum set of parameters can be found out from main effect plots.

For "the bigger, the better" characteristic:

$$\frac{\mathrm{S}}{\mathrm{N}} = 10\log\left[\frac{1}{\mathrm{n}}\sum_{\mathrm{n}=1}^{\mathrm{n}}\frac{1}{\mathrm{y}^2}\right].$$

For "the smaller, the better" characteristic:

$$\frac{\mathrm{S}}{\mathrm{N}} = -10\log\left[\frac{1}{\mathrm{n}}\sum_{\mathrm{n}=1}^{\mathrm{n}}\frac{1}{\mathrm{y}^2}\right].$$

For "the nominal values are better" characteristic:

$$\frac{\mathrm{S}}{\mathrm{N}} = -10\log\left(\overline{\mathrm{y}^2}/\mathrm{S}\right),$$

where n is the number of repeated experimental tests, y is the performance value, and S is the target value.

3. Results and Discussion

XRD. Figure 1 shows the indexed XRD pattern of purchased powder. It can be seen from the figure that various Mg-Al phases, along with oxides, were present in the material due to its high reactivity. $Al_{12}Mg_{17}$ is a hard phase of aluminum and magnesium that reduces the compressibility of AZ91 magnesium alloy. Various oxides phases (i.e., MgO and $Mg_{0.36}Al_{2.44}O_4$) present in the material reduce the sinterability due to the high thermal resistance of metallic oxides.

Green Density. The green density vs. compaction pressure graph of AZ91 magnesium alloy is shown in Fig. 2. It can be seen from the figure that the green density increased sharply with an increase in the compaction pressure. An increase in the green density can be attributed to the forced rearrangement of particles. The maximum average green density (1.527±0.02 g/cm3) was obtained for the samples compacted. Around 8% increase in green density was observed on increasing the compaction pressure from 350MPa to 550 MPa. The porosity of material (the samples compressed at 450MPa) was around 15-16 % (with respect to the as-cast AZ91 magnesium alloy) density. High porosity can be attributed to the poor compressibility of AZ91 magnesium alloy due to hard phases and oxides.

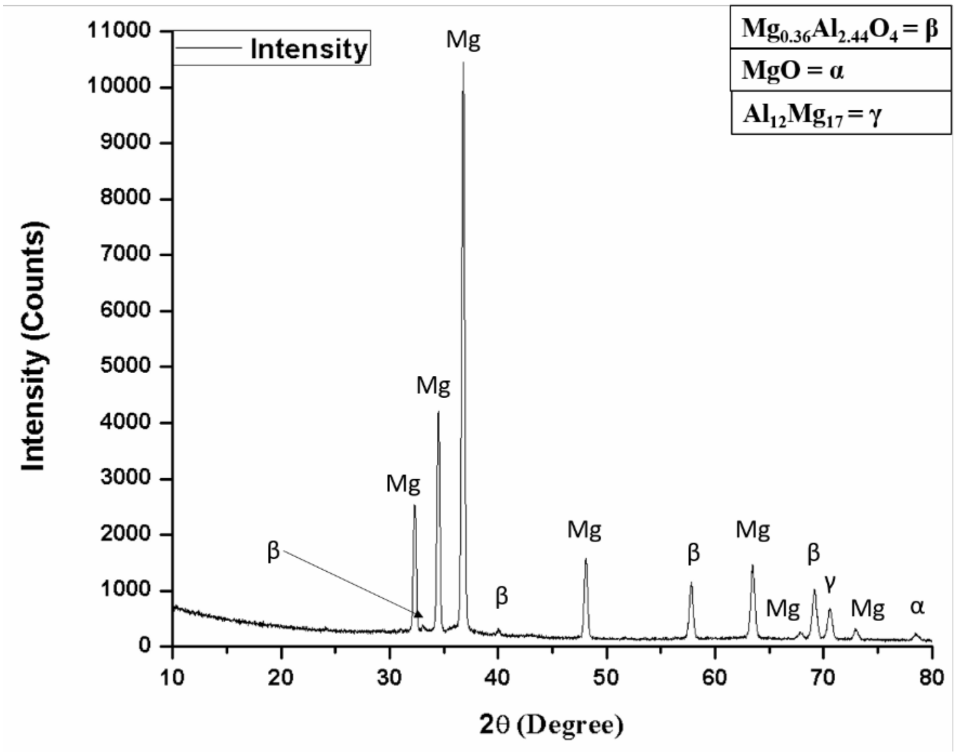

Fig. 1. Indexed XRD pattern of purchased AZ91 magnesium alloy

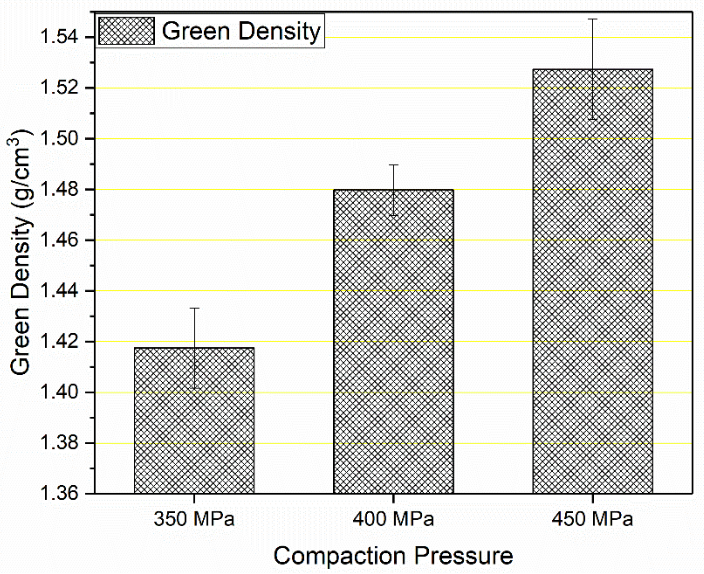

Fig. 2. Effect of compaction pressure on the green density of AZ91 magnesium alloy

Sintered Density. The sintered density of samples is shown in Fig. 3. It can be seen in the figure that the maximum sintered density (1.619 g/cm^3) was obtained for sample H (450MPa, 723K, 1h) while the minimum sintered density (1.496 g/cm^3) was obtained for sample A (350MPa, 673K, 1h). It shows that the density of samples increased after sintering. An increase in density after sintering can be attributed to samples' contraction due to inward diffusion of material during sintering. Minimum porosity in the sintered material (sample H) was around 10% (with respect to the as-cast AZ91 magnesium alloy) density. It shows that the porosity in the material reduced after sintering. But, the porosity in the material is still high. Poor compressibility and sinterability due to hard phases and oxides can be the reason for high porosity in the material.

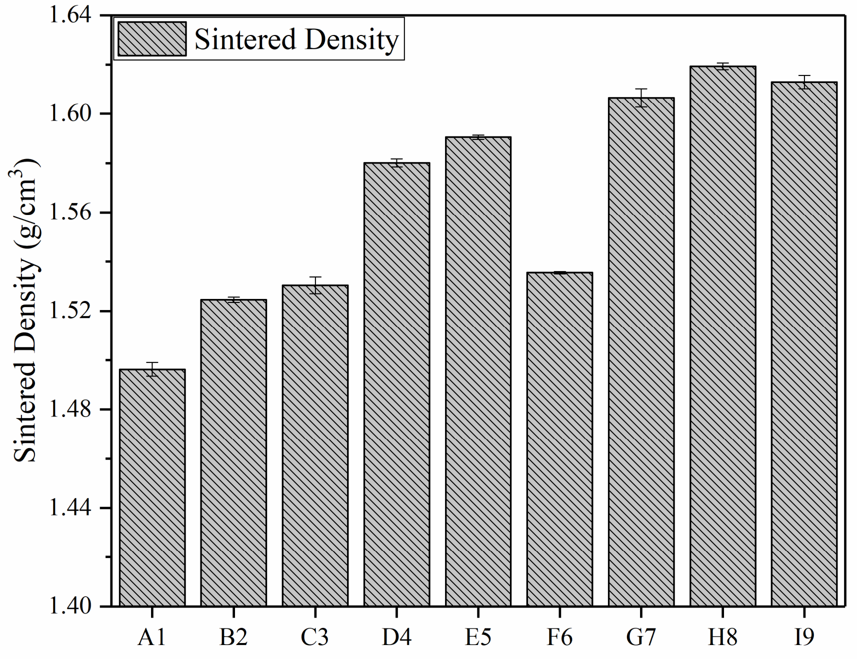

Fig. 3. Sintered density of AZ91 magnesium alloy samples

Statistical Results. Statistical results obtained during the Taguchi analysis of design with respect to the sintered density are given in Table 3. It can be observed from the table that the S/N ratio is maximum for sample H. It shows that a set of parameters (450MPa, 723K, 1h) used to fabricate sample H is the best set of parameters (from the present nine-sets of parameters.). But it is not sure that the optimum set parameters are available in the present nine-sets of parameters. An optimum set of parameters can be among the remaining eighteen-sets of parameters (left-out during the design of experiments). The optimum set of parameters can be found out by analyzing main effect plots.

Table 3. Statistical Results

S. No.	Production Parameters			Sintered Density (g/cm^3)	SN Ratio (Sintered Density)
	Compaction Pressure (MPa)	Sintering Temperature (K)	Sintering Time (h)		
1	350	673	1	1.496	3.500041
2	350	723	1.5	1.525	3.662863
3	350	773	2	1.530	3.696114
4	400	673	1.5	1.580	3.973635
5	400	723	2	1.591	4.030833
6	400	773	1	1.536	3.725178
7	450	673	2	1.607	4.117669
8	450	723	1	1.619	4.186453
9	450	773	1.5	1.613	4.152586

The main effect plots for sintered density are shown in Fig. 4. The main effect plot of compaction pressure indicates that the compaction pressure positively affects the sintered density of samples. Sintered density increases with an increase in the compaction pressure. Steep rise shows that the compaction pressure has a high effect on the sintered density. Maximum density is obtained for samples compacted at 450MPa, Therefore the optimum compaction pressure is 450MPa. The main effect plot of sintering temperature shows that the sintering temperature has a mixed effect on the sintered density. Initially, sintered increased with an increase in the sintering temperature due to the increased rate of inward diffusion at high temperatures. But, on further increasing the sintering temperature, sintered density decreased due to the dominance of thermal expansion over the diffusion phenomena. Therefore, the second level of sintering temperature (723K) is the optimum sintering temperature. The main effect plot for sintering time shows that the sintered density initially increased sharply on increasing the sintering time. But on further increasing the sintering time, there was not much effect on the sintered density due to the thermal expansion. Maximum sintered density was observed at a sintering time of 2h. Therefore, the optimum sintering time is 2h. Hence, the optimum set of parameters (450MPa, 723K, 2h) is not present in the parameters' present nine-sets.

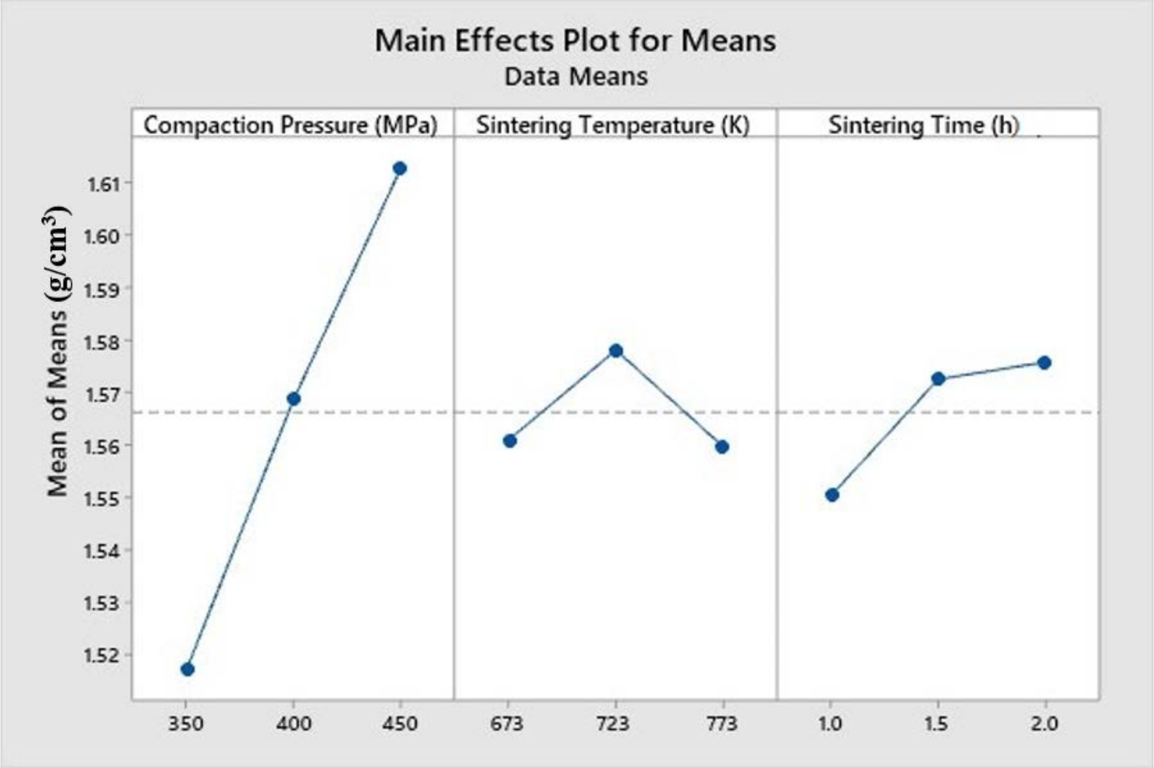

Fig. 4. Main effect plots for sintered density (means)

Response data for means (sintered density) is shown in Table 4. The ranking of parameters done based on the change in the mean sintered density (delta) is given in the response table for means. It can be seen in the table that the compaction pressure is ranked first, followed by sintering time and sintering temperature, respectively. It shows that the compaction pressure is the most influencing parameter, while sintering temperature is the least influencing parameter.

Table 4. Response data for means (Density)

Level	**Compaction Pressure (MPa)**	**Sintering Temperature (K)**	**Sintering Time (h)**
1	1.517	1.561	1.550
2	1.569	1.578	1.573
3	1.613	1.560	1.576
Delta	0.096	0.018	0.025
Rank	1	3	2

Analysis of variance (ANOVA) data is given in Table 5. The high value of F and the low value of P signifies the high effect of compaction pressure on the sintered density. Therefore, the ANOVA results confirm the results obtained during the Taguchi analysis of design (response table for means).

Table 5. Analysis of variance data for sintered density

Source	**DF**	**Adj SS**	**Adj MS**	**F-Value**	**P-Value**
Compaction Pressure (MPa)	2	0.013811	0.006905	20.74	0.046
Sintering Temperature (K)	2	0.000638	0.000319	0.96	0.511
Sintering Time (h)	2	0.001151	0.000576	1.73	0.366
Error	2	0.000666	0.000333		
Total	8	0.016266			

4. Experimental Validation

During statistical analysis using Taguchi analysis of design and ANOVA, it was observed that the optimal set of parameters (at which the sintered density of recycled AZ91 Mg alloy will be maximum) was not available in the current design of the experiment. The optimal set of the parameter is among the remaining 18 sets of parameters. The optimal set of parameters was obtained by analyzing mean effect plots. AZ91 Mg alloy scrap powders were recycled at the obtained optimal set of parameters to validate the statistical results experimentally. AZ91 Mg alloy scrap powders were compacted to a pressure of 450 MPa to produce the green compacts. Obtained green compacts were sintered at 723K temperature for 2h and the furnace cooled to room temperature. Sintered density of prepared samples was calculated. It was observed that the average sintered density (1.624 g/cm^3) of the sample prepared at the optimum set of parameters was higher than the sintered density of sample H (450MPa, 723K, 1h.). Therefore, the experimental results are in good agreement with the statistical results and the final optimal set of parameters for recycling of AZ91 Mg alloy is (450MPa, 723K, 2hr.).

5. Conclusions

Following conclusions are drawn after testing and analyzing conducted experiments:

- Scrap AZ91 magnesium alloy has been recycled successfully by the powder metallurgy route.
- The presence of hard phases ($Al_{12}Mg_{17}$) and oxides (MgO and $Mg_{0.36}Al_{2.44}O_4$) reduced the compressibility and the sinterability.
- The maximum value of green density and sintered density obtained was 1.527 g/cm^3 and 1.619 g/cm^3, respectively.
- Compaction pressure was found out to be the most influencing parameter, followed by sintering time in sintering temperature, respectively.
- The optimum value of compaction pressure was found out to be 450MPa.
- The optimum value of the sintering temperature was 723K.
- The optimum value of sintering time was 2h.
- The optimum set of parameters (450MPa, 723K, 2h) is not present in the experiments' current design.
- The sintered density of the sample prepared at the optimum set of parameters (450MPa, 723K, 1h.) was 1.628 g/cm^3.

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THE AUTHORS

Kumar N.
e-mail: chaudhary56naveen@gmail.com
ORCID: 0000-0002-6918-4384

Bharti A.
e-mail: abharti@mnnit.ac.in
ORCID: 0000-0003-2809-9674