Effect of annealing temperature and cutting angle of specimens on metallurgical and mechanical properties of Mg-7Li-1Zn alloy via Taguchi approach and response surface analysis

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Abstract. The main aim of this research is to assess the effect of annealing temperature and cutting angle of specimens on the metallurgical and mechanical properties of superlight Mg-Li-Zn alloy thin sheets. To this end, as-rolled LZ71 (Mg-7%Li-1%Zn) alloy was annealed at different temperatures (0, 250, and 350°C) for 3 hours. After that, the test specimens were cut at different angles relative to the rolling direction (0, 45, and 90°). Next, several experiments were accomplished to study the microhardness, microstructure, grain size, and tensile behavior of the LZ71 alloy. Finally, different Design of Experiment (DOE) techniques were used to determine the individual and interaction effects of annealing temperature and cutting angle of specimens on the material characterization of LZ71 alloy. In this regard, annealing temperature and cutting angle were considered as input parameters in both Taguchi Approach (TA) and Response Surface Analysis (RSA). Also, key parameters of the tensile test including Ultimate Tensile Strength (UTS), Yield Stress (YS), Elastic Modulus (EM), and Strain at BreakPoint (SBP) were considered as outputs. It was concluded that annealing temperature influence on the static behavior of LZ71 alloy is several times greater than the effect of cutting angle (i.e., the effect of annealing temperature on the UTS, YS, EM, and SBP is 92%, 76%, 61%, and 66%, respectively). Moreover, the results of the Response Surface Optimization Algorithm (RSOA) showed that to achieve the best static properties in different directions, the annealing temperature should be selected to be 350°C.

Keywords: Magnesium-lithium alloy, Microstructure, Mechanical properties, Taguchi approach, Response surface analysis, Optimization.

1. Introduction
Magnesium alloy applications are experiencing a sharp rise in various industries such as automotive, aerospace, and electronics due to their mechanical behavior [1-3]. Moreover, these alloys are well known as the lightest structural alloys having a density of 1.8 gr/cm³ [4, 5]. Pure magnesium is one of the most abundant metals and has considerable properties namely: high durability, very high strength
to weight ratio, high recyclability, desirable heat loss, and electromagnetic shielding [6, 7]. Magnesium-Lithium composite is one of the most ideal designing metals because of its incredible properties like high formability and ultra-low density [2]. Amongst all magnesium alloys, Mg-Li compounds are considerably more interesting than others due to their elongation besides the obvious low density [8]. To reach higher yield stress, adding rare earth elements to the compound is an effective solution. However, Mg-Li with around 5 to 12 weight percentage of lithium has the best structural behavior because, with this proportion of lithium, the microstructure of the alloy will be dual-phase with α-Mg (Mg solid solution, hcp structure) and β-Li (Li solid solution, bcc structure) [9]. Studies on microstructures and mechanical properties of magnesium alloys have been done because of their unique behavior under different situations of casting, forming, and annealing [5, 10-12]. Tang et al. have investigated the influences of the annealing process on microstructural and mechanical properties of Mg-XLi-3Al-2Zn-0.2Y [11]. In this study, experimental results have been reported taking into account three different values for the X parameter (5, 8, and 11). In other words, the effect of the percentage of lithium element in the alloy has been analyzed on the materials’ microstructure and mechanical properties. The results indicated that the ultimate and yield stresses increase and the elongation at breakpoint decreases by raising the weight percentage of lithium. Moreover, the surface microhardness reduces by increasing the lithium value. Also, they have shown that the surface microhardness variations have a direct relationship with the temperature if the amount of lithium in the alloy was high enough. Zou et al. have improved the mechanical behavior of superlight Mg-Li-Al alloys (LA36, LA96, and LA156) by duplex phases and fine precipitates [12]. The experimental results (tensile, compression, XRD, SEM, and TEM images) have been compared to show that the mechanical behavior of different Mg alloys is better than the pure magnesium. In the other words, the alloying with Li to improve ductility and the Al addition to enhance strength are proofed. To improve the mechanical and microstructural characteristics of as-cast alloys, extrusion and rolling processes have been known as the most prevalent work hardening methods [13-20]. Meng et al. had a survey on mechanical properties of Mg–8Li–1Al–1Zn, Mg–8Li–1Zn, and Mg–8Li–1Al [9]. According to their study, the strength of Mg–8Li–1Al–1Zn and Mg–8Li–1Al alloys improved due to the forming process. Also, Yu et al. have studied the rolling process temperature effects on mechanical properties and microstructures of AZ910 alloy [21]. They reported that the cutting angle impact on the results of tensile tests of the fine-grained rolled AZT910 alloy sheets is practically insignificant during deformation at 300 °C. In other studies, the microstructural and textural evaluation of Mg–X%Li–1%Zn (X=6, 8, and 12) has been performed [22]. Recently, the study on the fatigue behavior and fracture mechanisms of magnesium alloys has been considered by many scholars [23-29]. Also, in the previous study, the authors reported the high-cycle fatigue curves of LZ71 magnesium alloy and compared it with fatigue properties of pure magnesium. Moreover, they studied the high-temperature tensile behavior of LZ71 magnesium alloy in the laboratory [30]. In the present paper, for the first time, the individual and interactive effects of annealing temperature and cutting angle of specimens on the material characterization of as-rolled LZ71 alloy sheets were investigated using TA and RSA. To this end, test specimens were provided in three different cutting angles relative to the rolling direction and various annealing temperatures. Afterward, Optical Microscopy (OM) observations, X-ray Diffraction (XRD) analysis, microhardness measurement, and tensile tests were carried out to study the metallurgical and mechanical properties of the material. Eventually, the most effective parameter was reported based on the TA results. And, the optimal values were extracted using RSOA.

2. Methodology
In the present study, the commercial pure Magnesium (Mg), Zinc (Zn), and Lithium (Li) were used to produce LZ71 alloy through casting at almost 770°C under a protective atmosphere of Argon gas. The chemical composition of the cast alloy was Mg-7(Wt%)Li-1(Wt%)Zn. The initial ingot was flat-rolled from an initial thickness of 10 mm to a final thickness of 2 mm (total thickness reduction of almost 80% including 8 passes by 10% reduction in each pass) at a rolling temperature of 300°C [30]. Then, the sheets were annealed at two different temperatures including 250 and 350°C for 3 hours. Next,
tensile specimens were fabricated from the sheets of annealed and as-rolled materials with a thickness of 2 mm according to ASTM E8M [31] standard. Specimens were provided in different directions including 0, 45, and 90 degrees. The angle zero means that the longitudinal directions of specimens are parallel to the direction of the rolling process. Eventually, Figure 1 shows the working flowchart to clarify the research methodology in this study.

Figure 1. The working flowchart used in this study

3. Experimental procedure

3.1. OM observations
Microstructures of the specimens were observed utilizing the Optical Microscope (OM) before and after the annealing process at different temperatures. The specimens have been etched using a solution of CH3COOH (2 ml), HNO3 (2 ml), H2C2O4 (2 gr), and water (310 ml).

3.2. Microhardness measurement
Microhardness measurements were accomplished utilizing a SHAAB M5 tester. The applied load was set to 50 gf and the time duration was 15 seconds using Vickers indenter. To evaluate the trace of indenter accurately, surfaces of the specimens were prepared carefully, and four random indents have been considered for each specimen. Vickers microhardness of each indent was calculated based on HV=1.854P/d². In this equation, HV is Vickers microhardness value with the unit of Kgf/mm², P is the applied force with the unit of Kgf, and d is the average length of the diagonal remain by the indenter in millimeters.

3.3. XRD analysis
To obtain the grain size of the material before and after applying the annealing process at different temperatures, XRD measurements were carried out. X’Pert PRO MPD (PANalytical) X-ray diffractometer system was employed with Cu Kα radiation (λ=1.54060 Å). Also, Scherer’s equation was employed to calculate the crystallite size [32-34].

3.4. Tensile test
The STM-250 SANTAM universal testing machine was used to carry out the tensile test. All tests were performed at the constant strain rate of 0.1 mm/mm/min by applying a short travel extensometer.
with an accuracy of 0.001 mm at room temperature. All environmental conditions were controlled during the test following the ISO-17025 [35] standard.

4. Statistical analysis
Two different DOE techniques including TA and RSA were used to evaluate the data collected from experiments. Also, each of these methods was used for a specific purpose. However, the same conceptual structure was considered for both methods (Figure 2).

![Figure 2. Conceptual structural of DOE techniques according to the considered input and output parameters](image)

Taguchi sensitivity analysis was performed to find the most effective parameter (annealing temperature or cutting angle) on the static properties of the LZ71 superlight alloy sheet. Among all DOE techniques, the Taguchi algorithm uses the least number of samples for sensitivity analysis [36-39]. Considering the number of input parameters and their levels, the L9 orthogonal matrix was used to perform Taguchi analysis. Moreover, the current research seeks to improve the static behavior of the material (increasing the UTS, YS, EM, and SBP). Therefore, the viewpoint of larger is better was considered to calculate the mean of signal-to-noise ratio [40]. Next, response surface analysis was done to investigate the interaction effects of parameters on the static behavior of the material. Eventually, the RSM-based optimization algorithm [41] was employed to determine the optimal conditions to achieve the best static properties in different directions.

5. Results and discussion

5.1. Experimental results

0 presents the OM images of annealed specimens of the Mg-7Li-1Zn alloy. On the OM images of the sample before the annealing process, two different phases including α-Mg (bright regions) and β-Li (dark regions) are identified. Also, it could be noticed that the volume fraction of the alpha phase increase by raising the temperature for the annealing process. When the material is as-received (annealing temperature of zero or without annealing treatment), the microstructure is fiber shaped rolling formation. Then, the material can also find partial recrystallization by increasing the annealing temperature to 250 °C. Finally, consider the temperature of 350 °C for annealing treatment caused to create complete recrystallization, forming a fine recrystallized grains, and fibrous microstructure completely dis-appeared. According to the published researches, the alloy has completed the recrystallization, if continues to increasing annealing temperature, some grains will appear irregular
growth and into re-crystallization grain coarsening and growth stage [29]. In other words, the microstructure finds a more homogeneous distribution, because static recrystallizations occurred at the annealing temperature of 350°C. Thus, mechanical properties for annealed samples at 350 °C are expected to be the same in all directions.

![Rolling direction](image)

**Figure 3.** OM images of the different heat-treated specimens of LZ71: (a) Without annealing process, (b) Annealing temperature of 250 °C, and (c) Annealing temperature of 350 °C

After microstructural characterization, distributions of surface microhardness of the treated specimen were measured in 4 different points. It is clear from the results of hardness measurements that the surface microhardness of the specimens decreases by raising the temperature of the annealing process. Average surface microhardness of 65.51 ± 1.82, 62.12 ± 1.73, and 52.34 ± 3.77 have been obtained for annealed specimens at temperatures of 0, 250, and 350°C, respectively. The crystallite size of the top surface layer of treated specimens calculated using XRD analysis (Figure 4) and Scherrer-Debye equation are presented in Table 1.

![XRD results](image)

**Figure 4.** XRD results of the annealed specimens at different temperatures
Table 1. Crystallite size of the treated specimens

| Sample No. | Treatment                | Temperature (°C) | Crystallite size (nm) |
|------------|--------------------------|------------------|-----------------------|
|            |                          |                  | hcp       | bcc       |
| 1          | No annealing process     | 0                | 70.13     | 89.94     |
| 2          | Annealing process        | 250              | 79.07     | 103.37    |
| 3          | Annealing process        | 350              | 94.32     | 104.77    |

The key parameters of stress-strain diagrams as a result of tensile tests including ultimate stress, yield stress, elastic modulus, and elongation at breakpoint are reported in Table 2. The yield stress was calculated based on the strain offset of 0.2%. All tensile tests were repeated 3 times and the average values of key parameters with standard deviation calculation are presented in this section.

Table 2. Key parameters of tensile testing results

| Annealing temperature | Sheet angle relative to the rolling process | Ultimate stress (MPa) | Yield stress (MPa) | Elastic modulus (GPa) | Strain (%) |
|-----------------------|---------------------------------------------|-----------------------|-------------------|-----------------------|------------|
| As-rolled             | 0                                           | 196.64 ± 1.22         | 119.81 ± 0.78     | 12.44 ± 0.93          | 49.2 ± 1.09|
|                       | 45                                          | 192.51 ± 1.22         | 122.97 ± 0.84     | 14.44 ± 1.83          | 42.4 ± 0.94|
|                       | 90                                          | 188.48 ± 1.19         | 110.71 ± 0.92     | 11.81 ± 1.98          | 33.46 ± 0.8|
| 250 °C                | 0                                           | 170.39 ± 1.08         | 96.33 ± 0.97      | 12.22 ± 0.78          | 54.8 ± 0.91|
|                       | 45                                          | 163.57 ± 1.09         | 110.11 ± 1.17     | 13.03 ± 0.84          | 48.9 ± 0.81|
|                       | 90                                          | 168.99 ± 1.07         | 93.26 ± 1.42      | 10.93 ± 0.93          | 42.4 ± 0.7|
| 350 °C                | 0                                           | 142.9 ± 0.9           | 87.7 ± 0.63       | 13.49 ± 0.47          | 61.4 ± 0.38|
|                       | 45                                          | 140.68 ± 0.99         | 90.5 ± 0.69       | 14.88 ± 0.52          | 61.1 ± 0.42|
|                       | 90                                          | 138.85 ± 0.89         | 92.18 ± 0.7       | 15.19 ± 0.53          | 62.7 ± 0.41|

It is clear from the tensile testing results that the values of ultimate stress, yield stress, and elastic modulus are approximately the same at different sheet angles from each group and there is a significant change in their elongation at the breakpoint. The maximum and minimum values of elongation are for sheet angles of 0 and 90 relative to the rolling process, respectively. Finally, the experimental results indicated that the static properties of the sheet become homogeneous in different directions by raising the annealing temperature. Therefore, it can be stated that the material is utterly isotropic at the annealing temperature of 350 °C.

5.2. Results of statistical analysis

Taguchi sensitivity analysis was performed for each of the key parameters of the stress-strain diagram. Figure 5 presents the influences of S/N ratios at every level. Also, the ranking of the influence of different parameters (annealing temperature and cutting angle) is depicted in Figure 6. The results showed that the annealing temperature influence on the static behavior of LZ71 alloy is several times greater than the effect of cutting angle. Moreover, this parameter also has the greatest effect on the UTS (92%). Also, the effect of cutting angle is more visible on EM and SBP.

The response surface 2D graphs of annealing temperature and sheet angle on the various responses are demonstrated in 0. Also, Figure 7-a and Figure 7-b display the stress reduction in ascending order through the severity of the factors individually. On the other hand, the minimum value of stress components (ultimate and yield) is related to the maximum temperature of the annealing process. However, the minimum value of elastic modulus is related to a particular range including annealing temperature of 125-200°C and 70-220°C for sheet angle close to 0 and 90, respectively. However, the maximum elastic modulus occurs at the maximum annealing temperature as shown in 0-c.
Furthermore, 0-d shows that the two parameters have a different effect on the failure strain of the material. In the lower annealing temperature (less than 150°C), the sheeting angle has the most effect. But, its effect significantly decreases by increasing the annealing temperature.

Figure 5. Influences of S/N ratios of all parameters related to a) UTS, b) YS, c) EM, and d) SBP

Figure 6. The ranking of the influence of different parameters on static properties of LZ71 alloy sheet
Figure 7. Response surface 2D graphs of annealing temperature and sheet angle on the various responses: a) ultimate stress, b) yield stress, c) elastic modulus, and d) strain at the breakpoint

It is obvious that if the target function is to increase the elongation, the sample should be used with a sheet angle of zero and an annealing temperature of 350°C which has a significant disadvantage. This process reduces ultimate and yield stresses. In other words, it causes to reduce the static strength of the material. Response optimization was used to distinguish the combinations of variable settings which optimize a single response or a bunch of responses. While assessing the influence of numerous factors on a response is wanted, response optimization is altogether helpful [41-42]. Therefore, the optimization was provided by an RSM-based optimization algorithm, the annealing temperature of 350°C, and sheet angle of 90° are to be optimized with a composite desirability value of 0.7308 by considering the maximizing all the key parameters of the tensile test results as optimization target. Due to the optimized values, ultimate and yield stresses are optimized with 194.6347 and 122.7793 MPa, respectively by overcoming the desirability value of 0.96. elastic modulus is optimized with 13.5568 GPa, and elongation reaches a level of 47.58%.

6. Conclusion
In the current research, cast Mg-7Li-1Zn alloy specimens were fabricated from different sheet angles relative to the rolling direction. Then, the specimens were annealed at different temperatures. The study was aimed at investigating the effect of annealing temperature and cutting angle on the microstructure, microhardness, grain size, and mechanical properties of the material. Various experimental approaches were employed to explore the microstructural and mechanical properties of
the treated specimens. Next, the Taguchi approach and response surface method were used to analyze and optimize.

The volume fraction of the alpha phase increase by raising the temperature of the annealing process and the microstructure finds a more homogeneous distribution. Thus, mechanical properties for annealed samples at 350 °C are expected to be the same in all directions. The surface microhardness of the specimens decreases by raising the temperature of the annealing process. Moreover, the crystallite size increase by raising the annealing temperature in both hcp and bcc structures.

Tensile testing results showed that ultimate and yield stresses and elastic modulus are approximately the same at different sheet angles and there is a significant change in their elongation at the breakpoint. The maximum and minimum values of elongation are for sheet angles of 0 and 90 relative to the rolling process, respectively. However, the value of this parameter converges in different directions by increasing the temperature of the annealing process. Also, ultimate stress decreases by increasing the temperature. Also, the static properties of the sheet become homogeneous in different directions by raising the annealing temperature. Therefore, it can be stated that the material is completely homogeneous at the annealing temperature of 350°C.

TA results reveal that annealing temperature is the most effective parameter on the static behavior of LZ71 alloy. The effect of annealing temperature on the UTS, YS, EM, and SBP is 92%, 76%, 61%, and 66%, respectively. Moreover, RMS results indicated that the minimum value of elastic modulus is related to the annealing temperature range of 125-200°C and 70-220°C for sheet angles close to 0 and 90, respectively. However, the maximum elastic modulus occurs at the maximum annealing temperature. Moreover, the sheeting angle has the most effect in the annealing temperature less than 150°C. But, its effect significantly decreases by increasing the annealing temperature. Due to the optimized factors, ultimate and yield stresses are optimized with 194.6347, and 122.7793 MPa, respectively by overcoming the desirability value of 0.96. Elastic modulus is optimized with 13.5568 GPa, and failure strain reaches a level of 47.58%.

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