INFLUENCE OF HEAT TREATMENT ON MICROSTRUCTURE AND FATIGUE BEHAVIOR OF AZ61 MAGNESIUM ALLOY

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Abstract
The contribution is focused on the assessment of the influence of the heat treatment of AZ61 magnesium alloy on its microstructure, mechanical properties and fatigue behaviour in both the low- and high-cycle regimes. Experimental material was manufactured by the squeeze casting method and the heat treatment was performed at a temperature of 380 °C with various annealing time with subsequent water quenching. Optimal heat treatment conditions were established based on the results of tensile tests and metallographic analysis. Cyclic deformation behaviour and fatigue life of AZ61 magnesium alloys was studied under stress amplitude-control in a laboratory air at room temperature.

Keywords: AZ61, magnesium alloys, solution heat treatment, low cycle fatigue, high cycle fatigue

1 Introduction

Cast magnesium alloys are ideal for structural applications due to their low density, high specific strength and good damping capacity. An improvement of the mechanical properties of cast alloys can be achieved by using new manufacturing technologies, e.g. squeeze casting. Heat treatment, which results in reducing structural and chemical heterogeneity, is another option.

In AZ magnesium alloys, aluminium is partly in solid solution and partly precipitated in the form of γ phase ($Mg_{17}Al_{12}$) along grain boundaries as a part of lamellar eutectic or as a coarse particles [1–5]. Intermetallic γ phase is brittle and when it precipitates from grain boundaries, the heterogeneity of base material increases, which results in ductility decrease [6]. For AZ magnesium alloys the solution annealing conditions are chosen such that the γ phase is dissolved in all forms and that, after rapid cooling, the structure is formed by supersaturated solid solution δ with a minimum difference in the concentrations of additive elements [7, 8, 9].

Dissolution of the intermetallic γ phase leads to a significant improvement in the mechanical properties of same type of AZ alloys [1, 7, 10].

In the present study, influence of heat treatment on microstructure and mechanical properties of AZ61 magnesium alloy is evaluated. Based on metallographic analysis and tensile tests, optimal conditions of heat treatment are established. Additionally fatigue behaviour of as cast and optimally heat treated alloy are evaluated.

2 Experimental material and methods

The experimental material used was AZ61 magnesium alloy, fabricated by the squeeze casting in ZFW GmbH in Clausthal. A filling pressure of 97 MPa was applied, with subsequent squeeze and solidification at a pressure of 150 MPa. Material was supplied in the form of billet with dimension of 200 mm in diameter and 40 mm in height. The chemical composition measured by a Spectrumat GDS 750 optical emission spectrometer with glow discharge is shown in **Table 1**. The basic mechanical properties of as cast material established via tensile tests are given in **Table 2**.

Cylindrical specimens with a diameter of 6 mm and 20 mm in length were subjected to solution heat treatment at temperature of 360 and 380 °C with various annealing time from 1 to 16 hours and water quenched. The specimens were used for metallographic assessment and determination of the optimum annealing time from the viewpoint of structural and chemical homogeneity. Metallographic specimens were prepared in the usual way and etched with a mixture of picric acid (5 ml acetic acid, 6 g picric acid, 10 ml water, and 100 ml ethanol). As after brief examination of specimens was obvious that the temperature of 360 °C was too low to dissolve the γ phase, only specimens heat treated at 380 °°C were further evaluated. Microstructural evaluation, local analysis of chemical composition and analysis of elemental distribution were performed on scanning electron microscope PHILIPS XL30 with EDX analyzer.

For the determination of optimal conditions of heat treatment of AZ61 magnesium alloy from the viewpoint of mechanical properties the tensile test and hardness evaluation were proposed. The tensile test specimens were heat treated in the same way as specimens for metallographic analysis; tensile tests were conducted on a TIRA Test 2300 instrument according to EN ISO 6892-1 [11] with strain rate of 0.00025 s^{-1}

Fatigue test specimens were heat treated under conditions that were established to be optimal, test were performed on as cast and heat treated material to assess the influence of heat treatment on fatigue behaviour of AZ61 magnesium alloy.

Low-cycle fatigue behaviour in the stress amplitude control mode was determined on a servo-hydraulic testing system. Experiments were conducted in the range of proof stress amplitudes from 50 to 120 MPa at constant frequencies of 3 Hz and 20 Hz. To assess fatigue behaviour in the high-cycle regime, fatigue tests with proof stress amplitude in the range from 40 to 55 MPa and at a frequency of 130±5 Hz were conducted on a high-resonance pulsator. The symmetrical sine cycle ($R = -1$) was used, all tests were carried out under laboratory conditions in air at room temperature. Experimental data in low and high cycle fatigue region were fitted by Stromeyer regression function while Palmgren regression function was used to fit experimental data in whole fatigue life.

Table 1 Chemical composition of AZ61 magnesium alloy (wt%)

Al	Zn	Mn	Si	Fe	Ni	Zr	Sn, Cu, Pb	Mg
5.2	0.99	0.42	0.013	0.003	0.01	0.01	max. 0.01 %	bal.

Table 2 Tensile properties of AZ61 alloy in as cast (F) and heat treated conditions (T4)

	E [GPa]	$R_{p0.2}$ [MPa]	R_m [MPa]	A [%]
AZ61-F	42.9	73	175	5.0
AZ61-T4	43.1	75	237	10.9

3 Results

3.1 Tensile properties and microstructure

Results of the tensile tests of heat treated material with graded annealing time are expressed in **Fig. 1a**. It is evident from the results that 1-hour at the temperature leads to marked increase in tensile strength and elongation. Longer period of annealing time results in slight changes in tensile strength and elongation with local maxima at 6 hours. From the results it is also evident that with increasing annealing time the values of yield point practically do not change.

The results for the hardness of specimens after heat treatment at a temperature of 380 ºC with various times at the temperature are presented in **Fig. 1b**. With increasing annealing time there is a slow decrease in hardness.

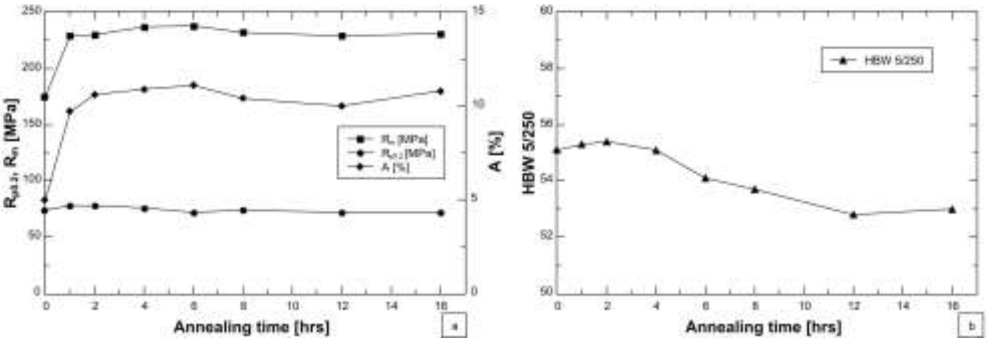

Fig.1 Effect of annealing time (a) on stress and strength characteristics of alloy AZ61, (b) on hardness of alloy AZ61

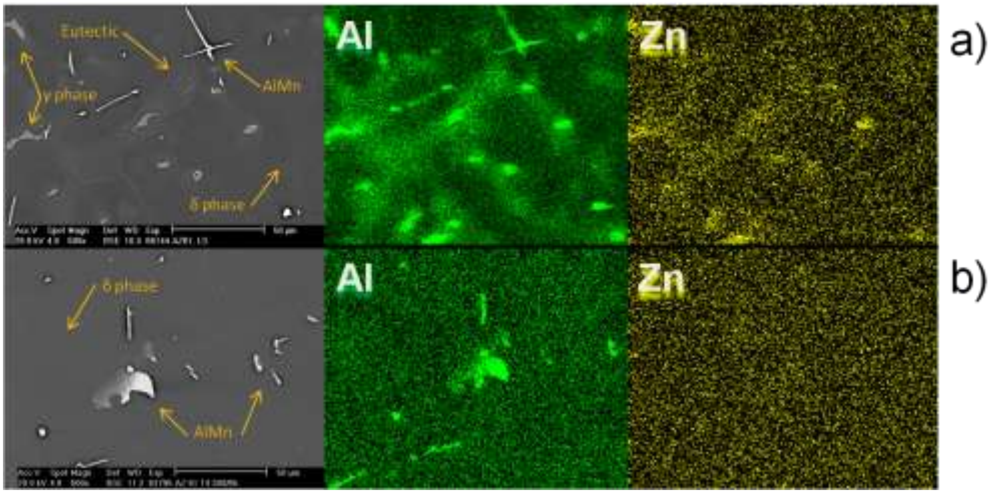

Fig.2 Microstructure and elemental distribution of (a) AZ61 as cast, (b) AZ61 solution heat treated

According to analysis of local chemical composition and binary Al-Mg [12] and ternary Mg-Al-Zn [13] diagrams the structure of squeeze cast AZ61 magnesium alloy (F) is formed by the δ solid solution, intermetallic γ phase ($Mg_{17}Al_{12}$), eutectic (δ + γ), and AlMn-based particles. The alloy in as cast condition is heterogeneous and the periphery of crystalline units is rich in Al and Zn (**Fig. 2a**).

The material was subjected to solution heat treatment (T4) to reduce structural and chemical heterogeneity.

Based on the evaluation of mechanical properties and changes in heterogeneity, the heat treatment conditions were established. A temperature of 380 °C and 6 hours of annealing were found to be optimal. The solution heat treatment results in the dissolution of the γ phase and in a significant decrease in heterogeneity. The structure of heat treated material is formed only by the solid solution δ and AlMn particles (**Fig. 2b**). The basic stress and strain characteristics of the AZ61 magnesium alloy in heat treated condition (T4) are given in Table 2.

3.2 Cyclic plasticity and low-cyclic fatigue

A comparison of static tensile curves and cyclic stress-strain curve, together with the cyclic yield strength $R_{p0,2}''$ is shown in **Fig. 3**. Experimental data of the CDC were fitted to the modified Ramberg-Osgood function (1) [12, 13]; the parameters are given in Table 3. As the cyclic plastic response was practically same for both, as cast and heat treated conditions, experimental data were fitted by single curve.

The derived Manson-Coffin curves (**Fig. 4**) for AZ61 in as cast and heat treated conditions were fitted by power law (2). The parameters are summarized in **Table 3**.

Due to dependence of fatigue parameters, the generally known equations (3) and (4) were used to establish parameters σ_f = 448.36 a b = −0.16.

$$\varepsilon(\sigma) = \frac{\sigma}{E} + \left(\frac{\sigma}{\sigma_0}\right)^m \tag{1.}$$

$$\varepsilon_{ap} = \varepsilon_f' \cdot (2N_f)^c \tag{2.}$$

$$b = \frac{1}{m} \cdot c \tag{3.}$$

$$\sigma_f' = \sigma_0 \cdot \left(\varepsilon_f'\right)^{n} \tag{4.}$$

where: ε [-] – strain
ε_{ap}[-] – plastic strain
σ [MPa] – stress
E [MPa] – Young´s modulus
m [-] – exponent of Ramberg-Osgood function
ε_f' [-] – fatigue ductility coefficient
N_f [-] – number of cycles to failure
c [-] fatigue ductility coefficient
b [-] – fatigue strength coefficient
σ_f' [MPa] fatigue strength coefficient
n [-] cyclic hardening exponent

Table 3 Regression parameters of Ramberg-Osgood and derived Manson-Coffin curves

Ramberg-Osgood – F + T4			**Manson-Coffin – F**		**Manson-Coffin – T4**	
σ_0 [MPa]	m	$R_{p0.2}''$ [MPa]	ε_f'	c	ε_f'	c
497.86	4.666	132	0.613	−0.749	1.565	−0.835

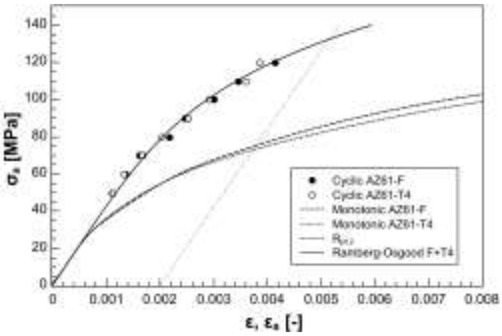

Fig.3 Static tensile and cyclic stress-strain curves of AZ61-F and AZ61-T4

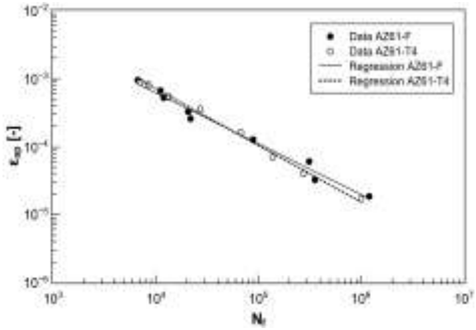

Fig.4 Derived Manson-Coffin curves of AZ61-F and AZ61-T4

3.3 Regression analysis of Wöhler curves (S-N curves)

The dependence of stress amplitude σ_a on the number of cycles to fracture N_f is given in **Fig. 5**. Experimental data in the low and high cycle fatigue regime for AZ61 in as cast and heat treated conditions were fitted by the Stromeyer function (5) and data in whole fatigue life were fitted by Palmgren function (6), where *a* is extrapolated value of function or of the tangent in the point of inflexion for N = l, *b* is the slope of oblique asymptote or of the tangent in the point of inflexion in log-log fit, σ_∞ is limit value of fatigue stress for infinite number of cycles and *B* the numbers of cycles in which the curve bends and its slope is equal to *b/2* in log-log fit [16].

$$\sigma(N) = a \cdot N^b + \sigma_\infty \tag{5.}$$

$$\sigma(N) = a \cdot (N + B)^b + \sigma_\infty \tag{6.}$$

where: a [-]parameter of the Basquin and some other functions
b [-] parameter of the Basquin and some other functions
N [-] number of cycles
σ_∞ [MPa] permanent fatigue limit
B [-] parameter of Palmgren function

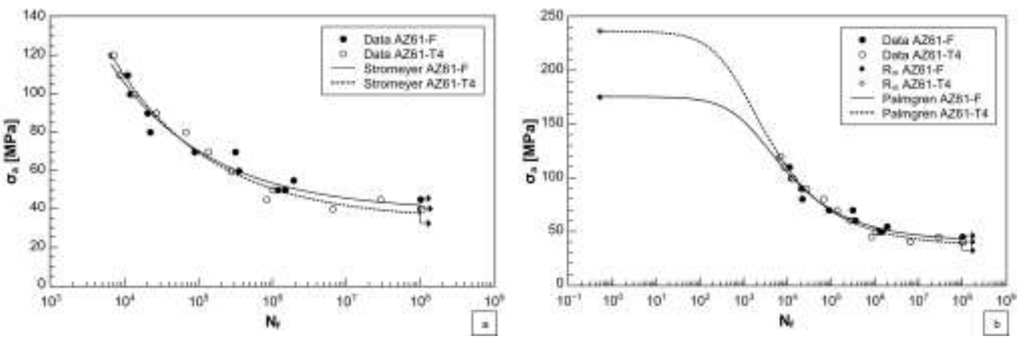

Fig.5 S-N curves using (a) Stromayer function and (b) Palmgren function

Regression parameters obtained by means of the squares of deviation minimization are summarized in **Table 4** and **Table 5**.

Table 4 Regression parameters of Stromeyer fit

Stromeyer AZ61-F			Stromeyer AZ61-T4		
a [MPa]	b	σ_∞ [MPa]	a [MPa]	b	σ_∞ [MPa]
1347.6	−0.326	38.5	1549.6	−0.328	34.0

Table 5 Regression parameters of Palmgren fit

Palmgren AZ61-F				Palmgren AZ61-T4			
a [MPa]	b	B	σ_∞ [MPa]	a [MPa]	b	B	σ_∞ [MPa]
1795.6	−0.351	1433.8	40.5	1992.9	−0.356	629.7	36.0

4 Discussion

The structure of AZ61 magnesium alloy in as cast condition is formed by the solid solution, intermetallic γ phase, eutectic, and AlMn particles which is in agreement with literature [17-19]. Heat treatment with optimal conditions (380 °C/6 hrs) leads to microstructure which is formed by the solid solution δ and AlMn particles.

The increase in tensile strength and elongation and the drop in hardness is the result of gradual dissolution of the γ phase and decreasing heterogeneity of the solid solution. Increase in tensile strength and ductility is in agreement with experiments performed on AZ magnesium alloys by Kleiner et al [9]. Authors of that paper also observed drop in yield strength due to grain coarsening. That was not observed in our experiment and the chosen temperature of solution heat treatment therefore can be seen optimal.

Dissolution of the γ phase in our experimental material led to increase in tensile strength by 35 % and insignificant increase in yield strength by 2.7 % which is in contrary with findings published by Liu et al [20]. They observed increase in tensile strength by 11.9 % and much higher increase in yield strength by 29 % resulting from dissolution of γ phase in AZ91 magnesium alloy.

Clear increase in cyclic yield strength (73 MPa for AZ61-F, 75 MPa for AZ61-T4) versus static yield strength (132 MPa) is again in contrary with AZ91 magnesium alloy [20], where increase in yield strength due to cyclic deformation was 29 % (AZ91-F) and 8.6 % (AZ91-T4) compared to increase in our material which was about 80 % for both conditions.

Also can be concluded, that heat treatment had a minimal effect on the cyclic response and subsequent strain hardening because of practically the same values of Young´s modulus and cyclic yield strength of the material in as cast and T4 conditions.

The significant increases in the deformation characteristics and tensile strength after heat treatment do not result in any significant increase in fatigue life at higher stress amplitudes. This is given by the similar values of plastic response and micro mechanism of fatigue failure.

The parameters of derived Manson-Coffin curves are in agreement with the trend of the Basquin function parameters. Due to dependence of fatigue parameters and identity of CDCs of material in both conditions, the expected trend of Wöhler-Basquin curves will be similar to trend of derived Manson-Coffin curves.

Based on results of regression analysis (table 4 and table 5), heat treatment results in increase in value of parameter a for both regression functions. The relative standard deviation of this parameter is significant because of far extrapolation due to deficiency of experimental points in the range of number of cycles $N_f < 10^4$.

Heat treatment of AZ61 magnesium alloy do not result in significant change of parameter *b* established by Stromayer and Palmgren regression functions. By comparing parameters *b* with parameters calculated from low cycle fatigue data was determined, that value of parameters *b* established by Stromayer and Palmgren regression functions was practically double. This finding is in agreement with results of Kohout et al [16].

When the experimental data in the whole fatigue life were fitted by the Palmgren function (**Fig. 5b**), decrease in the value of parameter B for material after heat treatment was observed. Parameter B is the numbers of cycles in which the curve bends in quasi-static and low-cycle fatigue regimes. Compared to as cast material, half of number of cycles to failure is predicted for the material after heat treatment. The Palmgren regression function well describes the increase in the value of tensile strength due to heat treatment, but description of fatigue behavior in quasi-static regime is only approximate.

The intersection of curves (**Fig. 4 and Fig. 5**) approximately at $8 \cdot 10^4$ numbers of cycles is probably caused by change of fatigue failure micro mechanism.

Increase in fatigue life time due to increase plasticity in the critical volume of material which is intensively plastically deformed was observed for material after heat treatment loaded at high stress amplitudes. In the high-cycle fatigue regime was observed decrease in fatigue strength for material after heat treatment.

5 Conclusions

The microstructure of as cast AZ61 alloy is formed by solid solution, eutectic, γ phase and AlMn particles. The dissolution of the γ phase and the decrease in heterogeneity result in a significant increase in static mechanical properties in the elastic-plastic response regime.

Material is cyclically hardening, which is proved by comparison of static tensile and cyclic stress-strain curves and furthermore by Manson rule ($R_m/R_{p0.2}$ greater than 1.4). Ratio $R_m/R_{p0.2}$ increased from 2.4 (material in as cast condition) to 3.2 due to heat treatment.

The fatigue behaviour is associated with the cyclic plastic response, which has practically the same values for both conditions. It follows that heat treatment has no significant effect on the low- and high- cycle fatigue behaviour.

Minor differences of both, F and T4 conditions of material are evident from derived Manson-Coffine curves. At higher stress amplitudes was observed slight improvement of fatigue life for material after heat treatment.

The fatigue limits for 10^8 cycles were obtained by each regression function. The fatigue limits obtained by the Stromeyer function are σ_c = 41.8 MPa (F) and σ_c = 37.6 MPa (T4). For Palmgren function are the fatigue limits σ_c = 43.1 MPa (F) and σ_c = 38.9 MPa (T4).

Based on the findings it was established that the fatigue ratio (σ_c/R_m) decreases from 0.24 in as cast condition to 0.16 in heat treated condition.

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