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Miroslava HORYNOVÁ*, Pavel DOLEŽAL, Pavel GEJDOŠ***, Josef ZAPLETAL****, Drahomíra JANOVÁ*****

INFLUENCE OF HEAT TREATMENT ON MECHANICAL PROPERTIES AND MICRO-STRUCTURE OF AZ61 MAGNESIUM ALLOY

OVLIVNĚNÍ STRUKTURY A MECHANICKÝCH VLASTNOSTÍ HOŘČÍKOVÉ SLITINY AZ61 TEPELNÝM ZPRACOVÁNÍM

Abstract

The contribution is focussed on the assessment of the influence of heat treatment of magnesium alloy AZ61 on its mechanical properties and chemical and microstructural heterogeneity. The experimental material was manufactured by the squeeze casting method using alternative two-stage casting. The microstructure of the alloy in as cast state was composed of solid solution δ, intermetallic phase $Mg_{17}Al_{12}$, and minor Mn-based phases. The heat treatment applied to the alloy was solution annealing at a temperature of 380 °C and with graded dwell time from 1 to 16 hours.

Chemical etching was employed in order to estimate the micro-segregation of the elements in the matrix qualitatively and to discern the phases in the microstructure. Differences in etching between the central part and the periphery of crystalline units are apparent in the metallographic images documented by light microscopy. The quantitative assessment of chemical heterogeneity was done via mapping and local chemical microanalyses. The intensity of chemical micro-heterogeneity of each specimen was further expressed using effective distribution coefficients.

The assessment of the influence of heat treatment on mechanical properties of the AZ61 magnesium alloy was performed by means of tensile tests on specimens of rational geometry. The basic stress and strain characteristics were assessed from the tensile test results. For a complex evaluation of the properties, the hardening test was performed.

It was found by metallographic analysis of the test specimens that longer dwell times cause gradual dissolution of the hard and brittle $Mg_{17}Al_{12}$ intermetallic phase, which dissolves completely after 6 hours. It was confirmed by local chemical analysis that simultaneously the micro-heterogeneity of individual elements decreases.

It further follows from the experimental results that tensile strength and deformation characteristics grow already after a 1-hour dwell at a temperature of 380 °C. The maximum values of tensile strength and deformation characteristics were achieved after 6 hours' dwell. The decrease in hardness and increase in tensile strength and deformation characteristics is caused by the dissolution of the hard and brittle intermetallic phase and by the decrease in heterogeneity of the matrix.

Abstrakt

Příspěvek je zaměřen na posouzení vlivu tepelného zpracování hořčíkové slitiny AZ61 na změnu chemické a strukturní mikroheterogenity a mechanických vlastností. Hořčíková slitina AZ61

* Bc. Miroslava Horynová, Brno University of Technology, Faculty of Mechanical Engineering, Institute of Materials Science and Engineering, Technická 2, Brno, e-mail kik.horynova@seznam.cz
** ng. Doležal Pavel, Ph.D., Brno University of Technology, Faculty of Mechanical Engineering, Institute of Materials Science and Engineering, Technická 2, Brno, tel. (+420) 54 114 3189, e-mail dolezal@fme.vutbr.cz
*** Ing. Pavel Gejdoš, Brno University of Technology, Faculty of Mechanical Engineering, Institute of Materials Science and Engineering, Technická 2, Brno, tel. (+420) 54 114 3466, e-mail gejdos@fme.vutbr.cz
**** Ing. Josef Zapletal, Brno University of Technology, Faculty of Mechanical Engineering, Institute of Materials Science and Engineering, Technická 2, Brno, tel. (+420) 54 114 3466, e-mail pepa.sanguis@centrum.cz
***** Ing. Drahomíra Janová., Brno University of Technology, Faculty of Mechanical Engineering, Institute of Materials Science and Engineering, Technická 2, Brno, tel. (+420) 54 114 3105 e-mail janova@fme.vutbr.cz

byla vyrobena metodou squeeze casting, alternativním dvoustupňovým litím. Mikrostruktura slitiny v litém stavu byla tvořena tuhým roztokem δ, intermetalickou fází $Mg_{17}Al_{12}$ a minoritními fázemi na bázi Al-Mn . Jako tepelné zpracování bylo použito rozpouštěcí žíhání při teplotě 380°C a s odstupňovanou výdrží 1 až 16 hodin.

Pro kvalitativní odhad mikrosegregace prvků v matrici hořčíkové slitiny a přítomných fází ve struktuře bylo využito leptání chemickými činidly. Na snímcích ze světelné mikroskopie byly zdokumentovány rozdíly heterogenity matrice ve středu a na krajích krystalických útvarů v závislosti na intenzitě naleptání. Kvantitativní hodnocení chemické mikroheterogenity bylo prováděno s využitím map rozložení prvků (mapping) a lokální chemické mikroanalýzy. Intenzita chemické mikroheterogenity jednotlivých vzorků byla vyjádřena pomocí efektivních rozdělovacích koeficientů.

Posouzení tepelného zpracování na mechanické vlastnosti hořčíkové slitiny AZ61 bylo prováděno pomocí zkoušky tahem na válcových poměrných zkušebních tělesech. Z výsledků zkoušky tahem byly stanoveny základní napěťové a deformační charakteristiky. Pro kompletní posouzení vlivu tepelného zpracování na mechanické vlastnosti bylo provedeno také hodnocení tvrdosti.

Metalografickým hodnocením bylo zjištěno, že s delší dobou výdrže na teplotě dochází k rozpouštění intermetalické fáze $Mg_{17}Al_{12}$, která se kompletně rozpouští až po 6 hodinách. Lokální chemickou mikroanalýzou bylo potvrzeno, že současně dochází i ke snižování chemické mikroheterogenity jednotlivých prvků.

Z experimentálních výsledků dále vyplývá, že již po jedné hodině výdrže na teplotě 380°C dochází k nárůstu meze pevnosti a deformačních charakteristik. Maximálních hodnot meze pevnosti a deformačních charakteristik bylo dosaženo u vzorků s výdrží 6 hodin na teplotě. Při delší výdrži na teplotě byl již dále prokázán postupný pokles těchto charakteristik. Pokles tvrdosti, zvýšení meze pevnosti a deformačních charakteristik je způsobeno právě rozpouštěním křehké intermetalické fáze a snížením heterogenity matrice.

1 INTRODUCTION – USED DRECE TECHNOLOGY

Magnesium alloys enable reducing the weight of machine parts while maintaining good mechanical properties and are therefore used in automotive and aircraft industries. However, the mechanical properties of magnesium alloys do not equal the values of alloys of higher-density metals and therefore it is necessary to focus on enhancing their mechanical properties. This can be achieved by subjecting them to heat treatment, reducing the occurrence of casting defects, and developing new manufacturing technologies. A serious problem of magnesium alloys can be seen in their poor corrosion resistance, which is influenced not only by the chemical composition of the alloy but mainly by the distribution of elements and the morphology of individual phases [1, 2, 3, 4].

In type 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. In the process of corrosion the γ phase functions as a barrier and reduces the corrosion rate but, at the same time, it can form with the solid solution a corrosion cell and accelerate the corrosion process. Thus, the corrosion of AZ alloys is greatly influenced by the aluminium distribution and morphology of the γ phase.

In castings, a big problem consists in the chemical heterogeneity, which affects not only the corrosion resistance of the material but also its mechanical properties. Solution annealing is used to reduce the irregular distribution of alloying elements in the alloy. For AZ magnesium alloys the solution annealing conditions are chosen such that the γ phase is dissolved in all forms and that, after cooling, the structure is formed by solid solution δ with a minimum difference in the concentrations of additive elements [5].

2 EXPERIMENTAL

As experimental material used was the AZ61 magnesium alloy fabricated by the squeeze casting method in ZWF GmbH in Clausthal. A filling pressure of 97 MPa was applied, with subsequent squeeze and solidification at a pressure of 150 MPa. Table 1 gives the chemical composition of the alloy, measured by a Spectrumat GDS 750 optical emission spectrometer with glow discharge, while Table 2 gives the mechanical properties of the as-cast alloy.

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

Element	Al	Zn	Mn	Si	Fe	Ni	Zr	Be	*
Measured	5.22	0.99	0.42	0.013	0.003	0.01	0.01	0.000	< 0.00

*** - Sn, Cu, Pb**

Table 2 Tensile properties of AZ61 Mg alloy fabricated by squeeze casting

	E [GPa]	$R_{p0,2}$ [MPa]	R_m [MPa]	A [%]	Z [%]
AZ61	41.1	74	175	5.0	5.3

Specimens for metallographic assessment 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). The micro-structure was evaluated on an Olympus GX71 light microscope equipped with an Olympus DP11 camera. A more detailed evaluation, local analysis of chemical composition and analysis of element distribution were performed on a PHILIPS XL30 scanning electron microscope with EDAX analyzer.

According to the results of the local analysis of chemical composition and literary data [1, 6], the structure of AZ61 alloy (Fig. 1) is formed by the solid solution δ, intermetallic γ phase ($Mg_{17}Al_{12}$), eutectic (δ + γ), and AlMn-based particles with different stoichiometric ratios. The material is heterogeneous; it is evident from the analysis of element distribution (Fig. 3) that there is Al-rich δ phase at the grain boundaries and around γ phases. On the basis of the results of local analyses of chemical composition, differences were established in the concentration of aluminium and zinc (Tables 3 and 4) and subsequently expressed in terms of effective distribution coefficients

$$k_{ef} = \frac{[\%i]_c}{[\%i]_p} , \qquad (1)$$

where [%i]c and [%i]p are the contents of elements -i- in the centre or on the periphery of crystalline configurations.

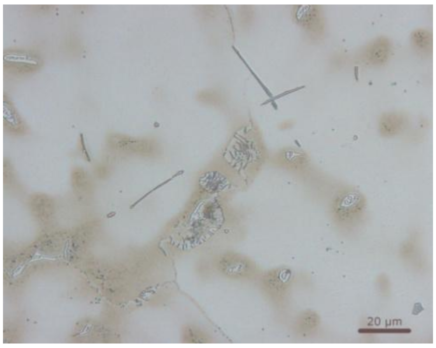

Fig. 1 Structure of as-cast alloy AZ61 (LM)

Fig. 2 Structure of as-cast alloy AZ61 (SEM)

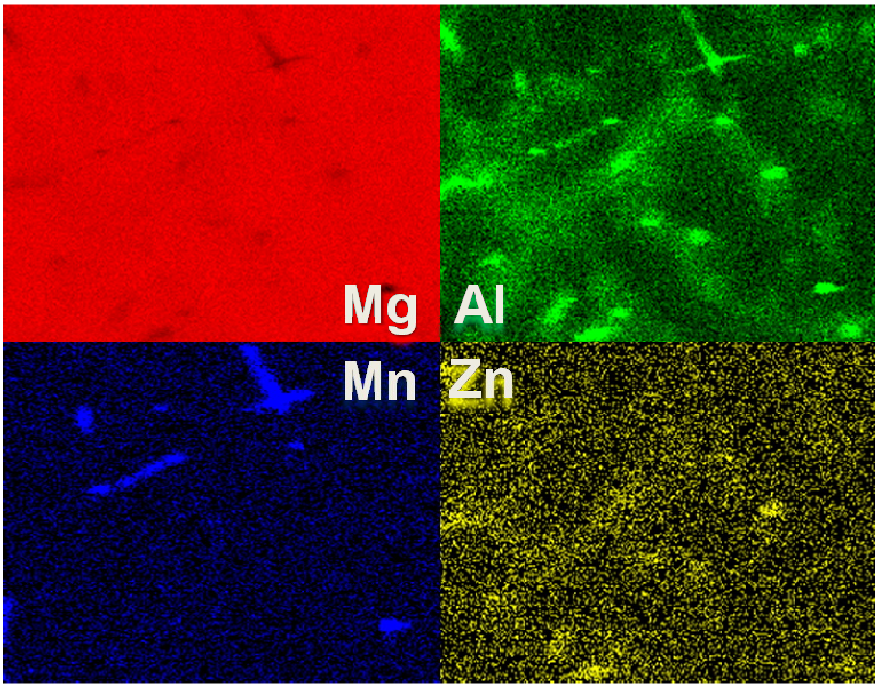

Fig. 3 Analysis of element distribution in AZ61 as-cast (SEM/EDS 500x)

Table 3 Effective distribution coefficients of Al in AZ61 as-cast

Al_c [wt%]		Al_p [wt%]		Al_p-Al_c [wt%]	k_{ef}
x	S	x	S		
2.943	0.092	9.037	0.301	6.094	0.326

Table 4 Effective distribution coefficients of Zn in AZ61 as-cast

Zn_c [wt%]		Zn_p [wt%]		Zn_p-Zn_c [wt%]	k_{ef}
x	S	x	S		
0.763	0.211	1.503	0.146	0.740	0.508

Cylindrical specimens of 6 mm in diameter and 20 mm in length were subjected to solution annealing at a temperature of 380 °C for periods of 1, 2, 4, 6, 8, 12, and 16 hours and water quenched. The specimens were used for metallographic assessment and determination of the optimum dwell time from the viewpoint of structural and chemical homogeneity.

For the assessment of an optimum procedure of the solution annealing of AZ61 magnesium alloy from the viewpoint of mechanical properties the tensile test and hardness evaluation were proposed. Test specimens for the tensile test were heat treated via solution annealing at a temperature of 380 °C with dwell times of 1 to 16 hours, with subsequent water quenching. The assessment of the basic stress and strength characteristics of the heat treated material was prepared from the results of tensile tests conducted on a TIRA Test 2300 instrument, on cylindrical proportional test pieces of diameter of 6 mm and length 30 mm. Specimen hardness was measured by the Brinell HBW 5/250 method on an HBE300 hardness testing machine.

3 REASULTS AND DISCUSSION

For the qualitative estimation of the microsegregation of elements it is usual to use etching by chemical agents; light microscope images are used as the output. In AZ magnesium alloys, areas rich in aluminium are, after etching, of a markedly darker colour than the surrounding matrix. In Fig. 4, the drop in heterogeneity with increasing dwell time at the temperature of solution annealing is evident. In comparison with the as-cast alloy (Fig. 1) a pronounced drop in heterogeneity is evident only after two hours of solution annealing. The structure is formed by the solid solution δ, residues of non-dissolved eutectic (Figs 4a and 4b), and AlMn-based inclusions. The inclusions can be spherical of up to 5 μm or elongated acicular shape or they can be of irregular shapes with rugged borders. According to literary sources [1] this is due to the growth of inclusions in the initial stage of alloy solidification.

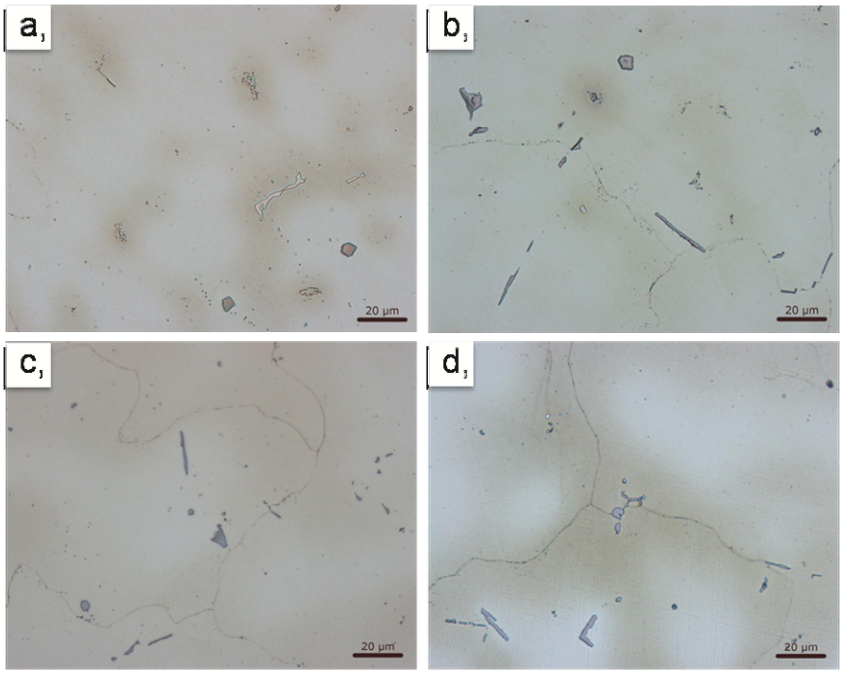

Fig. 4 Structure of AZ61 alloy after solution annealing at 380 ºC for periods of (a) 2 h, (b) 4 h, (c) 6 h, (d) 8 h (SM 500x)

The qualitative assessment of the structure and chemical microheterogeneity was done on the basis of the results of mapping. Figs 5 – 7 give the maps of element distribution for magnesium, aluminium and manganese. The amount of zinc in the AZ61 alloy fluctuates around 1 per cent, and in the heat treated alloy the changes in concentration are so low that they are within the measuring error range.

With increasing dwell time at the temperature the intermetallic phase starts to gradually dissolve and the microheterogeneity decreases. After two hours of solution annealing, the γ phase is still present in the structure, and the results of mapping (Fig. 5) still exhibit a difference in aluminium concentration in the matrix.

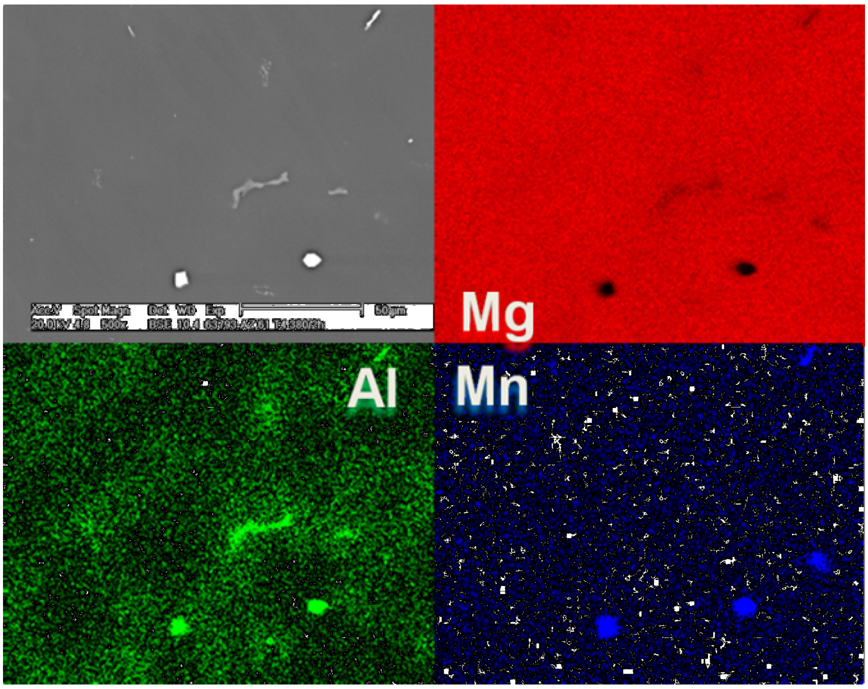

Fig. 5 Surface analysis of element distribution in alloy AZ61 T4 380 ºC 2 h

After four hours of solution annealing, most of the γ phase is dissolved, and sites with higher aluminium concentration only occur in the surroundings of non-dissolved residues of this phase (Fig. 6).

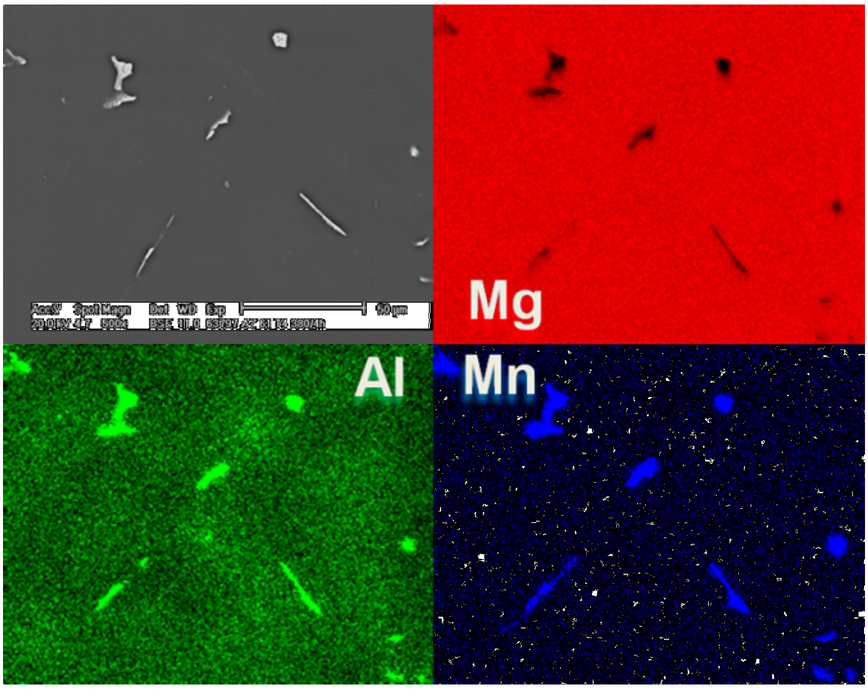

Fig. 6 Surface analysis of element distribution in alloy AZ61 T4 380 ºC 4 h

In the alloy with six hours of solution annealing (Fig. 7) the γ phase was completely dissolved, and chemical microheterogeneity changed only a little in comparison with the alloy after four hours of solution annealing.

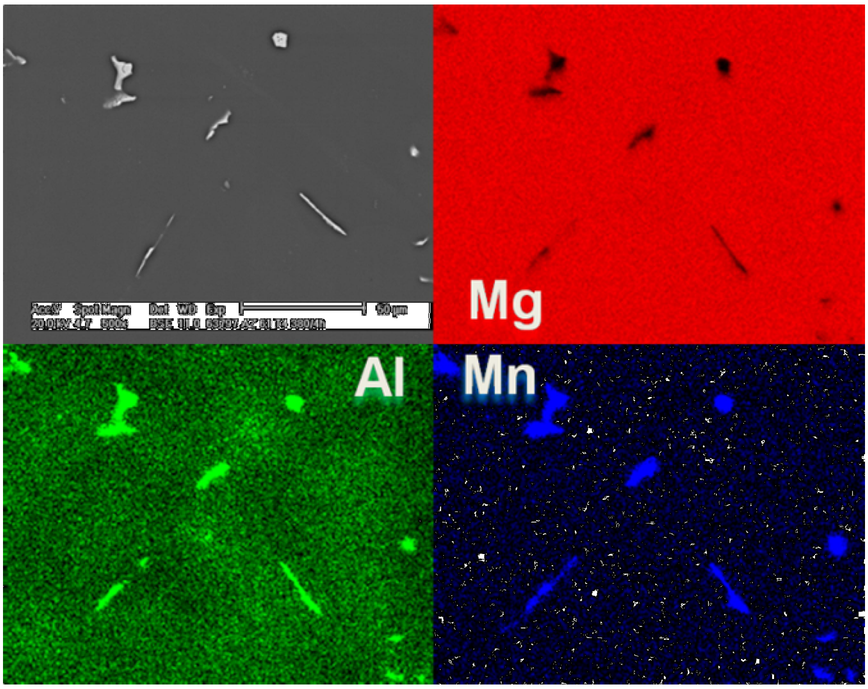

Fig. 7 Surface analysis of element distribution in alloy AZ61 T4 380 ºC 6 h

Changes in chemical microheterogenity are expressed quantitatively in Tables 5 and 6 in terms of differences in the concentration of Al and Zn and in Table 7 in terms of effective distribution coefficients. As expected, the values of effective distribution coefficients increase with the length dwell time, excepting the specimen with 6 hours of solution annealing, in which the deviation is due to the large scatter of the results measured.

Table 5 Differences in Al concentrations for AZ61 alloy after various periods of solution annealing

	Al_c [wt%]		Al_p [wt%]		Al_p-Al_c [wt%]
	x	S	x	S	
AZ61 T4 380°C 2hod	2.323	0.414	6.067	0.289	3.744
AZ61 T4 380°C 4hod	4.193	0.112	5.920	0.593	1.727
AZ61 T4 380°C 6hod	3.603	0.436	5.303	0.640	1.700

Table 6 Differences in Zn concentrations for AZ61 alloy after various periods of solution annealing

	Zn_c [wt%]		Zn_p [wt%]		Zn_p-Zn_c [wt%]
	x	S	x	S	
AZ61 T4 380°C 2hod	0.683	0.172	1.337	0.431	0.654
AZ61 T4 380°C 4hod	1.033	0.195	1.350	0.040	0.317
AZ61 T4 380°C 6hod	1.327	0.006	1.627	0.091	0.300

Table 7 Effective distribution coefficients of AZ61 alloy after various periods of solution annealing

	k_{Al}^{ef}	k_{Zn}^{ef}
AZ61 as cast	0.326	0.508
AZ61 T4 380°C 2hod	0.383	0.512
AZ61 T4 380°C 4hod	0.708	0.763
AZ61 T4 380°C 6hod	0.679	0.816

Results of the tensile test after heat treatment with graded dwell time are presented graphically in Figs 8 and 9. It is evident from the results that following a 1-hour dwell at the temperature there is a marked increase in the stress characteristics (tensile strength, yield strength) and an increase in the deformation characteristics (elongation and contraction). Longer dwell times subsequently showed in a slight increase in stress and strain characteristics, with local strength, elongation and contraction maxima being reached after 6 hours of solution annealing. From the results it is also evident that with increasing dwell time the values of yield point practically do not change.

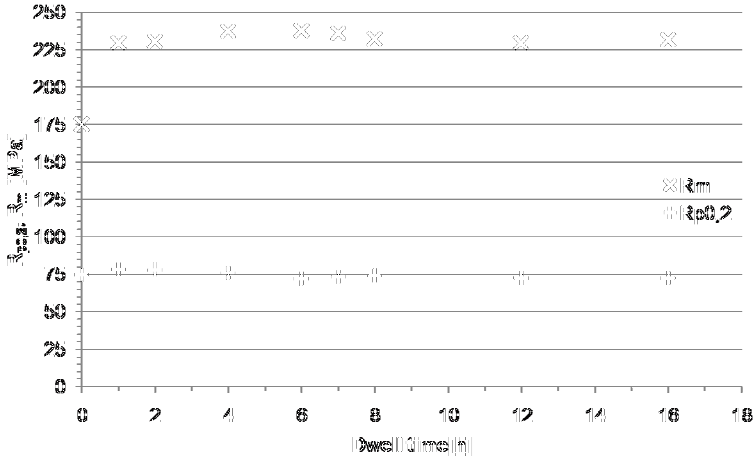

Fig. 8 Effect of dwell time on stress characteristics of alloy AZ61

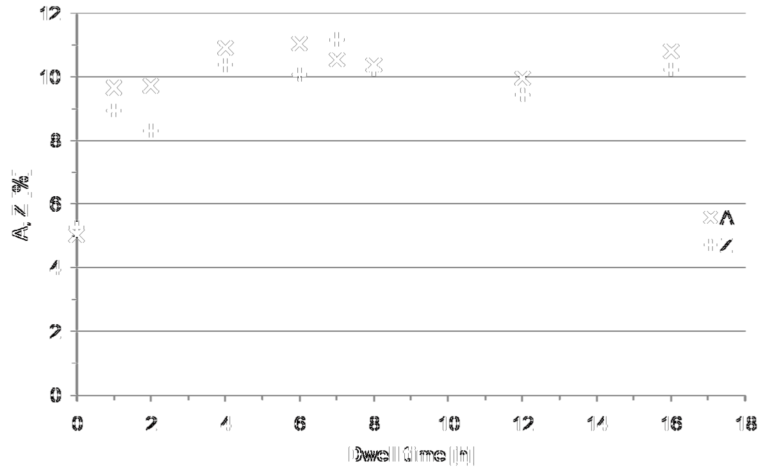

Fig. 9 Effect of dwell time on strength characteristics of alloy AZ61

For a full assessment of the influence of the heat treatment of AZ61 magnesium alloy on its mechanical properties, the hardness assessment was performed. The results for the hardness of specimens heat treated at a temperature of 380 ºC with various dwell times at the temperature are presented graphically in Fig. 10. With increasing dwell time at the temperature there is a slow decrease in hardness and after 10 hours the values are stabilized. The drop in hardness is the result of gradual dissolution of the γ phase and decreasing heterogeneity of the solid solution.

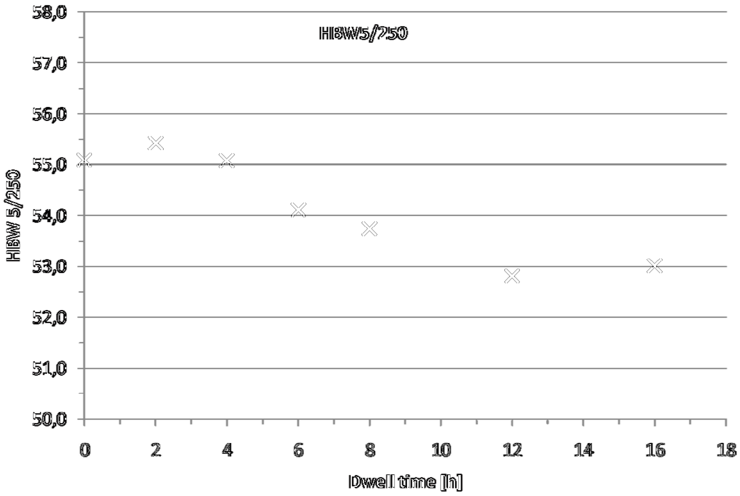

Fig. 10 Effect of dwell time on hardness of alloy AZ61

Optimum mechanical properties for the AZ61 alloy were established after the 6-hour dwell time at the solution annealing temperature. Metallographic assessment revealed that after the 6-hour dwell time at a temperature of 380 ºC the γ phase is completely dissolved and thus the maximum achievable values of strength and strain characteristics are obtained. The chosen temperature of solution hardening can be seen as optimal because there is no drop in the yield point due to grain coarsening.

4 CONCLUSIONS

The AZ61 alloy fabricated by the squeeze casting method consists of δ solid solution, coarse particles of γ phase and eutectic (γ+δ). The structure also contains Al-Mn-based inclusions with different stoichiometric ratios. The material is heterogeneous but solution annealing at a temperature of 380 ºreduces the heterogeneity. It was established that changes in the chemical heterogeneity occur already after a two-hour dwell time at the temperature. The peak change in the difference in the concentrations of additive elements comes after four hours, after that the concentration differences change only slightly.

The structure of AZ61 alloy after solution annealing is formed by δ solid solution, Al-Mn-based inclusions; depending on the dwell time at the temperature, the structure can also exhibit residues of the γ intermetallic phase, which is completely dissolved after six hours at 380 ºC.To establish the optimum solution annealing conditions from the viewpoint of mechanical properties, tensile tests were performed on specimens after heat treatment with various dwell times at a temperature of 380 ºC. From the stress and strength characteristics measured the maximum values for the alloy were established after six hours of solution annealing. Simultaneously with the dwell at the temperature there is a decrease in the hardness.

Experiments were conducted in order to establish the optimum conditions of solution annealing. A temperature of 380 ºC and a dwell time of 6 hours at the temperature were found to be optimal.

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