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Changes in hardness of magnesium alloys due to precipitation hardening

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Abstract
This paper deals with the evaluation of changes in hardness of magnesium alloys during precipitation hardening that are nowadays widely used in different fields of industry. It focuses exactly on AZ31, AZ61 and AZ91 alloys. Observing material hardness changes serves as an effective tool for determining precipitation hardening parameters, such as temperature and time. Brinell hardness measurement was chosen based on experimental needs. There was also necessary to make chemical composition analysis and to observe the microstructures of tested materials. The obtained results are presented and discussed in this paper.

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1. Introduction

Lightweight, low density, good machinability at higher temperatures and advantageous mechanical properties (mainly particular strength: density ratio) predispose magnesium alloys to the fact that they are nowadays very popular construction materials. Magnesium alloys have got good mechanical damping. The disadvantages are low hardness, limited ductility at room temperature caused by the hexagonal close packed (hcp) lattice of magnesium and low velocity of diffusion processes during heat treatment (BOLIBRUCHOVÁ, D. ET AL. 2005, LI, J. ET AL. 2015, YU, Z. ET AL. 2018). The use of magnesium in components and vehicles depends on numerous technical and economic factors. Investigated materials have got wide range of applications. They are used wherever lightness and good machinability are needed- in aircraft industry, in parts of cars and sports equipment to notebook or phone cases. Typical production methods for components made of magnesium alloys are casting methods. They offer advantages such as higher production rate, production of complex shapes and near net shape production (BAKER, H. ET AL. 1999, MURUGAN, G. ET AL. 2009). Casting of magnesium alloys is connected with microstructural defects such as casting porosity. It is limiting factor for fatigue properties. Presented defects serve as fatigue crack initiation sites (WOLF, B. ET AL. 2004, GU, X.N. ET AL. 2010).

The following study is focused on observation and evaluation of changes in hardness of AZ31, AZ61 and AZ91 magnesium alloys due to precipitation hardening. Magnesium alloy AZ31has got very good ductility, good weldability due to lower content of aluminum and it is very well castable. In practice it is not heat treated, because it is not hardenable (HUANG G. ET AL. 2008, CHEN L. ET AL. 2017, PENG, J. ET AL. 2018). For this research it was chosen for comparison of hardness with hardenable alloys. AZ61 alloy replaces AZ91 in numerous applications where higher fracture toughness and increased ductility are required. It is achieved by adding lower content of aluminum, thereby is reduced content of brittle phase γ in microstructure. Products made of AZ61 are determined for use up to 120°C. Over this temperature there is occurred worsening of mechanical properties occurs. AZ91 is nowadays one of the most used alloys in automotive industry. It has got the best combination of properties such as castability, mechanical strength and quite good corrosion resistance from all of magnesium alloys.

Precipitation is the process of aging where the metastable supersaturated solution is broken down into a stable system composed of two phases. During mentioned process the hardness of solid solution is increased. During artificial aging the γ phase is excluded as continuous and discontinuous precipitate. Precipitation hardening of materials is used to improve their mechanical properties. It is well known that the age hardening of alloys can have profound effects on fracture toughness, ductility, susceptibility to stress corrosion cracking, etc. An alloy is precipitation hardenable when its yield strength increases with time at a constant temperature, after rapid cooling from a much higher temperature. The presence of precipitates in the matrix contributes to an increase in strength. Continuous precipitate has got higher effect than discontinuous.

2. Experimental part

Research works were conducted for AZ31, AZ61 and AZ91 magnesium alloys. Specimens were cut from ingots produced by gravity casting into a sand mold. The first step of the research was chemical composition analysis. Spark emission spectrometer SPECTROMAXx was used in the process. The chemical composition of investigated specimens is shown in Tab.1. Afterwards, the specimens were heat treated at temperature 400°C for 22 hours to achieve solution annealing. Values of annealing temperatures were selected on the basis of literary research and expert consultations. Heat treatment was carried out in a resistance furnace without a protective atmosphere. After solution annealing the specimens were cooled in water with temperature 60°C in order to preserve the supersaturated solid solution. The next step of the experiment was precipitation hardening alone. The temperature was constant at 250°C; time intervals were 15, 30, 60, 120, 240, 480, 960, 1920, 3840 and 7680 minutes. Magnesium alloys require a longer time to start precipitation hardening, so short-term annealing times are not relevant in terms of more pronounced hardness changes. For this reason, it was necessary to choose long-term residence times at the precipitation curing temperature in order to reliably record all real changes in hardness of the alloy examined during annealing. The last time interval is relatively high in order to confirm the decrease in hardness due to material overheating. Brinell hardness was measured on individual samples in the initial state, after solution annealing and after each precipitation hardening interval. Hardness measurements were carried out under the following conditions: diameter of the sintered carbide spindle - 2.5 mm, load force - 62.5 kp, time - 10 seconds.

Table 1. Chemical composition of specimens

Element / Alloy	Al	Zn	Mn	Si	P	Mg
AZ31	2.980	0.655	0.202	0.067	0.002	balance
AZ61	6.880	1.200	0.229	0.079	0.004	balance
AZ91	8.280	0.554	0.202	0.007	0.004	balance

3. Results and discussion

Observing material hardness changes serves as an effective tool for determining precipitation hardening parameters, such as temperature and time. Brinell hardness measurement was chosen based on experimental needs, as the goal was to evaluate overall changes in hardness in materials. If it were intended to determine the hardness of the individual phases, it would be more appropriate to measure Vickers microhardness. Average hardness values calculated from the 5 measurements are shown in Table 2. The hardness of the AZ31 and AZ61 alloy after dissolving annealing is slightly lower than in the starting state. On the contrary, the hardness of the AZ91 alloy compared to its initial state has increased. During solution annealing, all inhomogeneities of the dendritic structure are dissolved to obtain a saturated solid solution.

Table 2. Average hardness values

Type of alloy		AZ31	AZ61	AZ91
Hardness in initial state		54	62	54
Hardness after solution annealing		53	58	60
Hardness after precipitation hardening	15 min	58	65	65
	30 min	57	62	62
	60 min	57	65	65
	120 min	57	71	71
	240 min	56	76	76
	480 min	55	78	78
	960 min	53	71	71
	1920 min	53	77	77
	3840 min	56	71	71
	7680 min	53	71	71

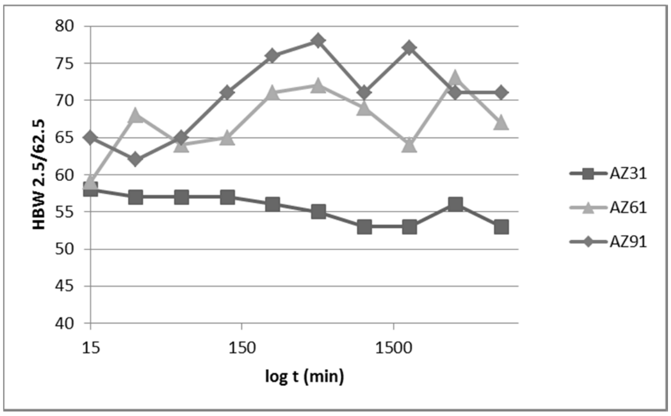
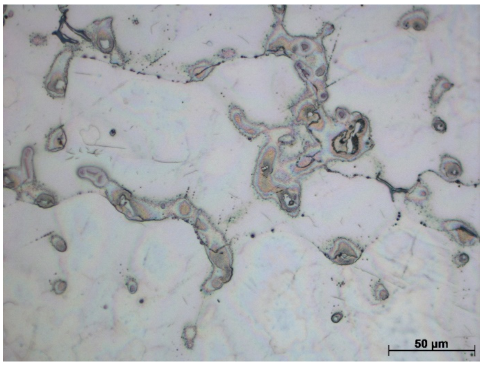


Fig 1. Changes in hardness of chosen magnesium alloys after heat treatment

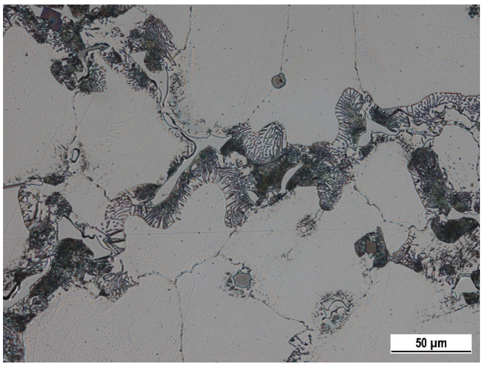
From the measured hardness values (Table 2) and the graph of the comparison of hardness changes (Fig 1.), it is evident that AZ31 is not precipitating hardenable. Hardness values are moving at values close to 55 HBW 2.5/62.5. The artificial aging process of AZ61 was similar to AZ91. The first increase in hardness was observed at 30 minutes when a discontinuous precipitate formed. Subsequently, the hardness dropped, but from 120 minutes it again increased up to 480 minutes. At this stage, the growth of the continuous precipitate was predicted. The last significant increase in hardness for AZ61 alloy was observed at 3840 minutes. During the last time interval, the hardness has decreased, which was caused by the material becoming stuck. The hardness of the AZ91 alloy increased gradually in time intervals between 30 to 480 minutes. At 960 minutes, the hardness decreased considerably, and increased again over the next time interval (1920 minutes). After this time, the hardness had only a decreasing character due to material overload.

It is important to observe microstructure of the materials for evaluation of changes taking place during heat treatment. Fig 2. shows microstructures of magnesium alloys AZ31, AZ61 and AZ91 in initial state. The microstructure of all three evaluated alloys in the initial state is formed by the δ phase. At the boundaries of the grains and in the interdendritic spaces, the exhausted phase γ is the electron-containing compound $Mg_{17}Al_{12}$. Other phases arising from the combination of additive elements in the matrix are present. For the observed types of magnesium alloys, the discontinuous precipitate is excreted along the borders of dendrites.

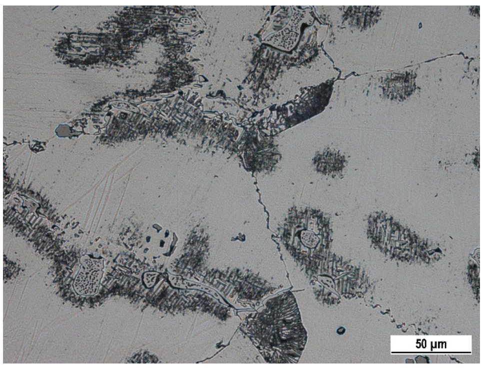
Images of magnesium alloy microstructures after T4 heat treatment under specified conditions: temperature 400°C, temperature endurance 22 hours, sample cooling to water with temperature 60°C, are shown in Fig 3. By heat treatment of T4, i.e., by dissolving annealing, parts of the intermetallic phases were dissolved in the microstructures AZ 31, AZ 61 and AZ 91 to produce polyhedral grains. Due to the absence of alloying elements which have not undergone heat treatment in the matrix, there is no discontinuous or continuous precipitate in the microstructures. The alloy microstructures consist of a primary solid magnesium solution and additive elements. In structures, there is a low percentage of undissolved phases at the grain boundaries.



a)

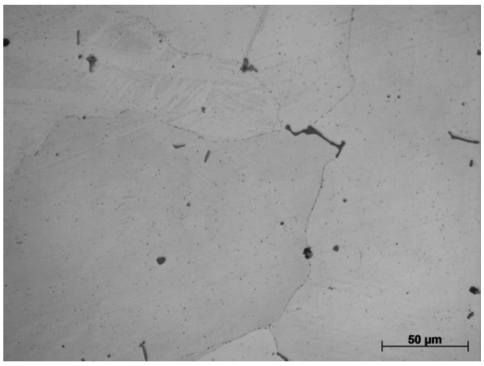


b)

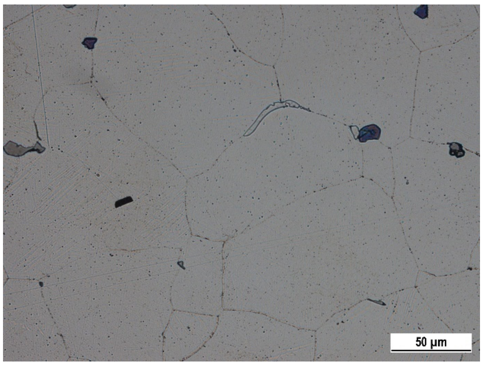


c)

Fig 2. Microstructures of magnesium alloys in initial state: a) AZ31, b) AZ61, c) AZ91



a)



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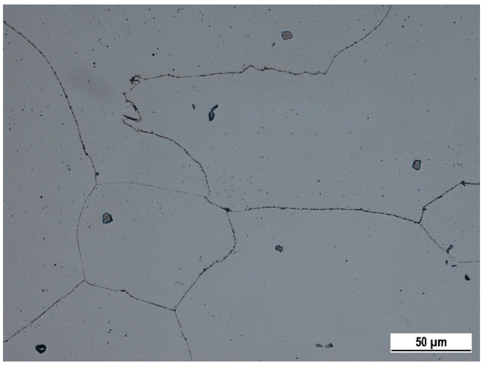

c)

Fig 3. Microstructures of magnesium alloys after T4 : a) AZ31, b) AZ61, c) AZ91

4. Summary and conclusion

The uneven increase in hardness during the precipitation hardening of the AZ61 and AZ91 alloys was probably due to structural changes that could have been caused by an inappropriately chosen temperature. For comparison, an example was given by the author Hlaváčová in her work (HLAVÁČOVÁ, I. 2014), that setting slightly different temperature intervals and the course of changes in alloy hardness AZ91 was of an exclusively increasing character. The dissolution annealing temperature was set at 420°C at room temperature, the temperature was 24 hours. The precipitation hardening temperature was 220°C, time intervals were similar to those in this study. It can be stated that the inappropriately chosen temperature significantly affects the results of the experiment.

Acknowledgements

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沉淀硬化引起的镁合金硬度变化

關鍵詞
镁合金
沉淀
硬度的变化
热处理

摘要
本文介绍了目前在不同领域广泛使用的沉淀硬化过程中镁合金硬度变化的评估。 它完全专注于 AZ31，AZ61 和 AZ91 合金。 观察材料硬度变化是确定沉淀硬化参数（如温度和时间）的有效工具。 根据实验需要选择布氏硬度测量。 还有必要进行化学成分分析并观察测试材料的微观结构。 所得结果在本文中给出和讨论。