UDC 669.721.5:621.785.7

Aikin M. Senior lecturer of Physical Materials Science Department, National University Zaporizhzhia Polytechnic, Zaporizhzhia, Ukraine, *e-mail: fitone14@gmail.com*, ORCID: 0000-0001-9513-2804

Shalomeev V. Doctor of Technical Sciences, Professor, Prorector, National University Zaporizhzhia Polytechnic, Zaporizhzhia, Ukraine, *e-mail: shalomeev@zntu.edu.ua*, ORCID: 0000-0002-6091-837X

OPTIMIZATION OF HEAT TREATMENT REGIME FOR A NEW BIODEGRADABLE MG-ZR-ND ALLOY WITH ENHANCED MECHANICAL PROPERTIES

Purpose. *To develop a rational heat treatment regime for a new biodegradable magnesium alloy of the Mg-Zr-Nd system, to ensure enhanced mechanical properties throughout the entire treatment period.*

Research methods. *Differential thermal analysis (DTA) was used to determine phase transformation temperatures. Microstructure analysis was conducted using optical microscopy ("Neophot 32" and "OLYMPUS IX 70") and scanning electron microscopy with energy-dispersive X-ray spectroscopy (SELMI REM-106I). Mechanical properties were determined using an INSTRON 2801 testing machine. The influence of cooling rate on microstructure and properties was studied using ProCAST simulation software. Heat treatment was carried out in a Bellevue type shaft furnace and a PAP-4M furnace. X-ray analysis was used to detect internal defects in samples.*

Results. *A new heat treatment regime was developed for the biodegradable Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy. Using differential thermal analysis and microstructure studies at various quenching temperatures, the optimal quenching temperature was established at 560 °C. Empirical relationships describing the influence of heat treatment parameters on the alloy's microstructure were calculated. The new heat treatment regime (quenching from 560 °C for 8 hours, air cooling + aging at 200 °C for 16 hours) resulted in improved mechanical properties (UTS = 276–282 MPa, δ = 5.2–5.8%) compared to the standard T6 regime.*

Scientific novelty. *For the first time, a comprehensive study of the influence of heat treatment parameters on the structure and properties of a new Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy with increased content of alloying elements was conducted. New dependencies describing the influence of quenching temperature on the alloy's grain size were established.*

Practical value. *A new heat treatment regime for the biodegradable magnesium alloy was developed, which ensures complete dissolution of the pseudoeutectic phase and formation of strengthening phases, resulting in improved mechanical properties compared to the standard alloy Mg-2.5Nd-0.4Zn-0.5Zr (wt%) and the standard T6 regime.*

Key words: *biodegradable magnesium alloy, Mg-Zr-Nd system, heat treatment, quenching temperature, mechanical properties, microstructure, pseudoeutectic phase.*

Introduction

Magnesium alloys of the Mg-Zr-Nd system have significant potential for use as biodegradable implants in osteosynthesis due to their unique combination of properties [1]. However, existing industrial alloys of this system, such as Mg-2.5Nd-0.4Zn-0.5Zr (wt%) (formerly known as ML10), do not always provide the optimal combination of mechanical characteristics and biodegradation rate required for effective application in medical practice [2, 3].

One of the key factors influencing the properties of magnesium alloys is their heat treatment regime. The standard T6 heat treatment regime [4, 5], widely used for Mg-Zr-Nd system alloys, does not always ensure complete dissolution of pseudo-eutectic phases and formation of optimal microstructure in alloys with increased content of alloying elements [5, 6].

The aim of this study is to develop a rational heat treatment regime for a new biodegradable magnesium alloy of the Mg-Zr-Nd system with increased content of alloying elements - Mg-3.15Nd-1.25Zr-0.6Zn (wt%) [3]. To achieve this goal, the following objectives were set:

1. Investigate the effect of the standard T6 heat treatment regime on the microstructure of the developed alloy.
2. Determine the phase transformation temperatures of the new alloy using computational methods and differential thermal analysis.
3. Study the influence of quenching temperature on grain size and the amount of secondary phases in the new alloy.
4. Develop an optimized heat treatment regime that provides an enhanced complex of mechanical properties for the new alloy.
5. Compare the mechanical properties of the new alloy after optimized heat treatment with the standard Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy.

DOI 10.15588/1607-6885-2024-3-5

The results of this study will allow the development of a rational heat treatment regime for the new biodegradable magnesium alloy, ensuring an optimal combination of strength, plasticity, and corrosion resistance necessary for effective application in osteosynthesis.

Analysis of research and publications

The development of biodegradable magnesium alloys for medical applications, particularly in osteosynthesis, has been a subject of extensive research in recent years. The Mg-Zr-Nd system alloys have shown promising results due to their favorable combination of mechanical properties, corrosion resistance, and biocompatibility [7, 8, 9, 10].

The standard heat treatment regime T6 (quenching + aging) has been widely used for Mg-Zr-Nd alloys to improve their mechanical properties and corrosion resistance [11]. This regime typically involves heating to 540 ± 5 °C, holding for 8 hours followed by air cooling, and then aging at 200 ± 5 °C for 16 hours with air cooling [4, 5]. The improvement in properties is achieved through the dissolution of non-equilibrium pseudo-eutectic and the precipitation of clusters of secondary phase particles, consisting of finely dispersed Zn_2Zr_3 intermetallics, β'' and β' phases [12, 13].

The influence of heat treatment parameters on the microstructure and properties of magnesium alloys has been a focus of several studies. It has been established that the quenching temperature can significantly affect the grain size of magnesium alloys, with higher temperatures generally leading to grain growth [14]. However, the relationship between quenching temperature and grain size can be complex, especially in alloys with high content of alloying elements.

The role of alloying elements, particularly Nd and Zr, in the formation of strengthening phases and their effect on the heat treatment process has been discussed in literature [15–18]. The concentration of Nd close to its solubility limit can lead to the formation of a larger amount of pseudo-eutectic in the alloy, which affects the mechanical properties and requires adjustment of the heat treatment regime.

Despite the extensive research in this field, there is still a need for systematic studies on the optimization of heat treatment regimes for new biodegradable Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy with increased content of alloying elements. This research aims to address this gap by developing a rational heat treatment regime for a new Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy, taking into account its specific composition and desired properties for osteosynthesis applications.

Purpose

The primary purpose of this study is to develop and optimize a heat treatment regime for a new biodegradable magnesium Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy with increased alloying content. This research aims to address the limitations of the standard T6 heat treatment when applied to alloys with higher concentrations of alloying elements. By systematically investigating the effects of heat treatment parameters on the alloy's microstructure and properties, this study seeks to establish a rational heat treatment process that ensures complete dissolution of pseudo-eutectic phases and formation of an optimal microstructure. The ultimate goal is to achieve an enhanced combination of strength, ductility, and corrosion resistance in the new Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy, making it more suitable for osteosynthesis applications compared to existing commercial alloys such as Mg-2.5Nd-0.4Zn-0.5Zr (wt%).

Research material and methodology

The research was conducted on newly developed biodegradable magnesium alloys of the Mg-Zr-Nd system [3]. The experimental alloys had varying contents of alloying elements within the following ranges: 1.2–1.3% Zr, 3.1–3.2% Nd, and 0.5–0.7% Zn. The standard alloy (0.4–1.5% Zr, 2.2–3.4% Nd, and 0.1–0.7% Zn) was used for comparison in various experiments.

Alloys were melted in an IPM-500 crucible furnace with a capacity of 0.5 tons, power of 140 kW, and productivity of 230 kg/hour. The melt was refined using VI 2 flux [19] and cast into removable crucibles at 650-730°C. Ligatures containing Zr, Nd, and Zn were added to adjust the composition.

Heat treatment was performed in a Bellevue type shaft furnace (112 kW, 95 kg/hour productivity) and a PAP-4M type furnace (50 kg/hour productivity) under an argon protective atmosphere. The standard T6 heat treatment regime for Mg-Zr-Nd alloys was applied according to existing standards [4, 5].

The ProCAST software package, including the COMPUTHERM module, was used to estimate material properties.

Chemical composition was determined using standard methods. Sample quality was assessed visually and using X-ray methods (RAP-150/300, RUP 150/300, RUP 400-5, and MIRA-2D devices).

Mechanical properties were determined using an INSTRON 2801 testing machine according to existing standards [4, 19].

Macro- and microstructures were studied using Neophot 32 and OLYMPUS IX 70 optical microscopes at magnifications of 100, 200, 350, and 500 times. Grain size was determined according to [20]. Phase composition was quantitatively assessed using the Rosiwal linear method.

Phase analysis of structural components was performed using a SELMI REM-106I scanning electron microscope with energy-dispersive microanalysis.

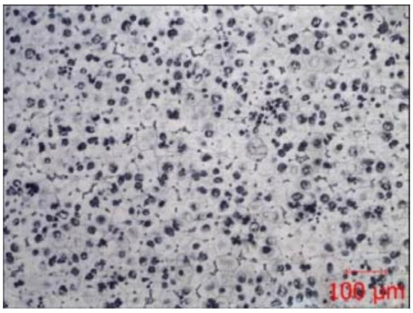
Differential Thermal Analysis (DTA) was performed using a VDTA-8 installation in an argon atmosphere with constant heating and cooling rates of 80 °C/min. The maximum heating temperature was 800 °C.

Results and their discussion

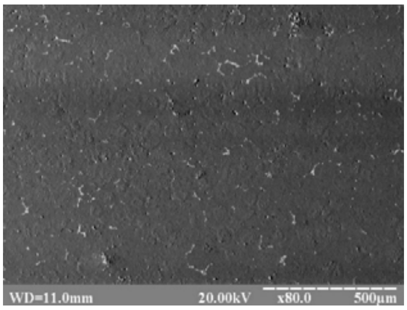
The study of the microstructure of the experimental biodegradable alloy after heat treatment showed that although fine dispersed strengthening phases were formed in

DOI 10.15588/1607-6885-2024-3-5

the structure (Fig. 1*a*), residual precipitates of pseudo-eutectic that did not dissolve during quenching were observed at the grain boundaries (Fig. 1*b*). This undesirable phenomenon was not observed in the standard Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy, indicating a potential issue specific to the experimental alloy's composition or heat treatment process.



a



b

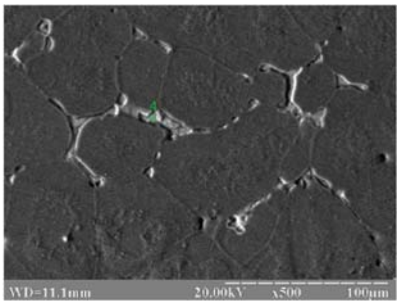
Figure 1. Microstructure of the experimental biodegredable alloy after standard heat treatment T6, ×100 (*a*) and pseudo-eutectic precipitates, ×80 (*b*)

Using micro-X-ray spectral analysis (Fig. 2), it was determined that the residual pseudo-eutectic at the grain boundaries has a composition of Mg-38.47Nd-1.06Zn-0.1Zr and corresponds to a (Mg, Zn)12Nd-type phase.

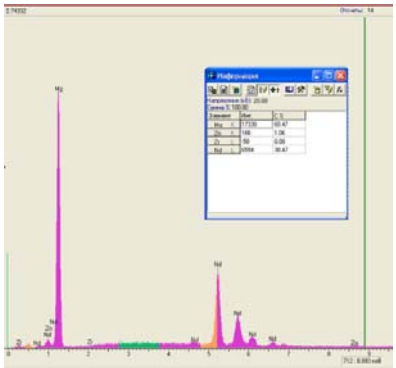
Such pseudo-eutectic precipitates after T6 heat treatment were observed in similar alloys [5, 6]. The authors of these works attribute this phenomenon to insufficiently high heating temperature of the alloy before quenching, insufficient holding time at this temperature, or slow cooling.

Given that the experimental biodegradable alloy contains a concentration of Nd (3.1%) close to the solubility limit (3.6%), this leads to the formation of a larger amount of pseudo-eutectic in the developed alloy compared to the standard Mg-2.5Nd-0.4Zn-0.5Zr (wt%). The presence of pseudo-eutectic in the structure reduces the threshold of mechanical properties of the alloy by decreasing the amount of Nd and Zn that form the strengthening phase. We investigated the potential for achieving complete dissolution of the residual pseudo-eutectic by exploring the effects of increased heating temperatures prior to quenching.

To determine the possibility of increasing the heating temperature before quenching, solidus and liquidus temperatures were calculated using the "COMPUTHERM" calculation module, which is part of the "ProCAST" software package.



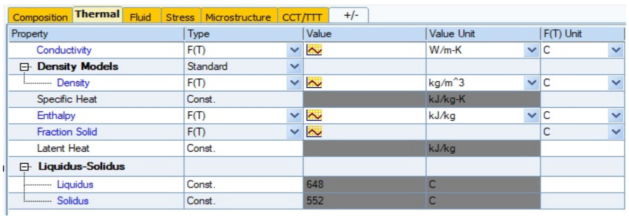
a



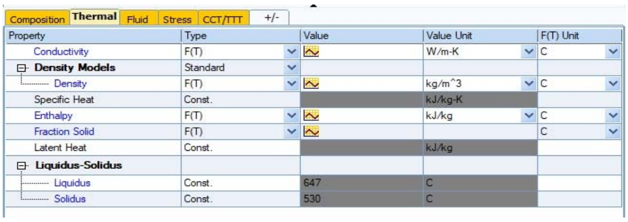
b

Figure 2. Micro-X-ray spectral analysis of the experimental biodegredable alloy after heat treatment T6

For the developed alloy, the solidus temperature was Tsol = 552 °C, and the liquidus temperature Tliq = 648 °C (Fig. 3, a). For comparison, temperature calculations were performed for the standard alloy of the Mg-Zr-Nd system — Mg-2.5Nd-0.4Zn-0.5Zr (wt%): Tsol = 530 °C, Tliq = 647 °C (Fig. 3*b*). The shift in solidus temperature was +22 °C, and in liquidus temperature +1 °C. Thus, the calculations determined that increasing the alloying elements Nd and Zr leads to an increase in the solidus temperature and has almost no effect on the liquidus temperature.



a



b

Figure 3. Working window of the "COMPUTHERM" module, showing the results for (*a*) the developed alloy and (*b*) the standard Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy

DOI 10.15588/1607-6885-2024-3-5

For practical determination of the optimal heating temperature before quenching of the new Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy, differential thermal analysis (DTA) was used. The graph (Fig. 4) shows DTA curves for the developed alloy in comparison with the standard Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy.

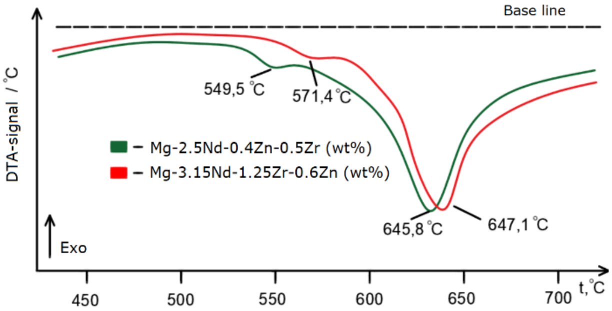


Figure 4. DTA curves of the standard Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy and the new non-heat-treated Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy

The curves for both alloys were similar and had 2 peaks: the first corresponds to the melting of the non-equilibrium pseudo-eutectic; the second corresponds to the melting of the alloy itself.

The DTA results (Table 1) correspond well with the "COMPUTHERM" calculations. The melting temperature of the pseudo-eutectic in the experimental biodegradable alloy was 571.4 °C, which is 21.9 °C higher than the corresponding temperature for the standard Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy – 549.5 °C. The melting temperature of the new Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy - 647.1 °C, slightly (by 1.3 °C) exceeded the Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy - 645.8 °C. Thus, to ensure the most complete dissolution of the pseudo-eutectic without overheating and melting, the heating temperature before quenching of the new Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy was set at Tquench = 560 °C.

Table 1 – DTA results

Alloy	Temperatures, °C		
	Heating for quenching	Melting of pseudo-eutectic	Melting of alloy
New Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy	560	571,4	647,1
Standard Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy	540	549,5	645,8

As being known, increasing the heating temperature before quenching can affect the grain size of magnesium alloys, causing it to increase [21], which negatively impacts mechanical properties, both strength and plasticity.

To determine the effect of heating temperature before quenching on the grain size of the new alloy, experimental samples in the form of discs with a height of 10 mm and a diameter of 20 mm were subjected to heat treatment according to the T4 regime (quenching, air cooling). To study the dynamics of changes in average grain size and the amount of secondary phase (pseudo-eutectic) in the new alloy, the following heating temperatures before quenching were selected: 400 °C, 450 °C, 500 °C, 540 °C, 560 °C (Fig. 5). The microstructure of the obtained samples was compared with samples of the standard Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy after quenching from 400 °C, 450 °C, 500 °C, and 540 °C (Fig. 6). The 50 °C step is sufficient to detect changes in the microstructure. Temperatures of 540 °C and 560 °C were chosen based on the DTA results (Table 1) and are optimal for heating before quenching for the developed alloy and Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy respectively.

The microstructure of the experimental alloys consisted of solid solution grains, precipitates of undissolved pseudo-eutectic $(Mg, Zn)_{12}Nd$ (dark areas) formed during quenching, and Zn_2Zr_3 intermetallics within the grain body that formed during quenching. At temperatures of 400 °C, 450 °C, 500 °C, precipitates of undissolved pseudo-eutectic were observed in the structures of both alloys (Fig. 5*a–c*, Fig. 6*a–c*), and in the structure of the new alloy, they were also observed at 540 °C (Fig. 5*d*), while at this temperature in the standard alloy, the pseudo-eutectic was almost completely dissolved (Fig. 6*d*). When quenching the new alloy from 560 °C, the eutectic at the grain boundaries was practically absent (Fig. 5*e*).

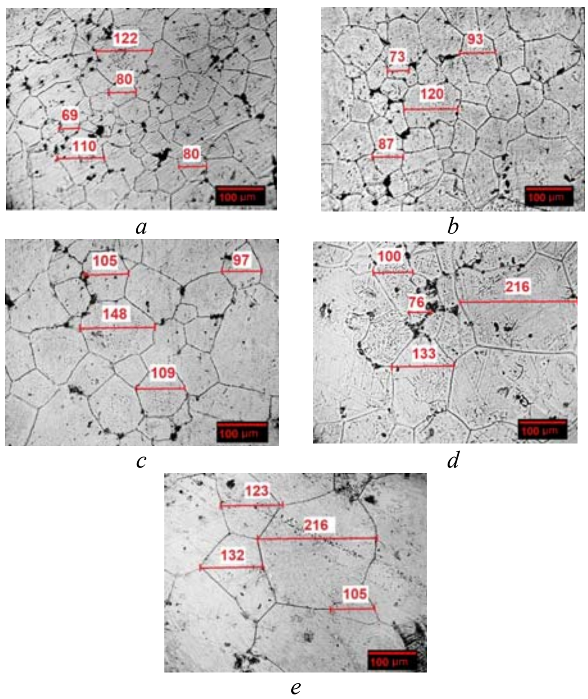


Figure 5. Microstructures of the developed alloy at different quenching temperatures, ×350

DOI 10.15588/1607-6885-2024-3-5

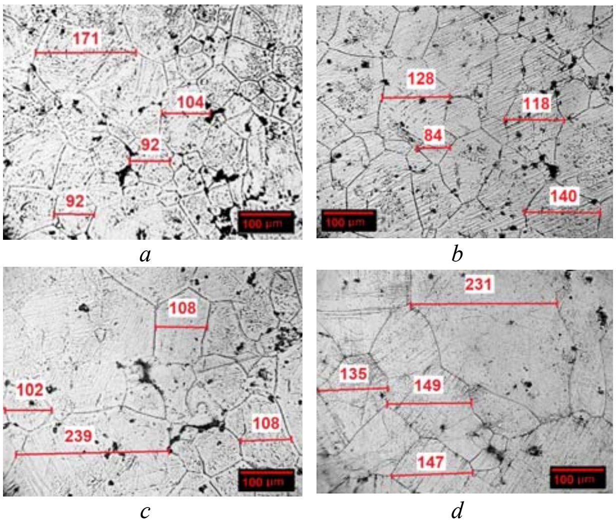


Figure 6. Microstructures of the standard alloy at different quenching temperatures, ×350

Quantitative analysis of the microstructures of the experimental alloys after different quenching regimes (Table 2) showed that the grain size of the developed alloy, at quenching heating temperatures from 400 to 540 °C, was lower compared to the Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy by 5.1–28.8 µm. When increasing the heating temperature before quenching of the experimental biodegradable alloy from 540 °C to 560 °C, the average grain size increased by 15.7 µm. At the same time, at a temperature of 560 °C, the grain size of the experimental biodegradable alloy was lower than the grain size of the Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy at 540 °C by 13.1 µm. Dependencies that most fully describe the change in the average grain size of the alloy with changing heating temperature during quenching were constructed for the experimental biodegradable alloy (1) and the standard Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy (2). The dependencies have the form of exponential regression equations.

Table 2 – Average grain size of experimental alloys at different quenching temperatures

Alloy	Average grain size at a certain heating temperature for quenching, µm				
	400 °C	450 °C	500 °C	540 °C	560 °C
New Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy	96,2	107,4	120	137,9	153,6
Standard Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy	101,6	112,5	129,9	166,7	-

$$D_{new\ alloy} = 30{,}4 \times e^{0{,}003\times T_{quench}} \pm 4{,}7,\ \mu m \qquad (1)$$

$R = 0{,}985$; $R^2 = 0{,}969$; $p = 0{,}95$

$$D_{STD} = 24{,}9 \times e^{0{,}003\times T_{quench}} \pm 9{,}1,\ \mu m \qquad (2)$$

$R = 0{,}966$; $R^2 = 0{,}932$; $p = 0{,}95$

The graphical representation of the dynamics of grain size changes (Fig. 7) shows that at heating temperatures < 500 °C, the rate of grain growth is almost the same. At heating temperatures > 500 °C, the grain of the standard Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy grows more significantly. These results can be explained by the increased modifying ability of Zr, the content of which is significantly higher in the new Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy, providing a finer initial structure due to an increase in the number of nuclei of new grains, as well as a greater amount of Nd in the solid solution and the formation of a larger number of Zn_2Zr_3 zirconides, which together leads to an increase in the heat resistance of the alloy [22].

The microstructure of the new Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy after the modified T6 regime—heating to 560 °C, holding for 8 hours, air cooling + aging at 200 °C for 16 hours (Fig. 8*a*) – exhibited clean, unmelted grain boundaries without noticeable residual pseudo-eutectic precipitates. The structure revealed a significant quantity of spherical clusters of secondary phase particles, composed of Zn_2Zr_3 intermetallics and strengthening β'' and β' phases of Mg_3Nd and Mg_7Nd types. The quantity and dispersion of secondary phases were notably higher compared to the alloy structure treated with the standard T6 regime (quenching from 540°C + aging) (Fig. 8*b*). Concurrently, no significant grain growth was observed relative to the standard regime.

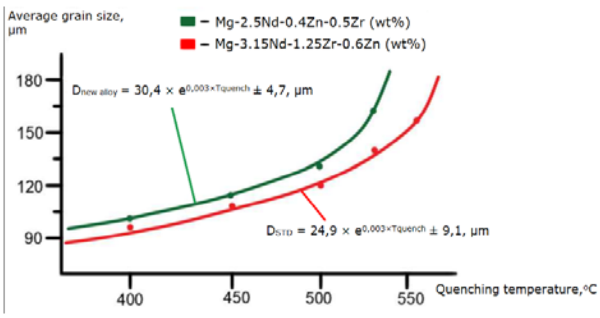


Figure 7. Dynamics of changes in the average grain size of experimental alloys at different heating temperatures for quenching

Following the increase in heating temperature before quenching, the ultimate tensile strength of the new alloy, compared to the standard T6 heat treatment, increased from 265–270 MPa to 276–282 MPa (an average increase of 9.5%), while the elongation increased from 4.5–5% to 5.2–5.8% (an average increase of 13%) (Table 3). Relative to the standard Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy (Fig. 8*c*), the property improvements were 18% and 31%, respectively. Thus, despite the slightly larger average grain size of the alloy treated with the new regime, the positive effect of more complete dissolution of the pseudo-eutectic outweighed the negative effect of increased grain size.

DOI 10.15588/1607-6885-2024-3-5

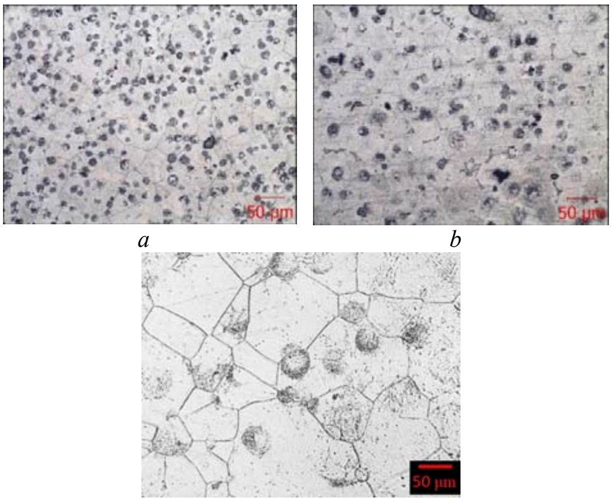


Figure 8. Microstructures of the studied alloys after aging, ×200: developed alloy processed using the new mode (*a*), developed alloy processed by the standard mode (*b*), standard Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy, processed according to the standard mode (*c*)

Table 3 – Mechanical properties of the studied alloys depending on the heat treatment mode

Alloy	Heat treatment regime	Physical and mechanical properties	
		UTS, MPa	δ, %
Standard Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy	Quenching from 540 ºC, aging	235–240	3,0–4,0
New Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy	Quenching from 540 ºC, aging	265–270	4,5–5,0
	Quenching from 560 ºC, aging	276–282	5,2–5,8

Based on the research results, the following heat treatment regime is recommended for the new Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy: heating to 560 ± 5 °C, holding for 8 hours followed by air cooling, and aging at 200 ± 5 °C for 16 hours followed by air cooling.

In conclusion, the study of the influence of technological factors on the microstructure and properties of the experimental biodegradable alloy has established their optimal parameters. To explore the possibility of further enhancing the overall property complex of the alloy, it is advisable to conduct industrial trials of the alloy with application of investigated methods.

Conclusions

The present study on the optimization of heat treatment for a new biodegradable Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy has yielded several important findings:

1. Increasing the concentration of chemical elements in the experimental biodegradable alloy leads to an increase in the amount of pseudo-eutectic $(Mg, Zn)_{12}Nd$ precipitates and insufficient dissolution during homogenization. This issue is addressed by increasing the holding temperature before quenching.

2. Using the "COMPUTHERM" module and differential thermal analysis, the melting temperatures of the pseudo-eutectic and the new alloy were predicted to be 571.4 °C and 647.1 °C, respectively. Based on these data, the optimal heating temperature before quenching for the experimental biodegradable alloy was determined to be T_{quench} = 560 °C.

3. The grain size of the new Mg-3.15Nd-1.25Zr-0.6Zn (wt%) alloy, at quenching temperatures from 400 to 540 °C, was lower compared to the Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy by 5.1–28.8 µm. When increasing the heating temperature before quenching of the experimental biodegradable alloy from 540 °C to 560 °C, the average grain size increased by 15.7 µm. At 560 °C, the grain size of the experimental biodegradable alloy was 13.1 µm smaller than that of the Mg-2.5Nd-0.4Zn-0.5Zr (wt%) alloy at 540 °C. Complete dissolution of the pseudo-eutectic in the microstructure of the experimental biodegradable alloy was observed only at a holding temperature of 560 °C.

4. For the experimental biodegradable alloy, the following heat treatment regime is recommended: heating to 560 ± 5 °C, holding for 8 hours followed by air cooling, and aging at 200 ± 5 °C for 16 hours followed by air cooling. This regime ensures a high complex of mechanical properties for the alloy when cast in a sand mold: ultimate tensile strength UTS = 276–282 MPa, elongation δ = 5.2–5.8%. Compared to the previous heat treatment regime, the alloy's ultimate tensile strength increased by 6–17 MPa, and the relative elongation increased by 0.2–1.3%.

References

1. Shalomeev, V.A., Tsyvirko, E.I., Aikin, M.D., Chornyy, V.V. (2017). Perspektyvy vykorystannya biorozchynnykh mahniyevykh splaviv dlya osteosyntezu [Prospects for using biodegradable magnesium alloys for osteosynthesis]. Metaloznavstvo ta obrobka metaliv, 1, 39–44.

2. Aikin, M.D., Shalomeev, V.A., Tsyvirko, E.I. (2019). Biorozchynnyy lyvarnyy splav medychnoho pryznachennya na osnovi mahniyu [Biodegradable cast magnesium-based alloy for medical purposes]. Metaloznavstvo ta obrobka metaliv, 25(92), 38–46.

3. Shalomeev, V.A., Tsivirko, E.I., Aikin, N.D. (2019). High-quality magnesium-based alloys with improved properties for engineering. Innovative materials and technologies in metallurgy and mechanical engineering, 1, 56–61.

4. DSTU EN 1753:2022 (EN 1753:2019, IDT). (2022). Mahniy ta mahniyevi splavy. Zlyvky ta vylyvky iz mahniyevykh splaviv [Magnesium and magnesium alloys. Magnesium alloy ingots and castings]. Kyiv: DP "UkrNDNTS".

5. DSTU ISO 16220:2008. (2013). Mahniy i mahniyevi splavy. Zlyvky ta vylyvky z mahniyevykh splaviv. Tekhnichni umovy [Magnesium and magnesium alloys. Magnesium alloy ingots and castings. Specifications] (ISO 16220:2005, IDT). Kyiv: Derzhspozhyvstandart Ukrayiny.

DOI 10.15588/1607-6885-2024-3-5

6. Wu, D., Ma, Y.Q., Chen, R.S., Ke, W. (2014). Effect of heat treatment on the microstructures and mechanical properties of the sand-cast Mg-2.7Nd-0.6Zn-0.5Zr alloy. Journal of Magnesium and Alloys, 2, 20–26.

7. Waizy, H., Diekmann, J., et al. (2014). In vivo study of a biodegradable orthopedic screw (MgYREZr-alloy) in a rabbit model for up to 12 months. Journal of Biomaterials Applications, 28(5), 667-675.

8. Feyerabend, F., Fischer, J., et al. (2010). Evaluation of short-term effects of rare earth and other elements used in magnesium alloys on primary cells and cell lines. Acta Biomaterialia, 6, 1834–1842.

9. Zhang, X., Yuan, G., et al. (2012). Effects of extrusion and heat treatment on the mechanical properties and biocorrosion behaviors of a Mg-Zn-Zn-Zr alloy. Journal of the Mechanical Behavior of Biomedical Materials, 7, 77–86.

10. Marukawa, E., Tamai, M., et al. (2015). Comparison of magnesium alloys and poly-L-lactide screws as degradable implants in a canine fracture model. Journal of Biomedical Materials Research B: Applied Biomaterials, 1–8.

11. Zheng, X., Luo, P., Dong, J., Wang, S. (2019). The effect of casting speed on microstructure, microsegregation, and mechanical properties of high-strength Mg-Nd-Zn-Zr alloy. Journal of Materials Engineering and Performance, 1–9. https://doi.org/10.1007/s11665-019-3878-0

12. Liu, S.J., Yang, G.Y., Luo, S.F., Jie, W.Q. (2015). Microstructure evolution during heat treatment and mechanical properties of Mg-2.49Nd-1.82Gd-0.19Zn-0.4Zr cast alloy. Materials Characterization, 107, 334–342.

13. Fu, P.H., Peng, L.M., Jiang, H.Y., Ma, L., Zhai, C.Q. (2008). Chemical composition optimization of gravity cast Mg-yNd-xZn-Zr alloy. Materials Science and Engineering: A, 496(1-2), 177–188.

14. Jie, W., Qudong, W., Bing, Y., Haiyan, J., Wenjiang, D. (2019). Effect of heat treatment on the microstructure and mechanical property of vacuum die-casting NZ30K Mg-alloy. Chinese Journal of Materials Research, 33(1), 1–8.

15. Shi, W., Ma, Y. (2013). Microstructure of ZM6 magnesium alloy with different Nd content. Rare Metals, 32(3), 234–240.

16. Penghuai, F., Liming, P., Haiyan, J., Jianwei, C., Chunquan, Z. (2008). Effects of heat treatments on the microstructures and mechanical properties of Mg-3Nd-0.2Zn-0.4Zr (wt. %) alloy. Materials Science and Engineering: A, 486, 183–192.

17. Zhu, S., Eastom, M.A., Abbot, T.B., Gibson, M.A., Nie, J. (2016). The influence of individual rare earth elements (La, Ce, of Nd) on creep resistance of die-cast magnesium aloe AE44. Advanced Engineering Materials, 18(6), 932–937.

18. Li, J., Chen, R., Ma, Y., Ke, W. (2013). Effect of Zr modification on solidification behavior and mechanical properties of Mg-Y-Re (WE54) alloy. Journal of Magnesium and Alloys, 1, 346–351.

19. DSTU ISO 6892-1:2019 (ISO 6892-1:2016, IDT). (2019). Metalevi materialy. Vyprobuvannya na roztyah. Chastyna 1. Metod vyprobuvannya za kimnatnoyi temperatury [Metallic materials – Tensile testing – Part 1: Method of test at room temperature]. Kyiv: DP "UkrNDNTS".

20. DSTU 8972:2019. (2021). Stali ta splavy. Metody vyyavlennya ta vyznachennya velychyny zerna [Steels and alloys. Methods for detecting and determining grain size]. Kyiv: DP "UkrNDNTS".

21. Avedesian, M., & Baker, H. (Eds.). (1999). ASM Specialty Handbook: Magnesium and Magnesium Alloys. Materials Park, OH: ASM International.

22. Wu, D., Chen, R.S., Ke, W. (2014). Microstructure and mechanical properties of a sand-cast Mg-Nd-Zn alloy. Materials and Design, 58, 324–331.

Одержано 12.09.2014

ОПТИМІЗАЦІЯ РЕЖИМУ ТЕРМІЧНОЇ ОБРОБКИ НОВОГО БІОДЕГРАДУЮЧОГО СПЛАВУ Mg-Zr-Nd З ПІДВИЩЕНИМИ МЕХАНІЧНИМИ ВЛАСТИВОСТЯМИ

Айкін М. Д. — Старший викладач кафедри фізичного матеріалознавства, Національний університет «Запорізька політехніка», Запоріжжя, Україна, *e-mail: fitone14@gmail.com*, ORCID: 0000-0001-9513-2804

Шаломєєв В. А. — Д-р техн. наук, професор, Національний університет «Запорізька політехніка», Запоріжжя, Україна, *e-mail: shalomeev@zntu.edu.ua*, ORCID: 0000-0002-6091-837X

Мета. *Розробити раціональний режим термічної обробки нового біодеградуючого магнієвого сплаву системи Mg-Zr-Nd для забезпечення підвищених механічних властивостей протягом всього періоду обробки.*

Методи дослідження. *Диференціально-термічний аналіз (ДТА) використовувався для визначення температур фазових перетворень. Аналіз мікроструктури проводився за допомогою оптичної мікроскопії («Neophot 32» та «OLYMPUS IX 70») та скануючої електронної мікроскопії з енергодисперсійною рентгенівською спектроскопією (SELMI PEM-106I). Механічні властивості визначалися на випробувальній машині INSTRON 2801. Вплив швидкості охолодження на мікроструктуру та властивості вивчався за допомогою програмного забезпечення ProCAST. Термічна обробка проводилася в шахтній печі типу Bellevue та печі ПАП-4М. Рентгенівський аналіз використовувався для виявлення внутрішніх дефектів у зразках.*

DOI 10.15588/1607-6885-2024-3-5

Результати. *Розроблено новий режим термічної обробки для біодеградуючого сплаву Mg-3,15Nd-1,25Zr-0,6Zn (мас.%). За допомогою диференціально-термічного аналізу та дослідження мікроструктури при різних температурах гартування встановлено оптимальну температуру гартування 560°С. Розраховано емпіричні залежності, що описують вплив параметрів термічної обробки на мікроструктуру сплаву. Новий режим термічної обробки (гартування від 560°С протягом 8 годин, охолодження на повітрі + старіння при 200°С протягом 16 годин) призвів до покращення механічних властивостей (σ_B = 276-282 МПа, δ = 5,2–5,8%) порівняно зі стандартним режимом Т6.*

Наукова новизна. *Вперше проведено комплексне дослідження впливу параметрів термічної обробки на структуру та властивості нового сплаву Mg-3,15Nd-1,25Zr-0,6Zn (мас.%) з підвищеним вмістом легуючих елементів. Встановлено нові залежності, що описують вплив температури гартування на розмір зерна сплаву.*

Практична цінність. *Розроблено новий режим термічної обробки біодеградуючого магнієвого сплаву, який забезпечує повне розчинення псевдоевтектичної фази та формування зміцнюючих фаз, що призводить до покращення механічних властивостей порівняно зі стандартним сплавом Mg-2,5Nd-0,4Zn-0,5Zr (мас.%) та стандартним режимом Т6.*

Ключові слова: *біодеградуючий магнієвий сплав, система Mg-Zr-Nd, термічна обробка, температура гартування, механічні властивості, мікроструктура, псевдоевтектична фаза.*

Список літератури

1. Перспективи використання біорозчинних магнієвих сплавів для остеосинтезу [Текст] / В. А. Шаломєєв, Е. І. Цивірко, М. Д. Айкін, В. В. Чорний // Металознавство та обробка металів. – 2017. – № 1. – С. 39–44.

2. Айкін М. Д. Біорозчинний ливарний сплав медичного призначення на основі магнію [Текст] / М. Д. Айкін, В. А. Шаломєєв, Е. І. Цивірко // Металознавство та обробка металів. – 2019. – Т. 25 (92). – С. 38–46.

3. Shalomeev V.A. High-quality magnesium-based alloys with improved properties for engineering [Text] / V.A. Shalomeev, E.I. Tsivirko, N.D. Aikin // Innovative materials and technologies in metallurgy and mechanical engineering. – 2019. – No 1. – P. 56–61.

4. ДСТУ EN 1753:2022. Магній та магнієві сплави. Зливки та виливки із магнієвих сплавів [Текст] [Чинний від 31.12.2023]. – К. : ДП «УкрНДНЦ», 2022.

5. ДСТУ ISO 16220:2008. Магній і магнієві сплави. Зливки та виливки з магнієвих сплавів. Технічні умови [Текст] [Чинний від 31.12.2023]. – К. : Держспоживстандарт України, 2013.

6. Wu D. Effect of heat treatment on the microstructures and mechanical properties of the sand-cast Mg-2.7Nd-0.6Zn-0.5Zr alloy [Text] / D. Wu, Y.Q. Ma, R.S. Chen, W. Ke // Journal of Magnesium and Alloys. – 2014. – No 2. – P. 20–26.

7. Waizy H. In vivo study of a biodegradable orthopedic screw (MgYREZr-alloy) in a rabbit model for up to 12 months [Text] / H. Waizy, J. Diekmann [et. al.] // Journal of Biomaterials Applications. – 2014. – No 28 (5). – P. 667–675.

8. Feyerabend F. Evaluation of short-term effects of rare earth and other elements used in magnesium alloys on primary cells and cell lines [Text] / F. Feyerabend, J. Fischer [et. al.] // Acta Biomaterialia. – 2010. – No 6. – P. 1834–1842.

9. Effects of extrusion and heat treatment on the mechanical properties and biocorrosion behaviors of a Mg-Zn-Zn-Zr alloy [Text] / X. Zhang, G. Yuan [et. al.] // Journal of the Mechanical Behavior of Biomedical Materials. – 2012. – No 7. – P. 77–86.

10. Comparison of magnesium alloys and poly-L-lactide screws as degradable implants in a canine fracture model [Text] / E. Marukawa, M. Tamai [et. al.] // Journal of Biomedical Materials Research B: Applied Biomaterials. – 2015. – P. 1–8.

11. The effect of casting speed on microstructure, microsegregation, and mechanical properties of high-strength Mg-Nd-Zn-Zr alloy [Text] / X. Zheng, P. Luo, J. Dong, S. Wang // Journal of Materials Engineering and Performance. – 2019. – P. 1–9.

12. Microstructure evolution during heat treatment and mechanical properties of Mg-2.49Nd-1.82Gd-0.19Zn-0.4Zr cast alloy [Text] / S.J. Liu, G.Y. Yang, S.F. Luo, W.Q. Jie // Materials Characterization. - 2015. – № 107. – P. 334–342.

13. Chemical composition optimization of gravity cast Mg-yNd-xZn-Zr alloy [Text] / P.H. Fu, L.M. Peng, H.Y. Jiang, L. Ma, C.Q. Zhai // Materials Science and Engineering: A. – 2008. – № 496 (1–2). – P. 177–188.

14. Effect of heat treatment on the microstructure and mechanical property of vacuum die-casting NZ30K Mg-alloy [Text] / W. Jie, W. Qudong, Y. Bing, J. Haiyan, D. Wenjiang // Chinese Journal of Materials Research. – 2019. – Vol. 33, No 1. – P. 1–8.

15. Shi W. Microstructure of ZM6 magnesium alloy with different Nd content [Text] / W. Shi, Y. Ma // Rare Met. – 2013. – No 32(3). – P. 234–240.

16. Penghuai F. Effects of heat treatments on the microstructures and mechanical properties of Mg-3Nd-0.2Zn-0.4Zr (wt. %) alloy [Text] / F. Penghuai, P. Liming, J. Haiyan, C. Jianwei, Z. Chunquan // Materials Science and Engineering: A. – 2008. – No 486. – P. 183–192.

17. The influence of individual rare earth elements (La, Ce, of Nd) on creep resistance of die-cast magnesium aloe AE44 [Text] / S. Zhu, M.A. Eastom, T.B. Abbot et. al. // Advanced engineering materials. – 2016. – Vol. 18, No 6. – P. 932–937.

18. Effect of Zr modification on solidification behavior and mechanical properties of Mg-Y-Re (WE54) alloy [Text] / J. Li, R. Chen, Y. Ma, W. Ke // Journal of magnesium and alloys. – 2013. – No 1. – P. 346–351.

19. ДСТУ ISO 6892-1:2019. Металеві матеріали. Випробування на розтяг. Частина 1. Метод випробування за кімнатної температури [Текст] [Чинний від 10.05.2019]. – К. : ДП «УкрНДНЦ», 2019.

20. ДСТУ 8972:2019. Сталі та сплави. Методи виявлення та визначення величини зерна [Текст] [Чинний від 01.01.2021]. – К. : ДП «УкрНДНЦ», 2019.

21. Avedesian M. ASM Specialty Handbook : Magnesium and Magnesium Alloys [Text] / M. Avedesian, H. Baker. – Materials Park, OH: ASM International, 1999. – 350 p.

22. Wu D. Microstructure and mechanical properties of a sand-cast Mg-Nd-Zn alloy [Text] / D. Wu, R.S. Chen, W. Ke // Materials and Design. – 2014. – No 58. – P. 324–331.

DOI 10.15588/1607-6885-2024-3-5