p-ISSN 1607-6885 Нові матеріали і технології в металургії та машинобудуванні. 2026/2
e-ISSN 2786-7358 New materials and technologies in metallurgy and mechanical engineering. 2026/2

СТРУКТУРОУТВОРЕННЯ. ОПІР РУЙНУВАННЮ ТА ФІЗИКО-МЕХАНІЧНІ ВЛАСТИВОСТІ

STRUCTURE FORMATION. RESISTANCE TO DESTRUCTION AND PHYSICAL-MECHANICAL PROPERTIES

UDC 669.721.5

INFLUENCE OF CHEMICAL COMPOSITION ON MECHANICAL PROPERTIES OF BIORESORBABLE MAGNESIUM ALLOY OF Mg-Nd-Zr SYSTEM FOR OSTEOSYNTHESIS

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Purpose. *Determination of the optimal chemical composition of a bioresorbable Mg-Nd-Zr magnesium alloy capable of providing a favourable balance of strength and ductility for osteosynthesis applications.*

Research methods*. The study employed a Box-Behnken experimental design. Specimens were prepared by induction melting followed by heat treatment. Tensile properties were measured in accordance with ISO 6892-1, and the results were processed statistically using the STATISTICA software package.*

Results*. Neodymium was identified as the main contributor to the tensile strength of the Mg-Nd-Zr alloy, with an optimal content of about 3.6 %. Zirconium improved ductility, most likely through the formation of a fine-grained structure, with an optimal content of about 1.8 %. A negative interaction between neodymium and zirconium was observed when content of both elements were increased simultaneously, and this effect should be considered when optimising the alloy composition. Within the zinc range studied (up to 0.8 %), zinc had no statistically significant effect on the mechanical properties. The resulting regression model predicts the mechanical properties of the alloy from the alloying-element content with good accuracy. Overall, the findings provide a basis for optimising the composition of bioresorbable magnesium alloys to achieve a favourable balance of strength and ductility for osteosynthesis.*

Scientific novelty*. The combined effect of Nd, Zr and Zn on the mechanical properties of a bioresorbable Mg-Nd-Zr alloy for osteosynthesis was established. Neodymium was identified as the principal strength-controlling element, whereas zirconium contributes to improved ductility and can also enhance strength. A negative Nd-Zr interaction at elevated concentrations was also revealed, with a substantial effect on the mechanical characteristics of the alloy.*

Practical value*. The regression model makes it possible to predict the mechanical properties of the alloy from the alloying-element content, thereby simplifying the selection of compositions for specific medical applications.*

Key words: *bioresorbable magnesium alloy, osteosynthesis, mechanical properties, chemical composition, neodymium, zirconium, Box-Behnken design, regression model, composition optimization, implants.*

Introduction

The full-scale war in Ukraine has sharply increased the demand for implants and osteosynthesis devices. Preliminary estimates suggest an increase of more than 800 %. This growth is associated with the sharp rise in traumatic injuries involving severe bone damage among both service personnel and civilians. According to the Ministry of Defence of Ukraine, about 40 % of wounded personnel sustain injuries to the extremities, many of which require complex surgery for bone reconstruction.

In the treatment of service personnel with traumatic bone injuries, temporary fixation of fractured bone fragments is often required. Standard clinical practice involves temporary fixation devices made of titanium alloys or special steels. The main drawback of this approach is the need for a second operation to remove the hardware.

A possible alternative is the use of materials that are gradually resorbed by the body, progressively lose their load-bearing capacity, and eventually dissolve completely. Magnesium-based alloys are among the most promising candidates. In particular, an alloy based on the ML10 grade has been developed for use in traumatology, orthopaedics and cardiac surgery, where it functions as a bioresorbable material that slowly dissolves in the surrounding tissue after fulfilling its medical role.

The main challenges associated with this concept are relatively low mechanical properties of magnesium alloys and need to control their dissolution rate in the body. Research aimed at improving these characteristics therefore remains highly relevant.

DOI 10.15588/1607-6885-2026-2-1

Literature review

Because of their biocompatibility and their ability to dissolve gradually in the body, magnesium alloys are widely regarded as promising candidates for temporary osteosynthesis implants [1]. Their comparatively low strength and ductility, however, remain an important limitation. These factors substantially restrict their use in load-bearing components of endoprosthetic systems.

Nevertheless, magnesium, whether used as a pure metal or in alloyed form, is receiving increasing attention in healthcare and biomedical research [2]. Over recent years, the number of publications devoted to magnesium as a temporary implant material has grown steadily owing to its bioresorbable nature. Magnesium degrades readily and does not leave long-term residues in the body [3, 4]. As a bioresorbable material, it gradually breaks down in the biological environment and can support tissue regeneration and the recovery of physiological function. In many medical applications, implants are required only temporarily to support healing. If conventional implants are not removed in time, adverse reactions may occur, including chronic irritation, inflammatory response and, in some cases, biotoxicity.

A considerable literature resources are available on the properties of magnesium alloys and the factors that influence them. Most studies, however, focus on Mg-Ca or Mg-Zn-Ca alloys, whereas Mg-Re systems, including neodymium-alloyed Mg-Nd-Zr [5, 6], have received less attention, particularly with the influence of chemical composition on mechanical properties.

Studies [7, 8] discuss the chemical composition, properties and applications of bioresorbable magnesium alloys and emphasise that a sound understanding of composition is essential for improving biocompatibility and bioresorbability. These studies analyse both chemical composition and corrosion behaviour, with particular attention to how individual alloying elements affect composition and corrosion resistance. In these and in similar studies [9, 10], however, the emphasis is placed on corrosion behaviour and biocompatibility rather than on mechanical properties. The authors of [11] review bioresorbable magnesium alloys as candidate materials for osteosynthesis and show that Ca, Zn, Mn, Sr and Zr are widely used as alloying additions to magnesium because of their non-toxicity. Elements such as Al, Ni, Ag, Cu and the rare earths (Nd, La, Ce) may also be used to improve the corrosion resistance and mechanical properties of magnesium alloys [12]. Lu et al. [13] examines alloying strategies for magnesium alloys intended for orthopaedic use. Zirconium is an efficient grain refiner and improves both strength and ductility. Zinc strengthens the alloy but can reduce ductility at high concentrations. Neodymium provides a pronounced increase in strength together with a favourable balance of strength and ductility. Calcium and strontium promote bioactivity and osteointegration. Compositions such as Mg-Nd-Zn-Zr show promising performance by combining high strength, adequate ductility and a controlled corrosion rate. Chen et al. [14] summarises the effects of critical alloying elements on the structure, mechanical properties and bioresorbable behaviour of magnesium alloys, with a focus on the Mg-Ca, Mg-Zn, Mg-Sr, Mg-Re and Mg-Cu systems. Zinc strengthens the alloy through solid-solution and precipitation hardening, calcium improves biocompatibility, strontium promotes osteogenesis, and rare-earth elements enhance corrosion resistance. Importantly, the content of alloying elements must be carefully controlled in order to maintain a workable balance between mechanical properties, biodegradation rate and biocompatibility.

Taken together, the available literature shows that, despite substantial research on magnesium alloys for medical applications, the selection of an optimal Mg-Nd-Zr composition with specific regard to mechanical properties remains insufficiently studied. Given the strong and complex influence of alloying elements on both strength and ductility, identifying their optimal content remains an important task in the development of bioresorbable materials for osteosynthesis.

Objective

This study is aimed to determine the optimal chemical composition of a bioresorbable Mg-Nd-Zr magnesium alloy that would provide a favourable balance of strength and ductility for osteosynthesis applications. To achieve this aim, an experimental design was developed, ingots of different chemical compositions were produced, the specimens were tested in tension, and the resulting data were analysed statistically.

The study examined how the mechanical properties of an Mg-Nd-Zr alloy – which contains no toxic alloying elements and is therefore a potential candidate for implant applications – depend on the concentration of its alloying elements. The property variables were ultimate tensile strength and elongation to failure under uniaxial tension.

Materials and methods

Ingots of the magnesium alloy were produced by melting in an IPM-500 induction furnace with a capacity of 0.5 t, a power rating of 140 kW and a throughput of 230 kg/h. A gas-fired holding furnace with a capacity of 150 kg was also used. Pre-heated charge materials were loaded into the induction furnace; after melting, the liquid alloy was transferred into removable crucibles at 650–730 °C. The crucibles were then placed in holding furnaces, where the alloy composition was refined with a flux consisting of 38–46 % $MgCl_2$, 32–40 % KCl, 5–8 % $BaCl_2$ and 3–5 % CaF_2 at 740–760 °C.

The cast blanks were heat-treated in a pit-type electric resistance furnace (112 kW, 95 kg/h capacity) and in a PAP-4M furnace (50 kg/h capacity). The heat-treatment regime consisted of solution treatment at 540 ± 5 °C for 15 h followed by air cooling, and ageing at 200 ± 5 °C for 8 h followed by final air cooling.

Tensile testing was performed on a modernised R10 testing machine (Fig. 1) in accordance with ISO 6892-1, using cylindrical specimens with a gauge-section diameter of 4 mm.

The extensometer gauge length was 25 mm. During testing, elongation of the gauge section was measured with

DOI 10.15588/1607-6885-2026-2-1

an accuracy of ±1 µm. Stress in the gauge section was applied by loading the movable crosshead with a force measured by a dynamometer. The measurement accuracy for stress in the gauge section was 3 MPa. The extensometer and elastic dynamometer signals were digitised at 0.01 s intervals. All tests were performed on series of identical specimens. Statistical analysis was conducted at a significance level of 0.05.


a *b* *c*

Figure 1. Specimens for tensile testing (*a*); general view (*b*) and working area (*c*) of the modernised R10 tensile testing machine equipped with a dynamometer

Statistical analysis and mathematical modelling were performed in STATISTICA (StatSoft) using the experimental data obtained from the designed experiments. The workflow combined correlation, analysis of variance and regression methods. Correlation analysis was first used to identify mutually independent factors for further study. ANOVA and Pareto charts were then used to identify the factors with the strongest influence on the response variables properties. Multiple regression analysis was subsequently used to determine the regression coefficients and construct the mathematical models. Model adequacy was assessed by comparing the adequacy variance with the reproducibility variance using Fisher's F-test. Because the number of experimental runs exceeded the number of regression coefficients to be estimated, the design was under-saturated. The reproducibility variance was determined from triplicate experiments at the centre point of the design, and its homogeneity was evaluated using Cochran's criterion.

The reproducibility variance was calculated from:

$$S_y^2 = \frac{\sum_{i=1}^{N}\sum_{j=1}^{n}\left(F_{i,j}^{\exp} - \overline{F_i^{\exp}}\right)^2}{N(n-1)}, \qquad (1)$$

where N is the number of replicate experiments and n is the number of repetitions per experiment.

Assuming uniform replication of experiments, the adequacy variance of the model was calculated from:

$$S_{ad}^2 = \frac{\sum_{i=1}^{N}\sum_{j=1}^{n}\left(T^{calc} - \overline{T^{\exp}}\right)^2}{f_{ad}}, \qquad (2)$$

where n is the number of replicate experiments in each series, T^{calc} is the predicted value of the response parameter, $\overline{T^{\exp}}$ is the mean experimental value from the replicates, and f is the number of degrees of freedom.

The homogeneity of the reproducibility variance was evaluated using Fisher's F-test:

$$F^{\exp} = \frac{S_{ad}^2}{S_y^2}. \qquad (3)$$

Results

To establish how the principal alloying elements influence strength and ductility, the upper concentration limit for each element was selected on the basis of its solubility in magnesium. The upper limits were thus 1.8 % for zirconium, 3.6 % for neodymium and 0.8 % for zinc.

The Box-Behnken design was selected for experimental planning. Compared with a full factorial design, it offers several advantages. It requires substantially fewer experimental points, especially when the number of factors is large, thereby reducing both material consumption and experimental cost. Unlike a first-order full factorial design, it also allows quadratic effects and factor interactions to be estimated, providing a more accurate approximation of the response surface within the region of interest. Because the design is spherically symmetric, the experimental points are distributed uniformly around the centre, which ensures uniform prediction variance throughout the factor space. This arrangement also makes the design more resistant to random disturbances and measurement error than a full factorial design.

The tensile-test results obtained for Mg-Nd-Zr alloy specimens of different chemical composition (Table 1) were used to construct the Box-Behnken experimental matrix with three factors at three levels (Table 2).

Table 1 – Levels of variation of the chemical composition of the experimental specimens

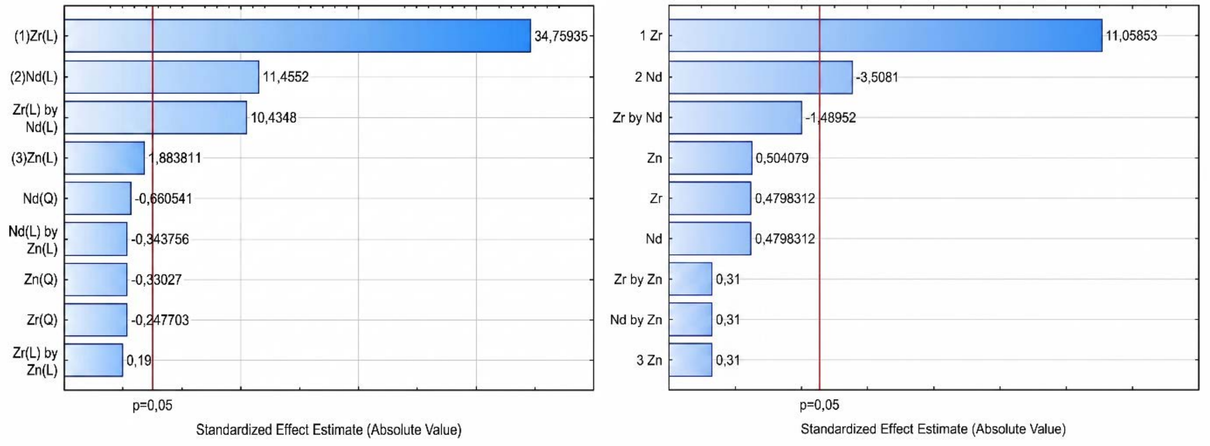
Level	Zr	Nd	Zn
-1	0,2	2,0	0,1
0	1,0	2,8	0,45
+1	1,8	3,6	0,8

Owing to the efficiency of the chosen experimental design, 15 experimental melts were sufficient for analysis of variance (Fig. 2) and for constructing a statistically significant second-order regression model with linear two-factor interaction terms (Tables 3 and 4).

Table 2 – Box-Behnken experimental design matrix (2^3)

Run No.	X_1 (Zr)	X_2 (Nd)	X_3 (Zn)	$\sigma_в$, МПа	δ, %
1	0,20	2,00	0,45	249,3	3,42
2	1,80	2,00	0,45	252,2	5,37
3	0,20	3,60	0,45	290,1	3,32
4	1,80	3,60	0,45	268,6	4,48
5	0,20	2,80	0,10	268,9	3,37
6	1,80	2,80	0,10	259,3	4,92
7	0,20	2,80	0,80	270,4	3,37
8	1,80	2,80	0,80	260,8	4,92
9	1,00	2,00	0,10	249,8	4,39
10	1,00	3,60	0,10	278,8	3,90
11	1,00	2,00	0,80	251,8	4,39
12	1,00	3,60	0,80	280,0	3,90
13	1,00	2,80	0,45	262,5	4,14
14	1,00	2,80	0,45	264,9	4,60
15	1,00	2,80	0,45	266,1	4,00

DOI 10.15588/1607-6885-2026-2-1



a *b*

Figure 2. Pareto charts showing the effect of the alloying elements on the tensile strength (*a*) and ductility (*b*) of the alloy

Table 3 – Parameters of the regression model for the effect of the alloying elements on tensile strength, including two-factor interaction terms

Factor	Regression coefficient	Standard error	t(5)	p	−95 % CI	+95 % CI
Constant	265.000	0.335907	788.9095	0.000000	264.1365	265.8635
Zr (linear)	−9.4500	0.822800	−11.4852	0.000088	−11.5651	−7.3349
Zr (quadratic)	−0.1500	0.605564	−0.2477	0.814215	−1.7067	1.4067
Nd (linear)	28.6000	0.822800	34.7594	0.000000	26.4849	30.7151
Nd (quadratic)	−0.4000	0.605564	−0.6605	0.538120	−1.9567	1.1567
Zn (linear)	1.5500	0.822800	1.8838	0.118296	−0.5651	3.6651
Zn (quadratic)	−0.2000	0.605564	−0.3303	0.754585	−1.7567	1.3567
Zr·Nd	−12.2000	1.163615	−10.4846	0.000136	−15.1912	−9.2088
Zr·Zn	0.0000	1.163615	0.0000	1.000000	−2.9912	2.9912
Nd·Zn	−0.4000	1.163615	−0.3438	0.745014	−3.3912	2.5912

Notes. Statistically significant values are shown in bold. t(5) is the value of the t-statistic with 5 degrees of freedom, used for hypothesis testing and for assessing statistical significance; p is the probability of observing the given effect under the null hypothesis that the effect is absent. Lower p-values indicate stronger evidence against the null hypothesis.

Table 4 – Parameters of the regression model for the effect of the alloying elements on elongation to failure, including two-factor interaction terms

Factor	Regression coefficient	Standard error	t(5)	p	−95 % CI	+95 % CI
Constant	4.145833	0.057314	72.33581	0.000000	3.998504	4.293163
Zr (linear)	1.552500	0.140389	11.05853	0.000105	1.191618	1.913382
Zr (quadratic)	0.049583	0.103324	0.47988	0.651567	−0.216019	0.315186
Nd (linear)	−0.492500	0.140389	−3.50810	0.017135	−0.853382	−0.131618
Nd (quadratic)	0.049583	0.103324	0.47988	0.651567	−0.216019	0.315186
Zn (linear)	0.000000	0.140389	0.00000	1.000000	−0.360882	0.360882
Zn (quadratic)	0.052083	0.103324	0.50408	0.635627	−0.213519	0.317686
Zr·Nd	−0.395000	0.198541	−1.98952	0.103314	−0.905365	0.115365
Zr·Zn	0.000000	0.198541	0.00000	1.000000	−0.510365	0.510365
Nd·Zn	0.000000	0.198541	0.00000	1.000000	−0.510365	0.510365

DOI 10.15588/1607-6885-2026-2-1

Discussion

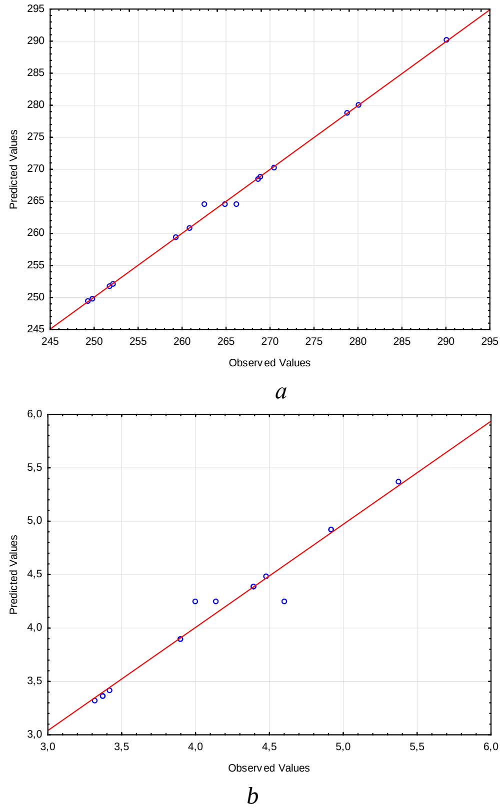
Analysis of the contribution of each factor to alloy strength (Fig. 2a) shows that neodymium exerts the strongest positive effect. By contrast, zirconium and Nd·Zr interaction both have a statistically significant negative effect on tensile strength.

The alloying elements affect ductility differently. Increasing the zirconium content improves ductility, whereas increasing the neodymium content reduces it (Fig. 2*b*). The positive effect of zirconium is more than three times larger than the negative effect of neodymium. The Zr·Nd interaction leads to an additional reduction in ductility. As in the case of tensile strength, zinc does not have a statistically significant effect on elongation to failure.

Overall, the results indicate that the strength of Mg-Nd-Zr alloys is governed primarily by neodymium content and by the nature of its interaction with zirconium. Increasing the neodymium content raises tensile strength, whereas an excessive simultaneous increase both Nd and Zr can reduce it because of the negative two-factor interaction. Within the concentration range studied, zinc does not have a statistically significant effect.

According to the regression model, the linear effect of zirconium on tensile strength is negative. This effect, however, cannot be interpreted in isolation, because zirconium also has a strong influence on ductility and participates in a two-factor interaction with neodymium. The low p-value supports the reliability of this estimate. The optimum zirconium content is therefore likely to lie in the upper part of the studied range (about 1.8 %). Tensile strength also increases with neodymium content, as indicated by the statistically significant positive regression coefficient and the low p-value. Its optimum is likewise expected to lie near the upper part of the studied range (about 3.6 %). The regression coefficients for zinc are not statistically significant, indicating that no detectable effect of this element on strength was observed.

The near-linear relation between the model-predicted and experimentally measured values of strength and elongation to failure (Fig. 3) indicates that the estimates are reliable and confirms the adequacy of the model.

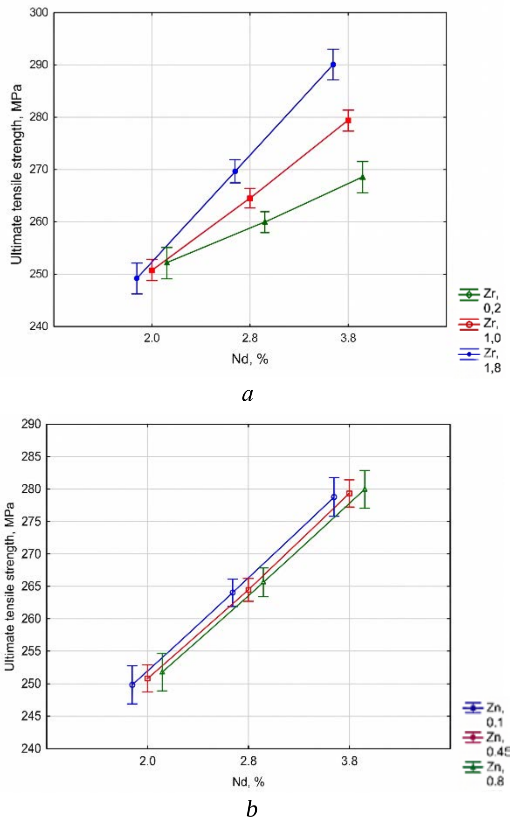


a

b

Figure 3. Model-predicted versus experimentally measured values of tensile strength (*a*) and elongation to failure (*b*) of the alloy

Analysis of the dependence of strength (Fig. 4) and ductility (Fig. 5) alloying-element content shows that increasing the neodymium content generally increases tensile strength while reducing ductility.



a

b

Figure 4. Tensile strength of the alloy as a function of neodymium content at 0.45 % Zn (*a*) and at 1 % Zr (*b*)

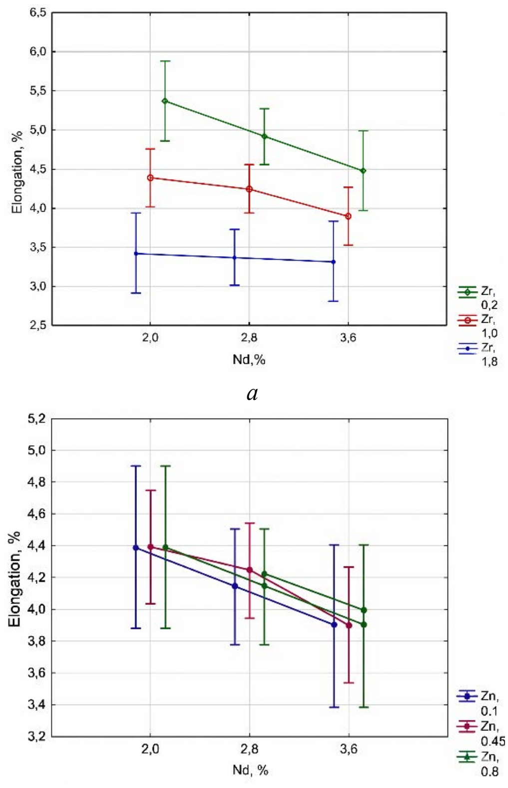
The increase in tensile strength most likely reflects two contributions from neodymium: solid-solution strengthening and the formation of strengthening

DOI 10.15588/1607-6885-2026-2-1

intermetallic phases. The solubility of zirconium in magnesium is low; nonetheless, zirconium restricts grain growth and thus promotes the formation of a fine-grained structure. As a result, zirconium can enhance strength and ductility simultaneously, which is particularly important because improvement of one of these properties is often accompanied by deterioration of the other.

Figure 4a shows that higher neodymium content is associated with increased tensile strength. At the same time, a simultaneous increase in neodymium and zirconium reduces strength, most likely because of excessive precipitation of secondary phases.

In the presence of zinc, zirconium may also influence the formation and distribution of secondary phases and thus affect the mechanical properties of the alloy. According to [15], zirconium has a beneficial effect on magnesium-alloy properties when its content remains below about 2 %. Although zinc may also contribute to an increase in strength, no such effect was observed within the zinc concentration range examined in present study.



a

b

Figure 5. Elongation to failure of the alloy as a function of neodymium content at 0.45 % Zn (*a*) and at 1 % Zr (*b*)

Consistent with the obtained results, Fig. 5a shows that ductility increases with increasing zirconium content, most likely because grain growth is restrained and a fine-grained structure forms. Changes in zinc concentration had almost no effect on alloy ductility.

Conclusions

Neodymium was found to be the key element controlling the tensile strength of Mg-Nd-Zr magnesium alloys, with the optimal content lying close to the upper limit of the studied range (3.6 %).

Increasing the zirconium content improved alloy ductility, most likely through its role in promoting a fine-grained structure. The optimal zirconium content likewise lies close to the upper limit of the range studied (1.8 %).

A negative interaction between neodymium and zirconium was observed at simultaneously elevated concentrations of both elements. This effect should be taken into account when optimising the alloy composition.

Within zinc concentration range (up to 0.8 %), zinc had no statistically significant effect on the mechanical properties of the alloy. Taking into consideration the possible positive effect of zinc on the corrosion resistance of magnesium alloys, however, its addition may still be useful and requires further investigation. At the same time, zinc additions may increase Young’s modulus [16].

The regression model developed predicts the mechanical properties of the alloy from its alloying-element content with good accuracy. These results provide a basis for optimising the chemical composition of the bioresorbable magnesium alloy to achieve a favourable balance of strength and ductility for osteosynthesis applications.

Further research should focus on how chemical composition influences the biodegradation rate and biocompatibility of the alloy in order to support its further optimisation for medical applications.

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Received 15.04.2026
Accepted 23.04.2026
Published 07.05.2026

УДК 669.721.5

ВПЛИВ ХІМІЧНОГО СКЛАДУ БІОРОЗЧИННОГО МАГНІЄВОГО СПЛАВУ СИСТЕМИ Mg-Nd-Zr ДЛЯ ОСТЕОСИНТЕЗУ НА МЕХАНІЧНІ ВЛАСТИВОСТІ

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Мета роботи. *Визначення раціонального хімічного складу біорозчинного магнієвого сплаву системи Mg-Nd-Zr, при якому забезпечується оптимальне поєднання міцності та пластичності, для застосування в остеосинтезі.*

Методи дослідження. *Застосовано комплекс методів, що включав планування експерименту за методом Бокса-Бенкена, виготовлення зразків шляхом виплавки та термічної обробки магнієвих сплавів, механічні випробування на розтяг згідно з ISO 6892-1, а також статистичну обробку отриманих даних за допомогою програмного забезпечення STATSOFT.*

Отримані результати. *Встановлено ключову роль неодиму у підвищенні границі міцності магнієвого сплаву системи Mg-Nd-Zr, з оптимальним вмістом близько 3,6 %. Виявлено позитивний вплив цирконію на пластичність сплаву, імовірно через формування дрібнозернистої структури, з оптимальним вмістом близько 1,8 %. Виявлено негативний ефект взаємодії між неодимом та цирконієм при їх одночасному підвищенні концентрації, що потребує врахування при оптимізації складу сплаву. Встановлено, що вплив цинку в досліджуваному діапазоні концентрацій (до 0,8 %) на механічні властивості сплаву є статистично незначущим. На основі отриманих даних розроблено регресійну модель, яка дозволяє з високою точністю прогнозувати*

DOI 10.15588/1607-6885-2026-2-1

механічні властивості сплаву залежно від вмісту легуючих елементів. Ці результати створюють основу для оптимізації хімічного складу біорозчинного магнієвого сплаву з метою досягнення оптимального балансу міцності та пластичності для застосування в остеосинтезі.

Наукова новизна. *Встановлено комплексний вплив легувальних елементів (Nd, Zr, Zn) на механічні властивості біорозчинного магнієвого сплаву системи Mg-Nd-Zr для остеосинтезу. Виявлено, що неодим є ключовим елементом, який підвищує границю міцності сплаву, тоді як цирконій сприяє одночасному зростанню міцності та пластичності. Виявлено негативний ефект взаємодії між Nd та Zr при їх високих концентраціях, що суттєво впливає на механічні характеристики сплаву.*

Практична цінність. *Розроблена регресійна модель дозволяє прогнозувати механічні властивості сплаву залежно від вмісту легуючих елементів, що значно спрощує процес підбору оптимального складу для конкретних медичних застосувань.*

Ключові слова: *біорозчинний магнієвий сплав, остеосинтез, механічні властивості, хімічний склад, неодим, цирконій, план Бокса-Бенкена, регресійна модель, оптимізація складу, імплантати.*

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DOI 10.15588/1607-6885-2026-2-1