Adrian BARYLSKI[*]**, Krzysztof ANIOŁEK**[*]**, Michał DWORAK**[*]

THE INFLUENCE OF SOLUTION TREATMENT ON THE STRUCTURE AND MECHANICAL AND TRIBOLOGICAL PROPERTIES OF MAGNESIUM ALLOY WE54

WPŁYW PROCESU PRZESYCANIA NA STRUKTURĘ ORAZ WŁAŚCIWOŚCI MECHANICZNE I TRIBOLOGICZNE STOPU MAGNEZU WE54

Key words:

WE54, solution treatment, mechanical properties, wear

Słowa kluczowe:

WE54, przesycanie, właściwości mechaniczne, zużycie

Abstract

The paper presents the influence of solution treatment on the mechanical and tribological properties of the WE54 magnesium alloy. The investigated alloy was solution treated at a temperature of 545°C for 8 hours and cooled in ice water (0°C), in room temperature water (20°C), and in hot water (95°C). Depending on the applied solution treatment parameters, a diversified decrease in hardness and Young's modulus was obtained. The lowest values of hardness H and modulus E were obtained when cooling in ice water. Abrasive wear of alloy WE54 was tested using a ball-on-disc tribometer (with a ZrO_2 ball). The

[*] Institute of Materials Science, Faculty of Computer Science and Materials Science, University of Silesia, ul. 75 Pułku Piechoty 1A, 41-500 Chorzów, Poland; e-mail: adrian.barylski@us.edu.pl.

tests have shown more than a threefold reduction in the volumetric wear and a twofold reduction in the linear wear, as well as favourable changes of the friction coefficient (a 20% decrease) as compared to the material in the as-received condition.

INTRODUCTION AND RESEARCH METHODOLOGY

Following the development of materials over the past 20 years, it can be noticed that magnesium alloys are more and more often used as constructional materials **[L. 1]**. A vast majority of these alloys are used for die-casting, and a few percent are subjected to mechanical working. Magnesium alloys are suitable for casting elements with complicated shapes and are quite easy to treat and process **[L. 2–3]**. Recently, a growing interest has been observed in magnesium alloys for biomedical applications (e.g., in orthopaedic or cardiovascular implants) **[L. 4]**. They have a significant advantage over conventional metallic biomaterials, ceramics, or biodegradable polymers, due to their low density (comparable to human bone density (ca. 1.75 g/cm^3), a relatively high specific strength, a low Young's modulus (44.1 GPa), and resistance to brittle cracking **[L. 4–6]**. Magnesium alloys are also characterised by high biocompatibility **[L. 7]**, and their biggest advantage is that there is no necessity for reoperation to replace the implants made of them **[L. 4]**. The presence of rare earth elements in magnesium alloys is mainly aimed at their strengthening and improving their corrosion resistance **[L. 8]**. For biomedical applications based on rare earth elements, the following alloys are anticipated: Mg-Y **[L. 9]**, Mg-Gd **[L. 10]**, and alloys based on the Mg-Y-Nd – Zr system, identified as WE43 and WE54 **[L. 11–14]**. Magnesium alloys for biomedical applications are still in the research phase; therefore, it seems purposeful to obtain a material with an optimum set of mechanical, tribological, and corrosive properties. The aim of this paper is to determine the influence of solution treatment on the mechanical and tribological properties of the WE54 magnesium alloy.

The research material was the WE54 magnesium alloy produced by Magnesium Elektron (England), supplied in the form of rods, 25.4 mm in diameter. Heat treatment was performed in an electric resistance furnace, in an atmosphere of air. The specimens were 5 mm thick discs. Solution treatment was performed at a temperature of 545°C for 8 hours. Cooling from the solution treatment temperature took place in ice water (0°C – specimen designation – CIW), in room temperature water (20°C – designation – CCW) and in hot water with a temperature of 95°C (designation – CHW).

Grinding and polishing of the specimens was performed according to the standard procedure Metalog Guide A, with etching of the metallographic specimens in a 3% Nital solution (HNO_3 in ethyl alcohol). The structure was observed using a light optical microscope Olympus GX-51.

Phase composition of the material was examined by X-ray diffraction, using a Philips X'Pert diffractometer fitted with a copper anode tube (λCuKα – 1,54178 Å), operating at 30 mA/40 kV, and a diffracted-beam graphite monochromator for a wave length coming from the copper anode. Registration was performed using the "step scanning" method with a 0.04° step and a counting time of 25 s/step, in the angular range of 10° – 140° 2θ. The divergence slit was 1/2°, for the diffracted beam 1/2°, and 2° Soller slits were applied.

Mechanical properties were examined with a nanohardness tester, NHT[2] manufactured by Anton-Paar (Switzerland). Measurements were performed in accordance with the recommendations of the ISO 14577 standard. A Berkovich indenter was used, with a maximum load of 100 mN, loading and unloading time of 30 s, and time of maximum loading of 10 s. Hardness H and elasticity modulus E were determined with the standard Oliver-Pharr method [L. 15]. The measurement results were averaged for 10 indentations made on each of the tested specimens.

Tribological tests of alloy WE54 were performed for the as-received alloy and for an alloy subjected to heat treatment. In each case, 4 specimens (ϕ25.4 mm discs) were prepared by cutting them out of rods provided by the manufacturer. ZrO$_2$ balls with a diameter of 6 mm were used as counter-specimens. The surfaces of the specimens were prepared so as to obtain a roughness in the order of Ra = 0.1 μm. The tests were carried out under dry friction conditions. The pressure in the friction pair was 10 N. The sliding speed in a one-way rotary motion with a 7 mm radius was 0.1 m/s at a friction distance of 100 m. Ambient conditions (temperature of 21±1°C, and humidity of 50±5%) were in compliance with the recommendations of VAMAS [L. 16]. The average area of the wear trace, P, was determined using a Mitutoyo Surftest SJ-500 profilographometer. Volumetric wear, V_W, was determined from the formula (1):

$$V_W = \frac{V}{F_n \cdot s} \left[\frac{mm^3}{N \cdot m} \right] \tag{1}$$

where: F_n – pressure force [10 N], s – friction distance [100 m], V – volume of the wear trace of the disc determined from the formula: $V = P \cdot 2\pi r$ [mm^3], P – average area of the wear trace [mm^3], r – radius of the friction distance [7 mm].

The linear wear, L_w, was determined as the difference between the indications of the micrometric sensor before the test and after cooling. The friction coefficient was determined as a quotient of the recorded friction force, F_t, and the normal force applied F_n.

RESEARCH RESULTS AND THEIR ANALYSIS

Magnesium alloy WE54 in its as-received condition had a fine-grained structure consisting of a solid solution, α-Mg, and intermetallic phase $Mg_{41}Nd_5$, with precipitates of, inter alia, $Mg_{24}Y_5$ (**Fig. 1a**). On a diffraction pattern of the as-received WE54 alloy (**Fig. 2a**), apart from peaks coming from the α-Mg solution, only reflexes derived from phase $Mg_{41}Nd_5$ could be observed. The fact that there were no reflexes coming from other precipitates indicated that their content was lower than the detectability threshold in the method. During the solution treatment process, a significant part of precipitates dissolved, which can be seen on the diffraction pattern in **Fig. 2b**, and an intensive growth of the grain occurred. In the image of the microstructure (**Fig. 1b**), fine precipitation particles were also visible, which did not dissolve.
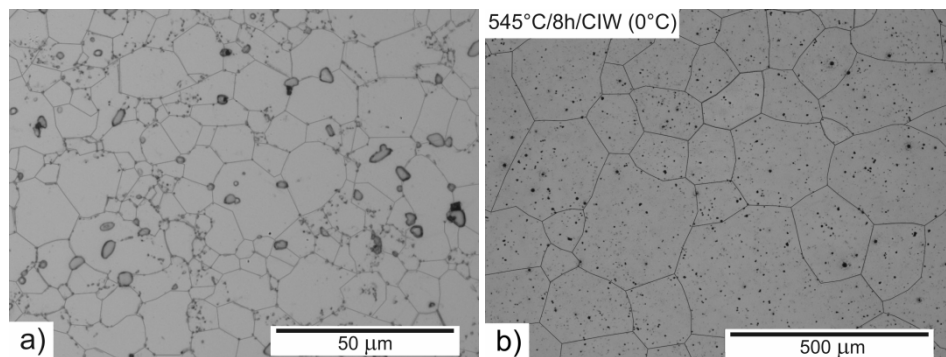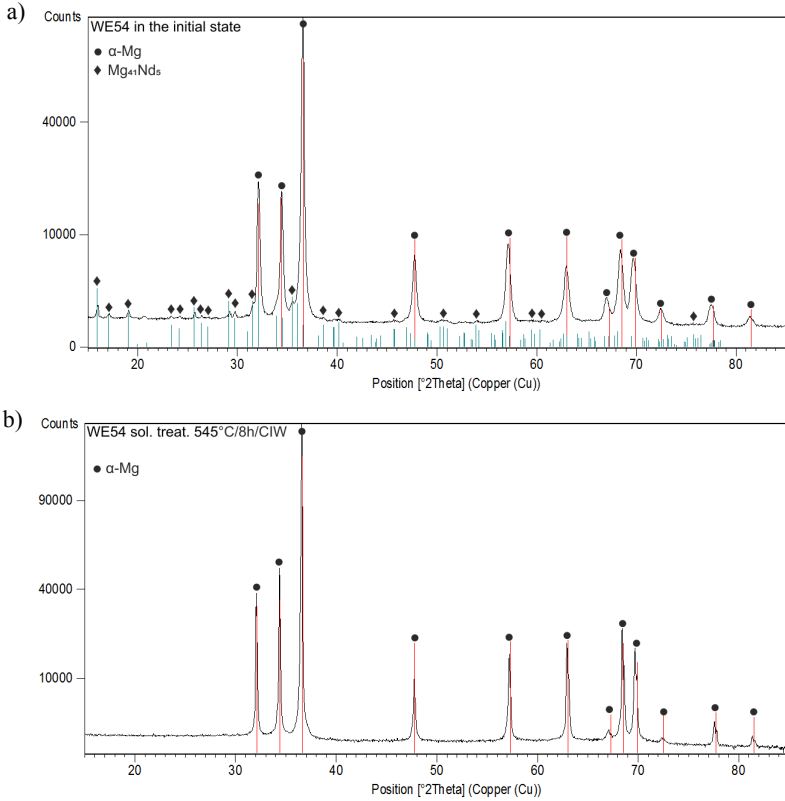

Fig. 1. **Example of the microstructure of alloy WE54 in the as-received condition – a) and after solution treatment at 545°C, and cooling in ice water (0°C)**
Rys. 1. Przykładowa mikrostruktura stopu WE54 w stanie wyjściowym – a) i po przesycaniu w temperaturze 545°C i chłodzeniu w wodzie z lodem (0°C)

 Figure 3 presents the results of hardness H measurements for the material in its as-received condition and after heat treatment.

These data show that, in its as-received condition, the investigated magnesium alloy had hardness of 1.08 GPa. The hardness after solution treatment at 545°C was lower by ca. 10–18% compared to the as-received condition, and reached the lowest value of 0.91 GPa for a specimen cooled in ice water. Another observation made was that Young's modulus of the solution treated specimens decreased in comparison with the as-received condition (**Fig. 4**). The changes were similar to those in hardness, i.e. the lowest value of modulus E was obtained for the specimen cooled in ice water.

Fig. 2.　Diffraction pattern (XRD) of the surface of alloy WE54 in the as-received condition (a), and after solution treatment (b) at 545°C, and cooling in ice water (0°C)

Rys. 2.　Dyfraktogram (XRD) z powierzchni stopu WE 54 w stanie dostawy – a) i po przesycaniu w temperaturze 545°C i chłodzeniu w wodzie z lodem (0°C)
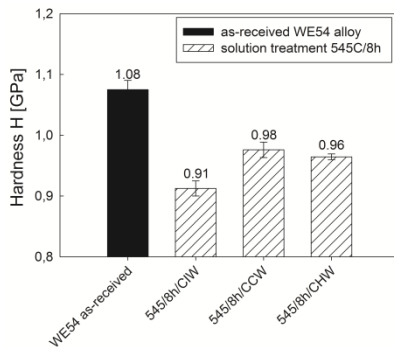
Fig. 3.　Hardness of alloy WE54 in the as-received condition and after solution treatment at 545°C followed by cooling in water with a temperature of 0°C – CIW, 20°C – CCW and 95°C – CHW

Rys. 3.　Twardość stopu WE54 w stanie wyjściowym oraz po przesycaniu w temperaturze 545°C z chłodzeniem w wodzie o temperaturze: 0°C – CIW, 20°C – CCW i 95°C – CHW

Changes of mechanical properties caused by solution treatment of an alloy depend on a number of factors, including, inter alia, the phase composition and special features of the alloy's structure in its as-received condition and after solution treatment, as well as solution treatment conditions and previous treatment. Generally, the solution treatment process may lead to both strengthening and softening of an alloy. In the case of alloy WE54 investigated in this study, the dissolution of precipitates during solution treatment could have led to solution strengthening but, on the other hand, the considerable increase in the grain size could have been the cause of the decrease of hardness. Measurements of hardness and Young's modulus performed after solution treatment showed that the overall effect of the two factors led to softening of the WE54 alloy, thereby showing that the contribution to the decrease of hardness, coming from the grain growth, outclassed the reinforcement effect induced by the dissolution of the precipitate phases in the matrix.
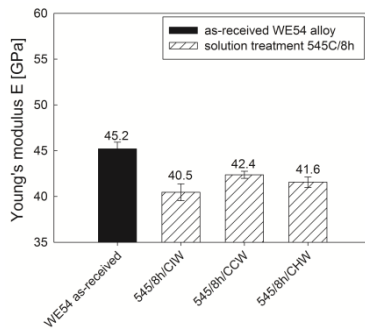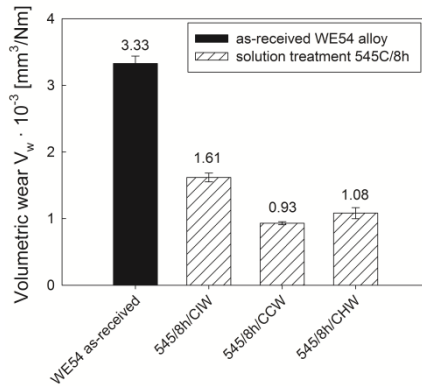

Fig. 4.   Young's modulus E of alloy WE54 in the as-received condition and after solution treatment at 545°C, followed by cooling in water with a temperature of 0°C – CIW, 20°C – CCW, and 95°C – CHW
Rys. 4.   Moduł Younga E stopu WE54 w stanie wyjściowym oraz po przesycaniu w temperaturze 545°C z chłodzeniem w wodzie o temperaturze: 0°C – CIW, 20°C – CCW, and 95°C – CHW

The results of tests of the effect of solution heat treatment on the tribological properties of alloy WE54 are presented in **Figures 5–8**.

In its as-received condition, alloy WE54 was characterized by volumetric wear: $V_w = 3{,}33 \cdot 10^{-3}$ mm^3/Nm. Tribological studies showed a more than threefold reduction in the volumetric wear for alloy WE54 subjected to solution treatment, as compared to the initial material. The alloy subjected to solution treatment at 545°C and cooled in room temperature water of 20°C $(0.93 \cdot 10^{-3}$ mm^3/Nm) was characterized by the lowest volumetric wear, and the nature of wear has also changed. The alloy in its as-received condition abraded mainly through machining (**Fig. 6a**), while after solution treatment, part of the material was deformed (the so-called "ploughing" appeared) and did not undergo wear (**Fig. 6b**).

Fig. 5. Volumetric wear V_w **of alloy WE54 in the as-received condition and after solution treatment at 545°C, followed by cooling in water with a temperature of 0°C – CIW, 20°C – CCW, and 95°C – CHW**

Rys. 5. Zużycie objętościowe V_w stopu WE54 w stanie wyjściowym oraz po przesycaniu w temperaturze 545°C z chłodzeniem w wodzie o temperaturze: 0°C – CIW, 20°C – CCW i 95°C – CHW
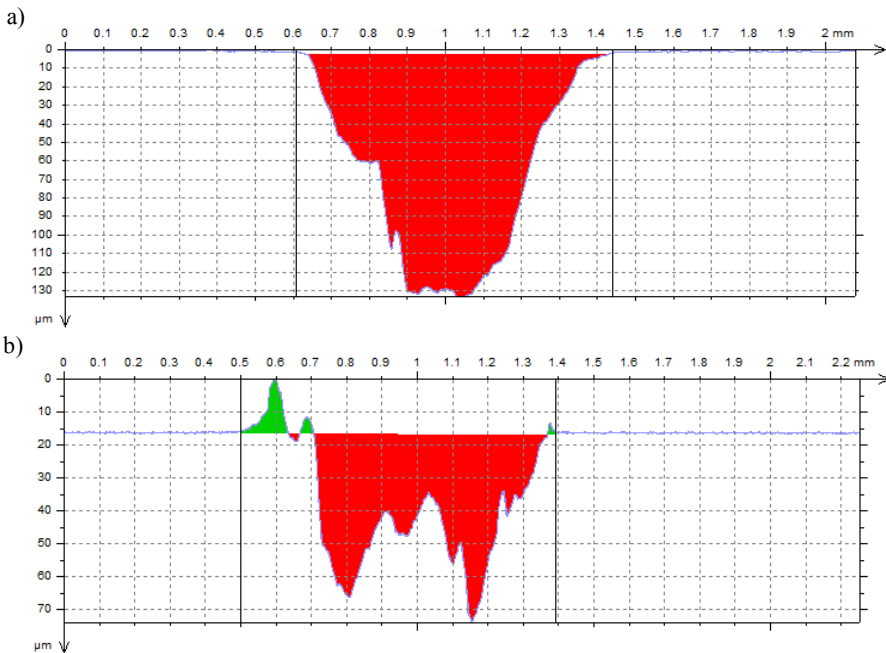
Fig. 6. Example of the friction surface profile of alloy WE54 in the as-received condition **(a), and after solution treatment at 545°C, followed by cooling in room temperature water of 20°C (b)**

Rys. 6. Przykładowy profil powierzchni tarcia stopu WE54 w stanie wyjściowym – a) oraz po przesycaniu w temperaturze 545°C z chłodzeniem w wodzie o temperaturze pokojowej 20°C – b)

A similar dependence can be observed for linear wear, L_w, where more than a twofold reduction in wear was observed for alloy WE54 subjected to heat treatment (**Fig. 7**). There was also a significant decrease, by 20%, of the friction coefficient in the material subjected to solution treatment (**Fig. 8**). No wear of the ceramic balls was observed.
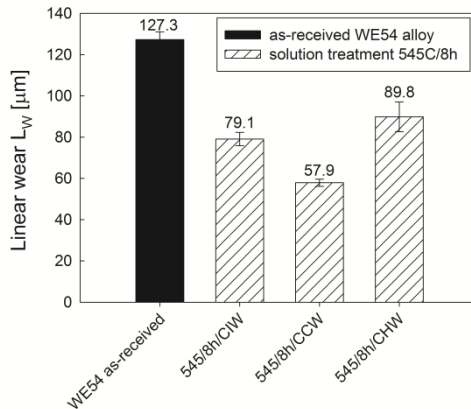

Fig. 7. Linear wear L_w of alloy WE54 in the as-received condition and after solution treatment at 545°C, followed by cooling in water with a temperature of 0°C – CIW, 20°C – CCW, and 95°C – CHW

Rys. 7. Zużycie liniowe L_w stopu WE54 w stanie wyjściowym oraz po przesycaniu w temperaturze 545 °C z chłodzeniem w wodzie o temperaturze: 0°C – CIW, 20°C – CCW i 95°C – CHW
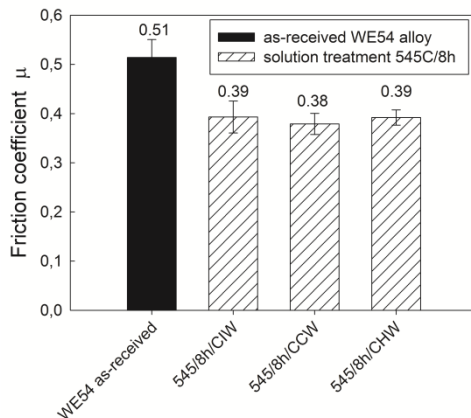

Fig. 8. Friction coefficient μ of alloy WE54 in the as-received condition and after solution treatment at 545°C, followed by cooling in water with a temperature of 0°C – CIW, 20°C – CCW, and 95°C – CHW

Rys. 8. Współczynnik tarcia μ stopu WE54 w stanie wyjściowym oraz po przesycaniu w temperaturze 545°C z chłodzeniem w wodzie o temperaturze: 0°C – CIW, 20°C – CCW i 95°C – CHW

CONCLUSIONS

- Magnesium alloy WE54 in its as-received condition had a fine-grained structure consisting of a solid solution, α-Mg, with precipitates of $Mg_{24}Y_5$ and intermetallic phase $Mg_{41}Nd_5$; however, after solution treatment at a temperature of 545°C, the intermetallic phases dissolved and an intensive growth of the grain occurred.
- The solution treatment process caused a considerable decrease in hardness of alloy WE54 – from 1.08 GPa (as-received) to 0.91 GPa for the alloy solution-treated at 545°C, followed by cooling in ice water. The decrease in hardness was caused mainly by the grain growth in the alloy during solution treatment.
- Tribological tests showed a more than threefold reduction of the volumetric wear, a twofold reduction of the linear wear, and a 20% reduction of the friction coefficient for alloy WE54 subjected to solution treatment, as compared to the initial material. The increased resistance to abrasive wear resulted mainly from the change in the nature of wear. The alloy in its as-received condition abraded mainly through machining, while after solution treatment, part of the material was deformed (the so-called "ploughing" appeared) and the wear was reduced.

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Streszczenie

W pracy przedstawiono wpływ przesycania na właściwości mechaniczne i tribologiczne stopu magnezu WE54. Badany stop przesycano w temperaturze 545°C, w czasie 8 h, z chłodzeniem w wodzie z lodem (0°C), w wodzie o temperaturze pokojowej (20°C) i w wodzie gorącej (95°C). W zależności od zastosowanych parametrów procesu przesycania uzyskano zróżnicowany spadek twardości oraz modułu Younga. Najmniejsze wartości twardości H i modułu E uzyskano dzięki zastosowaniu chłodzenia w wodzie z lodem. Badania zużycia ściernego stopu WE54 przeprowadzono na tribometrze o skojarzeniu kula (ZrO$_2$) – tarcza. Wykazano ponad 3 krotne ograniczenie zużycia objętościowego i 2-krotne zmniejszenie zużycia liniowego, korzystne zmiany odnotowano także w przypadku współczynnika tarcia (spadek o 20%) w stosunku do materiału w stanie dostawy.