DOI: 10.5604/01.3001.0016.1603

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EFFECT OF DEEP CRYOGENIC TREATMENT TIME ON MICROMECHANICAL AND TRIBOLOGICAL PROPERTIES OF MAGNESIUM ALLOYS WE43 AND WE54

WPŁYW CZASU GŁĘBOKIEJ OBRÓBKI KRIOGENICZNEJ NA WŁAŚCIWOŚCI MIKROMECHANICZNE I TRIBOLOGICZNE STOPÓW MAGNEZU WE43 I WE54

Key words: magnesium alloys WE43 and WE54, deep cryogenic treatment, micromechanical properties, compression test, friction, wear.

Abstract: The paper presents the effect of deep cryogenic treatment time on micromechanical and tribological properties of magnesium alloys, WE43 and WE54. The alloys were subjected to deep cryogenic treatment at a liquid nitrogen temperature (-196°C) for 2 to 48h. Tribological tests were performed in a rotational and a reciprocating linear motion, and wear trace studies were performed by profilometric and microscopic measurements. The tests indicate that deep cryogenic treatment has a favourable effect on the micromechanical, mechanical and tribological parameters of the two investigated alloys. It has also been shown that sub-zero treatment time significantly impacts the cryogenic treatment result. Among other things, there was a nearly 10% increase in hardness, Young's modulus, and a 35% reduction in tribological volumetric wear resulting from the improvement in mechanical properties, as well as a 2-fold reduction in linear wear with an increase in sub-zero treatment time relative to the material in its as-delivered state. Deep cryogenic treatment with appropriately selected sub-zero treatment time allows for improving the service life of magnesium alloys with rare earth metals.

Słowa kluczowe: stopy magnezu WE43 i WE54, głęboka obróbka kriogeniczna, właściwości mikromechaniczne, próba ściskania, tarcie, zużycie.

Streszczenie: W artykule przedstawiono wpływ czasu głębokiej obróbki kriogenicznej na właściwości mikromechaniczne oraz tribologiczne stopów magnezu WE43 i WE54. Stopy poddano głębokiej obróbce kriogenicznej w temperaturze ciekłego azotu (-196°C) w czasie od 2 do 48 h. Testy tribologiczne wykonano w ruchu obrotowym oraz w ruchu posuwisto-zwrotnym liniowym. Badania śladów zużycia wykonano za pomocą pomiarów profilografometrycznych oraz mikroskopowych. Przeprowadzone testy wskazują, że głęboka obróbka kriogeniczna wpływa korzystnie na parametry mikromechaniczne, mechaniczne i tribologiczne obu badanych stopów. Wykazano również, że istotne znaczenie na efekt obróbki kriogenicznej ma czas wymrażania. Stwierdzono między innymi blisko 10% wzrost twardości modułu Younga oraz wynikające z poprawy właściwości mechanicznych – 35% ograniczenie zużycia tribologicznego objętościowego i 2-krotne obniżenie zużycia liniowego wraz ze wzrostem czasu wymrażania w stosunku do materiału w stanie dostawy. Głęboka obróbka kriogeniczna o odpowiednio dobranym czasie wymrażania pozwala na poprawę trwałości eksploatacyjnej stopów magnezu z metalami ziem rzadkich.

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INTRODUCTION

Due to the increasing awareness of manufacturers in an effort to reduce greenhouse gas emissions related to dangerous climate changes and the huge increase in the price of fossil fuels over the past two decades, lightweight materials with high strength are in demand **[L. 1]**. Magnesium alloys are an excellent alternative to conventional engineering materials. In the automotive and aerospace industries, among other things, they allow weight reduction, which has a major impact on reducing fuel consumption (a 10% reduction in weight leads to up to a 30% reduction in fuel consumption) **[L. 2]**. The unique properties of magnesium alloys with rare earth metals make them suitable for applications not only in the industrial, automotive or aerospace sectors but also in the biomedical area (including orthopaedics **[L. 3]**, general surgery, and cardiovascular implants – Edward Huse presented the first medical report as early as 1878 after using magnesium wires to ligate bleeding vessels **[L. 4]**). The density of magnesium alloys (1.75––2.1 g/cm^3) is similar to that of the cortical bone (1.8–2.1 g/cm^3) **[L. 5, 6]**, which is of great importance in orthopaedics. Studies have further shown that biodegradable magnesium alloy rods yielded a significantly higher bone-implant interface strength than titanium controls **[L. 7, 8]**, and their biocompatibility promotes the formation of new bone tissue **[L. 9, 10]**. The high machinability of magnesium alloys (easy machining), on the other hand, makes it possible to produce complex shapes with high dimensional accuracy, which is an added value in biomedical applications **[L. 11–14]**. The main problem in industrial and medical applications is the high degree of degradation caused by low corrosion resistance **[L. 15, 16]** and significant tribological wear of Mg and its alloys **[L. 17]**.

Deep cryogenic treatment (DCT) is one of the possible methods leading to the improvement of the mechanical as well as tribological properties of engineering materials **[L. 18–20]**. It is most often conducted at a liquid nitrogen temperature (-196°C) **[L. 21]**. The effect of DCT on the properties of steels **[L. 22–24]** and aluminium alloys **[L. 25–27]** is quite well documented. Few scientific reports deal with magnesium alloys containing rare earth metals. Moreover, in magnesium alloys forming systems with limited solubility of the other component, sub-zero treatment contributes to streamlining the heat treatment process by increasing the number of precipitates **[L. 21, 28]**, as shown, inter alia, in our previous studies on WE54, in which we investigated the effect of deep cryogenic treatment and precipitation hardening on the structure, micromechanical properties and wear **[L. 29]**; we also determined sclerometric and mechanical properties **[L. 30]**, conducted tribological tests under various loads **[L. 31]** and started research on alloy WE43 **[L. 32]**. However, the literature lacks specific recommendations on the duration of deep cryogenic treatment for magnesium alloys. Therefore, in this paper, we have decided to check the effect of sub-zero treatment time on the properties of the WE43 and WE54 alloys (Mg-Y-Nd).

RESEARCH METHODOLOGY

The test materials were magnesium alloys with rare earth metals manufactured by Luxfer MEL Technologies (Manchester, UK), designated WE43 and WE54. The alloys were supplied in the form of rods with a diameter ∅ = 25.4 mm (1 in.). For micromechanical and tribological tests, specimens with a nominal diameter of the rod and a height of 5 mm were prepared. Specimens prepared for the compression test had a diameter ∅ = 6 mm and a height of 9 mm. The chemical compositions, as per the manufacturer's certificate, are presented in **Table 1**.

The alloys were subjected to deep cryogenic treatment (DCT) at a temperature of – 196°C (in liquid nitrogen). Each alloy was sub-zero treated for 2, 4, 8, 24 and 48h, respectively. Next, the micromechanical and tribological testing specimens

Table 1. Chemical composition of alloys WE43 and WE54 in the as-delivered state
Tabela 1. Skład chemiczny stopów WE43 i WE54 w stanie dostawy

Element content, wt-% [%]										
Alloy type	**Y**	**Re**	**Nd**	**Zr**	**Zn**	**Cu**	**Mn**	**Li**	**Ni**	Other impurities
WE43	4.0	3.0	2.3	0.49	0.01	0.002	0.02	0	0	<0.2
WE54	5.2	2.6	1.6	0.5	0.01	0.002	0.009	0.08	0.001	<0.2

were ground using abrasive paper with a grit size ranging from 320 to 4000 to obtain the same surface roughness of Ra = 0.05 μm. The roughness of the specimens was measured using a Surftest SJ-500 contact surface tester (Mitutoyo, Tokyo, Japan) in compliance with EN ISO 4288 (replaced with EN ISO 21920 in 2022), using a sampling length λc = lr = 0.25 mm and an evaluation length ln = 1.25 mm; three measurements were taken for each test specimen.

Micromechanical properties were measured using an MCT^3 (Micro Combi Tester) (Anton Paar, Corcelles-Cormondrèche, Switzerland) according to the recommendations of ISO 14577 **[L. 33]**. The maximum load on the Berkovich indenter was 250 mN, and the loading and unloading rate of the indenter was 500 mN/min, with the hold time under a maximum load of 10 s. The measurement results were averaged for each sample for nine indents (3x3 indentation matrix). By using the Oliver-Pharr method **[L. 34]**, the following quantities were determined: instrumental hardness H_{IT}, instrumental elastic modulus E_{IT}, and indentation work, including the percentage of the elastic component of indentation work η_{IT}.

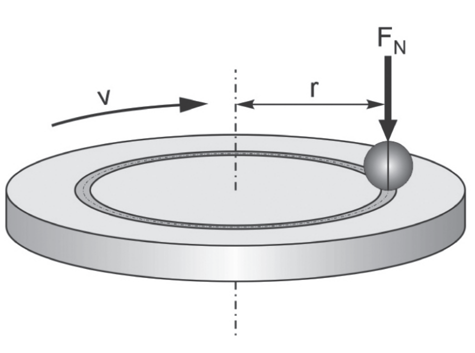
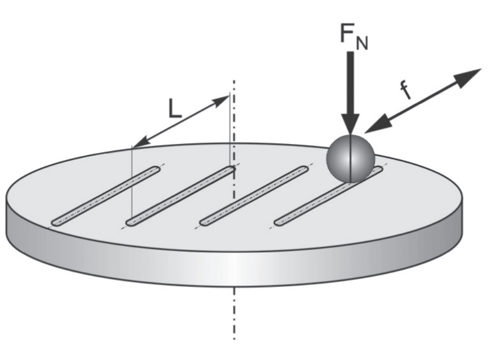
The effect of deep cryogenic treatment time on the mechanical properties of the WE43 and WE54 was examined in a uniaxial compression test performed with an Instron 5982 strength testing machine. The specimens with ∅ = 6 mm and initial height h_0 = 9 mm were compressed with the cross-bar speed of 1 mm/min. During the test, the dependence of the force (stress) on the shortening Δh (of the specimen) was recorded continuously. Next, such quantities as compressive strength – UCS (R_c), proof stress – UYS ($R_{c0,2}$) and relative shortening (A_c) were determined.

Tribological tests were carried out using a TRN tribometer (Anton Paar, Corcelles-Cormondrèche, Switzerland) in the counter-specimen (ball) – specimen (disc) system in rotary and reciprocating linear motion. The measurement conditions are shown in **Table 2**. ZrO_2 balls with a diameter ∅ = 6 mm were used as counter-specimens (for rotary motion) and ∅ = 3 mm (reciprocating motion). The wear was examined under technically dry friction conditions at room temperature of 21±1°C and air humidity of 40±5%, in accordance with the recommendations of VAMAS Technical Note and the ASTM G99 and ASTM G133 standards **[L. 35–37]**. During tribological tests, the following parameters were determined: average area of the wear trace P, volumetric wear V_w, linear wear L_W, and mean stabilised friction coefficient μ_{mean}.

The average area of the wear trace, *P*, was determined using a Surftest SJ-500 profilometer (Mitutoyo, Tokyo, Japan). Surface area measurements were averaged for four 2D profiles acquired every 90° (for rotary motion) and four 2D profiles from the central part of the linear wear trace (ASTM G133). Volumetric wear, V_W, was determined from the formula (1):

$$V_W = \frac{V}{F_N \cdot s} \left[\frac{mm^3}{Nm}\right] \qquad (1)$$

Table 2. Tribological test parameters
Tabela 2. Parametry testów tribologicznych

Rotary motion		Reciprocating linear motion	
			
Load F_N	10 N	Load F_N	5 N
Linear velocity v	0.15 m/s	Amplitude L	10 mm
Friction distance radius r	7 mm	Frequency f	2.5 Hz
Friction distance s	100 m	Friction distance s	50 m

(where: F_N – the load applied [N], s – friction distance [m], V – the volume of the wear trace calculated for rotary motion from the formula: $V = P \cdot 2\pi r$ [mm^3], P – the average area of the wear trace [mm^2], r – friction distance radius [mm]; and for reciprocating linear motion from the formula: $V = P \cdot L$ [mm^3], P – the average area of the wear trace [mm^2], L – friction distance amplitude [mm].

The morphology of the wear traces was examined using a JEOL JSM-6480 scanning electron microscope (Jeol, Tokyo, Japan). In order to identify the wear mechanisms, SEM images were acquired at a magnification from 30x to 2000x. This allowed taking images of the entire area of the wear trace in reciprocating linear motion and half of the wear trace in rotary motion.

RESEARCH RESULTS AND DISCUSSION

First, the effect of deep cryogenic treatment (DCT) time on the micromechanical properties of the WE43 and WE54 alloys was determined. Microindentation tests showed that the alloys had hardness H_{IT} = 916 MPa (WE43) and = 950 MPa (WE54). As the sub-zero treatment time increased, an increase in the hardness of both alloys was observed (**Fig. 1**). The best properties were obtained in the case of sub-zero treatment conducted for 24 hours, which resulted in an approx. 10% increase in hardness H_{IT} = 955 MPa (WE43) and = 1058 MPa (WE54). After exceeding the 24 h time, a decline in micromechanical properties was observed again.

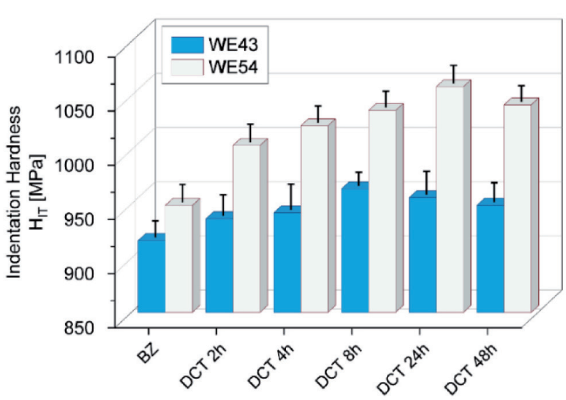


Fig. 1. Changes in hardness H_{IT} of alloys WE43 and WE54 with increasing time of deep cryogenic treatment

Rys. 1. Zmiany twardości H_{IT} stopów WE43 i WE54 wraz ze wzrostem czasu głębokiej obróbki kriogenicznej

Micromechanical tests also showed that deep cryogenic treatment did not significantly alter the elasticity of the two magnesium alloys tested (causing only a slight increase in Young's modulus). Young's modulus E_{IT} was approx. 49 GPa in the as-delivered state, and approx. 53 GPa after sub-zero treatment in liquid nitrogen for 24 h (**Fig. 2a**). The share of the elastic component of indentation work η_{IT} was 13–14% and also did not change significantly under sub-zero treatment (**Fig. 2b**).

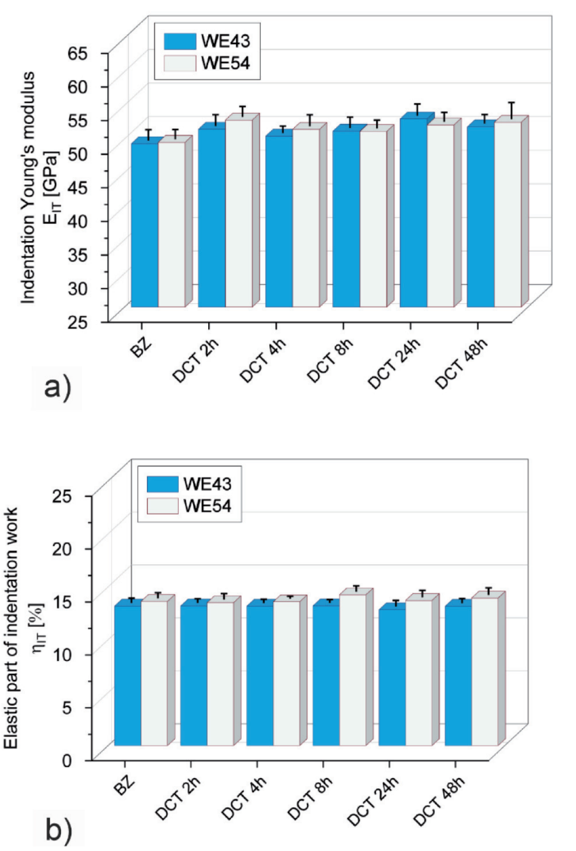


Fig. 2. Changes in Young's modulus E_{IT} – a) and in the percentage of the elastic component of indentation work η_{IT} – b) of alloys WE43 and WE54 with increasing time of deep cryogenic treatment

Rys. 2. Zmiany modułu Young'a E_{IT} – a) oraz sprężystej części pracy indentacji η_{IT} – b) stopów WE43 i WE54 wraz ze wzrostem czasu głębokiej obróbki kriogenicznej

Improved mechanical properties of magnesium alloys with rare earth metals after deep cryogenic treatment were also found during a uniaxial compression test (**Fig. 3**). Compressive strength, UCS (R_c), was shown to increase with increasing sub-zero treatment time for both magnesium alloys tested, with little change in plastic properties represented by proof stress, UYS ($R_{c0,2}$). The most favourable results, like in the case of micromechanical tests, were obtained for the specimens sub-zero treated for 24 h, where about 6–7% increase was found in their compressive strength, UCS (**Fig. 3a**), with an approx. 1-2% decrease in plastic properties for the WE43 alloy and an approx. 3–4% increase in UYS for the WE54 (**Fig 3b**), with relative shortening (A_c) ranging from 23 to 26%. Examples of curves recorded during the compression test for the specimens in the initial state and after the deep cryogenic treatment are shown in **Fig. 3c**. The improvement in the micromechanical and mechanical properties of Mg alloys WE43 and WE54 after sub-zero treatment has to do with a reduction in the number of defects, which, in turn, reduces voids in the crystalline lattice, the latter becoming more homogeneous. In addition, residual stresses are reduced during the sub-zero treatment **[L. 21]**.

Changes in mechanical and micromechanical properties induced by deep cryogenic treatment

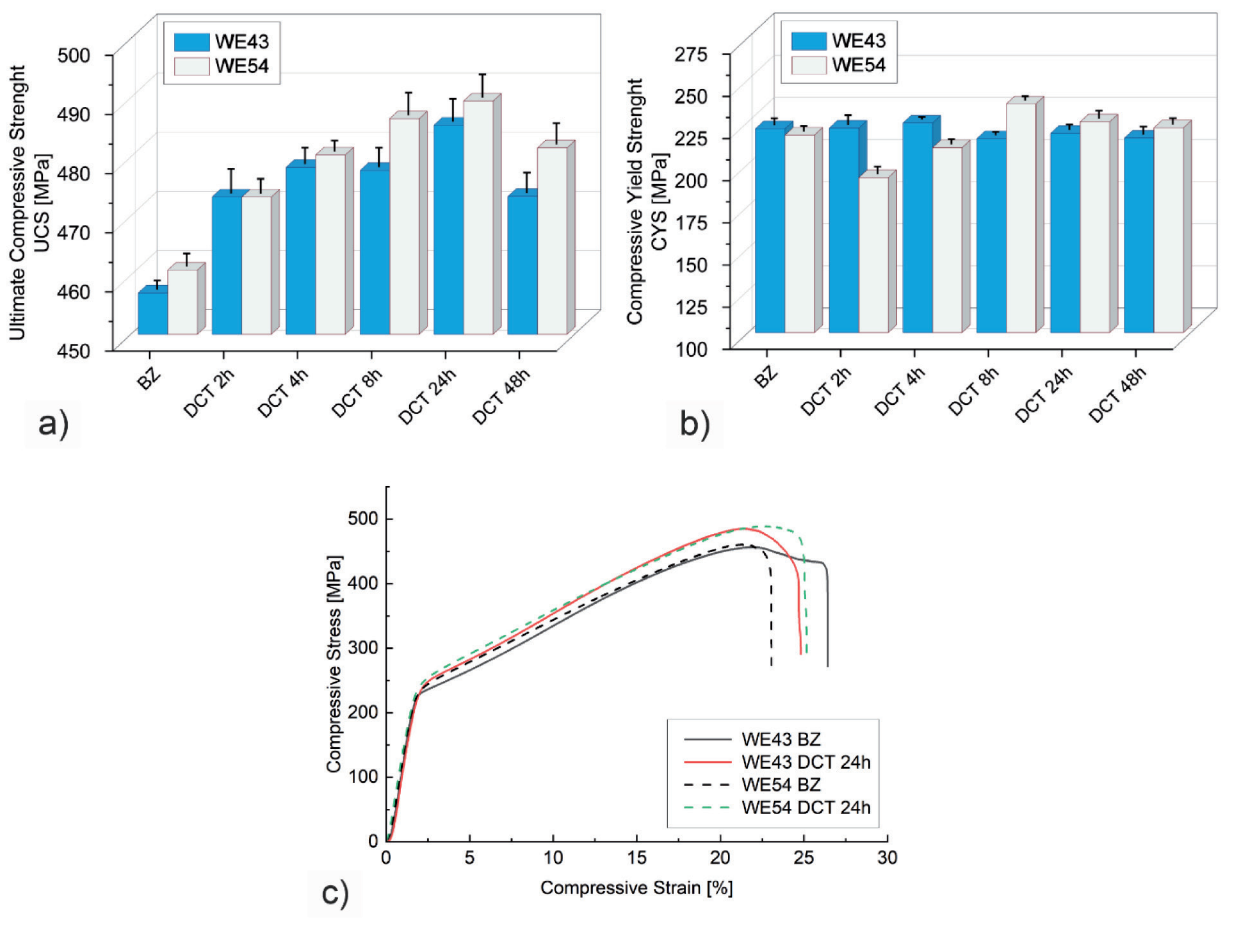


Fig. 3. Compression test parameters of Mg alloys WE43 and WE54 after deep cryogenic treatment: compressive strength UCS (R_c) – a), proof stress UYS ($R_{c0,2}$) – b) and examples of compression curves for the alloys in their initial state and after sub-zero treatment for 24 h – c)

Rys. 3. Parametry próby ściskania stopów magnezu WE43 i WE54 po procesie głębokiej obróbki kriogenicznej: wytrzymałość na ściskanie UCS (R_c) – a), umowna granica plastyczności UYS ($R_{c0,2}$) – b) oraz przykładowe krzywe ściskania dla stopów w stanie wyjściowym i po wymrażaniu przez 24 h – c)

directly affect the tribological properties of the alloys studied. For tribological tests in reciprocating linear motion and rotary motion, alloys in the as-delivered state were selected, as well as alloys after 24 h DCT with the most favourable mechanical properties. **Figure 4** shows the quantities recorded during tribological tests, including volumetric wear V_W (**Fig. 4a**), linear wear L_W (**Fig. 4b**), and mean stabilised friction coefficient μ_{mean} (**Fig. 4c**). Both investigated alloys in the initial state are characterised by high volumetric wear of about $2.3 \cdot 10^{-3}$ (mm^3/Nm) and linear wear of

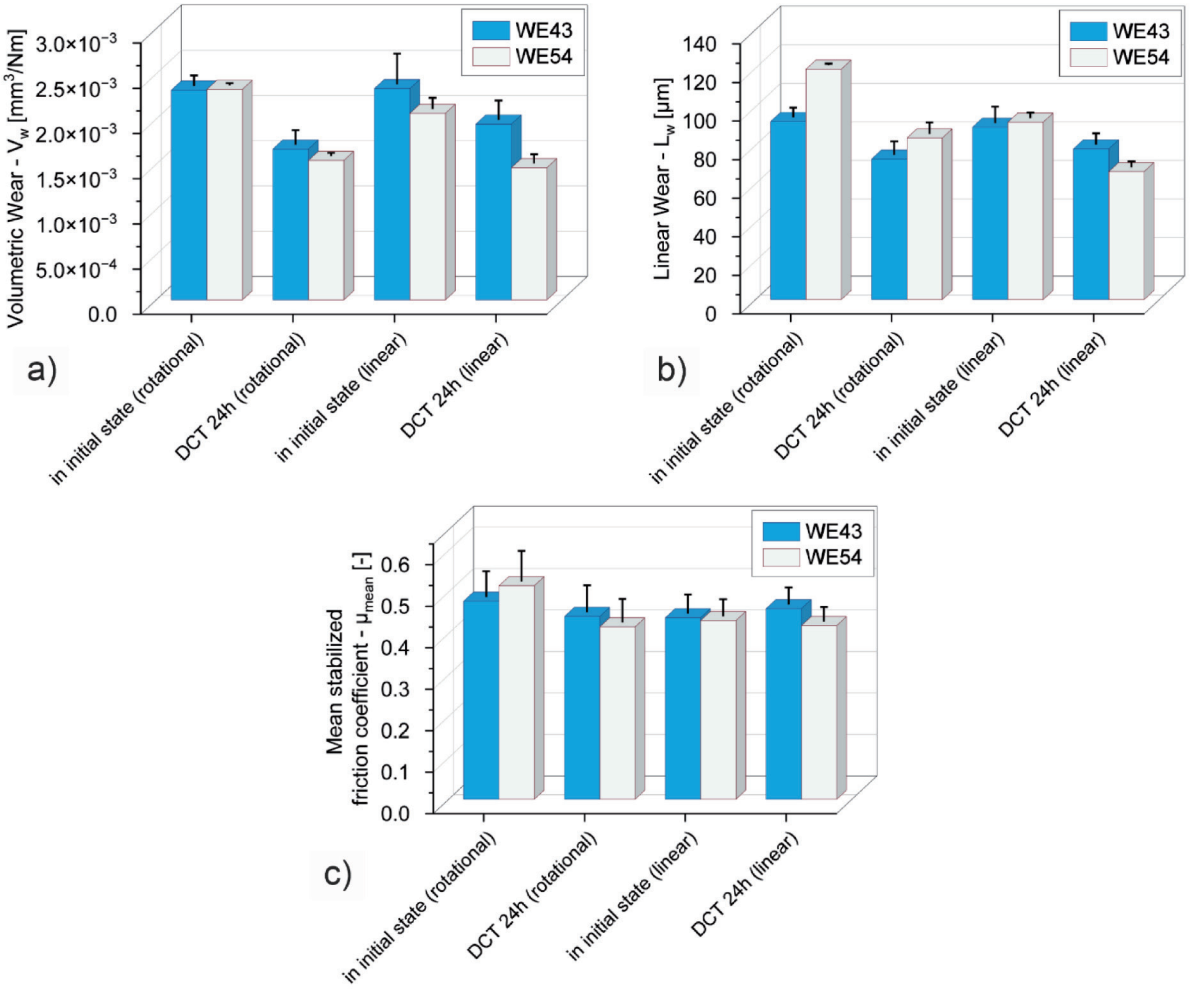


Fig. 4. Changes in volumetric wear V_W – a), linear wear L_W – b) and stabilised friction coefficient μ_{mean} of Mg alloys WE43 and WE54 in their initial state and after deep cryogenic treatment for 24 h

Rys.4. Zmiany zużycia objętościowego V_W – a), zużycia liniowego L_W – b) oraz ustabilizowanego współczynnika tarcia μ_{mean} stopów magnezu WE43 i WE54 w stanie wyjściowym i po procesie głębokiej obróbki kriogenicznej przez okres 24 h

90-120 μm. The introduction of deep cryogenic treatment for the Mg alloys allows for an approx. 30-35% reduction in volumetric wear, as well as a considerable reduction in linear wear, which, for the WE54 alloy, was nearly double (**Fig. 4a, b**). No wear of the balls (ZrO_2 counter-specimens) was recorded due to, inter alia, the large difference in hardness of the materials tested.

In addition to the improvement in mechanical and micromechanical properties, the decrease in tribological wear was influenced by the reduction in the mean stabilised friction coefficient, μ_{mean}, observed after the deep cryogenic treatment (DCT). In the case of the WE43 alloy, the sub-zero treatment caused a reduction of the friction coefficient in rotary motion from 0.48 to 0.44 and from 0.52 to 0.43 for the WE54. In reciprocating linear motion, the stabilised coefficient of friction (dynamic) was in the range from 0.41 to 0.43.

Examination of the wear trace morphologies, in turn, provided information on the wear mechanisms which occurred during tribological tests of alloys WE43 and WE54. **Figure 5** presents examples of SEM images of the wear traces of Mg alloy WE54 in the as-delivered state and after deep cryogenic treatment for 24 h in reciprocating linear motion, and **Figure 6** shows images of the wear traces of the alloy in rotary motion.

In both reciprocating linear motion and rotary motion, the dominant wear mechanism was sliding wear manifested in the form of numerous grooves and depressions formed parallel to the direction of sliding. In the case of reciprocating motion, areas of microcutting and adhesion are also present,

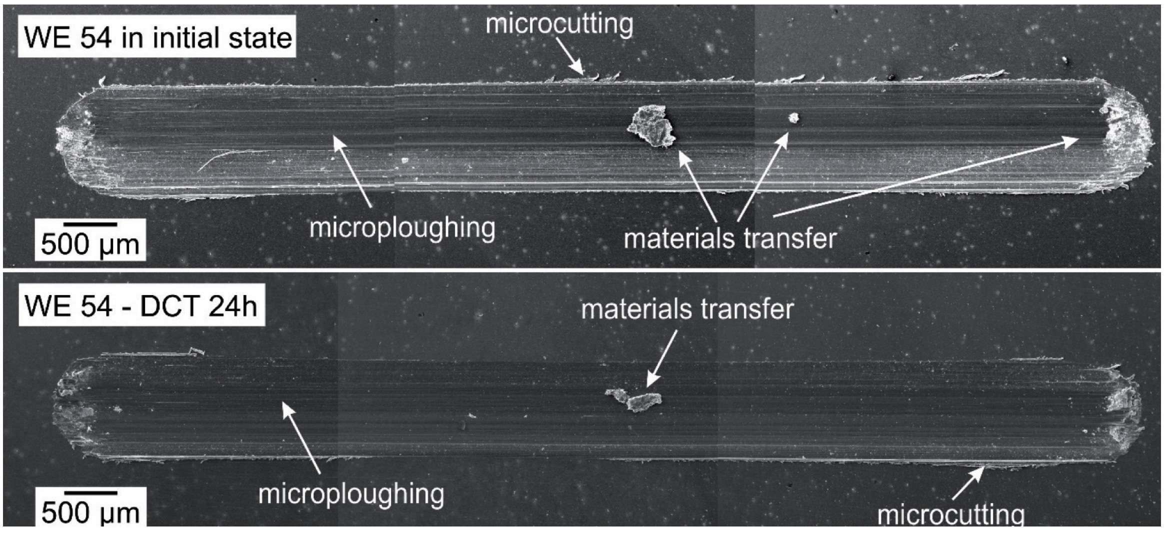


Fig. 5. Morphologies of the wear traces of Mg alloy WE54 formed in reciprocating linear motion in the as-delivered state and after deep cryogenic treatment for 24 h

Rys. 5. Morfologia śladów wytarcia stopu magnezu WE54 powstałych w ruchu posuwisto-zwrotnym w stanie dostawy oraz po głębokiej obróbce kriogenicznej w czasie 24 h

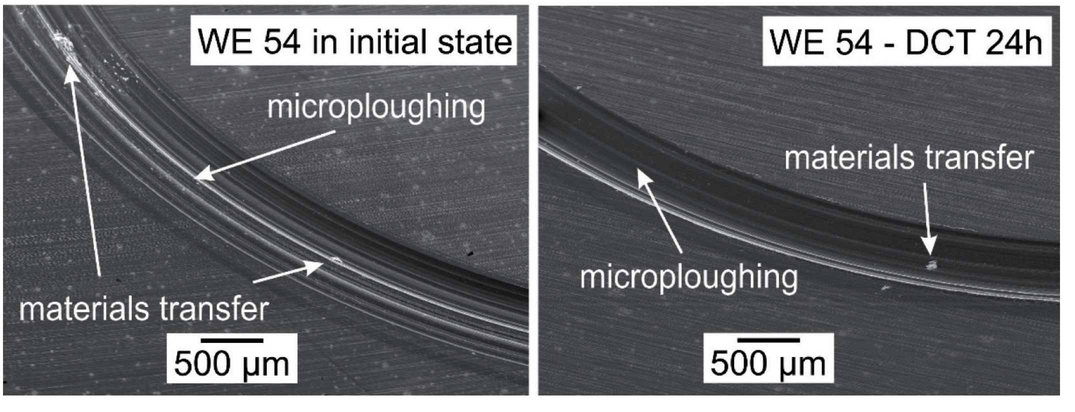


Fig. 6. Morphologies of the wear traces of Mg alloy WE54 formed in rotary motion in the as-delivered state and after deep cryogenic treatment for 24 h

Rys. 6. Morfologia śladów wytarcia stopu magnezu WE54 powstałych w ruchu obrotowym w stanie dostawy oraz po głębokiej obróbce kriogenicznej w czasie 24 h

especially at the ends of the trace deposited when the direction of motion changes. Deep cryogenic treatment of magnesium alloys with rare earth metals (WE43 and WE54) effectively reduces the cutting process and the formation of deep scratches, directly reducing tribological wear (**Fig. 5** and **6**). Together with the improvement in mechanical and micromechanical properties, this promises much higher durability for the two alloys tested.

CONCLUSIONS

- Gradually increasing the duration of deep cryogenic treatment of WE43 and WE54 from 2 to 24 h induces changes in their micromechanical properties (increase in microhardness and Young's modulus), while longer (48h) sub-zero treatment causes a decrease in the recorded parameters. The most favourable properties were found for the alloys subjected to sub-zero treatment for 24 hours, in which case an approx. 10% increase in the micromechanical properties was observed compared to the alloys in the as-delivered state.
- Improvement in the mechanical properties induced by a change in the duration of sub-zero treatment was also noted during uniaxial compression testing. Similarly to the

micromechanical properties, the mechanical properties increase with increasing sub-zero treatment time; the most favourable results were obtained for specimens subjected to deep cryogenic treatment for 24 hours. In that case, the alloys showed a 6–7% increase in compressive strength, USC, with little variation in plastic properties and relative shortening.

- Changes in magnesium alloys' micromechanical and mechanical properties after deep cryogenic treatment consequently improved their tribological properties: a 35% reduction in volumetric wear, a 2-fold reduction in linear wear, and a 20% decrease in the coefficient of friction were observed relative to the initial material. The best results of tribological tests were obtained for the WE43 and WE54 alloys after a 24 h period of sub-zero treatment.
- Examination of the wear trace morphologies showed that during friction in rotary motion, the acting wear mechanisms are: abrasion, microcutting and adhesion, while in reciprocating motion, in addition, traces of plastic deformation and areas of delamination appear. The main wear mechanism for the WE43 and WE54 alloys was abrasive wear.
- Increasing the duration of deep cryogenic treatment effectively reduces the cutting process and the formation of deep scratches during abrasive wear of both tested alloys (WE43 and WE54), which, combined with the improvement of micromechanical and mechanical properties, testifies to the enhancement of their service life.

FUNDING

This work was supported by the Polish National Science Centre (Polish: Narodowe Centrum Nauki, abbr. NCN) under the research project no. 2020/04/X/ST5/00256.

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