

INFLUENCE OF DEEP CRYOGENIC TREATMENT
ON THE MECHANICAL AND PLASTIC PROPERTIES
OF AlMg4.5 ALUMINIUM ALLOY

Plamen Stoyanov[✉], Daniela Spasova

Received on October 13, 2025

Presented by Ch. Roumenin, Member of BAS, on February 24, 2026

Abstract

In this article, the influence of deep cryogenic treatment (DCT) on the microstructure, as well as on the strength and ductile mechanical characteristics, of the AlMg4.5 (EN AW-5083) aluminium alloy is investigated. The DCT process consists of the gradual cooling of the investigated material to a temperature of -197°C using liquid nitrogen, maintained for 24 and 48 hours, followed by gradual tempering back to room temperature. The changes in mechanical properties resulting from DCT were analyzed through Vickers hardness measurements and tensile testing, while the structural changes were identified by means of microstructural analysis and X-ray diffraction analysis (XRD). The results indicate structural-level changes after DCT, accompanied by an improvement in mechanical properties.

Key words: aluminium alloy AlMg4.5, deep cryogenic treatment, mechanical properties, hardness, microstructure, X-ray diffraction analysis

Introduction. Aluminium alloys from the Al–Mg system are widely used in modern industry due to their low density, high corrosion resistance, good ductility, and favourable strength-to-weight ratio. These properties make them a preferred material in applications requiring a combination of lightness and strength, such

The authors express their gratitude for the support of Research Project NP-10/2025, funded through the state budget, within the framework of the research activities at Technical University of Varna.

<https://doi.org/10.7546/CRABS.2026.04.07>

as shipbuilding, transportation engineering, aerospace structures, as well as the production of tanks, pipelines, and architectural components [1, 2].

Among them, the AlMg4.5 alloy (EN AW-5083) occupies a particularly important place, as it combines excellent resistance to marine corrosion, good weldability, and satisfactory mechanical properties without the need for heat strengthening. However, under intensive operation and dynamic loading conditions, the material may undergo structural changes that lead to metal fatigue and deterioration of its service performance [3–5].

In the pursuit of improving the properties of aluminium alloys, various innovative technological approaches have been explored including modification of the chemical composition, incorporation of nano-additives, optimization of thermomechanical processes, and the use of deep cryogenic treatment (DCT) as an additional stage following conventional processing.

Deep cryogenic treatment is a process in which the material is cooled to temperatures below -197°C , typically using liquid nitrogen, in order to modify the internal structure of the metal. During this treatment, several microstructural transformations may occur – reduction of residual stresses, increase in dislocation density, refinement of grain structure, and possible redistribution of secondary phases [6, 7]. As a result, treated materials often exhibit increased hardness, dimensional stability, improved wear resistance, and enhanced fatigue strength.

While the effects of DCT are well established and routinely applied in tool steels, research on non-ferrous metals – particularly aluminium alloys – remains limited and often yields contradictory results [8, 9]. Some studies report improvements in hardness and tensile strength after cryogenic treatment, whereas others observe negligible changes or even a reduction in ductility. These inconsistencies are likely due to differences in chemical composition, microstructure, degree of deformation, and cooling parameters, highlighting the need for more detailed experimental investigations [10].

Materials and methods. The present study aims to analyze the effect of deep cryogenic treatment (DCT) on the structural and mechanical properties of the aluminium alloy AlMg4.5. Through a comparative approach between treated and untreated specimens, the research seeks to provide a deeper understanding of the potential for optimizing material characteristics by applying cryogenic processing technologies.

Materials. The investigated material is a cold-rolled, strain-hardened AlMg4.5 alloy, typically supplied in plate form. Similar to other high-magnesium alloys from the 5000 series, AlMg4.5 exhibits high corrosion resistance – including in marine environments – good weldability, and increased strength achieved through cold working. With increasing magnesium content, the mechanical properties improve, although corrosion resistance tends to decrease. The chemical composition of the AlMg4.5 aluminium alloy is as follows (in wt.%): Al – 92.545–95.55; Mg – 4.0–4.9; Mn – 0.4–1.0; Fe \leq 0.4; Si \leq 0.4 [11].

Methods. *Deep cryogenic treatment.* The influence of DCT was studied on the strain-hardened AlMg4.5 alloy. Pre-machined specimens were subjected to slow and gradual cooling to -80°C , followed by immersion in a Dewar vessel containing liquid nitrogen for deep cryogenic treatment lasting 24 and 48 h, respectively.

The preliminary cooling to -80°C was carried out by immersion in a thermos vessel containing a solid CO_2 (dry ice)–acetone medium. After reaching -80°C and holding for 10 min, the specimens were transferred into a liquid nitrogen environment, achieving a temperature of -197°C [12, 13].

Tempering back to room temperature was also performed gradually and uniformly: the specimens were removed from the Dewar vessel and placed in the thermos for approximately 10 min to ensure a controlled temperature rise.

Tensile and hardness testing. Tensile strength testing was performed according to BDS EN ISO 6892-1:2019 – Metallic materials – Tensile testing – Part 1: Method of test at room temperature [14]. In compliance with the standard, three specimens of each type were prepared: untreated reference samples, and samples subjected to cryogenic treatment for 24 and 48 h, respectively.

Hardness measurements were carried out using the Vickers method on a digital microhardness tester HVS-1000 under a load of 1 kg. For each specimen, hardness was measured in three different regions, and the average value was calculated.

Structural analysis. Samples for metallographic examination were prepared from the tested specimens. Microstructural observations were conducted using a trinocular inverted metallographic microscope IM-3MET.

In addition, a qualitative X-ray diffraction (XRD) phase analysis was performed using a TDM-10 Tongda diffractometer. $\text{Cu K}\alpha$ radiation with a wavelength of $\lambda = 1.79026 \text{ \AA}$ was used at room temperature. XRD patterns were recorded in the 2θ range of 30° – 90° under a coupled-scan mode. Phase identification and peak matching were carried out using the X'Pert HighScore Plus software package for XRD analysis [15].

Results and analysis. *Results from tensile testing and hardness measurements.* The obtained results show that, for the AlMg4.5 alloy, deep cryogenic treatment has a positive effect on the improvement of mechanical strength properties (Table 1). A proportional increase in the strength-related mechanical characteristics is observed, as the structure of the base material is also mechanically strengthened. The greatest increase in tensile strength and hardness occurs after 24 h of DCT (deep cryogenic treatment), where the average tensile strength (R_m) rises from 305 MPa to 321 MPa (Fig. 1). For the samples subjected to 48 h of DCT, the tensile strength increases by only 9 MPa compared to the reference samples. Slight changes are also observed in the elongation at break after 48 h of DCT – approximately by 1.5%. Furthermore, in the AlMg4.5 samples, no significant change in hardness is observed after 48 h of DCT – increasing only from

T a b l e 1

Values of the mechanical properties obtained after tensile strength testing and hardness measurements of the AlMg4.5 alloy

Processing	F_m N	R_m MPa	$R_{m\text{ av.}}$ MPa	A %	$A_{\text{av.}}$ %	Hardness HV1	Hardness HV1 av.
Reference	15000	306		6.68		85	
	14950	304	305	6.42	6.01	84	85
	14980	305		6.14		86	
24-h DCT	15500	322		6.92		98	
	15200	320	321	6.84	6.91	99	99
	15300	320		6.68		99	
48-h DCT	15150	313		7.12		91	
	15000	315	314	7.19	7.42	90	91
	15130	314		7.05		92	

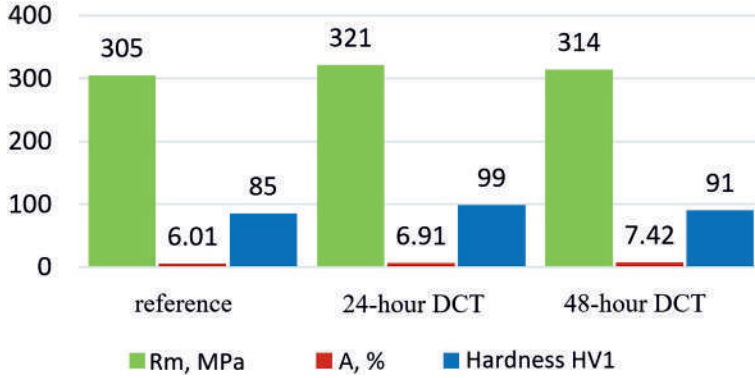


Fig. 1. Comparative diagram of the measured hardness (HV1), tensile strength (R_m , MPa), and elongation at break (A , %) of the AlMg4.5 alloy

85 HV to 91 HV – whereas after 24 h of DCT, the hardness increases by 14 units, reaching 98 HV.

Results from the microstructural analysis. At higher magnification, as seen in Fig. 2b, after 24 h of DCT (Deep Cryogenic Treatment), the texture fibres appear more pronounced and denser compared to those of the initial structure. This is attributed to the segregation and densification of the secondary phase along the fibre boundaries, which leads to an increase in the mechanical strength properties. This process is also confirmed by the conducted X-ray diffraction phase analysis.

After 48 h of DCT (Fig. 2c), the fibre density is less pronounced compared to the structure treated for 24 h, but still more distinct than that of the reference sample. This explains the observed decrease in mechanical strength after 48 h of DCT. Prolonged DCT leads to the breakdown of the coherent bond between the secondary phase and the matrix, resulting in a reduction of dislocation density, and consequently, a decrease in the mechanical strength properties.

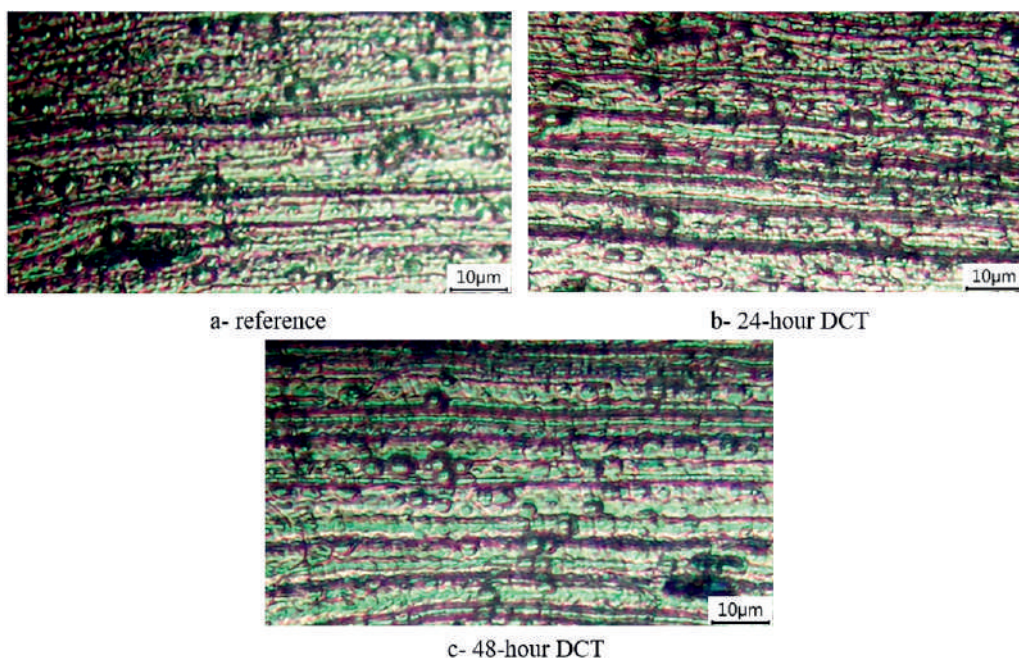


Fig. 2. Microstructure of the AlMg4.5 aluminium alloy, $\times 800$

Results from the X-ray diffraction phase analysis. The X-ray diffraction phase analysis of the investigated AlMg4.5 samples also showed no new phase formations after deep cryogenic treatment (Fig. 3). After 24 and 48 h of DCT, as seen in Fig. 3b and c, the diffraction peaks shift toward higher 2θ angles, i.e., toward smaller interplanar spacings. This indicates that the α -solid solution becomes depleted in dissolved Mg, resulting in a reduction of the lattice parameter of Al. This effect is explained by the contraction of the α -solid solution lattice due to DCT.

It is also observed that the integral intensity of the diffraction line of α -Al with the (220) crystallographic planes increases after DCT. Calculations performed using Graph software show that the integral intensity is 37.88 for the reference sample, 65.02 after 24 h of DCT, and 50.42 after 48 h of DCT. The highest value is observed after 24 h of DCT, and a larger integral intensity indicates an increase in dislocation density and microstresses, which leads to structural strengthening. After 48 h of DCT, the integral intensity decreases compared to the 24-h treatment, indicating a reduction in third-order microstresses, which explains the observed increase in elongation at break.

Conclusion. The conducted investigations show that deep cryogenic treatment exerts a noticeable influence on the microstructure and mechanical properties of the AlMg4.5 aluminium alloy. It was established that a technological regime involving a 24-h DCT duration leads to improved results in terms of increased tensile strength and hardness, while the ductility is preserved. This effect

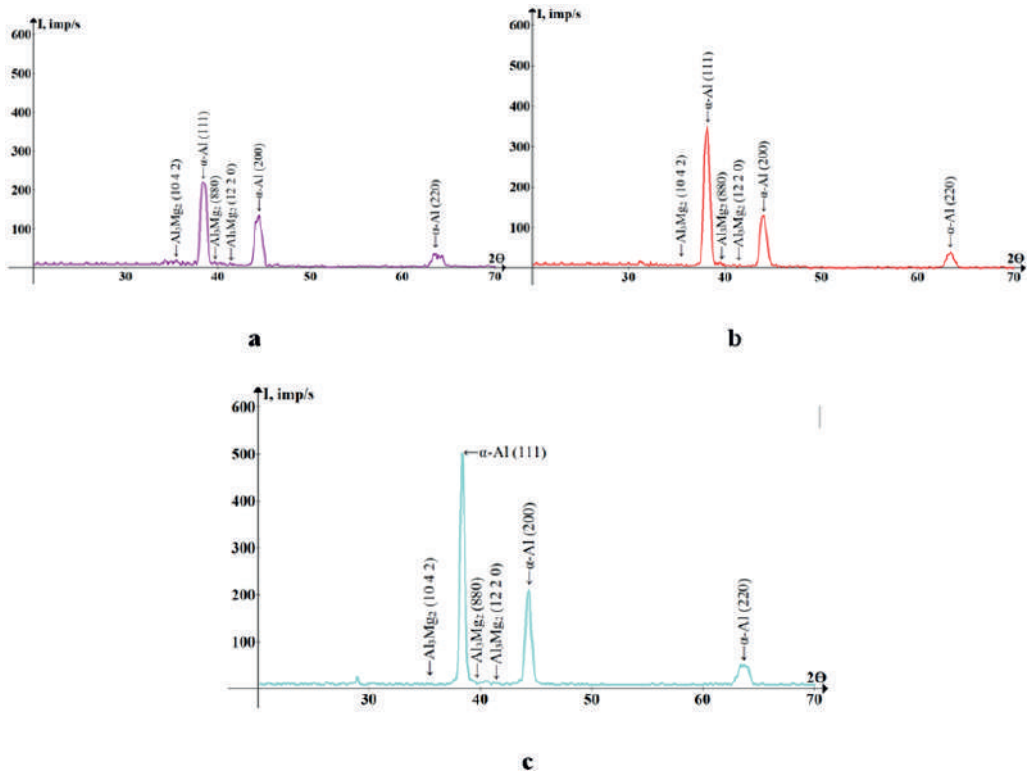


Fig. 3. Results from the X-ray diffraction phase analysis of the AlMg4.5:
 (a) reference, (b) 24-h DCT, (c) 48-h DCT

is attributed to the redistribution and densification of the secondary phase along the grain boundaries, as well as to an increase in dislocation density, which results in hardening of the material. During DCT for 48 h, a partial disruption of the coherent interfaces between the secondary phase and the matrix is observed, leading to a reduction in microstresses and, consequently, to a slight decrease in the strength characteristics, while simultaneously resulting in an increase in the value of the relative elongation A (an indicator of ductility). The shift of the peaks toward higher values of the 2θ angle, i.e., toward smaller interplanar spacings, registered by the conducted X-ray diffraction (XRD) phase analysis, indicates that the α -solid solution becomes depleted of the Mg dissolved in it. Furthermore, the observed increase in the integrated area of the diffraction peaks after DCT confirms an increase in dislocation density, which in turn leads to higher values of the mechanical properties.

REFERENCES

- [1] WAHID M. A., A. N. SIDDIQUEE, Z. A. KHAN (2020) Aluminum alloys in marine construction: characteristics, application, and problems from a fabrication viewpoint, *Mar. Syst. Ocean Technol.*, **15**, 70–80.

- [2] GRABNER F., J. A. ÖSTERREICHER, B. GRUBER et al. (2019) Cryogenic forming of Al-Mg alloy sheet for car outer body applications, *Adv. Eng. Mater.*, **21**(8), <https://doi.org/10.1002/adem.201900089>.
- [3] LEE B. H., S. H. KIM, J. H. PARK et al. (2016) Role of Mg in simultaneously improving the strength and ductility of Al-Mg alloys, *Mater. Sci. Eng. A* **657**, 115–122, <https://doi.org/10.1016/j.msea.2016.01.089>.
- [4] BALDISSERA P., C. DELPRETE (2008) Deep cryogenic treatment: a bibliographic review, *Open Mech. Eng. J.*, **2**(1), 1–11, <https://doi.org/10.2174/1874155x00802010001>.
- [5] MUKHINA I. YU. (2011) Structure and properties of new cast magnesium alloys, *Foundry Production*, **12–14**, 6, (in Russian).
- [6] MUKHINA I. YU., V. V. STEPANOV, Z. P. URIDIYA (2013) Influence of melting and casting technology on the properties of magnesium alloys, *Coll. reports of the conference “Problems of creating new materials for the aerospace industry in the 21st century”*, Moscow, VIAM, p. 74, (in Russian).
- [7] SPASOVA D., P. STOYANOV, P. PETROV (2024) Effect of deep cryogenic treatment on microstructure and mechanical properties of AlMg6 aluminum alloy, *Int. J. “NDT Days”*, **7**(2), 67–74.
- [8] SPASOVA D., P. STOYANOV, P. PETROV et al. (2024) Effect of deep cryogenic treatment on the mechanical properties of AlSi7Mg casting alloy, *Innovative Manufacturing Engineering and Energy – IManEE2024*, 260–268, <https://doi.org/10.21741/9781644903377-34>.
- [9] KARKI B. S., A. MISRA (2024) A technical review on deep cryogenic treatment of aluminum alloys: microstructural and performance enhancements, *Int. Res. J. Eng. Technol. (IRJET)*, **11**(9), 580–586.
- [10] ZHOU C., Q. SUN, D. QIAN et al. (2022) Effect of deep cryogenic treatment on mechanical properties and residual stress of AlSi10Mg alloy fabricated by laser powder bed fusion, *J. Mater. Process. Technol.*, **303**(11), 117543, <https://doi.org/10.1016/j.jmatprotec.2022.117543>.
- [11] Information on https://www.splav-kharkov.com/en/e_mat_start.php?name_id=1418.
- [12] LIU X., K. GU, Z. WENG et al. (2020) A review on deep cryogenic treatment of aluminium alloy, *Materials Reports*, **34**(3), 3172–3177, <https://doi.org/10.11896/cldb.19040288>.
- [13] PAGIDIPALLI B., C. NASHINE, P. BHANSALI (2019) Influence of deep cryogenic treatment on the wear behaviour of different Al–Si Alloys: Select Proceedings of ICEMMM 2018. In book: *Advances in Materials and Metallurgy*, 217–225, https://doi.org/10.1007/978-981-13-1780-4_22.
- [14] Information on <https://www.iso.org/standard/78322.html>.
- [15] YANKOVA R., I. RUSEV (2025) Corrosion resistance and antifouling performance of selective plated nickel-based coatings with silver particles, *C. R. Acad. Bulg. Sci.*, **78**(3), 419–427, <https://doi.org/10.7546/CRABS.2025.03.12..>

Technical University – Varna, 1 Studentska St, 9010 Varna, Bulgaria
e-mails: plamen.stoyanov@tu-varna.bg, danielats@tu-varna.bg