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CONTENTS

Impact of deep cryogenic treatment on microstructural and electrical properties of recycled aluminium alloys

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ABSTRACT: This study investigated the impact of Deep Cryogenic Treatment (DCT) on the microstructural and electrical properties of a recycled aluminium alloy, aiming to enhance its performance. Samples were subjected to DCT at temperatures below -196°C, and their properties were compared to those of the untreated samples. Microstructural analysis using optical microscopy revealed a significant reduction in the size and density of the inclusions and defects after DCT, indicating a more refined and homogeneous microstructure. Electrical resistivity measurements demonstrated a substantial decrease in both resistance and resistivity following DCT, suggesting improved electrical conductivity. These enhancements are attributed to the phase redistribution, impurity dissolution, stress relaxation, and reduced dislocation density induced by the cryogenic treatment. These findings highlight the potential of DCT to effectively optimise the microstructural and electrical properties of aluminium alloys derived from scrap materials, making them suitable for applications requiring enhanced conductivity and mechanical performance. This research suggests that DCT is a viable approach for improving the quality and applicability of recycled aluminium alloys.

Keywords: Cryogenic treatment, electrical resistivity, microstructure, material properties, scraps aluminium alloy

Aluminium and its alloys are indispensable materials across diverse industries due to their favourable strength-to-weight ratio, corrosion resistance, and electrical conductivity. Sustainable material utilization necessitates efficient scrap aluminium recycling, yet impurities and microstructural inconsistencies in recycled alloys can negatively impact their electrical resistivity. Cryogenic treatment (CT), an emerging technique, offers a potential pathway to refine microstructural properties and enhance material performance. This study investigates the influence of cryogenic treatment on the electrical resistivity of scrap aluminium alloys and explores its industrial significance.

Cryogenic treatment of aluminium alloys can induce a decrease in electrical resistance through several mechanisms. Cryogenic treatment has been shown to induce significant microstructural changes in various alloys, including aluminium alloys. For instance, in Ti-6Al-4V alloy, cryogenic treatment led to refinement of the microstructure, reduced β-phase volume fraction, and increased dislocation density (Kang *et al*., 2024). Similar microstructural alterations could potentially occur in aluminium alloys, affecting their electrical properties.

Interestingly, cryogenic treatment has been found to improve thermal conductivity in some materials (Kalsi *et al*., 2010). Since thermal and electrical conductivity are often correlated in metals, this suggests that cryogenic treatment might also enhance electrical conductivity (i.e., decrease electrical resistance) in aluminium alloys. These include microstructural changes, such as the formation of fine precipitates and reduction of lattice defects, leading to a more ordered crystal structure. Thermal contraction can reduce dislocation density. Stress relief, which improves conductivity, is achieved by minimizing internal stresses.

Cryogenic treatment induces significant microstructural changes in alloys, including refinement of grain structure, reduction in retained austenite, and precipitation of fine carbides (Jovičević-Klug *et al*., 2021; Kang *et al*., 2024; Li *et al.,* 2018). These changes can potentially affect the electrical properties of the material. For instance, the reduction in grain size and increased dislocation density observed in Ti-6Al-4V alloy after cryogenic treatment (Kang *et al.,* 2024) could impact electron scattering and thus electrical resistance.

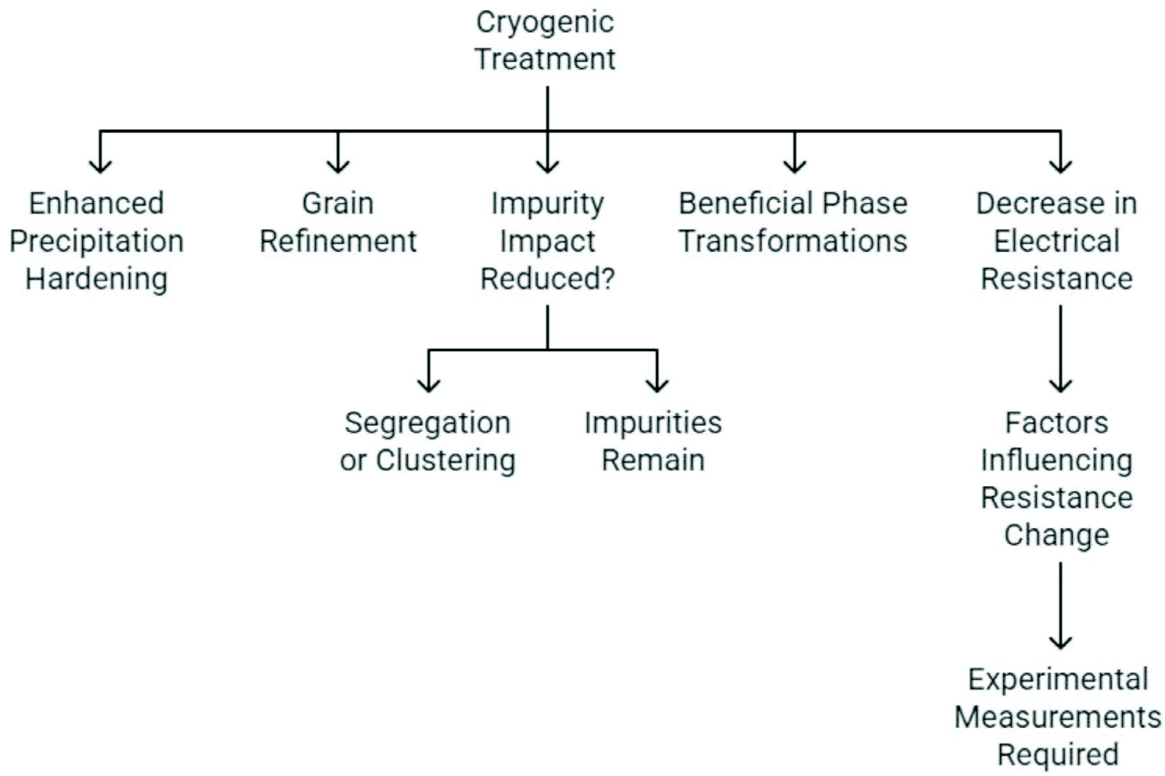


Fig. 1: Effect of Cryogenic on Aluminium Alloy

Interestingly, the papers highlight that the effects of cryogenic treatment vary depending on factors such as alloy composition, treatment temperature, duration, and post-treatment processes. For example, (Barylski *et al.,* 2023) shows that lowering the aging temperature and introducing deep cryogenic treatment led to favourable microstructural changes in WE43 alloy. (Çakir and Çelik, 2020) demonstrate that the duration of deep cryogenic treatment (24 vs. 36 hours) affects the plasticity and microstructure of Ti6Al4V alloy differently.

In some alloys, cryogenic treatment can enhance precipitation hardening, resulting in a more uniform distribution of strengthening particles. Grain refinement can also occur. The impact of impurities may be reduced through segregation or clustering. Finally, beneficial phase transformations can occur in specific alloys. The magnitude of the electrical resistance decrease depends on factors such as the specific alloy composition, initial heat treatment condition, cryogenic treatment temperature and duration, and any post-cryogenic heat treatment processes. Quantifying this change requires experimental measurements of electrical resistivity on specific alloy samples before and after cryogenic treatment.

Properties of Aluminium Wire

i. Material Considerations: Aluminium is a relatively low-resistance material compared to other metals like copper. However, the resistance of aluminium is higher than copper, so you'll need to account for that.

ii. Effect of Heat Treatment and Cryo-Treatment:

a) Heat Treatment: Aluminium alloys are commonly heat-treated to improve mechanical properties, such as strength and hardness. However, heat treatment can also increase the wire's resistance due to changes in microstructure.

b) Cryogenic Treatment: Cryogenic treatment (deep freezing) typically aims to reduce residual stresses and enhance the material's strength, but it may slightly alter the electrical properties, potentially reducing resistance in some cases, as it could influence the electron mobility within the wire.

The impact of these treatments might make the wire's resistance slightly different from untreated aluminium. Heat-treated or cryo-treated aluminium may have a slightly higher or lower resistance compared to untreated material, depending on the specific alloy and treatment process used.

While some studies have focused on the impact of CT on mechanical properties like wear resistance, which is often increased by CT (contrary to an initial assumption that it might decrease resistivity), the direct relationship between CT and electrical resistivity in aluminium alloys requires further investigation. For instance, research has demonstrated improvements in wear resistance and mechanical properties in alloys like AZ91 magnesium alloy (Asl *et al.,* 2009), unspecified aluminium alloys (Steier *et al*., 2016), and 7075 aluminium alloy (Wei *et al.,* 2018) following cryogenic treatment. These improvements are attributed to microstructural modifications, including changes in precipitate distribution, dissolution of secondary phases, and stress relief. While these studies highlight the impact of CT on related properties, the precise influence on electrical resistivity remains an open question. Some research suggests a decrease in electrical resistivity after CT in alloys like AA7020 (Eivani *et al.,* 2009) and Cu-11Fe composites (Liu *et al*., 2022), attributed to factors like the depletion of alloying elements from the matrix. However, the effect can be alloy-specific, as seen in AA 6061 where CT influenced electrical conductivity, but the nature of this influence requires further clarification (Gogte *et al.,* 2014).

This study aims to address this gap by systematically investigating the effect of cryogenic treatment on the electrical resistivity of architectural scrap aluminium alloys. The research will explore the underlying mechanisms responsible for any observed changes and assess the potential of CT for enhancing the electrical performance of recycled aluminium alloys in low-resistivity, high-conductivity applications. This introduction highlights the need for further research, setting the stage for the experimental investigation and analysis that will follow in the subsequent sections of the paper.

MATERIALS AND METHODS

Materials and Sample Preparation

The scrap aluminium alloy samples were collected and cast with the addition of silicon and copper (5 wt% each) to enhance the strength and cast ability of the material. The compositional analysis results are presented in Table 1. Based on the chemical composition—dominated by aluminium, with notable amounts of magnesium, copper, and minor elements such as iron, manganese, zinc, and trace silver—the alloy resembles a modified 2xxx or 7xxx series, but due to the mixed-source scrap origin and added elements, it is best categorized as a non-standard, recycled aluminium alloy with similarities to Al-Cu-Mg or Al-Zn-Cu systems.

The samples were cut into cylindrical specimens with dimensions of 4 mm diameter and 50 mm length for electrical resistivity testing, providing a consistent geometry for evaluating the influence of alloying elements and microstructure on electrical performance.

Table 1: Compositional Analysis of Alloys

Spectrum 3				
Element	Signal Type	k Ratio	Wt%	Atomic %
Al	EDS	0.11372	88.82	95.74
Mn	EDS	0.00022	0.16	0.07
Mg	EDS	0.0062	5.1	1.8
Fe	EDS	0.00147	1.06	0.48
Cu	EDS	0.00595	4.48	1.78
Zn	EDS	0.00039	0.29	0.11
Ag	EDS	0.00010	0.09	0.02
Total			100.00	100.00

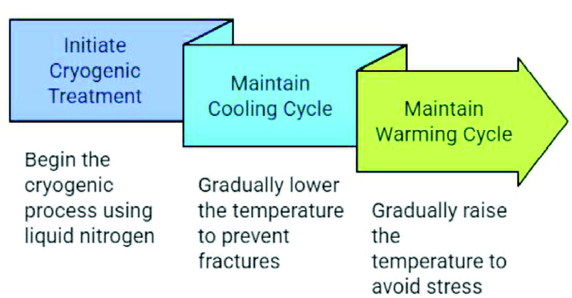


Fig. 2: Cryogenic Treatment Process

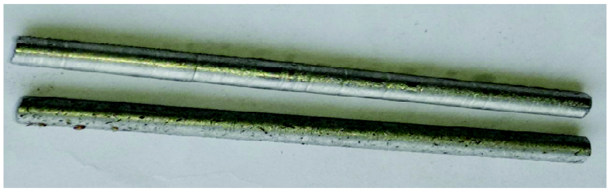


Fig. 3: Aluminium Alloy Electric Wire Sample

Cryogenic Treatment Process

The samples underwent controlled cryogenic treatment at temperatures below -196°C using liquid nitrogen immersion for 24 hours. A gradual cooling and warming cycle were maintained to prevent thermal stress-induced fractures.

Electrical Resistivity Measurement

The electrical resistivity of the treated and untreated samples was measured using a multimeter. The resistivity values were recorded before and after cryogenic treatment to determine the variations.

To measure the resistance of a 4 mm diameter, 50 mm long heat-treated and cryo-treated aluminium wire using a multimeter, a detailed and precise procedure must be followed. The following is an expanded explanation of the steps involved, key considerations, and potential challenges specific to aluminium and heat-treated/cryo-treated materials. To measure the electrical resistivity of the treated and untreated aluminium samples, a multimeter was employed to record the resistance values before and after the cryogenic treatment. This method facilitates a direct comparison of the electrical properties of the material and aids in quantifying the effects of the cryogenic process. When measuring a 4 mm diameter, 50 mm long aluminium wire (as shown in fig. 2), several factors must be considered to ensure accurate results. The wire dimensions play a crucial role in calculating resistivity from the measured resistance, as resistivity is an intrinsic property of the material independent of sample size.

The procedure involved carefully positioning the multimeter probes at precise points on the wire to measure resistance over a known length. It is essential to maintain consistent contact pressure and positioning to minimise measurement errors. Additionally, the temperature of the wire during measurement should be controlled and recorded, as the resistivity of aluminium is temperature-dependent. Potential challenges specific to aluminium include its high thermal conductivity, which can lead to rapid temperature changes, and its tendency to form an oxide layer on the surface, which may affect the electrical contact between the probes and the wire. To mitigate these issues, multiple measurements may be taken and averaged, and the wire surface may need to be cleaned or treated to ensure good electrical contact.

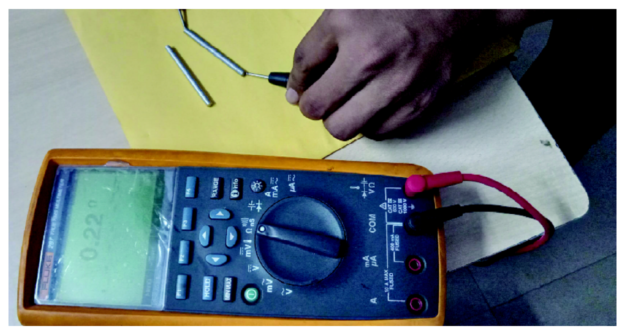


Fig. 4: Testing With Millimeter

Methodology for Measuring Resistance Using a Digital Multimeter

To measure the resistance of an aluminium wire, a digital multimeter is used. The multimeter should be set to the resistance (Ω) mode by turning the rotary dial to the appropriate setting. The resistance

Table 2: Before Cryogenic Treatment

Specimens	Length	Diameter	Cross-section area	resistance	resistivity
1	56.08 mm	4.023 mm	12.71 mm^2	0.45	0.102 Ω.mm
2	56.6mm	4.22 mm	13.99 mm^2	0.47	0.116 Ω.mm

Table 3: After Cryogenic Treatment

Specimens	Length	Diameter	Cross-section area	resistance	resistivity
1	56.08 mm	4.023 mm	12.71 mm^2	0.22	0.04986 ΩΩ.mm
2	56.6 mm	4.22 mm	13.99 mm^2	0.23	0.05213 ΩΩ.mm

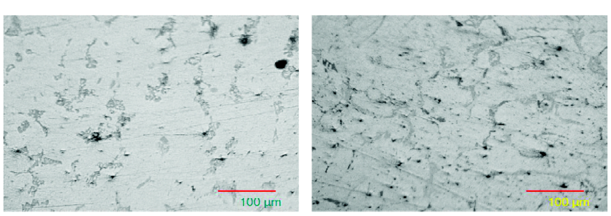
measurement mode is typically denoted by the Ω symbol. If the multimeter is auto-ranging, it will automatically select the appropriate resistance range. Otherwise, it is recommended to manually set the range to the lowest available setting (e.g., 200Ω or 2kΩ) to ensure accurate measurement of low-resistance materials such as aluminium.

Since aluminium is highly conductive, the resistance of a short wire segment (50mm in length, 4mm in diameter) is expected to be in the milliohm (mΩ) to ohm range. If the initial reading is too low or unstable, adjusting the range accordingly can provide better resolution.

Before taking measurements, the aluminium wire should be cleaned to remove any oxidation or contamination that may interfere with accurate readings. This can be done using fine-grit sandpaper or a wire brush to clean the ends of the wire where the probes will be placed. Ensuring that the wire is straight and free from mechanical stress is also important, as these factors can alter the resistance.

For accurate measurement, the two multimeter probes should be placed firmly at the ends of the aluminium wire. Maintaining stable and secure contact with the wire minimizes contact resistance. Since aluminium tends to form an oxide layer, good electrical contact should be ensured by cleaning the probe contact points with a fine abrasive pad if necessary.

Once the probes are in place, the resistance value displayed on the multimeter screen should be observed. The measured resistance will be in ohms (Ω). Due to aluminium's high conductivity, the resistance of a 50mm, 4mm diameter wire is expected to be very low. If required, a four-wire (Kelvin) measurement method can be used to minimize the effects of lead and contact resistance, improving measurement accuracy for very low resistance values.



a) Without DCT b) DCT

Fig. 5: Optical Microscopy of Without DCT after DCT

Table 4: The Grain Size in Microns

	Without DCT	With DCT
Avarage Particle Size	26 µm	18 µm
Standard Deviation	approx.19.2 µm	approx.8.5 µm

To ensure accurate results, several considerations must be taken into account. Contact resistance should be minimized by ensuring the probes have firm and clean contact with the wire. Temperature variations can affect resistance, so measurements should be conducted in a controlled temperature environment. Additionally, any variations in wire dimensions can impact resistance readings, so sample preparation should be uniform.

Following this methodology ensures precise and repeatable resistance measurements of aluminium wire, which is essential for analyzing its electrical properties, particularly in relation to cryogenic treatments.

The formula to calculate electrical resistivity (ρ) is:

$$\rho = R\left(\frac{A}{L}\right)$$

Where,

ρ: Electrical resistivity (Ω·mm)

R: Electrical resistance of the material (Ω)

A: Cross-sectional area of the material (mm^2)

L: Length of the material (mm)

To examine the cryo- effect on the resistivity two samples were tested having dimensions as given below

RESULTS AND DISCUSSION

Microstructural Changes

Fig. 5 presents a comparative analysis of optical microscopy images of the recycled aluminium alloy before and after Deep Cryogenic Treatment (DCT), captured at a magnification indicated by the 100/µm scale bar. The untreated sample ("Without DCT") exhibits a coarser and more heterogeneous grain structure with visible inclusions and microstructural defects, typical of recycled aluminium. In contrast, the DCT-treated sample shows a significantly more refined and uniform microstructure, with sharper grain boundaries and reduced visible defects. These visual observations are quantitatively supported by the data in Table 5, where the average grain size decreased from 26/ µm to 18/ µm and the standard deviation dropped from approximately 19.2/ µm to 8.5/ µm after DCT. The reduction in both grain size and variability indicates effective grain refinement and microstructural homogenization induced by DCT. These changes can be attributed to the cryogenic-induced mechanisms such as thermal contraction, dislocation rearrangement, impurity redistribution, and stress relief, confirming the potential of DCT to significantly enhance the microstructural quality and consistency of recycled aluminium alloys.

In the "Without DCT" image (Fig. 5a), numerous dark spots and lines are observed, which likely correspond to impurities, precipitates, or surface defects present in the material prior to treatment. These features appear to be randomly distributed across the surface, and their low contrast against the background makes it challenging to discern finer details. The presence of such inclusions or defects suggests inherent microstructural heterogeneity, which may influence the material's mechanical and functional properties.

In contrast, the "DCT" image (Fig. 5b) exhibits a noticeable reduction in the size and density of dark spots and lines. This suggests that the DCT process has facilitated the refinement or dissolution of inclusions and defects, leading to a more homogeneous microstructure. The overall uniformity of the treated surface is significantly improved, and the enhanced contrast allows for a clearer visualization of the remaining features. These microstructural changes indicate that DCT has induced transformations within the material, potentially through mechanisms such as phase redistribution, stress relaxation, or impurity dissolution.

The observed refinement of the microstructure suggests that DCT may contribute to enhanced material properties. The reduction in defects and inclusions can lead to improved mechanical characteristics, such as increased hardness, wear resistance, and fatigue life. Additionally, the potential for stress relief due to cryogenic treatment may further enhance the performance of the material in demanding applications.

However, further analysis is required to establish a more comprehensive understanding of these microstructural modifications. The specific material composition, along with the detailed parameters of the DCT process (e.g., temperature and duration), plays a crucial role in determining the extent of these changes. Higher magnification imaging techniques, such as Scanning Electron Microscopy (SEM) or Transmission Electron Microscopy (TEM), could provide a more detailed insight into the microstructural transformations occurring at a finer scale. Additionally, a quantitative assessment of feature size, density, and distribution would offer an objective evaluation of the impact of DCT on the material's microstructure.

The comparative optical microscopy analysis suggests that DCT has a significant influence on the microstructure of the material, promoting the refinement or dissolution of inclusions and defects. These changes are likely to contribute to improved material properties, although further investigation is necessary to confirm the underlying mechanisms and optimize the treatment parameters for specific applications.

Electrical Resistivity Analysis

The results showed a significant increase in electrical resistivity in cryogenically treated samples compared to untreated ones. The increase in resistivity can be attributed to reduced dislocation density and alterations in the electron scattering mechanism due to microstructural modifications.

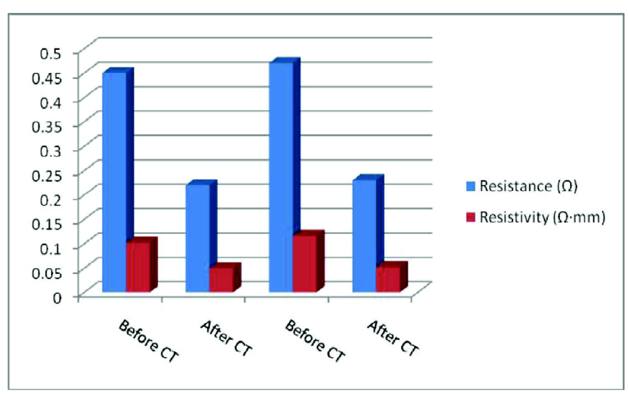


Fig. 6: Bar Chat of Resistance and Resistivity

The bar graph provides a visual comparison of the resistance (Ω) and resistivity (Ω·mm²/mm) of samples before and after cryogenic treatment (CT). The blue bars, representing resistance, show a clear decrease after CT, indicating that the samples exhibit lower opposition to electrical current flow. Similarly, the red bars, representing resistivity, also demonstrate a reduction after CT, signifying a decrease in the material's inherent resistance to current flow per unit length and cross-sectional area. This reduction in both resistance and resistivity suggests that cryogenic treatment enhances the electrical conductivity of the material. The noticeable decrease in resistance implies that the material experiences improved electron mobility, leading to increased electrical conductivity. These findings highlight the potential of cryogenic treatment in optimizing the electrical properties of the material, which could be beneficial for various engineering applications requiring enhanced conductivity.

CONCLUSION

This study investigated the effects of Deep Cryogenic Treatment (DCT) on the microstructural and electrical properties of an aluminium alloy fabricated from scrap material. The experimental analysis included compositional evaluation, microstructural characterization using optical microscopy, and electrical resistivity measurements. The microstructural observations revealed significant changes after DCT, with a reduction in the size and density of inclusions and defects. The treated samples exhibited a more uniform and refined microstructure, suggesting improved material homogeneity. These changes can be attributed to phase redistribution, impurity dissolution, and stress relaxation induced by cryogenic treatment. Such modifications have the potential to enhance mechanical properties, including hardness, wear resistance, and fatigue life.

The electrical resistivity analysis showed a considerable decrease in resistance and resistivity after DCT, indicating enhanced electrical conductivity. This improvement is likely due to reduced dislocation density and modifications in the electron scattering mechanism, leading to better electron mobility. The results demonstrate that cryogenic treatment can effectively optimize the electrical performance of aluminium alloys, making them more suitable for applications requiring superior conductivity.

Overall, this study highlights the potential of DCT in refining microstructural features and enhancing the electrical properties of aluminium alloys derived from scrap materials. Future studies should focus on advanced characterization techniques, such as Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM), to further elucidate the underlying mechanisms of microstructural transformations. Additionally, exploring different cryogenic treatment parameters could help optimize the process for specific industrial applications.

REFERENCES

Asl, K. M., Tari, A. and Khomamizadeh, F. (2009). Effect of deep cryogenic treatment on microstructure, creep and wear behaviors of AZ91 magnesium alloy. *Materials Science and Engineering: A*, *523*(1–2): 27–31.

Barylski, A., Dercz, G., Matuła, I., Mazur, I., Aniołek, K. and Rak, J. (2023). The Effect of Changes in the Aging Temperature Combined with Deep Cryogenic Treatment on the Structure, Phase Composition, and Micromechanical Properties of the WE43 Magnesium Alloy. *Materials*, 16(23):7447.

Çakir, F. H. and Çelik, O. N. (2020). Influence of Cryogenic Treatment on Microstructure and Mechanical Properties of Ti6Al4V Alloy. *Journal of Materials Engineering and Performance*, 29(10): 6974–6984.

Eivani, A. R., Duszczyk, J., Ahmed, H. and Zhou, J. (2009). Correlation between Electrical Resistivity, Particle Dissolution, Precipitation of Dispersoids, and Recrystallization Behavior of AA7020 Aluminium Alloy. *Metallurgical and Materials Transactions A*, 40(10): 2435–2446.

Franco Steier, V., Reißig, L., Ashiuchi, E. S. and Araújo, J. A. (2016). Effect of a Deep Cryogenic Treatment on Wear and

Microstructure of a 6101 Aluminium Alloy. *Advances in Materials Science and Engineering*, 2016: 1–12.

Gogte, C. L., Likhite, A., Peshwe, D., Bhokarikar, A. and Shetty, R. (2014). Effect of Cryogenic Processing on Surface Roughness of Age Hardenable AA6061 Alloy. *Materials and Manufacturing Processes*, 29(6): 710–714.

Jovičević-Klug, P., Jovičević-Klug, M., Feizpour, D., Podgornik, B. and Sever, T. (2021). Impact of steel type, composition and heat treatment parameters on effectiveness of deep cryogenic treatment. *Journal of Materials Research and Technology*, 14: 1007–1020.

Kalsi, N. S., Sehgal, R. and Sharma, V. S. (2010). Cryogenic Treatment of Tool Materials: A Review. *Materials and Manufacturing Processes*, 25(10):1077–1100.

Kang, H.-H., Han, S.-C., Ji, M.-K., Lee, J.-R. and Jun, T.-S. (2024). Achieving synergistic improvement of wear and mechanical properties in Ti–6Al–4V alloy by multiple cryogenic treatment. *Journal of Materials Research and Technology*, 29: 5118–5125.

Li, B., Jin, X., Li, C. and Wang, Y. (2018). Effect of Cryogenic Treatment on Microstructure and Wear Resistance of Carburized 20CrNi2MoV Steel. *Metals*, 8(10): 808.

Liu, K., Wen, J., Fu, K., Shen, Z., Li, M., Zhang, Y., Shi, J., Jin, Y. and Huang, Z. (2022). Impact of cryogenic treatment on the microstructure and properties of a thermo-mechanically processed Cu-11Fe composite. *Journal of Physics: Conference Series*, 2342(1): 012007.

Wei, L., Zheng, G., Li, H., Song, R., Ye, F., Wang, D., Xie, D. and Wu, S. (2018). Effects of Cryogenic Treatment on the Microstructure and Residual Stress of 7075 Aluminium Alloy. *Metals*, 8(4): 273.

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