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CONTENTS

Experimental study on the enhancement of fabricated 6101 Aluminium alloy through Cryogenic treatment

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ABSTRACT: The Cryogenic treatment is an old technique for material enhancement; however, it has become a popular method. It is an optional and one-time permanent treatment process that involves exposing materials to extremely low temperatures at -196 °C and has piqued interest because of its potential to greatly improve mechanical properties and is a well-established technique for material enhancement; however, its recent popularity stems from its potential to significantly improve mechanical properties. This study investigated the effect of cryogenic treatment on aluminium alloy 6101, focusing on enhancing its mechanical and microstructural properties for potential structural applications. The fabricated samples were cooled to approximately -196 °C and then spontaneously heated to ambient temperature. Compression tests were conducted on both the treated and untreated samples to evaluate the changes in yield strength, ultimate compressive strength and ductility. Optical microscopy was employed to analyse the grain structure, dislocation density and precipitate distribution. The results demonstrated that the cryogenic treatment led to a notable increase in the compressive strength, although the ductility decreased. The microstructural analysis revealed a finer grain structure, which contributed to the improved load-bearing capacity of the material. These findings highlight the potential of cryogenically treated aluminium alloy 6101 for applications that require superior compressive strength and structural integrity.

Key words: Cryogenic treatment, grain structure, material enhancement, mechanical properties, microstructural analysis, optical microscopy.

Aluminium alloy 6101 belongs to the 6000 series. This series includes aluminium alloys known for their high strength, good resistance to corrosion and good machinability. Typically, these alloys contain Mg and Si as their main elements. They form magnesium silicon, which allows heat treatment and improves mechanical properties (Ikumapayi *et al.,* 2021).

This alloy is primarily utilized as feedstock for AAAC-type aluminium conductors and extrusions, particularly for compression joints and connectors in the electrical field (Karabay *et al.*, 2005). AA6101 scrap can be effectively recycled and repurposed for architectural applications, capitalizing on the widespread use of 6XXX series aluminium alloys, including AA6101, in engineering and architectural fields owing to their desirable mechanical properties (Silva *et al.,* 2004). These alloys typically undergo thermal treatments such as solution heat treatment and artificial aging to achieve the desired microstructural state and mechanical characteristics. The recycling of aluminium scrap, including AA6101, has gained significant importance for both economic and ecological reasons. Utilizing scrap as a raw material reduces energy consumption and total costs, while minimizing environmental impacts (Silva *et al.,* 2004). A study on AA6060 alloy, which exhibits similarities to AA6101, revealed that a higher proportion of scrap material can be incorporated during production without compromising the mechanical properties compared with conventional compositions. However, recycling and manufacturing processes for AA6101 present challenges, including variations in tensile strength and hardness along extrusions, decreased tensile strength and elongation in wires post-T6 treatment, wire breakage during repeated drawing and stranding and reduced tensile strength and hardness in extrusions used for electrical connectors (Karabay *et al.*, 2005). Despite these challenges, the recycling process conserves resources and maintains the desired properties of the alloy, making it a sustainable and eco-friendly option for architectural applications, aligning with the increasing focus on green materials for construction and design.

Aluminium alloy 6101 mainly has aluminium (Al), but it also includes magnesium (Mg) and silicon (Si) as key alloying elements. The typical chemical composition of this alloy is listed in Table 1.

Table 1: Composition of AA 6101 (wt. %).

Elements	%weight
Aluminium (Al)	97.8%-99.4%
Magnesium (Mg)	0.35% - 0.8%
Silicon (Si)	0.3% - 0.7%
Copper (Cu)	$\leq$ 0.10%
Iron (Fe)	$\leq$ 0.50%

Other elements, such as manganese (Mn), chromium (Cr) and zinc (Zn), also appear in smaller quantities (Aluminium Association).

For aluminium alloy 6101, the recrystallisation temperature is typically between 400 °C and 450 °C (Ikumapayi *et al.,* 2021). The alloy experienced major microstructural changes in this temperature range, which improved its ductility and decreased its hardness. Depending on the particular processing conditions and intended qualities of the material, the precise temperature may change. During compressive testing, the microstructure was modified as described below.

a) Grain distortion: During the compressive force, the grains of the aluminium alloy expand or deform. This is an illustration of plastic deformation, in which granules undergo shape changes without shattering. The metal can accept deformation because the grains slip and tangle under the compressive stress.

b) The dislocation density increases: the grains multiply and increase the dislocation density within them.

Work hardening is the outcome of dislocations accumulating at the grain boundaries owing to compression. This can render a material more brittle and increase its strength and hardness (Rodrigues *et al.,* 2020).

Cryogenic Treatment

Cryogenic treatment is a metallurgical process. It uses liquid nitrogen or other comparable fluids to chill samples at low temperatures, typically around -196°C (77 K). This approach is mostly used for metals with FCC and HCF lattice structures (Dash and Chen, 2023). The aim was to make them tougher and stronger. Because of the low temperature, the microstructure of the material changed. These adjustments can result in improved performance, a longer life and higher dependability.

Cryogenic treatment was done to an Al alloy that had already been heat treated in the following manner:

Cool slowly without thermal shock to approximately -196°C.

Store at approximately -300°F (-184°C) for 24 hours.

Return to ambient temperature gradually, without thermal shock.

To slow down atomic and molecular activity, test temperatures were decreased from ambient to -196°C with liquid nitrogen (LN$_2$) (Baldissera and Delprete, 2008; Desai *et al.*, 2016; Sonia, 2020; Singh and Pandey, 2022). Slow temperature fluctuations result in equivalent thermal compression and expansion from the core to the surface, releasing residual tensions and stabilizing the alloy uniformly. Maintaining homeostasis during temperature cycling may take up to 48 hours or longer (Callister and Rethwisch, 2020).

MATERIALS AND METHODS

The material used for the experiment was aluminium alloy 6101 sourced from Hindalco Industries

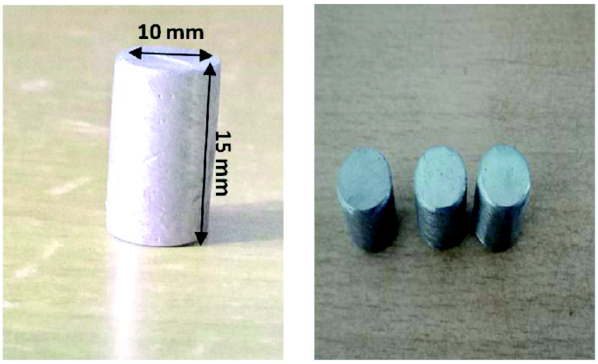

Fig. 1: Compressive Test Specimens

Limited, which is a metal flagship of the Aditya Birla Group. Because it was initially supplied in the form of a plate, it was cast-processed to meet the ASTM E9 standard applicable for compressive testing. The final specimen had a diameter of 10 mm and length of 15 mm, as shown in Figure 1.

The samples were split into two batches, to which cryogenic treatment was applied to the former and the latter was only a cast. Each batch contained three samples and the average of each group was used for comparative analysis. After testing in the UTM, the samples were ground and polished to analyse their grain structure using Optical Microscopy.

Cryogenic Treatment Procedure

The samples underwent Deep Cryogenic Treatment (DCT) by cooling them to -196°C using liquid nitrogen and placing them in a Dewar flask for 24 h (Figure 2). Following the treatment, the samples were gradually warmed to room temperature to minimize the thermal stress, thereby preventing potential cracking or structural issues. This treatment refines the microstructure of the materials, enhances their mechanical properties and improves the overall performance (Karki *et al.*, 2024). Cryogenic treatment is a supplementary process to conventional heat treatment, which exposes materials to extremely low temperatures, usually approximately " 196°C, for a particular period (Razavykia *et al.,* 2019; Sonar *et al.,* 2024).

The process is usually divided into the following stages:

1. Quenching: The product in the initial stage of the overall heat treatment process is quenching.
2. Cryogenic cooling: Finally, the material is cooled at cryogenic temperatures usually by liquid nitrogen to about -196°C.
3. Imbibitions: This is permitted at cryogenic temperature for an established number of hours typically 24 hours (Sonar *et al.,* 2024).
4. Tempering: The material then undergoes tempering, which relieves the internal stresses and removes more impurities from the microstructure. Interestingly, the sequence of cryogenic treatment with respect to tempering influences material properties. A few studies have argued that cryogenic treatment prior to tempering enhances wear resistance and tool life (Vimal *et al.,* 2008; Yan *et al.*, 2012). On the other hand, cryogenic treatment after tempering has been reported to enhance fracture toughness for certain steels (Sola *et al.,* 2017).

Fig. 2: Cryo-Treated Specimens

Compressive Testing

Compressive tests were conducted using a Servo Universal Testing Machine in the Farm Machinery Department (GPBUAT) with a load cell capacity of 200 kN as shown in figure 3.

Aluminium components are used in many applications, such as columns, beams, or panels, to withstand compressive loads. The ability of aluminium to resist these forces without buckling or deformation was determined through testing. The Compressive strength is a crucial element to consider when designing aluminium structures that will bear the expected loads with safety in mind, thus avoiding structural failures and ensuring conformity to safety standards. Samples were then tested at a constant strain rate of 0.5 mm/min until failure. Stress-strain curves were generated for both treated and untreated

Fig. 3: Servo Universal Testing Machine

samples.

Preparation for Microstructural Analysis

For microstructure analysis, the compressed specimen is grind using finer abrasive sheets to eliminate surface imperfections and scratches. The materials used are shown in figure 4.

- After grinding, samples are polished using diamond suspensions of decreasing particle size to obtain a mirror-like quality.
- The polished samples are etched with Keller's reagent (2 mL HF, 3 mL HCl, 5 mL HNO_3 and 190 mL H_2O) to reveal the grain boundaries and microstructure through chemical interaction, creating contrast between phases or grains.

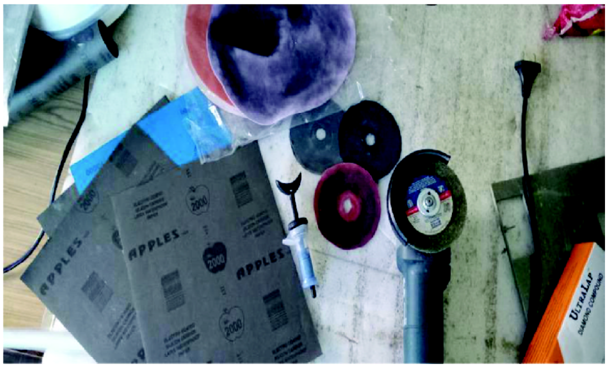
Fig. 4: Surface Cleaning Equipments

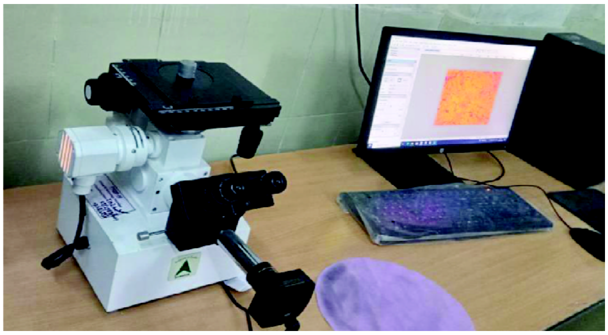
Fig. 5: Optical Microscope Setup

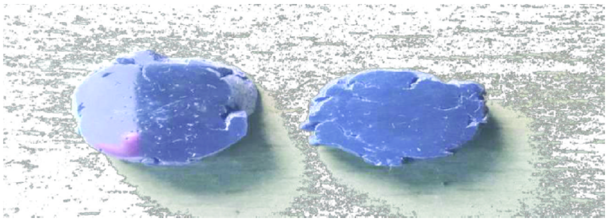
Fig. 6: Prepared Samples for Microscopic Analysis

The prepared samples were examined using an optical microscope (Figure 5).

Images of the microstructure were captured for both cryogenically treated and untreated specimens, shown in figure 6.

Optical microscopy was performed using an optical microscope. This technique uses a magnifying lens to visually examine the microstructure of materials. It is especially useful for examining the grain structure, phase distribution and any defects or inclusions in metals such as aluminium alloys. This approach uses visible light to illuminate the sample, with subsequent magnification being affected by the arrangement of the lenses. The aim was to observe the transformations in the grain structure before and after the cryo-treatment.

RESULTS AND DISCUSSION

An average of 3 samples was used for each observation. The cryogenically treated samples demonstrated a significant increase in compressive strength compared to their untreated counterparts. Analysis of the stress-strain curves revealed improvements in both yield strength and ultimate compressive strength for the treated samples. The

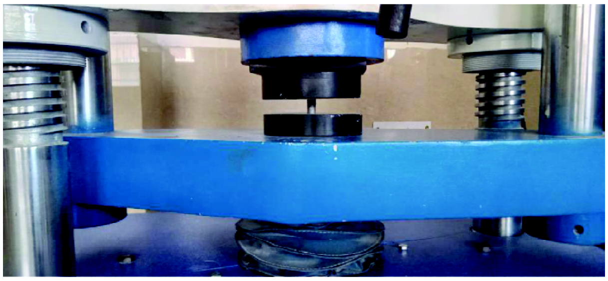
Fig. 7. (a) Compression Test

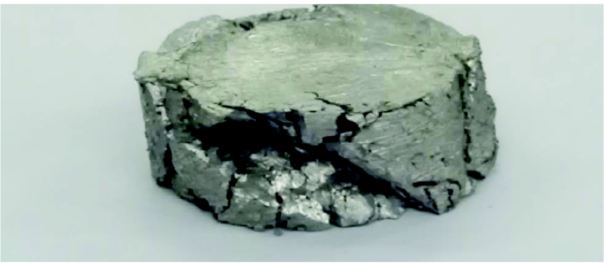
Fig. 7. (b) Compression Test

treated and untreated specimens were compressed in the UTM, as shown in figure 7 (a) and (b), respectively.

To enhance this description further, we consider the following:

1. Specific numerical values or percentage increases for the observed improvements in compressive strength, yield strength and ultimate compressive strength.
2. Details of cryogenic treatment process, such as temperature and duration.
3. Information about the material composition of the samples.
4. Statistical analyses were performed to validate the significance of observed differences.

Table 2: Comparison Data

Samples	Yield strength	Ultimate strength	strain
Untreated	235 MPa	460 MPa	0.258
Treated	404 MPa	511 MPa	0.17

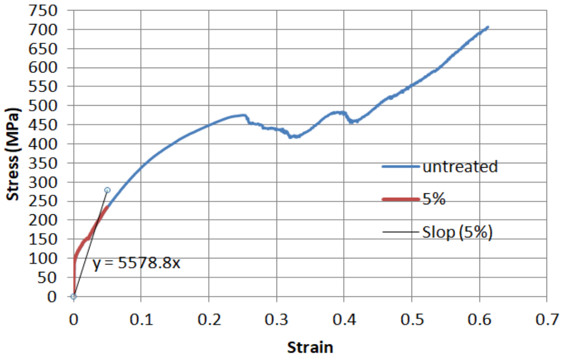

Fig. 8. (a) Compresition Graph of Untreated Specimens

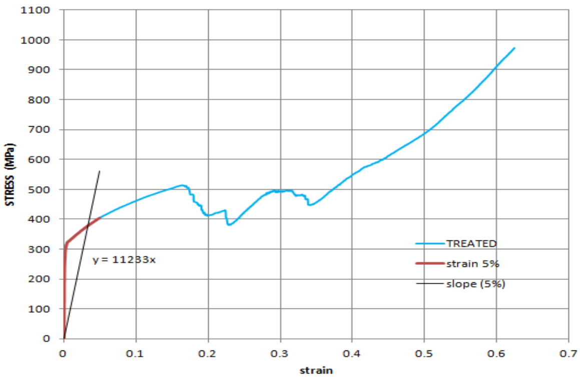

Fig. 8. (b) Compression Graph of Treated Specimens

5. Potential explanations for the improved mechanical properties resulting from cryogenic treatment.

These additions would provide a more comprehensive and scientifically rigorous account of the experimental findings, as shown in figure 8 (a) and (b).

The specimen broke at a point in both types of specimens owing to its brittleness.

The increased yield and ultimate strength of the cryogenically treated specimens can be correlated with the microstructural changes induced by deep cryogenic treatment. Such deep freezing at -196°C leads to a higher transformation of retained austenite into martensite, resulting in an increased dislocation density, which is harder and stronger. This treatment causes grain refinement, which tends to decrease the internal stress and increase mechanical strength. Nevertheless, this causes decreased ductility and the strain values are lower because of the increased brittleness and loss of the ability of the material to deform plastically.

Microstructure Observations

Microscopic examination revealed a finer grain

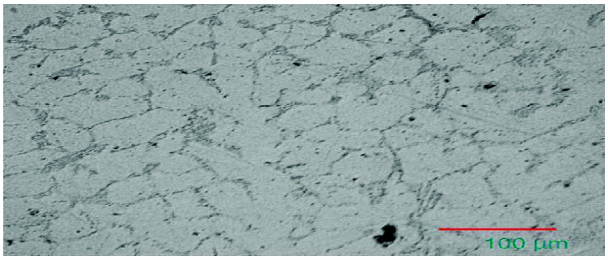

Fig. 9: (a) Cryogenic Treated Sample

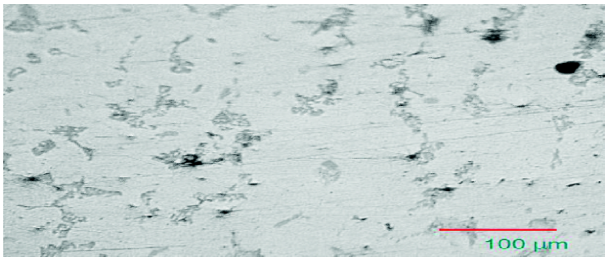

Fig. 9: (b) Cryogenic Untreated Sample

structure and reduced porosity in cryogenically treated samples. This is shown in figure 9 (a) and (b).

Although well-aligned grains were evident in the treated sample, broken and ill-aligned grain boundaries were observed in the untreated sample. The phase transformations present in this cryogenic treatment correlated well with the observed improvements in mechanical properties. The cryogenic treatment refined the grain size, with the average grain size in the treated samples being 15 µm compared with the untreated samples, as presented in Table 3. Therefore, a reduction in residual stress increases the mechanical strength and hardness of the material. The efficiency of the material was confirmed through microstructural analysis, which was directly correlated with grain refinement arising from the structure

As shown in Table 3, particle sizes were obtained using an optical microscopy software generator. Thus, the cryogenically treated samples significantly reduced the average grain size of the particles from 24 µm in the as-received sample to 15 µm in the treated sample, a reduction of approximately 37.5%. This reduction in grain size has implications for the improvement of the mechanical strength and hardness of the material through the minimization of defects at the grain boundaries and residual stresses.

The finer grain structure observed in the treated sample caused an increase in dislocation movement. This, in turn, leads to increased resistance to plastic deformation. A larger grain boundary area was present in the refined grains, which limited their motion as a dislocation carrying barrier. This leads to an enhanced mechanical performance, as reported for the treated sample. Again, this microstructural refinement resulted in comparable improvements in the yield strength and ultimate strength, as discussed earlier. The fine-grained structure is a direct result of the phase transformations induced by cryogenic treatment and the overall mechanical behaviour of the material is enhanced.

Table 3: Grain Boundary Size

conditions	Average Particle Size
Treated	15 µm
Untreated	24 µm

The cryogenic treatment effectively reduced the residual stresses and refined the microstructure, which mainly accounted for the improved compressive strength achieved in the aluminium alloy 6101 samples. As shown in Table 2, the yield strength in the cryogenically treated samples increased by an exceptional 71.9%, which clearly indicates an excellent improvement in the ability of the material to resist permanent deformation due to the applied loads. Finally, the ultimate compressive strength increased by 11%

However, such a significant strength improvement occurs at the cost of a noticeable reduction in strain, as it decreases by 51% at the point of ultimate compressive strength. This reduction in strain was due to the phase transformation caused by the cryogenic treatment, which caused a change from a more ductile to a more brittle material. However, the improved tensile strength in the critical tensile direction is beneficial to the bearing capacities and the consequent loss in ductility suggests that this material might fracture more easily in brittle mode under appropriate circumstances.

Figure 10 shows the stress-strain relationship variation between cryogenically treated and untreated samples. It is visually appreciated to distinguish the mechanical behaviour. From the graph, it can be concluded that there is a gain in the

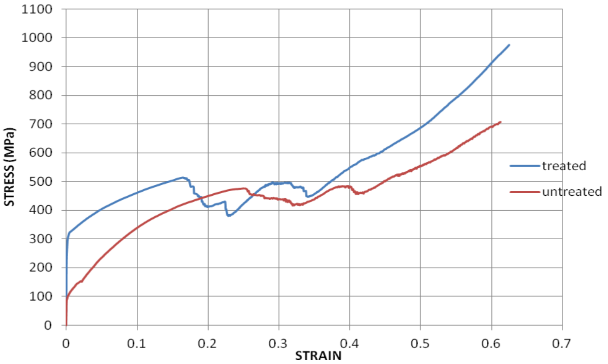

Fig. 10: Stress-Strain Variation between Cryogenically Treated and Untreated Samples

yield strength and ultimate strength by treatment at the expense of a steeper slope in the stress-strain curve, reflecting the loss of ductility.

Further analysis of the grain boundaries showed notable changes due to the cryogenic treatment. For example, there was a reduction of approximately 37.5% in the grain boundary size of the treated samples. The microstructure should be refined because smaller grains increase the strength of the material via the Hall-Petch relationship, which further indicates that finer grains may hinder the movement of dislocations more than larger grains. With an increase in the grain boundary area, there were more obstacles to the motion of dislocations and thus an increase in the strength and hardness of the aluminium alloy.

CONCLUSION

The Cryogenic treatment of aluminium alloys, in this case scrap architectural aluminium alloys, has been shown to improve their mechanical properties, specifically their compressive strength, yield strength and ultimate compressive strength. The treated specimens exhibited a yield strength of 404 MPa and ultimate strength of 511 MPa. It was found to be significantly better than that of the untreated specimens, which yielded 235 and 460 MPa, respectively. Microstructure changes due to deep cryogenic treatment, such as the refinement of grains and further transformation of retained austenite into martensite, increase the density of dislocations and strengthen the materials; therefore, this increased strength compromises some amount of ductility, such that their reduced strains are seen in the cases with heat treatment. The brittle nature of the treated specimens is a critical consideration for applications where plastic deformation is important.

Microscopic analysis further confirmed these improvements, with a finer grain structure and reduced porosity in the treated samples. The average grain size decreased by approximately 37.5% from 24 µm in the untreated samples to 15 µm in the treated samples. This grain refinement increases the mechanical strength and hardness by reducing the defects along the grain boundaries and lowering the residual stresses. Moreover, in refined structures, a larger area of grain boundaries acts as a barrier to movement for dislocation, which resists further deformation in the plastic range, contributing to the mechanical properties.

While the mechanical properties of aluminium alloys significantly improve with cryogenic treatment, their ductility is reduced; therefore, its value must be considered in application-specific environments. The choice of cryogenically treated materials will balance the strength and hardness gained against the possibility of brittleness, depending on the functional requirements of the end product. In general, cryogenic treatment appears to be a valuable process for enhancing the structural integrity and performance of aluminium alloys, especially in demanding applications where strength and durability are crucial.

FUTURE SCOPE

Further studies should focus on optimizing the cryogenic treatment settings and investigating the long-term impact of treatment on the performance of the alloy in diverse applications. Long-term performance evaluation of cryogenically treated alloys under diverse environmental conditions such as high humidity, extreme temperatures and corrosive environments is crucial for assessing their reliability and durability. Studies focusing on the microstructural evolution of alloys over time, particularly under cyclic loading or thermal cycling, can provide valuable information regarding their stability and lifespan in real-world applications.

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