A Technical Review on Deep Cryogenic Treatment of Aluminum Alloys: Microstructural and Performance Enhancements

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Abstract - *Aluminum alloys are essential in industries like aerospace, automotive, and manufacturing due to their lightweight and excellent performance. With the growing demand for enhanced properties in these alloys, choosing effective treatment methods has become crucial. Deep cryogenic treatment (DCT) offers a green, low-cost, and efficient solution to improve the microstructure and performance of aluminum alloys without altering their dimensions. This review explores how DCT influences the structural and performance evolution of these alloys. When parameters are carefully optimized, DCT can refine grain structures, increase dislocation density, and optimize secondary phase distribution, resulting in improved mechanical properties, reduced residual stress, enhanced corrosion and wear resistance, and better electrical conductivity. By summarizing key research findings, this paper also provides recommendations for future studies on DCT-treated aluminum alloys.*

Key Words: **Aluminum alloys, Aerospace industry, Lightweight, Green technology, Structural evolution, Grain refinement, Dislocation density**

1. INTRODUCTION

Cryogenic treatment modifies the microstructure of metallic materials at ultra-low temperatures to elevate property levels. Two types are seen: DCT and SCT (1). DCT is the most widely studied form, usually an auxiliary process to conventional heat treatments. It is attractive because of its low cost, simple energy efficiency, and friendliness toward the environment (2). DCT is widely used in the aerospace, automobile, and manufacturing industries. It usually operates within a temperature range of 143K and 77K (3). It commonly uses liquid nitrogen as a refrigerant, due to its safety, availability, and relatively economic factor (4). The process occurs in three stages: cooling, holding, and warming (5). In some processes, DCT is used along with the conventional heat treatment to enhance performance. Among the critical process parameters influencing the efficiency of the process are the soaking temperature, holding time, cooling/warming rates, and the treatment sequence (6).

The significant part of work established was on the DCT's effects on ferrous metals, focusing on transforming austenite to martensite (7) and secondary carbides precipitation for strengthening the steel alloy (8). Nevertheless, the latest works emphasize the benefits achieved in the non-ferrous metal, such as in aluminum alloys. Aluminum alloys are used extensively in structural applications for aerospace, defense, and many other purposes due to their specific strengths, stiffer rigidity, toughness, low corrosion, and low cost. For instance, Al alloys 2024 and 7050 are of particular interest for aerospace applications (10). Alloy 2519 is used to create armor for combat vehicles, while rocket parts for Falcon 9 are created using alloy 2198. Demand for betterment is increasing owing to higher mechanical performance required in aerospace applications and other fields.

DCT provides several advantages to aluminum alloys in terms of residual stress removal and improvement in dimensional stability without degrading their mechanical properties (13). Some aluminum alloys, such as 2219 and 3102, are used to function at ultra-low temperatures. Therefore, the microstructure of these aluminum alloys should be strengthened at ultra-low temperatures (14). Research on DCT applied to aluminum alloys is highly undertaken in this direction. This review will summarize the DCT influence on the microstructure of aluminum alloys as well as performance improvements obtained through such processing and discuss insights and perspectives for further material post-treatment development..

2. MICROSTRUCTURAL EVOLUTION

Recent research shows that deep cryogenic treatment (DCT) can significantly enhance the overall performance of aluminum alloys, primarily by altering their microstructures. The key microstructural elements affected by DCT include grains, dislocations, and secondary phases.

2.1. Effect of DCT on Grain Size

The effects of DCT on grain size are closely related to the structure of dislocations that develops during the treatment. First, the dynamic recovery of dislocations suppressed, therefore the growth of dislocation density; thus, the DCT causes grain refinement. For instance, Li et al.(17) indicated that the mean grain size of 2024-T351 aluminum alloy reduced from 33.46 μm through RT-LP to 28.30 μm after CLP at -130 ± 2°C. Later on, they found that, in a separate experiment, the grain size was 28.20 μm after CLP but it further reduced to 27.13 μm as DCT for 4 hours before CLP was used (CLP-4) (28).

At the same time, recrystallization sites nucleated during DCT contribute to finer grains after subsequent heat treatment. For example, size of grain after solution heat treating has decreased from 70 μm upon room temperature deformation to 53 μm at cryogenic deformation (29).

In addition, in DCT, sub-grain boundaries often preferentially align along dislocations, thus reducing the internal energy and contributing to finer grains (30). The grain structure of aluminum alloys is, therefore, also strongly influenced by the DCT parameters: temperature and time. Zhou et al. demonstrated that the grain size of 2024-T351 aluminum alloy continued to decrease with increases in DCT time due to lattice contraction caused by the longer time, which promoted dislocation motion that led to further grain refinement (31). As for DCT temperature, Zhang et al. indicated that the mean grain size in the 7055 Al matrix composite reduced from 40 μm at -70°C to 10 μm at -150°C (32). Prangnell et al. report a decreasing grain size for the Al-0.1%Mg alloy with decreasing deformation temperature from 298 K to 213 K and 77 K; it is probably because an increased driving force for grain refinement exists at lower temperatures.

However, DCT leads to grain coarsening in aluminum alloys according to some researchers. For instance, Jiang et al. reported that the grain size of 3102 Al was increased from 0.1 μm to 0.5 μm after DCT while being in O-state. Grain coarsening, at a slight extent, was also seen in the weld nugget zone of 2219 Al in dissimilar friction stir welding; in that, the grain size increased from 2 μm to 2.4 μm after DCT at -196°C for 24 hours (34).

This grain coarsening can be associated with the initial microstructure of aluminium alloys. Both of them reveal high content of fine-grained, subgrained, and heterogeneous grain sizes that would allow migration and coalescence of sub-grain boundaries during DCT. What is more important, these alloys included lesser content of secondary phase particles, which would guarantee less "nailing" effect usually restricting grain and sub-grain boundary movement and favouring grain coarsening.

2.2. Effect of DCT on Grain Orientation

The anisotropic internal stress resulted from the DCT might favor the orientation of the grains with a crystal lattice rotation that has aligned in the favorable direction for slip initiation. For example, Liu et al. found higher intensity for the diffraction peak of the [220] orientation for the DCT-treated composites of the (FeCoNi1.5CrCu) p/Al samples, compared to the corresponding untreated samples (35). Wei et al. had submerged the 7075 aluminum alloy in specific apparatus at temperatures of between 25°C and -196°C for 2 hours, leading to discovering the fact that DCT reduces the intensity of diffraction peaks of the [111], [220] and [311] crystal planes, but increases the intensity of the [200] (36). Zhang et al. observed that the preferred grains orientation was better in dissimilar aluminum alloys undergoing friction stir welding, as they reported that the max of concentration orientation for nugget zone grains of DCT samples was better than for samples without the treatment. (34). the authors thought the much stronger formation of texture was induced because of continuous lattice rotation based on internal stress driven by DCT.

2.3. Effect of DCT on Dislocations

Deep cryogenic treatment (DCT) introduces cold shrinkage and stress in aluminum alloys. Chen et al. suggested that the volume shrinkage rate can reach 1.37% if pure aluminum and its alloys are subjected to cryogenic treatment at 200K (37). Plastic deformation and dislocation movement accompany cold shrinkage when internal stress approaches yield stress. (38). In addition to the surface layer, cryogenic laser peening further increased dislocation density at a subsurface depth of 300 μm in 2024-T351 aluminum alloy compared to RT-LP.

Cabibbo et al. carried DCT processing on 6012 aluminum alloy for successive ECAP processing wherein it was observed that a suppressive effect occurs on the pre-DCT aluminium alloy with respect to dislocation movement and cell recombination during ECAP, showing higher dislocation density than those samples which are not subjected to DCT ECAP processing (40). However, in a few instances, the dislocation density will be decreased by performing DCT. Chatterjee et al. Zhang et al. also report a reduction in dislocation density in the weld nugget zone of 2219 Al in a friction stir weld with ceramic Al. According to the authors, this is attributed to the absorption of some of the dislocations into grain boundaries that reduces the dislocation density (34).

2.4. Effect of DCT on Secondary Phase

Alloys of aluminum contain a matrix and secondary phases, whose relation to mechanical properties is critical. DCT leads to lattice shrinkage resulting in increased supersaturation of the solute atoms, thus enhancing the driving force for precipitation. Simultaneously, high dislocation density acts as nucleation sites to accelerate aging kinetics and the precipitation of secondary phases. DCT proved to stimulate the precipitation of the β'' phase in 6026 aluminum alloys (25) and to stimulate the formation of MgZn2 phase in 7050 aluminum alloy (33). Wang et al. confirmed that DCT brings about more dense precipitates of MgZn2 phase in 7A85 aluminum alloy (37). DCT increases lattice distortion in 7000 series aluminum alloys. As such, Mg precipitates out to relieve the stress. Because Zn has a higher solubility in Al, Zn precipitates out during DCT since the solubility decreases at lower temperatures and thus favors the precipitation of $MgZn_2$. In the 2000 series alloys, DCT increases dislocations. This works in favor for the precipitation of θ' phases (Al_2Cu) but leads to preferential precipitation of the S phase (Al_2CuMg) in Mg in the case of 2024 aluminum alloy.

DCT also decreases the size of coarse secondary-phase particles. For instance, multiaxial forged 7085 Al-Zn-Mg-Cu alloy showed more reduced particles after cryogenic deformation at -180°C as compared to high-temperature deformation (17). The area fraction of large Al_2Cu particles in 2219 aluminum alloy was decreased with lower temperatures (35).

DCT temperature and time significantly impact the secondary phase evolution. Longer DCT time promotes precipitation and dissolution of coarse particles, as seen with the S phases in 2024-T6I4 Al(35) and smaller precipitates in Al-Mg-Si alloy with longer DCT time (34). Lower temperatures also contribute to these mechanisms, as seen in 7075 aluminum alloy, with a decrease in DCT temperature. Effect of DCT on Aluminum Alloy Performance Lower temperatures also contribute to these mechanisms, as seen in 7075 aluminum alloy, where decreased DCT temperature decreased coarse phases and increased fine phase density (36). Major alterations in the performance of aluminum alloys are brought about by deep cryogenic treatment. These encompass mechanical properties, residual stress, corrosion resistance, wear resistance, and electrical conductivity.

3. MECHANICAL PROPERTIES

There are mainly two important mechanisms that explain improvements in mechanical properties after DCT:

a) Grain Boundary Strengthening: DCT increases the refinement of grain size that based on the Hall-Petch relationship. Various studies have reported that DCT increases the hardness, yield strength, and elongation of a number of different aluminum alloys including friction stir welded 7050 and melt inert gas welded 5A06 alloys due to grain refinement (34). Grain refinement in cryorolled Al-Cu alloys has also been demonstrated (31).

b) Precipitation and Dispersion Strengthening: DCT offers the better distribution and higher density of secondary phases. It also brings strength increase in aluminum alloys. For instance, when this DCT was applied on 2024 aluminum alloy at -196°C for 24 hours, fine homogenously dispersed S(S') precipitates were formed. These precipitates gave rise to greater mechanical properties (23). Similar improvements in ultimate tensile strength, yield strength, and elongation were reported for 7A85 aluminum alloy when aged at -196°C compared to other elevated temperatures (28). Additionally, a synergism that improves the dispersion of secondary phases is achieved when low-temperature aging is combined with DCT as in the case of 2024-T351 (29)

3.1. Effect of DCT on Aluminum Alloy Performance

Deep cryogenic treatment (DCT) significantly impacts the performance of aluminum alloys, affecting their mechanical properties of aluminium alloy, residual stress, corrosion resistance, wear resistance, and electrical conductivity.

3.1.1. Mechanical Properties of aluminium alloy

a) Secondary Phase Strengthening: the alloy is strengthened because of the enhanced ability of fine secondary phases to pin dislocations while reducing coarse secondary particles, which enhances the mechanical properties.

b) Dislocation Strengthening: DCT-induced dislocations result in an improvement of the mechanical properties via dislocation strengthening. The increasing dislocation density during DCT leads to higher hardness and yield strength. For example, cryo-ECAP of pure aluminum led to an increase in hardness by 27 % as compared with the case when processed at room temperature due to increasing dislocation density (31). On the contrary, the hardness for cryo-rolled 7075 Al was remarkably higher than that for the room temperature-rolled at 172 Hv as against 152 Hv, due to the increased dislocation density caused by cryo-rolling processing (63). Additionally, a higher dislocation density may lead to the acceleration of precipitation kinetics, thereby making the precipitates more hardened (28).

c) Optimum Cryogenic Time: Some researches indicated that increasing cryogenic time usually leads to improving mechanical properties, however elongated times led to losing the strength. Jin et al. revealed that hardness and tensile strength of 5083 Al-Mg alloys increased up to DCT times, however, afterward decreased with more elongated DCT times due to decomposition of the $Al3Mg_2$ phase and owing to relaxation of stress concentration (29). Similarly, Gogte et al. reported that the hardness of 6061 aluminum alloy increases initially, then decreases with larger values of DCT times, which may result from the precipitation and dissolution of a phase (30).

3.1.2. Residual Stress

The residual stress from plastic deformation and thermal stress during machining can influence the mechanical properties and dimensional stability of aluminum alloys (31). DCT is an effective method for releasing residual stress, often at a lesser cost and time compared to the conventional methods.

As an example, DCT at -196°C for 24 hours reduced residual stress in AlSi10Mg alloy by 72.7%, which equals the results of the traditional heat treatment processes. This can be attributed to the volume shrinkage mismatches that lead to compressive residual stress that offsets part of the original tensile residual stress (23). Zhou et al. found that DCT of 2024-T351 aluminum alloy resulted in the reduction of tensile residual stress from 26 MPa to 10 MPa with slight compressive stress formed (31). Araghchi et al. also reported such trend by reducing the residual stress of 2024 aluminum alloy from 140 MPa to 40 MPa after DCT. According to the authors, this is because a temperature gradient always remains between the surface and core during such treatment (26).

3.1.3 DCT Influence on Corrosion Protection

Aluminium alloys show increased resistance to corrosion after DCT in a different ways.

a) Intergranular and Exfoliation Corrosion: DCT modifies secondary phases thereby weakening intergranular and exfoliation corrosion. The treatment commonly leads to the dissolution or breakdown of precipitates along grain boundaries, severely inhibiting the formation of corrosion micro-batteries which are generally responsible for pitting. For example, Zhang et al. observed that in LC4 aluminum alloy, DCT minimized the dissolution of secondary phase particles, thereby delaying further corrosion (27).

b) Precipitate-Free Zones Enlargement DCT has been shown to increase the width of PFZs with resultant improvements in corrosion resistance in the alloy. Su et al. reported that DCT increased the width of PFZ in the 7075 aluminum alloy coupled with a decrease in the degree of corrosion (38). Ma et al. reported that DCT increased the width of PFZ in the 7075 aluminum alloy. The anti-corrosion resistance was thereby significantly improved (27).

c) Induction of Compressive Stress and Nanoprecipitation: DCT introduces compressive stress and creates minor nanoprecipitation in grain boundaries. This decreases the gradients of concentration and potential difference, thus reducing the occurrence of inter-granular corrosion (31). Cabeza et al. reported in 2017-T4 aluminum alloy, DCT introduced non-coherent nanoparticles at grain boundaries, which improved stress corrosion cracking performance (40).

4. ELECTRICAL CONDUCTIVITY

Much attention in recent research has been paid to the DCT's influence in the preparation of aluminum alloys, since it is well recognized for its capability to improve the mechanical properties without significantly losing conductive properties. This approach stands sharply against standard strengthening techniques, such as alloying and precipitation hardening, as the latter approach would result in an increase in electron scattering by means of dislocation generation, grain boundaries, and larger precipitates, causing this method to lose electrical conductivity. While DCT introduces such minor microstructural changes that do not contribute much to the alteration of electrical conductivity. Research has demonstrated that DCT leads to the development of finer nanoscale precipitation products such as β'', h', MgZn2, and Al3Zr precipitates, which increase strength with relative preservation of high electrical conductivity. Cai et al. investigated CDDT, a variant of DCT, in aluminum wire. They observed an increase in the ultimate tensile strength by more than 150% - from 137 MPa to 190 MPa - while showing only slight degradation in electrical conductivity compared with the wire treated at room temperature. This would suggest that DCT improves a more refined microstructure that facilitates electron flow as smaller precipitates offer pathways for electrons to flow through, whereas larger precipitates or grain boundaries would impede the flow.

In another study, Zhao et al. investigated the cumulative effects of multiple DCT cycles on aluminum alloys. They found that repeated DCT treatments led to further grain refinement and the stabilization of the microstructure, resulting in incremental improvements in both mechanical properties and electrical conductivity (35). Although there were diminishing returns after several cycles, the overall effect showed that repeated cryogenic treatments could be used to fine-tune the balance between strength and conductivity. This approach could be particularly beneficial in applications requiring long-term material stability and performance under mechanical and thermal stress.

These results are supported by other studies which proved that DCT promotes the precipitations of nanoscale precipitates in alloys 7A85 and 6061. Wang et al. (38) found that such precipitates especially β'' and Al_3Zr phases strengthen without significant loss in electrical conductivity, that degrades with an approximately 1%. This is a benign contrast to classical precipitation hardening where the strength and conductivity balance can become a significantly more important issue.

5. CONCLUSIONS

It has been shown that Deep Cryogenic Treatment is an effective, non-destructive method with favorable cost benefits for enhancing properties of aluminum alloys by refining microstructures. Low temperatures in DCT cause lattice shrinkage, stopping atomic oscillations and by sealing the structure of the alloy, increases locked-in energy due to stored deformation, which then restricts atomic and dislocation motions and leads to higher strength and hardness material. Grain refinement is a result of grain growth suppression during rapid cooling, which results in smaller and more uniform grains. The stored deformation energy increases; however, the higher dislocation density acts to strengthen the alloy by preventing subsequent plastic deformation. DCT can improve the secondary phase morphology and distribution in two ways-in precipitation-hardened aluminum alloys, it encourages the formation of finer, uniformly distributed precipitates such as β'', MgZn2, and Al3Zr. This precipitates further improve strength, hardness, and wear properties.

The scope of potential DCT-induced microstructural changes is very vast for industries such as automotive, marine, and aerospace in which strength and durability are equally challenging. Wear resistance improves because of the higher density of dislocations and finer grains that avoid wear-related defects. This leads to resistance from corrosion during secondary phases stabilized and uniformly distributed grain boundaries, and reduces the tendency of localized corrosion in the alloy. On the contrary, DCT-treated aluminum alloys maintain their electrical conductivity or decrease only slightly despite significant improvements in their mechanical properties, which enable them to be applied in place where there is a significant demand for both structure integrity and conductivity, such as aerospace wiring or electronic components.

Yet some aluminum alloys can show undesirable behaviour after DCT, which is grain growth and reduced dislocation density. This commonly occurs in systems where the stored deformation energy exceeds the threshold for substructure movement, thus allowing grain boundary migration either during or after cryogenic treatment. Grain boundary migration leads to a coarsening of the grain structure that makes one lose the acquired benefits by the refinement of grains. The mechanical properties are further degraded. This behavior also has a relation with the strain rate sensitivity of the alloy at the low temperatures wherein such a deformation under stress becomes a function of strain. The grains that have higher strain rate sensitivity are those most prone to grain growth during DCT, causing a weakness in such strength as well as hardness.

In some cases, the strength reduction due to DCT can be caused by the relaxation of the stored energy within the material. Cooling and subsequent reheating allow dislocations to annihilate or reconfiguration into a lower-energy state, eventually resulting in a relaxed microstructure that has fewer obstacles for dislocations to overcome and that reduces the strength of the material.

Although the treatment through DCT may enhance the properties of aluminum alloys, the application should be properly optimized in terms of specific alloy composition and the intended usage. On the other hand, the possibility of grain growth and concomitant reduction in dislocation density underscores the necessity of studying the value of stored deformation energy and strain rate sensitivity of the alloy that would determine the efficiency of treatment. Further work would be conducted to match DCT parameters to alloys and, thus ensure that the improvements in the mechanical properties, wear resistance, and electrical conductivity are not accompanied by grain coarsening or reduced strength.

6. FUTURE SCOPE

Future Research Directions

Even further research is aimed at boosting the comprehension of intrinsic mechanisms that control modifications in microstructure and properties related to DCT treated aluminum alloys. In spite of promising improvements in mechanical strength, wear resistance, as well as electric conductivity among others, the governing processes underlying the improvement are not well understood. It may be able to explore these fundamental mechanisms to provide an opportunity to achieve full potential for DCT and its varied applications.

One area where further work is needed concerns the impact of various initial alloy conditions on microstructure and properties subsequent to DCT. The condition at which the alloy begins cryogenic treatment can significantly influence its response to cryogenic treatment and in turn perhaps might be a factor in how the material responds concerning grain refinement and dislocation density-a property that the alloy possesses to some extent after DCT. Understanding the mechanisms and properties affected by DCT may lead to tailoring the treatments to maximize the desired property levels in different alloys.

Another important area of research is examining the microstructural stability resulting from multiple cyclic cryogenic treatments. DCT typically is accomplished as a single-step treatment; cyclic cryogenic treatments may actually further improve and stabilize the refinement of the microstructure. Repetitive cycling through cryogenic and ambient temperatures may promote more orderly precipitate formation and grain refinement, which could result in alloys that are better at being fatigue-resistant, wear performance, and long-term microstructural stability. However, it is important to establish whether such cyclic treatments result in diminishing returns or have unintended consequences, such as embrittlement or grain growth.

Computational simulations and modeling could also lead to major breakthroughs in the cryogenic treatment field. Simulations may be applied to predict the effect of DCT on the various aluminum alloys' compositions and initial conditions besides optimizing the temperature profile, cooling rates, and holding time. Using computational tools, the optimization of DCT parameters will be taken more rapidly than in the case of long trial-and-error experiments. This could speed up further improvements toward more efficiently enhancing material performance as well as increasing industrial applications.

Conclusion Future work should, therefore, center on some of these areas to further advance the processes of DCT in relation to aluminum alloys. As our comprehension of how DCT influences microstructural and property changes improves, so too will our ability to realize the full potential of this cost-effective and sustainable heat treatment method across all types of industries.

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