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Cryogenic treatment of 7050 aluminum alloy to improve corrosion resistance

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Abstract

Cryogenic treatments represent an innovative technology developed with the aim of improving the performance of metallic alloys. The beneficial effects on steels are well documented in the literature, whereas their influence on other materials, such as aluminum alloys, is still not completely clarified. Even if the scientific literature reports conflicting data and conclusions, the industrial applications of such treatments are constantly growing. In the present experimental work, the mechanical and corrosion properties of a high-performance 7050 aluminum alloy were studied after cryogenic treatment at - 196 °C in liquid nitrogen. Whereas no appreciable variation was observed in the mechanical resistance, the corrosion tests showed a positive effect of the cryogenic treatment. The microstructure was investigated by FEG-SEM analysis, revealing a different distribution of precipitates near the grain boundaries, which was able to reduce the electrochemical potential difference among these regions and the center-grain.

Keywords: cryogenic treatment, aluminum alloys, mechanical resistance, corrosion resistance, metallography

1. Introduction

Aluminum alloys are one of the most important materials in engineering structures. Among them, the so-called 7xxx series, based on the Al-Zn-Mg (-Cu) system, is characterized by the highest mechanical strength. They are widely used in high-performance structural, aerospace and transportation applications. Together with the chemical composition and the fabrication process, their properties are significantly influenced by the heat-treating procedure, which involve three basic steps: high-temperature solubilization, water quenching and natural or artificial aging. In the precipitation response, the peak hardening is obtained at precise time-temperature conditions [1-5]. Depending on the chemical composition, in standard heat treatments [1-2], the precipitation sequence can be very complicated and can involve many stable and/or metastable compounds [1-2].

In recent years, a further and innovative possibility to improve the service performance of metallic alloys has been identified in the cryogenic treatment (CT). It consists of a gradual cooling of the material until the defined temperature, holding it for a given time and then progressively returning it to room temperature. The aim is to induce a microscopic modification of the material resulting in improved mechanical, wear and corrosion resistance [6-8]. In high-alloyed steels and cast irons, several authors have demonstrated an increase in the mechanical, electrical and corrosion response after CT [9-11], linked to microstructural changes promoting the conversion of retained austenite into martensite and inducing carbide precipitation [12-23]. This supplementary treatment has also been used to improve some properties in other non-ferrous

alloys, such as magnesium, copper and titanium [24-26]. The adoption of this heat treatment to increase the mechanical properties of aluminum alloys is quite recent [27,28], but the real influence of CT has not yet been clarified. Several studies were found in the technical literature [29-45], but the obtained results are often contradictory and not always justified by microstructural modifications. Recent studies have suggested the formation of hard secondary phases [33,34], grain-size effects [35] and the generation of dislocations [36]. Baldissera et al. [30] presented a review of CTs of several alloys resulting in a finer grain size and optimum dispersion of secondary phases. The study of Cabezea et al. [31] on 2xxx aluminum alloys showed that the CT increases the secondary incoherent nanoparticles in intergranular regions, and this slight precipitation did not produce any modification of the mechanical properties but an improvement in stress-corrosion cracking resistance.

In this investigation, the influence of cryogenic treatment on the corrosion resistance of a 7050 aluminum alloy was studied, and the resulting properties were related to microstructural changes. The mechanical strength and hardness were also investigated. This paper aims for clarify the microstructural transformation induced by CTs and justify the variation in corrosion and mechanical properties in high-performance 7xxx aluminum alloys.

2. Materials and methods

The present experimental investigation is focused on a 7050 aluminum alloy. The material was delivered as 100 mm thick plates in the T7451 temper, and the nominal chemical composition is reported in Table 1.

% Zn	% Mg	% Cu	% Zr	% Al
6.21	2.10	2.19	0.10	Bal.

Table 1. Nominal chemical composition in wt. % of the 7050 aluminum alloy.

The cryogenics treatment (CT) was performed at - 196 °C by dipping the specimens in liquid nitrogen. The heating back to room temperature was carried out slowly by means of an insulated box. To study the influence of low temperature on corrosion and mechanical performance, laboratory heat treatments were carried out to obtain T76 temper samples with and without CTs. The investigated conditions are described in Table 2. The influence of soaking time was investigated by hardness tests, and 72 hours of soaking was selected for mechanical and corrosion tests.

Id. code	Treatment procedure
T7451	As-delivered
T7451C	As-delivered, 72 h cryogenic soaking
T76	Solution treatment at 477 °C , aging at 121 °C 4.5 h + 166 °C 14 h
T76C	Solution treatment at 477 °C , aging at 121 °C 4.5 h + 166 °C 14 h, 72 h cryogenic soaking
T76SC	Solution treatment at 477 °C , 72 h cryogenic soaking, aging at 121 °C 4.5 h + 166 °C 14 h

Table 2. Details of the laboratory heat-treating procedures. The solution treatment and aging temperatures satisfy the ASTM B918/B918 M – 17a standard [1].

To determine the mechanical resistance of the investigated conditions, tensile tests were carried out on specimens from the long transverse (LT) direction using an Instron 4507 H universal machine according to

standard ISO 6892-1:2016 [44]. Intergranular corrosion tests were performed on prismatic specimens with dimensions equal to 25 mm x 25 mm x 20 mm. According to standard ASTM G110-92 [45], they were immersed for 24 hours in a solution composed of NaCl (57 g) and H₂O₂ (10 mL) diluted to 1 L with reagent water. All the plate surfaces (L-LT, L-ST, LT-ST – L is the longitudinal direction, LT is the long transverse direction and ST is the short transverse direction) were exposed to the corrosive environment after grinding with 120-grit paper. Figure 1 shows a scheme of the sample preparation for microscopic observation after corrosion tests.

Figure 1. Procedure for the analysis of the corroded profiles: (a) sample immersed in the testing solution; (b) cut along the L-ST plane; (c) observed corroded profile.

The evaluation of the corrosive attack was performed by image analysis according to the procedure described in literature [47]. Referring to Figure 2, each pit has been described by measuring its depth, width and roundness, which is defined according to Equation (1).

Figure 2. Image analysis of a corrosion pit.

$$R_i^* = R_i \frac{H_i}{L_i} \quad (1)$$

$$R_i = \frac{P_i^2}{4\pi S_i} \quad (2)$$

where R_i^* is the modified roundness, R_i is the standard roundness defined by Equation (2), P_i is the perimeter of the i -th pit and S_i is the area of the i -th pit. Since the mechanical properties of the alloy can be decreased by the presence of multiple corrosion pits, it is important to find a parameter representative of the surface condition. This value, called C_s and defined in Equation (3), is defined as the ratio between the total length of the defects and the total length of the original profile.

$$C_s = 100 \frac{\sum_i L_i}{L_{tot}} \quad (3)$$

where L_i is the length of the i -th corrosion pit and L_{tot} is the total length of the original profile (with no corrosion pits). Finally, Equation (4) provides a parameter representative of the whole surface damage in the presence of corrosion:

$$R_C = C_s R^* \quad (4)$$

where R^* is the average modified roundness. The corrosion resistance increases when R_C decreases.

3. Experimental results and discussion

The influence of soaking time at -196 °C was initially investigated by hardness tests (HBW 2.5/62.5) on T76-temper specimens. Varying the initial heat-treating condition, Figure 3 shows the influence of cryogenic treatment on hardness as a function of time.

Figure 3. Influence of soaking time at -196 °C on hardness.

The experimental data showed no appreciable difference among the tested conditions. In the technical literature, the influence of soaking time is debated. Dobbins and Barron [47,48] claim that the exposure time at cryogenic temperature directly affects the final material properties. Lulay et al. [25] studied the response of a 7075 aluminum alloy after cryogenic treatment of 2 and 48 hours, but no significant changes were observed in the mechanical resistance. The research conducted by Li et al. [39] showed that the exposure time at cryogenic temperatures can significantly affect the mechanical properties of an Al-Zn-Mg-Cu alloy, even if the mechanical strength is not linearly related to the treatment time. In the present paper, the authors decided to perform CTs at -196 °C with 72 hours soaking, which is the maximum investigated time. Figure 4 shows the results of tensile tests.

Figure 4. Ultimate tensile strength (UTS) and yield strength (Rp0.2) in the investigated conditions.

The obtained results show no or a very limited influence of the cryogenic treatment. This behavior, confirmed by the technical literature, was already observed by other authors [47,48] on a 7075 aluminum alloy, which has a chemical composition similar to that of 7050 aluminum alloys.

After the mechanical tests, the intergranular corrosion resistance was studied. All the rolling planes (L-LT, L-ST and LT-ST) were exposed to the test solution. Figure 5 shows some examples of corrosion pits developed from the L-ST plane.

Figure 5. Corrosion pits observed in the T7451C (a), T76 (b), T76C (c) and T75CS (d) specimens.

The images acquired after corrosion were investigated by quantitative analysis. Figure 6 shows a comparison among the investigated conditions as a function of the R_C parameter evaluated in all the rolling directions.

Figure 6. R_C parameter in all the rolling directions in each heat-treating condition after corrosion test.

The obtained results show that the R_C value is influenced by the cryogenic treatment. In particular, in the T76 temper, its value especially decreases in the presence of cryogenic treatment carried out between solubilization and aging (T76SC condition). This observation is confirmed by the average R_C values shown in Figure 7.

Figure 7. Average R_C values for T76, T76C and T76SC tempers.

For a better understanding of the metallurgical mechanisms involved, the pit morphology was investigated by calculating the modified roundness, R_i^* , in all the experimental conditions. The analysis concentrated on the most critical rolling direction according to the results shown in Figure 6, the L-ST plane. Figures 8 and 9 show the modified roundness values as a function of the area for each pit in all the experimental conditions. Figures 8 and 9 show that the pit morphology is similar in all the investigated conditions. However, the main difference is related to the number of pits and, in the presence of cryogenic treatment, this value decreases significantly, as shown in Figure 10.

In conclusion, the metallurgical quantitative analysis of the corroded samples shows that the cryogenic treatment is able to produce a decrease in the pit number, while it has a negligible influence on the pit morphology.

Figure 8. Comparison among the modified roundness values in T7451 and T7451C tempers as a function of the pit area.

Figure 9. Comparison among the modified roundness values in T76, T76C and T76SC tempers as a function of the pit area.

Figure 10. Comparison among the number of pits in all the heat-treating conditions.

For a better understanding of the metallurgical phenomena involved, the corroded T76, T76C and T76SC samples were investigated by scanning electron microscopy (Zeiss Sigma 500), and the precipitate distribution at the grain boundaries was observed. Images at very high magnifications were acquired and compared, as shown in Figures 11-13.

Figure 11. Precipitate distribution at the grain boundaries - T76 temper. Picture b) is the magnification of picture a).

Figure 12. Precipitate distribution at the grain boundaries - T76C temper. Picture b) is the magnification of picture a).

Figure 13. Precipitate distribution at the grain boundaries - T76SC temper. Picture b) is the magnification of picture a).

The comparison among the previous images shows a significant difference in the precipitate distribution. In fact, the cryogenic treatment produces a finer and more homogeneous precipitation of secondary phases in the whole grain. According to the technical literature, this phenomenon enhances the corrosion resistance

of aluminum alloys [1-5], as also recently observed by Bouzada et al. [42]. The homogenous precipitate distribution induces a lower electrochemical potential difference among the grain boundaries and the center-grain. This behavior generates a higher corrosion resistance of grain boundaries, and finally, a higher intergranular-corrosion resistance of the whole material.

4. Conclusions

In the present paper, the influence of cryogenic treatment on a 7050 aluminum alloy was investigated from the mechanical and corrosion points of view. Two treatment tempers were considered: T7451, the as-delivered, and T76, obtained by laboratory solubilization and aging. Cryogenic soaking at - 196 °C was carried out on both the as-delivered sample and the T76 temper. In the latter, cryogenic treatment was performed both after aging and between solubilization and aging. The corrosion properties indicated a certain influence of the cryogenic treatment, especially in the T76 temper. Microscopic observations revealed a different precipitate distribution at the grain boundaries in the presence of cryogenic treatment. The best corrosion resistance was obtained when soaking at - 196 °C was carried out between solubilization and aging. Instead, tensile tests did not reveal any influence of such different precipitate distributions. In fact, the mechanical strength was very similar in both T451 and T76 tempers. Future development of this project will be focused on stress-corrosion cracking tests to confirm the obtained results. Moreover, different soakings at - 196 °C will be considered to study the influence of the exposure time on the corrosion resistance.

Statements and declarations

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