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EFFECT OF CRYOGENIC AND HEAT TREATMENT ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Al-7Si-1,5Cu-Mg

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An Al-7Si-1.5Cu-Mg alloy was synthesized through the modification of a commercial A356 alloy with the inclusion of alloying elements. This alloy underwent a treatment regime comprising solid solution, cryogenic treatment, and ageing. The results indicate that cryogenic treatment increased nucleation rate of precipitates, which increases the number of precipitates and reduces their sizes, ultimately improving the mechanical properties and reduce secondary dendrite arm spacing of the alloy. Under the optimal treatment conditions (solid solution at 520 °C for 10 hours, followed by deep cooling for 48 hours, and ageing at 160 °C for 10 hours), the secondary dendrite arm spacing of the alloy was reduced by approximately 37,5 %, exhibiting hardness of 102,8 HV and plasticity of 4,2 %.

Key words: Al-7Si-1,5Cu-Mg, cryogenic treatment, precipitation, secondary dendrite arm spacing, Mechanical properties

INTRODUCTION

Aluminum alloys, characterized by their superior mechanical properties, boast high tensile strength, wear and corrosion resistance, coupled with notable plasticity and reduced weight. Their versatility has led to their extensive utilization in diverse sectors including automotive, aerospace, defense, and medical device industries [1].

Ultra-low temperature treatment of aluminum alloys, commonly referred to as cryogenic treatment, involves subjecting the alloy to temperatures typically below -150 °C. This treatment has been recognized for its potential to refine the microstructure, particularly enhancing the distribution of precipitates and optimizing the size and density of secondary phases. Consequently, cryogenic treatment can lead to significant improvements in the alloy's mechanical properties, wear resistance, and overall performance, making it a focus of interest in advanced materials research.

While cryogenic treatment of A356 aluminum alloy has been addressed in existing reference [2], there is a notable dearth of studies focused on its application to the modified Al-7Si-1,5Cu-Mg alloy. This research gap is particularly significant due to the potentially influential role of copper (Cu) in shaping the alloy's microstructure and performance during cryogenic treatment. As a result, the need for comprehensive investigation in this area becomes evident.

This study undertook a systematic exploration of the Al-7Si-1,5Cu-Mg alloy's microstructural evolution and enhancement of mechanical properties following its solid solution and subsequent cryogenic ageing treatment under varied conditions. The objective was to elucidate the underlying mechanisms of how cryogenic processing influences the alloy, ultimately deriving an optimal treatment process. It is anticipated that these insights will catalyze the broader integration of cryogenic treatment methodologies in industrial aluminum alloy production.

EXPERIMENTAL MATERIALS AND METHODS

The composition of the new Al-7Si-1,5Cu-Mg alloy is shown in Table 1.

The surface of the alloy was ground and polished and then melted in a pit-type resistance furnace at a preset temperature of 740 °C. After melting of the alloy, Al-50Cu intermediate alloy was added at 750 °C. It was left to stand for three minutes and then stirred with a quartz rod for five minutes. Al5TiB, Al10Sr and Al20La, Al20Ce intermediate alloys were added at 740 °C and 760 °C respectively and stirred well. Sprinkle a layer of slagging agent on the surface of the alloy and hold for 15 minutes. Reduce the temperature to 750 °C, add the refining agent and hold for 10 minutes. The alloy was slagged and poured into a 200 °C mold at 720 °C to obtain an Al-7Si-1,5Cu-Mg alloy, which was subjected to the cryogenic treatment process shown in Table 2.

The samples' surfaces were sanded with sandpaper and then polished using a semi-automatic polishing machine until they were mirror-like and free of scratches.

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Table 1 **Chemical composition of Al-7Si-1,5Cu-Mg alloy WT. %**

Element	Si	Mg	Cu	La	Ce	Ti	B	Sr	Fe
Content (ω/%)	6,5	0,34	1,6	0,19	0,1	0,12	0,03	0,03	0,1

Table 2 **Cryogenic treatment process**

Name	Solid solution	Cryogenic	Aging
S12	520 °C×10 h	liquid nitrogen×12 h	160°C×10 h
S24	520 °C×10 h	liquid nitrogen×24 h	160°C×10 h
S36	520 °C×10 h	liquid nitrogen×36 h	160°C×10 h
S48	520 °C×10 h	liquid nitrogen×48 h	160°C×10 h

The samples were put into the X-ray diffraction (XRD) diffractometer. The "MEASUREMENT" option was chosen, with a scanning angle range of 10-90°. The voltage was set to 40 kilovolts kV, the current to 40 milliamperes (mA), and the scanning rate to 4° per minute. The specimens were immersed in an electrolyte solution consisting of a mixture of HCl_4 and C_2H_5OH. The electrolysis process was conducted for a duration of 10 seconds, under controlled conditions of a temperature of -10 °C and a voltage of 20 V. The samples were observed by Electron Backscatter Diffraction (EBSD) and Scanning Electron Microscopy (SEM) using a Gemini SEM electron microscope (voltage: 20Kv, step size: 5um, scanning area: 0,17 mm). The microstructure of the samples was observed using a Zeiss microscope. The samples were hardness tested using HV-1000A Vickers hardness tester. Tensile testing of specimens conforming to ASTM E8-2009 was carried out on a Shenzhen Rigorous RGM2010 model tensile machine.

EXPERIMENTAL RESULTS AND ANALYSIS

Microstructure characterization

As shown in Figure 1, the phase composition of the alloy is basically the same under different cryogenic treatment durations, which mainly consists of a large amount of greyish-white α-Al matrix, and greyish-black eutectic silica small particles phase. It mainly consists of a large amount of greyish-white α-Al matrix as well as greyish-black eutectic silicon small particle phase. Eutectic silicon tends to be distributed along the grain boundaries of α-Al and the boundaries of secondary dendrite arms (SDAS). Under the conditions of cryogenic, the eutectic silicon undergoes a transformation, causing a more rounded shape and the replacement of sharp corners with a passivated morphology. This alteration significantly diminishes the tearing impact of eutectic silicon on the alloy. As the duration of deep cooling is extended, the solidification dendrite arm spacing SDAS in the alloy structure exhibits a pattern of initial increase followed by subsequent decrease. The SDAS of aluminum alloys shows a trend like increasing and then decreasing as the duration of cryogenic treatment increases. After 48 h of cryogenic treatment, the SDAS size of the aluminum alloy was reduced to a minimum value of 20,1 um (as shown in Figure 1d), which is about 35 % lower than the maximum value. And more α-Al dendrites appeared in the alloy.

Figure 2 shows the EBSD image of the alloy after cryogenic treatment. As can be seen from Figure 2, the grain morphology of the alloy is basically the same after cryogenic treatment for different times, and its size is in the range of 200 - 300 um. As can be seen from the figure, the cryogenic treatment with different time lengths does not cause significant changes in the grain size of the Al-7Si-1,5Cu-Mg alloy.

X-ray diffractogram (XRD) of the alloy is shown in Figure 3, from which it can be seen that α-Al matrix, Si phase as well as Mg_2Si phase are mainly present inside the alloy. This data is consistent with the microstruc-

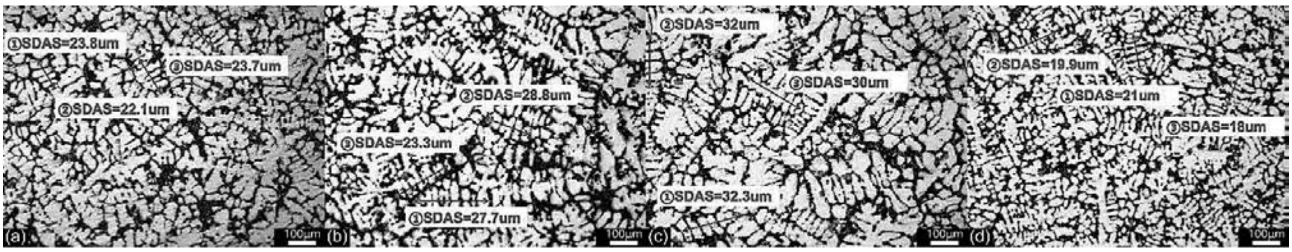

Figure 1 Microstructure (a) S12, (b) S24, (c) S36, (d) S48.

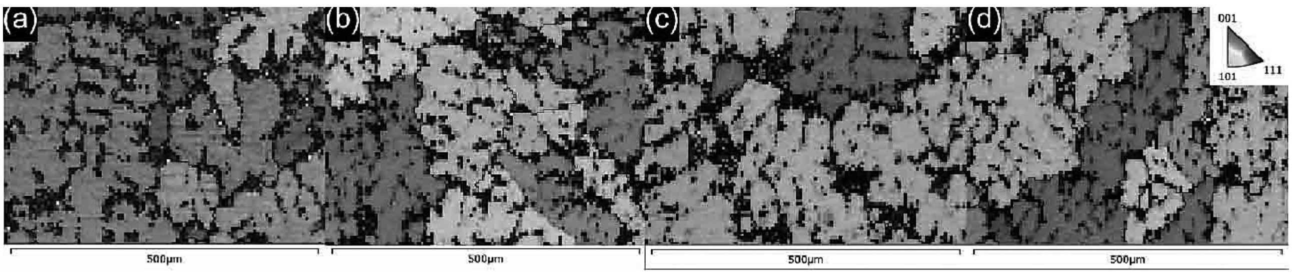

Figure 2 EBSD diagram of the alloy (a) S12, (b) S24, (c) S36, (d) S48

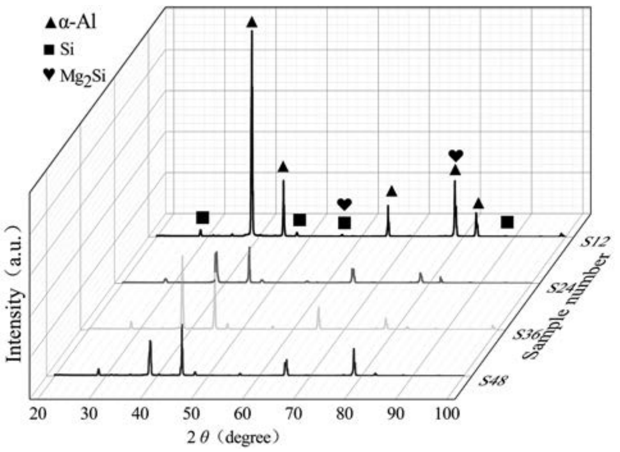

Figure 3 XRD diffraction pattern (a) S12, (b) S24, (c) S36, (d) S48.

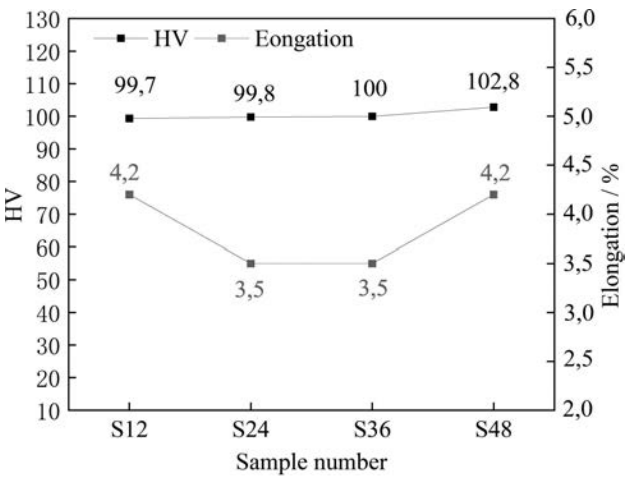

Figure 4 Mechanical properties (a) S12, (b) S24, (c) S36, (d) S48.

tural composition of the alloy as described above and further supports the compositional composition of the alloy. Cryogenic treatments can also cause changes in the strength of the X-ray diffraction peaks of the alloy. As in the direction of (111), the intensity of the (111) diffraction peak shows a significant decrease when the cryogenic treatment time exceeds 12 h. This suggests that cryogenic treatment reduces the tendency of Al-Si alloys to undergo selective orientation at the (111) crystal plane.

Mechanical properties

Figure 4 shows the variation of hardness and plasticity of the alloy at different cryogenic treatment times. It can be seen from the graph that the hardness of the alloy shows a gradual increase with the increase in deep cooling time. The hardness reached its highest value of 102,8 HV after 48 hours of cryogenic treatment. Its plasticity shows a tendency to decrease and then increase with the cryogenic time. A comprehensive analysis of the hardness and plasticity of the alloy shows that the alloy has the best performance when the heat treatment process of solid solution 520 °C × 10 h + cryogenic × 48 h + aging 160 °C × 10 h is used.

The analysis of the tensile fracture of the alloy was conducted with varying cryogenic treatment times, as depicted in Figure 5. The results show the presence of a large number of tear ribs (black arrows as shown) as well as a small number of tough fossae (green arrows as shown) at the fracture. Combined with the tensile properties of the alloy it can be deduced that the alloy is brittle fracture at this point. suggest that the alloy exhibits brittle fracture characteristics at this particular time. This finding is consistent with Fiedler's, T et al [3] findings that low temperatures lower the plasticity of Al-Si-Mg alloys. With the increase of cryogenic time, the number of dimples at the fracture showed a tendency to decrease and then increase, while the tear rib showed a tendency to increase and then decrease. The alloys S12 and S48 show a large number of dimples and a small number of tear ribs. This phenomenon indicates that its elongation is better than that of S24, S36, and this result is in line with the trend of elongation of the four alloys in Figure 6.

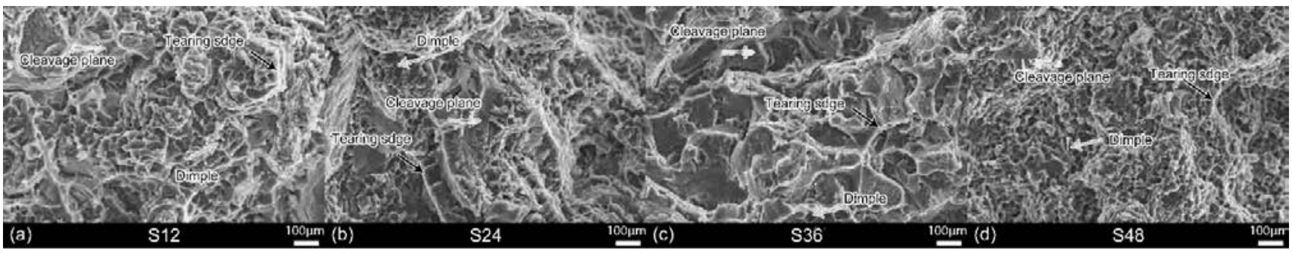

Figure 5 Tensile fracture

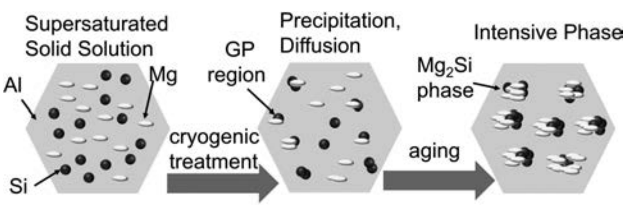

Figure 6 Intensive precipitates principle

Mechanistic analysis

From the equation for the nucleation rate I versus the temperature T: $I = \beta \exp\frac{-\Delta G_A}{kT} \times \exp\frac{-\Delta G}{kT}$ [4] (β: potential nucleation site density parameters, ΔG_A: diffusion activation energy, ΔG: nuclear activation energy, k:

Boltzmann's constant). Cryogenic treatment increases the nucleation rate of the alloy's reinforcing precipitation phase, thereby increasing the strength of the alloy's alloy.

When the alloy is solution treated, the temperature is raised to 520 °C. Phases such as Mg_2Si and Al_2Cu in the alloy begin to dissolve, forming atoms such as Mg, Si and Cu. These solute atoms solidify into the aluminum matrix (Figure 6). After quenching treatment, the alloy is in a supersaturated solid solution state. The alloy is then placed in liquid nitrogen at -196° C. At low temperatures, the supersaturation of solute atoms is increased, with a consequent increase in the driving force for the precipitation of atoms such as Si, Mg and Cu. Prolonged low temperatures reduce the concentration of vacancies and further facilitate the precipitation of solute atoms. Due to the slow molecular motion in the low-temperature environment, these solute atoms are unable to move over long distances, and can only be biased towards the nearby zone. Many diffuse and uniformly distributed solute atom-enriched micro-zone appear within the matrix. This process is known as the GP zone core formation process. A large number of GP zone cores converge, culminating in a diffuse and evenly distributed GP zone. The cryogenic treatment at this point acts as a pre-ageing process. During subsequent artificial aging at 160 °C. The high-density GP zone becomes a precipitation core for transition phases such as β''-Mg_2Si, and a large number of Mg_2Si reinforced phases begin to form in the matrix [4].

Therefore, the cryogenic treatment promotes the formation of a large number of diffuse and homogeneous GP zones within the alloy. This will increase the nucleation point of the Mg_2Si phase and improve its nucleation rate. This change leads to the generation of a large number of diffuse and homogeneous Mg_2Si phases in the alloy, which enhances the precipitation strengthening of the alloy.

CONCLUSION

Upon incorporation of Cu and Sr elements, cryogenic treatment retains its efficacy in the modified alloys without exacerbating any adverse impacts of Cu.

The alloy's secondary dendritic arms witnessed a reduction of approximately 37,5 % from their peak values post a 48-hour cryogenic treatment.

Subsequent to cryogenic treatment, the alloy's plasticity initially declines, exhibiting a brittle fracture mode. However, after an extended deep cooling period of 48 hours, the alloy's plasticity begins to recover.

For the newly constituted Al-Si-Mg alloy, the optimal cryogenic treatment protocol is: solid solution at 520 °C for 10 hours, cryogenic processing for 48 hours, followed by aging at 160 °C for 10 hours. This regimen imparts the alloy with hardness of 102,8 HV and plasticity of 4,2 %.

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Note: The responsible translator for English language is C. Yang, Ningbo, China