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Production of AlSi10Mg alloy materials: SLM technology and cryogenic treatment

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Abstract

Aluminum alloys have recently gained significant importance in the industry due to their increasing use in various applications. Among them, AlSi10Mg alloys are especially utilized in the automotive, aerospace, and defense sectors, where the need to balance low weight with desirable thermal and mechanical properties is critical. The rapid advancement of additive manufacturing (AM) technologies has further driven research efforts to develop new alloys that are both environmentally sustainable and economically viable. Selective Laser Melting (SLM), a key AM technology, is particularly advantageous for producing complex components, as it enables the creation of lightweight parts with optimized topology. Despite these advantages, post-processing requirements remain a challenge in AM. Cryogenic treatment, which has shown promising effects on AlSi10Mg parts, is sometimes chosen even when specialized heat treatments are needed. This study examined the mechanical properties of AlSi10Mg alloy produced using SLM. The blocks were manufactured using a stripe scan strategy with 700W laser power, dual laser, and a 60 µm layer thickness. After production, samples were extracted from these blocks and subjected to cryogenic treatment in cycles of 24 and 36 hours at -196°C. Following the cryogenic treatment, tensile and hardness tests were conducted to investigate the mechanical properties. The results indicated that the hardness of the cryogenically treated samples improved over their as-built state after a 36-hour cycle, but decreased after a 24-hour cycle. While there were no significant differences in hardness based on the length of the cryogenic treatment, the percent elongation values increased with longer treatment cycles. However, tensile and yield strength values were higher in samples treated for 24 hours, with a slight decrease observed in those treated for 36 hours. In conclusion, although the cryogenic process did not significantly alter the hardness of the SLM AlSi10Mg samples, it had a notable impact on their tensile strength, yield strength, and percent elongation values.

Keywords: Additive Manufacturing, Selective laser melting, Cryogenic treatment, Al alloys, AlSi10Mg

1. Introduction

AlSi10Mg alloy has begun to be preferred especially in the aviation and aerospace industry due to its lightness, which is one of its important advantages. Aluminum-magnesium alloys are both lighter than other aluminum alloys and much less flammable than alloys containing very high amounts of magnesium. For this reason, today Al-Mg alloys have become one of the most popular and valuable materials as they are used in many engineering applications [1].

Cryogenic treatment process has become more interesting in recent years as an answer to existing questions in the industry. This method involves cooling a material to a temperature lower than 0˚C to induce certain qualities in the material. A few studies available in the literature indicate that there is a positive increase in the mechanical, electrical and corrosion properties of high alloy steels and cast irons after cryogenic treatment.

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Cryogenic treatment has also been used to improve some properties in other non-ferrous alloys such as magnesium, aluminum, copper and titanium. The adoption of this heat treatment to increase the mechanical properties of aluminum alloys is quite new in the literature. However, it is stated that the actual effect of the cryogenic process on this material still continue [2,3].

Additive manufacturing, also known as 3D printing, is a production method that allows rapid prototype production while facilitating the production of complex shaped parts that are not possible with traditional manufacturing methods. Briefly, the desired shape is created by depositing material layer by layer through a nozzle [4,5,6]. This production technology, which enables production from powder or wire and is based on a system that provides heat input, enables fast production [4] . In this study, the production method was selected as the Selective Laser Melting Selective Laser Melting method, which is one of the additive manufacturing technologies. The SLM method involves the layer-by-layer melting of the powder bed of the material composition to be produced with the help of laser power. The biggest advantage of this method is the ability to produce directly from powder properties without the need for secondary metallurgical processes and alloying. The intense preference for this method is a focus of great interest for the industry, as it can produce lightweight parts and provides economic advantages by eliminating other secondary metallurgical methods.

Selective Laser Melting is gaining worldwide attention thanks to the possibility of achieving a free-form fabrication combined with high mechanical properties associated with a very thin microstructure. However, SLM is very complex from a physical perspective due to the interaction between a concentrated laser power and metallic powders and the extremely rapid melting and subsequent rapid solidification. Despite these advantages, the desired part properties cannot always be achieved “as-built”, that is, in its untreated form, with the Selective Laser Melting method. To give an example, heat treatment is one of the most preferred methods, as it is quite possible to achieve goals such as enhancing surface features and producing dense structures through secondary (post-processing) methods [1,7]. Since the production of AlSi10Mg alloys by traditional methods (e.g. casting) is a very laborious process and requires secondary metallurgical processes to create the final part produced different methods have become a subject of interest in the literature and industry. For example, in the casting production method, special mold designs/production and processes are required for complex structured parts, as well as special molds are used for casting aluminum alloys due to their reactivity, and these incur extra costs for the industry [7,8]. In the literature, it is indicated that SLM plays a crucial role when producing these alloys. SLM technology is frequently utilized in AlSi10Mg alloy additive manufacturing because it produces high density components [9].

However, traditional heat treatments can weaken mechanical properties while eliminating residual stress, which is still one of the biggest challenges in additive manufacturing. In recent years, a more advanced and innovative possibility to improve the service performance of metallic alloys has been identified in studies on cryogenic processing [2,7]. The aim of the cryogenic process is to cause a microscopic modification in the material and achieve improved mechanical, wear and corrosion resistance [8]. In a study, it was indicated that the deep cryogenically treated samples showed changes in the values of yield strength, tensile strength, and percent elongation compared to EBM as-built samples [11].

SLM technology is frequently used in additive manufacturing of AlSi10Mg alloy as it enables the production of high-density components. When producing metal parts of the required quality with SLM, the manufacturing parameters used to melt the metal powder in the powder bed and solidify it as desired are very important [12]. In the literature which compared that SLM and as-cast condition of AlSi10Mg alloy materials and, significant performance variations were observed between SLM AlSi10Mg and as-cast. A higher tensile strength was achieved with the separate precipitated phases and finer grain of SLM AlSi10Mg. The brittle SLM AlSi10Mg fatigue limit was lowered by more internal defects and coarser precipitates [13]. Additionally, because SLM material has a finer microstructure than cast material, it has a greater hardness value. Tensile studies, however, show that cast parts has higher strength than SLM produced part [14]. This study aims to improve mechanical properties such as yield strength, tensile strength, percent elongation, and hardness by applying cryogenic treatment to the parts produced by SLM.

2. Materials and Method

2.1 SLM Production

From Table 1 it is possible to see the standard powder composition belongs to AlSi10Mg alloys by %weight. It contains 9-11 % Si, 0.20-0.45 Mg, 0.55% Fe, 0.45% Mn, 0.05% Cu-Ni-Pb-Sn, 0.10% Zn, 0.15% Ti and balanced (~89%) Al.

Table 1. The standard powder chemical composition corresponds to AlSi10Mg alloy [14].

Elements	**Al**	**Si**	**Fe**	**Cu**	**Mn**	**Mg**	**Zn**	**Ti**	**Ni**	**Pb**	**Sn**
%weight	Balance	9-11.00	0.55	0.05	0.45	0.20-0.45	0.10	0.15	0.05	0.05	0.05

First, part production was carried out with SLM Solutions SLM280HL device using the production parameters can be seen in Table 2. The scanning strategy was stripe and laser power was 700W with 1850 mm/s. The beam diameter 85 µm and 0.17 mm hatch distance. And then, 8 test samples removed from this part for mechanical examinations according to ASTM E8/E8M − 13a standard size.

Table 2. Production parameters of the SLM parts.

Name of the parameter	**Type/Value**
Scanning strategy	Stripe
Laser power	700 W
Scanning speed	1850 mm/s
Beam diameter	85 µm
Hatch distance	0,17 mm

The particle sizes of the powder used in production are between 20 - 63 µm and the layer thickness of the production is 60 µm.

2.2 Cryogenic Treatment Conditions

After test samples production, cryogenic treatment was carried out to those samples as the four test samples 24 hours and 4 test samples 36 hours cycle at -196˚C with nitrogen fluid, respectively. In this work, deep cryogenic treatment was applied to the samples for the best results. From Figure 1 it can be seen that cryogenic treatment steps that applied to as-built AlSi10Mg samples. Firstly, samples were cooled down to -196˚C from room temperature and heated up to the room temperature. ~~carried out~~. After this step, tempering was carried out at 200˚C, followed by soaking at that temperature for 1 hour. The final step was cooling under atmospheric conditions to room temperature.

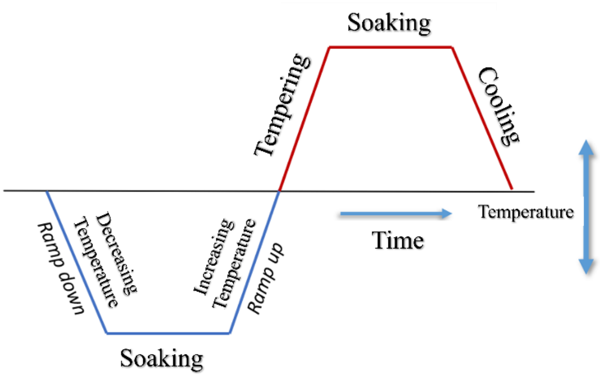

Fig. 1. Cryogenic treatment steps that applied in AlSi10Mg samples.

2.3 Mechanical Characterization

For mechanical examination tensile tests were carried out in horizontal testing device at room temperature according to ASTM E8/E8M − 13a standard. In addition, hardness tests were performed Future Tech FM-800 device 100 gf and 10 seconds with the Vickers indentation.

2.4 Microstructural Characterization

For microstructural investigations Nikon Eclipse Upright Optical Microscope was used. AlSi10Mg samples were etched with Keller etchant. The vertical section of the samples was examined.

3. Results and Discussion

3.1 Microstructural Examinations

Figure 2 shows the microstructural images belonging to AlSi10Mg samples. All examinations were carried out within the vertical section of the samples. It has been observed that a different formation was obtained in the microstructural examination compared to the known conventionally produced AlSi10Mg material microstructure. This has been determined to be due to the Selective Laser Melting process, which involves intense heat input and rapid cooling. In Figure 2.a, it is shown that microstructure of the as-built AlSi10Mg sample. The formation of a melt pool displaying a "fish scale" morphology was observed. It was noted that these melt pools were regular, with their geometry resembling a circular segment. OM images of the samples subjected to cryogenic treatment for 24 hours are shown in Figure 2.b, and for 36 hours are shown in Figure 2.c. It was observed that the scanning tracks exhibited similar characteristics in both as-built and cryogenic treated samples (Figure 2.a).

In the cryogenically treated AlSi10Mg samples (Figure 2.b and Figure 2.a), a finer microstructure was observed compared to the as-built sample (Figure 2.a). This is related to the reduction of internal stresses in the material and the triggering of grain refinement after cryogenic treatment. It was particularly noted that the melt pool structures between the grains in the microstructure were prominent and located at the grain boundaries. Additionally, the yellow-orange discoloration observed in some regions of the microstructure was due to the appearance of silicon phases in different colors on the surface after the etching process. These colored silicon phase structures may have concentrated within cellular and dendritic structures and could be observed in these tones as a result of chemical interactions. While these formations were more prominent in Figures 2.a-b, they were observed to be less intense in Figures 2.c.

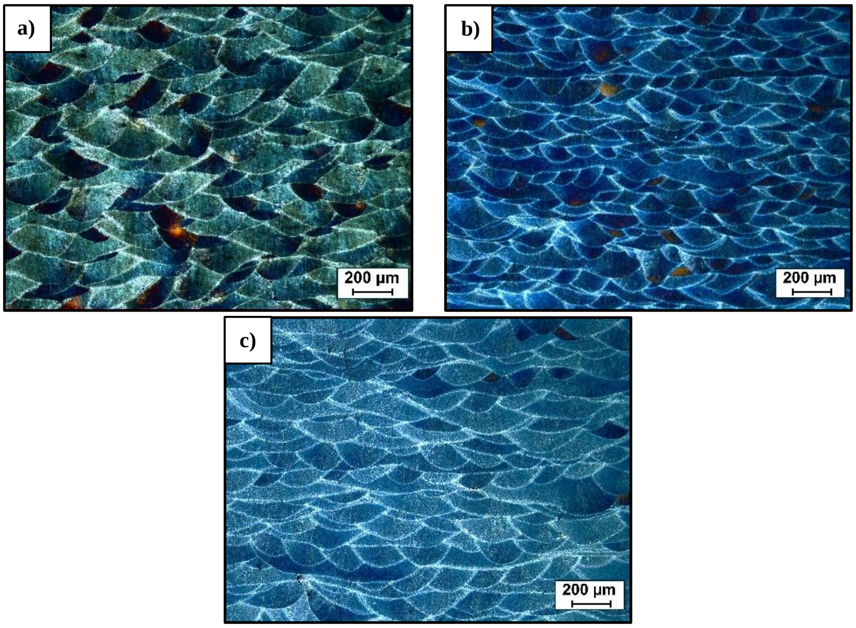

Fig. 2. Optical Microscope images of AlSi10Mg -196°C **a)** as-built, **b)**24 h, **c)** 36 h cycle time cryogenic treated samples at 50x magnification.

It is well-known from the Al-Si phase diagram that the formation of α-Al and Si particles is very common in AlSi10Mg structures [15]. The dark-colored regions are referred to as Si particles, while the light-colored regions represent α-Al areas. Cryogenic treatment was found to not affect removing the scanning tracks in parts produced by the SLM method. Apart from that, it was observed that the sample subjected to 36 hours of cryogenic treatment had more irregular melt pools compared to the 24-hour sample.

3.2. Mechanical Test Results

Test samples were removed from the completed block production part with the production parameters of which are given in Table 1 and subjected to mechanical tests. Mechanical test results of the produced parts after cryogenic treatment are shown in Table 2. It was observed that although the parts are produced with the same production parameters, it was observed that the yield strength, tensile strength, and elongation values significantly differed between the 24-hour and 36-hour cryogenic treatments.

Table 2. The mechanical test results belong to the samples.

Sample Name	Yield Strength (MPa)	Tensile Strength (MPa)	%Elongation	Hardness (HV)
As-Built	259.2 ± 6.9	406.4 ± 5.2	2.9 ± 0.7	112.5 ± 0.9
24h Cryogenic (A)	279.5 ± 25.8	426.6 ± 19.5	4.9 ± 1.5	106.7 ± 6.6
36h Cryogenic (B)	255.2 ± 15.8	419.5 ± 13.2	5.9 ± 2.1	116.5 ± 12.6

According to the results, the as-built samples had a yield strength of 259.2 ± 6.9 MPa. After the 24-hour cryogenic treatment, this value increased to 279.5 ± 25.8 MPa. Tensile strength values also exhibited the same behavior as yield strength, which was 406.4 ± 5.2 in the as-built state was observed to be 426.6 ± 19.5 MPa after 24 hours of cryogenic treatment. However, just like the yield and tensile strength values, also the percent elongation values increased from 2.9 ± 0.7 % in the as-built state to 4.9 ± 1.5 % after 24-hour cryogenic treatment.

It was observed that the 36-hour cryogenic treatment caused a decrease in the yield strength, causing the value to become 255.2 ± 15.8 MPa from 259.2 ± 6.9 MPa compared to the as-built state, which is nearly similar. It was observed that there was an increase in the tensile strength from 406.4 ± 5.2 MPa to 419.5 ± 13.2 MPa, but it was still lower than the 24-hour cryogenic treatment. Percent elongation values belong to 36h cryogenic treated samples observed as 5.9 ± 2.1 which more than as-built state and 24 hour treated samples. When 12-hour and 24-hour cryogenic treatments are compared in the literature, it is stated that the highest UTS value was ~~is~~ observed in the 24-hour cryogenic treatment [1]. It is indicated that only second phase precipitation is found to be responsible for improvement in mechanical properties of nonferrous material [10]. In addition, in a study where cryogenic treatment was applied to AlSi10Mg parts produced by additive manufacturing was interpreted as having similar microstructure therefore, showed similar mechanical properties with untreated as-built samples [14].

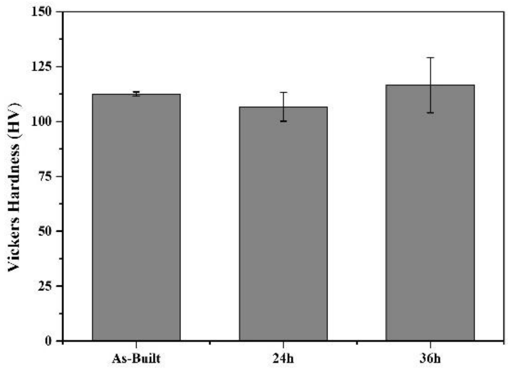

Fig. 2. Hardness graph belongs to the AlSi10Mg samples.

In Figure 2, it is possible to see the hardness graph of the as-built, 24-hour treated, and 36-hour treated AlSi10Mg samples. If the hardness values for 24-hour cryogenic treatment and 36-hour cryogenic treatment are compared, it is seen that the values are 106.7 ± 6.6 HV and 116.5 ± 12.6 HV, respectively. The 36-hour treatment had a greater effect on the hardness than the 24-hour treatment. The increase in hardness, especially in the 36-hour cryogenic treatment, can be associated with the increase in the precipitate phase as the cryogenic treatment increases. Although cryogenic treatment may occasionally influence hardness, some studies in literature report little or no effect [10]. The reason for the hardness value sometimes not changing at all can be related to whether there is a sufficient amount of precipitate in the structure after the cryogenic treatment. However, it is indicated that, after cryogenic treatment of AlSi10Mg samples for 12h, and 24h the hardness increased to 18.4% and 21.47% respectively. This increase is attributed the grain refinement during the cryogenic treatment [1]. Furhermore, according to literature, it is observed that the same amount of tensile is obtained with the heat treatments currently applied samples. In this way, it can be concluded that both hardness values and tensile and yield strengths are improved [12].

4. Conclusion

In this study, the effect of 24-hour and 36-hour cryogenic treatment on the mechanical properties of parts produced by the SLM method was examined and the results are listed as follows:

- As the cryogenic treatment time increases, grain coarsening is observed. Finer grains are present in the 24-hour cryogenic treatment compared to the as-built sample.

- It was observed that the samples exposed to 24-hour cryogenic treatment caused an increase in yield and tensile strength values, and also percent elongation value increased compared to the as-built samples. The improvement in yield and tensile values in the 24-hour sample can be explained by grain refinement.
- While decreasing in yield strength, tensile strength; in percent elongation values increasing was observed parts that exposed to 36-hour cryogenic treatment compared to 24 hours, and there was a noticeable increase in the hardness value.
- When comparing the as-built samples with those subjected to 36-hour cryogenic treatment, it was observed that there was a decrease in yield strength, while tensile and hardness values increasing, also there was increasing in percent elongation values. Finally, it can be said that the most important effect of increasing cryogenic treatment soaking time on the samples was the increase in hardness and percent elongation values, this effect was observed as a decrease for tensile and yield strength.
- It is inevitable that all these observed mechanical properties should be verified with a complementary microstructure analysis. In particular, it was concluded that in order to understand the effects of the cryogenic treatment on the sample more clearly, a complementary heat treatment should be selected, and the effects should be compared in the continuation of the study.

Author Contribution Statement

Conceptualization: T.K., and S.K..; Methodology: T.K., S.K. and P.S.; Investigation: S.K. and P.S.; Resources: P.S., T.K and S.K.; Data Curation: P.S., S.K. and T.K.; Writing – Original Draft: P.S. and S.K.; Writing – Review & Editing: P.S. and S.K.; Visualization: P.S.; Supervision: T.K. and S.K.

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