




# Effect of Niobium on the Size and Morphology of Fe-rich Intermetallic Phases in an AlSi5Cu2Mg Alloy

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## Abstract

The presence of iron (Fe) in foundry Al-Si alloys significantly impacts the properties of the final castings. Iron tends to form hard, brittle, needle-like  $\beta$ -Al<sub>3</sub>FeSi phases that act as stress concentrators, leading to reduced ductility and toughness. Transforming the morphology of Fe phases is essential, especially for aluminum alloys intended for large-scale castings where structural integrity is crucial. Niobium acts as a potential inoculating element and modifier that favorably influences the morphology and size of Fe phases. This study focuses on the effect of niobium (Nb) on the morphology and size of iron (Fe) phases. The study showed that adding Nb resulted in favorable fragmentation and size reduction of the needle-like  $\beta$ -Al<sub>3</sub>FeSi phases. Changes in the morphology of Fe-rich phases can positively impact the foundry, mechanical, and physical properties of the alloy under investigation. From this perspective, niobium appears to be a promising inoculating element for aluminum alloys.

**Keywords:** AlSi5Cu2Mg, niobium, Fe intermetallic phases

## 1. Introduction

The aluminum alloy AlSi5Cu2Mg is used for highly stressed automotive components. Its specific chemical composition, which is characterized by a low silicon content, makes it an ideal structural material for large and complex castings. Due to its hypereutectic composition ( $\leq 6$  wt.% Si), the AlSi5Cu2Mg alloy has a wide solidification range [1]. However, this range makes the alloy more susceptible to hot tearing and reduces its ductility. This significantly risks the structural integrity and operational reliability of large castings with complex geometry.

The final properties of aluminum alloys are significantly influenced by impurities. One of the most critical impurities is iron (Fe). Even at low concentrations, precipitation of Fe-based intermetallic phases occurs. According to the phase

thermodynamics of the Al-Si-Fe system, under normal cooling rates, the undesirable monoclinic  $\beta$ -Al<sub>3</sub>FeSi phase forms preferentially [2]. These hard, brittle  $\beta$ -Al<sub>3</sub>FeSi phases precipitate in the matrix as sharp-edged needles. These phases act as stress concentrators, creating favorable sites for crack initiation and further propagation. The presence of  $\beta$ -Al<sub>3</sub>FeSi phases reduces the ductility and fracture toughness of the alloy. Controlling the morphology of the Fe phases is essential for improving the material's mechanical properties [2, 3].

One way to optimize the properties of aluminum alloys is through metallurgical melt treatment. Melt treatment can significantly impact the size of the primary phase and the morphology of the eutectic silicon (Si) and iron (Fe) phases, as well as the width of the solidification interval [4]. Studies have shown that inoculation is an effective way to reduce the susceptibility of Al-Si-Cu-Mg alloys to hot tearing [4]. However, inoculation of an



AlSi5Cu2Mg alloy with a standard Ti-based inoculant is limited by the maximum Ti content of 0.03 wt.%. Therefore, this technological limitation stimulates research into new, non-standard inoculating elements that are not based on the conventional Al-Ti-B system. Research has shown that inoculating aluminum alloys with the transition element niobium (Nb) is becoming increasingly important [5, 6].

Niobium is being considered as a potential inoculating element for Al-Si alloys [5, 6]. Grain refinement in aluminum alloys improves mechanical and casting properties and reduces porosity and susceptibility to hot tearing [7]. Nb also tends to form thermodynamically stable Al<sub>3</sub>Nb-based phases, resulting in favorable improvements in mechanical and physical properties at elevated operating temperatures [8]. A literature review has shown that adding Nb has a positive effect on Fe-rich phases [5]. Due to the addition of Nb, needle-like β-Al<sub>3</sub>FeSi phases transform into more compact Fe phases. This change in the morphology of the Fe phases reduces internal stresses, resulting in increased ductility. [9]

This work is part of broader research focused on the effects of niobium (Nb) on the microstructure and properties of the aluminum alloy AlSi5Cu2Mg. The goal is to develop an aluminum alloy based on AlSi5Cu2Mg that meets the specific requirements of structural materials used in large castings. This study focuses exclusively on analyzing the effect of graded Nb addition on the morphology and size of Fe-rich phases.

## 2. Materials and Methods

AlSi5Cu2Mg was chosen as the experimental alloy. Its chemical composition is shown in Table 1. Based on its chemical composition, it is evident that the alloy was supplied in a pre-modified state.

Table 1.

Chemical composition of AlSi5Cu2Mg alloy [wt.%].

Si	Cu	Mg	Mn
5.58	1.92	0.29	0.013
Fe	Sr	Nb	Al
0.19	0.03	-	Bal.

The AlSi5Cu2Mg alloy was melted in an electric resistance furnace. The alloy's melting temperature was 750 °C ± 5 °C. Niobium was then added to the melt in the form of an AlNb10 master alloy. Al-Nb-based master alloys are classified as “difficult-to-melt” master alloys. To ensure complete dissolution of the master alloy in the melt, the AlNb10 master alloy was melted separately in an electric induction furnace and then added to the melt. The experimental alloys were obtained by adding 0.10, 0.15, and 0.20 wt.% of niobium (Nb). The alloys were labeled according to their nominal Nb content as follows: AlSi5Cu2Mg + X wt.% Nb (X = 0.10, 0.15, or 0.20). The chemical composition of the experimental alloys is shown in Table 2.

Table 2.

Chemical composition of experimental alloys [wt.%].

	AlSi5Cu2Mg + 0.10 wt.% Nb	AlSi5Cu2Mg + 0.15 wt.% Nb	AlSi5Cu2Mg + 0.20 wt.% Nb
Si	5.82	5.88	5.86
Cu	2.40	2.46	2.34
Mg	0.25	0.19	0.19
Mn	0.012	0.014	0.012
Fe	0.23	0.23	0.22
Nb	<b>0.049</b>	<b>0.063</b>	<b>0.076</b>
Sr	0.004	0.005	0.006
Al	Bal.	Bal.	Bal.

The experimental samples were prepared by pouring molten metal into a graphite-coated metallic mold. The mold was preheated to 150 °C ± 5 °C. The casting temperature was set at 750 °C ± 5 °C. A set of five test samples was prepared for each experimental variant to determine the mechanical and physical properties. Microstructural analysis, and thus evaluation of the size and morphology of the iron (Fe) phases, was performed on the test samples with the optimal combination of mechanical and physical properties. This analysis was performed using a Keyence VHX-7000 digital microscope and a TESCAN LMU II scanning electron microscope with a Bruker EDX analyzer. The size of the Fe phases was determined using the Keyence VHX-7000 digital microscope's image analysis software.

As part of the microstructural evaluation, we assessed the effect of graded Nb addition on grain refinement based on the SDAS (Secondary Dendrite Arm Spacing) index. The SDAS index determines the distance of secondary dendrite arms; the smaller the SDAS index value, the finer the structure and the smaller the segregation distances. The SDAS index is calculated as the ratio of dendrite length (L) to the number of secondary dendrite arms (n):

$$SDAS = L/(n-1) \quad (1)$$

## 3. Results and Discussion

### 3.1. Microstructural analysis

The microstructure of the aluminum alloy in the as-cast state without Nb addition consisted of the primary α-(Al) phase, eutectic Si, and Cu- and Fe-based intermetallic phases (Fig. 1). The eutectic silicon (Si) precipitated in the form of imperfectly rounded grains. The typical plate-like shape of the eutectic silicon was not observed due to the alloy's pre-modification by the producer using strontium (Sr). Cu-based phases were also detected in the metallographic section. They were observed in the form of compact-morphology ternary eutectics (Al-Al<sub>2</sub>Cu-Si) and isolated Al<sub>2</sub>Cu particles. The Fe-based intermetallic phases in the reference alloy were observed in two morphologies: plate-like β-Al<sub>3</sub>FeSi structures and segmented chinese script Al<sub>13</sub>(FeMn)<sub>3</sub>Si<sub>2</sub>.

Needle-like phases were observed in the matrix of the AlSi5Cu2Mg alloy by scanning electron microscopy (Fig. 2). EDX analysis identified these phases as  $\beta$ -Al<sub>3</sub>FeSi phases.

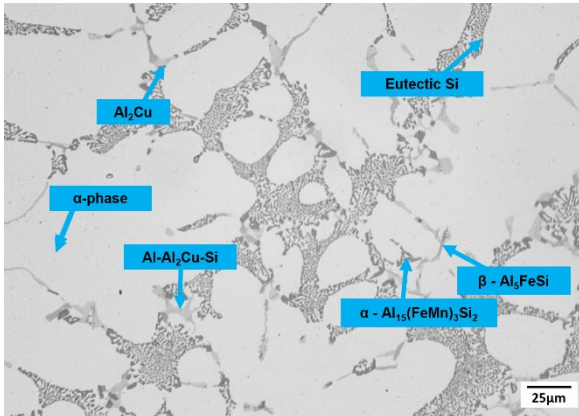


Fig. 1. Microstructural evaluation of AlSi5Cu2Mg alloy in as-cast state, etch. 0.5% HF

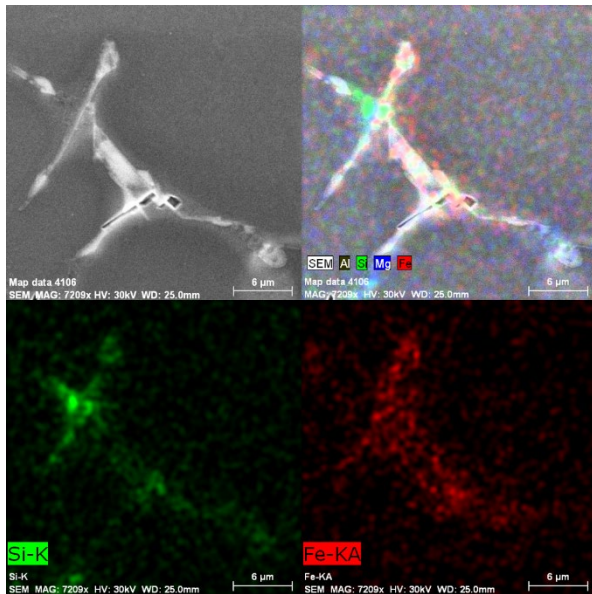


Fig. 2. Fe-rich intermetallic phases with corresponding EDX mapping of chemical elements (AlSi5Cu2Mg, as-cast state, etch. H<sub>2</sub>SO<sub>4</sub>), SEM

Similar to the alloy without Nb addition, the microstructure of the experimental alloys with Nb addition in the as-cast state consisted of the primary  $\alpha$ -(Al) phase, eutectic Si, and Fe- and Cu-based intermetallic phases (Fig. 3). Microstructural analysis revealed that Nb addition resulted in grain refinement of the primary  $\alpha$ -(Al) phase. This analysis also demonstrated a clear correlation between Nb addition and refinement of the AlSi5Cu2Mg alloy's microstructure. The most significant refinement was achieved at the highest Nb content. Studies indicate that refinement of the primary phase directly influences the morphology of the eutectic silicon. In the alloy without Nb addition, Si segregates into coarse networks in the interdendritic

regions (Fig. 1). In contrast, alloys with graded Nb additions have a fine-grained structure that leads to a substantially more homogeneous distribution of eutectic Si in interdendritic areas (Fig. 3). Microstructural analysis revealed Nb's favorable modifying effect on the Fe phases of the  $\beta$ -Al<sub>3</sub>FeSi type. In the metallographic section, fragmentation and size reduction of brittle, needle-like  $\beta$ -Al<sub>3</sub>FeSi phases were observed. The Fe phases were more uniformly distributed in the aluminum alloy matrix. These phases preferentially precipitated in more compact forms, such as Chinese script morphology.

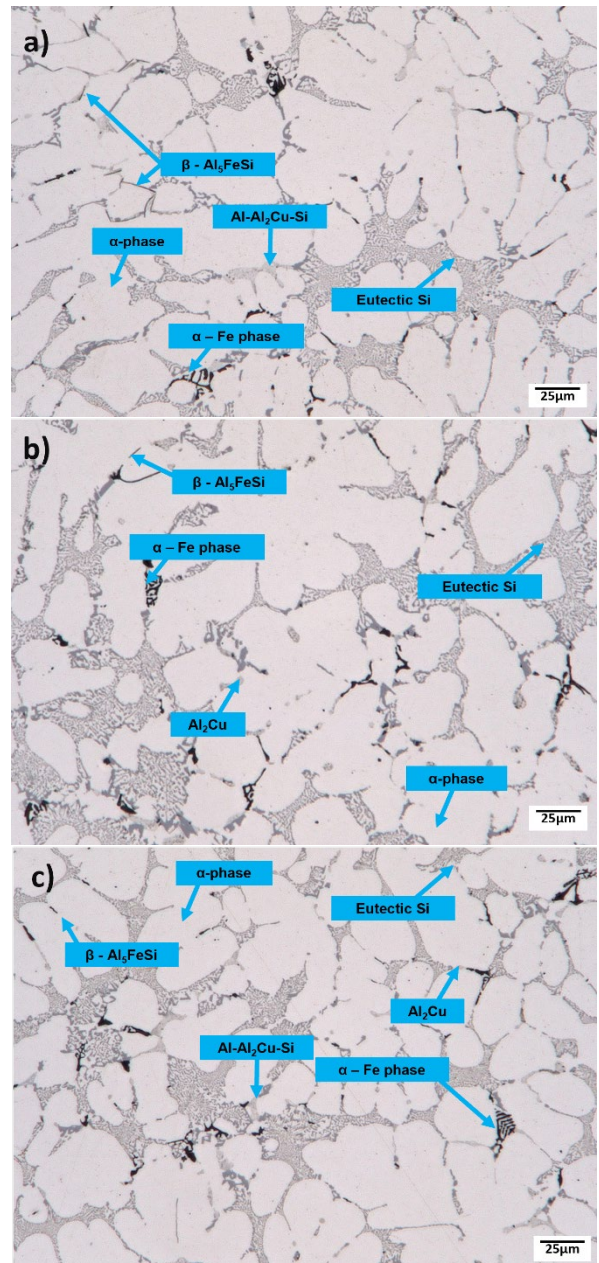


Fig. 3. Microstructural evaluation of aluminum alloys with nominal addition of: a) 0.10 wt.% Nb, b) 0.15 wt.% Nb, c) 0.20 wt.% Nb, in as-cast state, etch. 20 ml H<sub>2</sub>SO<sub>4</sub> + 100 ml H<sub>2</sub>O

EDX analysis revealed the presence of Fe-rich phases in the metallographic section. These phases either precipitate in a more favorable chinese script morphology (Fig. 4a) or form as small, compact structures resulting from the fragmentation of needle-like  $\beta$ -Al<sub>3</sub>FeSi phases (Fig. 4b). Unlike unfavorable needle-like phases, such compact Fe-based phases do not disrupt the aluminum alloy matrix to the same extent. The fragmentation and size change of the Fe phases can positively influence the mechanical and physical properties of the aluminum alloy under investigation, a topic that will be explored further.

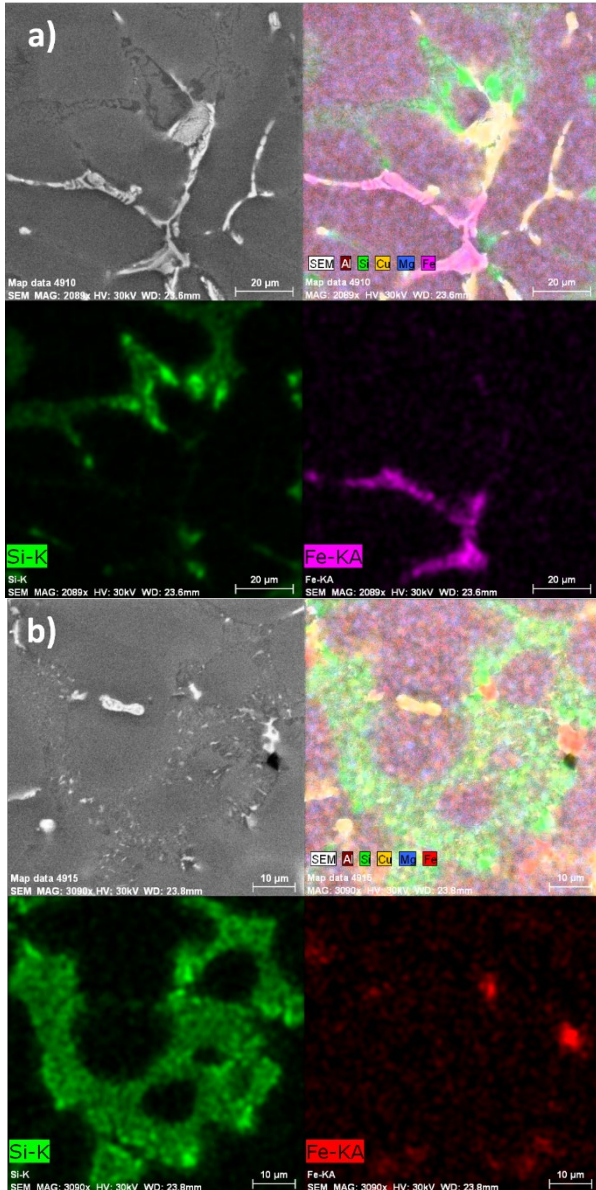


Fig. 4. Fe-rich intermetallic phases with corresponding EDX mapping of chemical elements in AlSi5Cu2Mg alloy with nominal addition of: a) 0.10 wt.% Nb, b) 0.20 wt.% Nb, as-cast state, etch. H<sub>2</sub>SO<sub>4</sub>, SEM

### 3.3. Size and Area Fraction of Fe Phases

The size of the Fe phases was determined within the microstructural evaluation using the image analysis software of the Keyence VHX-7000 digital microscope. The evaluation focused on  $\beta$ -Al<sub>3</sub>FeSi phases. The presented values are averages of five measurements.

Table 3. Size of  $\beta$ -Al<sub>3</sub>FeSi phases [ $\mu$ m]

AlSi5Cu2Mg g	AlSi5Cu2Mg g + 0.10 wt.% Nb	AlSi5Cu2Mg g + 0.15 wt.% Nb	AlSi5Cu2Mg g + 0.20 wt.% Nb
22.59	19.06	14.89	11.64

Based on the results, Nb addition was shown to positively affect the size of  $\beta$ -Al<sub>3</sub>FeSi phases. The size of the studied Fe phases decreased with increasing Nb addition. The alloy with 0.20 wt.% nominal Nb addition exhibited the most significant size reduction, nearly 48% compared to the alloy without Nb addition.

Fig. 5 shows the graph of the area fraction of the Fe phases ( $\alpha$ - and  $\beta$ -Fe phases) depending on the Nb addition. According to the literature, selecting the optimal Mn/Fe ratio suppresses the precipitation of brittle  $\beta$ -Fe phases [10]. Some studies state that, at an Mn/Fe ratio of 0.5, the transformation of  $\beta$ -Al<sub>3</sub>FeSi into more spheroidized Fe phases is favourable [11]. However, in experimental alloys with Mn/Fe  $\approx$  0.055, optimal suppression of  $\beta$ -Al<sub>3</sub>FeSi crystallization did not occur. Based on the results, the addition of AlNb10 reduces the fraction of the undesirable  $\beta$ -Al<sub>3</sub>FeSi phases.

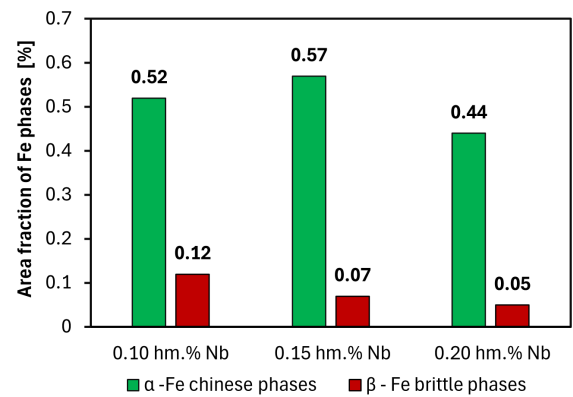


Fig. 5. Area fraction of Fe phases [%]

Nb tends to form Nb-rich intermetallic phases that act as potential nucleation sites for the primary  $\alpha$ -(Al) phase. In their study, Narducci et al. [12] state that heterogeneous nucleation occurs through the wetting of nucleation site surfaces by the liquid phase, which reduces the energy barrier for forming new crystals. Consequently, the growth of dendrites of the primary  $\alpha$ -(Al) phase is limited, resulting in refinement of the aluminum alloy microstructure. They further state that the favorable inoculating effect of Nb positively influences the morphology of  $\beta$ -Al<sub>3</sub>FeSi phases. In fine-grained aluminum alloys, grain boundaries act as

barriers that prevent Fe atoms from clustering into large, coarse needles. Due to the refining effect, Fe tends to precipitate in the form of smaller, more isolated, and more spheroidal Fe-rich phases. These phases are uniformly distributed in the matrix. The area fraction evaluation results of the Fe phases are consistent with the aforementioned study.

### 3.4. Analysis of DAS Index

A metallographic evaluation of the DAS index was performed on AlSi5Cu2Mg and Nb-added alloys using image analysis software from the Keyence VHX-7000 digital microscope. The DAS index values of the reference alloy and the Nb-added alloys are shown in Table 4. The presented values are averages from five DAS index measurements.

Table 4.  
DAS index of experimental alloys [ $\mu\text{m}$ ].

AlSi5Cu2Mg	AlSi5Cu2Mg	AlSi5Cu2Mg	AlSi5Cu2Mg
g	g + 0.10 wt.% Nb	g + 0.15 wt.% Nb	g + 0.20 wt.% Nb
32.41	28.44	26.71	21.55

Sentence correction: The alloy without Nb addition had a DAS index value of 32.41  $\mu\text{m}$ . Due to the addition of Nb, a decrease in the DAS index values of the studied alloys was observed. The addition of nominal 0.10, 0.15, or 0.20 wt.% Nb decreased the DAS index by 12%, 18%, or 33%, respectively, compared to the alloy without Nb addition.

A decrease in the DAS index value indicates a finer structure has been achieved. The observed refinement of the dendritic structure due to the addition of niobium (Nb) is consistent with the study by Bolzoni et al. [13], which reports that Nb addition leads to the formation of thermodynamically stable Al<sub>3</sub>Nb particles that simultaneously act as nucleation sites for the primary  $\alpha$ -(Al) phase. A key advantage of Nb over standard Ti-based inoculants is its high chemical stability in Al-Si melts. In Ti-inoculated alloys, Ti's high affinity for Si leads to TiAlSi-based phases that do not act as nucleation sites [5]. For this reason, the effectiveness of Ti inoculation in aluminum alloys depends on the wt.% Si. Nb-based particles do not show affinity to Si; this ensures the chemical integrity of their surface and keeps it active for heterogeneous nucleation. Therefore, it can be concluded that the inoculating effect of Nb is independent of Si content in aluminum alloys. [5, 13]

## 4. Conclusions

This paper investigated the effect of graded additions of Nb on the size and morphology of iron-bearing intermetallic phases, as well as its impact on the primary  $\alpha$ -(Al) phase and the overall microstructural evolution of the AlSi5Cu2Mg alloy. Based on the summarized results, the following conclusions can be drawn:

- The addition of Nb led to a positive refinement of the primary  $\alpha$ -(Al) phase. The refinement of the primary  $\alpha$ -(Al) phase was quantitatively confirmed by a decrease in the DAS index

value. The DAS index of the experimental alloys decreased with increasing Nb addition. The largest decrease in the DAS index, by approximately 33%, and consequently the most significant refinement of the primary  $\alpha$ -(Al) phase, was observed in the alloy with the nominal addition of 0.20 wt.% Nb. The refining effect of Nb is attributed to the formation of thermally stable Al<sub>3</sub>Nb particles, which serve as heterogeneous nucleation sites for the primary  $\alpha$ -(Al) phase.

- The inoculating effect of Nb is not dependent on the Si content in the aluminum alloy.
- As a result of the refining effect of Nb, a more homogeneous distribution of Si in the interdendritic regions is achieved.
- A correlation between the Nb addition and the morphology and size of the Fe-rich phases was detected. The addition of Nb led to the fragmentation of long, brittle, needle-like  $\beta$ -Al<sub>3</sub>FeSi phases into smaller and more compact particles. The average size of these  $\beta$ -phases in the alloy with a nominal addition of 0.20 wt.% Nb was approximately 11.64  $\mu\text{m}$ . Compared to the AlSi5Cu2Mg alloy without Nb addition, a reduction in the size of the detrimental  $\beta$ -phases of almost 48% was achieved. The size reduction and the decrease in the volume fraction of  $\beta$ -phases in the matrix of the alloys with Nb addition is a consequence of Nb's refining effect. The refinement of the primary  $\alpha$ -(Al) phase prevents the formation of coarse and brittle needle-like  $\beta$ -phases.

Based on the achieved results, Nb appears to be a potential inoculant for large-scale structural castings, which simultaneously neutralizes the harmful effect of impurities such as Fe. The refinement of the microstructure and the fragmentation of brittle Fe-phase needles leads not only to higher ductility but, above all, to a significant increase in fatigue life and resistance to crack propagation. A more homogeneous and finer microstructure also improves melt fluidity and reduces the susceptibility to microporosity formation in complex sections of the casting. This increases not only the overall strength but also the reliability and safety of these critical components. Future research will focus on evaluating the effect of Nb addition on the casting, mechanical, and physical properties of the AlSi5Cu2Mg aluminum alloy in the as-cast and T6 heat-treated conditions.

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