RESEARCH ARTICLE

Influence of NbC Addition in Aluminium Alloy A380 on Microstructure at Semi-Solid Processing Temperature

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Semi-solid processing is a promising technique used to fabricate parts. The globular structure is the key in this process, a characteristic achieved with the partial melting material to temperatures between solidus and liquidus. In this sense, the aim of this research is to evaluate the microstructure quality of the aluminium matrix composite (AMC) reinforced with NbC, after semi-solid treatment. Micron-sized NbC powder was employed as reinforcement to fabricate a composite through the stir casting method. Furthermore, was used an Al-5Ti-1B alloy grain refinement. Globularization heat treatment at 562 °C, with a holding time of the 90s, was realized. An optical microscope under conventional and polarized light and a scanning electron microscope (SEM) allows the microstructure analyses. The dendritic cell size (DCS), grain size (GS), shape factor (SF), and rheo quality index (RQI) were used to analyse the morphology and microstructure. The results show a general reduction of GS and DCS and more globular microstructure parameters with a higher amount of NbC, when compared with non-reinforced alloy.

Introduction

Semi-solid metal (SSM) is a promising forming technique supported by a non-dendritic microstructure [**1**]. The slurry with thixotropic behaviour is used to produce parts with low porosity and better mechanical properties. Two essential routes can process semi-solid conditions: thixoforming and rheoforming. Independent of the route, the main characteristic of the process is the presence of a microstructure consisting of spheroids (globules) of solid in a liquid matrix when the material is in a semi-solid state [**2**]. Many technologies have been developed to process SSM. Chemical grain refinement is a well-known technique in the casting industry. This technique is generally used in Al-based alloys to produce the appropriate globular microstructure [**3**]. In the processing of composite materials in a semi-solid state, the use of the refinement technique is not very common. Mainly they were formulated based on shear [**4**]. This work then focuses on exploring grain refinement as part of the methodology to process the composite at a semi-solid temperature.

The heterogeneous nucleation agent causes suppression of dendritic growth and produces equiaxed fine grain size microstructures, with better distribution of α-Al phases in the cast product or solidified ingot. Further, the heat treatment of partial remelting promotes the globularization of α-Al particles [**5**].

In SSM production, the chemical grain refinement method is highlighted as an alternative route, with the possibility of using various refining alloys [**2**]. Al–5Ti–1B ternary master alloys have been commonly used in aluminium alloys [**6**].

The properties of the semi-solid slurries make them excellent starting materials for processing metal matrix composite [**1**]. A while ago, Moon *et al.* [**4**] produced an Al-Si composite reinforced with SiC and analysed the effects after the semi-solid process. They found a possible relationship between the presence of the ceramic and solidified metal particles toward more perfect spheroids. The study of microstructural evolution in partial remelting is one of the keys to understanding the properties of the composites. Because this has a considerable impact on the final semi-solid microstructure. However, studies focused on composites are scarce compared to matrix alloys as demonstrated in the work of Zhang *et al.* [**7**]. Some studies on partial remelting have been published in the last few years, focusing on matrices reinforced with SiC **[8]**, Mg_2Si [**7**], and TiB_2 [**9**]. Nevertheless, there is a gap in studying the incorporation of NbC in A380 aluminium alloy modified by Sr to change the eutectic Al-Si morphology [**10**], and Mg to improve wettability [**11**]. NbC has great potential to use as a composite reinforcement [**12**]. It is a highly brittle ceramic with a high melting point and hardness. In addition, it can be applied as wear protection [**13**].

In this sense, this work seeks to elucidate the knowledge gap to understand the semi-solid processing of composites with NbC reinforcement in aluminium matrix. To study the application of aluminum matrix reinforced with NbC, some decisive parameters need to be analysed. Shape factor (SF) and dendritic cell size (DCS) are some of the main to evaluate and measure the quality of the semi-solid microstructure. Rheo Quality Index (RQI) is another important parameter to understand the complexity of the material, with the relation between microstructure and morphology [**14**].

Here in this research was analysed the microstructure evolution of the new A380 matrix reinforced with NbC after partial remelting. An analysis of the changes provided by the insertion of ceramic particles aiming to improve the quality of the semi-solid material.

Experimental

This section will show the materials, procedures to made and evaluate the composite.

Preparation of the composite

The commercial hypoeutectic A380 aluminium matrix alloy, was melted at 750 °C in a 35 KW GRION induction furnace. Four amounts of micron-sized NbC powder were added to the matrix. The powder was supplied by the Brazilian Metallurgy and Mining Company (CBMM). The casting conditions can be visualized in **Table 1**.

The stir casting process was carried out with the addition of Sr as a eutectic phase modifier, Mg as a wettability agent, commercial scorifier, and desgasefier. Al-5Ti-1B alloy also was added as a chemical grain refiner. Preheated NbC particles at 200 °C were added to molten, and then stirred for 5 min at 400 rpm. The composite was poured into a 200 °C preheated cylindrical steel mould.

Table 1. Casting conditions (wt. %).

	Al0 NbC	Al5 NbC	Al10 NbC	Al15 NbC
A380	98.075	93.075	88.075	83.075
Sr	0.025	0.025	0.025	0.025
Mg	1	1	1	1
Al5Ti1B	0.2	0.2	02	0.2
Desgasefier	0.3	0.3	0.3	0.3
Scorifier	0.4	0.4	0.4	0.4
NbC	0	5	10	15

Globularization heat treatment

Previous work of Ferreira *et al.* [**15**] from the research group obtained 562 °C as a temperature from a 60 % solid fraction. It was from differential scanning calorimetry (DSC), resulting from integrating the area under the curve and Scheil equation.

Characterization of composites

Samples for microstructure analysis were prepared by the standard metallurgical technique, followed by etching in a Barker's reagent.The metallographic examination was made using an optical microscope (OM) and scanning electron microscope (SEM) images. The polarized images were obtained using polarizing filters to obtain the colour images of their grains, so that grains with the same crystal orientation presented similar colouring, thus facilitating their identification and characterization [**16**].

Grain size (GS) was obtained from OM images with polarizing filters and dendritic cell size (DCS) from black and white images. According to ASTM E112 [**17**], Heyn's intercept method was used to measure DCS and GS. X-ray diffraction (XRD) analysis was performed on as-cast samples using a Shimadzu XRD-7000 diffractometer. The shape factor (SF) was obtained according to **Equation 1**, in which A_α is the area of the single rated entity, and P_α the perimeter.

$$SF = \frac{4\pi A_\alpha}{P_\alpha^2} \tag{1}$$

The rheo quality index (RQI) was calculated according to **Equation 2** [14].

$$RQI = \frac{DCS}{GS \cdot SF} \tag{2}$$

Results and discussion

In this section, the results about microstructure changes after partial remelting and quality parameters are analysed.

As-cast microstructure

The initial microstructure of the composite obtained through the stir casting method was a typical of hypoeutectic Al-Si alloy. The presence of the phases was detected by the XRD. The majority phase was primary α-Al, (PDF number 85-1327). The Si in the Al-Si fibrous eutectic phase is due to the addition of the Sr modifier [**18**] (PDF number 75-589) [**19**]. A Fe-based phase was founded, an acicular intermetallic β-Fe phase, distributed in all conditions [**15**], (PDF number 49-1499) [**20**]. The presence of Cu in the alloy provided a formation of the intermetallic phase θ-Cu [**21**], (PDF number 25-12) [**22**]. NbC peaks are the second quantity in the Al15NbC, decreasing with the reduction in the percentage inserted in the respective alloys (PDF number 38-1364) [**23**].

Microstructure after partial remelting

Fig. 1 shows the SEM images from samples after partial remelting. At the bottom of the images are shown the respective EDS maps of Nb of the three reinforcement conditions to identify and prove the presence of NbC in the composite. The microstructure of all samples shows a primary α-Al, surrounded by the eutectic Al-Si (liquid in the processing temperatures). Most of the NbC particles were localized in the eutectic phase. It observed a lower quantity of big clusters. Moreover, the formation of more globular structures is observed with an increase in the reinforcement fraction. In all conditions, a lower quantity of entrapped liquid was observed, its presence is undesirable [**24**].

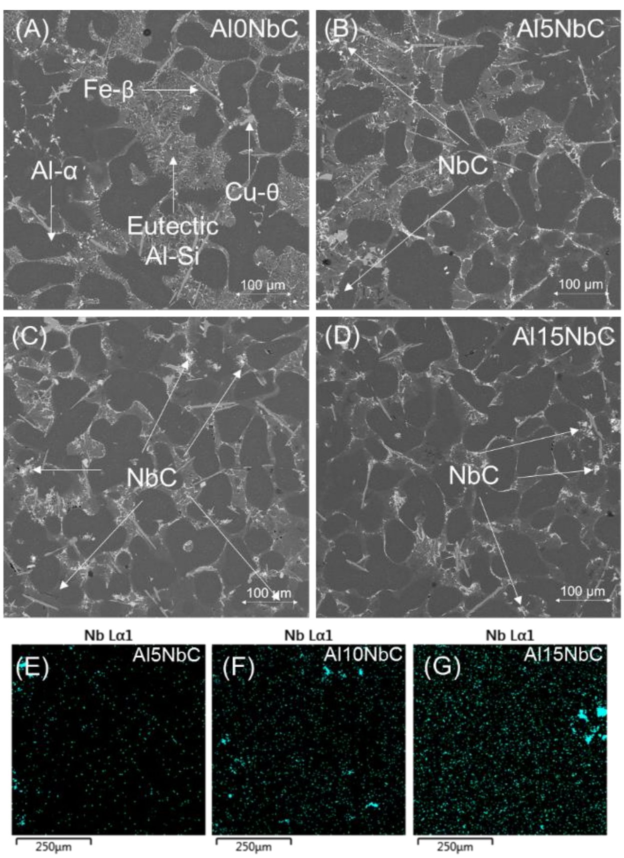

Fig. 1. SEM images after partial remelting samples showing the NbC particles in (A) Al0NbC (B) Al5NbC (C) Al10NbC (D) Al15NbC, and EDS maps of Nb from (E) Al5NbC, (F) Al10NbC, (G) Al15NbC

In **Table 2** the information on DCS, GS, SF, and RQI is shown. There is a decrease in DCS with NbC incorporation. However, no significant difference can be noted with the reinforcement content increase. In GS analysis, the respective addition of 5 wt. % and 10 wt. % of NbC promotes a reduction in average values of 40.8 % and 57.1 % in relation to unreinforced alloy (Al0NbC). There is no additional reduction in the GS with the Al15NbC condition. The differences were amortized by standard deviation. In their work Dantas *et al.* [**25**], related that the breakdown of dendritic arms and subsequent dissolution of thinner particles, were caused by Oswald ripening as the main mechanisms of GS reduction in partial remelting. This mechanism is one of the responsible for the microstructure after remelting, presenting a more globular morphology, with an increase in the NbC fraction.Which depends on the holding time [**14**]. In DCS/GS, closer to “1,” a lower complexity structure was expected, minimizing the interaction between solid particles when the material has been subjected to thixoforming [**16**]. The major fraction of NbC promoted an increase in this relation. On the other hand, a comparison between the SF values reveals an improvement trend only with 10 wt. %. NbC. However, there is no significant difference between the Al5NbC and Al15NbC conditions. Dantas *et al.* [**25**] also verified that adding Al_2O_3 particles did not promote an alteration in the shape factor of the A356 aluminium matrix grains.

Table 2. Microstructural parameters of A380/NbC after partial remelting at 562 ° C (average ± standard error).

Sample	DCS (µm)	GS (µm)	DCS/ GS	SF	RQI
Al0NbC	101.48 ± 8.46	176.21 ±13.24	0.57	0.45 ± 0.02	0.26 ± 0.05
Al5NbC	78.43 ± 9.15	125.20 ± 4.01	0.63	0.43 ± 0.01	0.27 ± 0.05
Al10NbC	76.57 ± 4.49	112.86 ± 4.77	0.68	0.51 ± 0.02	0.35 ± 0.02
Al15NbC	74.28 ± 2.87	106.03 ± 9.69	0.70	0.46 ± 0.04	0.32 ± 0.03

Comparative microstructure globularization supported by RQI is a critical analysis [**26**]. RQI index shows an improvement with the addition of 5 wt. % and 10 wt. % NbC. In the Al15NbC sample, a decrease in RQI was obtained. This factor linked to GS reveals that grain size reduction is essential in getting a quality semi-solid material. Salleh *et al.* [**20**] in their work show a similar result, emphasizing the importance of grain refining in obtaining semi-solid material

Conclusion

The addition of NbC particles improves the quality of the semi-solid A380 alloy. The reduced quantity of entrapped liquid reveals the quality of the material to apply this process, bearing in mind that its presence is undesirable. DCS was decreased with the addition of the reinforcement, but there was no significant difference in the amount of NbC. NbC promotes a 57.1 % reduction in GS with 10 wt. % nevertheless, no extra reduction was observed in the 15 wt.% of reinforcement. SF and RQI show the best values with 10 wt. % of reinforcement. Based on the results, it can be concluded that the holding time tested is adequate to promote the globularization of the aluminum matrix composite with NbC reinforcement. The proposed methodology allows for reducing the time of the thixoforming process. Thus, the present methodology is promising for use in the thixoforming or rheoforming process of composite materials. It is suggested, then, a deepening in the evaluation of different solid fractions in the microstructure and the properties of thixoformed composites.

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Conflicts of interest

There are no conflicts to declare.

Keywords

Aluminium matrix composite, NbC, semi solid treatment; microstructure.

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Authors biography

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Graphical abstract

A380 aluminium alloy with NbC was utilized to make a composite through stir casting. The heat treatment of globularization was performed at 562 °C for the 90s, which corresponds to 60 % of the solid fraction. Metallographic analysis of DCS, SF, GS and RQI index was carried out to evaluate the quality and changes promoted by NbC addition. The results show an improvement in RQI with an increase in the NbC content.