

**RESEARCH**

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

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**ABSTRACT**

Semi-solid processing is a promising technique used to fabricate parts, minimize manufacturing steps post preparation, and reduce casting defects. The globular structure is the key in this process, a characteristic achieved with the partial remelting of material to temperatures between solidus and liquids. 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 with 0 wt. %, 5 wt. %, 10 wt. %, and 15 wt. % 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 parameters with a higher amount of NbC. The AMC with NbC shows more globular microstructure when compared with non-reinforced alloy.

**KEYWORDS**

Aluminium matrix composite, niobium carbide, stir casting; globular.

**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 [2]. 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 [3]. Many technologies have been developed and applied worldwide to produce this appropriate globular microstructure. Chemical grain refinement is a well-known technique in the casting industry. This route is usually used in Al-based alloys [4]. The heterogeneous nucleation agent

equiaxed, fine grain size microstructures with better distribution of  $\alpha$ -Al phase in the cast product or solidified ingot. Further, the heat treatment of partial remelting promotes the globularization of  $\alpha$ -Al particles [5].

In the SSM production, this method is highlighted as an alternative route, with the possibility of using various refining alloys [3]. Al-5Ti-1B ternary master alloys have been commonly used in aluminium alloys [6]. In their work, [7] analysed the influence of Al-5Ti-1B on A357 aluminium alloy for producing semi-solid material in different pouring temperatures. The authors found that the addition of the grain refiner promoted more nuclei for solidification. Dendritic morphology was changed to rosette and globular after the globularization heat treatment.

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causes suppression of dendritic growth and produces

The properties of the semi-solid slurries made them excellent starting materials for processing metal matrix composite [1]. A while ago, [8] 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 [9].

Some studies on partial remelting have been published in the last years, focusing on matrices reinforced with SiC [10], Mg<sub>2</sub>Si [9], and TiB<sub>2</sub> [11]. 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 [12] and Mg to improve wettability [13]. NbC has great potential to use as a composite reinforcement [14]. It is a highly hard ceramic with a high melting point and hardness. Also, it can be applied as wear protection [15]. Some parameters like shape factor (SF) and dendritic cell size (DCS) are crucial 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 relation between microstructure and morphology [16]. In addition, it can be expected that a modification in that parameters resulted in some critical information to verify further the microstructural evolution process [9].

In this sense, this research available 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.

### Materials

Commercial hypoeutectic A380 aluminium alloy was selected as a matrix material. The chemical composition of the alloy can be visualized in **Table 1**. Micron-sized NbC powder was supplied from the Brazilian Metallurgy and Mining Company (CBMM).

**Table 1.** Chemical composition A380 aluminium alloy.

Element	Wt. %	Element	Wt. %
Si	8.159	Cr	0.017
Fe	0.786	Pb	0.183
Cu	3.281	Ti	0.082
Mn	0.139	Sr	0.001
Mg	0.068	Ca	0.002
Zn	2.961	Al	Balance
Ni	0.197		

### Preparation of the composite

The alloy was melted at 750 °C in a 35 KW GRION induction furnace. Four conditions of reinforcement addition were made, named Al0NbC unreinforced, Al5NbC, Al10NbC, and Al15NbC, respective with 5 wt. %, 10 wt. % and 15 wt. % of NbC.

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

### Globularization heat treatment

Previous work [17] 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. For isothermal globularization heat treatment, the samples were machined to diameter 27.5 mm and 25 mm high from casting ingots. A 2 mm × 20 mm hole was made to insert the K-thermocouple and monitor the temperature. Globularization heat treatment was realized in the same induction furnace, but now coupled to an induction coil. An optimum 50 °C heating rate was utilized to achieve the final temperature of 562 °C [19]. The samples were held at this temperature for the 90s and finally rapidly cooled in water to freeze the microstructure.

### Characterization of composites

After the heat treatment, the samples were cut longitudinally in Buehler Isomet 4000 in the middle. A quarter cut was made in the circular samples to mount and prepared metallographically for the microstructural characterization.

The samples were sanded with 220, 400, 800, and 1200 mesh sandpaper. 3 polishing steps were utilized, diamond suspension of 3 µm and 0.25 µm, and final vibratory polishing with 0.04 µm silica suspension in Buehler Vibromet 2. After polishing, the samples that were not etched were analysed. In the sequence, the composites were etched using an electrolytic solution of fluoboric acid 6%, applying a voltage of 25 V for about 150 seconds under moderate and constant agitation.

Metallographic analysis was performed in the optical microscope Axios Imager.A2m and scanning electron microscope (SEM) Tescan Vega 3. 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 [18].

Grain size (GS) was obtained from an optical microscope with polarizing filters and dendritic cell size (DCS) from standard images. According to ASTM E112 [20], Heyn intercept method was used. Five images from

different localizations for each sample were utilized, and a simple average was performed as a final value. The hardness of the composites was evaluated by the Rockwell B test using a Rockwell test machine (PANTEC RBS) according to ASTM E18 [21]. X-ray diffraction (XRD) analysis was performed on as-cast samples, using a Shimadzu, XRD-7000 diffractometer (40 kV) with Cu K $\alpha$  radiation (wavelength of 1.54060 nm)

The shape factor (SF) was obtained according to **Equation 1**, in which  $A_{\alpha}$  was the area of the single rated entity and  $P_{\alpha 2}$  the perimeter.

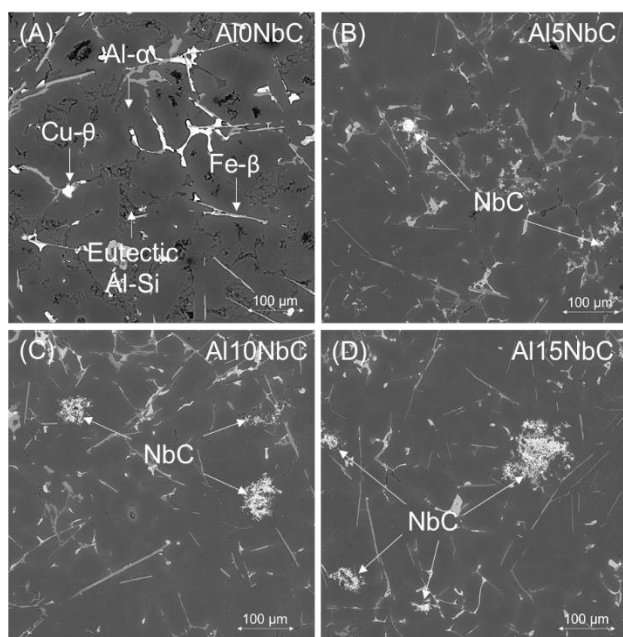
$$SF = \frac{4\pi A_{\alpha}}{P_{\alpha}^2} \quad (1)$$

The rheo quality index (RQI) was calculated according to **Equation 2** [16].

$$RQI = \frac{DCS}{GS \cdot SF} \quad (2)$$

## RESULTS AND DISCUSSION

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



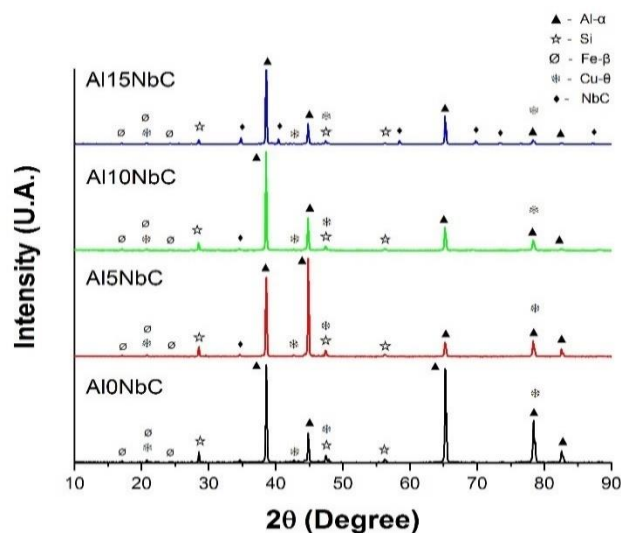
**Fig. 1.** SEM images of as-cast samples showing the NbC particles in (A) Al10NbC (B) Al15NbC (C) Al110NbC (D) Al115NbC.

### As-cast microstructure

The initial microstructure of the composite obtained through the stir casting method can be visualized in **Fig. 1**. A typical microstructure of hypoeutectic Al-Si alloy was found. The Al-Si eutectic surrounds most of the primary  $\alpha$ -Al phase. According to the Heyn intercept method, the

grain size values are: Al10NbC  $232.82 \pm 4.97 \mu\text{m}$ , Al15NbC  $173.78 \pm 6.98 \mu\text{m}$ , Al110NbC  $142.72 \pm 2.14 \mu\text{m}$ , Al115NbC  $143.46 \pm 3.6 \mu\text{m}$ . It can be observed that the largest fraction of NbC interacts with  $\alpha$ -Al and eutectic Al-Si, promoting a reduction in its size. This phenomenon is possibly associated with the nucleating effect provided by the interaction of carbides with Ti and the aluminium matrix [28]. On the other hand, it has been shown that during cooling, the eutectic Al-Si phase can change its morphology from plate to fibrous as a consequence of the addition of Sr as a modifier [22]. Also, the morphology of  $\alpha$ -Al presents itself in irregular shapes associated with the fact that NbC particles can restrict growth in some directions. Some pores were observed within the  $\alpha$ -Al phase. A Fe-based phase was founded, acicular  $\beta$ -Fe phase was distributed in all conditions [17]. The presence of Cu in the alloy provided a formation of the intermetallic phase  $\theta$ -Cu [23].

The presence of the phases was also detected by the XRD, showed in the **Fig. 2**.



**Fig. 2.** X-ray diffraction spectrum of composites.

The peaks of  $\alpha$ -Al are the major presence in the XRD pattern in the base alloy (PDF number 85-1327). 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) [25]. Other phases were also confirmed: Si (PDF number 75-589) [26],  $\theta$ -Cu (PDF number 25-12) [24], and  $\beta$ -Fe (PDF number 49-1499) [27].

### Hardness

**Fig. 3** is shown the hardness variation of the NbC fraction in the composite at the as-cast condition and after partial remelting. The general values show an increase in the hardness with the NbC fraction increase.

An increase in the hardness could be attributed to some mechanisms: reinforcements acting as a barrier to the movement of dislocations [28], the Orowan mechanism [29], and the Hall-Petch relation [30]. In the as-cast

condition, the addition of 5 wt. %, 10 wt. % leads to a respective hardness increase of 47 % and 55 %. The same analysis increases by 23 % and 37 % in the remelting process, both compared to unreinforced alloy. The standard deviation analysis showed no significant changes between 10 wt. % and 15 wt. % for both conditions. The uniform distribution of hard ceramic particles has an essential effect on composite hardness [22]. The incorporation of the NbC particles provides restriction to the dislocation movement. With a major level of reinforcement, the distance between the particles decreases, which leads to an increase in the required tension for dislocation movement [31]. As a result, the agglomeration has a significant problem. The particles coming together reduce the number of obstacles to dislocation movement and increase the probability of pore formation. Compared to the as-cast condition, the increase in the hardness after partial remelting could be linked to better particle distribution. The partial remelting could provide a better distribution of NbC particles and increase the hardening mechanism, as shown in Fig. 5.

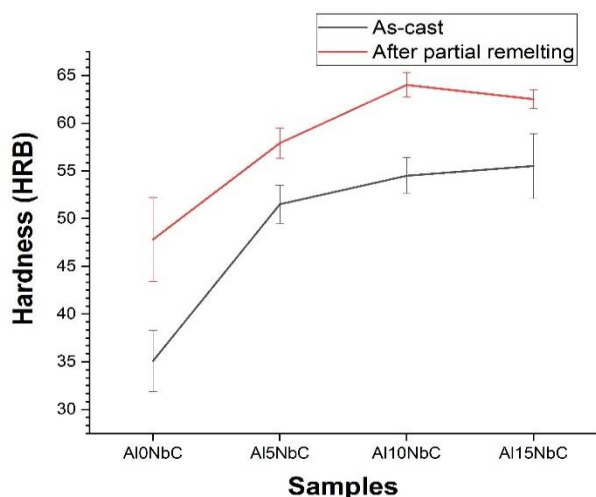


Fig. 3. Rockwell B hardness test results of A380/NbC, in as-cast condition and after partial remelting at 562 °C.

An increase in hardness also is obtained in the research of [11]. The Mg–11Al–0.5Zn alloy was reinforced with TiB<sub>2</sub> and analyzed on partial remelting at 510 °C and 545 °C, obtaining the highest hardness value of the matrix alloy at the lowest temperature and opposite situation in the case of the composite. In their work, [10] obtained a similar behavior in the Mg alloy AZ91HP reinforced with SiC. The authors also observed that increasing isothermal holding time has a deleterious effect on the hardness.

### Solid fraction

The relation between the  $\alpha$ -Al and the solid fraction, was used to verify the solid fraction calculated by the DSC analysis and Scheil equation [32]. SEM images were processed using the open software Image J, to determine the solid fraction by area comparison.

Fig. 4 shows the analysis result concerning the globularized samples. It is noticed that all conditions present close solid fractions, around 60 % - 64 %. A liquid fraction of around 40% was judged to be the most appropriate [34]. Higher solid fractions limit the benefits of the thixoforming process, while solid fractions below this value led to shape distortions before the forming step and create die entry problems [2].

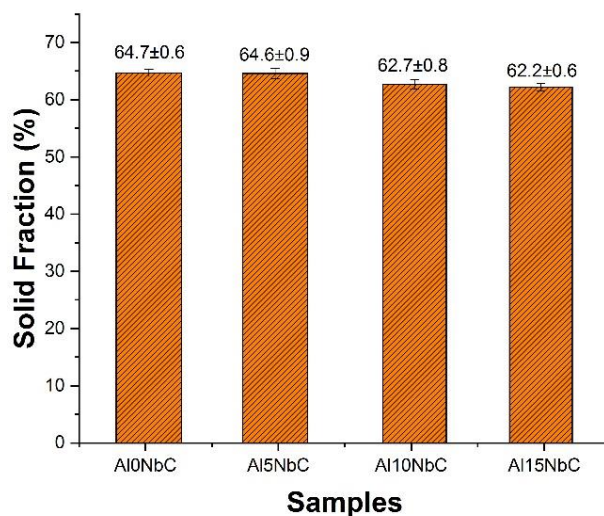


Fig. 4. Solid fraction results from image analysis after partial remelting at 562 °C.

### Microstructure after partial remelting

Fig. 5 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. Most of the NbC particles were localized in the eutectic phase. Also, it was observed a lower quantity of clusters. This fact could be one of the responsible for the hardness increase. The microstructure of all samples shows a primary  $\alpha$ -Al, surrounded by the eutectic Al-Si (liquid in the processing temperatures) and the presence of acicular formations of the  $\beta$ -Fe phase, present most of the time within the eutectic phase, where most of the  $\theta$ -Cu phase was also found. The  $\alpha$ -Al morphology was changed to a more globular and refined when compared to the as-cast condition. This reduction in size and morphology alteration could be one of the responsible for the hardness increase. With a more refined and globular microstructure, mainly due to the Hall-Petch relation, the hardness increases after partial remelting [30]. As explained by [16], the microstructure after partial remelting tends to be more globular by the Ostwald ripening and coarsening, both depending on the holding time. The work of [9] proposed a microstructural evolution based on steps of initial rapid coarsening, structural separation, the spheroidization, and the final coarsening owing to the mergence and subsequent Ostwald ripening. Also, the globular and refined

microstructure of  $\alpha$ -Al after partial remelting could be linked to a more regular distribution of NbC. A reduction in the number of clusters in the microstructure also allows the phase growth to be more regular in all directions when compared to the as-cast condition.

The Al-Si eutectic phase presents more evidence after partial remelting. The efficient distribution of NbC after partial remelting, generally present in the eutectic phase, also helps the Al-Si be more refined and present in the microstructure. Another fact is the increase in the cooling rate in the sample after partial remelting. Rapidly cooling in water may be another reason for this change. The thermal properties of Al and Si are different, so in high cooling rates, Al probably will solidify faster than Si [33].

In Fig. 6, it is observed that more globular structures were formed with an increase in the reinforcement fraction. The higher NbC fraction also promoted a reduction in DCS (conventional micrographs (A) and (B)) and GS (micrographs under polarized light (C) and (D)), which adds to the better distribution of the NbC after heat treatment. This fact could be linked to the function of the NbC particles, acting as nucleation sites for the grains [28]. In this way, when more particles are dispersed in the material, more grains are formed, and because they are in the same space, they have a smaller size than the alloy without NbC. The greater number of clusters in Al15NbC may be responsible for the slight difference with an Al10NbC condition.

In all conditions, a lower quantity of entrapped liquid was observed. The presence of entrapped liquid is undesirable for thixoforming. The liquid trapped inside the  $\alpha$ -Al phase formations hinders the lubrication process, essential in the thixotropic behavior during thixoforming [35].

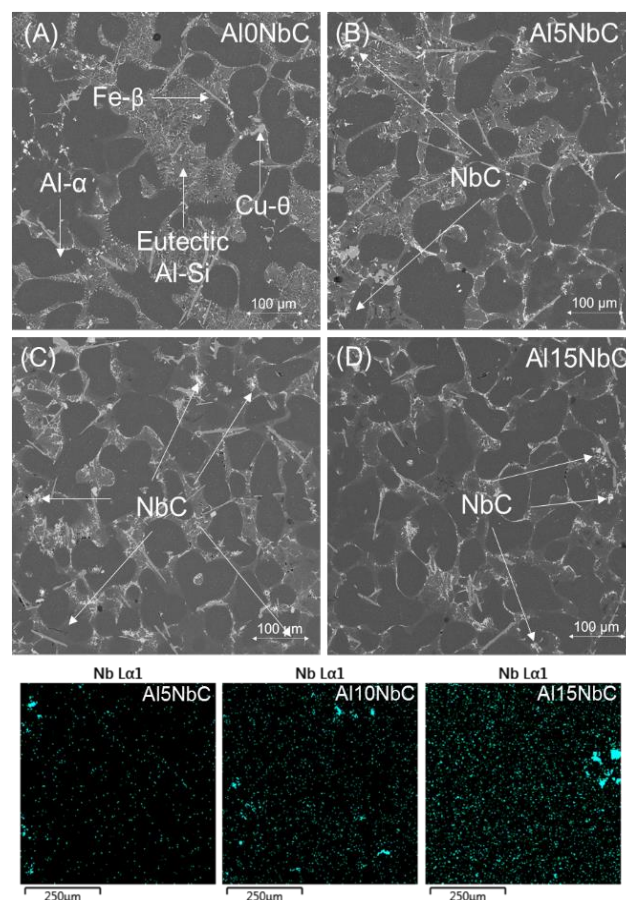
From Table 2 the information on DCS, GS, SF, and RQI is shown.

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

Sample	DCS ( $\mu\text{m}$ )	GS ( $\mu\text{m}$ )	DCS/GS	SF	RQI
Al0NbC	101.48 $\pm$ 8.46	176.21 $\pm$ 13.24	0.57	0.45 $\pm$ 0.02	0.26 $\pm$ 0.05
	78.43 $\pm$ 9.15	125.20 $\pm$ 4.01		0.63	0.43 $\pm$ 0.01
Al10NbC	76.57 $\pm$ 4.49	112.86 $\pm$ 4.77	0.68	0.51 $\pm$ 0.02	0.35 $\pm$ 0.02
	74.28 $\pm$ 2.87	106.03 $\pm$ 9.69		0.70	0.46 $\pm$ 0.04

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 %. There is no additional reduction in the GS with the Al15NbC condition. The differences were amortized by standard deviation. In their work [36], related breakdown

of dendritic arms and subsequent dissolution of thinner particles caused by Oswald ripening are the main mechanisms of GS reduction in partial remelting. In DCS/GS, closer to “1,” a lower complexity structure was expected, minimizing the interaction between solid particles, which is good to fluidity [18]. The major fraction of NbC promoted an increase in this relation. On the other hand, 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. In their research, [36] also verified that the addition of Al<sub>2</sub>O<sub>3</sub> did not promote an alteration in the SF on the A356 aluminum matrix.



**Fig. 5.** SEM images after partial remelting samples showing the NbC particles in (A) Al0NbC (B) Al5NbC (C) Al10NbC (D) Al15NbC.

Comparative microstructure globularization supported by RQI is a critical analysis [38]. RQI index shows an increasing improvement with the addition of 5 wt. % and 10 wt. % NbC. Although, in the Al15NbC sample, a decrease in RQI was obtained. This factor linked to GS reveals that grain size reduction and morphology is essential in obtaining a quality semi-solid material [27]. In their work show a similar result, emphasizing the importance of grain refining in obtaining semi-solid material.

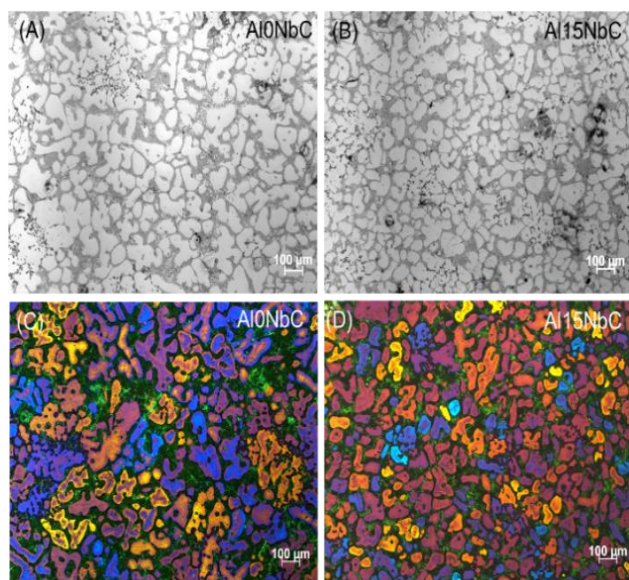


Fig. 6. Simple Optical images from normal conditions Al0NbC (A), Al15NbC (B) and polarized light microscopy Al0NbC (C), Al15NbC (D).

## CONCLUSION

The addition of NbC particles improves the quality of the semi-solid A380 matrix. An average increase in hardness was obtained with NbC addition after partial remelting. DCS was reduced 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. % NbC nevertheless, no extra reduction was observed in the 15 wt.% of reinforcement. SF and RQI show the best values with 10% of reinforcement. Hence, the present investigation particle and matrix are promising for potential use in the thixoforming or rheoforming process.





## ACKNOWLEDGEMENTS

The authors would like to thank the Graduate program (DIRPPG) from Technological Federal University of Paraná (UTFPR). CNPq/Araucaria Foundation for aid in promotion. The CBMM to supply the NbC powder. José Roberto de Campos for building the stir casting system and, Coordination for the Improvement of Higher Education Personnel - Brazil (CAPES) - Financing Code 001, for providing financial support for this study.

## CONFLICTS OF INTEREST

There are no conflicts to declare.

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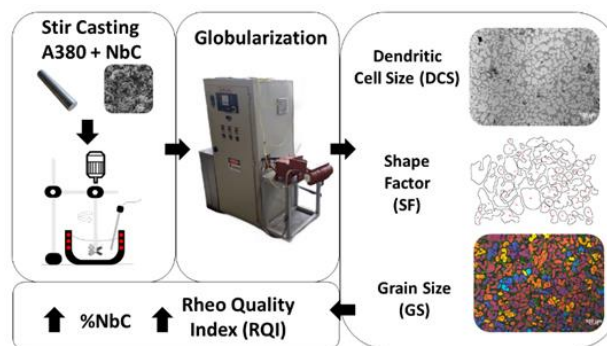
## SUPPORTING INFORMATION

**Table 3.** Casting conditions (wt. %).

	Al0 NbC	Al5 NbC	Al10 NbC	Al15 NbC
<b>A380</b>	98.075	93.075	88.075	83.075
<b>Sr</b>	0.025	0.025	0.025	0.025
<b>Mg</b>	1	1	1	1
<b>Al5Ti1B</b>	0.2	0.2	0.2	0.2
<b>Degassing</b>	0.3	0.3	0.3	0.3
<b>Slag agent</b>	0.4	0.4	0.4	0.4
<b>NbC</b>	0	5	10	15

## 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.



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