



Development of Al–Nb–B master alloys using Nb and KBF₄ Powders



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ARTICLE INFO

Article history:

Received 18 September 2014

Revised 5 March 2015

Accepted 7 March 2015

Available online 11 March 2015

Keywords:

Aluminium alloys

Grain refinement

Heterogeneous nucleation

Solidification microstructure

Nb–B inoculation

ABSTRACT

We recently reported that the combined employment of niobium and boron (i.e. Nb-based intermetallics formed in the melt by the addition of powders), instead of niobium or boron individually, is a highly effective way to refine the grain size of Al–Si alloys without the inconvenience of the poisoning effect typical of commercial Al–Ti–B master alloys. In this work the progress concerning the development of Al–xNb–yB master alloys, which are much more suitable for its use in aluminium foundries, is reported and discussed. Precisely, a first approach to produce Al–xNb–yB master alloys as well as its characterisation by means of EDS mapping and TEM is presented. The study is completed by testing the effectiveness of the produced Al–xNb–yB master alloys on pure aluminium and binary Al–10Si alloy as well as commercial hypoeutectic and near-eutectic Al–Si alloys. It is found that the approach employed to produce the Al–xNb–yB master alloys is suitable because the size of the primary α -Al dendrites is significantly reduced in each of the case investigated.

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1. Introduction

Aluminium (Al) cast alloys are common materials used to fabricate engineering components for the transportation industries, especially the automotive, due to the easiness of their shaping by means of casting processes and the intrinsic reduction of weight of structural components that their employment involve. Moreover, the stringent requirement for the reduction of fuel consumption and, therefore, exhausted gas pollution as well as the design of structural components with lower weight and enhanced mechanical performances are pushing the automotive industry towards the employment of a greater amount of light metals, and Al will definitively play a major role. It is well known that a way to improve static and dynamic mechanical properties of metals is by achieving fine grain structures [1–3]. In the Al industry the practise of grain refinement is well established [1–3] and it generally carried out by the addition of master alloys available in the market which were developed on the ternary Al–Ti–B system [4–15], where different theories to explain the mechanism governing their refinement have been proposed [2,3,10]. In this way, an equiaxed as-cast structure in Al direct chill (DC) casting ingots is achieved which makes the material more suitable for its subsequent downstream processing. This leads to semi-finished products with improved mechanical properties and less cold and hot

cracking phenomenon. In the case of Al cast alloys, where silicon (Si) content is generally higher than 4–5 wt.%, the refinement by means of master alloys based on the Al–Ti–X phase diagram (where X = B and C [16–19]) is drastically inhibited due to the formation of titanium silicides. These intermetallics form from the reaction of titanium (Ti) present in the grain refiner and the Si of the alloy. This phenomenon, which is identified as poisoning effect, has been studied in details by many researchers [5,7,20]. Despite this fact, the grain refinement of Al–Si cast alloys is commonly carried out, if done, using commercial Al–Ti–B master alloys due to the lack of effective alternative. We recently reported that efficient and reliable grain refinement of hypo-eutectic and near-eutectic Al–Si cast alloys can successfully be done by employing Nb and B [21–23]. Precisely, the addition of 0.1 wt.% of Nb powder and 0.1 wt.% of B through KBF₄ flux leads to the formation of niobium borides (NbB₂) and niobium aluminides (Al₃Nb) which are responsible for the grain refinement of Al–Si cast alloys (i.e. Nb–B inoculation). Specifically, NbB₂ has a lower lattice mismatch with the Face-Centred Cubic (F.C.C.) structure of Al with respect to TiB₂ whilst Al₃Nb has the same lattice mismatch of Al₃Ti with Al. The greatest difference is, nonetheless, the higher chemical stability of the niobium silicides with respect to titanium silicides. The former intermetallics form at higher temperatures than those generally employed to cast Al–Si alloys. Consequently, Nb–B inoculation should not present any poisoning effect. It is worth mentioning that the addition of a grain refinement in the form of powders at industrial level is not of practical implementation and it is why

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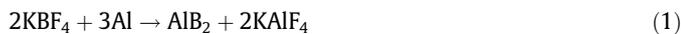
Table 1
Chemical composition of the commercial Al–Si alloys used in the study.

Alloy	Element (wt.%)							
	Al	Si	Fe	Cu	Mn	Mg	Zn	Ti
Pure Al	Balance	0.02	0.08	<0.001	0.001	<0.001	0.002	0.006
Al–10Si	Balance	10.02	0.08	–	0.01	–	0.02	–
LM25 (A356)	Balance	6.5–7.5	0.2	0.2	0.1	0.2–0.4	0.1	0.2
LM6 (A413)	Balance	10.0–13.0	0.6	0.1	0.5	0.1	0.1	0.2

they are generally provided in the form of master alloys. Therefore, the aim of this work is to report and discuss the development of Al–xNb–yB master alloys focusing on the characterisation of the phenomena that take place during their production. The produced Al–xNb–yB master alloys are used to introduce Nb–B inoculants in different Al-based materials (i.e. pure Al, binary Al–Si alloy as well as Al–Si commercial alloys) in order to assess their grain refining potency.

2. Experimental procedure

The materials used to carry out the study about the development of Al–xNb–yB master alloys were pure Al, Nb powder (<45 µm) and potassium tetrafluoroborate (KBF₄). The employment of salt flux like the KBF₄ is a common industrial practise for the production of master alloys (such as the ones based on the Al–Ti–B ternary system). Salt fluxes promote the in-situ formation of borides (i.e. AlB₂ and TiB₂) and titanium aluminide (Al₃Ti) particles (intermetallics) in the Al matrix which constitute the master alloy. Once the master alloy is added to the casting Al alloy, these intermetallic particles (inoculants) act as heterogeneous sites for the nucleation of primary α-Al grains. In the case of the development of the Al–xNb–yB master alloys, the employment of the KBF₄ flux has the advantage that when it reacts with Al generates a significant amount of energy (due to the fact that the reaction is highly exothermic) for a short period of time which locally increases the temperature and helps to dissolve the Nb powder particles. Specifically, the chemical reaction taking place during the mixing of pure Al, pure Nb and KBF₄ are:



Three Al–xNb–yB master alloys were produced following the same fabrication route: Al–4Nb–1B, Al–2Nb–1B and Al–1Nb–1B. It is worth mentioning that the real content of Nb and B of the Al–xNb–yB master alloys is thought to be lower because some Nb powder gets oxidised during its addition at high temperature and B recovery from KBF₄ flux at lab scale is not very efficient. That is why the compositions are labelled as “targeted” addition of Nb throughout the whole manuscript. The correct amount of pure Al was placed inside a clay-bonded graphite crucible and melted at 850 °C and left to homogenise during 2 h inside an electric furnace. Subsequently, the Nb powder and the KBF₄ flux were added meanwhile manually stirring the melt with an alumina rod. Stirring was repeated every 15 min during the following 2 h. Finally, the slag present on the surface of the molten metal was removed and the master alloy poured into a pre-heated steel mould. The complete dissolution and reaction of the Nb particles with Al was checked by means of superconductivity experiments.

In particular, the magnetic moment was measured as a function of the temperature under a magnetic field of 100 Oe applied by means of a SQUID magnetometer. The cast master alloys were characterised and, therefore, optical micrographs (Axioscope A1 optical microscope), SEM-EDS semi-quantitative chemical analyses (Zeiss Supra 35VP FEG) and TEM (JEOL 2200F) study of the nucleant intermetallic particles were considered. The refining potency and effectiveness of Nb–B inoculation via Al–xNb–yB master alloy addition was tested on different materials like commercially pure Al, binary Al–10Si alloy and commercial Al–Si alloys (i.e. LM25 (A356) and LM6 (A413) alloys). As it can be seen from the chemical composition of the commercial Al–Si alloys shown in Table 1, LM25 is a hypo-eutectic alloy whilst LM6 is a near-eutectic alloy.

Different steel moulds were employed to cast the materials without and with the addition of the Al–xNb–yB master alloys. Specifically, a cone-shaped steel mould (cooling rate ~0.5 °C/s), a 30 mm cylindrical steel mould (cooling rate ~2 °C/s) and the TP-1 mould of the Aluminium Association (cooling rate ~3.5 °C/s) were employed. The classical metallographic route of SiC papers grinding plus OPS polishing was used to prepare the samples for their microstructural analysis. In the case of the determination of the grain size, the polished samples were also anodised passing a current of approximately 10 V/1 A and using a tetrafluoroboric acid (HBF₄) solution. Image analysis to measure the grain size of the cast specimens was carried by means of an Axioscope A1 optical microscope equipped with a dedicated program.

3. Results

3.1. Characterisation of the Al–xNb–yB master alloys

Fig. 1 shows the results of the magnetic moment tests carried out to confirm the complete reaction of Nb with the Al matrix by detection of the superconductivity transformation.

It is well-known that Nb is characterised by a transition (T_c) in its superconductive behaviour at 9.2 K. From the results of the magnetic moment measurements shown in Fig. 1a superconducting transition temperature was detected at 9.2 K when testing the elemental Nb powder. After the combined addition of Nb and potassium tetrafluoroborate powders to Al, the Al–xNb–yB master alloys have not shown the typical transition behaviour to the superconductive state of Nb. This indicates and confirms that Nb completely transforms into Nb-based compounds and it is not present as pure elements in the master alloys anymore.

Fig. 2 shows a representative micrograph of the Al–xNb–yB master alloys produced by mixing pure Al with Nb powder and KBF₄ flux along with the EDS elemental mapping results showing the distribution of the elements that constitute the master alloy.

As it can be seen from the analysis of the micrograph of the Al–xNb–yB master alloys (Fig. 2a), the materials is mainly constituted by the Al matrix and some uniformly dispersed particles are present. The elemental mapping reveals that, as expected, Al is the main constituent (Fig. 2b), Nb is concentrated in many different particles whose distribution is rather uniform (Fig. 2c) and B is

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