Journal of Alloys and Compounds 580 (2013) 232–238

Contents lists available at SciVerse ScienceDirect

Journal of Alloys and Compounds

journal homepage: www.elsevier.com/locate/jalcom

A Taguchi optimisation for production of Al–B master alloys using boron oxide

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ARTICLE INFO

Article history:
Received 14 March 2013
Received in revised form 14 May 2013
Accepted 16 May 2013
Available online 23 May 2013

Keywords:
Boron oxide
Aluminium boride
Al–B master alloys
Taguchi method

ABSTRACT

Al–B master alloys have been produced by liquid state reaction between aluminium and boron oxide in liquid aluminium. Taguchi design method has been employed to examine the effects of four process parameters of holding temperature, holding time, cooling rate and matrix type on the extent of boron dissolved and size distribution of the resulting AlB_2 intermetallic flake structure. In the experiments, melting, casting, solidification, metallography, optical microscope, scanning electron microscope (SEM) and wet chemical analysis techniques have been used. Results showed that maximum 2.14 wt.% boron has been dissolved in the aluminium through direct addition of boron oxide (B_2O_3). It is concluded that the cooling rate is the most effective factor on the size of AlB_2 particles.

1. Introduction

Al–B master alloys have been used mainly for grain refinement of aluminum alloys in Al–Ti–B form. Because AlB_2 particles introduced from Al–B master alloys are thought to act as nucleating substrates for aluminium alloys [1]. Al–B master alloys were also known to be effective alloys in the production of electrical conductive grade aluminiums to remove transition metals impurities, such as Ti, V, Cr and Zn [1–8]. Recently, it has been shown that Al/AlB_2 type composites are being produced with Al–B master alloys, because of the potential of reinforcement phase for high mechanical properties, such as high aspect ratio, homogeneous distribution and excellent interfacial bond with the matrix [9–14].

The Al–B system is shown in Fig. 1. According to the phase diagram, a peritectic reaction ($L + AlB_{12} \leftrightarrow AlB_2$) takes place at 980 °C [15]. AlB_{12} boride structure is the first phase to form in liquid Al–B, during subsequent cooling, AlB_{12} boride structure transforms to AlB_2 boride structure at the peritectic reaction temperature [16–19]. Therefore, stable AlB_2 boride crystals can be produced in the final aluminium matrix during cooling of an Al–B liquid. AlB_2 and AlB_{12} boride phases are reported to have an hp3 structure, which hexagonal and complex tetragonal crystal structure, respectively. The lattice parameters of AlB_2 is a = 0.3006 nm and c = 0.3252 nm, whereas AlB_{12} has a = 1.0161 nm and b = 1.4238 nm [20].

Deppisch and co-workers [21] reported that boride phase type and shape are highly dependent on the cooling rate from liquid (L) to the $L + AlB_{12}$. AlB_{12} phase is cuboidal shaped which is formed with slow cooling processes while the AlB_2 phase is flake shaped formed with high cooling processes. The width of AlB_2 flakes varies between 10 and 2000 μm, and thickness between 0.3 and 6 μm, depending on the cooling rate and alloying elements [21,22].

Although Al–B master alloys are produced by using KBF_4, it can also be produced by using inexpensive boron salts such as boron oxide or borax ($Na_3B_4O_7$) [23,24]. Because the Gibbs free energy of aluminum oxide is lower than that for boron oxide (B_2O_3), chemical reactions occurs at the interface between molten aluminum and boron oxide at 800 °C [25]. The reaction with boron oxide can be represented as:

$$2Al + B_2O_3 = \alpha Al_2O_3 + 2[B]\ \text{dissolved}, \quad \Delta G_{298^\circ} = -416.9\ \text{kJ/mol},\ \Delta H_{298^\circ} = -402.7\ \text{kJ/mol}. \tag{1}$$

As boron atoms go into solution in the molten aluminium, aluminium oxide covers the top of melt as slag. Since density of boron oxide is lower than aluminium (1.844 g/cm^3 and 2.69 g/cm^3 respectively) it floats on the liquid aluminium and hence the reaction takes place at the top of the crucible.

Past studies on the fabrication of Al–B master alloys by adding boron oxide (B_2O_3) are very limited [23,24]. In addition, there is no accessible research works which include the studies on amount of boron and size (in terms of width and thickness) of the boride particles in the Al–B master alloys which are produced by using boron oxide. Savas and co-workers reported that the amount of boron in the Al–B master alloys, produced by liquid reaction of the aluminium with boron oxide (B_2O_3) at 1400 °C, was 1.3 wt.%. AlB_2 flake

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0925-8388/$ - see front matter
http://dx.doi.org/10.1016/j.jallcom.2013.05.112

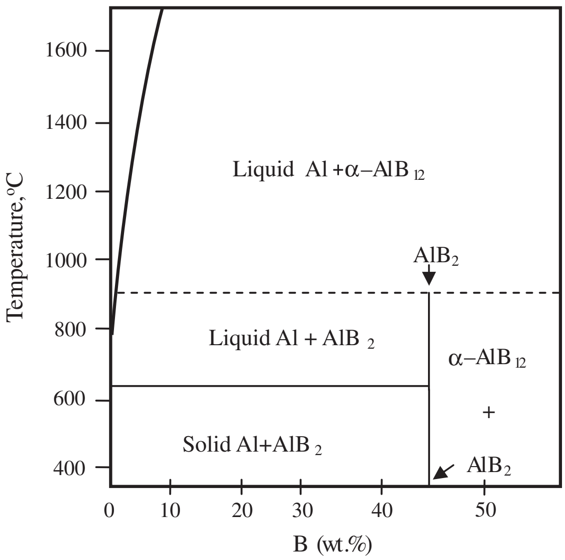

Fig. 1. Al–B phase diagram [15].

widths were varied between 10 μm and 420 μm, the thickness was varied approximately between 0.5 μm and 4.15 μm [22].

Past works on aluminium borides have generally dealt with their grain refining effects on casting aluminium alloys. Thus, studies on their sizes and shapes are limited. This study has been aimed to examine the effects of process parameters on the size of aluminium boride phases in Al–B alloys. An experimental design has been set to optimize some important parameters affecting the amount of dissolved boron and the final boride sizes and their shapes within the Al matrix.

2. Experimental Procedure

2.1. Materials

This study aims at Al–B or Al–Si–B solution have been prepared by synthesising boron oxide (B_2O_3) directly in liquid aluminium at 1300 or 1400 °C with 30 or 90 min of holding time, then after, in order to form AlB_2 from reacting boron with molten Al, the solution is cooled to room temperature with fester or lower od cooling rate. Taguchi method has been applied to examine the effects of parameters which holding time, cooling rate, alloying elements on the size of borides and the amount of boron in the Al–B master alloys.

In the experiments two different alloys were used. These are a commercially pure aluminium (99.98% Al) and Al7Si0.3 Mg (A356) alloy. Their chemical compositions were analysed by a Foundry-Master Pro atomic absorption spectrometer and are given in Table 1. Ingots of 100 gr were cut and placed in a crucible with 15 gr of boron oxide (B_2O_3). Before being added to aluminium, the boron oxide was heated to 400 °C for 15 min to remove any water constituents followed by milling.

The mixture of boron oxide and aluminium was heated in an induction coil with a steel crucible as explained in a previous study [22]. To obtain two different cooling rates as slow and fast, two different crucible geometries were used which are 75 × 25 × 20 mm and 75 × 50 × 10 mm. Each crucible was placed in the middle of the induction coil as shown in Fig. 2. The inner faces of the crucible was coated with boron nitride (BN) based coating to prevent any reaction between the molten aluminum alloys and steel.

Since boron cannot be quantified by Energy Dispersive Spectrometer (EDS), measurement of total weight per cent of boron in the Al–B master alloys was carried out by a wet chemical analysis method, as explained elsewhere [22].

Table 1
Chemical composition of the matrix alloys.

Alloys type	Elements (wt%)							
	Si	Fe	Cu	Mn	Mg	Cr	B	Al
Pure Al	0.13	0.29	0.00	0.00	0.00	0.002	0.00	Balance
A356	5.93	0.82	0.12	0.24	0.04	0.00	0.03	Balance

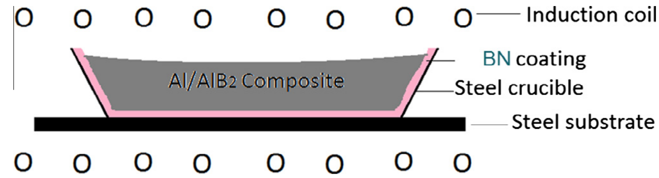

Fig. 2. Schematic view of the setup for melting process.

Small samples (5 × 5 × 5 mm) were extracted from the Al–B master alloys. For the microstructure analysis the samples were ground up to 1200 grid by using SiC paper followed by polishing, using 0.2 μm diamond paste. For detailed evaluation of the boride structure, the samples of Al–B master alloys were deep etched using HCl solution followed by an examination with JEOL JSM 6060LV scanning electron microscope (SEM).

2.2. Taguchi's experimental design

Taguchi's method is a well-known and reliable tool for the design of a high-quality system without increasing the cost. Taguchi's method is being used for understanding of the individual and combined effects of design parameters. In this study, experiments were designed to apply the Taguchi's methods to establish the effects, the optimal combination and the interaction effects of the process parameters on the amount of boron and the size of boride particles in the Al–B master alloys. The process parameters involve alloying elements, holding temperature of the liquid alloy, holding time and cooling rate. The four process parameters and their levels are given in Table 2.

An L8 orthogonal array table has been employed in the design of the experiments which includes only 8 trials. At the end of each trial, the average size of the boride phase and the amount of boron in the Al–B master alloys were measured. The measurements were carried out at least in three different regions of each sample. A signal-to-noise (S/N) ratio was calculated by using the average values of the measurements. The larger-the-better quality characteristics was considered since the performance was measured in terms of amount of boron, a property which is generally expected to be as high as possible as represented by Eq. (2).

$$S/N = -10\log\left(1/n\sum_{i=1}^{n}y^2\right) \quad (2)$$

where n is the number of measurements in a trial and y is the value of measurement in a trial. Analysis of variance statistical method (ANOVA) employs Eqs. (3)–(7) [26,27], was prepared to determine the effect and the optimal combination of the parameters by using the signal-to-noise (S/N) ratio values.

$$SS_T = \left[\sum_{I=1}^{N}(S/N)i^2\right] - \frac{T^2}{N} \quad (3)$$

$$SS_A = \left[\sum_{I=1}^{K_A}\left(\frac{Ai^2}{n_{A_i}}\right)\right] - \frac{T^2}{N} \quad (4)$$

$$\upsilon_{Total} = N - 1 \quad (5)$$

$$V_{Factor} = \frac{SS_{Factor}}{\upsilon_{Factor}} \quad (6)$$

$$F_{Factor} = \frac{V_{Factor}}{V_{Error}} \quad (7)$$

where SS_T is the sum of squares due to total variation, N is the total number of experiments, SS_A represents the sum of squares due to factor A, K_A is the number of levels for factor A. A_i stands for the sum of the total i^{th} level of the factor A, n_{Ai} is the number of samples for ith level of factor A. T is the sum of total (S/N) ratio of the experiments, υ_{total} is the degrees of freedom, V_{factor} is the variance of the factors, SS_{factor} represents the sum of squares of the factor and F_{factor} is the F ratio of the factor.

Table 2
Process parameters and their levels.

Factors	Symbols	Level 1	Level 2
Holding temperature	A	1300 °C	1400 °C
Holding time	B	30 min	90 min
Cooling rate	C	Slow	Fast
Matrix type	D	Al7Si0.3 Mg	Pure Al