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Processing and microstructural characterisation of AA 1070 and AA 6063 matrix B_4C_p reinforced composites

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ABSTRACT

Wetting problem between B_4C and liquid Al at lower temperatures has to be overcomed for promised mechanical and physical properties, such as high hardness, high wear resistance and stiffness. In the present work, AA 1070 and AA 6063 matrix B_4C_p reinforced composites were produced by casting route at 850 °C and titanium-containing flux (K_2TiF_6) was used to overcome the wetting problem between B_4C and liquid aluminium metal. AA 6063/B_4C_p composite samples were then subjected to solution treatment. The microstructure of matrix/reinforcement interfaces of both as-cast and heat treated samples with or without Ti additions were investigated with SEM studies. The reaction layer was also characterized with EDS analysis and X-ray mapping. It was found from the microstructural observations by high resolution field emission gun SEM (FEG-SEM) that the wetting improved by the formation of very thin TiC and TiB_2 reaction layers.

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1. Introduction

Metal matrix composites (MMCs) have been developed to respond the demand for lighter materials with high specific strength, stiffness and wear resistance [1,2]. Aluminium is favoured as matrix material in MMCs because of its low density, easy fabricability and good engineering properties [3]. Particulate reinforced aluminium matrix composites (AMCs), especially hardenable aluminium alloys matrix, such as AA 2XXX, AA 6XXX and AA 7XXX groups are attractive MMC materials due to their strength, ductility and toughness as well as their ability to be processed by conventional methods. Therefore, AMCs have been applied successfully to structural components, especially in the automotive and aviation industries, and the number of applications is expected to increase with the development of low-cost processing methods [1,4].

AMCs can be reinforced with various oxides, carbides, nitrides and borides in particulate, whisker or fiber form such as SiC, Al_2O_3, B_4C, TiC, TiB_2, MgO, TiO_2, AlN, BN and Si_3N_4 [5–10]. While SiC and Al_2O_3 are common reinforcing materials in AMCs, limited research has been conducted on B_4C reinforced AMCs due to the higher cost of B_4C powders [3,11]. B_4C is an attractive reinforcement material because of its excellent chemical and thermal stability; most importantly, B_4C has lower density (2.52 g/cm^3) and higher hardness (Hv = 30 GPa) relative to Al_2O_3 and SiC [11–15].

Al–B_4C composites can be processed with low-cost casting routes [6,16,17]. But poor wetting between Al and B_4C below 1100 °C means that it is difficult to produce Al–B_4C composites by mixing particles into the liquid phase. In order to enhance the wettability of ceramics and improve their incorporation behaviour into Al melts, particles are often heat treated or coated [18]. Apart from wetting, controlling of the interphases occurring at the Al–B_4C interface is also important in the production of cast Al–B_4C composites. At least nine phases have been reported in the Al–B–C ternary system [19].

It has been reported that for a given liquid metal, transition metal carbides, borides and nitrides are better wetted than covalently and ironically bonded ceramics [20]. Titanium is one of the reactive metals that can be used to increase wettability in Al–B_4C system [18,21]. But there is very limited data related to the matrix/reinforcement interface of the Al–B_4C composites produced with Ti addition. Therefore, in the present work, AA 1070 and AA 6063 matrix, B_4C reinforced composites were processed through a casting route with addition of K_2TiF_6 flux to form a reaction layer containing TiC and TiB_2 at the interface to improve the wettability and interface was carried out in detail.

2. Experimental procedure

B_4C particles with an average particle size 32 μm were used as a reinforcement and AA 1070 and AA 6063 alloys were used as a matrix material in this study. In order to enhance the wettability of

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boron carbide powders and improve their incorporation behaviour into aluminium melts, potassium fluotitanate (K_2TiF_6) flux was used.

Ten percentage of (wt.) B_4C particulate reinforced AMCs were produced using 400 g of aluminium (AA 1070 and AA 6063) charges which were melted in a boron nitride coated graphite crucible utilizing induction furnace for each specimen. Mixture of B_4C particles and the same amount of K_2TiF_6 flux (with 0.1 Ti/B_4C ratio) were added into the melt within 4 min at 850 °C with mechanical stirring at 500 rpm for five minutes. Finally the melt was poured into a metal mould with 850 °C casting temperature.

The T6 heat treatment was performed to AA 6063 matrix composites by solution treatment at 510 °C for 24 h, followed by quenching in water, then aging at 180 °C for 4 h.

Metallographic samples were prepared using the diamond grinders and water based diamond and colloidal silica suspensions. As-polished microstructures were examined under the JEOL JSM 7000F field emission gun scanning electron microscope (FEG-SEM) and JEOL JSM-5410 LV scanning electron microscope (SEM) both equipped with EDS analysis.

3. Results and discussion

The aim of Ti addition in the casting of Al–B_4C composites is to form a reaction layer on the interface that contains titanium carbide (TiC) and titanium boride (TiB_2). When Ti is added in the form of K_2TiF_6, K and F contribute to remove the oxide film from the Al surface [6,18]. Wetting increased on K_2TiF_6 added AA 1070/B_4C samples with different boron carbide particle sizes (below and above 20 μm) and on the other hand, no trace of bonding or interaction was observed on the samples produced without Ti addition, as mentioned earlier [6,22].

Comparatively homogeneous particle distribution was seen on low magnification SEM images of AA 1070/B_4C and AA 6063/B_4C composites as shown in Fig. 1.

At least nine ternary and binary phases have been reported for the Al–B–C system. On the other hand, in Al–B_4C system, Al_3BC and AlB_2 interphases can be expected between 660 °C and 868 °C and detrimental Al_4C_3 phase starts to occur just below the 1000 °C [23–26]. Since the process performed under 900 °C in the present work, Al_4C_3 phase would not expected. The potential reactions that could take place in the Al–Ti–B_4C system are reported by Shen et al. [27] are given as follows:

$$\frac{3}{4}Al + \frac{1}{4}Ti \rightarrow \frac{1}{4}TiAl_3 \quad (1)$$

$$\frac{10}{13}Al + \frac{3}{13}B_4C \rightarrow \frac{6}{13}AlB_2 + \frac{1}{13}Al_4C_3 \quad (2)$$

$$\frac{3}{4}Ti + \frac{1}{4}B_4C \rightarrow \frac{1}{2}TiB_2 + \frac{1}{4}TiC \quad (3)$$

$$\frac{3}{4}TiAl_3 + \frac{1}{4}B_4C \rightarrow \frac{1}{2}TiB_2 + \frac{1}{4}TiC + \frac{9}{4}Al \quad (4)$$

$$\frac{1}{2}AlB_2 + \frac{1}{2}Ti \rightarrow \frac{1}{2}TiB_2 + \frac{1}{2}Al \quad (5)$$

$$\frac{1}{4}Al_4C_3 + \frac{3}{4}Ti \rightarrow \frac{3}{4}TiC + Al \quad (6)$$

Shen et al. reported the changes in the Gibbs free energy, ΔG, and the reaction enthalpy, ΔH in the Al–Ti–B_4C system (Fig. 2). As they reported, all the reactions are thermodynamically favorable ($\Delta G < 0$) and exothermic ($\Delta H < 0$). Since the reaction (3) has the lowest Gibbs free energy, ΔG, in our process temperatures, in the Al–Ti–B_4C system, the most favorable reaction products are TiB_2 and TiC [27]. From that point, in the Al–B_4C composite processed with Ti addition, a reaction layer that consists of TiB_2 and TiC can be expected on the matrix/reinforcement interface. That reaction layer also acts as a "reaction barrier" on the surface of B_4C particulates and limits the interfacial reactions between the B_4C and the aluminium matrix responsible for the production of the undesirable interphases [16]. X-ray maps taken from both AA 1070 and AA 6063 matrix composite specimens shows the Ti containing regions surrounding boron carbide particles as shown in Fig. 3.

It was found from the microstructural observations by high resolution field emission gun SEM (FEG-SEM) that a very thin (80–180 nm in thickness) reaction layers formed on the B_4C surfaces. The EDS analysis taken from the reaction layer confirmed that the reaction layer consists of TiB_2 and TiC. Fig. 4 shows the SEM image and matching Ti elemental map and Fig. 5 shows the high magnification FEG-SEM image of the matrix/reinforcement interface and the EDS analysis of the interface taken from AA 6063/B_4C (10% wt.) composite specimen. Same results observed for from AA 1070/B_4C (10% wt.) composite as reported elsewhere [22].

The hardness values increased from 48 to 68 HV_{100} from as-cast specimens to heat treated specimens. On the other hand, no changing was observed on the reaction layers after solution treatment or aging. Fig. 6 shows the SEM image and matching Ti elemental map and Fig. 7 shows the high magnification FEG-SEM image of the matrix/reinforcement interface and the EDS analysis of the interface taken from the heat treated AA 6063/B_4C composite specimen.

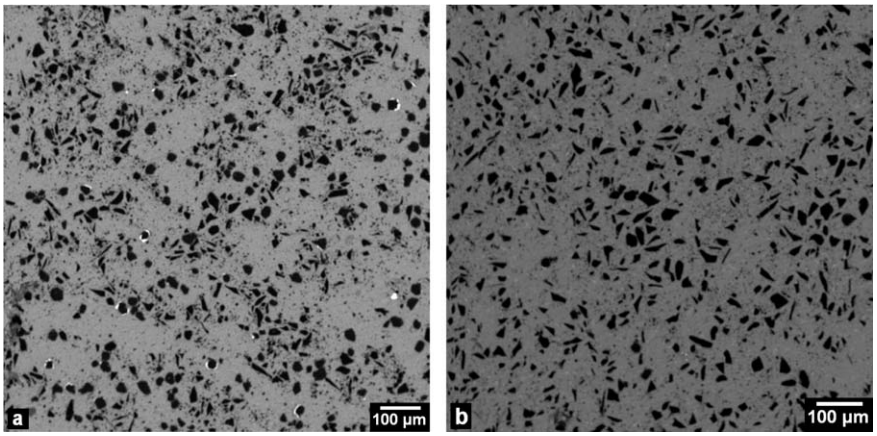


Fig. 1. BSI SEM images of: (a) AA 1070 matrix and (b) AA 6063 matrix 10% (wt.) B_4C reinforced composites.

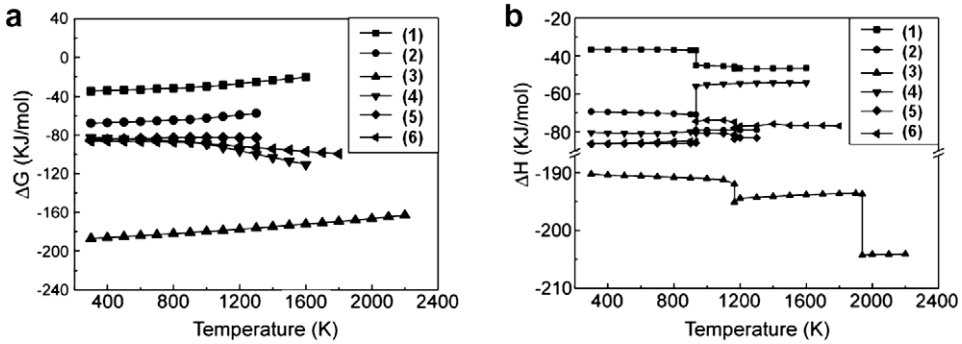


Fig. 2. Changes in: (a) Gibbs free energy, ΔG and (b) reaction enthalpy, ΔH, for reactions in the Al–Ti–B_4C system [27].

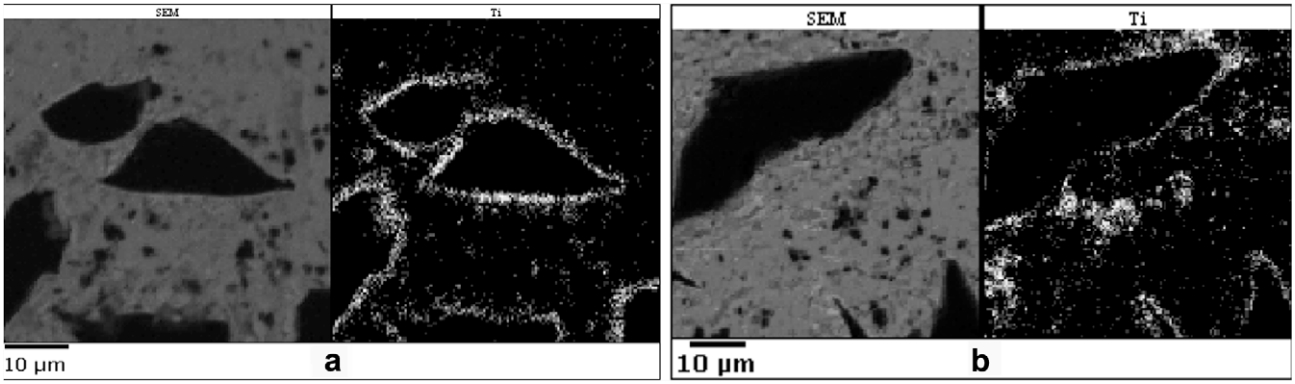


Fig. 3. SEM images and matching Ti elemental maps of: (a) 1070 matrix and (b) 6063 matrix B_4C reinforced composites.

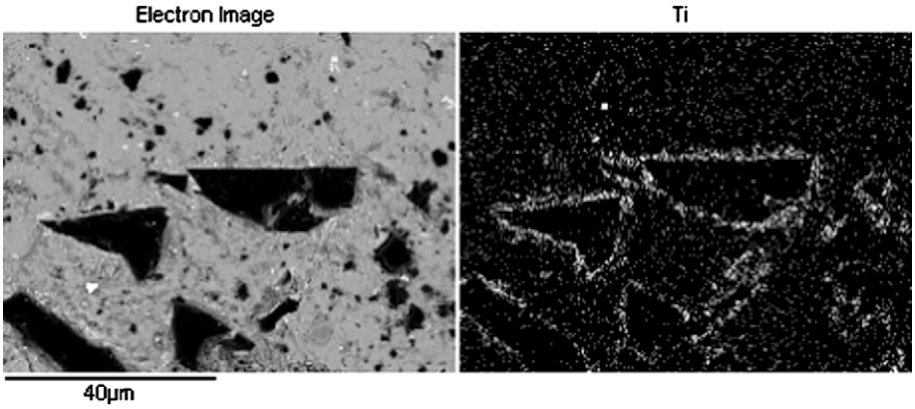


Fig. 4. BSI SEM image and matching Ti elemental map of AA 6063/B_4C composite.

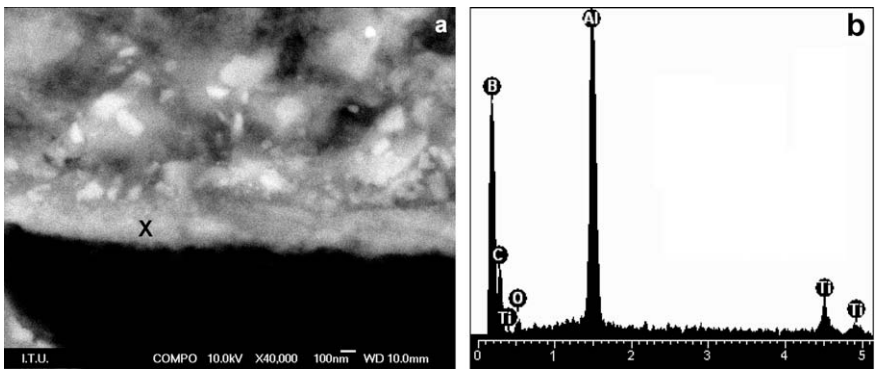


Fig. 5. (a) SEM image of the interface at AA 6063/B_4C composite and (b) EDS spectrum taken from "x" marked point in (a).

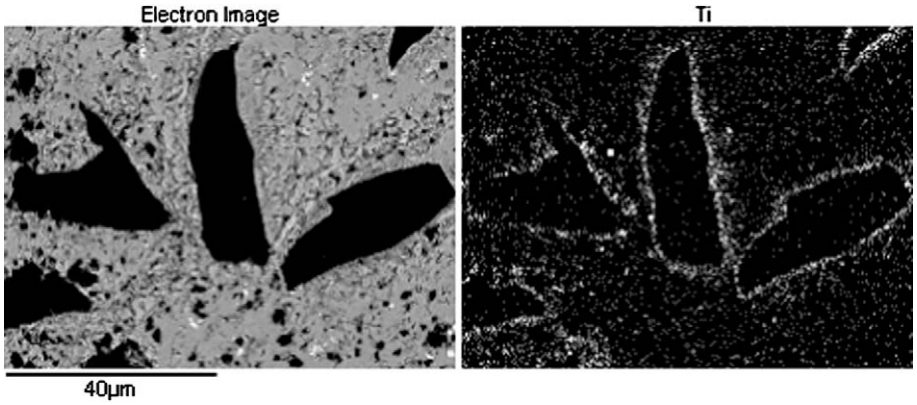


Fig. 6. BSI SEM image and matching Ti elemental map of AA 6063/B_4C composite (after aging).

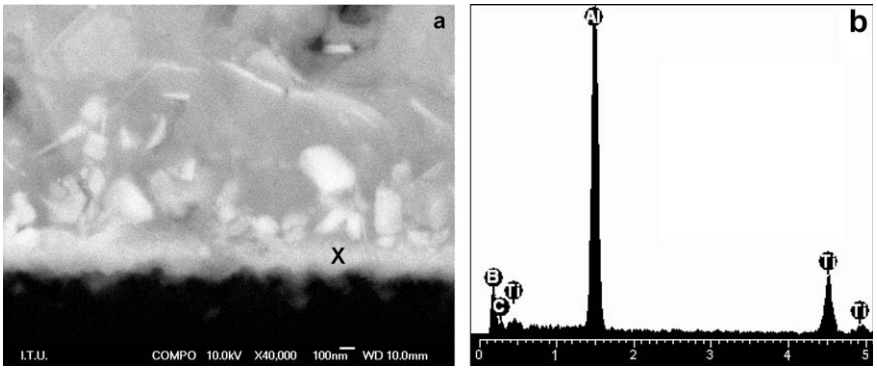


Fig. 7. (a) SEM image of the interface at AA 6063/B_4C composite and (b) EDS spectrum taken from "x" marked point in a (after aging).

Since the specimens were processed under open atmosphere with vortex casting, porosity values were high. Due to the porosity, desired mechanical properties could not be reached although wetting is improved. Therefore, future experiments will be carried out under vacuum conditions in order to reduce the porosity index.

4. Conclusions

Ten percentage of (wt.) B_4C reinforced AA 1070 and AA 6063 matrix composites are processed with casting route with addition of K_2TiF_6 flux to form a reaction layer contains TiC and TiB_2 at the interface for increasing wettability and interface bonding. AA 6063 matrix composites were subjected to T6 heat treatment and matrix/reinforcement interfaces characterised in both as-cast and heat treated composites. From this study the following can be concluded:

- Due to the poor wetting of B_4C particles by liquid aluminium, an effective bonding could not be formed on the matrix/reinforcement interface in Al–B_4C composites produced at relatively lower temperatures like 850 °C.
- The wetting improved by the formation of very thin (80–180 nm in thickness) TiC and TiB_2 reaction layers with addition of K_2TiF_6 flux.
- As to the forming of a reaction layer, it was seen that no significant differences between AA 1070 and both as-cast or heat treated conditions in AA 6063 matrix composites.

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