Effect of Ti addition on the wettability of Al-B₄C metal matrix composites

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In this study, the wettability of B₄C by molten Al, as well as Al-Ti alloys, with different Ti contents has been studied using the sessile-drop method. In general, the contact angle decreases with increasing temperature and time. The results showed that an appropriate amount of Ti addition improves the wettability of B₄C by Al. Analyses of the interfaces of solidified drops revealed that Al₃BC and AlB₂ particles form around the B₄C particles when they are in contact with pure Al. However, when Ti is added to Al, AlB₂ particles disappear and a new Ti-rich layer composed of TiB₂ particles forms around B₄C surfaces. In addition, the effect of Ti addition on the surface tension of aluminum has also been examined.

Keywords: Metal Matrix Composites; Al-B4C; Wettability; Contact angle; Interfacial microstructure; Surface tension, Scanning electron microscopy
1. Introduction

Metal matrix composites (MMCs) are made of one continuous phase (metal matrix) and a solid reinforcement which can be discontinuous (particle, whisker, short fiber or other) or continuous (monofilament or multifilament). The properties of the MMC depend on the properties of its components. This material group offers new alternatives when the properties of available materials do not meet the demands of a specific application. Aluminum is one of the major industries in Quebec and Canada. Compared to conventional aluminum materials, aluminum-based metal matrix composites have the advantage of a relatively low weight with improved mechanical properties such as better strength, stiffness, fatigue resistance, corrosion resistance, lower coefficient of thermal expansion, and higher thermal conductivity. These composites have applications in the automotive, aerospace and military industries, as well as in the production of high voltage electric cables, and in recreational products such as golf clubs, bicycle frames, etc. [1, 2].

Al-B₄C composites are made of reinforcing B₄C particles dispersed in Al matrix. As B₄C has a capacity to absorb radioactive neutrons, these composites are used in the fabrication of nuclear waste storage containers as a main neutron absorber component [3, 4]. During the preparation of Al-B₄C composites using the liquid mixing process, B₄C particles in the form of fine powder injected into and are mixed with liquid aluminum in the temperature range of 700 – 850°C. This is difficult because of the poor wettability between B₄C and Al [5, 6, 7]. Also, B₄C is not stable when in contact with liquid aluminum [8]. Interfacial reactions take place extensively and Al continues to react with B₄C until the reaction is complete. Chen [7] reported that the following reaction between B₄C and Al is thermodynamically possible:
10Al + 3B₄C → 6AlB₂ + Al₄C₃ \hspace{1cm} (1)

Later on, the following reaction and formation of Al₃BC and AlB₂ has been proposed [8, 9, 10]

9Al + 2B₄C → 2Al₃BC + 3AlB₂ \hspace{1cm} (2)

Interfacial reactions decrease the fluidity of the melt and deteriorate its mechanical and physical properties [10]. To prevent these adverse effects, Ti is added into Al during the liquid mixing process [3, 7]. The Ti forms a protective layer on the B₄C particles through an interfacial reaction which facilitates the incorporation of B₄C into the melt and prevents its further reaction with molten Al. The possible thermodynamic reaction suggested is [7]:

3Ti + B₄C → 2TiB₂ + TiC \hspace{1cm} (3)

The ΔG of the reactions with and without Ti (Equations (3) and (1)) is -638 and -335 kJ at 750°C, respectively. The reaction taking place in the presence of Ti has a lower ΔG; consequently, it is thermodynamically more favourable than the first reaction without the addition of Ti [7].

Recently, it is found in Al-Ti-B₄C system that secondary reaction phases Al₃BC and TiB₂ are produced at Al/B₄C interface through the following reaction [11, 12]:

6Al + 3Ti + 2B₄C → 2Al₃BC + 3TiB₂ \hspace{1cm} (4)
As previously explained, Ti limits the undesired interfacial reaction and facilitates the incorporation of B₄C into molten Al. The latter effect is attributed to the reduction of the interfacial tension of the liquid metal, and consequently, the improvement of the wettability of reinforcement particles (B₄C) by the Al matrix. However, there is no systematic study on the wetting of B₄C particles at the temperatures of interest (700 to 850°C) as used in the industry.

The wettability and reactivity of B₄C by molten Al have been studied by several researchers [5, 13-16]. Kennedy and Karantzalis [5] studied the incorporation of B₄C ceramic particles into molten aluminum and its relationship to contact angle data. The initial contact angle was reported to be 135° at 900°C but the experimental conditions were not specified in the publication.

Halverson et al [13] studied the wetting of hot pressed and polished B₄C plates by industrial grade pure aluminum between 900 and 1300°C under vacuum. The initial contact angle at 900°C was 145° and dropped to 110° at 1000°C. Naidich et al [14] reported a contact angle as high as 147° at 1000°C. However, it decreased sharply with increasing temperature and a value of 33° was obtained at 1150°C. Lin et al [16] found the strong temperature and time dependence of wettability of B₄C by molten Al, which was attributed to the interaction of Al with B₄C and the formation of various reaction products at the interface.

It is well known that the wetting studies are not easy to carry out with Al since Al oxidizes rapidly even if a trace amount of O₂ is present in the atmosphere within the temperature range used in the industry. Therefore, most of the research is carried out under vacuum or at very high temperatures where, according to the following reaction, the oxide layer on Al dissociates with the production of aluminum suboxides which are volatile [17]:
However, the conditions under which these contact angles were measured are not the same as those used in the industrial MMC production. Besides, none of those studies reported the effect allying elements on the wettability of B₄C by Al. Thus, there is a need for a wetting study to test the effect of Ti on the wettability of B₄C particles by Al within the temperature range of interest in the industry.

Wettability can be defined as the ability of a liquid to spread on a solid surface and indicates the extent of contact between the liquid and the solid. It is characterized by the contact angle (θ) which is the angle between the liquid-vapor interface and the solid, as shown in Figure 1(a). The contact angle at equilibrium is determined by the Young-Dupre equation [18]:

$$\gamma_{sv} = \gamma_{sl} + \gamma_{lv} \cos \theta$$

(6)

where $\gamma_{sv}$, $\gamma_{sl}$, $\gamma_{lv}$ are the interfacial tensions between solid and gas, solid and liquid, and liquid and gas in “N/m”, respectively. $\gamma_{lv}$ is also known as the surface tension (force per unit length or energy per unit area). If $\theta = 0^\circ$, the solid is completely wetted by the liquid. However, for all practical purposes, the system is said to be wetting if $\theta<90^\circ$ and non-wetting if $\theta>90^\circ$. The sessile-drop method is the most common method used in measuring contact angles at high temperatures [19].
Ideally, contact angles should be measured using solid substrates with smooth surfaces (plates). However, they are very frequently measured for beds of fine powders [20,21]. In certain applications such as composite preparation where powders are mixed with molten metal the wettability of the powder by the metal is crucial. Therefore, the powder bed measurements represent better the situation encountered during the industrial application. Although, the contact angle measured in this case is an apparent angle, it is still an invaluable tool to carry out comparative study of particle wettability.

Two methods are commonly used to determine the surface tension from the shape of a sessile-drop: the Basforth and Adams method [22] and the Dorsey method [23]. There are also numerical methods that find the best fit to the drop profile over the relevant region [24, 25]. With these methods, the drop symmetry, the contact angles, and the local shape variations can be readily determined. In this study, the Dorsey method is utilized for the determination of surface tension using the proposed empirical correlation [23]:

\[
\gamma_{lv} = \frac{g \rho_L d_m^2}{4} \left( \frac{0.0520}{f} - 0.1227 + 0.0481 \right)
\]

\[
f = \left( \frac{2H'}{d_m} \right) - 0.4142
\]

where \( \gamma_{lv} \) is the surface tension in “N/m”, \( \rho_L \) is the density of liquid in “kg/m³”, \( g \) is the gravitational acceleration in “m/s²”, \( d_m \) is the diameter of the widest section of the drop in “m”, and \( H' \) is the distance between the intersection of the 45° line tangent to the drop and the vertical line passing through the center of the drop in “m”. Figure 1(b) describes the geometrical dimensions of the drop used for this calculation.
In the present study, the wetting experiments were carried out for B₄C/Al as well as B₄C/Al-Ti using the sessile-drop method under conditions similar to those used in the Al-B₄C MMC production. The effect of the Ti content on the wettability of B₄C by Al was investigated. The interfacial reactions of sessile-drop samples were studied using an optical microscope and a scanning electron microscope (SEM).

2. Methodology

2.1. Sessile-drop experimental set-up

The sessile-drop experimental system is shown in Figure 2(a). This system consists of a tube furnace (Thermolyne 21100), an inconel tube with a metal injection system (Figure 2(b)), a graphite sample crucible with three compartments (Figure 2(c)), a digital video camera (B/W) (APPRO, model KC), and a secondary rotary vacuum pump (GE, Precision Vacuum Pump, Model D25). The details of the experimental system are given elsewhere [26]. B₄C powder is placed in the middle compartment of the sample crucible. The other two compartments contain titanium particles that are used for the removal of residual oxygen. The graphite injection chamber holds the solid metal or alloy sample. This chamber has a small hole at the bottom and is placed just above the ceramic sample. In this study, commercial pure Al (99.8%) as well as Al-1wt%Ti, Al-1.5wt%, and Al-3wt%Ti alloys were used. B₄C powder (Micro Abrasives Co., F320) with an average particle size of 23 microns was tested.

The experiments were conducted under an argon (Ar) atmosphere. Ar is chosen because N₂ can be absorbed by the metal. There were two entry lines for argon. The main line was directly
connected to the inconel tube for maintaining the inert atmosphere inside this tube. The other line, connecting the injection chamber to the inert gas supply, carried the Ar gas necessary for slightly pressurizing this chamber in order to force the liquid metal/alloy out onto the solid sample. Research grade Ar (99.999%) was used during the experiments. In order to decrease the O₂ and humidity content of the Ar, it was passed through O₂ (Chromatographic Specialties, Oxygen Trap C36084) and humidity traps (Chromatographic Specialties, Glass Moisture Trap - C36150) before it entered the system.

This set-up has various advantages:
a) The molten metal or alloy droplet can be directly dropped on a solid substrate. In most sessile-drop systems, a solid piece of metal is placed on the substrate and it melts as the temperature is increased. However, due to oxidation, the contact angles measured from the form of the molten piece can be erroneous and the errors could be significant.
b) The drop size can be changed by adjusting the hole size of the holder.
c) The sample holder can be removed by using the specially designed mechanism from the hot region of the furnace, and the sample can be quenched for further analysis.

2.2. Experimental procedure
Before starting the experiments, the system is evacuated using a diffusion pump and purged with Ar several times. It is then heated to 600°C under an Ar atmosphere. When the temperature stabilizes at 600°C, a secondary argon line is opened to purge the injection chamber. The furnace is heated to the desired temperature and the molten aluminum which is in the injection chamber is pushed down gently by applying a small pressure to the line leading to this chamber. A sessile-drop forms on the surface of the B₄C powder bed and the experiment starts. A video of the drop
is captured for 20 minutes. The system can capture both static and dynamic behaviors. To measure the contact angle, the FTA 32 software is used. When the experiment is completed, the furnace is shut down, and the sample crucible is pulled back from the hot region of the furnace by using a specially designed sample removing mechanism in order to instantaneously cool the sample. The experiments are repeated a number of times to assure the reproducibility and contact angles given in the text are the average of the results of these experiments. The solidified drops are sectioned vertically and polished for metallographical analysis using an optical microscope, a scanning electron microscope (SEM) and an energy dispersive spectrometer (EDS).

3. Results and discussion

In this project, the wetting characteristics of B₄C/Al as well as B₄C/Al-Ti alloys were investigated at 750 and 850°C. Three different Al-Ti alloys (1wt%Ti, 1.5wt%Ti, 3wt%Ti) were studied using a sessile-drop system in order to understand the effect of the Ti content on the wetting of B₄C particles. The surface tension of Al and Al/Ti alloys were also determined.

3.1. Contact angle

Change of the drop shape for Al/B₄C at different times is shown in Figure 3. This figure also illustrates how the software measures the contact angles.

Figure 4 shows the contact angle data for B₄C/Al as well as B₄C/Al-Ti alloys with different Ti contents as a function of time and temperature. Figures 5(a) and 5(b) show the initial contact angles and contact angles after 20 minutes respectively for the same systems. Initial contact angles are measured 20 second after the drop is placed on the surface of the solid bed and end of this period is taken as the time zero for the experiment. This is necessary for the drop to stabilize
before the contact angle measurement is taken. However, within this stabilization period there is not any significant oxidation of molten metal (pure Al or Ti-Al alloy) nor there is any significant reaction between the metal and the solid powder. The initial contact angle is the angle between the initial solid surface and the metal. The angles after 20 minutes show the effect of reaction between the solid bed and the metal interface on the contact angle. Within this period the metal drop keeps its original color. If the experiment is continued for periods longer than 20 minutes the drop becomes darker showing that an extensive oxidation has taken place. This also manifests itself in contact angle values which remain the same because the shape of the drop can not freely change due to the oxidation. Therefore, the contact angles are presented at the beginning (after the stabilisation period) and after 20 min of reaction. It is inevitable that there is some oxidation within this period, however, the drop stays flexible enough so that the contact angle change can be followed with the time during which interfacial reactions take place. In general, it is observed that the contact angle decreases with increasing temperature and time which are directly proportional to the extent of the reaction. It can be seen from these figures that the contact angles measured with the alloy are lower (more wetting) than those measured with pure aluminum at high temperatures. The results show that B₄C is partially wetted by Al. At the lower temperature (750°C), the initial contact angles for pure aluminum and Al-1wt%Ti alloy are the same. Pure aluminum wets the B₄C particles more when compared to the alloy up to 1000s at this temperature (Figure 4). Subsequently, the Al-1wt%Ti alloy wets the particles more than pure Al, and the contact angle decreases to 125° at the end of the experiment. At higher temperatures, the Ti addition improves the wetting. It is observed that increasing the titanium content of the alloy to 1.5wt% improves the wetting compared to that of the Al-1wt%Ti alloy. When the Ti content is increased from 1.5wt% to 3wt%, the contact angles also slightly rises; consequently, the wetting is decreased compared to that of 1.5wt% Ti. Although the wetting is still better with the Al-
3wt%Ti alloy compared to the wetting with pure Al and the Al-1wt%Ti alloy, there is no further improvement by increasing the Ti content. Among the three concentrations tested, the wetting is best with the Al-1.5wt%Ti alloy at both temperatures compared to the other Al-Ti alloys and pure Al. B4C powder is best wetted by the Al-1.5wt%Ti alloy at 850°C. Although, the change in contact angle is small, there is a definite change as can be seen from the error bars (Figure 5). The results also show that the precautions taken to reduce the reactivity of the system do not affect the wettability in adverse direction. They clearly show that the Ti addition improves the wetting of B4C. However, it is important to select an appropriate Ti level to obtain the best results.

Experimental conditions, such as the duration of the experiment, the surface roughness, and the gas atmosphere have a great impact on the wetting characteristics. Figure 6 compares the initial contact angles measured during this study with the results of other researchers for the B4C-pure aluminum system. The results of the present study seem to be in good accordance with the cited literature [5, 13, 14] in spite of the differences in the experimental conditions used (see Table 1).

3.2. Surface Tension Determination for Al and Al-Ti Alloys

Accurate information on the surface tension of aluminum and its alloys are necessary for many MMC applications. In this study, the effect of titanium addition on the surface tension has been also studied. The surface tensions are calculated by image analysis using the empirical relations proposed by Dorsey [23] given in Equation (7). The results show that the surface tension decreases with increasing temperature (see Figure 7). Since the Ti content changes the liquid properties, it therefore affects the surface tension. The surface tension of the Al-1wt%Ti alloy is lower than that of pure aluminum. The surface tension decreases with increasing Ti content up to 1.5wt%. At both temperatures, however, it starts to increase when the Ti content of the alloy is
further increased, in this case to 3wt%. This is in agreement with the contact angle data (see Figure 5).

As it is well known the surface tension is the property of a liquid when it is in contact with a gas and it is the function of the cohesive forces between the liquid molecules. The reported surface tension of Ti is higher than that of Al [21, 27] Therefore, it can be questioned that how addition of Ti to Al can reduce the surface tension. The nature of the gas phase can play a role in the results. The presence of oxygen and its adsorption on the solid surface can modify the surface tension of the alloy. When the liquid is also in contact with a solid, the forces ($\gamma_{sv}$, $\gamma_{sl}$, $\gamma_{lv}$, see eqn. 6) have to balance at liquid-solid-gas interface (triple point). When the reactions are taking place at the solid-liquid interface this balance changes continuously due to new phases forming at the interface. Also oxidation of the liquid surface when molten metals are concerned affects the surface tension [28]. In this study ultra high purity Ar (99.999 %) is used, however, it is impossible to prevent oxidation entirely due to the high oxygen affinity of aluminum. Therefore, the interfacial reactions or the oxidation of the molten metal might play a role in the surface tension modification observed during this study.

These surface tensions measured with pure Al are generally in agreement with the results reported in the literature shown in Table 2 [21, 29-31]. The surface tensions of pure aluminum measured in this study are in excellent agreement with the results of Ergin [21] who also measured the surface tension of pure aluminum under an argon atmosphere. These results show that the method used give reasonable values for surface tensions; therefore, the surface tensions of the Ti containing aluminum alloys, although they are not reported in the literature, can be used with confidence.
3.3. Microstructural Analyses

In this study, the microstructural analyses are carried out after a 20 minute solid-liquid contact time at 850°C. Figures 8 and 9 present the micrographs of the cross-sections of the drop-substrate interface taken with the optical microscope and the SEM, respectively, for the B$_4$C/Al system. In a reactive system, the wetting frequently occurs as a result of the chemical reactions occurring at the interface (chemical wetting). Due to the interfacial reaction, new reaction induced solid compounds form at the interface. For the B$_4$C/Al system, the formation of a new interface layer of 2-3 µm thickness around the B$_4$C particle surface is clearly observed. However, it was not possible to accurately examine the reaction induced phases in the cross-section of the samples using XRD due to the small volume of the interface. Therefore, it was impossible to identify any phase other than B$_4$C and Al with the XRD technique. As can be seen from the micrographs shown in Figure 8, two types of reaction products, gray and yellow particles, can be distinguished under the optical microscope. The EDS analysis on the grey phase revealed the presence of Al, B, and C elements which indicates the formation of a Al-B-C compound. Several yellow particles were also examined using EDS, and only Al and B elements were detected. According to the chemical stoichiometries and its morphology the yellow phase was suggested as AlB$_2$ compound.

The nature of the interfacial reaction of the B$_4$C/Al system has been extensively studied using transmission electron microscopy (TEM) and electron probe microanalysis (EPMA) in the recent literature [10-12]. When B$_4$C was in contact with liquid aluminum for prolonged times, two reaction products, grey and yellow phases, formed at the interface. The grey phase was identified as Al$_3$BC using TEM and the yellow phase was recognized as AlB$_2$ using EPMA. It has been confirmed that the interfacial reaction between B$_4$C and pure Al follows the reaction shown in Eqn.( 2) to form Al$_3$BC and AlB$_2$ products [8, 10-12]. Careful observations on the interfacial
microstructure under SEM (Fig. 9) reveal that mainly Al$_3$BC crystals cover the entire interface of B$_4$C and even grow into the inside of the B$_4$C particles. The presence of reaction phases implies a considerable dissolution of the B$_4$C surface by the invasion of molten Al. With regard to that the contact angle in the B$_4$C/Al system decreases with increasing time and temperature (Figures 4 and 5), it is reasonable to correlate this time and temperature dependence of the wettability with the interface chemistry, particularly with the formation Al$_3$BC phase. It is evident that the interfacial reaction improves the wetting of B$_4$C with the increasing contact time due to the better wettability of the reaction products by aluminum, which is confirmed by the recent study of Lin et al [16].

Figure 10 shows optical micrographs of the B$_4$C/Al-Ti alloy with different Ti contents. It is observed that with the presence of Ti in aluminum, AlB$_2$ particles disappear and new Ti rich needle-like particles form around the B$_4$C particle at the interface. The needle-like particles are approximately 0.1 to 3µm in size. It is also observed that, with increasing Ti level, the amount of gray particle (Al$_3$BC) is decreased (see Figures 10(a) and (b)). However, further increase in the Ti levels to 3wt% causes the formation of large plates of Al$_3$Ti intermetallics in the aluminum matrix (Figure 10(c)).

Figure 11 presents the SEM micrographs for the alloys containing different Ti percentages. The EDS analysis of the needle-like phase revealed the presence of Ti and B elements, suggesting a TiB$_2$ compound according to its chemistry. The interfacial reaction between the B$_4$C and Al-Ti alloys has been recently established [10-12]. When B$_4$C was brought into contact with the molten Al-Ti alloy, a Ti-rich layer composed of fine TiB$_2$ crystals and needle-like TiB$_2$ particles formed around the B$_4$C particle surfaces, as described by SEM and TEM investigations [10,11]. The Ti addition resulted in the formation of TiB$_2$ which is more stable than AlB$_2$, and the AlB$_2$ was
gradually replaced by TiB$_2$ as the Ti content of the alloy increases, followed by the reaction given by Eqn. (4) [12]. This TiB$_2$ layer isolated the B$_4$C from molten Al and acted as an efficient diffusion barrier at the B$_4$C/Al interface. Consequently, the wetting behaviour of B$_4$C by molten Al-Ti alloy changed considerably.

Since TiB$_2$ is almost completely wettable by molten Al [32], the wettability of B$_4$C improves remarkably with the Ti addition and a prolonged contact time. The wettability of B$_4$C by Al-Ti alloys is better than that of by pure Al. It is evident that the presence of Ti increases the stability of the B$_4$C particles in the Al matrix through the formation of a protective barrier of TiB$_2$ around the B$_4$C particles which not only prevents further reaction of B$_4$C with Al but also improves the wettability of B$_4$C by Al. This phenomenon is in good accordance with the measured contact angle data (Figures 4 and 5).

4. Conclusions

1. Wetting of B$_4$C particles by molten Al as well as Al-Ti alloys with different Ti contents has been studied using the sessile-drop technique at 750 and 850°C. In general, it is observed that the contact angle decreases with increasing temperature and time.

2. The wettability of B$_4$C by Al increases as the Ti content increases up to 1.5wt%. The optimum Ti level is around 1.5wt%.

3. The wetting of B$_4$C by molten Al is influenced by the interfacial reaction products formed around the B$_4$C particles, namely the Al$_3$BC and AlB$_2$ phases in B$_4$C/Al system and the TiB$_2$ and Al$_3$BC phases in B$_4$C/Al-Ti Alloy system.
4. The surface tension of Al is reduced by increasing the temperature and the Ti content of the alloy up to 1.5wt% Ti. If the Ti content is further increased to 3wt%, the surface tension begins to increase.

5. It is difficult to compare the initial contact angle data with those found in the literature due to the different experimental conditions used. However, the initial contact angles measured during this work seem to be in the same range as those cited in the literature for the B₄C/Al system [4, 10].

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References


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Table 2: An Overview of Surface Tension Data for Pure Aluminum Reported in Literature
### Table 1: An Overview of Wetting Data Reported in Literature for B₄C/Pure Al System

<table>
<thead>
<tr>
<th>Author</th>
<th>Experimental Medium</th>
<th>Metal (Al) Purity</th>
<th>Ceramic Substrate</th>
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<td>99.99%</td>
<td>Particles</td>
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Table 2: An Overview of Surface Tension Data for Pure Aluminum Reported in Literature

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<th>Method</th>
<th>Environment</th>
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<td>707-817</td>
<td>760-720</td>
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*SD : Sessile-drop
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