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Aluminium- Titanium Diboride (Al-TiB₂) Metal Matrix Composites: Challenges and Opportunities

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Abstract

A review of Al based metal matrix composite reinforced with TiB₂ particles developed after 1997 is presented in this paper. This paper presents an overview of Al- TiB₂ MMC on aspects relating to the formation of TiB₂, development of Al- TiB₂, mechanical characteristics, thermodynamic calculation, wear behavior of Al- TiB₂, cycle fatigue response of in-situ Al based composite, processing, microstructure, properties and application are discussed.

Aluminium alloys are widely used for commercial applications in the transportation, construction and similar engineering industries. Nowadays, main focus is given to Aluminium as matrix material because of its unique combination of good corrosion resistance, low electrical resistance and it possesses excellent mechanical properties in addition to good corrosion resistance due to which the alloy finds extensive application in naval vessels manufacturing. Al-TiB₂ composite is a metal matrix composite (MMC) that can be manufactured using the in-situ salt-metal reaction. With TiB₂ as the particulate addition the properties of Al 6061 alloy can be greatly improved. The addition of TiB₂ to aluminium increases the strength of the aluminium. The wear resistance of this material is due to the lack of pull out of this material from the aluminium matrix. Also, such material when machine with non-conventional methods like EDM will show better machinability due to its high conductivity. In the last few years, Al-TiB₂ have been utilized in high-tech structural and functional applications including aerospace, defense, automotive, and thermal management areas, as well as in sports and recreation. The future research is summarized finally.

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Keywords: Al- TiB₂ MMC, Aluminum alloys, Wear behavior, Thermodynamic calculation microstructure and application of MMC

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1. Introduction

Metal matrix composites (MMCs) have emerged as an important class of materials for structural, wear, thermal, transportation and electrical applications, primarily as a result of their ability to exhibit superior strength-to-weight and strength-to-cost ratio when compared to equivalent monolithic commercial alloys [8]. Aluminum-based particulate reinforced metal matrix composite have emerged as an important class of high performance materials for use in aerospace, automobile, chemical and transportation industries because of their improved strength, high elastic modulus and increased wear resistance over conventional base alloy [10].

Al based composite reinforced by hard ceramic particles have become more and more attractive in the research of structural composites. The addition of ceramic particles such as TiB₂, SiC, Al₂O₃, B₄C to an Aluminum based matrix does not considerably change the density of the material but instead it usually leads to a significant rise in specific strength and modulus of composite. This improvement of composite plays a role as weight saving material for use in structural applications [6]. In the addition of above ceramics, TiB₂ has emerged as an outstanding reinforcement. This is due to the fact that TiB₂ exhibits outstanding features such as high melting point (2790°C), high hardness (86 HRA or 960 HV) and high elastic modulus (530 × 10⁵ GPa) and good thermal stability. It has resistance to plastic deformation temperature. TiB₂ particles do not react with molten aluminum, thereby avoiding formation of brittle reaction products at the reinforcement-matrix interfaces. Thus the Al-TiB₂ composites exhibit certain useful and unique characteristics [3, 9].

Fabrication of in situ composite involves synthesis of reinforcing phases directly with in the matrix. This approach is in contrast to ex situ composite where the reinforcements are synthesized separately and then introduced into the matrix during a secondary process such as melting, infiltration (or) powder processing [1]. The main problem lies in ex situ composite is thermodynamic instability of reinforcing ceramic phases with the matrix [2]. This problem can be solved by using in situ technique. And also there are several potential advantages associated with insitu composite such as thermodynamic stability, kinetic control of the reaction, reduced size, uniform distribution of the reinforcing particles, good interfacial bond and economy in processing. The incorporation of TiB₂ particles in the matrix by insitu processing offer special advantages such as clean matrix-particle interface resulting in effective load transfer and minimizing the wear rates [4].

2. Experimental Procedures

As discuss earlier, MMCs can be have been manufactured by two methods one is ex situ method and another is insitu method. But insitu process gave several potential advantages over exsitu process. Some of experimental methods that involves in the development of insitu Al-TiB₂ is discuss below.

The A16063 base matrix alloy and two master alloy Al-10% Ti and Al-3%B are melted in the electric resistance furnace with a stoichiometric Ti:B ratio 2:1. The percentage weight of Al-10% Ti was varied from 10 wt% to 20 wt% in steps of five with stoichiometric weight ratio intact [1, 2]. Here the TiB₂ reinforcement has been created by an in situ reaction using an alloy – alloy technique. In another method, stir-casting technique is used to prepare in situ Al-TiB₂ composite. Here Ti sponge powder (99.5% purity; average size 150 μm), amorphous B powder (96.1 % purity; average size 1 μm) and commercial aluminium AA1100 wire were used as the initial materials. [9] These powders of stoichiometric compositions based on equation (1).

\[2\text{Ti} + 2\text{B} + 3\text{Al} \rightarrow \text{TiB}_2 + \text{Al}_5\text{Ti}\]  

(1)
In another process Al6063 base matrix alloy and salts such as potassium hexafluorotitanate \{K₂TiF₆\} and potassium tetrafluoroborate \{KBF₄\} are used. In this system, the exothermic reactions between the salts yield in situ formed TiB₂ particulates in the alloy. \[10\]. In another method pure aluminium was used as the base metal and two types of salt K₂TiF₆ and KBF₄ was used to synthesise the TiB₂ reinforcement. The overall reaction showing the formation of TiB₂ can be written as \[8\],

\[
K₂TiF₆ (l) + KBF₄ (l) + Al (l) \rightarrow TiB₂ (in Alloy) + KFAlF₃ (l) + KF (l) + AlF₃ (l)
\]  

(2)

The synthesis of in situ Al-TiB₂ can also carried out from the Al-TiO₂-B and Al-TiO₂-B₂O₃ systems using RHP method (Reactive hot processing). Using Al-TiO₂-B system three types of in situ reinforcements, i.e. Al₂O₃, TiB₂ and Al₃Ti can be created during hot pressing process. The synthesized reactions can be summarized as:

\[
3TiO₂ + 13Al \rightarrow 2Al₂O₃ + 3Al₃Ti
\]  

(3)

\[
3TiO₂ + 6B + 4Al \rightarrow 2Al₂O₃ + 3TiB₂
\]  

(4)

From the above reaction we can summarized that the amount of Al₂O₃, TiB₂ and Al₃Ti can be control by varying the B content. Using Al-TiO₂-B₂O₃ system the following reactions are expected:

\[
B₂O₃ + 2Al \rightarrow Al₂O₃ + 2B
\]  

(5)

\[
3TiO₂ + 4Al \rightarrow 2Al₂O₃ + 3 Ti
\]  

(6)

It is suggested that Ti can also react with Al and B to form titanium aluminium borides (Ti, Al)ₓ Bᵧ by following reaction:

\[
xTi + x Al + yB \rightarrow \{Ti, Al\}ₓ Bᵧ
\]  

(7)

There are some experiments for the comparison of Al-4Cu alloy and Al-4Cu-TiB₂ composite, on the basis of wear, grain structure, micro hardness etc.

3. Formation of TiB₂ particles in the in situ composite:

The formation of TiB₂ particles under various reactants are discuss below. In alloy-alloy technique after the start of reaction, further cracks and voids initiate from the external surface of the Al₃Ti particles and propagate into the bulk. The number of sites for the reaction is increased so that the rate of the process is raised with the resulting formation of finer TiB₂ particles. In addition, the reaction between Ti and B is highly exothermic in behaviour and the heat generated helps to accelerate the formation of TiB₂ readily. The sequence of TiB₂ formation can be proposed in five steps.

(i) Movement of boron atoms towards Al₃Ti particles
(ii) Reaction of Ti and B in a gap from Al₃Ti surface to form TiB₂
(iii) Diffusion of boron atoms through TiB₂ particles due to its small size
(iv) Natural cracks on Al₃Ti particles and formation of Al₃Ti which enhances dissolution of Al₃Ti particles, will lead to increased rate of TiB₂ formation
(v) Formation of ring like TiB₂ particles \[1, 2\].
Studies indicate that the primary TiB₂ particles on the surface of TiAl₃ are appreciably free and movable and because of boron diffusion across boundary layer towards TiAl₃, TiB₂ particles produces during growth with the primary ones formed agglomeration ring. This can be expressed as [2]

\[ \text{TiAl}_3 + 2\text{B} \rightarrow \text{TiB}_2 + 3\text{Al} \]  \hspace{1cm} (8)

\[ \Delta G^\circ = -43.4 \times 10^3 + 15.8 \text{ T} \]  \hspace{1cm} (9)

From the thermodynamic equation, it is expected that TiB₂ is stable at applied temperatures [2]. The formation of in situ TiB₂ ceramic particles on the basis of equation (2) is described below. The overall reaction is essentially divided into three parts.

(i) The decomposition of K₂TiF₆ and KBF₄ to KF liquid, TiF₄ and BF₃ gases at the molten flux-liquid metal interface to form TiB₂ and AlF₃.
(ii) The aluminothermic reduction of TiF₄ and BF₃ gases at the molten flux-liquid metal interface to form TiB₂ and AlF₃.
(iii) Formation of a cryolite (3KFAIF₃ and KF mixture

The formation of TiB₂ ceramic particles in the stir casting technique is shown below [9].

\[ \text{Ti} + 3\text{Al} \rightarrow \text{Al}_3\text{Ti} \]  \hspace{1cm} (10)

\[ \text{Ti} + 2\text{B} \rightarrow \text{TiB}_2 \]  \hspace{1cm} (11)

\[ \text{Ti} + 2\text{B} + 3\text{Al} \rightarrow \text{TiB}_2 + \text{Al}_3\text{Ti} \]  \hspace{1cm} (12)

Figure. 1: Schematic representation of fragmentation and dissolution of TiAl₃ particle and formation of ring-like TiB₂ particles

The formation of in situ TiB₂ particulates from the Al-TiO₂-B and Al-TiO₂-B₂O₃ systems using reactive hot pressing technique (RHP) is based on the equations (3,4,5,6). From that equation the amount of Al₂O₃, TiB₂ and Al₃Ti phases can be controlled by varying the boron content. The studies indicate that by adjusting the molecular ratios of B/TiO₂ to 6/3 in the Al-TiO₂-B system can completely eliminate Al₃Ti phase [3].
4. Thermodynamic calculations:

Using thermodynamic laws, the calculated values for the enthalpy and Gibbs free energy for the reactions at 1080°C in stir casting technique and the calculated value of Gibbs free energy at 1000K on the basis of equation (2) is shown in table (1) and (2), [8, 9].

<table>
<thead>
<tr>
<th>Reactions</th>
<th>$\Delta H^o_{1353}$ (KJ/mol)</th>
<th>$\Delta G^o_{1373}$ (KJ/mol)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti + 3Al $\rightarrow$ Al$_3$Ti</td>
<td>-12738</td>
<td>-12694</td>
</tr>
<tr>
<td>Ti + 2B $\rightarrow$ TiB$_2$</td>
<td>-4429</td>
<td>-4400</td>
</tr>
<tr>
<td>Ti + 2B + 3Al $\rightarrow$ TiB$_2$ + Al$_3$Ti</td>
<td>-16620</td>
<td>-17020</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Reactions</th>
<th>$\Delta G^o$ (J/mol)</th>
<th>$\Delta G^o_{1000}$ (J/mol)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$K_2$TiF$_6$(l) + 2KBF$_4$(l) = 4KF (l) + TiF$_4$(g) + 2BF$_3$(g)</td>
<td>432207 - 318.4T</td>
<td>113807</td>
</tr>
<tr>
<td>TiF$_4$(g) + BF$_3$(g) + 3½ Al(l) = TiB$_2$(s) + 3½AlF$_3$</td>
<td>-1424936 + 617.2T</td>
<td>-807736</td>
</tr>
<tr>
<td>3KF (l) + AlF$_3$(l)</td>
<td>8075 - 139.5T</td>
<td>-131425</td>
</tr>
<tr>
<td>Ti + 2B = TiB$_2$</td>
<td>-284510 + 20.5T</td>
<td>-264010</td>
</tr>
<tr>
<td>TiB$_2$ (s) = TiB$_2$(l)</td>
<td>100690 - 31.5T</td>
<td>69190</td>
</tr>
</tbody>
</table>

In general, the excess free energy $\Delta G$ has been used to determine the possibility of a reaction. The free energy of a system at a temperature $T$, can be written as,

$$\Delta G = \Delta H - T\Delta S$$

(13)

Where $\Delta H$ and $\Delta S$ are the change in enthalpy and entropy of two states, $\Delta G$ is only a function of temperature and also dependent on the concentration of the reactants, the influence of composition on the reaction must be taken into consideration.

In the reaction, $\Delta G$ in a reaction may be expressed as

$$\Delta G^o_{T, p} = \Delta G^o_{T, p} + \sum_i n_i RT \ln \alpha_i = \Delta G^o_{T, p} + \sum_i n_i RT \ln x_i + \sum_i n_i RT \ln \gamma_i$$

(14)

Where, $\Delta G^o_{T, p}$ is the standard free energy, $x_i$, the molar concentration of the component $i$ in the solution, and $\gamma_i$ is the activity coefficient component $i$. 
Lupis presented a feasible method to find $\gamma_i$. By carry out the experiments between the salts KBF$_4$, K$_2$TiF$_6$ and Al. The above equation is compared with the relative intensity of XRD spectra, D is evaluated,

$$D_i = \frac{I_i}{I_i + I_j + I_k} \times 100\%$$  \hspace{1cm} (15)

Where $I_i$ is the intensity of compound $i$. It can be concluded that the present model is suitable for the analysis of in-situ reaction of Al- TiB$_2$ composites [6].

5. Relation between the reaction holding time and the formation of TiB$_2$ particulates.

In stir casting technique, a holding time of 30 minute was found to produce the optimal result with the highest mechanical properties. When the holding time was too short, the formation of TiB$_2$ particulates was low and the reinforcing particles were hardly discernible. On the other hand, when the holding time was extended beyond 30 minute, coarse Al$_3$Ti flakes were produced in the composites. In addition the viscous melt made casting difficult [9]. On the basics of equation (2), it can be seen that the weight percentage of TiB$_2$ first increase to 14.7 up to a reaction holding time of 20 minute and subsequently decreases as the reaction holding time is increased to 40 minute. The variation in weight percentage of TiB$_2$ can be attributed to an increase in the volume of cryolite slag with an increase in holding time. The studies indicate that there is always a balance between the rate of formation of TiB$_2$ and the volume of cryolite slag. When the holding time less than 20 minute, the rate of formation of TiB$_2$ produced via chemical reaction was greater when compared to the formation of cryolite slag containing TiB$_2$ particles. Once the holding time was increased beyond 20 minute, the amount of slag produced was too great enabling the small particles of TiB$_2$ to become oxidized and trapped in the slag. The variation of weight percentage of TiB$_2$ particulates on the basics of reaction holding time is shown in table (3), [8].

<table>
<thead>
<tr>
<th>Matrix</th>
<th>Additions made</th>
<th>Reaction temperature ($^\circ$C)</th>
<th>Reaction holding time (min)</th>
<th>Resultant composite</th>
<th>Reinforcement (weight %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>K$_2$TiF$_6$ and KBF$_4$ salts</td>
<td>850</td>
<td>10</td>
<td>TiB$_2$/Al</td>
<td>12.7</td>
</tr>
<tr>
<td>Al</td>
<td>K$_2$TiF$_6$ and KBF$_4$ salts</td>
<td>850</td>
<td>20</td>
<td>TiB$_2$/Al</td>
<td>14.7</td>
</tr>
<tr>
<td>Al</td>
<td>K$_2$TiF$_6$ and KBF$_4$ salts</td>
<td>850</td>
<td>30</td>
<td>TiB$_2$/Al</td>
<td>11.5</td>
</tr>
<tr>
<td>Al</td>
<td>K$_2$TiF$_6$ and KBF$_4$ salts</td>
<td>850</td>
<td>40</td>
<td>TiB$_2$/Al</td>
<td>7.7</td>
</tr>
</tbody>
</table>

6. How temperature affects the formation of TiB$_2$ particulates:

The temperature at which the Ti and B powder were introduced played an important role in synthesis of TiB$_2$. In stir casting technique, when the mixture was introduced at temperatures below 1000$^\circ$C and raised to 1080$^\circ$C after all the powder had been added, no TiB$_2$ particle was detected. However, when the temperature of introducing the mixture was increased to 1060$^\circ$C and then ramped to 1080$^\circ$C, TiB$_2$ particlars was found and the intensity of TiB$_2$ particulates increased with the increase in volume fraction of composite. When the temperature was maintained 1060$^\circ$C or 1080$^\circ$C throughout the entire duration of
adding the mixture and holding until the completion of reaction, insignificant TiB$_2$ phase was again detected in the former case, but in later case, the recovery of composite was low. This was because solid lump was formed immediately after a batch of mixture was introduced and continual holding at this temperature caused the melt to change into a silvery residue. The choice of introducing the mixture at 1060°C had prevented the formation of a solid lump from taking place whilst racing the e temperature to 1080°C the permitted the reaction to take place smoothly. The analysis showed that at 1080°C should be used in order to effect an optimum formation of TiB$_2$ in the composite [9].

7. Loses occur in preparation of Al-TiB$_2$ composite:

In stir casting technique, there was marginal but inconsistent difference between the actual and measured volume fraction of TiB$_2$ in the composite. The 1.3-3.6% difference may be attributed to two factors: the loss of unreacted Ti and B powders on the melt surface leading to the formation of slag and the removed of dross from the melt surface during the skimming process. The variation of designing and measured value of volume fraction of TiB$_2$ is shown in the table (4), [9]. As discuss earlier the TiB$_2$ particulates is losses by the presence of cryolite slag is the one type of loss found on the basis of equation (2). So more efficient method is looking for the preparation of Al-TiB$_2$ composite.

<table>
<thead>
<tr>
<th>Designed Vf$_{%}$ (%)</th>
<th>Measured Vf$_{%}$ (%)</th>
<th>Difference Vf$_{%}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>3.7</td>
<td>1.3</td>
</tr>
<tr>
<td>10</td>
<td>7.3</td>
<td>2.7</td>
</tr>
<tr>
<td>15</td>
<td>11.8</td>
<td>3.6</td>
</tr>
</tbody>
</table>

8. Characterization of composite:

8.1. Interfacial Characteristics:

In situ composites exhibit cleaner particle- matrix interface, thereby yielding strong interfacial- matrix bonding. The absence of voids and reaction products, indicating good integrity between the in situ produced TiB$_2$ particles and the aluminum matrix. The formation of a good Al-TiB$_2$ interface can be attributed to several factors.

(i) The in situ formation of TiB$_2$ with in the melt itself reduces the opportunity for the oxidation of particles and hence the surfaces of the TiB$_2$ particles tend to be free from oxides, which improves interfacial integrity.

(ii) The exothermic reaction between the salt and the melt result in an increase in the local melt temperature, which improves the interfacial conditions for wetting of the reinforcement particles.

(iii) The K-Al-F slag of cryolite type formed during the reaction has high solubility for Al$_2$O$_3$. This slag assists particle wetting by dissolving the aluminum oxide on the surface and within the melt.

(iv) The removal of the oxide reduces the wetting angle between the liquid Al and TiB$_2$ and improves the wetting condition and hence produced good interfacial integrity [8].
8.2. Microstructure:

The TiB₂ particle size on the basis of equation (2) decrease with increase in the reaction holding time up to 30 min, beyond which the particle size increases slightly. It is due to the possibility of the presence of some TiAl₃ amongst the TiB₂ particles for longer reaction holding time; TiAl₃ tends to decompose to form TiB₂. The TiAl₃ in the composite casts would not contribute much as a reinforcement phase when compared to TiB₂ [8]. Table 5 shows the results of TiB₂ particle-size measurement conducted using Quantimet Image analysis.

Table: 5 Results of grain-size and particle-size measurement using Quantimet image analysis:

<table>
<thead>
<tr>
<th>Matrix</th>
<th>Additions made</th>
<th>Reaction holding time(min)</th>
<th>Resultant composite</th>
<th>Grain size (µm)</th>
<th>Particle size (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>-</td>
<td>-</td>
<td>Al</td>
<td>73.58 ± 16.25</td>
<td>-</td>
</tr>
<tr>
<td>Al</td>
<td>K₂TiF₆ and KBF₄ salts</td>
<td>10</td>
<td>TiB₂/Al</td>
<td>23.52 ± 6.30</td>
<td>0.57 ± 0.15</td>
</tr>
<tr>
<td>Al</td>
<td>K₂TiF₆ and KBF₄ salts</td>
<td>20</td>
<td>TiB₂/Al</td>
<td>18.48 ± 4.19</td>
<td>0.53 ± 0.17</td>
</tr>
<tr>
<td>Al</td>
<td>K₂TiF₆ and KBF₄ salts</td>
<td>30</td>
<td>TiB₂/Al</td>
<td>12.75 ± 2.94</td>
<td>0.43 ± 0.14</td>
</tr>
<tr>
<td>Al</td>
<td>K₂TiF₆ and KBF₄ salts</td>
<td>40</td>
<td>TiB₂/Al</td>
<td>23.11 ± 6.51</td>
<td>0.54 ± 0.21</td>
</tr>
</tbody>
</table>

Lai and Coworker reported that the size of in situ TiB₂ particles formed in the matrix of Al based composites varies from 0.5 to 1.5 µm. It is generally accepted that the strengthening of particulate-reinforced MMCs depends significantly on the shape, size and amount of the reinforcing particles as well as the matrix microstructure. In case of particle size of several micrometers would act as stress concentrators, giving rise to the reinforcement fracture during tensile loading. The propensity to particle cracking tends to diminish with decreasing the size of reinforcing particles, especially for submicron TiB₂ and Al₂O₃ particulates seen in Al-TiO₂-B₂O₃ and Al-TiO₂-B system. The yield strength of the aluminum-based MMCS reinforced with submicron (or) nanometric particles is related to the particulate-dislocation interaction by means of the orowan bowing mechanism. Residual dislocation loops are left around each particle after the dislocation passes the particles. In other words, the high hardness of the TiB₂ and Al₂O₃ submicron particles causes the dislocations to loop around them during tensile loading. Mathematically, the Orowan stress (σ) can be expressed as,

$$\sigma = \frac{M G b}{L}$$  \hspace{1cm} (16)

Where, M the orientation factor, G the material’s shear modulus, b the burger’s vector and L the inter particle spacing. The value of L can be determined from the equation,

$$L = \left(\frac{6V}{\pi} \right)^{1/3} d$$  \hspace{1cm} (17)

Where V and d are the volume fraction and mean diameter of fined particles.
9. Summary and Future Research:

Based on the author's investigation, it has been found that the research on the following aspects of the Al-TiB₂ MMC is relatively limited and may attract more interests in the future research.

(i) It has been shown that the addition of TiB₂ particles to aluminium casting alloys has a direct effect on some important features such as microstructure, thermal properties and mechanical properties. Contents higher than those traditionally used for grain refining purposes (only around 0.2 vol. %) provide additional advantages that may compensate the higher cost of the material.

(ii) Furthermore properties may be tailored and adjusted to the specific requirements of the industry based on the TiB₂ content within the alloy. In order to advance in the use TiB₂ reinforced aluminium alloys for industrial applications some additional works that might be useful are following pointed out: i). Optimization of the production of the reinforced material, ii). Study and optimization of thermal treatments, iii). Study of the behavior of the reinforced materials with other production processes, iv). Use of other additives like silicon modifiers, fluxes or grain refiners.

(iii) The wear rate of the composites improves significantly with the TiB₂ content. The study also shows that wear rate is a strong function of TiB₂ content rather than the overall hardness of the composite. It is suggested that the wear rate per unit amount of reinforcement (here, TiB₂) should be taken into consideration to explain the load carrying capacity of MMCs.

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