Microstructure and mechanical characteristics of an in-situ synthesis of AA7075/TiB$_2$ metal matrix composite

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Abstract. In the present study, AA7075/TiB$_2$ aluminium metal matrix composite (AMCs) was prepared by stir casting method using in-situ reactions of inorganic salts KBF$_4$ and K$_2$TiF$_6$. In this process AA7075 alloy is reinforced with different weighted percentages of (5 %wt, 10 %wt, and 15 %wt) Titanium Diboride (TiB$_2$) particles. X-ray Diffraction (XRD) investigation reveals the presence of TiB$_2$ particles without any formation of the intermediate phase. An optical microscope was used to examine the microstructure, which revealed that the TiB$_2$ particles are equally distributed and that grain size reduces as the weighted percentage of reinforcement particles increases. When the weighted percentage of TiB$_2$ reinforcement particles increased, the microhardness and ultimate tensile strength of the AA7075/TiB$_2$ AMCs increased. Furthermore, the ductile mode of failure of the tensile specimen has been observed by fractography analysis.

Keywords: Aluminium metal matrix composite, in-situ reaction, stir casting, AA7075 alloy, TiB$_2$

1. Introduction

Metal matrix composites (MMCs) are composites made up of metallic matrix (copper, magnesium, cobalt, iron, and aluminium) and a scattered phase. The primary benefits of aluminium-based MMCs materials are extraordinary strength, controlled thermal coefficient, wear resistance, improved stiffness and improved abrasion, and enhanced damping capabilities. [1-3] Aluminium-based metal matrix composites are the composites where Al is used as matrix, and the other components are inserted into the matrix, which is known as reinforcement, for example, (TiB$_2$, SiC, Al$_2$O$_3$, C, B$_4$C,) Al-based MMCs are broadly utilized in automobiles and Aerospace applications. It is because of its properties like high strength, improved thermal properties, and improved durability [8]. Particulates reinforced aluminium matrix composites (PRAMCs) feature a unique set of properties, including high thickness, high hardness, high specific modulus, and a low magnification coefficient at low temperatures [10]. Because of its high hardness, great thermodynamic stability, high corrosion resistance, high modulus, high melting point, and low density, the TiB$_2$ class is considered to be an excellent reinforcement in aluminium among the various possible reinforcement particles such as Al$_2$O$_3$, Si$_3$N$_4$, TiC B$_4$C, SiC, and TiB$_2$. Aluminium-based MMCs are considered to synthesize monolithic material, including titanium alloys, aluminium alloys, polymer composites and ferrous alloys in numerous applications. In this investigation, stir casting was chosen over other processes like powder metallurgy, foil diffusion bonding, stir casting, and physical vapour deposition.

In the casting process, a liquid substance is poured into a mould, which contains a hollow chamber of the desired shape, and then allowed to harden. Stir casting is a sort of casting method in which a mechanical stirrer is used to create a vortex in the matrix material to combine reinforcement. The main advantages of stir casting are its simplicity, flexibility, and possibility of producing large volumes. This process is the most economical among all the available approaches. It also allows a substantial component to be fabricated.

In-situ composites are multiphase materials in which the reinforcing phase is produced within the matrix while the composite is being made. Ex-situ composites are those in which the reinforcement is synthesized outside of the matrix and then incorporated in the matrix using a secondary process like penetration or powder metallurgy. [9]

Another method was built in this study to manufacture TiB$_2$ particulate aluminium in-situ composites with enhanced molecular dispersion, which included mechanical stirring on the salt/aluminium interface. Typical work on changing aluminium-based
compounds (ex-situ composites) entails adding reinforcement to matrix alloys from the outside, such as SiC, Al₂O₃, and TiC. This mechanism can result in thermodynamic instability of the reinforcement and poor interfacial attachment. In-situ composites have a more homogenous dispersion of the scattered phase particles because the reinforcement is combined inside the matrix. The bonding between in-situ-shaped dispersed particles and matrix is better than in ex-situ MMCs. Types of equipment for preparing in-situ composites are less expensive than the other. Many researchers have explored the impact of the weighted percentage of TiB₂ particles on the microstructure and mechanical behavior of composite material and saw that as the weighted percentage of reinforcement particles changes, the mechanical and microstructural behavior also changes. Soltani et al. [5] studied Al-SiC composites (3% wt) and found that a shorter stirring period is needed for ceramic amalgamation to achieve metal/ceramic bonding. Srivatsan et.al [6] investigated the influence of particulate silicon carbide on the mechanical properties of Al 6061 MMC. Sethi et al [7] studied the synthesis of AA7075/TiB₂ aluminium composite and found that the ultimate tensile strength increases, hardness increases, and the size of grains decreases with an increase in the weighted percentage of the reinforcement particles.

In the current investigation, AA7075 alloys were chosen for matrix material and TiB₂ as reinforcement with different weighted% (5%wt, 10%wt, and 15%wt). Synthesis is done through an in-situ reaction of KBF₄ and K₂TiF₆ salt by the stir casting method. The impact of reinforcing particle weighted percent on microstructure and mechanical qualities is investigated.

2. Experimental Procedure

In the present study, AA7075/TiB₂ composite was synthesized by the bottom pour stir casting method. Casting is done in vacuum condition in the presence of Argon gas. An advanced ultrasonic vibrator is used as a stirring mechanism for the uniform distribution of reinforcement particles. Inside a graphite crucible, AA7075 rods were placed and then heated using an electrical furnace. To remove the moisture content a combination of K₂TiF₆ and KBF₄ salt of required weighted% was preheated at 250 °C for 30 mins. On molten aluminium alloy, preheated salts were added at 850 °C and stirred intermittently for 40 mins. After complete expulsion of slag at temperature 850 °C, the composite melt was bottom-poured in the preheated die.

\[
K_2TiF_6 + \frac{13}{3} Al \rightarrow Al_3Ti + \frac{4}{3} AlF_3 + 2KF \quad (1)
\]

\[
2KBF_4 + 3Al \rightarrow AlB_2 + AlF_3 + 2KF \quad (2)
\]

\[
Al_3Ti + AlB_2 \rightarrow TiB_2 + 4Al \quad (3)
\]

Some gases and intermetallic compounds Al₃Ti and AlB₂ were formed when KBF₄ and K₂TiF₆ salts were added with molten aluminium. Again, Al₃Ti and AlB₂ react with each other to form TiB₂ particles. For the complete conversion of TiB₂ and Al₃Ti, an excess amount of KBF₄ was added. In Fig.1. An experimental setup of stir casting is shown. Using the standard metallographic technique, the specimens were polished. Etching of specimens was done by using Keller's reagent. For microstructure analysis of etched specimens, a scanning electron microscope and Leica light optical microscope was used. The tensile samples were produced according to the ASTM-E8 standard for tensile strength testing. In the INSTRON 8801 UTM, the specimens were evaluated at a strain rate of 0.5mm/min. The composite's microhardness was tested using Vicker microhardness estimation with a 100g load and a 30s dwell duration. Scanning electron microscopy was used to investigate the fracture surface of the failed tensile sample (SEM). In Fig. 2. The weighted percentages of the as-cast AA7075/TiB₂ composite are depicted.

| Element | Mg  | Mn  | Cu  | Cr  | Fe  | Ni  | Si  | Ti  | Zn  | Al   |
|---------|-----|-----|-----|-----|-----|-----|-----|-----|-----|------|
| Weighted % | 2.11| 0.07| 1.48| 0.22| 0.23| 0.01| 0.10| 0.07| 5.29| bal  |

Table 1: Chemical composition of AA7075 alloy

Fig. 1. Experimental stir casting setup  
Fig. 2 Different weighted percent of as-cast AA7075/TiB₂ composite
3. Results and discussions

3.1. X-Ray Diffraction (XRD) analysis of composite material

The in-situ formation of the TiB\textsubscript{2} phase is formed during the synthesis of KBF\textsubscript{4} and K\textsubscript{2}TiF\textsubscript{6} salts and molten aluminium. In Fig 3 X-Ray diffraction (XRD) pattern of the AA7075/ TiB\textsubscript{2} reinforcement composite specimen is shown. The complete reaction has been observed as no intermediate phase is formed only Al and TiB\textsubscript{2} are present. This indicates that the TiB\textsubscript{2} particles in-situ are thermodynamically stable. Undesirable compounds will be produced by reaction with matrix alloy if particles are not in a thermodynamically stable state. The absence of other compounds in significant quantity indicates that the interface between AA7075 and TiB\textsubscript{2} is free from contamination. From the pattern, it is also observed that as the weighted percentage of reinforcement particles increases, the intensity of peaks of TiB\textsubscript{2} also increases. This will eventually accumulate at the particles-matrix alloy interface, lowering the load-bearing capacity. It is also seen that the AA7075 diffraction peaks were higher because of the presence of a large quantity of matrix material in the composites.

![Fig 3: XRD analysis of AA7075/TiB\textsubscript{2} insitu composites](image)

3.2. Microstructure of AA7075/TiB\textsubscript{2} AMCs

Microstructures of varied weighted percentage (5% wt, 10% wt, and 15% wt) reinforcement AA7075/TiB\textsubscript{2} are illustrated in Fig.4. Both intergranular and intragranular zones have an equal distribution of TiB\textsubscript{2} reinforcement particles. The microstructures of as-cast AA7075 are dendritic structures, these dendritic structures are formed due to solidification [8]. The dendritic structure of cast AA7075 has been adjusted to grainy structures, this is because of the grain refining activity of TiB\textsubscript{2} particles [12, 13]. From the figure below, it can be seen that the amount of agglomeration in 10% wt and 15% wt reinforcement is insignificant. Because the nucleation sites grow, grain formation of the aluminium matrix is restricted, the size of the composite grains reduces as the weighted percent of reinforcement increases [10, 11]. The presence of TiB\textsubscript{2} particles, which increases the nucleation rate, is also seen in the microstructure, indicating that the composite is better attributed to their existence. Synthesizing TiB\textsubscript{2} particles within the melt minimises the chance of oxidation, resulting in a cleaner and stronger interface.

![Fig 4: Optical photomicrographs of AA7075/TiB\textsubscript{2} composites with varying weighted percents of TiB\textsubscript{2} reinforcing particles: (a) 5 %, (b) 10 %, (c) 15 %](image)

3.3. Microhardness analysis of AA7075/TiB\textsubscript{2} aluminium matrix composites

The microhardness of various weighted percent (5%, 10%, and 15%) reinforcement as-cast AA7075/TiB\textsubscript{2} AMC was displayed in Fig. 5. It was seen that as the weighted% of TiB\textsubscript{2} reinforcement particle increases, the microhardness of the composite also increases. This is due to the fact that the rigidity of ceramic reinforcement increases as the weighted percent of TiB\textsubscript{2} reinforcement particles increases. The improved hardness of the composite is due to the good amalgamation between matrix and reinforcement, as well as the reinforcement’s fine size and uniform distribution. An increase in density of dislocation occurs as a result of the decrease in grain size which improves the resistance to plastic deformation [11]. The improvement in mechanical property is due to fine grain, according to the Hall-Petch relationship [14]. 5%wt reinforcement as-cast AA7075/TiB\textsubscript{2} had a minimum microhardness of 100 HV\textsubscript{0.1}, 10 %wt reinforcement as-cast AA7075/TiB\textsubscript{2} AMCs had a microhardness of 130 HV\textsubscript{0.1} and 15 %wt reinforcement as-cast AA7075/TiB\textsubscript{2} AMCs had a maximum microhardness of 150 HV\textsubscript{0.1}.

![Fig 5: Microhardness analysis of AA7075/TiB\textsubscript{2} with various weighted% of TiB\textsubscript{2} reinforcement particle](image)
3.4. Tensile strength analysis of AA7075/TiB₂ aluminium matrix composite

A tensile test was used to determine the mechanical characteristics of an AA7075/TiB₂ aluminium matrix composite material with varied weighted percents of reinforcement particles. The ultimate tensile strength (UTS) of 5% wt, 10% wt, and 15% wt reinforcement is illustrated in Fig. 6 (a). The ultimate tensile strength of the composite was found to grow as the percent weightage of the reinforcement increased. It is because of the increase in the quantity of high-strength TiB₂ particles. This is also due to the presence of reinforcement particles which have a different coefficient of thermal expansion property than the matrix composite. During the deformation of particulate reinforced composites, the matrix material bears the majority of the load; however, the reinforcing particles within the matrix bear the same stress and prevent matrix distortion. When dislocations bind to particle-matrix interfaces, the mobility of the dislocations is halted, resulting in stress concentration at the particle level. By which, improvement in UTS can be achieved. During in-situ synthesis, because of the evenly distributed TiB₂ particles, it gives Orowan strengthening mechanism as a result there is an increase in strength of AA7075/TiB₂ composite [15]. The polished interface and excellent joining delay the separation of particles from the AMCs. The minimum ultimate tensile strength of as-cast AA7075/TiB₂ aluminium matrix composite was 220 MPa, and the ultimate tensile strength of 10 weighted percent reinforcement as-cast AA7075/TiB₂ aluminium matrix composite was 260 MPa, and the maximum ultimate tensile strength of 15 weighted percent reinforcement as-cast AA7075/TiB₂ aluminium matrix composite was 290 MPa. From this observation, it can be concluded that there is a significant increase in UTS with the increment in weighted% of reinforcement particle.

In Fig.6 (b) the percentage elongation of 5 wt%, 10 wt% and 15 wt% reinforcement is shown. The percentage elongation diminishes with the increment in wt% of reinforcement. This is because of a decrease in ductile matrix content when wt % of TiB₂ particles increases in the matrix material. A decline in percentage elongation of AA7075/TiB₂ aluminium matrix composite likewise happens because of an increase in hard ceramic content. Ranjan et al. [8] also reported similar results. Minimum elongation of 3% was obtained in 15 wt percent reinforcement as-cast AA7075/TiB₂ AMC, 3.2 percent in 10% wt of reinforcement, and 4.9 percent in 5 %wt of reinforcement of as-cast AA7075/TiB₂ AMC.

3.5. Fractography analysis of AA7075/TiB₂ AMCs

The wrecked tensile specimens were examined in an electron microscope to investigate the reason for failure and fracture morphology. The fracture surface of the composite is shown in Fig.7 at various weighted percents of reinforcement (5 % wt, 10% wt, and 15% wt). Brittle fractures of AA7075/TiB₂ composite have been observed macroscopically and ductile fracture has been observed microscopically. The void size of the AA7075/TiB₂ composite was reduced during in-situ synthesis. This is due to TiB₂ reinforcement's grain refining activity. The ductile behavior of the fracture surface in A7075/TiB₂ material reduces as the grain size decreases. It was observed that in composite with 5wt% reinforcement, the fracture surface has voids with dimple indicating a ductile failure. In composite with 10wt% of reinforcement, large shape voids with fine dimple have been observed which indicates the failure in ductile mode. In composite with 15wt% of reinforcement,-dimple shaped void with layered morphology has been observed which indicates failure in ductile mode.
4. Conclusions

AA7075/TiB₂ AMCs were made in this study using in-situ reactions of KBF₄ and K₂TiF₆ salts. The impact of TiB₂ particles on the microstructural and mechanical properties of AA7075/TiB₂ AMCs has been thoroughly investigated. The following are some of the inferences that can be drawn:

- The AA7075/TiB₂ composite was effectively produced using TiB₂ reinforcing particles of 5%, 10%, and 15% by weight. There is no intermediary phase formed and only Al and TiB₂ are present, indicating that the reaction is complete.
- It can also be seen in the XRD pattern that when the weighted percent of reinforcing particles increases, the intensity of TiB₂ peaks increases.
- The reinforcement particles are evenly dispersed throughout the matrix, and the grain size decreases as the percentage weight of TiB₂ reinforcement particles increases.
- The ultimate tensile strength of the composite has improved as the percent weight of reinforcement has increased.
- Microhardness has increased as the percent weight of reinforcement in the composite has increased.
- Fractography examination of tensile specimens revealed a ductile mode of failure.

5. References

1. Nami H, Adgi H, Sharifitabar M, Shamabadi H., Mater Des 32, 976 (2010)
2. Alidokht SA, Zadeh AA, Soleymani S, Assadi H., Mater Des 32, 2727 (2011)
3. Sharifitabar M, Sarani A, Khorshahian S, Afarani MS., Mater Des 32(4), 64 (2011)
4. Kala, H., Mer, K. K. S., & Kumar, S., Procedia materials science, 6, 1951 (2014)
5. Soltani, S., Khosroshahi, R. A., Mousavian, R. T., Jiang, Z. Y., Boostani, A. F., & Brabazon, D., Rare Metals, 36(7), 581 (2017)
6. Srivatsan, T. S., Al-Hajri, M., Hotton, B., & Lam, P. C., Applied Composite Materials, 9(3), 131 (2002)
7. D. Sethi, S. Kumar, S. Choudhury et al., Materials Today: Proceedings, 2, 418 (2020) doi: 10.1016/j.matpr
8. Rajan, H. M., Ramabalan, S., Dinaharan, I., & Vijay, S. J., Materials & Design, 44, 438 (2013)
9. Fishman SG. In-situ composites. Science and Technology: TMS; 1994.
10. Gao, Q., Wu, S., Shulin, L. Ü., Duan, X., & An, P., Materials & Design, 94, 79 (2016)
11. A.K Gajakosh, R.Keshavamurthy, Ugrasen.G, Adarsh.H., Materials Today: Proceedings 5, 25605 (2018)
12. Liu, Z., Rakita, M., Wang, X., Xu, W., & Han, Q., Journal of materials research, 29(12), 1354 (2014)