

Evolution of Microstructure during the Preparation of TiB_2 Reinforced In-Situ Aluminium Matrix Composites

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Abstract: AA6082 aluminium alloy was reinforced with 5wt% of TiB_2 particles using the Flux Assisted Technique (FAS) using inorganic K_2TiF_6 and KBF_4 salts at 800°C. The evolution of the microstructure and the grain size effect was investigated by varying the reaction time of the salt with aluminium alloy melt. The reaction time was varied from 0-90min in steps of 30min. At lower reaction time the presence of more number of Al_3Ti , AlB_2 particles were predominantly observed. As the reaction time increased the formation of more number of TiB_2 particles in the matrix alloy was evident. At lower reaction time of 30min the grain size of the composite decreased compared to the matrix alloy due to the presence of more number of effective Al_3Ti nucleant particles, whereas at higher reaction time increase in grain size was observed due to the absence of Al_3Ti particles, which dissociates to form TiB_2 particles.

Keywords: AA6082- TiB_2 composite, in-situ processing, microstructure evolution.

I. INTRODUCTION

Aluminium metal matrix composites (AMCs) are known for their superior strength to weight ratio and strength to cost ratio compared to the monolithic aluminium alloys. Due to the above mentioned characteristics of AMCs they are mostly preferred in aerospace and automobile industries where materials with superior properties with less weight are preferred. Depending on the nature of the reinforcements added, AMCs can be classified as fiber reinforced AMCs and particulate reinforced AMCs [1-4] [1-4]. Although the fiber reinforced AMCs were found to be superior compared to particulate reinforced AMCs, tedious processing methods and high cost of fiber reinforced AMCs have made researchers to find new ways, which resulted in the development of particulate reinforced AMCs [5]. Since then considerable attention has been paid to development of particulate reinforced AMCs having superior mechanical properties such as high specific strength, specific modulus and hardness [6].

Based on the processing method, the particulate reinforced AMCs can be classified as ex-situ and in-situ composites. The ex-situ composites are prepared by adding prior prepared particulates into the aluminium matrix [7], whereas the in-situ composites the reinforcements are precipitated inside the matrix by a suitable exothermic chemical reaction [8]. The in-situ composites were developed to overcome the discrepancies such as interfacial reaction and poor wettability between the matrix and reinforcements observed among the ex-situ composites [9, 10]. Among the available processing methods to prepare in-situ composites, the liquid metallurgy has been found to be more economical and easy method for the processing of particulate reinforced AMCs. Compared to the ex-situ processed composites the in-situ processed particulate reinforced AMCs offer the following advantages such as, the formation of

thermodynamically stable reinforcements, good bonding between the matrix and reinforcements because of the clean interface between the particle and matrix and particulates with fine size leading to uniform distribution of particles. In-situ composites, in particular TiB_2 reinforced AMCs by flux assisted synthesis (FAS) developed by London Scandinavian Metallurgical company (LSM) has been the latest topic of research in the field of metal matrix composites (MMCs). In the FAS technique the TiB_2 reinforcements are formed inside the matrix melt by suitably adding Ti and B based K_2TiF_6 and KBF_4 salts. The high exothermic reaction between the added salts and the melt helps in the dissociation of the salts thereby forming TiB_2 reinforcements [11, 12]. The formation of TiB_2 reinforcements in the final composite is not a direct process. There is always a possibility of the intermediate phases such as Al_3Ti and AlB_2 to be present in the matrix alloy [13]. Presence of these intermediate phases can be avoided by carefully controlling the processing parameters. Among the available processing parameters the reaction time of the salts with the aluminium alloy melt plays a major role in the formation of the TiB_2 reinforcements. Hence in the present investigation the effect of reaction time on the microstructural changes in the processed composite has been carried out.

II. EXPERIMENTAL PROCEDURE

In the present study Al-Si-Mg aluminium alloy was selected as the base alloy since this alloy offers a wider range of mechanical properties, good machinability, weldability, formability and highly corrosion resistant. The chemical composition of the procured alloy is shown in Table.1. Among the variety of ceramic reinforcements available TiB_2 has been chosen as reinforcement since it

possesses good properties like high hardness, high strength to density ratio, high electrical conductivity, and high thermal conductivity and majorly it avoids the formation to interfacial reaction products with aluminium. The procured alloy was melted in the graphite crucible in a resistance furnace, which was set to a temperature of 800°C. The temperature of melt was carefully observed by using K-Type thermocouple. Commercially pure K₂TiF₆ and KBF₄ fluoride salts were blended so as to achieve a stoichiometry of Ti and B ratio of 2.2:1. The blended fluoride salts were heated to a temperature of 250°C for 60 min to remove the moisture from the fluoride salts. When the melt temperature reached thermal equilibrium with the chosen temperature on the furnace, the melt was degassed with hexachloroethane (C₂Cl₆) tablet. The preheated K₂TiF₆ and KBF₄ were added to the aluminium melt by wrapping the powders in aluminium foil. The K₂TiF₆ and KBF₄ reacted with the molten metal to form TiB₂ particles by the exothermic reaction.

The melt was stirred every 10 minutes using zirconia coated mild steel rod. The reaction time of the fluoride salts with aluminium melt was varied from 30-90 minutes in steps of 30 min. With the completion of the reaction time the dross was decanted and the melt was degassed with hexachloroethane tablet and poured into a preheated (250°C) mild steel mould of diameter 30mm and height 170mm.

The base alloy and prepared composites were cut and machined to obtain samples for characterization studies. The cut samples were characterized by X-ray diffraction (XRD) techniques for the identification of the phases. The samples were polished and etched using Keller's reagent. The etched samples were used for microstructural studies using Optical microscopy (OM), scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS).

Table 1 Chemical composition of as-cast AA6082 alloy

Alloy	Matrix Chemical Composition (Wt. %)					
	Mg	Si	Mn	Cr	Fe	Al
AA6082	1.02	1.30	0.47	0.15	0.17	Bal

III. RESULTS AND DISCUSSION

Microstructure of the as cast matrix alloy shown in Fig.1 (a) clearly exhibits the formation of grain with dendritic in nature moreover it is evident that these dendrites are primary dendrites which have an elongated morphology. Optical micrograph of the composite prepared at 30min shows the presence of Al₃Ti, AlB₂ and TiB₂ particles (Fig.1b).

The Al₃Ti particles were found to be blocky in morphology with the different sizes ranging from 20 to 40µm. AlB₂ particles formed at this reaction time were of fine blocky morphology with size ranging from 4 to 10µm. The TiB₂ particles were found to be very fine in size ranging from sub micrometer to a maximum of 3µm.

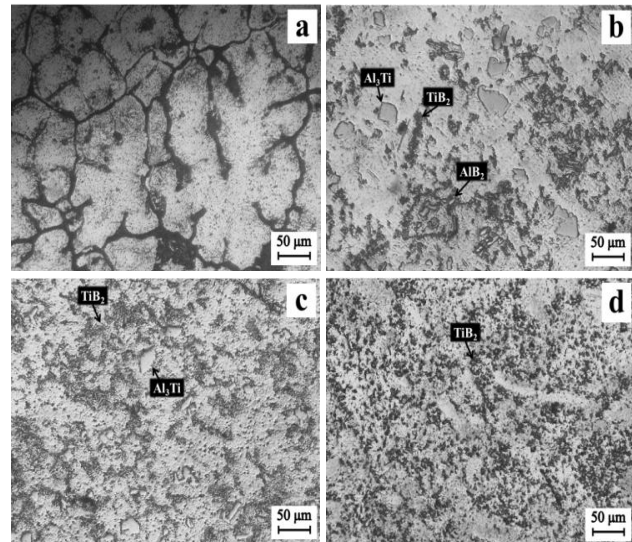


Fig. 1. Optical microstructure of alloy and composite: (a) AA6082, (b)30min, (c)60min, (d)90min.

The TiB₂ particles formed at this temperature range were found to be clustered. The X-ray diffraction pattern obtained from the composite prepared at 30min of reaction time shown in Fig.3a further supports the results obtained from the micrographs. The presence of Al₃Ti peaks with higher intensity indicates that more of Al₃Ti particles are present in the composite. The reduced intensities of TiB₂ and AlB₂ further confirm that they were present less in quantity at this reaction temperature. Upon the addition of the K₂TiF₆ and KBF₄ salts to the melt, Ti and B get dissolved in the molten Al by exothermic reaction of the fluoride salts. The dissolved Ti and B in the aluminium melt precipitate as AlB₂, Al₃Ti, and TiB₂ particulates.

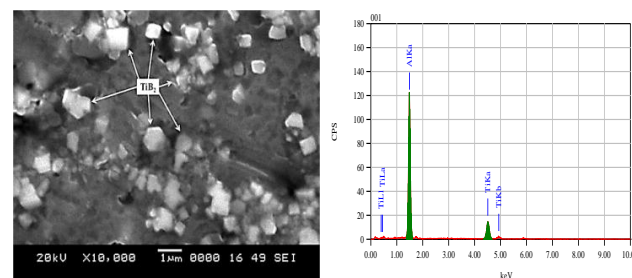


Fig. 2: SEM photomicrograph (a) TiB₂ particle (b) EDS spectrum of TiB₂ particle in alloy.

As the reaction time was increased from 30 to 60min, the Al₃Ti particles present in the composite were found to be smaller than 15µm (Fig.1c). The AlB₂ particles in the microstructure are found to be absent at this reaction temperature. The X-ray diffraction analysis on the composite prepared at 60min shows the presence of only two type of particles Al₃Ti and AlB₂ present in the matrix alloy.

This XRD results (Fig 3c) further validates the results obtained from the microstructural analysis. The decrease in the intensity of Al₃Ti peaks in the composite prepared at 60 min confirms that for this reaction time the Al₃Ti and AlB₂ particles have begun to dissociate thereby transforming into TiB₂ particles.

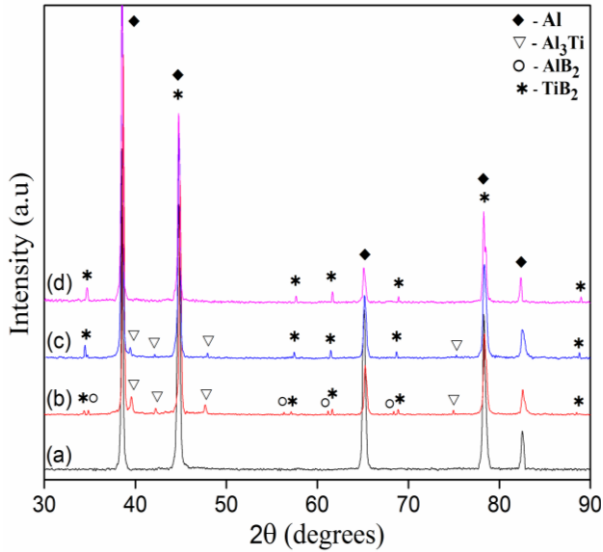


Fig. 3. XRD pattern of as cast base alloy and Al composite for a different reaction time: (a) AA6082, (b) 30min, (c) 60min, (d) 90min.

The increase in intensities of TiB_2 peaks with a reaction time of 60 min further confirms the formation of more number of TiB_2 particles compared with the reaction time of 30min. Fig.1 (d) shows the optical micrographs of composite prepared with a reaction time to 90min at a temperature of $800^\circ C$. The micrograph shows the presence of only TiB_2 particles in the matrix alloy. Moreover the presence of only TiB_2 peaks in the XRD pattern obtained from composite further confirms the complete formation of TiB_2 particles (Fig.3d). The increase in intensities of TiB_2 peaks (Fig.3b-d) as the reaction time is increased clearly suggest that more of TiB_2 is formed in the matrix alloy.

The SEM micrograph shown in Fig.2 (a) confirms the presence of TiB_2 particles which were further confirmed by the EDS technique. The TiB_2 particles had an average size of $1\mu m$ where both hexagonal and cubic morphology of the particles were observed. The presence of Al peaks along with Ti peaks is mainly due to the signals received from the surrounding matrix alloy. The absence of boron peaks in the EDS spectrum indicates is due to its lower atomic number and hence it is not being detected by the detector (Fig. 2b). The micrographs and the XRD pattern obtained from the composites prepared at different reaction time suggest that with the addition of salts to the matrix melt Al_3Ti and AlB_2 primarily precipitated in the melt. On further increase in the reaction time the primarily precipitated particles slowly begins to dissociate and this leads to the formation of TiB_2 particles.

Fig. 4 shows the grain size distribution histogram obtained from the as cast alloy and composite prepared at different reaction time. The grain size of as cast alloy was found to be $151\mu m$. The grains were found to dendritic in nature. The composite prepared at 30min showed a grain size of $63.5\mu m$. There has been a drastic decrease in grain size in the composite prepared at 30min compared to the base alloy.

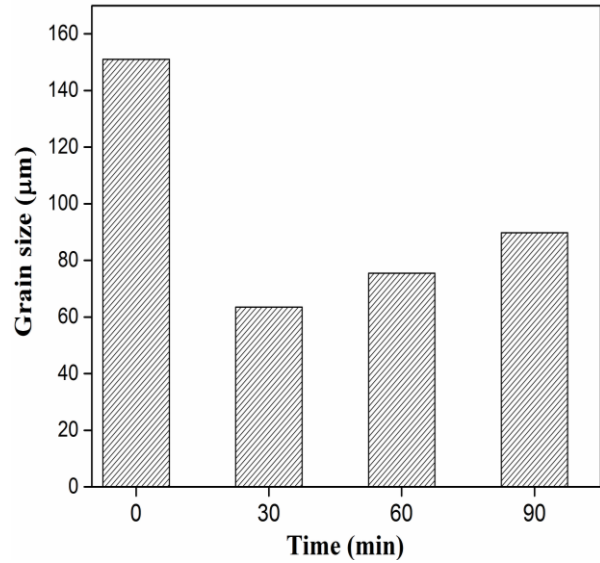


Fig.4. Histograms showing the grain size distribution of Alloy and composite prepared at different reaction time.

The grain size of composite prepared at 60min showed a grain size of $75.5\mu m$. The composite prepared at 90min showed grain size of $89.8\mu m$ which is marginally higher than the composite prepared at 60min.

From the histogram it is evident that as the reaction time increases, there is also an increase in the grain size in the composites. The cause for the decrease in the grain size of the composite prepared at 30min is due to the presence of large number of Al_3Ti , AlB_2 and TiB_2 particles.

As the reaction time increased the dissociation of Al_3Ti and AlB_2 particles led to the formation of TiB_2 particles, thereby decrease in the number of particles needed for the nucleation of the grains.

Hence there is an increase in the grain size of composite prepared at 90min compared to the composite prepared at 30min. It is also well established that the Al_3Ti particles were found to a better nucleant than the TiB_2 particles.

IV. CONCLUSION

The microstructural evolution during the preparation of in-situ AA6082-5wt. % TiB_2 composite has been investigated at different reaction time of 30, 60 and 90min for a constant temperature of $800^\circ C$. The presence of Al_3Ti and TiB_2 phases were confirmed by XRD, SEM and EDS. At lower reaction time the formation of Al_3Ti , AlB_2 and TiB_2 was observed.

At the reaction time of 90min, only TiB_2 particles were observed. This confirms that there has been a complete dissociation of Al_3Ti and AlB_2 particles forming TiB_2 particles. Upon the addition of particles the grain size of the matrix alloy decreased from 151 to $63.3\mu m$. The grain size of the composite prepared at higher reaction time of 60 and 90min increased due to absence of Al_3Ti and AlB_2 particles.

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