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Influence of process parameters of aluminothermic reduction process on grain refinement of in-situ Al/ TiB₂ composites

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Abstract

This paper reports the effect of process variables such as holding time, addition time on grain refinement on in-situ Al/TiB₂ composite and also size of the formed TiB₂ particle size. The in-situ composites were fabricated by flux assisted synthesis route using potassium hexafluorotitanate (K₂TiF₆) and potassium fluoroborate (KBF₄) salts as precursors. The composites were produced with respect to different holding time varies from 20 min to 40 min and also for different addition times of precursors 10 min to 30 min at 700°C. The resulting composites were characterised by microstructure examination and size of particles. The TiB₂ particles are segregated by crushing and followed by acidic immersion. From the results, effects of the process parameter on the particle formation and particle sizes on grain refinement of in-situ Al/TiB₂ were investigated.

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Keywords: in-situ reactions, Al/TiB₂ composite, Grain refinement.

1. Introduction

Aluminium based metal matrix composites (Al MMCs) are widely used in structural and electronic packaging application due to its characteristics such as high strength to weight ratio, high wear resistance and also high temperature resistance.

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Al MMCs have been successfully produced by several techniques including liquid state metallurgy[1]. However, the composite fabricated by this method suffers due to few technological issues such as non-uniform distribution of particles, improper wettability between reinforcement and matrix, interface reactions. In order to overcome these problems, several in-situ reactions were developed. The recent studies show that the composite formed by in-situ routes resolved most of problems faced by ex-situ route[2]. Further, it is demonstrates that the in-situ formations of particles refine the grain during solidification [3].

In recent years, most of researchers have studied Al/ TiB₂ in-situ composite due to its characteristics such as high wear resistance, chemical and thermal stability. It can be synthesised from various in-situ methods such as solid-solid, solid-liquid, liquid-liquid and vapour-liquid-solid reaction. Among these solid-liquid route has been widely employed to produce Al/TiB₂ composite. Madhavan et al.[4] fabricated Al/TiB₂ composite through in-situ reaction with respect to different process parameters such as temperature and holding time. They reported that grain refinement occurred even at higher holding time and also found out no boron loss occurred at higher temperature. Wang et al.[5] investigated about effect of Ti and B ratio on grain refinement of Al/ TiB₂ composite. They reported that higher grain refinement was achieved when there was excess amount of titanium than stoichiometry ratio. Mallikarajuna et al.[6] reported that grain size of Al/TiB₂ composite was decreased efficiently upto 30 min holding time, afterwards it is followed by gradual reduction of the grain sizes . They also found out that co-efficient of friction was decreased when there is an increase in holding duration. However, a detailed study of influence of each of the process parameters on the grain refinement of Al/TiB₂ composite has not been reported.

This paper reports that influence of process parameters on grain refinement of in-situ Al/TiB₂ composites and also an effect of particle size on grain refinement of com

2. Experimental procedure

Al/5wt.%TiB₂ composite was fabricated by using halide salts such as Potassium Hexafluorotitanate (K₂TiF₆) and potassium Potassium Fluoroborate (KBF₄). Initially weight quantity of Al (A380) alloy was melted in electric resistance furnace which was used as matrix material. The weight quantity of K₂TiF₆ and KBF₄ as per the stichiometric calculation to form 5wt. % TiB₂. The mechanical stirring was carried out while addition of salts for effective reaction and also achieve the good dispersion of TiB₂ particles in the matrix. The stirring time and holding time were also same, which was 20, 30 and 40 minutes. The K₂TiF₆ and KBF₄ were added with different addition time of salts which were 10, 20 and 30 minutes. After completing the reaction, the composite slurry was poured into a steel die which was preheated upto 300°C. The microstructure analysis of as cast samples was carried out by using optical microscope (Metavis model MM25 IS, Japan) with image analysis software. The particle formation was confirmed by using XRD.The size distributions of extracted particles were estimated out using Malvern Zetasizer nano analyser (Zetasizer ver.7.1, UK).

3. Results and discussions.

3.1. XRD analysis

Fig. 1 shows the XRD peaks for Al/TiB₂ composites processed at 20 min holding time and 20 min addition time. From this result, formationTiB₂ particles were confirmed.

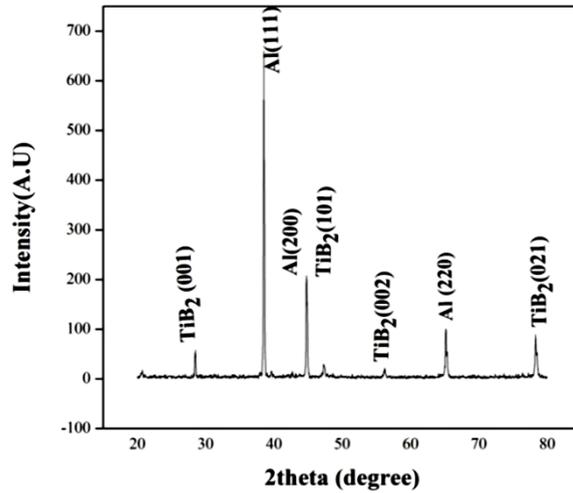


Fig. 1. X-ray diffraction pattern for Al/TiB₂ composite processed at 20 min holding and addition time.

3.2. Microstructure analysis

Fig. 2a and 2b represent the microstructure and grain size distribution of Al (A380) alloy. From this result, dendrite structure dominantly formed in the Al alloy and measured grain size was 45 μ m which was higher than the Al/TiB₂ composites.

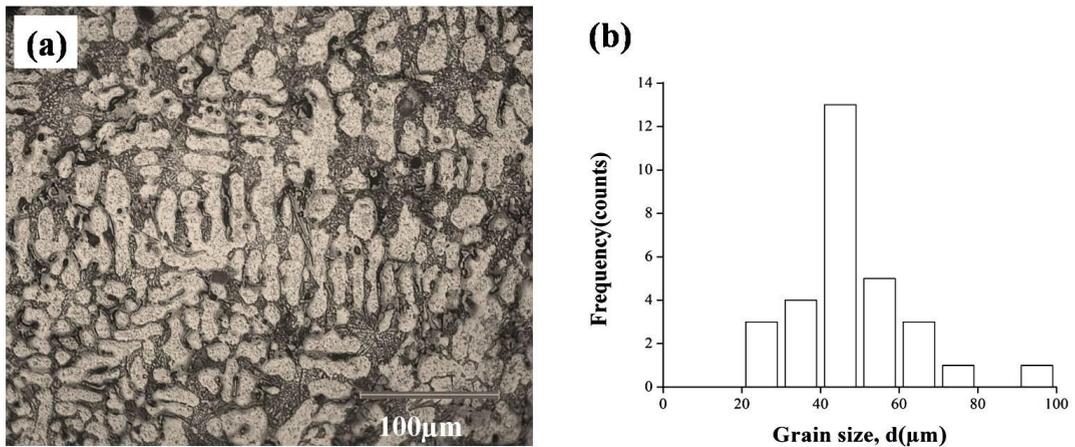


Fig. 2. (a) Microstructure of Al (A380) Alloy (b) Grain size distribution of Al alloy.

In Al/TiB₂ composites, growth of Al dendrites was restricted by formation of TiB₂ particle during solidification which was shown in fig. (3-5). Fig 3a and 4a, the grain size was decreased when there was an increase in the addition time from 10 min to 20 min respectively. This is due to formation of finer particles lead to increase in number of particles which nucleates more amount of α -Al grains. Further increase in addition time, grain size might be increased due to higher holding time with formation of bigger size particles and more particle formation which was shown in fig 3c and 5c. Therefore refinement of grains was due to combine effect of holding time and addition time.

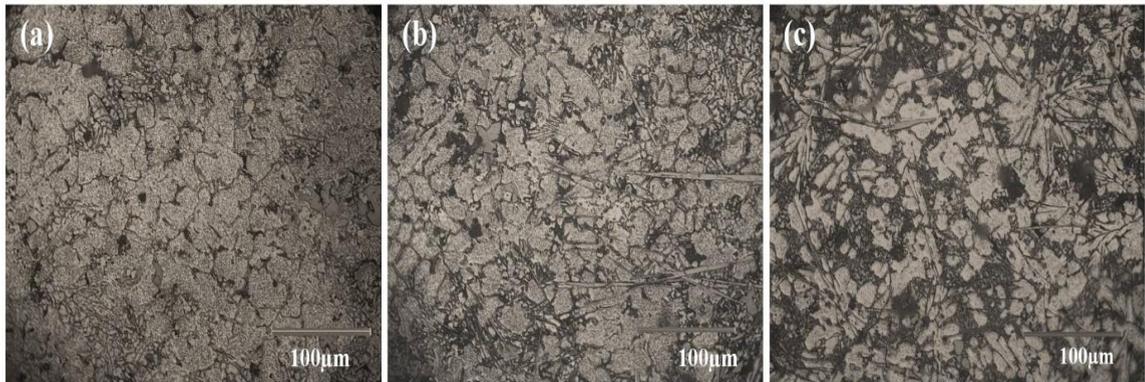


Fig. 3. Microstructure of Al/TiB₂ composite fabricated at 10 min addition time. (a) 20 min holding time (b) 30 min holding time (c) 40 min holding time

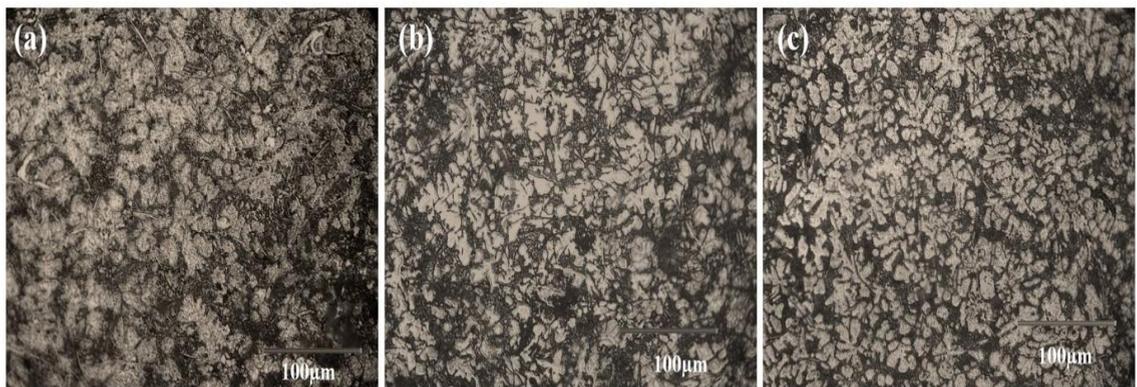


Fig. 4. Microstructure of Al/TiB₂ composite fabricated at 20 min addition time. (a) 20 min holding time (b) 30 min holding time (c) 40 min holding time.

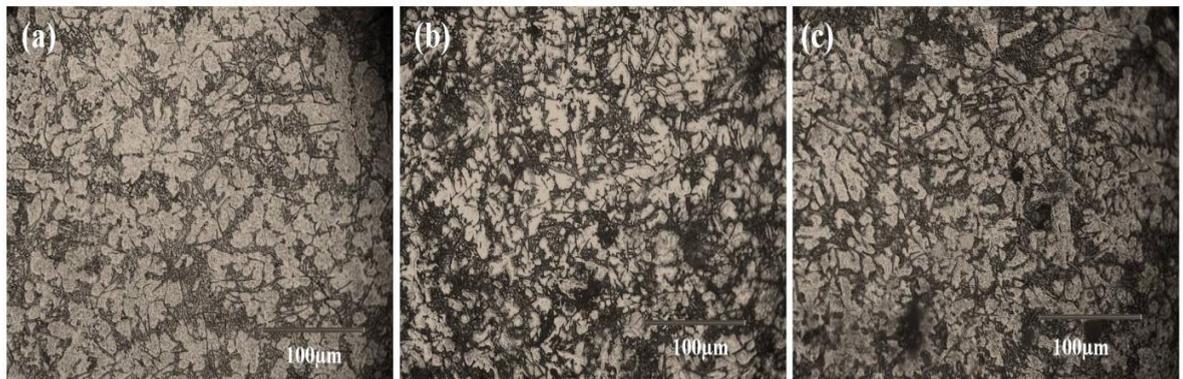


Fig. 5. Microstructure of Al/TiB₂ composite fabricated at 30 min addition time. (a) 20 min holding time (b) 30 min holding time (c) 40 min holding time.

Fig 3(a-b) and 4(a-b) shows that grain size was decreased when there was increase in holding time from 20min to 30 min, further it increases, grain size was increased due to formation of more TiB₂ particles leads to settling the particle at bottom of the cast to form agglomeration during solidification. But Fig.4c, grain size was reduced even at higher holding time due to addition time leads to formation of finer particles, followed by an increasing the number of particles in the melt[7]. Fig. 6a and 6b shows the average particle size and average grain size of composite respectively.

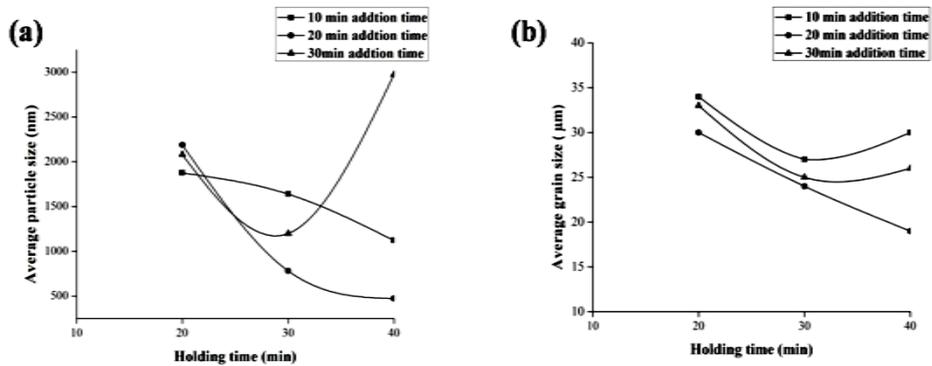


Fig. 6 (a) Average particle size of TiB₂ particles (b) Average grain size of Al/TiB₂ composite.

Conclusion.

Al/ 5wt. %TiB₂ composite fabricated through in-situ reactions of halide salts. The grain refinement was low at higher (40 min) and lower holding time (20 min), due to incomplete reaction and formation of higher particle sizes.

Grain refinement was good at 30 min holding time and 40 min holding time with 20 min addition time due to formation of finer particles, complete reaction with uniform dispersion of particles.

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