

Effect of Titanium and Boron weight ratio on the microstructure of Al1050-5%TiB₂ in-situ composites

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Abstract

A thorough investigation on the salt stoichiometry of the added fluoride salts to the melt was carried out in an attempt to prepare In-situ Al1050-5wt.% TiB₂ composite. It is well known that addition of fluoride based K₂TiF₆ and KBF₄ salts to the aluminium melt produces TiB₂ particles. The stoichiometric ratio of the fluoride salts which were added to the melt was changed and the obtained results were analyzed. Addition of slightly more KBF₄ salts to the melt produced better results yielding only TiB₂ particles. However more addition of KBF₄ salts resulted in formation of more amount of entrapped slag in the composite. The stoichiometry of the salts decides the final reinforcement product in the composite.

Keywords – stirring, particulate, manual, mechanical.

1 INTRODUCTION

Metal matrix composites were developed in response to the increasing mandate for weightless and tougher materials. When compared to monolithic alloys, MMCs were created by adding the beneficial properties of certain metals and specific ceramics reinforcements, resulting in higher

strength-to-cost ratios and strength-to-weight. MMCs are unusual in that the desired qualities for a specific application may be modified by choosing the right matrix, reinforcements, and manufacturing processes. MMCs are classified as continuous fiber reinforced MMCs or discontinuously reinforced MMCs based depending on the type of reinforcements incorporated into the matrix [1].

2 Experimental Details

The studies were accepted out to determine the impact of fluoride salts in the melt on the production of Al-5wt% titanium di boride composite. Using a resistive furnace, the Al rods in the graphite crucible was melted. Throughout the experiment, a consistent composite with a melt holding time of sixty minutes was maintained. To the aluminium melt, the stoichiometrically weighed salts with different Ti:B ratio, which has been preheated K₂TiF₆ and KBF₄ salts was added. The interaction between the melt and the salts has been catalysed by discontinuous stirring at equal intervals of eight minutes. The slag which has been floating on upper part of the composite melt was detached after the required melt holding temperature was reached, and the melt of the composite was casted in the warmed die which was made of mild steel[6]. At over a temperature of 850°C for sixty minutes, the composite castings were made with different Ti:B ratio. The as-prepared samples were characterized using X-ray diffractometer (XRD), and scanning electron microscopy (JEOL JSM-840A EDX attached with, SEM).

3. Results and discussions

3.1 XRD analysis on the composite processed with different Ti:B ratio

The X-ray diffraction studies which were performed during the processing of Al1050-5%TiB₂ composites with a Ti:B ratio of 0.55 and with a processing temperature of 850°C for a constant time of 60 minutes is shown in Fig.1.1. The K₂TiF₆ and KBF₄ salts were added according to the stoichiometric calculations. The XRD pattern obtain from the composites processed from the above conditions shows the peaks of Al, TiB₂, Al₃Ti and AlB₂ phases which indicate that at the above mentioned conditions there is no complete transformation of the fluoride salts to the TiB₂ phase[2]. The presence of intermediate phases which further indicates that the transformation is

not complete at Ti:B ratio of 0.551. Moreover the high intensity of Al_3Ti peaks in the Fig. 1.1 further confirm that there is more formation of Al_3Ti particles than the AlB_2 particles. The high intensity of Al_3Ti peaks in the XRD analysis also indicated that the boron in the melt has been considerably reduced; hence there is formation of more Al_3Ti particles than AlB_2 particulates. The presence of TiB_2 peaks shows that there is meager amount of TiB_2 particles have formed during the reaction process.

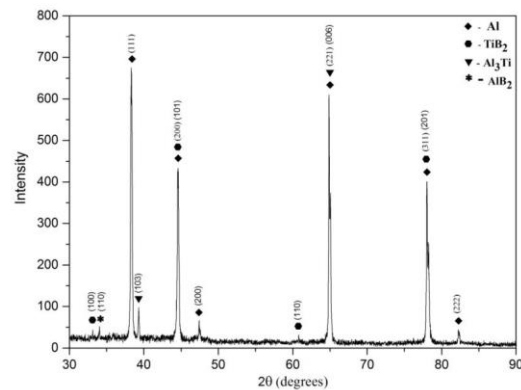


Fig.1.1 XRD pattern of *in-situ* composite prepared at stoichiometric mixture of K_2TiF_6 and KBF_4 salt

The X-ray diffraction studies which were performed during the processing of $Al_{10}50-5\%TiB_2$ composites with a Ti:B ratio of 0.503 and with a processing temperature of $850^\circ C$ for a constant time of 60 minutes is shown in Fig.1.2.

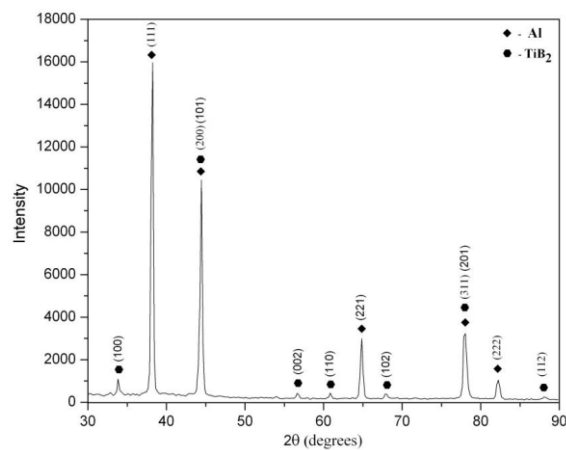


Fig. 1.2 XRD pattern of *in-situ* composite prepared at 10wt.% of excess KBF₄ (Ti:B molar ratio of 0.503)

In the above conditions the during the preparation of K₂TiF₆ and KBF₄ salts an excess of 10% KBF₄ salts is added. Under these processing conditions the X-ray diffraction image shows the only TiB₂ peaks which indicate that even at 850°C of melt temperature there is complete transformation of the fluoride salts to the TiB₂ phase[3]. The absence of intermediate phases which further indicates that the transformation is complete with the addition of excess 10% KBF₄. Moreover the absence of other intermediate peaks in the Fig. 1.2 has reduced indicating that the transformation is proceeding in the right direction.

The reduction in the peaks of AlB₂ and Al₃Ti peaks to TiB₂ in the XRD analysis as shown in Fig.1.2 compared to the peaks in Fig.1.1 clearly indicated that the more of boron in the melt has been considerably reduced to TiB₂ particles, which is further confirmed by the XRD analysis.

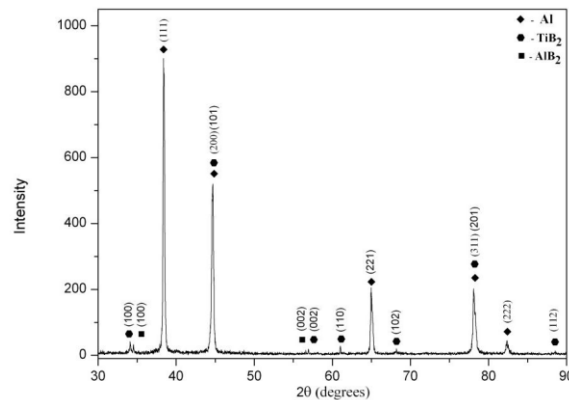


Fig. 1.3 XRD pattern of *in-situ* composite prepared at 20% KBF₄ salt to the stoichiometry mixture of K₂TiF₆ and KBF₄ salts (Ti:B molar ratio of 0.459)

The X-ray diffraction studies which were performed during the processing of Al1050-5%TiB₂ composites with a Ti:B ratio of 0.459 and with a processing temperature of 850°C for a constant time of 60 minutes is shown in Fig.1.3. In the above conditions the during the preparation of

K₂TiF₆ and KBF₄ salts an excess of 20% KBF₄ salts is added. Under these processing conditions the X-ray diffraction image shows peaks of both AlB₂ and TiB₂ peaks which indicate that even at 850°C of melt temperature there is incomplete transformation of the fluoride salts to the TiB₂ phase[4]. The presence of intermediate phases which further indicates that the transformation is incomplete with the addition of excess 20% KBF₄. It can be seen that on excess addition of Boron to the melt not only resulted in the formation of TiB₂ particles, it has also resulted in the formation of more AlB₂ particles, because of boron.

The X-ray diffraction studies which were performed during the processing of Al1050-5%TiB₂ composites with a Ti:B ratio of 0.424 and with a processing temperature of 850°C for a constant time of 60 minutes is shown in Fig.1.4.

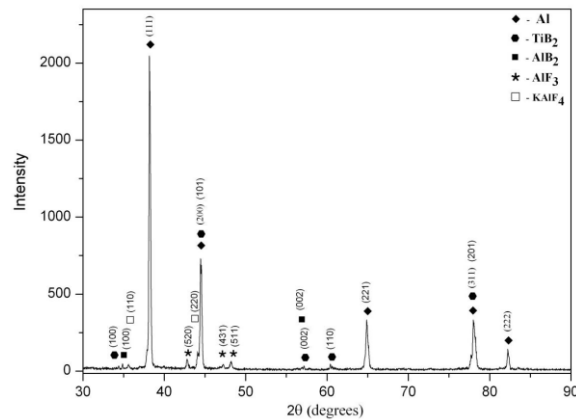


Fig. 1.4 XRD pattern of *in-situ* composite prepared with 30wt.% of excess KBF₄ salts (Ti:B molar ratio 0.424)

In the above conditions the during the preparation of K₂TiF₆ and KBF₄ salts an excess of 30% KBF₄ salts is added. Under these processing conditions the X-ray diffraction image shows peaks of AlB₂, TiB₂, KAlF₄

and AlF₃ peaks. The XRD on the prepared composite confirm that with the excess addition of KBF₄ salts more amount of slag is formed in the composite, which tends to get trapped inside the melt[5]. The presence of slag in the melt will deteriorate the material properties, hence more addition of salts than the required level should be avoided. Moreover, addition of more number of salts resulted in high viscosity of the melt which created problems during the pouring of the melt.

3.2 Scanning Electron Microscopy

Scanning electron microscope plays a vital role to study the surface property of the samples.

The temperature of the melt has a huge influence on the preparation of the composite. The above photomicrograph obtained using SEM were from the prepared composite at different temperatures. The sample processed with K_2TiF_6 and KBF_4 salts which were added to the alloy melt according to the stoichiometric calculations i.e. Ti:B molar ratio of 0.551 is shown in Fig.1.5a clearly shows that the particles have formed during the process.

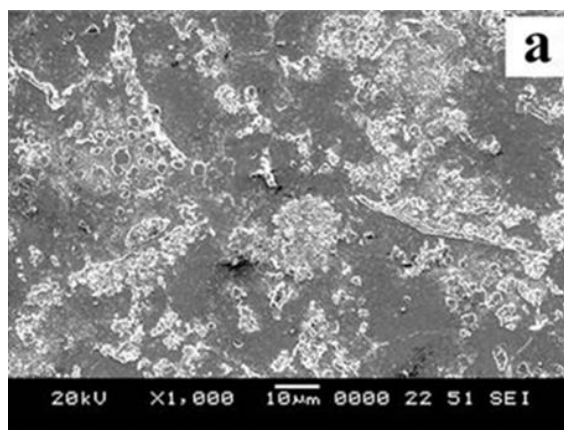


Fig.1.5a SEM, from the prepared composite at different Ti:B ratio stoichiometric mixture of K_2TiF_6 and KBF_4 salt

The formed particles were of different size and morphology. The string like particles which are seen the photomicrograph was found to be having the elemental signature of Ti, Al and Si. The energy dispersive spectrum confirmed that some of the particles were TiB_2 and Al_3Ti . In the Fig.1.5b, fine blocky type particles are seen.

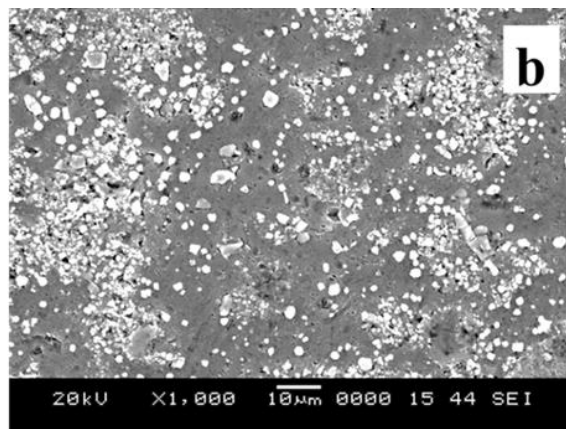


Fig.1.5b SEM, from the prepared composite at different Ti:B ratio of excess 10% KBF_4 .

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The EDS analysis on the particles confirmed that they were all TiB₂ particles. The sizes of the TiB₂ particles were found to be in the range of nano meter to less than 1 μm . The TiB₂ does not form directly from the exothermic reaction of the salts with the melt. First as the exothermic reaction between the salts and melt occur the Ti and B is released from the salts. The Ti and B react with aluminium and form Al₃Ti and AlB₂. As the melt temperature is held for a consistent amount of time, the Al₃Ti and AlB₂ further decomposes and forms as TiB₂. The SEM image of composite processed with 20%KBF₄ which has a Ti:B molar ratio of 0.459 is shown in Fig.1.5c. Analysis on the image shows that there is formation of more number of AlB₂ particles along with TiB₂ particles.

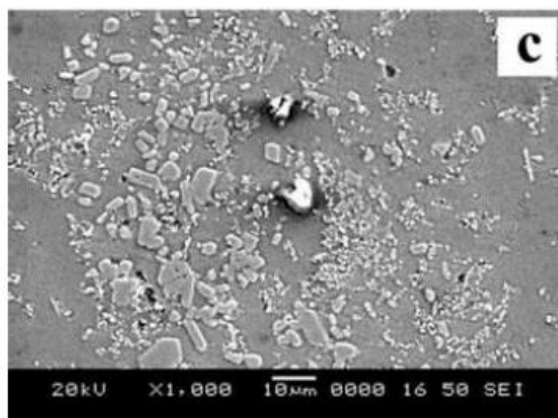


Fig.1.5c SEM, from the prepared composite at different Ti:B ratio of 20%KBF₄.

The observation on the image also reveals that along with the formed particles the presence of slag is also evident. It can also be observed that with excesses addition of salts more amount of slag is formed.

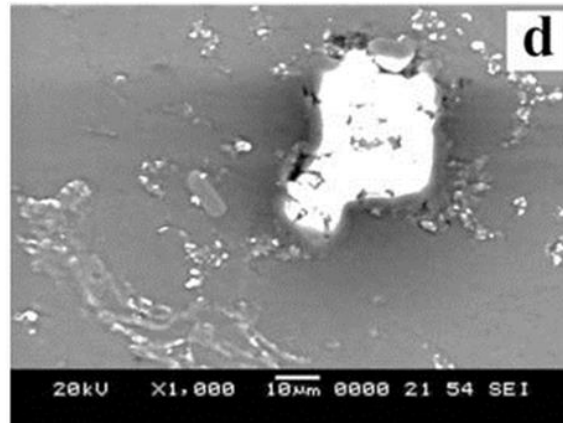


Fig.1.5d SEM, from the prepared composite at different Ti:B ratio of 30% KBF₄

4. CONCLUSION

Overall, the prepared composites at different Ti:B molar ratio had greater impact on the microstructural and mechanical properties than the unreinforced alloy, according to the findings. Several causes can be attributed to the properties of the composites:

- (i) Addition of 10% excess KBF₄ salts to the stoichiometry mixture of K₂TiF₆ and KBF₄ salts was found to be desirable condition for production Al1050-5%TiB₂ composite.
- (ii) Addition of excess KBF₄ salts to the stoichiometry mixture of K₂TiF₆ and KBF₄ salts has led to formation of more unwanted AlB₂ particles and
- (iii) Excess addition of more KBF₄ salts to the stoichiometry mixture of K₂TiF₆ and KBF₄ salts led to presence of more unwanted slag in the prepared composite.
- (iv) The presence of slag in the composite has considerably reduced the properties of the composite.
- (v) Adding excess amount of KBF₄ salts increased the viscosity of the melt which led to the difficulty in pouring of the composite melt

References

1. Abdel Hamid, A. and Durand, F. (1985). "Liquid-Solid equilibria of Al-rich Al-Ti-B alloys, liquid-Solid Equilibria of Al-Rich Al-Ti-B Alloys, Part 2: Crystallization sequence leading to TiB₂ crystals." *Z. Metallkd.*, 76, 739-746.
2. Birol, Y. (2006). "The effect of processing and Mn content on the T5 and T6 properties of AA6082 profiles." *J. Mater. Process. Technol.*, 173(1), 84-91.
3. Brinkman, H. J., Duszczek, J. and Katgerman, L. (1997). "In-situ formation of TiB₂ in a P/M aluminium matrix." *Scripta Mater.*, 37(3), 293-297.
4. Chen, Z. Y., Chen, Y. Y., Shu, Q., An, G. Y., Li, D. and Liu, Y. Y. (2000). "Microstructure and properties of in situ Al-TiB₂ composite fabricated by in-melt reaction method " *Metall. Mater. Trans. A*, 31A, 1959-1964.
5. Christy, T. V., Murugan, N. and Kumar, S. (2010). "A comparative study on the microstructures and mechanical properties of al 6061 alloy and the MMC al 6061/TiB₂/12P." *Journal of minerals & Materials Characterization & Engineering.*, 9(1), 57-65.
6. Rahul Mitra and Yashwant R. Mahajan, "Interfaces in discontinuously reinforced metal-matrix composites," *Defence Sci J*. Vol 43, pp. 397-418, 1993.
7. A. E. Karantzalis, A. Lekatou, E. Georgatis and H. Mavros, "Solidification behaviour of ceramic particle reinforced Al alloy matrices," *J. Mater. Sci*. Vol. 45, pp. 2165-2173, 2010.
8. A.K.Kuruvilla, K.S.Prasad, V.V. Bhanu prasad and Y.R.Mahajan, " Microstructure-property correlation in Al/TiB₂ (XD) composites, " *Scripta Metall Mater*. Vol. 24, pp. 873-878, 1990.