Research Paper

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The effect of Melt Temperature on changes in the microstructure of Al1050-5%TiB_2 in-situ composites

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Abstract

In-situ Al1050-5wt.% TiB2 composite was prepared by flux assisted synthesis (FAS) technique using potassium hexaflurotitanate (K2TiF6) and potassium tetrafluroborate (KBF4) salts at different melt holding temperatures of 750ºC to 900ºC for a constant holding time of 60 minutes. The decomposition of salts with respect to temperature was determined by DTA analysis. The influence of holding temperature on porosity, microstructure and particle morphology on the prepared composite was investigated. The composite prepared at 800ºC, showed an increase the particles average size. The porosity present in prepared composite was found to increase with higher melt holding temperature. The results confirmed that at higher holding temperatures a complete reaction between the halide salts and the aluminium occurred, leading to the nucleation and growth of only TiB2 particles in the final alloy composite. Moreover, at higher temperatures the particles which were formed in the melt were dispersed more homogenously in the matrix of the processed composite.

Keywords – stirring, particulate, manual, mechanical.

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1 INTRODUCTION

The FAS process evolved from a well-known technique for producing master alloys such as Al-Ti-B, which were employed in refinement of grains in aluminium and related alloys [4]. In-situ Al/ Al3Ti composites are commonly made using the FAS technique by adding Ti-bearing fluoride salts like potassium hexafluortitanate (K2TiF6) into the melt of aluminium, where reinforcements such as Al3Ti are nucleated and grown by the separation of Ti which is existing in the halide salts via a chemical reaction which is exothermic between the melt of aluminium and the added halide salts [1]. The resulting reinforcements which are TiB2 were found to be very fine, distributed well, and well-bonded to the matrix alloy. Due to a lack of understanding of the process parameters, partial reactivity of the fluoride fluxes with the alloy matrix occurs, resulting in the development of intermetallic phases which are intermediate during the reaction such as AlTi3 in the composite. The inclusion of intermediary phases in composites is proven to be detrimental to their characteristics. People have been attempting to manufacture in-situ Al/ TiB2 composites without the generation of intermediate reaction products since FAS developed in-situ Al/ TiB2 composites, but only a few have succeeded [2-5]. The main challenge was determining the ideal holding duration; therefore the main difficulty was to establish an optimal holding period for Al-5wt% TiB2 composites in order to totally avoid the production of AlTi3 phases.

2 Experimental Details

The studies were accepted out to determine the impact of holding temperature of 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 which has been preheated K2TiF6 and KBF4 salts with a Ti:B ratio of two point two is to 1 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 composite melt was casted in the warmed mild steel die. At

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over a temperature of 750, 800, 850 and 900ºC, the composite castings were made. 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

The X-ray diffraction studies which were performed on the Al1050-5%TiB_2 composites with a processing temperature of 750°C for a constant time of 60 minutes is shown in Fig.1.1. Under these processing conditions the X-ray diffraction image shows the peaks of Al_3Ti and AlB_2 phases which indicate that even after sixty minutes of processing time at a temperature of 750°C there is no complete transformation of the fluoride salts to the TiB_2 phase. The presence of intermediate phases which further indicates that the transformation is not complete 750°C. Moreover the high intensity of Al_3Ti peaks in the Fig. 1.1 further confirm that there is more formation of Al_3Ti particles that 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.

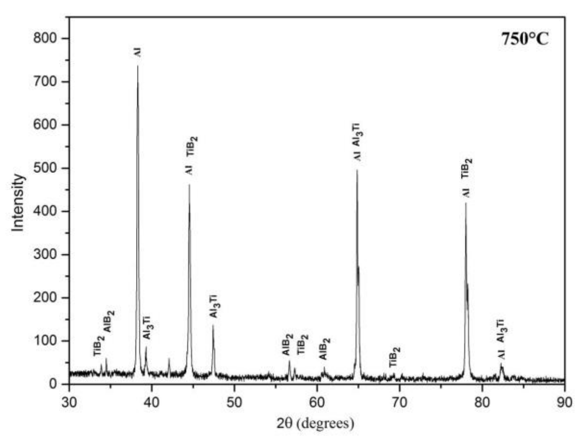

Fig. 1.1 XRD pattern of *in-situ* composite prepared at 750°C at 60 minutes.

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The X-ray diffraction studies which were performed on the Al1050-5%TiB2 composites with a processing temperature of 800°C for a holding time of sixty minutes is shown in Fig.1.2. Under these processing conditions the X-ray diffraction image shows the peaks of Al3Ti AlB2 and TiB2 phases which indicate that even at 800°C of melt temperature there is no complete transformation of the fluoride salts to the TiB2 phase[4]. The presence of intermediate phases which further indicates that the transformation is not complete even at 800°C. Moreover, the intensity of Al3Ti peaks in the Fig. 1.2 has reduced indicating that the transformation is proceeding in the right direction.

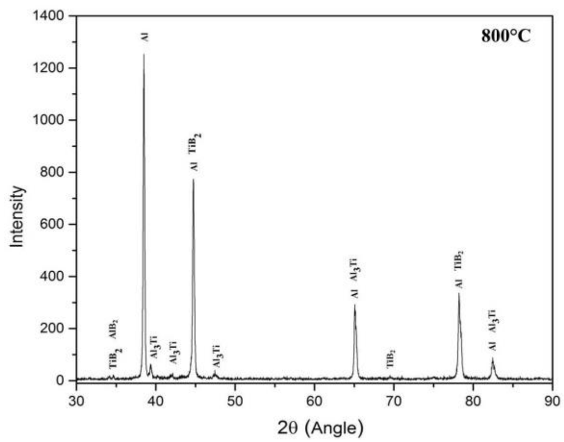

Fig. 1.2 XRD pattern of *in-situ* composite prepared at 800°C at 60 minutes.

The reduction in the peaks of AlB2 and Al3Ti peaks in the XRD analysis as shown in Fig.1.3 compared to the peaks in Fig.1.2 clearly indicated that the more of boron in the melt has been considerably reduced to TiB2 particles, which is further confirmed by the reduction in intensity of Al3Ti peaks. This reduction in Al3Ti peaks is because more of Ti is used in the formation of TiB2 particles.

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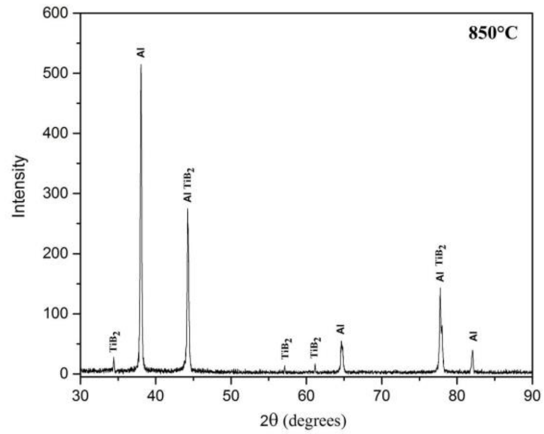

Fig. 1.3 XRD pattern of *in-situ* composite prepared at 850°C at 60 minutes.

The X-ray diffraction studies which were performed on the Al1050-5%TiB2 composites with a processing temperature of 850°C for a holding time of sixty minutes is shown in Fig.1.3. Under these processing conditions the X-ray diffraction image shows the peaks of TiB2 phases which indicate that at 850°C of processing temperature the transformation of the fluoride salts to the TiB2 phase is complete which confirms that only TiB2 particles are present in the composite.

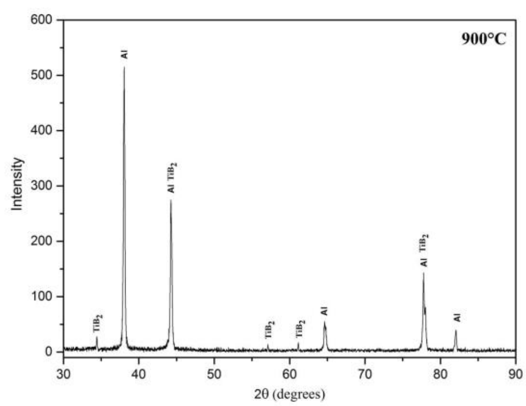

Fig. 1.4 XRD pattern of *in-situ* composite prepared at 900°C at 60 minutes.

The X-ray diffraction studies which were performed on the Al1050-5%TiB2 composites with a processing temperature of 900°C for a holding time of sixty minutes is shown in Fig.1.4.

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The diffraction pattern confirms that the reaction between the fluoride salts and the aluminium melt is complete thereby indicating the presence of only TiB2 peaks. From the XRD results it is very much evident that the reaction between the fluoride salts and melt is complete at 850°C and 900°C. The results confirm that 850°C is sufficient for the processing of the Al1050-TiB2 composites.

3.2 Scanning Electron Microscopy

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 at 750ºC which is shown in Fig.1.5a clearly shows that the particles have formed during the process.

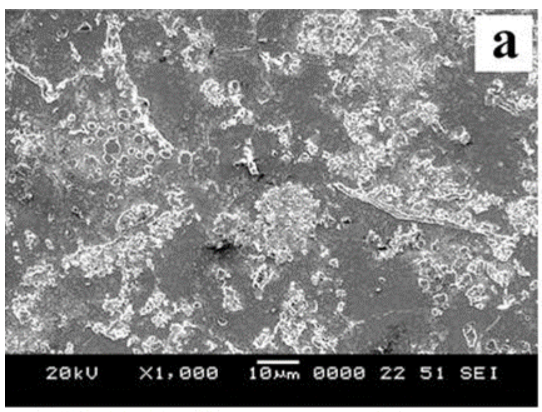

Fig.1.5a SEM Photomicrograph from the prepared composite at 750 ºC.

The formed particles were of different size and morphology. The string like particles which are seen the photomicrograph were found to be having the elemental signature of Ti, Al and Si[6]. The energy dispersive spectrum confirmed that the particles were AlTiSi. In the Fig.1.5b, fine blocky type particles are seen. The EDS analysis on the particles confirmed that they were all Al3Ti particles. Similarly the EDS analysis on particles which were seen the photomicrographs of Fig.1.5c and Fig.1.5d were of TiB2 and AlB2.

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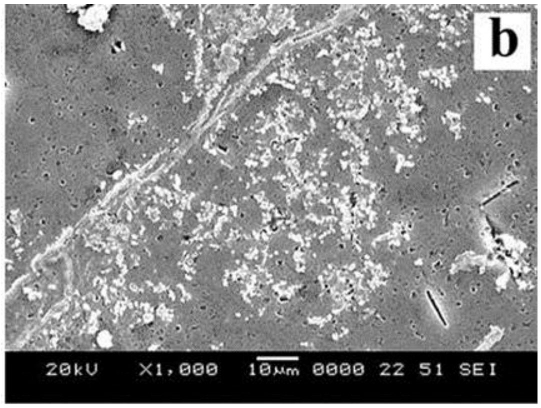

Fig.1.5b SEM Photomicrograph from the prepared composite at 800 ºC.

The sizes of the TiB2 particles were found to be in the range of nano meter to less than 1 µm [5]. 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.

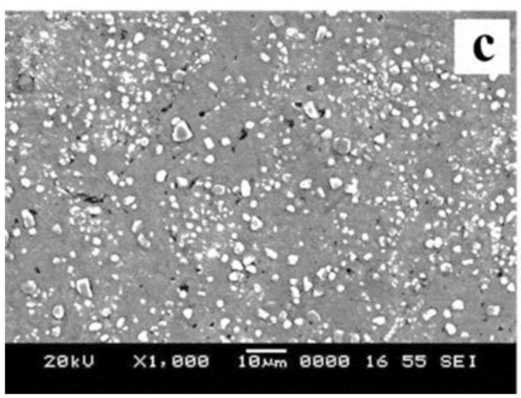

Fig.1.5c SEM Photomicrograph from the prepared composite at 850 ºC

The large AlB2 and AlTiSi in the matrix as seen in Fig.1.5b confirms that the aluminium alloy melt don’t have the sufficient temperature for this particles to transform it into TiB2. The string like particles which were found to be Al3Ti, seen in Fig.1.5c of the composite processed at

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800ºC for 60 min confirms that at this temperature most of the Si has diffused out of AlTiSi particles, hence we were able to see only Al3Ti particles at this temperature. The presence of Al3Ti particles further indicate that the holding temperature is still insufficient for the particles to further dissociate and form as TiB2[7].

The photomicrograph of the composite processed at 800ºC shows that the stability of Al3Ti particles was due to depletion of high amount of boron in the melt. Although at this processing temperature higher amount of TiB2 particles were seen, the presence of Al3Ti particles confirms that the reaction between the salts and melt is incomplete.

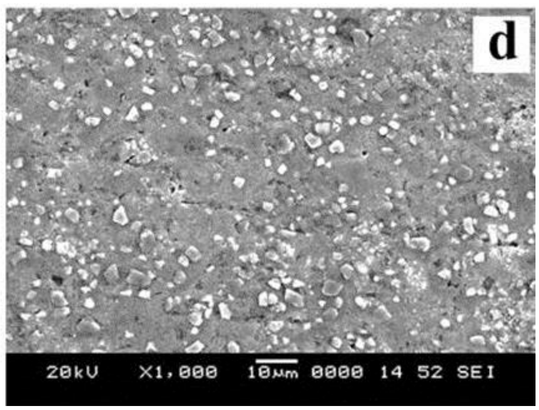

Fig.1.5d SEM Photomicrograph from the prepared composite at 900 ºC.

The SEM photomicrographs obtained from the composite processed at 850 and 900 ºC is shown in Fig 1.5c and Fig.1.5d No presence of AlB2 and Al3Ti were seen We can observe that only TiB2 particles were present in the composites which confirms that the reaction between the halide salts and melt was complete. The particles in the composite processed at 850°C and 900°C were having an average size of 2.2 and 2.7 µm. Some of the TiB2 particles in the composite showed hexagonal morphology. However, the observations also confirmed that large particles which were present showed morphology which was irregular. The size of the TiB2 particles in the composite processed at 850°C and 900°C show that the increase in temperature of the aluminium alloy melt do have a major role in the growth of the TiB2 particles [8]. The temperature of the melt had a series effect on the formation the TiB2 particles in the melt.

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4. CONCLUSION

Overall, the prepared composites at different temperature had greater mechanical properties than the unreinforced alloy, according to the findings. Several causes can be attributed to the composites' improved properties:

(i) Absence of slag which is undesirable and unwanted, which is entrapped during solidification.

(ii) Increase in strengthening of the matrix alloy and

(iii) Presence of more number of TiB2 particles compared to Al3Ti particles

The composites' characterization shows that there is no slag in the matrix alloy. As a result, the above-mentioned causes could be to blame for the composites' improved qualities. Based on the knowledge gathered from this opinion, it was decided to reduce the occurrences of particles of Al3Ti.

The overall observations also confirmed that the temperature of the melt had an important role in improving the properties of the unreinforced alloy.

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