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Research Paper

Microstructural effects on the Intermittent stirring during the processing of Al1050-5%TiB_2 in-situ composites

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Abstract

In the present work particulate reinforced Al1050-5wt.%TiB_2 *in-situ* composites were processed under three time of 5 minutes, 10 minutes and 15 minutes of intermittent stirring was investigated. The microstructural characterization of the prepared composites was systematically examined by optical, electron microscopy and X-ray diffraction. Composite processed at 5 minutes of Intermittent stirring showed inhomogeneous distribution of particles present along with the unreacted salt and slag. Moreover, along with TiB_2 particles the presence of intermediate phases like Al_3Ti and AlB_2 confirm that the reaction between the salts and the melt was incomplete. The composite processed 10 minutes of intermittent stirring showed uniform distribution of particles. Composite prepared with 15 minutes of Intermittent stirring of the composite also showed the presence of both intermediate phases along with fine TiB_2 particles confirmed that high melt stirring conditions hindered the formation of TiB_2 particles. The microstructural observations were confirmed by the X-ray diffraction studies. The percentage of porosity present was found to be higher among the composites prepared with 5 minutes of intermittent stirring.

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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.

2 Experimental Details

The studies were carried out to determine the impact of different intermittent stirring time 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 salts with standard 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 different time interval of 5 minutes, 10 minutes and 15 minutes were carried out. 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. At over a temperature of 850ºC for sixty minutes, the composite castings were made at different intermittent stirring. 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

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The X-ray diffraction studies which were performed during the processing of Al1050-5%TiB_2 composites processed with 5 minutes of intermittent stirring with a processing temperature of 850°C for a constant time of 60 minutes is shown in Fig.4.6. 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, TiB_2, Al_3Ti and AlB_2 phases which indicate that at the above mentioned conditions 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 at 5 minutes of intermittent stirring. 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.

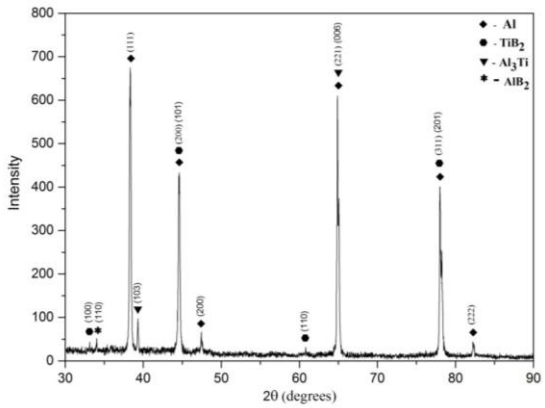

Fig.1.1 XRD pattern of *in-situ* composite prepared at 5 minutes of intermittent stirring

The X-ray diffraction studies which were performed during the processing of Al1050-5%TiB2 composites with 10 minutes of intermittent stirring with a processing temperature of 850°C for a constant time of 60 minutes is shown in Fig.1.2.

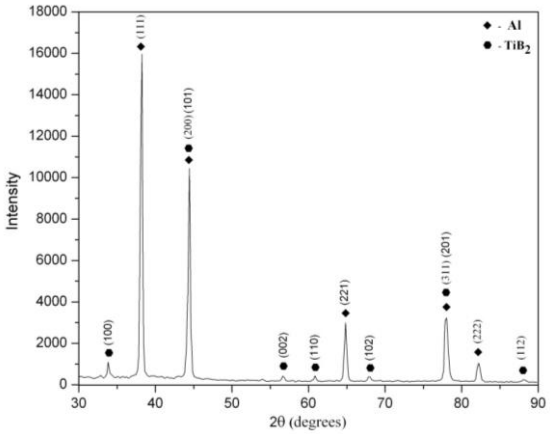

Fig.1.2 XRD pattern of *in-situ* composite prepared at 10 minutes of intermittent stirring

In these conditions the the preparation of K_2TiF_6 and KBF_4 salts an excess of 10% KBF4 salts is added. Under these processing conditions the X-ray diffraction image shows the only TiB_2 peaks which indicate that even at 850℃ of melt temperature there is complete transformation of the fluoride salts to the TiB_2 phase. The absence of intermediate phases which further indicates that the transformation is complete with the 10 minutes of intermittent stirring. Moreover the absence of other intermediate peaks in the Fig. 1.3 has reduced indicating that the transformation is proceeding in the right direction.

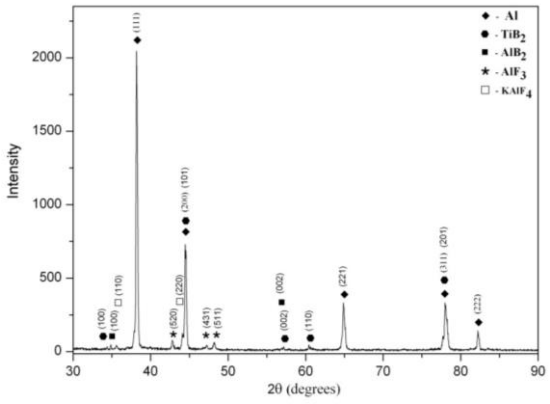

Fig.1.3 XRD pattern of *in-situ* composite prepared at 15 minutes of intermittent stirring

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.

3.2 Scanning Electron Microscopy

Scanning electron microscope plays a vital role to study the surface property of the samples. 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 5 minutes of intermittent stirring is shown in Fig.1.4a clearly shows that the particles have formed during the process. 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

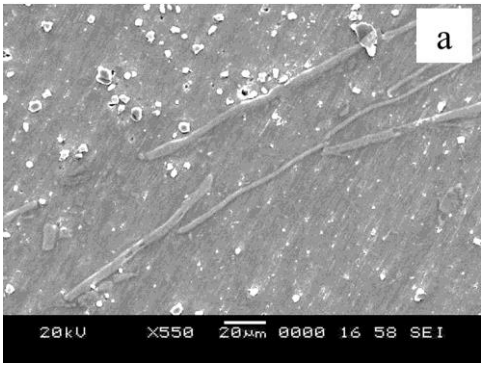

Fig.1.4a SEM image of composite processed at 5 minutes of intermittent stirring

The EDS analysis on the particles confirmed that they were all TiB_2 particles. The sizes of the TiB_2 particles were found to be in the range of nano meter to less than 1 µm. The TiB_2 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 Al_3Ti and AlB_2.

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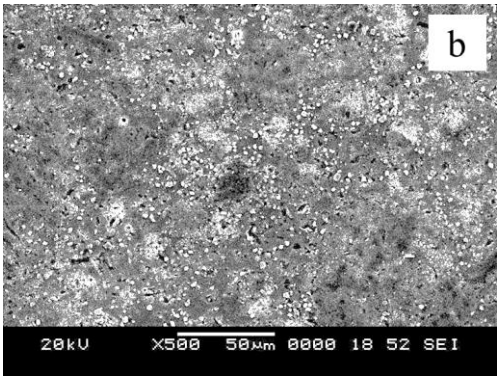

Fig.1.4b SEM image of composite processed at 10 minutes

As the melt temperature is held for a consistent amount of time, the Al_3Ti and AlB_2 further decomposes and forms as TiB_2. The SEM image of composite processed with 10 minutes if intermittent stirring is shown in Fig.1.4b. Analysis on the image shows that there is formation of only TiB_2 particles.

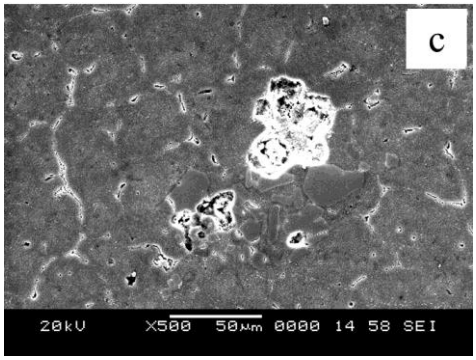

Fig.1.4a SEM image of composite processed at 15 minutes

The SEM image of composite processed with 10 minutes if intermittent stirring is shown in Fig.1.4c. 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 at lesser time of intermittent stirring of salts the mixing of salts the salts do not mix completely with the melt. Hence the slag remained in the composite.

4. CONCLUSION

Overall, the prepared composites at different intermittent stirring time 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) Intermittent stirring of 10 minutes of the melt with salts was found to be desirable condition for production Al1050-5%TiB2 composite.

(ii) Intermittent stirring of 5 minutes of the melt with mixture of K2TiF6 and KBF4 salts has led to formation of more unwanted AlB2 particles and Al3Ti particles.

(iii) 15 minutes of Intermittent stirring of the melt to the stoichiometry mixture of K2TiF6 and KBF4 salts led to presence of more unwanted slag in the prepared composite.

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