



# Processing of Mg-3.2 Al-2.4 Zn Alloy Matrix In-situ Composites by Reactive Infiltration Technique

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**Abstract:** Mg alloy matrix composites having a combination of their indispensable and superior properties have drawn an attention for various implementations especially in automotive and aerospace industries. Mg in-situ composites were synthesized using Mg-3.2Al-2.4Zn alloy ingot, coarse Ti and B<sub>4</sub>C powder. Ti and B<sub>4</sub>C powders were mixed in a plastic bottle with zirconia balls in Ar atmosphere by ball milling. The resulting mixture of these powders was compacted into a cylindrical preform which was infiltrated by Mg-3.2Al-2.4Zn alloy under capillary force. The infiltration of the preform was performed in an electric resistance furnace at the temperatures of 800°C and 900°C for different holding time in Ar atmosphere. The microstructure of the in-situ composites was investigated with a field emission scanning electron microscope (FESEM) equipped with Energy Dispersive X-ray (EDX). The formation of phases in the in-situ composites during processing was identified using XRD with Cu K $\alpha$  radiation. SEM revealed different phases in the in-situ composites. It was found in XRD pattern that TiC, TiB<sub>2</sub>, TiB, MgB<sub>2</sub>, MgB<sub>4</sub>, B<sub>13</sub>C<sub>2</sub> and Ti<sub>2</sub>AlC compounds were formed during infiltration of Mg-3.2 Al-2.4 Zn alloy matrix in the composites. The effect of processing parameters on bulk density and Brinell hardness was also discussed.

**Keywords:** Mg Alloy, In-situ Composites, XRD, Bulk Density, Hardness

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## 1. Introduction

Mg alloy has been considered as a potential matrix material for metal matrix composite (MMC) for its lightweight properties i.e. pure Mg is 35% lighter than Al and over four times lighter than steel [1-3]. Ceramic particles reinforced Mg alloy MMCs has been effective for the automotive and aerospace applications because of having their high specific modulus and high wear resistance, low coefficient of thermal expansion, tensile strength and excellent electrical and thermal conductivities [4-8]. The fabrication of the composites with isotropic properties can easily be achieved by reinforcing the particulate as reinforcement in the composites. However, the properties of MMCs depend upon the particle stability, volume fraction, size, particle bonding with the matrix and extent of dispersion in the matrix. Conventional methods are known as ex-situ process, such as powder metallurgy, spray deposition, mechanical alloying, preform infiltration and various casting

methods such as rheocasting, squeeze casting and compo-casting have been generally used to fabricate Mg alloy MMCs. The interface characteristics between matrix and reinforcement have the significant effects on the properties of the composites fabricated by these ex-situ processes.

In the in-situ process, reinforcement particulates in the matrix metal alloys are formed by the reactions of the starting materials during processing. The formation of ultra-fine homogeneously dispersed and a thermodynamically stable ceramic reinforcement with clean reinforcement-matrix interfaces in-situ MMCs exhibit excellent mechanical properties [9-10]. TiC and TiB<sub>2</sub> ceramic particles having high melting points, good thermal and chemical stability, high hardness, low densities and excellent wear resistance are considered as good potential reinforcing elements for Mg alloy matrix [11-12]. Moreover, TiB<sub>2</sub> is considered as a proper reinforcement for their coherency of a crystal lattice

with Mg alloy matrix [13]. Mg matrix composites reinforced with in situ TiC and TiB<sub>2</sub> particles have been fabricated using self-propagating high-temperature synthesis (SHS), stir casting method and remelting and dilution (RD) [14-15]. The in-situ reactive infiltration process can be used for the synthesis of in-situ composites as an innovative process. However, a few researchers had followed this technique using Ti and C as starting materials to produce TiC reinforcements in Mg-based alloy matrix composites [8, 16-18]. Using the starting materials Ti and B<sub>4</sub>C particles via a reactive infiltration technique has been found to be limited in the open literature for synthesizing the Mg alloy matrix TiC and TiB<sub>2</sub> in-situ composites [16-19]. Thus, the present work has been performed to the synthesis of Mg alloy matrix composites in-situ MMCs reinforced with stable ceramic particles TiC and TiB<sub>2</sub> through infiltration process using Ti and B<sub>4</sub>C particles as starting materials.

## 2. Materials and Experimental Procedure

### 2.1. In-situ Composites Fabrication by Reactive Infiltration Technique

Mg-Al-Zn alloy ingot (94.03 wt.% Mg, 3.24 wt.% Al and 2.40 wt.% Zn) was used as matrix materials and coarse Ti (average particle size 250 µm) and B<sub>4</sub>C (average particle size 40 µm) powders were used as starting materials for synthesizing the in-situ Mg alloy composites. Ti and B<sub>4</sub>C powders with a molar ratio of 3:1 were taken in a polypropylene jar with 210 gm zirconia balls. In order to prevent oxidation of Ti, B<sub>4</sub>C powders and zirconia balls were taken in a Perspex box filled with Ar gas. The powders were mixed in a planetary ball mill with a speed of 400 rpm for 4 hours. After blending, the mixture of Ti and B<sub>4</sub>C powders was compacted into the green preforms of the cylindrical shape of 15 mm diameter and various heights (150-167mm) using hardened steel dies under the pressure of 80 Mpa with the help of the universal tensile testing machine. The compacted Ti-B<sub>4</sub>C preform was then placed in a perforated mild steel tube. The pieces of Mg-Al-Zn alloy ingot were placed on the top of the compacted preform in the tube. The steel tube containing powder preform and Mg alloy ingots was then placed in a stainless steel chamber and the chamber was then closed with the lid containing two openings where one was used as an inlet for Ar gas and another was used as an outlet. The stainless steel chamber was placed in the furnace and the chamber was purged with Ar for a few minutes before heating and then backfilled with argon gas. The infiltration process was carried out in the furnace in the presence of a continuous flow of argon gas with a flow rate of 5 L.min<sup>-1</sup>. The synthesis process was carried out at temperatures of 800°C and 900°C for 1 and 4 hours. The heating rate 13°C/min was used to reach synthesis temperature. At the end of predetermined infiltration time,

the furnace was switched off and the samples were cooled in the furnace in an argon atmosphere.

### 2.2. Characterization of the Nanostructures

The samples were polished by following standard metallographic technique and were investigated under a field emission scanning electron microscope (FESEM) equipped with Energy dispersive X-ray (EDX). For phase analysis, some selective samples were analyzed by X-ray diffraction technique using Cu Kα radiation. The relative density of the synthesized composites was determined. Brinell hardness test was also carried out to determine the mechanical properties of the in-situ composites.

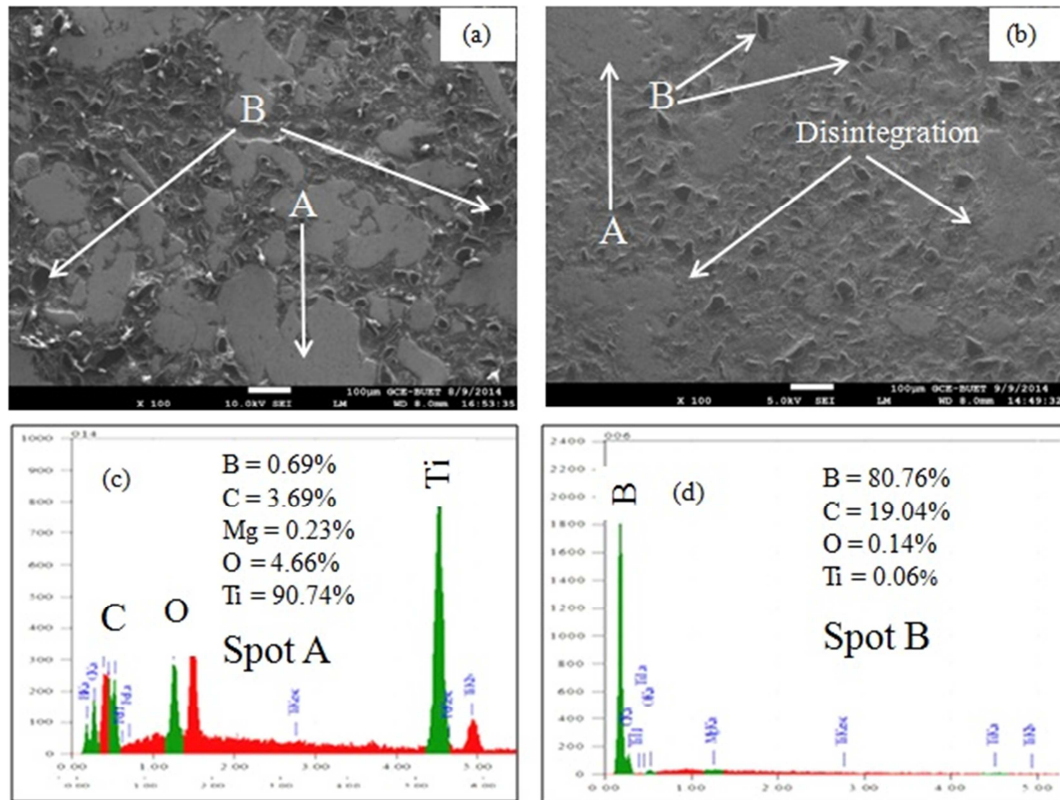
## 3. Results and Discussion

### 3.1. Micro Structural Characterization of the In-situ Composites

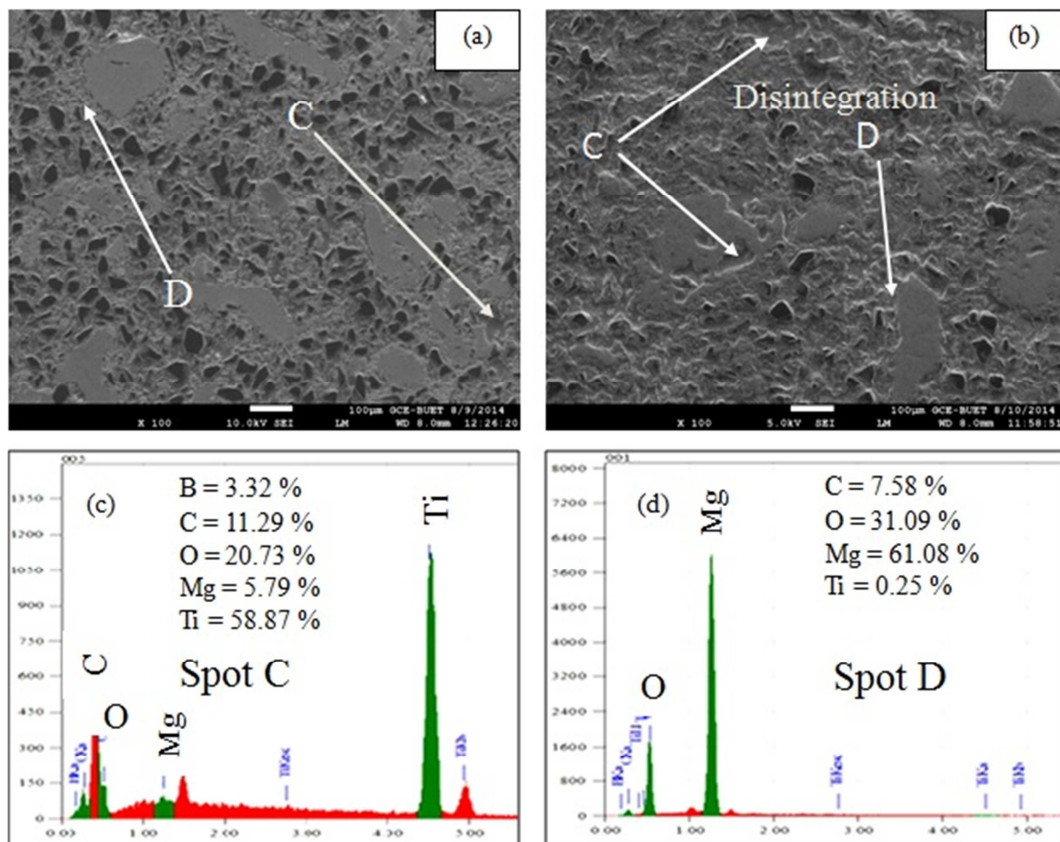
SEM micrographs of the Mg-Al-Zn alloy in-situ composites synthesized at the temperature of 800°C and 900°C for holding time 1 and 4 hours investigated using FESEM are shown in Figure 1, Figure 2 and Figure 3. Microstructures reveal that the samples infiltrated at different processing parameters contain grey particles (A), black particles (B), grey at deep ditches (C), porous structure (D) and white particles (E). The various sizes of the phases ranging from the submicron to several hundred microns are observed for all the in-situ composites.

The chemical compositions of the phases were measured by EDX analysis and the typical spectra of different phases are shown in Figure 1 (c) and Figure 1 (d). According to the EDX results, it is seen that the grey dense phases (A) consists of 0.69% B, 3.69% C, 4.66% O, 0.23% Mg and 90.73% Ti while black phases (B) consists of 80.76% B, 19.04% C, 0.14% O and 0.06% Ti. Based on the analysis of EDX result, the grey dense phase (A) is indicated as Ti and the black particles (B) as B<sub>4</sub>C. It is seen from the Figure 2, EDX analysis at spot C shows that grey phase at deep ditches consists of 3.32% B, 11.29% C, 20.73% O, 5.79% Mg and 58.87% Ti while the porous structure (spot D) consists of 7.58% B, 31.09% O, 61.08% Mg and 0.25% Ti. Grey phase at deep ditches (spot C) in the matrix is indicated to be infiltrated Mg but the porous structure (spot D) is basically B, C, O and Ti enriched structure. This O is due to the oxidation of Mg which may be produced during polishing.

From the Figure 3, spot E consists of 1.69% B, 13.71% C, 57.35% O, 26.70% Mg and 0.55% Ti. Some bright white small particles (E) observed in the matrix through infiltrated in-situ composites as an oxide of Mg is identified. At least eight spots analysis from each of the phases of the in-situ composites synthesized at different temperature and time were taken and an average value of above-mentioned phases is given in Table 1.



**Figure 1.** Showing SEM image of Mg alloy in-situ composites synthesized at 800°C for (a) 1 hr and (b) 4 hr, and EDX spectra of (c) spot A and (d) spot B in the image.



**Figure 2.** Showing SEM image of Mg alloy in situ composites synthesized at 900°C for (a) 1 hr and (b) 4 hr, and EDX spectra of (c) spot C and (d) spot D in the image.

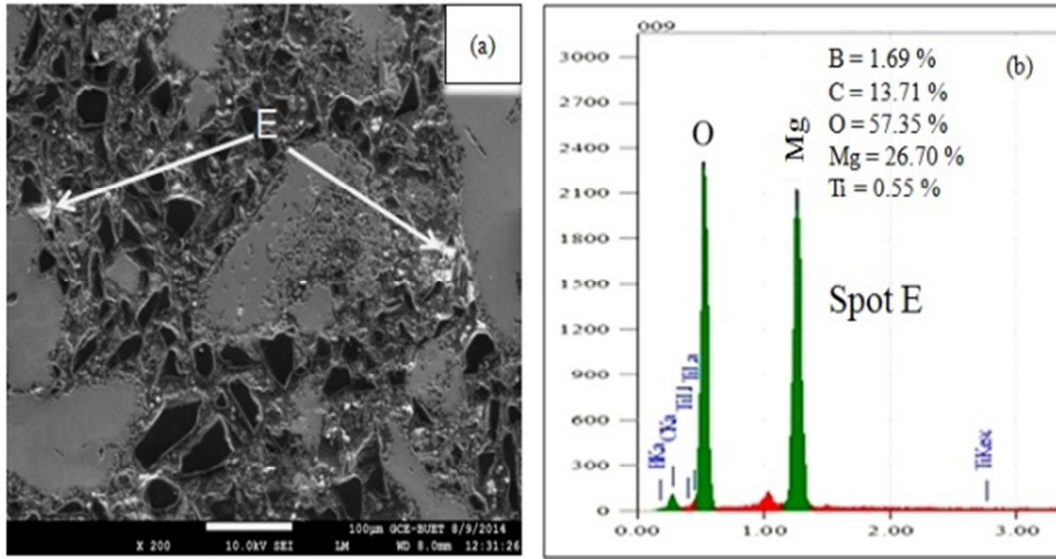


Figure 3. Showing (a) SEM image of Mg alloy in-situ composites synthesized at 900°C for 1 hr, and EDX spectra of (b) spot E in the image.

Table 1. Chemical composition of different phases present in Mg-Al-Zn alloy in-situ composites synthesized at different temperatures and time.

Phase	B (wt.%)	C (wt.%)	O (wt.%)	Mg (wt.%)	Ti (wt.%)
Grey particles (A)	1.31	3.33	14.51	0.72	80.13
Black particles (B)	82.83	16.81	0.11	0.04	0.21
Grey phase at deep ditches (C)	-	11.99	22.34	65.26	0.41
Porous structure (D)	17.53	8.78	14.34	3.18	56.17
White particle (E)	-	15.97	57.89	26.08	0.06

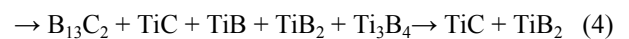
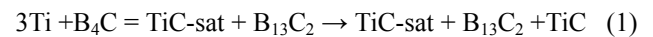
It is observed from Figure 1 (b) and Figure 2 (b), Ti phase is disintegrated with the rise of the holding time [compare Figure 1 (a) and Figure 1 (b)] and temperatures [compare Figure 1(b) and Figure 1 (d)] because more reactions occur and new compounds are formed in a large quantity. From the micrographs, it is observed that the extent of disintegration of Ti and B<sub>4</sub>C and reactions are found to be the highest at the infiltration temperature of 900°C for 4 hours (Figure 2d). The disintegration of the vicinity of the Ti phases is normally higher in the Mg alloy in-situ composites because the addition of Al in Mg acts as a reaction intermediary to facilitate the reaction between Ti and B or Ti and B<sub>4</sub>C [20]. It was reported that Al addition to the Ti-C preform enhances the reaction between Ti and C in Mg alloy [21]. The viscosity and wettability facilitate Mg alloy in infiltration through the preform of Ti-B<sub>4</sub>C to form the in-situ TiC compared to the performance of Mg in-situ composites. As a result, more infiltration of Mg alloy into the pores of the Ti-B<sub>4</sub>C preform conducted by the capillary force results in the densified microstructure of in-situ composites.

### 3.2. XRD Analysis of In-situ Composites

The XRD pattern of the samples of Mg-Al-Zn alloy infiltrated in the Ti-B<sub>4</sub>C preforms at 800°C and 900°C for 4 hours is shown in Figure 4. From the XRD pattern analysis of the in- situ composites, MgB<sub>2</sub>, MgB<sub>4</sub>, TiC, TiB, TiB<sub>2</sub>, B<sub>13</sub>C<sub>3</sub> and Ti<sub>2</sub>AlC phases are identified in addition to the starting materials of Ti, B<sub>4</sub>C and Mg alloy. It was reported that C-

saturated Ti, (Ti<sub>C</sub>-sat), formed prior to titanium carbide due to the diffusion of C from B<sub>4</sub>C into Ti.

With increasing temperature, the substoichiometric TiC forms while the lattice parameters of the retained Ti were decreased. The range of the lattice parameters of the boron carbide is found to be consistent with that of the standard lattice constants of B<sub>13</sub>C<sub>2</sub> according to Pearson's Handbook [22]. Based on the results and the crystallographic data of phases from Pearson's handbook [22], the reaction sequence of the Ti-B<sub>4</sub>C system can be presented as follows:



The formation of substoichiometric TiC prior to the titanium borides was proposed by Zhao and Chang [23]. This attribute to the diffusivity of carbon in Ti was being much greater than that of boron [24]. Despite strong covalent bonds between the atoms in the boron carbide structure, carbon diffused away from boron carbide faster than that of boron. Thus TiC phase formed prior to titanium borides liberating a B-rich boron carbide B<sub>13</sub>C<sub>2</sub> as it is also evident in XRD analysis in Mg matrix system. This was also reported by Shen et al. [24-25].

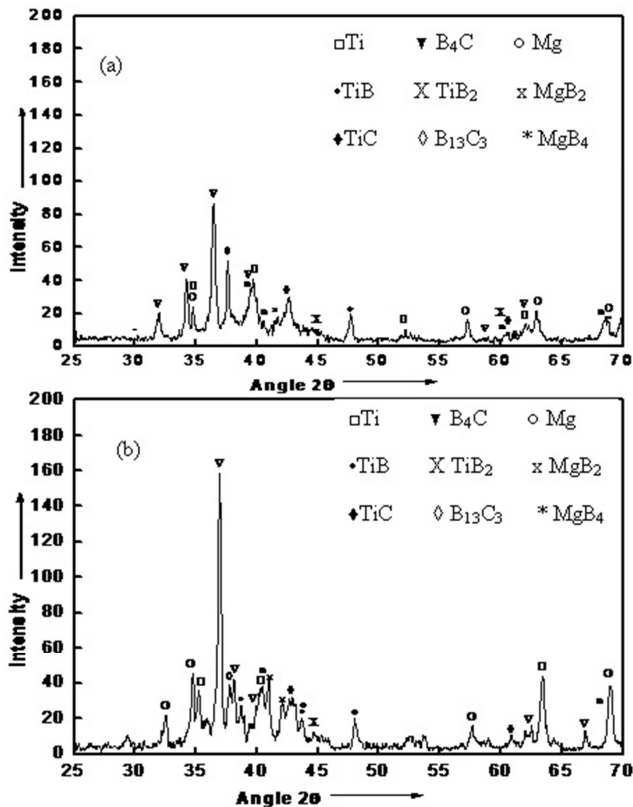
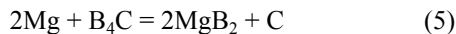
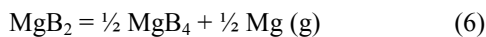


Figure 4. XRD patterns of heat treated Mg-Al-Zn-(Ti-B<sub>4</sub>C) samples synthesized at different temperatures of (a) 800°C and (b) 900°C after 4 hour holding time.

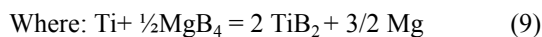
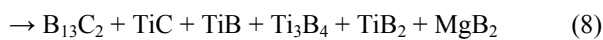
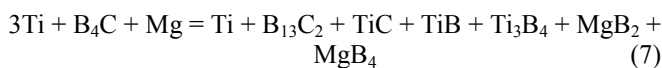
Based on the XRD results, it can be suggested that molten magnesium alloy reacts with B<sub>4</sub>C to form magnesium diboride liberating carbon as the following reaction. Kevorkijian and Skapin revealed the same [26]. This reaction is an exothermic reaction evolving heat for further reaction to occur in the system. Hence, the reaction mechanism is as follows:



The presence of MgB<sub>4</sub> peaks in the XRD pattern is also observed because MgB<sub>2</sub> partially decomposes forming MgB<sub>4</sub> and Mg gas that diffuses through the thin film of MgB<sub>4</sub> as follows:

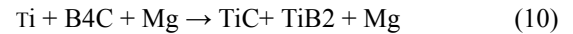


This agrees with the results obtained by Brutti et al. [27]. TiC is formed due to the reaction between Ti and B<sub>4</sub>C (1) and also the diffusion of atomic C liberated from the reaction between Mg and B<sub>4</sub>C (3) into Ti according to the following equation:



Since MgB<sub>2</sub> reacts with TiB forming TiB<sub>2</sub>, TiB and Ti<sub>3</sub>B<sub>4</sub>, react with the retained boron carbide as presented earlier in

the reaction mechanism of a Ti-B<sub>4</sub>C system, the reaction between Ti, B<sub>4</sub>C, and infiltrated molten Mg alloy is finally obtained as follows:



It is found in XRD pattern that TiC formed at first in both the samples while the percentage of TiB<sub>2</sub> increased with increasing synthesis temperature, and this is clear when comparing the peaks of the phases because of a strong effect of processing temperature on increasing the in situ reaction rate in XRD. Only one ternary compound Ti<sub>2</sub>AlC is also identified in Mg alloy composites synthesized at both temperatures and time. But, at a higher temperature, the Ti<sub>2</sub>AlC peak is found to be increased. This peak is formed due to the presence of Al in the alloy which reacts with Ti and dissociated C from B<sub>4</sub>C. The atomic bonding in the ternary compound is a combination of metallic, ionic and covalent [28]. The covalent-ionic Ti-C bonds are comparable to bonds in the binary TiC but they are stronger than the metallic Ti-Al bonds in the ternary structure [28]. Because of the relatively weak bonds between the TiC and Al layers in the basal planes, these materials have an anisotropic character [29].

MgB<sub>2</sub>, MgB<sub>4</sub>, TiC, TiB, TiB<sub>2</sub>, and B<sub>13</sub>C<sub>3</sub> are found in both the in situ composites synthesized in the present study. It was also reported that the compounds of MgB<sub>2</sub>, MgB<sub>4</sub>, TiB, and B<sub>13</sub>C<sub>3</sub> were thermodynamically unstable and these are known as intermediate compounds [30]. These compounds dissociate and form the more stable compounds TiC and TiB<sub>2</sub> [11-12, 31]. In the present case, the presence of Ti and B<sub>4</sub>C with intermediate compound MgB<sub>2</sub>, MgB<sub>4</sub>, TiB and B<sub>13</sub>C<sub>3</sub> in the both in situ composites indicates that the reactions were incomplete.

### 3.3. Effect of Temperature and Time on Relative Density

The relative density of Mg alloy in-situ composites synthesized at 800°C for 1 and 4 hours, and 900°C for 1 and 4 hours shows 2.50 gm/cm<sup>3</sup>, 2.68 gm/cm<sup>3</sup>, 2.60 gm/cm<sup>3</sup> and 2.77 gm/cm<sup>3</sup>, respectively. It also shows that the relative density increases with increasing infiltration temperature and time. With increasing temperature, more Mg alloy is infiltrated into the preform by capillary action and yields to high density. The small addition of Al from Mg-Al-Zn alloy increases the wettability of Mg and infiltration into the preforms with increasing holding time [20-21, 32]. Secondly, the presence of a lot of carbon vacancies in the TiC structure orderly can help Al to fill the spaces forming a ternary compound Ti<sub>2</sub>AlC [33-34]. This ternary compound may form due to the diffusion of Al from the Mg-Al-Zn alloy melt into the sub-stoichiometric TiC compound in the in-situ composites infiltrated with the rise of infiltration time.

### 3.4. Effect of Temperature and Time on Hardness

Brinell hardness of Mg alloy matrix in-situ composites using Ti-B<sub>4</sub>C preform infiltrated at different processing parameters is presented in Table 2. It is observed that the



hardness of Mg alloy infiltrated in-situ composites increases with the increase of temperature and time. More Mg alloy infiltration into the preforms by capillary action make the in-situ composites dense and simultaneously more reaction products formed at high temperature and time with the higher amount of stable TiC and TiB<sub>2</sub>. High infiltration temperature and time resulted in high infiltration and high intermediate compounds with stable TiC and TiB<sub>2</sub>.

**Table 2.** Brinell hardness values of Mg alloy matrix in-situ composites using Ti-B<sub>4</sub>C preform infiltrated at different processing temperature and time.

Temperature (°C)	Time (hr)	Brinell hardness
800	1	127
	4	191
900	1	142
	4	198

## 4. Conclusion

Reactive infiltration process was used to synthesize Mg alloy matrix in-situ composites. The infiltration of Mg in Ti-B<sub>4</sub>C preform was evident from the SEM investigation and increment of relative density with infiltration temperature and time. Different types of stable and unstable compounds such as MgB<sub>2</sub>, MgB<sub>4</sub>, TiB, TiC, TiB<sub>2</sub>, and B<sub>13</sub>C<sub>2</sub> are formed in the in situ composites during the infiltration process. The reactions in the in situ composites at synthesizing temperature and time used in the study were found to be incomplete as the evidence of the presence of intermediate compounds. The dissolution of less stable intermediate compounds will be changed into thermodynamically stable TiC and TiB<sub>2</sub> using high infiltration time at high temperature. The hardness of the composites was found to increase with the infiltration temperature and time.

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