

● Research Articles

Analysis of *in situ* Reaction and Pressureless Infiltration Process in Fabricating TiC/Mg Composites

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An innovative processing route, *in situ* reaction combined with pressureless infiltration, was adopted to fabricate magnesium matrix composites, where the reinforcement TiC formed *in situ* from elemental Ti and C powders and molten Mg spontaneously infiltrated the preform of Ti and C. The influences of primarily elemental particle sizes, synthesizing temperature, holding time *etc* on *in situ* reactive infiltration for Mg-Ti-C system were systematically investigated in order to explore the mechanism of this process. In fabricating TiC/Mg composites, Mg can not only spontaneously infiltrate the preform of reinforcement and thus densify the as fabricated composites as matrix metal, but also it can accelerate the *in situ* reaction process and lower the synthesizing temperature of Ti and C as well. *In situ* reaction of Ti and C and Mg infiltration processes are essentially overlapping and interacting during fabrication of TiC/Mg composites. The mechanism proposed in this paper can be used to explain the formation and morphologies of the reinforcement phase TiC.

KEY WORDS: Magnesium matrix composite, *In situ* reactive infiltration, Titanium carbide, Mechanism

1. Introduction

Recently, magnesium matrix composites have been paid much attention due to their superior specific properties, and they can be the most attractive candidate for applications in aerospace, electronic and automotive industries^[1~3]. However, among the several conventional processing routes, they are sometimes inevitably suffering from poor wettability of the reinforcement with matrix metal, interfacial reaction and processing cost *etc*. To overcome the limitations of these *ex situ* processes, an innovative process, *in situ* reactive infiltration, has been developed, in which the reinforcement phases are synthesized *in situ* and the molten metal spontaneously infiltrates into the preform of the formed reinforcement^[4~6]. This process contains the stage of self-propagating high-temperature synthesis (SHS)^[7], during which microscopically fine and thermodynamically stable reinforcing ceramic phases are formed *in situ*. As we know that in case of combustion synthesis it is very difficult to control the reaction among elemental reactants due to their very high exothermicity and reaction rate^[8,9]. For *in situ* reactive infiltration process in Mg-Ti-C system, the reaction rate can be controlled by infiltration of magnesium melt through diluting the density of the compacted elemental powder. The porous preform can further be densified by infiltration of the magnesium melt driven by capillary force and composites synthesized completely.

The advantages of the metal matrix composites (MMCs) produced by this process can be ascribed to as follows: (1) thermodynamically stable and homogeneously dispersed fine reinforcement formed *in situ* within the matrix lead to an increase in their mechanical properties, (2) bonding strength is greatly improved with existence of clean interface between reinforcement and matrix, and (3) the composites can be easily produced to a near-net shape with a high ceramic volume percentage (>50%) and cost effectively. By utilizing this processing route, several kinds of aluminum and magnesium matrix composites have been successfully manufactured^[4~7].

In analyzing the *in situ* reactive infiltration process, however, problem always arises as the sequence of *in situ* reaction and the successive process associated with the infiltrating behavior. As a matrix metal, in essence Mg does not take part in the reaction with C and no stable magnesium carbide appears, and also forms no intermetallic compound with Ti in Mg-Ti-C system. That the Mg does play an important role but how it influences the *in situ* reactive process remains

unclear. For this purpose, in this paper some experimental works relating to the effect of processing parameters on reaction products were carried out in order to investigate the mechanisms involved in synthesizing the TiC particulate reinforced magnesium matrix composites and clearly understand the *in situ* reactive infiltration process.

2. Experimental

Two sets of experiments were designed to explore the mechanisms relating to the *in situ* reaction and infiltration process in fabricating TiC/Mg composites for Mg-Ti-C system. Firstly, various sizes of titanium and carbon particles and processing conditions were taken into consideration. Table 1 shows the initial materials parameters and processing conditions used in fabricating TiC/Mg composites.

Secondly, the experiments through controlling the processing temperatures and holding time were conducted so that we can evaluate the reaction process of Ti and C and the infiltration capability of molten Mg into Ti_p+C_p preform. In this experiment, a relatively lower processing temperature, 953 K, slightly above Mg melting point and a very short holding time 15 min were adopted. Moreover, the occurrence of *in situ* reaction between Ti and C without Mg was examined in order to make a better comparison with the cases of Mg involved in this process. The Ti and C particle sizes were all 38 and 1.5 μm, respectively.

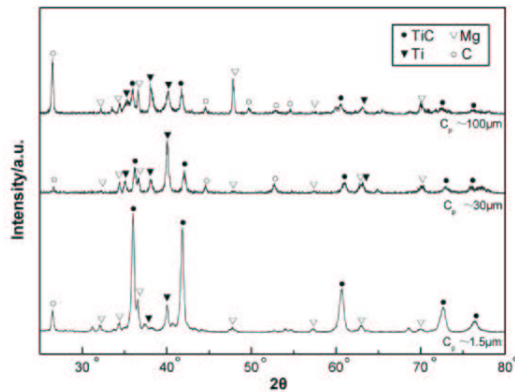
The powders of Ti_p and C_p, after mechanical blending, were first compacted into green body of cylindrical shape (φ16.5 mm×15 mm) with porosity of approximately 28%. Assuming that all Ti and C powders will transform into TiC particles with the pre-designed molar ratio of Ti/C=1:1 during *in situ* reactive infiltration process, TiC/Mg composites with reinforcement of about 55 vol. pct could be obtained according to the product of TiC phase formed *in situ* in the magnesium matrix. The preform together with a magnesium ingot on it was then put into an alumina tube. Small holes were drilled at the bottom of alumina tube in order to release the air. *In situ* reactive infiltration experiments were finally carried out in an electric furnace under a flowing argon (99.999% pure) atmosphere. The heating temperature and holding time were designed as mentioned above. After that, the samples were cooled down to room temperature with the furnace.

The phase composition of the reaction products and the microstructures for all as fabricated composite specimens were characterized by XRD in an X-ray diffractometer (D/max-2500 PC, Rigaku, Japan), scanning electron microscopy

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Table 1 List of materials parameters and processing conditions used in fabricating TiC/Mg composites

Composite	Mg	Ti		C		Processing conditions
		Size/ μm	Purity/%	Size/ μm	Purity/%	
C1		74	99.0	100	90.0	1023 K/1.5 h
C2	Ingot,	38	99.5	30	98.5	1023 K/1.5 h
C3	99.5%	38	99.5	1.5	98.5	1023 K/1.5 h
C4	pure	38	99.5	1.5	98.5	1073 K/1.5 h
C5		38	99.5	1.5	98.5	973 K/1.5 h

**Fig.1** XRD spectra of the TiC/Mg composite specimens synthesized at 1023 K/1.5 h. Ti/carbon particle sizes being $\sim 74/100$, $\sim 38/30$ and $\sim 38/1.5$ μm , respectively**Fig.2** XRD spectra of the TiC/Mg composite specimens fabricated at 1073, 1023 and 973 K/1.5 h. T_{ip} : ~ 38 μm , C_{p} : ~ 1.5 μm

(JSM-6301, Japan) equipped with energy dispersive spectroscopy (EDS). In addition, differential thermal analysis (DTA) (Shimadzu, Japan) was utilized to investigate the possible interactions among Ti-C, Mg-C and Mg-Ti-C for the Mg-Ti-C system. The measuring temperature ranged from the ambient up to 1473 K at a constant heating rate of $10 \text{ K} \cdot \text{min}^{-1}$ under an argon atmosphere.

3. Results and Discussion

3.1 Influence of Ti and C particle sizes and processing temperature on *in situ* reaction

TiC/Mg composites were fabricated with different C particle sizes and synthesizing temperatures so that the influences on the *in situ* reaction process between Ti and C can be investigated. Figures 1 and 2 showed the XRD patterns of the as fabricated TiC/Mg composite specimens as suggested in Table 1.

XRD spectra in Fig.1 indicated that magnesium spontaneously infiltrated into the $T_{\text{ip}}+C_{\text{p}}$ preform and TiC formed at temperature of 1023 K *via* the *in situ* reaction. As the

particle sizes of Ti and C decreases, XRD peaks of Ti and C of the starting materials were abruptly reduced and the composite synthesized with the smallest carbon particle size (~ 1.5 μm) displayed the largest peaks of TiC. When larger Ti and C particles were used, there were retained or unreacted Ti and C particles present in the samples. It is clear that molten magnesium can infiltrate the preform spontaneously and the *in situ* reaction between Ti and C with smaller particle sizes could proceed more completely and there were almost not any starting elemental powder to be preserved throughout the process.

From Fig.2, it can be seen that it is also important to consider the influence of the synthesizing temperatures on these processes. The higher the synthesizing temperature, the more complete the *in situ* reaction of Ti with C. Thus, there is more TiC reinforcement formed *in situ*. After 1073 K/1.5 h, no Ti or C was detected by XRD and the reaction nearly finished. It can be found that the *in situ* Ti and C reaction easily takes place when C and Ti powder particles of smaller sizes and a relatively lower synthesizing temperature down to 973 K are used for Mg-Ti-C system.

Figure 3 showed the SEM micrographs corresponding to the synthesizing conditions mentioned above for *in situ* reactive infiltration process. For the microstructures of the TiC/Mg composites, the reinforcement phase TiC has several morphologies such as interpenetrating network and spheroid-shape *etc.* The particle size of the reinforcement TiC decreases with reducing carbon particle size, and there are more TiC_{p} of $0.5\sim 2$ μm with spheroid-like morphology formed when carbon particle size was reduced to 1.5 μm .

These results suggest that for the reactive infiltration carbon sizes and the processing temperature play significant role in the morphologies of TiC formed by *in situ* process. It can be concluded that the contact area between titanium and carbon particles could be increased by hundreds of times when smaller carbon particle size is employed, and this is beneficial to the combustion synthesizing reaction at a relatively lower temperature. Illumination in detail about these reactions will be discussed associated with the mechanism of the reaction.

3.2 Influence of holding time on *in situ* reaction

To investigate the influence of the holding time on the combustion reaction, the synthesizing experiments were carried out of heating time for 15 min and 1.5 h at 953 and 973 K, respectively.

Figure 4 shows the XRD spectra for the TiC/Mg composites processed under such conditions. When holding time was 15 min (Fig.4(a)), a little TiC as the phase of reaction product was detected. This confirms that the reaction took place and reaction product was produced at such a low temperature 933 K just above the melting point of magnesium. As the holding time increased to 1.5 h (Fig.4(b)), XRD peaks of TiC became stronger gradually. When the synthesizing temperature was raised to 973 K (Figs.4(c) and (d)), XRD patterns of the specimens compared to those at 953 K with the same conditions indicated that the *in situ* reaction synthesis proceeded quickly, though the holding time is only 15 min. It is clear that the holding time at 953 K has less influence on *in situ* reaction of Ti and C than that at 973 K.

Consequently, it is believed that the synthesis initiates at 953 K with a slow velocity and as temperature is increased

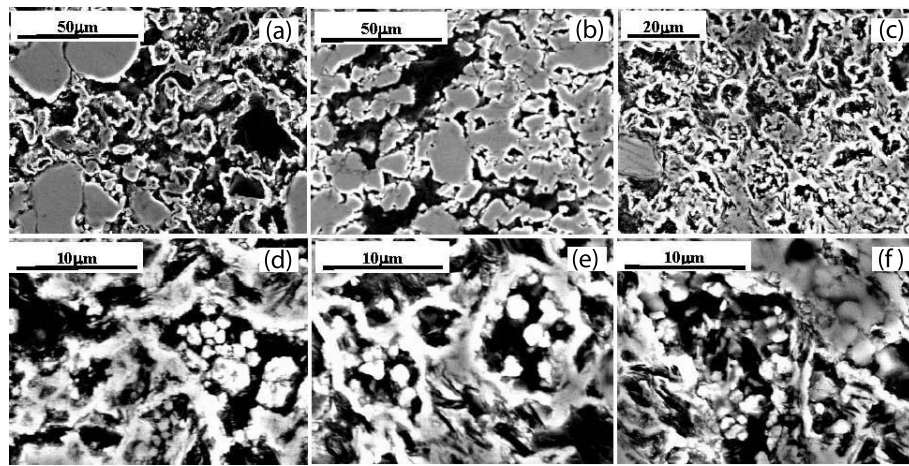


Fig.3 SEM micrographs of the as fabricated TiC/Mg composites by *in situ* reactive infiltration process. (a) T_{i_p} : $\sim 74 \mu\text{m}$, C_p : $\sim 100 \mu\text{m}$, 1023 K/1.5 h, (b) T_{i_p} : $\sim 38 \mu\text{m}$, C_p : $\sim 30 \mu\text{m}$, 1023 K/1.5 h, (c) T_{i_p} : $\sim 38 \mu\text{m}$, C_p : $\sim 1.5 \mu\text{m}$, 1023 K/1.5 h, (d) T_{i_p} : $\sim 38 \mu\text{m}$, C_p : $\sim 1.5 \mu\text{m}$, 1073 K/1.5 h; (e) T_{i_p} : $\sim 38 \mu\text{m}$, C_p : $\sim 1.5 \mu\text{m}$, 1023 K/1.5 h, (f) T_{i_p} : $\sim 38 \mu\text{m}$, C_p : $\sim 1.5 \mu\text{m}$, 973 K/1.5 h

Fig.4 XRD patterns of the specimens synthesized with T_{i_p} : ~ 38 and C_p : $\sim 1.5 \mu\text{m}$, (a) 953 K/15 min, (b) 953 K/1.5 h, (c) 973 K/15 min, (d) 973 K/1.5 h

Fig.5 XRD patterns of the specimens synthesized at 973 K/1.5 h, T_{i_p} : $\sim 38 \mu\text{m}$; C_p : $\sim 1.5 \mu\text{m}$, without Mg (a) and with Mg (b)

up to 973 K or above, reaction proceeds more quickly and completely.

3.3 Influence of magnesium melt

To investigate the influence of magnesium melt during and after the spontaneous infiltration, the experiments that the preform containing Ti and C powder with size of $1.5 \mu\text{m}$ was infiltrated by the magnesium at 973 K. A comparison was made with the $T_{i_p}+C_p$ preform without magnesium ingot. XRD patterns of the specimens are shown in Fig.5. In addition, to determine the igniting temperature for the combustion reaction between Ti and C and the role of the magnesium melt in the Mg-Ti-C system, DTA experiments were also carried out. The compacted reactants were designed in term of mole ratios of (a) Ti/C=1:1, (b) Mg/C=1:4 and (c) Mg/Ti/C=0.625:1:1, respectively. The contents of magnesium powder added to other reactants were chosen so as to simulate the composition of the TiC/Mg composites while fabricated by reactive infiltration process. These results are presented in Figs.5 and 6.

In case of Ti/C=1:1 shown in Fig.6(a), an exothermic reaction was detected in measuring temperature range of 300 to 1373 K, *i.e.* the combustion reaction between Ti and C occurred. Therefore, it is confirmed that the igniting temperature for the reaction in the Ti-C binary system was near 953 K and it largely took place around 1123 K. Figure 5(a) also revealed that although a little TiC was detected at 973 K by XRD, the reaction of titanium with carbon really happened.

Fig.6 DTA curves of compacted powder mixtures with the mole ratio of (a) Ti/C=1:1, (b) Mg/C=1:4, (c) Mg/Ti/C=0.625:1:1

For Mg/C=1:4 (Fig.6(b)), only an endothermic reaction was detected due to the melting of magnesium around 923 K. It is known that magnesium can react with carbon and the unstable product Mg_2C_3 decomposes to Mg vapor and C above 933 K. However, it was not detected in this experiment because the reactions did not take place simultaneously, some Mg_2C_3 forms while the others decompose. As a result, no reaction product of Mg with C was detected.

As for the case of Mg/Ti/C=0.625:1:1 (Fig.6(c)), there was a strong exothermic reaction observed after the endothermic reaction near the melting point of magnesium. Therefore, it was revealed that after magnesium melted by heating, the exothermic reaction between Ti and C was mostly synthesized at a lower temperature, approximately 1023 K compared to Fig.6(a). Clearly, the existence of magnesium melt is of great importance to the reaction, which makes it synthesize at a low temperature and to accelerate the reaction rate. As can be seen in Fig.5(b) that the reaction of Ti with C took place at temperature lower than 973 K and it was accelerated and reacted more completely near the temperature of 1023 K after the infiltration of magnesium melt.

3.4 Mechanism of *in situ* reactive infiltration

As an important technique for fabricating metal-matrix composites, *in situ* reactive infiltration process itself generally consists of two steps. One is the *in situ* reaction of the reinforcement from their elemental powders, and the other is the infiltration of the molten metal into the preform of the reinforcement. However, they cannot be distinguished from one another since they are always essentially overlapping and interacting during fabricating MMCs. Only one step predominates when processing conditions favor it.

For system Mg-Ti-C, the penetration of molten Mg depends mainly on its wettability to Ti and C or TiC, and its viscous behavior. Without any externally applied pressure, the capillarity acts as a driving force. The wettability of a solid by a liquid is usually indicated by the contact angle θ . This angle is related to the three surface tensions γ_{sg} , γ_{sl} and γ_{lg} of the interfaces of solid-gas, solid-liquid and liquid-gas, respectively, by the well-known Young equation^[10]:

$$\gamma_{lg}\cos\theta = \gamma_{sg} - \gamma_{sl} \quad (1)$$

a liquid is said to wet a solid surface when $\cos\theta > 0$, *i.e.* when $\gamma_{sg} > \gamma_{sl}$. It can be expressed by Young-Kelvin equation^[10]:

$$\Delta P = 2\gamma_{lg}\cos\theta/r \quad (2)$$

where, ΔP and r are the capillary attraction and the radius of pores, respectively. One can calculate from Eq.(2) that a pressure of 2~5 MPa would be generated by the capillarity with pores down to a radius r of 1.0 μm .

For metal-metal system, it is well known that molten metal with lower melting point can easily infiltrate into the preform of higher melting metal. We also found that molten Mg penetrated compacted Ti powder without any externally applied pressure. Since no intermetallic compounds form between Mg and Ti, they are physically wettable. At the initial stage, Mg can infiltrate the preform only along the pores of Ti in Mg-Ti-C once Mg gets melted. However, most Ti particles are surrounded by small C particles, *i.e.* this process will be hindered or slowed down by C participation so that Ti-C-Mg interface coexisted. The reaction of Ti with C belongs to solid-solid reaction and the reaction rate is predominantly controlled by the slowest step among diffusion of reactants toward interface, interfacial chemical reaction and transfer of products outwards. If only Ti and C do exist, a subtle TiC can form even if at 973 K due to long distance diffusion. This can be seen in Fig.5(a). The case is different when molten Mg existed in the Ti-C system and it can be interpreted from two aspects. Mg can react with C to produce unstable Mg_2C_3 , on one hand, liable to decompose into Mg and C in such a way that atomic C in molten Mg soon accumulated around Ti particles and the transfer of product TiC will be speeded up toward interface. On the other hand, the product TiC can be easily transferred outwards by diffusion through liquid phase to increase their contact area with reactants so that at 953 K there is a little TiC formed, this was confirmed by DTA result. Molten Mg has strong wettability with ceramic TiC^[11], occurrence of TiC made molten Mg further infiltrate along capillary holes till new layer of unreacted Ti and C. This

process will be spontaneously repeated till the whole preform was penetrated by molten Mg.

Next, we can estimate the infiltration time of magnesium melt. The distance x traveled by the melt along the capillaries after the time t under the capillary attraction can be described as^[12]:

$$x = [r^2t/(4\eta)]^{1/2} \quad (3)$$

where η is the viscosity of the melt. Here liquid magnesium has a viscosity of less than 10^{-3} Pa·s. If a minimum pressure of 1 MPa is applied, use of Eq.(3) shows that the liquid penetrates 15 mm of small pores with a radius about 0.5~1.0 μm in 4 s. In fact, the capillary pores are irregular, which will be influenced by many factors, such as sizes of mixture powders, distribution of Ti and C powder in preform and interfaces, *etc.* All these make the process more complicated. It will take longer time for the spontaneous infiltration to the end than that from calculation. In the experiments, 5 min or less was needed for the simultaneous infiltration of molten Mg.

As mentioned above, during *in situ* reaction Mg reacts with C and C in atomic form in molten Mg can accumulated around Ti. At this time, the infiltration of Mg nearly finished and C concentration around Ti was greatly increased. Smashed C with Ti formed numerous diffusion/reaction couples, which made diffusion distance short and provided driving force for diffusion of C and Ti. In such case, the activation energy for reaction of Ti and C will be further reduced and hence the synthesizing temperature of TiC lowered^[13]. These combinative effects lead to ease of SHS reaction. On the other hand, there is a little TiC formed at the initial stage and the infiltrated Mg homogeneously distributed within the preform, which could reduce the virtual density of the preform and slowed down the reaction rate. Obviously, the synthesizing temperature played a significant role in *in situ* reaction, as differed from the holding time affecting the diffusion process particularly at the primary stage. Due to reactive activation energy, a prolonged holding time had minor effect on the synthesizing reaction, as can be seen in Fig.4(b). At an even higher elevated temperature, SHS reaction will accomplish in a short time, which makes holding time insignificant and that is in well agreement with these experimental results. According to literature [14], the wettability of Mg to TiC varies with increasing temperature and a prolonged time will also modify the wettability. At 1173 K for holding time from 15 to 30 min, the contact angle will change from 45 to 20° and this will lead to a dense TiC/Mg composite through an extended holding time after SHS.

Based on the reaction mechanism proposed above in this paper, an explanation could be made for the morphologies of the product TiC. As shown in Fig.7, some TiC (bright part) formed around coarse Ti particles (gray part) and a mixture of Mg and C (dark part) was found to be present in capillary pores by EDS analysis. One can also find that some Ti particles cracked while molten Mg was infiltrating. This suggested that interconnected TiC occurred due to the presence of reaction between Ti and C at the initial stage of Mg infiltration.

A comparison of Figs.5(a) with (b) indicated a left shift of the XRD peaks for Ti. It was due to the dissolution of C within Ti accelerated by Mg and thus TiC formed with ease. TiC, a fcc structural compound with an alternative stoichiometry is derived from hcp Ti with octahedral interstitials filled with C. From binary diagram of Ti and C^[15], interstitial TiC compound exists in a certain region at nearly 1:1 atomic ratio and there is a (Ti+TiC) two-phase region on Ti-rich side. Molten Mg raised the solubility of C in Ti to transform toward TiC. There is a transitional composition range of C in Ti, which showed the TiC formed during primary stage not in Ti/C=1:1. With the progress of reaction of TiC and infiltration of Mg, some Ti were melted and cracked or split due to interfacial stress and high temperature. Reaction of the rest C with melted Ti consequently formed the fine particulate TiC at adiabatic temperature during the final stage of SHS,

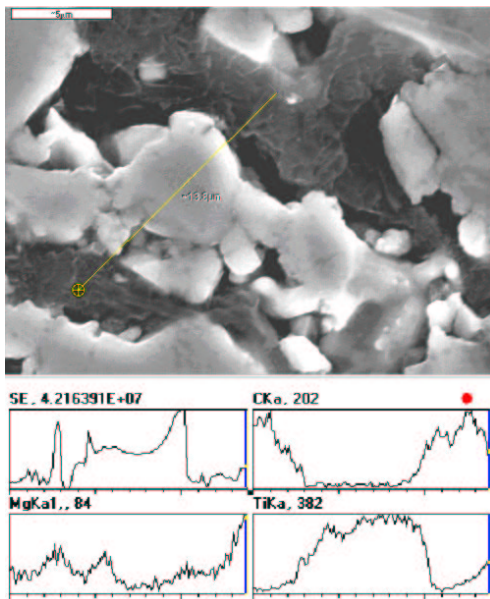


Fig.7 EDS analysis results of the composite synthesized by *in situ* reactive infiltration process (T_p : $\sim 38 \mu\text{m}$, C_p : $\sim 30 \mu\text{m}$, 973 K/15 min)

as observed in Figs.3(d)~(f).

4. Conclusions

TiC/Mg composites were fabricated by *in situ* reactive infiltration process for the Mg-Ti-C system. The mechanisms of the *in situ* reactions and infiltration process were analyzed through investigating the influences of elemental particle sizes, synthesizing temperature, holding time *etc.* The following conclusions could be drawn.

(1) Fabrication of TiC/Mg composites by *in situ* reactive infiltration depends mostly on carbon particle size, synthesizing temperature and holding time of the process. When carbon particle size of about $1.5 \mu\text{m}$ was used, the composites were well synthesized at 1023 K for 1.5 h.

(2) In fabricating TiC/Mg composites, Mg has two-fold effects. Not only it can spontaneously infiltrate the preform of reinforcement and thus densify the fabricated composites as matrix metal, but also it can accelerate the *in situ* reaction

process and lower the synthesizing temperature of Ti and C as well.

(3) *In situ* reaction of Ti and C and Mg infiltration process cannot be separated from one another. They are always essentially overlapping and interacting during fabrication of TiC/Mg composites.

(4) The reinforcement phase TiC has several morphologies such as inter-penetrating network, spheroid-shape. The *in situ* reactive infiltration mechanism proposed in this paper can be used to interpret their formation.

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REFERENCES

- [1] M.R.Krishnadev, R.Angers, C.G.Krishnadas and G.Huard: *JOM*, 1993, **8**, 52.
- [2] V.Laurent, P.Jarry, G.Regazzoni and D.Apelian: *J. Mater. Sci.*, 1992, **27**, 4447.
- [3] J.N.Hryn: *Magnesium Technology 2001*, A Publication of TMS, 2001.
- [4] N.Omura, M.Kobashi, T.Choh and N.Kanetake: *J. Japan Inst. Metals*, 2002, **66**, 1317.
- [5] N.Omura, M.Kobashi, T.Choh and N.Kanetake: *Mater. Sci. Forum*, 2002, **396-402**, 271.
- [6] Q. Dong, L.Q. Chen, M.J. Zhao and J. Bi: *Mater. Lett.*, 2004, in print.
- [7] Liqing CHEN, Qun DONG, Mingjiu ZHAO and Jing BI: *Chin. J. Mater. Res.*, 2004, in print. (in Chinese)
- [8] F.D.S.Marquis and C.A.C.Sequeira: *Key Engineering Materials*, 2002, **230-232**, 350.
- [9] P.Persson, A.E.W.Jarfors and S.Savage: *J. Mater. Process. Tech.*, 2002, **127**, 131.
- [10] Y.J.Kwon, M.Kobashi, T.Choh and N.Kanetake: *Mater. Trans., JIM*, 2002, **43**, 2769.
- [11] F.Delannay, L.Froyen and A.Deruyttere: *J. Mater. Sci.*, 1987, **22**, 1.
- [12] J.Chen and M.Y.Gu: *J. Mater. Res.*, 2002, **17**, 911.
- [13] K.A.Semlak and F.N.Rhines: *Trans. Met. Soc. AIME.*, 1958, **212**, 325.
- [14] P.G.McCormick: *Mater. Trans., JIM*, 1995, **36**, 169.
- [15] A.Contreas, C.A.Leon, R.A.L.Drew and E.Bedolla: *Scripta Mater.*, 2003, **48**, 1625.
- [16] T.B.Massalski: *Binary Alloy Phase Diagrams*, 2nd edn, ASM, The Materials Information Society, Vol.1, 1990, 890.

Synthesis and Characterizations of Nanocrystalline WC-Co Composite Powders by a Unique Ball Milling Process

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In order to explore the high efficiency of fabricating nanocrystalline WC-Co composite powders, this paper presented a unique high energy ball milling process with variable rotation rate and repeatious circulation, by which nanocrystalline WC-10Co-0.8VC-0.2Cr₃C₂ (wt pct) composite powders with mean grain size of 25 nm were prepared in 32 min, and the quantity of the powders for a batch was as much as 800 grams. The as-prepared powders were analyzed and characterized by chemical analysis, X-ray diffraction (XRD), transmission electron microscopy (TEM) and differential thermal analysis (DTA). The results show that high energy ball milling with variable rotation rates and repeatious circulation could be used to produce nanocrystalline WC-Co powder composites with high efficiency. The compositions of the powders meet its specifications with low impurity content. The mean grain size decreases, lattice distortion and system energy increase with increasing the milling time. The morphology of nanocrystalline WC-Co particles displays dominantly sphere shape and their particle sizes are all lower than 80 nm. The eutectic temperature of the nanocrystalline WC-10Co-0.8VC-0.2Cr₃C₂ composites is about 1280°C.

KEY WORDS: Nanocrystalline material, WC-Co composite, High energy ball milling

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