CURRENT RESEARCH STATUS IN THE STATE KEY LABORATORY OF METTAL MATRIX COMPOSITES

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Abstract.

Nowadays, advanced materials with high structural efficiency and structural/functional integral properties are needed by the development of technology to cope with the energy and environmental crisis. Typically, metal matrix composites (MMCs) based on light metals and alloys, with their high specific strength and high specific stiffness, have been widely used in aerospace, automotive, power, electronic and other industrial applications. The State Key Laboratory of Metal Matrix Composites (SKLMMC), China, was founded in 1991 and specialized in MMCs research and development during the last two decades. In this paper, *in situ* processes developed in SKLMMC to synthesize magnesium and titanium based MMCs are reviewed.

1 Introduction

It is widely acknowledged that the properties of MMCs are controlled mainly by the size and volume fraction of the reinforcements and the nature of interfaces between the matrix and reinforcements. Excellent mechanical properties can be achieved with fine and thermally stable ceramic particles dispersed uniformly in the matrix. Great efforts have been made to meet such requirements. However, discontinuously reinforced MMCs have been prepared traditionally by processes such as powder metallurgy, spray deposition, mechanical alloying (MA) and other casting techniques, i.e. squeeze casting, rheocasting and compocasting et al. Most of these techniques are on the basis of the adding of ceramic reinforcements into the molten or powder matrices at various temperatures. Conventional processes of MMCs can be viewed as *ex situ* techniques, because the reinforcements are prepared separately prior to the composite fabrication process. By these means, the reinforcing phase size is limited to the order of microns to tens of microns and rarely below 1 μm. Besides, the interfacial reactions between the reinforcements and the matrix, the poor wettability between the reinforcements and the matrix due to surface contamination of the reinforcements are harmful for the properties of the resulted composites [1].

Therefore, *in situ* techniques were developed for the fabrication of novel composites, in which the reinforcements are synthesized simultaneously in the matrix by chemical reactions between element(s) and compound added in the composite preparation. Compared to the conventional *ex situ* formed MMCs, the *in situ* MMCs with uniformly distributed reinforcements in the matrix exhibit many advantages, including the finer and thermodynamically stable reinforcements, cleaner interfaces and stronger interfacial bonding between the reinforcements and the matrix, thus to yield better mechanical properties. In this article, *in situ* processes of Mg and Ti based MMCs developed in the State Key Laboratory of Metal Matrix Composites (SKLMMC), China, are reviewed.

2 Mg based MMCs

As the “Green Engineering Materials in 21st century”, Mg alloys have the advantages of low density, high specific strength and stiffness, good electromagnetic shielding and damping capacities, good machining property and easy recycling capacity [2]. However, due to low elastic modulus, limited strength, poor abrasion and creep resistance, the field and range of Mg alloys application is restricted. In the meantime, Mg and Mg alloys can’t be improved effectively even by using aging strengthening, because no phase transformation occurs between solidification temperature and room temperature and the solid solubility of alloying elements is low. It is one of the effective ways to improve the performance of Mg alloys by introducing other reinforcements into Mg alloys. Traditionally, magnesium matrix composites have been produced by *ex situ* methods. Recently, the *in situ* technique has been received more and more attentions since *in situ* Mg MMCs exhibit thermodynamic stable reinforcements and cleaner and stronger bonding of the matrix-reinforcement interfaces. However, this technique is still relatively new for *in situ* Mg MMCs. Here in this article, the investigations of *in situ* reaction, fabrication, microstructure, damping capacities and creep resistance of *in situ* magnesium matrix composites are briefly summarized. The effect of reinforcement controls and the microstructure optimization on the properties of TiC/Mg and ($AlN+Mg_2Si$)/Mg matrix composites were investigated.

2.1 TiC/Mg composites

Among widely used reinforcements, TiC ceramic particles possess many desirable properties, such as high hardness, low density, high modulus and high wear resistance. Recently, Al-Ti-C as a typical system to synthesize TiC particles has been extensively studied by a number of researchers. In our works, molten Mg alloy was spontaneously infiltrated into Al-Ti-C preforms, simultaneously the *in situ* reaction happened and TiC particles were formed in the liquid of Mg alloy. Then the semisolid slurry stirring technique was used to fabricate Mg MMCs.

In this process, Ti reacted with Al to form $TiAl_3$ in the initial stage, and then C reacted with $TiAl_3$ to form TiC. Al in the preforms serves not only as a reactant and participates in the *in situ* reaction to decrease reaction temperature and TiC particle size, but also as a diluent to facilitate the diffusion and distribution of TiC particles. Fig. 1(a) shows XRD patterns of different volume fraction of TiC/AZ91D composites. The results of X-ray diffraction (XRD) confirm the presence of TiC in the composites. The as-cast microstructure of the *in situ* composites revealed the uniform distribution of TiC particulates with spherical sizes, as can be seen from Fig. 1b [3]. Fig. 1b shows a TEM bright field (BF) image of 3 vol% TiC/AZ91D composite and the corresponding selected area diffraction pattern (SADP) on particle/matrix interface. The good and clean interface is shown between TiC particle and AZ91D matrix.

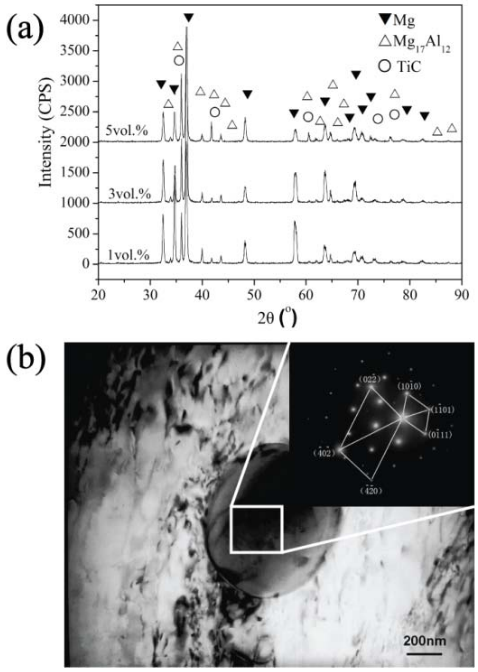

Fig. 1 (a) XRD of TiC/AZ91D composites and (b) TEM bright field image of 3vol% TiC/AZ91D composite with selected area diffraction pattern (as an inset)

Moreover, damping capacities of Mg MMCs were investigated at room temperature and elevated temperatures, as shown in Fig. 2. Damping capacities of AZ91D magnesium alloy and TiC/AZ91D composites were found to be decreased when vibration frequency increased at room temperature, while they rise with increasing temperatures with testing frequency of 0.1 Hz. Damping capacities of TiC/AZ91D composites were higher than that of AZ91D alloy. With the increase of reinforcement volume fraction, damping capacities of TiC/AZ91D composites increased. It is inferred that the increase of reinforcement is beneficial to the improvement of damping capacities of composites. Further, microstructure analysis showed high dislocation density around the reinforcements. Introducing TiC particles into magnesium matrix also improved the mechanical properties of the matrix material. Compared with AZ91D Mg alloy, the magnesium MMCs were greatly strengthened, and work hardening occurred at low temperature condition. Fig. 3 is the peak true stress of AZ91D and TiC/AZ91D [2]. Apparently, the strength of TiCp/AZ91D MMCs is higher than that of AZ91D alloy at 250 °C. The high density of dislocation, caused by the difference of coefficients of thermal expansion between reinforcement and matrix, played a vital role in the strengthening mechanism.

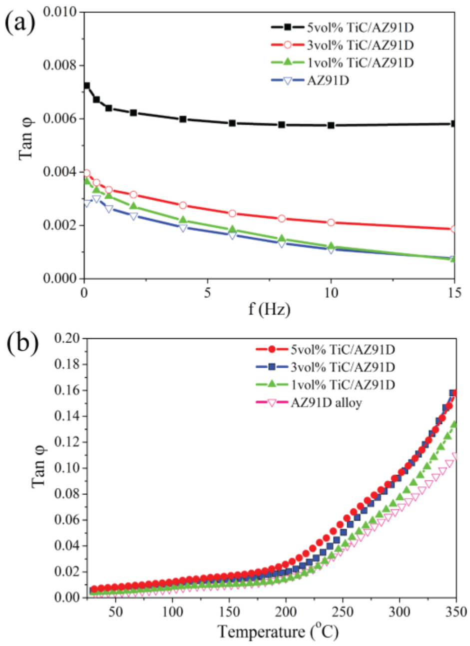

Fig. 2 Damping capacities of AZ91D alloy and TiC/AZ91D composites at: (a) room temperature with the vibration frequency; (b) various temperatures with testing frequency of 0.1 Hz

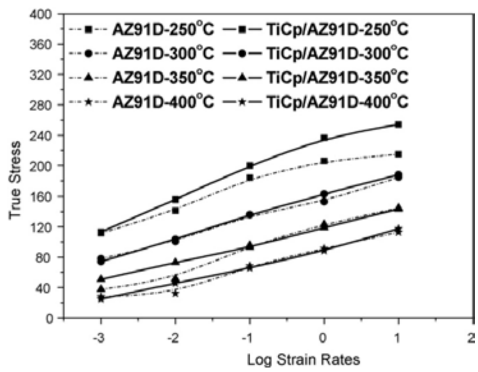

Fig. 3 Peak true stress of AZ91D and TiC/AZ91D

2.2 (AlN+Mg_2Si)/Mg composites

After fully considering the wetting between the reaction materials and Mg alloy melts, the wetting between the reinforcements and Mg alloy melts, the effective reaction systems and Mg alloys characterization, AlN and Mg_2Si particle reinforced Mg MMCs has been designed. Si_3N_4 particles and AZ91 alloys were chosen as the reaction materials and the matrix alloy, respectively.

On the basis of nucleation and growth control, the approach of selecting the effective alloying elements was developed to control the size of *in situ* formed Mg_2Si in (AlN+Mg_2Si)/Mg MMCs. An extended Miedema model and Wilson equation was used to determine the effect of alloying addition on the nucleation of Mg_2Si in Mg-Al-Si melts [4]. To verify the theoretical prediction of the effect of alloying addition on the size of in situ formed Mg_2Si phase in (AlN+Mg_2Si)/Mg composites, Ti alloying element is selected to add into the (AlN+Mg_2Si)/Mg composites. Two kinds of (AlN+Mg_2Si)/Mg composites, one without the addition of Ti denoted as S1 and the other with the addition of Ti denoted as S2, were synthesized. A schematic representation of the synthesis process of (AlN+Mg_2Si)/Mg composites is shown in Fig.4. In composites S2, 5.3wt.% Ti powders (25um) are added into the starting materials, resulting in that the mole percent of Ti in the Mg-Al-Si-Ti melts is 3% after completely reaction in Stage 3.

The X-ray profile of composites S1 and S2 revealed that whether with the addition of Ti or not, the AlN and Mg_2Si phases can be formed and the starting material Si_3N_4 disappeared, indicating that Si_3N_4 is completely converted to AlN and Mg_2Si in both composites. The difference between S1 and S2 is the formation of Ti_5Si_3 in composite S2, indicating the reaction between Ti and Si [3].

Fig. 5a, c and b, d shows the microstructure of composites S1 and S2, respectively. It can be seen that these composites yield a relatively uniform distribution of AlN (shown in A) and Mg_2Si (shown in B and C) particles, which are confirmed by energy dispersive spectrometer (EDX) equipped on SEM. With the addition of Ti, the amount and size of the first primary Mg_2Si are decreased and the size of the second primary Mg_2Si is slightly increased, respectively. Creep behaviors and compression creep properties of extruded Mg alloy and (AlN+Mg_2Si)/Mg composites were investigated, as shown in Fig.6. Creep resistance of (AlN+Mg_2Si)/Mg composites is greatly enhanced compared to that of Mg alloy. Besides, damping capacities of (AlN+Mg_2Si)/Mg MMCs were also studied at room temperature and elevated temperatures [5].

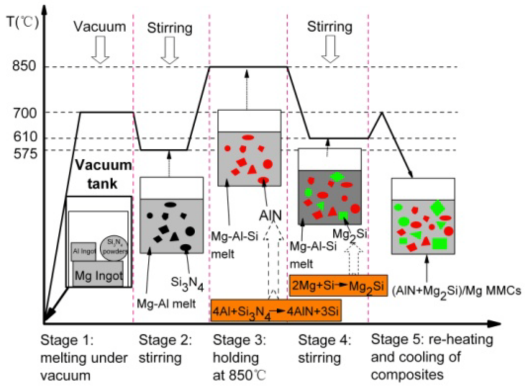

Fig. 4 Schematic rjepresentation of the synthesis process

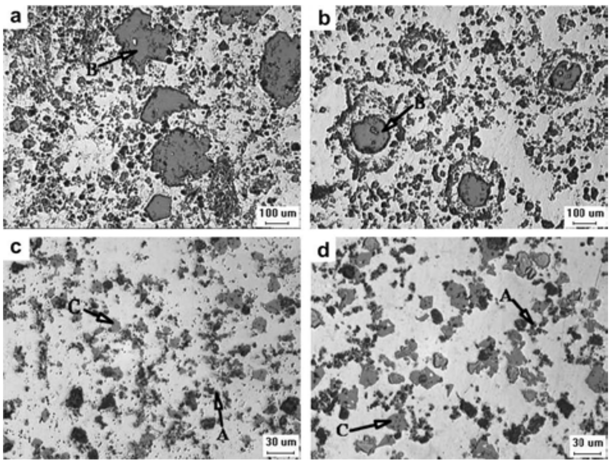

Fig. 5 Microstructure of (a), (c) composite S1, without the addition of Ti and (b), (d) composite S2, with the addition of 5.3 wt.% Ti (A: AlN particles; B: first primary Mg_2Si particles; C: second primary Mg_2Si particles)

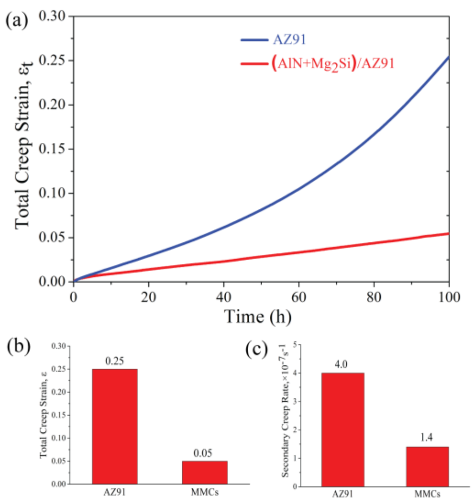

Fig. 6 Creep curves (a) and compression creep properties of extruded AZ91 and $(AlN+Mg_2Si)/Mg$ composites: (b) total creep strain at 100h; (c) secondary creep rate

3 Ti based MMCs

We also studied *in situ* formed titanium matrix composites systematically and the microstructure and mechanical properties of the *in situ* synthesized Ti MMCs has been investigated. Generally, the titanium matrix composites (TMCs) reinforced with whisker or particle are conventionally prepared by powder technology or liquid metallurgy [6], where the ceramic particles are directly incorporated into solid and liquid matrices, respectively. However, titanium metal matrices reinforced with ceramic particles formed *in situ* are an emerging group of discontinuously reinforced composites that have distinct advantages over the conventional TMCs.

Here, we highlight a novel *in situ* process, in which traditional ingot metallurgy plus self-propagation high-temperature synthesis technique were used to produce (TiB+TiC)/Ti composites. The TMCs were fabricated by common casting techniques in nonconsumable vacuum arc remelting and consumable vacuum arc remelting furnace. In order to ensure the chemical homogeneity of the composites, the ingots were melted at least three times. After casting, the ingots prepared by consumable vacuum arc remelting furnace were hot-forged into a rod of 20mm diameter. To acquire excellent mechanical properties, some rare earth elements or compounds (Nb, LaB_6, Y, et al) were added prior to the *in situ* processes.

The XRD results show that TiB, TiC or TiB and TiC reinforced titanium matrix composites can be synthesized by the common casting utilizing the chemical reaction between Ti and B, C, B_4C [7]. The reinforcements are distributed uniformly in the titanium matrix alloy [8, 9]. Fig.7 show XRD and SEM image of *in situ* formed $(TiB+TiC+Nd_2O_3)/Ti$ composites. From Fig.7b, TiB grows in whisker, while TiC grows in equiaxed shape and Nd_2O_3 in sphere [7, 10]. The microstructures of *in situ* formed $(TiB+TiC+Y_2O_3)/Ti$ and $(TiB+TiC+La_2O_3)$ composites were also shown in Fig. 8 and Fig.9, respectively, indicating good interfacial bonding between Re_2O_3 and Ti matrix [11].

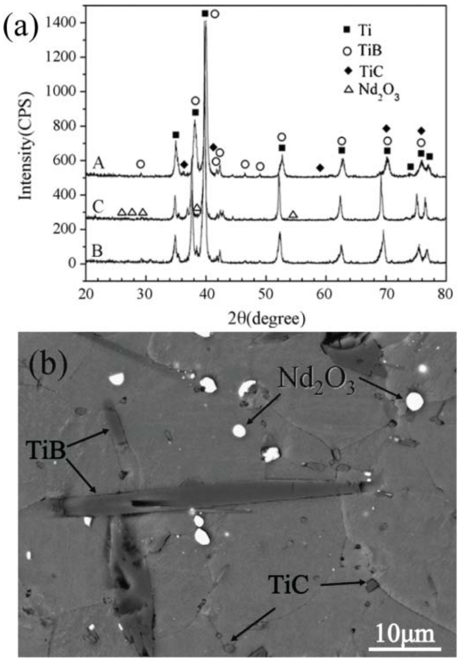

Fig.7 XRD and SEM image of *in situ* formed ($TiB+TiC+Nd_2O_3$)/Ti composites

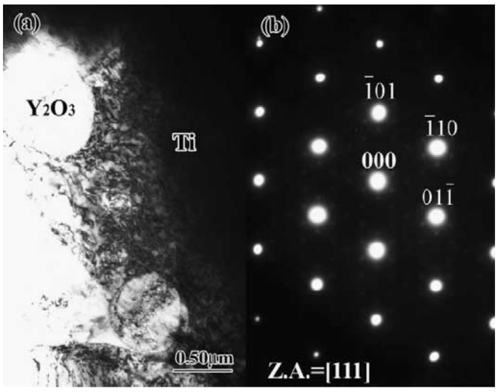

Fig. 8 TEM of Y_2O_3 and Ti and corresponding SAD patterns: (a) Bright field image; (b) SAD of Y_2O_3

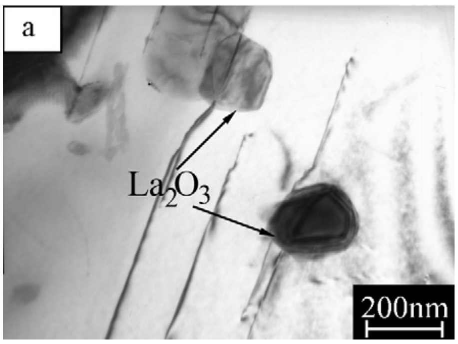

Fig. 9 The TEM micrograph of the reinforcements after 700 °C thermal exposure (a) La_2O_3 particle and (b) TiB whisker.

After the addition of rare earth elements (Nb_2O_3, La_2O_3, Y_2O_3, et al.), the properties of TMCs are improved [12-14]. Fig. 10 shows the stress-strain curves of the matrix alloy and the composites during tensile. It could be found that in situ synthesized reinforcements significantly increased the modulus and strength of matrix alloy while the plasticity decreased. Comparing the mechanical properties of TMC1 and TMC2, it was found that multiple-reinforced ($TiB+TiC+La_2O_3$)/Ti composites has higher strength and plasticity than hybrid-reinforced (TiB+TiC)/Ti composites with the same volume fraction of reinforcements, but the modulus slightly decreased. The increasing on strength and plasticity of ($TiB+TiC+La_2O_3$)/Ti may attribute to the dispersion strengthening of fine La_2O_3 particles in the matrix alloy and the decreasing of oxygen content in the matrix alloy caused by the addition of LaB_6. Other mechanical properties were also studied in our tasks [15, 16].

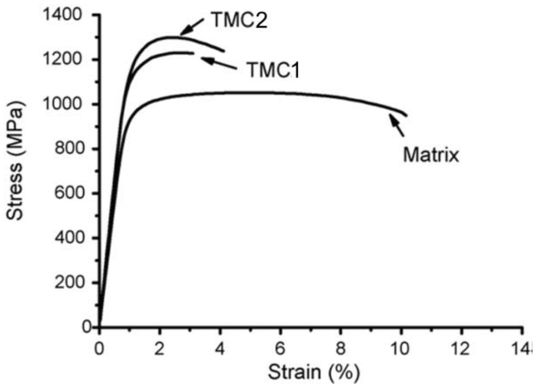

Fig. 10 Stress-strain curves of matrix alloy and titanium matrix composites (TMC1: (TiB+TiC)/Ti; TMC2: ($TiB+TiC+La_2O_3$)/Ti)

4 Summary

In summary, novel *in situ* processes developed in SKLMMC to synthesize magnesium and titanium based MMCs are reviewed. *In situ* formed Mg and Ti matrix composites with thermodynamic stable reinforcements and cleaner and stronger bonding of matrix-reinforcement interfaces were investigated. Promising microstructures and properties of *in situ* formed Mg and Ti based composites were acquired. *In situ* process has shown great potentials to obtain excellent mechanical and functional properties and to extend their industrial applications.

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