Microstructure, mechanical properties and fracture mechanism of Ti₂AlC reinforced AZ91D composites fabricated by stir casting

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Abstract: Mg-alloy (AZ91D) based composites reinforced by Ti₂AlC MAX phases particles were **firstly** fabricated by the stir casting technology. The mechanical properties in relationship with the microstructure were carefully investigated. The Ti₂AlC particles were generally distributed along α -Mg grain boundaries and strongly impeded the dendritic growth of α -Mg. Yield strength, Vickers hardness, ultimate compressive strength, and Young's moduli increased with increasing Ti₂AlC fraction in composites, while the optimized ultimate tensile strength was found for 10vol% of Ti₂AlC. **The theoretical calculation on tensile yield strength reveals that individual strengthening contribution was mainly from Hall-Petch strengthening and Forest strengthening, followed by Orwan strengthening effect.** The elastic moduli were predicated by Halpin-Tsai approach the mixture rule and the Hashin and Shtrickman equation. One aspect ratio of 3 was found by fitting Halpin-Tsai approach with experimental results. In-situ tensile test revealed the cracks were initiated in Ti₂AlC particles and propagated along α -Mg grain boundaries. During this process, no decohesion occurred at the Ti₂AlC and AZ91D interfaces.

Key words: Ti₂AlC; Mechanical properties; In-situ fracture; Microstructure; Casting.

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1. Introduction

Magnesium–alloy matrix composites are characterized by a low density, high specific strength and stiffness from room temperature to high temperature, a superior wear resistance and damping capacity.[1, 2] The requirement of these high performance materials in aerospace and automobile industries has led to extensive investigations on MMCs, as well as cost-effective fabrication technologies.[3]

To date, apart from the traditionally ceramic particles(such as TiC[4, 5], SiC[6, 7] and AlN[8]), kinking nonlinear elastic (KNE) ternary Ti_2AlC and Ti_3SiC_2 MAX phases[9, 10] are scarcely used as MMCs reinforcement [11-13]. In these so-called MAX phases, the $M_{n+1}X_n$ layers are characterized by strong covalent M–X bonds interleaved with A layers through weaker M–A bonds.[14, 15] This inherent nano-layered structure provides a unique combination of metal-like and ceramic-like properties. From their metal-like side, they possess good machinability, good electrical and thermal conductivities, as well as high thermal shock resistance. From their ceramic-like side, some of them are elastically rigid (Young's moduli >300GPa) with light weight ($4g/cm^3$) and good oxidation and fatigue resistances[16, 17]. In previous reports, MAX phases have been introduced into different metal matrices to optimize their mechanical properties, e.g. Ti_3AlC_2 –Cu[18], Ti_2AlC –Cr[19] and Ti_3AlC_2 –Al[20] composites. Recently, Anasori *et al.* have reported that the fabricated Ti_2AlC/Mg composites are not only machinable, stiff and light, but also exhibit higher damping capabilities.[11, 21] In comparison with **TiC reinforced Mg composites**, the stronger interfacial bonding **endows Ti_2AlC/Mg** with better mechanical properties than TiC/Mg[11]. Furthermore, it has been reported that molten Mg can wet the

Ti₂AlC, especially with increasing Al content in Mg-alloy.[12] In addition, sub-micron Mg grains can be formed on Ti₂AlC grain surface in melt infiltration method.[12]

Concerning the Ti₂AlC/Mg MMCs fabrication methods, only melt infiltration and powder metallurgy methods have been reported for 50vol% Ti₂AlC/pure Mg or Mg alloy and 5-20vol% Ti₂AlC/**pure Mg** composites, respectively.[11] In comparison, stir casting is much more adaptable and economically applicable due to its low processing cost and high production rate. In addition, this process enables near-net shape formation.[8, 22] To date, there are no reports on the fabrication of Ti₂AlC/AZ91D composites by stir casting. Therefore, the objective of the present work was to fabricate Ti₂AlC reinforced MMCs by stir casting, and to investigate their microstructure and mechanical properties. Furthermore, *in-situ* scanning electron microscopy (SEM) was adopted to observe the crack initiation and propagation during tensile test.

2. Experimental method

2.1 Casting process

In this study, AZ91D magnesium alloy (Al:9.0, Zn:0.81, Si:0.1, Mn:0.24, Fe:0.022, Cu:0.030, Ni:0.005, Mg: rest) was selected as the matrix. Laboratory-made Ti₂AlC particles with diameter below 10 μm were selected as the reinforcement. First, AZ91 alloy was melt under protective atmosphere of mixed CO₂/SF₆. Second, the melt was cooled down to the semi-solid condition under stirring. At the same time, preheated Ti₂AlC particles were added into the melt. After semi-solid stirring, the melt was reheated to 700°C and then stirred again in the liquid condition without the vortex formation. Finally, the melt was poured into a preheated metal mold and solidified under about 100MPa. For convenience, the samples were labeled and

summarized in Table 1. Furthermore, the densities of the matrix alloy and composites, were measured using the Archimedes' principle.

2.2 Microstructure characterization

For SEM observation, the specimens were ground with silicon carbide and then successively polished with diamond suspensions ranging from 6 μ m to 0.25 μ m. Chemo-mechanical polishing was performed using Al₂O₃ suspension (particle size: 0.04 μ m) to avoid the work-hardening caused by conventional grinding. The samples were etched with a diluted acetic acid solution (50 ml distilled water, 150 ml anhydrous ethyl alcohol and 1 ml glacial acetic acid) in order to observe and estimate Mg grain size,. Phase identification was performed by X-Ray Diffraction (XRD) using a Bruker (Karlsruhe, Germany) D8 diffractometer with CuK_a radiation. Microstructures and phase compositions were examined by Optical Microscopy (OM), Scanning Electron Microscopy (SEM), **For TEM observation, cross-sectional laminae were sliced with FIB in a dual-beam FIB/SEM (Carl Zeiss Auriga Cross Beam FIB-SEM Workstation, Germany) with a Ga ion source operating at 30 kV.**

2.3 Property evaluation

Mechanical properties were evaluated in term of tensile and compressive properties, as well as microhardness. Tensile and compression tests were performed on a universal servo-hydraulic mechanical testing machine (Instron 5600, Norwood, MA) with a strain rate of 0.5mm/min at room temperature in air. The samples were fabricated following **GB228 flat specimen standard**. The dimensions were 18mm long (gauge), 3mm wide and 2mm thick for tensile test. For the compression test, cylindrical specimen of 8mm diameter and 12mm height were prepared. The microhardness measurements were performed using a Vickers microindenter (LECO

Corp., St. Joseph, MI) at a load of 10 N held for 15 s. Each test was repeated six times for each composite in order to evaluate the corresponding mechanical properties.

2.4 Fracture mechanism

To investigate the crack initiation and propagation, the *in-situ* tensile test is widely used.[23, 24] As shown in Fig.1, a servo-hydraulic loading system was installed in the vacuum chamber of a scanning electron microscope (Shimadzu Corporation, Japan). The tensile experiment was performed by fixing one side of the specimen, while moving the other one at constant speed. During a regular test, the loading speed was set constant at 0.001 mm/min.

3. Results and discussion

3.1 Microstructure of the composites

Fig.2a-d shows the SEM images of four composites with different Ti_2AlC volume fraction. The A5 and A10 composites show uniform particle distribution. However, with the increasing volume of reinforced particles, the agglomeration of Ti_2AlC particles appears in A15 and A20, especially in A20. Fig.2e-f reveals the morphologies of cast AZ91D and A10 under BSE mode. Fig.2e depicts distinguishable primary α -Mg and primary β - $\text{Mg}_{17}\text{Al}_{12}$ phases along the grain boundaries, and the another high Al content eutectic α -Mg in primary β - $\text{Mg}_{17}\text{Al}_{12}$ phases.[25] Like the distribution of $\text{Mg}_{17}\text{Al}_{12}$, Fig.2f indicates that Ti_2AlC particles distributes along the primary α -Mg grain boundaries. Furthermore, the enlarged area in Fig.2f reveals that Ti_2AlC and $\text{Mg}_{17}\text{Al}_{12}$ could co-exist together. The sharp and clear interface indicates no reaction between Ti_2AlC and $\text{Mg}_{17}\text{Al}_{12}$.

The XRD analysis corresponding to Fig.2 was given in Fig.3a. It reveals that there is no detectable trace about impurities except of AZ91D (α -phase and β -phase) and Ti_2AlC . Furthermore, a region - wherein Mg penetrated a pre-existing crack in a Ti_2AlC particle in a Mg- Ti_2AlC composite - was imaged in the TEM (Fig.3b). It shows that Mg could penetrate into the sub-micron cracks among Ti_2AlC particles. The backscatter electron STEM micrograph (Fig.3c) confirms no chemical reaction happens between Ti_2AlC and AZ91D. In addition, the enlarged area in Fig.3a indicates Mg peak shifted to the higher angle with increasing Ti_2AlC content in composites. In Fig.8, the OM images indicate that Ti_2AlC particles segregated within dendritic α -Mg intergranular regions due to the “push” effect of solidification front. During the solidification of MMS composites by stir casting, most particles are segregated in the intergranular regions during the impingement with other growing grains.[26, 27] As no chemical reaction happens, the shift of Mg peak could be ascribed to the compressive force resulting from the restriction of dendritic α -Mg growth because of intergranular dispersed Ti_2AlC particles. Furthermore, the zone shown in Fig.3c was milled and the corresponding TEM images were given in Fig.4. It reveals that the molten AZ91D can wet the Ti_2AlC . This result is similar to the previous report, the molten Mg can wet the Ti_2AlC , especially in Mg-Al alloy. [12], In addition, sub-micron Mg grains was found among the Ti_2AlC particles, which was also found in melt infiltration method.[12]

The fracture surfaces of specimens after tensile test are given in Fig.5. Less plastic matrix deformation happens with the increasing Ti_2AlC particles volume in

composites. The enlarged area in Fig.5a reveals that Ti_2AlC particles are surrounded by a thin layer of Mg except for the clean fracture zone. This result is in agreement with the reported work on Mg- Ti_2AlC composite produced by hot pressing and melt infiltration methods. The latter results suggested that this thin Mg layer could create stronger interlocks between matrix and reinforcement, than in the case of TiC and Mg. [11, 28] Finally, no Ti_2AlC -matrix interfacial decohesion was found, unlike the cases of TiC and SiC[11, 29, 30]. In this work, the composites only revealed a mixed fracture mode of matrix plastic deformation and Ti_2AlC particles delamination (enlarged area of Fig.5b).

Furthermore, the fracture surfaces of specimens after compressive tests are given in Fig.6. For all the AZ91D and AZ91D- Ti_2AlC composites, the fracture occurred 45° with respect to the compression loading axis. The fractographic images indicate the presence of shear bands in all samples. Due to the “pinning effect” of Ti_2AlC in AZ91D matrix, the serious torn zone appeared above the Ti_2AlC in the glide direction, as shown in Fig.6c. With the increase in Ti_2AlC volume, above 10%, cracks propagate perpendicular to compression axis, i.e. roughly corresponding to the maximum resolved shear stress. All types of crack locations are observed, that is, cracks at matrix-particle interfaces. As already observed in MAX phases,[31, 32] Fig.6d displays the typical kink bands with very sharp radii of curvature and delamination of Ti_2AlC MAX phases. This suggests that Ti_2AlC particles reinforced the AZ91D matrix and also participated in the plastic deformation. For example, Anasori *et al.*

pointed out that using Ti_2AlC could improve the mechanical properties of Mg composites as much as TiC , and also achieve/maintain high damping properties.[33]

3.2 Mechanical properties

Elastic modulus

In order to compare the experimental results of the composites with the theoretical evaluations of elastic modulus, the means of the mixture rule expression (Eq. (1)), the Halpin-Tsai equation [34] (Eqs. (2) and (3)) and the Hashin and Shtrickman equation (Eq. (4))[1] were adopted:

$$E_c = E_m V_m + E_r V_r \quad (1)$$

$$E_c = \frac{E_m(1 + 2sqV_r)}{1 - qV_r} \quad (2)$$

$$q = \frac{E_r/E_m - 1}{E_r/E_m + 2s} \quad (3)$$

$$E_c = E_m * \frac{E_m V_m + E_r(V_r + 1)}{E_r V_m + E_m(V_r + 1)} \quad (4)$$

where E_c = elastic modulus of composite (GPa), given the E of Ti_2AlC is 278GPa[35]. V_m = % volumetric matrix, V_r = % volumetric reinforcement, E_m = matrix modulus (GPa), E_r = reinforcement modulus (GPa), s = aspect ratio.

The measurements of elastic modulus measured, as well as the values from mixture rules, Hashin–Shtrickman, and Halpin-Tsai equations for different aspect ratio are shown in Fig.7. The mixture rule is generally used for continuous fiber-reinforced composites. Typically, Halpin-Tsai model is a mathematical model for the prediction of elasticity of composite material based on the geometry and orientation of the reinforcement and the elastic properties of the reinforcement and

matrix. Several aspect ratios were adopted in order to observe the effect of aspect of the reinforcement. The best results were obtained for aspect ratio of 3.

Yield strength (YS) and Vickers hardness (VH)

The evolution of VH and YS is directly related to the capacity of the material to delay the dislocation motion. Three sources of dislocation mobility hindering are available[5]: the grain boundaries can be estimated through the Hall-Petch law(σ_{HP})[36], the presence of Ti_2AlC particles can be estimated from Orowan strengthening effect (σ_{Or}), and the thermal strain due to the mismatch of thermal coefficients (σ_{CTE}).

$$\sigma = \sigma_{Mg} + \sigma_{Orowan} + \sigma_{Hall-petch} + \sigma_{CTE}$$

$$= \sigma_{Mg} + M \frac{0.4Gb}{\pi\bar{\lambda}} \frac{Ln(\bar{d}/b)}{\sqrt{1-v_{Mg}}} + Kd^{-0.5} + AMGb \left(\frac{12\sqrt{2}\Delta\alpha\Delta Tf}{bd(1-f)} \right)^{0.5} \quad (5)$$

$$\bar{d} = d \sqrt{2/3} \quad (6)$$

$$\bar{\lambda} = \bar{d} \left(\sqrt{\pi/4f} - 1 \right) \quad (7)$$

where, M is the mean orientation factor for Mg (6.5) [37], A is a constant of dislocation forest for Mg (0.2)[37], G is the shear modulus of Mg(17.3GPa)[38], b is the burgers vector of Mg (3.21×10^{-10} m), d is average particle size of Ti_2AlC (6 μ m), v_{Mg} is the Poisson's ratio of Mg (0.35)[2], f is the volume fraction of Ti_2AlC , ΔT is temperature change during matrix solidification (assumed to be 475°C), K is the constant value of the material ($K=280$ for AZ91). CTE ($7.1 \pm 0.3 \times 10^{-6} K^{-1}$ and $10.5 \pm 0.5 \times 10^{-6} K^{-1}$ for a- and c- direction in Ti_2AlC , $26 \times 10^{-6} K$ for Mg) [39]

Fig.8 (a) plots the individual strengthening effect theoretically calculated by eqs.(5-7). It was found the Hall-petch strengthening effect is the most dominant. As can be seen in Fig.8 (b-f), the α -Mg grain size strongly decreased with increasing content of reinforcement. Similarly, it indicates that σ_{CET} has also a strong contribution to yield strength. This should be introduced by the mismatches of CTE between Ti_2AlC and Mg-alloy ($7.1\pm0.3\times10^{-6}K^{-1}$ and $10.5\pm0.5\times10^{-6}K^{-1}$ for a- and c-direction in Ti_2AlC , $26\times10^{-6}K$ for Mg), which could produce high dislocation densities and thermal strain in the vicinity of matrix/reinforcement.[11, 39]. In the case of micro-composites, the effect of Orowan strengthening is not significant due to the presence of coarser particulates and large inter particulate spacing.[40].

Ultimate tensile strength (UTS) and Ultimate compressive strength (UCS)

Table 2 compares the tensile yield strength, UCSs, UTSs and Vickers hardness values of pure AZ91D alloy and the composites fabricated. In this work, the tensile yield strength, UCSs, UTSs and Vickers hardness except of UTSs increase with the increasing Ti_2AlC volume fraction. The A10 composite presents the optimal value of UTS, which is also higher than the reported ones in SiC reinforced AZ91D composites fabricated by stir casting.[41] [42] In addition, the elongations of composites clearly decreased with the increasing Ti_2AlC volume in composites in this work. As shown in Fig.5, the shorter tear edges suggest that less Mg participated to the plastic deformation with the increasing volume fraction of reinforcement. For easier capturing the fracture detail and classifying the role of reinforcement in tensile test, the in-situ observation experiment was carried out on A5 specimen with lowest

content of Ti_2AlC .

4. Fracture behavior observation by **in-situ tensile test**

Fig.9 presents the curve of applied load as a function of displacement of the A5 composite. The aslant discontinuous tails of the line are related to the several interruptions performed during the mechanical test for the purpose of capturing the constantly changing morphology of the specimen.

Fig.10 (a-h) represents the detailed deformation about crack initiation and propagation in Ti_2AlC reinforced AZ91D composite and corresponding to the interruption points of solid line indicated in Fig.9. First, at the yielding point marked as “b”, no cracks were found. With increasing loads, one “spring curve” appears in tensile curve. Corresponding to the point “c-e” in Fig.9, Fig.10 (c-e) reveals some broken Ti_2AlC particles into two parts. **This may be aroused by the shape and orientation of Ti_2AlC particles.** Meanwhile, no interfacial decohesion occurred between Ti_2AlC and AZ91D. After that, slipping bands appears in the vicinity of Ti_2AlC -matrix interface. Due to the mismatch in CTE, some plastic deformation occurs in Mg-alloy matrices close to the interface leading to the nucleation of dislocations and concomitant strengthening.[11] As shown in Fig.10(g), the shearing deformation of AZ91D matrix among the broken Ti_2AlC particles is clearly exposed. Fig.10(h) reveals cracks **propagated along grain boundaries where exists the $\text{Mg}_{17}\text{Al}_{12}$ (β -phase),** but not in the whole sheared Mg-alloy matrix interface. Finally, the tendency of the curve dropped at the end of the plateau because of the connection of cracks among the cracked Ti_2AlC particles. Contrary to SiC-Mg alloy where the

interface decohesion was observed in SiC–Mg alloy,[43, 44], here, the Ti_2AlC remains combined to the Mg-matrix. It confirms that the Ti_2AlC and AZ91D have a strong interfacial bonding.

For the Ti_2AlC particles to fracture, they must be loaded to their fracture stress. This is achieved globally by the tensile stress and locally by shear loading through the interface. The extent of particle loading by the shear mechanism is dependent on the aspect ratio of the reinforcing particles. For the case of symmetrically packed particles in the metal matrix, the aspect ratio (S) for maximum loading is[45]:

$$S = \frac{\sigma_{Ti_2AlC}}{\tau_i} \quad (8)$$

where σ_{Ti_2AlC} is the strength of the particle (above 1000MPa)[46] and τ_i is the interfacial shear strength.

With the aspect ratio ($S=3$) predicated by Halpin-Tsai approach, we can predict the interfacial shear strength (about 330MPa). This value is higher than the UTS of fabricated composites (215MPa for A10), it is reasonable that no Ti_2AlC -AZ91D interface decohesion happened in this work. Finally, the cleaned delaminated Ti_2AlC surface was observed in [Fig.5](#).

5. Conclusion

The primary conclusions of this study are as follows.

- 1) AZ91D reinforced with 5, 10, 15, 20vol% Ti_2AlC were successfully fabricated under 700°C by the stir casting method. Yield strength, Vickers hardness, ultimate compressive strength, and Young's moduli increased with increasing Ti_2AlC fraction in composites, while the optimized ultimate tensile strength

was found when the Ti_2AlC fraction is 10%.

- 2) No interfacial reaction happened between Ti_2AlC and AZ91D. One thin nano-Mg layer surrounding Ti_2AlC was found, which could support a strong interfacial bonding between Ti_2AlC and AZ91D. The elastic moduli were close to the values predicated by Halpin-Tsai approach with an aspect ratio of 3.
- 3) The calculation of individual contribution strengthening effects reveals Hall-Petch strengthening and Forest strengthening are the dominate roles on the tensile yield strength while not Orowan strengthening effect.
- 4) The reinforcement could strongly restrict the α -Mg grain growth. During the compression test, the typical kink band behavior of Ti_2AlC occurred, which promises this composite a good damping property.[21, 35]
- 5) In-situ observation reveals the cracks were initiated in Ti_2AlC particles and propagated along α -Mg grain boundaries. As the Ti_2AlC - AZ91D interfacial shear strength was predicated above 330MPa, no interfacial decohesion occurred between Ti_2AlC and AZ91D. This behavior is different from the cases of SiC-AZ91D[43], SiC/Mg-6%Zn[44], TiC-Mg[11].

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7. References.

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Fig.1: Configuration of *in-situ* tensile test with specimen (unit: mm).

Fig.2: The SEM images of the composites with different Ti₂AlC fraction (a)5%, (b) 10%, (c)15% and (d)20% in SE mode; (e) cast AZ91D alloy and (f) 5% in BSE mode.

Fig.3: (a) XRD pattern of AZ91D alloy and the composites (b) and (c) TEM and STEM morphologies of AZ91D and Ti₂AlC particles.

Fig.4: TEM micrographs of AZ91D-Ti₂AlC interface.

Fig.5: Fracture surfaces of the composite with different Ti₂AlC reinforcement volume (a) 5%, (b) 10%, (c) 15% and (d) 20% after tensile tests.

Fig.6: Fracture surfaces of the composite with different Ti₂AlC reinforcement volume (a) 5%, (b) 10%, (c) 15% and (d) 20% after compressive tests, (c) and (d) magnified zone marked in (b), the compressive axis direction was marked in image.

Fig.7: Elastic modulus results in function of the volume fraction of reinforcement.

Fig.8: (a) The calculated contribution of σ_{HP} , σ_{CTE} and σ_{OR} to the yield strength with the increasing volume fraction in composites; (b-h) morphologies of the as

casted alloy and composites after being etched.

Fig.9: The typical tensile curves of 5% Ti₂AlC reinforced AZ91D.

Fig.10: (a)~(h) represents the detailed deformation morphologies of the specimen corresponding to the interruption noted on solid line indicated in Fig.9.

Table 1: Material designation and characterization results obtained from the alloy and composites.

Table 2: Mechanical properties of fabricated alloy and composites with the reported, including Elastic moduli (E), 0.2% yield strengths (YS), ultimate tensile strengths (UTS), ultimate compression strengths (UCS) and Vickers hardness, VH.

Fig.1

Fig.2

Fig.3

Fig.4

Fig.5

Fig.6

Fig.7

Fig.8

Fig.9

Fig.10

Table 1

Material	Designation	Theoretical density (g.cm ⁻³)	Experimental density (g.cm ⁻³)	Mg grain size (μm)
AZ91D	A0	1.810	1.807	400
AZ91D/5%vol.Ti ₂ AlC	A5	1.9245	1.912	200
AZ91D/10%vol.Ti ₂ AlC	A10	2.039	2.013	100
AZ91D/15%vol.Ti ₂ AlC	A15	2.1535	2.132	70
AZ91D/20%vol.Ti ₂ AlC	A20	2.268	2.197	30

Table 2

Materials	Reinforcement particle size (μm)	Processing	E (GPa)	0.2% YS (MPa)	UTS (MPa)	Elongation (%)	UCS (MPa)	V _H (GPa)	Ref.
A0		cast	42	73	180	5.3	275	0.65	This work
A5	Below 10	cast	52	108	200	3.4	367	0.85	This work
A10	Below 10	cast	56	135	215	1.9	382	1.10	This work
A15	Below 10	cast	62	145	185	1.2	395	1.21	This work
A20	Below 10	cast	69	160	165	0.7	400	1.32	This work
AZ91D		cast	40		152-200				[42]
10-SiC-AZ91D	10	Powder metallurgy	45-50		135	0.47			[42, 47]
10-SiC-AZ91D	10	cast		120	172	1.3			[41]
15-SiC-AZ91D	15	Rheocast-T4		182	192	1.8			[30]
5-Ti ₂ AlC-Mg	Below 40	Hot Pressing	54	128	-		339	0.9	[11]
5-TiC-Mg	7	HP	42	123	-		337	0.88	[11]