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TiC/AZ91D composites fabricated by in situ reactive infiltration process and its tensile deformation

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Abstract: An innovative processing route was adopted to fabricate 42.1%(volume fraction) TiC/AZ91D magnesium matrix composites. The reinforcement TiC was in situ synthesized from elemental powders of Ti and C and the matrix magnesium alloy AZ91D pressurelessly infiltrated into the preform of Ti and C. A comparative tensile deformation tests were conducted on the as-synthesized TiC/AZ91D composites and magnesium alloy AZ91D. The true strain—stress curves were fitted by Hollomon relation and their failure mechanisms were finally analyzed. The results show that the in situ formed TiC can increase the tensile strength, and is especially effective at elevated temperatures. Theoretical calculation of the strain hardening exponent (n) for TiC/AZ91D composites indicates that the n value ranges from 0.71 to 0.82 when tensile deformation was carried out at 423−723 K and shows fracture with brittle characteristic. However, the n value of 0.11−0.32 obtained for the matrix alloy AZ91D shows typical ductile features at elevated temperatures.

Key words: magnesium-matrix composites; TiC/AZ91D; in situ reactive infiltration; tensile deformation; strain hardening; fracture mechanism

1 Introduction

Relative to aluminum matrix composites, magnesium-matrix composites are receiving interests increasingly in recent years due to their low densities and high specific properties. They are potentially attractive for the applications in aerospace, electronic and automotive industries.

Although the conventional processing routes, i.e. powder metallurgy technique and stirring casting, can be adopted to fabricate magnesium matrix composites with deliberate operations, they are inevitably causing extra costs. Moreover, the reinforcement phases associating with these methods are ex situ brought and detrimental to the reinforcement/matrix interface. In situ formed reinforcement phases are generally considered to be ideal, since they have clean interface excluded from outside pollution and can avoid the interfacial chemical reactions so as to achieve good bonding with matrix and thermodynamic compatibility[1]. Sometimes, the secondary forming process, i.e. hot-extrusion and/or rolling process, is required to obtain dense composites. Low-cost pressureless infiltration technique is an alternative to produce metal-matrix composites(MMCs)[2−5], the same problem as low interfacial bonding strength in ex situ routes also exists in this process. Other in situ processing techniques also appeared associating with stirring casting route, e.g. the preparation of preform and immersing into the metal melt[6,7], which still consists of complicated procedures and is unable to be made in a near-net shape.

Recently, a novel processing route, in situ reactive infiltration technique, has been developed for synthesizing MMCs, by which aluminum and magnesium matrix composites have been successfully fabricated[8−13]. The advantages of this method can be ascribed to the following. Firstly, it combines the in situ reaction with pressureless infiltration process, so the thermodynamically stable and homogeneously dispersed

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fine reinforcing phases are in situ formed within the matrix, or the matrix and ceramic phase are inter-penetrated or co-continuous, which could lead to a significant increase in their mechanical properties. Secondly, the preform can be easily shaped and the composites produced into a near-net shape with a high ceramic volume percentage and cost effectively. Dense composites can be obtained by infiltration of molten metal into the preform and no further forming procedure is required. Finally, the volume percentage of the ceramic reinforcement can be tailored just by simply controlling the relative density of the preform compacted from elemental powders and by considering the intrinsically volumetric contraction before and after in situ reaction[14].

In this paper, we adopted this processing route and synthesized 42.1% TiC/AZ91D magnesium matrix composites. The tensile deformation behaviors were then comparatively investigated for the as-fabricated TiC/AZ91D composites together with the unreinforced matrix magnesium alloy AZ91D at room and elevated temperatures up to 723 K. Particularly, the strain hardening effects and fracture mechanism were analyzed on the basis of the Hollomon relation in terms of strain hardening exponent and fractured surfaces.

2 Experimental

The experimental raw materials are Ti powder particles (about 38 μm in size, purity ＞99.5%), C particles (about 1.5 μm in size, purity ＞98.5%) and AZ91D magnesium alloy cast ingot (Mg-9.0Al-0.6Zn-0.2Mn, mass fraction, %). The TiC/AZ91D magnesium matrix composite was synthesized by in situ reactive infiltration process and the details can be described as follows.

Firstly, the mixed Ti and C powders (molar ratio of Ti to C is 1:1), after full mechanical blending, were compacted into preform with dimension of 25 mm×60 mm×15 mm in a steel mould and relative density of about 56.6%. Assuming that all Ti and C powders will fully transform into TiC phase in pre-designed molar ratio during in situ reactive infiltration process, the TiC/Mg composites with reinforcement volume fraction of 42.1% could be obtained according to the product of TiC phase formed in situ in magnesium matrix and by considering the intrinsically volumetric shrinkage of the reactants due to the in situ reaction. Under the condition of 1 073 K, 1.5 h, the practical volumetric contraction of the reactants is about 14.5%, while the theoretically calculated value is approximately 13%[14].

Secondly, the (Ti_p+C_p) preform together with magnesium alloy ingot on it was put into a graphite crucible. Several small holes were drilled at the bottom of the crucible in order to release the air during fabricating TiC/Mg composites. The reaction chamber was degassed prior to heating and then backfilled with Ar gas (purity ≥99.999%). In situ reactive infiltration experiments were finally carried out in a resistance heating furnace under the flowing Ar atmosphere. The heating temperature was set as 1 073 K with holding time 1.5 h. Two-stage heating rate was used, i.e. 10 K/min when T＜933 K and 5 K/min when T≥933 K. Following these, the samples were cooled down to room temperature together with the furnace. T6 heat treatment (686 K, 18 h air cooling followed by 441 K, 16 h furnace cooling) was applied to all samples.

The tensile specimens with gauge of 20 mm×4 mm ×2 mm were polished prior to testing and the experiments were carried out in a hydraulically driven fatigue testing machine (Schenck- PZV1795, Germany) with a strain rate of 0.001 s^{-1} at room and elevated temperatures of 423−723 K. Holding time was set to be 5 min for magnesium matrix composites and 2min for magnesium alloy. The load-displacement curves were automatically plotted by computerized system and the true stress—true strain (σ_{tr}−ε_{tr}) curves were converted by relations as

$$\sigma_{tr}=\frac{Fl_0}{S_0(\Delta l+l_0)} \tag{1}$$

$$\varepsilon_{tr}=\ln[(l_0+\Delta l)/l_0] \tag{2}$$

where F is load, S_0 is the initial cross-sectional area, l_0 is the initial length of the gauge and Δl is the displacement.

3 Results and discussion

3.1 Tensile deformation behavior

Fig.1 shows the true tensile stress—strain curves for AZ91D alloy and TiC/AZ91D composites with a tensile strain rate of 0.001 s^{-1} at room and elevated temperatures, from which we can see that the tensile strength decreases with increasing deformation temperatures for both materials. The tensile strength of TiC/AZ91D composites is always higher than that of magnesium alloy AZ91D, which can be attributed to the introduction of TiC phases in composites. At room temperature (298 K), the tensile strength of AZ91D alloy is 175 MPa and that of TiC/AZ91D composites is 203.4 MPa, only increased by 16.2%. However, the tensile strength of the TiC/AZ91D composites is significantly improved as compared with the unreinforced matrix alloy at elevated temperatures. For example, at 723 K the tensile strength of the TiC/AZ91D composites is 95.2 MPa, increased by 180%, while that of the AZ91D alloy is only 34 MPa. This can be clearly illustrated in Fig.2.

As expected, during the whole tensile deformation process the TiC/AZ91D composites possess rather limited plasticity, less than 2% at the experimental

temperatures. This differs from the case of the matrix alloy AZ91D. For the latter, the elongation to failure increases with increasing deformation temperature and exceeds 20% when deformed at 723 K.

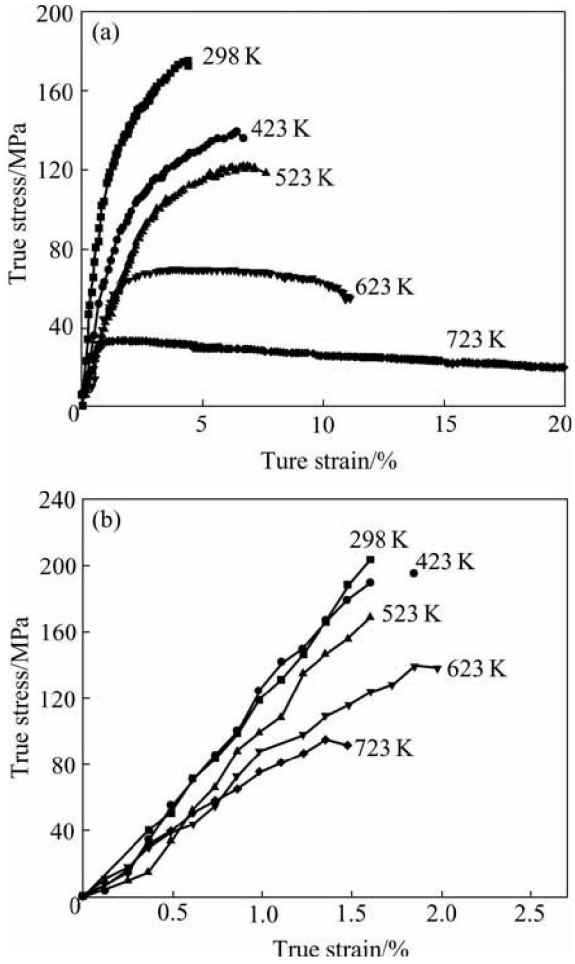

Fig.1 True tensile stress–strain curves for magnesium alloy AZ91D(a) and 42.1% TiC/AZ91D composites(b) at strain rate of 0.001 s^{-1} and different deformation temperatures

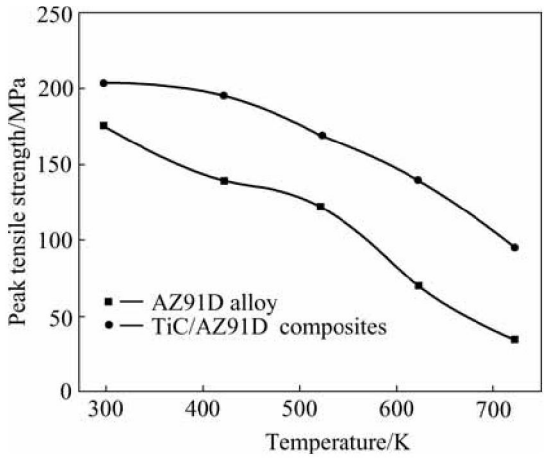

Fig.2 Plot showing peak true tensile stress versus deformation temperature for AZ91D alloy and TiC/AZ91D composites at strain rate of 0.001 s^{-1}

3.2 Strain hardening behavior

Provided that the plastic deformation in metal or alloys is performed at a given temperature with a constant strain rate, we can calculate the strain hardening exponents with the following Hollomon relation[15]:

$$\sigma = K\varepsilon_p^n \tag{3}$$

where σ is true stress, ε_p is true plastic strain, n is strain hardening exponent and K is a constant related to strain hardening. If we take natural logarithm for the both sides of the above equation, the linear relation ($\ln\sigma$–$\ln\varepsilon_p$) can be obtained as

$$\ln\sigma = \ln K + n\ln\varepsilon_p \tag{4}$$

where the slope, n, denotes the strain hardening exponent. For the AZ91D magnesium alloy and TiC/AZ91D composites, the strain hardening exponents have been calculated, respectively, on the basis of the true tensile stress—strain curves (Fig.1) and Eqn.(2). The results are presented in Fig.3 and Table 1.

From Fig.3, one may notice that the linear relationship between $\ln\sigma$ and $\ln\varepsilon_p$ is well established. It

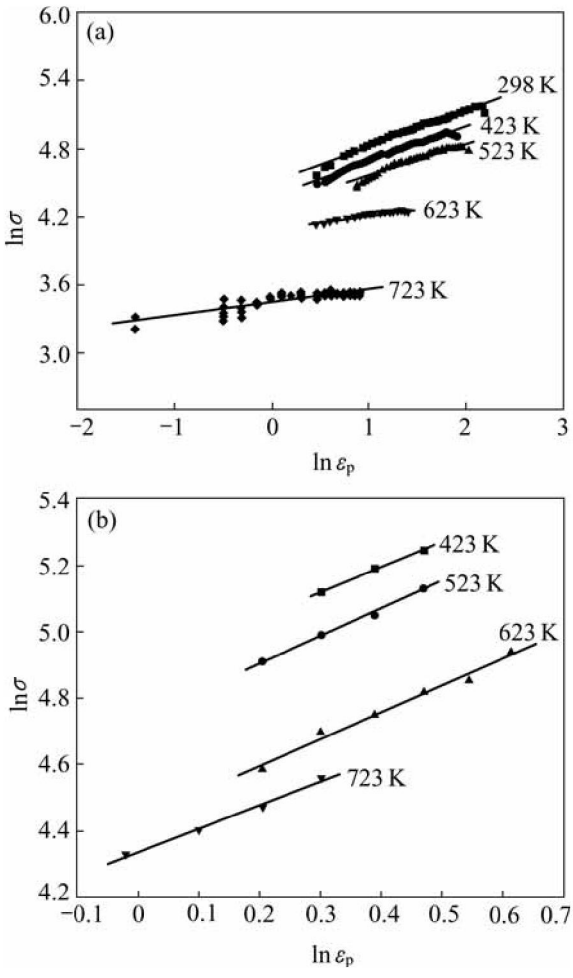

Fig.3 $\ln\sigma$–$\ln\varepsilon_p$ curves of AZ91D(a) and 42.1%TiC/AZ91D (b) composites in tensile test

indicates that Hollomon relation is applicable to the tensile deformation of AZ91D magnesium alloy and TiC/AZ91D composites at a given deformation temperature with constant tensile strain rate. The strain hardening exponents, n, for both materials were calculated and the results show that the AZ91D magnesium alloy has lower n values ranging from 0.11 to 0.32, which decreases with increasing deformation temperature. For 42.1% TiC/AZ91D composites, the n values are greater than that of the matrix magnesium alloy which ranges from 0.71 to 0.82. It is well known that the ideal elastic deformation yields value of n=1. Since the calculated values of 0.71−0.82 for TiC/AZ91D composites is approximately equal to 1, this interprets that the tensile behavior of TiC/AZ91D composites mainly adopts elastic deformation at elevated temperatures. This can also be seen from Fig.1(b) that the magnesium matrix composites possess very limited plasticity and only a small elastic deformation occurs over all the experimental temperature range.

3.3 Fracture features

The fracture features of magnesium alloy AZ91D and TiC/AZ91D composites at room and elevated temperatures can be explored by analyzing the fractured surfaces as shown in Fig.4. For AZ91D alloy, the fractured surfaces after tensile deformation are rather flat at room temperature and behave brittle characteristics (Fig.4(a)), which differs from the case at elevated temperatures displaying dimples with ductility (Fig.4(b)). Also, we can easily find that there is intermetallic $Mg_{17}Al_{12}$ phase co-existing with α-Mg in AZ91D alloy and some cracks (indicated by arrows) occur in intermetallic $Mg_{17}Al_{12}$ during tensile deformation at room temperature. These intermetallic compounds due to tensile stress concentration form the origin of crack, further extension could lead to the final failure of the AZ91D alloy. Since slip deformation can take place on non-basal crystal planes at elevated temperatures, the α-Mg has improved plasticity.

For TiC/AZ91D composites, the macroscopically flat fractured surfaces can be observed at all deformation temperatures. Although the matrix α-Mg at elevated temperatures show a little ductility, it is certain that the failure of the composites during tensile deformation originates from interface debonding between reinforcement phase TiC and matrix α-Mg, which are clearly shown in Figs.4(c) and (d).

Table 1 Strain hardening exponent(n) of AZ91D alloy and 42.1%TiC/AZ91D composites at different temperatures

Material	n				
	298 K	423 K	523 K	623 K	723 K
AZ91D	0.317 84	0.308 23	0.279 35	0.117 54	0.113 90
42.1%TiC/AZ91D	–	0.754 95	0.822 74	0.808 84	0.705 89

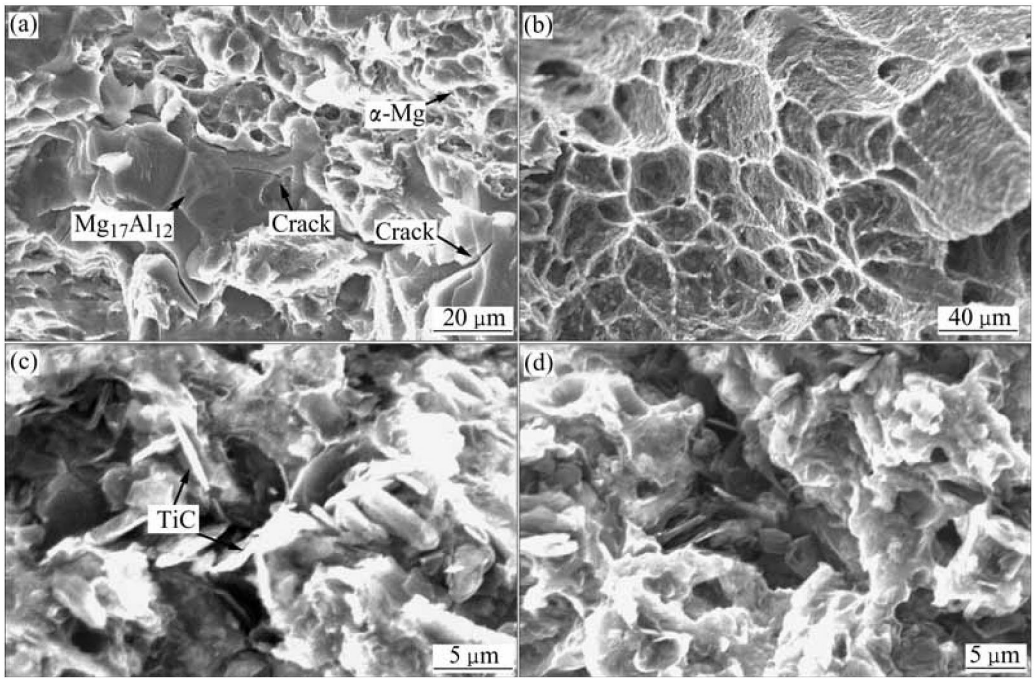

Fig.4 SEM images of fractured surfaces for AZ91D alloy at 298 K(a) and 623 K(b) and for TiC/AZ91D composites at 298 K(c) and 623 K(d)

4 Conclusions

1) The in situ formed TiC can increase the tensile strength of magnesium alloy, especially effective at elevated temperatures. At room temperature, the tensile strength of AZ91D alloy is 175 MPa and that of TiC/AZ91D composites is 203.4 MPa, only increased by 16.2%. At 723K the tensile strength of the TiC/AZ91D composites is 95.2 MPa, increased by 180%, while that of the AZ91D alloy is only 34 MPa.

2) The tensile deformation of AZ91D alloy and TiC/AZ91D composites follows Hollomon relation. Theoretical calculation of the strain hardening exponent n has been performed for both materials. The n values of TiC/AZ91D magnesium matrix composites range from 0.71 to 0.82, close to 1.0, which shows fracture with brittle characteristic. However, the n values of 0.11−0.32 obtained for matrix alloy AZ91D show typical ductile features at elevated temperatures.

3) The failure of magnesium alloy AZ91D during tensile deformation is generally associated with cracking of intermetallic compound $Mg_{17}Al_{12}$, while that of TiC/AZ91D composites originates from their interface debonding.

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