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ARTICLE

Preparation of $NiTi_p$/WE43 Magnesium Matrix Composites by Friction Stir Processing

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Abstract: An effective technique of friction stir processing (FSP) to fabricate the NiTi particle reinforced WE43 magnesium (Mg) matrix composite was proposed. The microstructure of FSP specimens was examined by scanning electron microscopy (SEM) coupled with an energy-dispersive X-ray spectroscope (EDS). X-ray diffraction (XRD) was used for phase analysis. The results show that the composite possesses the shape memory effect. The low processing temperature effectively prevents the interfacial reaction between the NiTi particles and Mg matrix during FSP. Regardless of particle size, the NiTi particles are homogeneously distributed in the Mg matrix. In addition, compared with the Mg matrix, the yield strength, ultimate tensile strength, and elongation of the $NiTi_p$/WE43 composite are reduced by 33%, 12%, and 18%, respectively. Both the tensile strength and elongation reduces with increasing the size of as-received NiTi particles. The failure mechanism of the composite is interface debonding and the fracture of reinforced particles.

Key words: friction stir processing; NiTi particles; magnesium matrix composite; microstructure; shape memory effect

Particle reinforced metal matrix composites (MMCs) are widely applied in aerospace, aircraft, marine and many other industries, due to their splendid strength, elastic modulus, toughness, fatigue resistance, thermal conductivity, electric conductivity and abrasion resistance[1]. However, particle reinforced MMCs have some defections, such as high processing temperature and costly manufacturing process.

There are many techniques to fabricate MMCs, such as casting[2], powder metallurgy[3,4], and infiltration[5]. However, these conventional techniques are commonly performed at high temperature, which accelerates the element diffusion and interfacial reaction between the reinforced particles and metal matrix. As a result, intermetallic compounds are easily generated, and interfacial bonding strength of MMCs decreases[6-8]. Therefore, in order to improve the performance of MMCs, it is a key issue to diminish, and even avoid the interfacial reaction between the reinforced particles and metal matrix during the manufacturing process.

Friction stir processing (FSP) is a new kind of solid-state processing technique based on the principle of friction stir welding, which can achieve materials refinement, densification and homogenization[9,10] simultaneously. In the recent years, FSP is mainly applied in the microstructural modification of casting alloys[11-13], and the fabrication of ultrafine-grained materials[14,15] and particle reinforced MMCs. Mishra et al[16] used FSP to fabricate the SiC particle reinforced MMC on the top surface of aluminum (Al) alloy, and demonstrated that FSP has the advantage of effectively preventing the interfacial reaction between SiC particles and Al matrix. Besides, FSP has been used to prepare the Al and magnesium (Mg) matrix surface composites[17-20]. The reinforced particles mainly include carbon nanotube, SiO_2, TiN, SiC, etc[21]. The

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results showed that both the microhardness and wear resistance of the prepared surface composites are significantly improved. In order to obtain the shape memory effect (SME) on the surface of Al alloy, Dixit et al[22] adopted FSP to prepare $NiTi_p$/1100Al surface composite, which exhibits the SME due to the martensitic phase transformation of NiTi particles. Similarly, Ni et al[23] successfully fabricated $NiTi_p$/6061Al surface composite with the SME.

Mg alloys are widely used in aerospace, transportation, 3C and medical fields[24]. Among them, WE43 Mg alloy is one of the most attractive biological materials due to its good biological compatibility[25,26]. In order to satisfy the demand of biological material for shape memory function, the preparation of WE43 matrix MMCs with SME is practically needed. Therefore, the aim of this study is to use FSP to prepare $NiTi_p$/WE43 composite with addition of NiTi particles, which possess the shape memory function, and to investigate the effect of FSP on the microstructure, mechanical properties and SME of $NiTi_p$/WE43 composite.

1 Experiment

Rolled WE43 Mg plates with the chemical composition (wt%) of Mg-4.2Y-2.7Nd-0.5Zr-0.3Ce-0.2La were used in this study. Fig.1 shows the morphologies of as-received $Ni_{55.2}Ti_{44.8}$ (at%) particles. In order to study the effect of the size of NiTi particles on the properties of $NiTi_p$/WE43 composites, two kinds of particles with different diameters of 2~50 and 100~150 μm were used, which were named as fine and coarse particles, respectively.

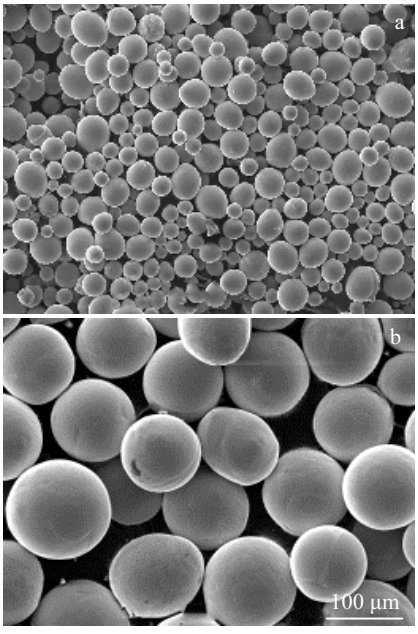

Fig.1 Morphologies of as-received fine (a) and coarse (b) NiTi particles

FSP was performed on a modified milling machine. A series of blind holes with 3 mm in diameter and 5 mm in depth were machined on the surface of as-received WE43 plates with the dimension of 135 mm×60 mm×6 mm. Then the NiTi particles were embedded within the blind holes. A stir tool without the pin was adopted to embed NiTi particles into the plate, followed by four-passes reciprocating FSP. The stir tool with a flat shoulder with 18 mm in diameter and a threaded cylindrical pin with 6 mm in diameter and 4.8 mm in length were used during the reciprocating FSP. Two kinds of stir tools were made of W18Cr4V. During FSP, a rotation rate of 600 r/min and a traverse speed of 100 mm·min^{-1} were used at the reduction per pass of 0.2 mm. The processing direction was parallel to the rolling direction of WE43 plate (Fig.2). During FSP, the temperature distribution in the processed region was measured by the non-contact infrared thermometer.

Microstructures of the FSP specimens were examined by scanning electron microscopy (SEM, JSM-6390A) coupled with an energy-dispersive X-ray spectroscope (EDS). X-ray diffraction (XRD, D8ADVANCE A25) was used for phase analysis. Specimens for SEM were cut perpendicular to the processing direction (PD) through the original position of blind holes, and then ground, polished and etched with etching reagent (10 mL acetic acid, 10 mL distilled water, 4.2 g picric acid, and 100 mL ethanol). Differential scanning calorimeter (DSC, 204F1) was used to measure the transformation temperature of $NiTi_p$/WE43 composite. The testing temperature ranged from −150 °C to 150 °C at a rate of 10 °C/min in argon atmosphere. The specimens for DSC were cylindrical and had a size of Φ5 mm×2 mm.

The specimens for tensile test with the gauge section of 4 mm×4 mm×1.6 mm were machined parallel to the PD direction. Tensile tests were conducted at an initial strain rate of 1×10^{-3} s^{-1} at room temperature using a computerized

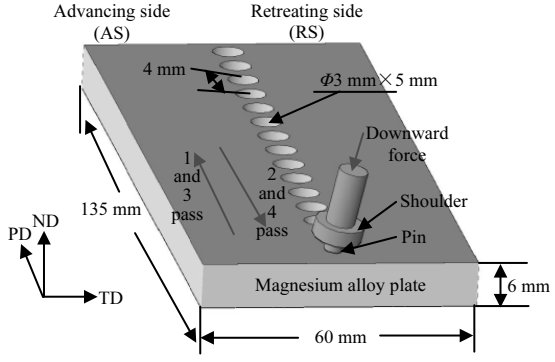

Fig.2 Schematic of FSP $NiTi_p$/WE43 composite (PD, TD and ND represent processing, transverse and normal direction, respectively; AS of the plate is where the rotation speed of stir tool has the same direction as its travel speed; RS means that the two speed components have opposite directions)

Instron-8801 testing system. Three specimens were tested at each condition to guarantee accuracy of tensile test data. Before tensile test, specimens were ground and polished to achieve a smooth surface. After tensile test, the fracture surface of the tensile specimens was observed by SEM.

2 Results and Discussion

2.1 Microstructure

Fig.3a and 3b show the SEM images of the $NiTi_p$/WE43 composites, and three results can be obtained. Firstly, NiTi particles are homogeneously distributed in the Mg matrix, and do not exhibit agglomeration phenomenon regardless of the diameter of as-received particles. Secondly, the distribution of fine NiTi particles in the Mg matrix is more homogeneous than that of coarse particles (Fig.3a). Thirdly, compared with the as-received particles (Fig.1), the NiTi particles distributed in the matrix are slightly damaged, indicating that the NiTi particles suffer from the shearing and squeezing under the combined effect of shoulder and threaded cylindrical pin during the FSP (Fig.3).

Fig.3c shows the interfacial element distribution between NiTi particles and Mg matrix. It can be clearly seen that there is no discernible interfacial reaction between the NiTi particles and Mg matrix. This is mainly attributed to the following reasons. On the one hand, the peak temperature of the FSP processed region is about 360 °C, which is much lower than the processing temperature of casting[2] as well as powder metallurgy[3,4]. On the other hand, FSP does not provide enough time for interfacial reaction, because the time of duration is only 20 s during the single pass FSP.

The XRD results also demonstrate that there is no new phase generated in the $NiTi_p$/WE43 composite (Fig.4). This suggests that FSP is a good method to fabricate $NiTi_p$/WE43 composite in terms of avoiding the interfacial reaction between NiTi particles and Mg matrix.

2.2 Shape memory effect

Fig.5 shows the DSC curves of as-received NiTi particles and $NiTi_p$/WE43 composite. It is explicit that the as-received NiTi particles undergo a reversible phase transformation between the austenite and thermoelastic martensite. The corresponding transformation temperatures of A_s, A_f, M_s, and M_f are 19.2, 40.7, 8.0, and 61.7 °C, respectively (Fig.5a). This reversible thermo-elastic martensitic transformation indicates that as-received NiTi particles possess the shape memory function[22].

The $NiTi_p$/WE43 composite fabricated by FSP exhibits the similar characteristics of phase transformation, indicating that the composite inherits the shape memory function of as-received NiTi particles. Generally, the shape memory function of NiTi particles can be damaged when the reaction of L+NiTi →$NiTi_2$ occurs at the high temperature of 984 °C. However, in this study, the low processing temperature induced by FSP does not deteriorate the SME of the as-received NiTi particles. It should be noted that compared with the as-received NiTi particles, the SME of $NiTi_p$/WE43 composite is relatively weaker. This is mainly because the volume fraction of the additive NiTi particles is low. Therefore, a further research to improve the SME of the $NiTi_p$/WE43 composite by increasing the volume fraction of particles during the FSP is required.

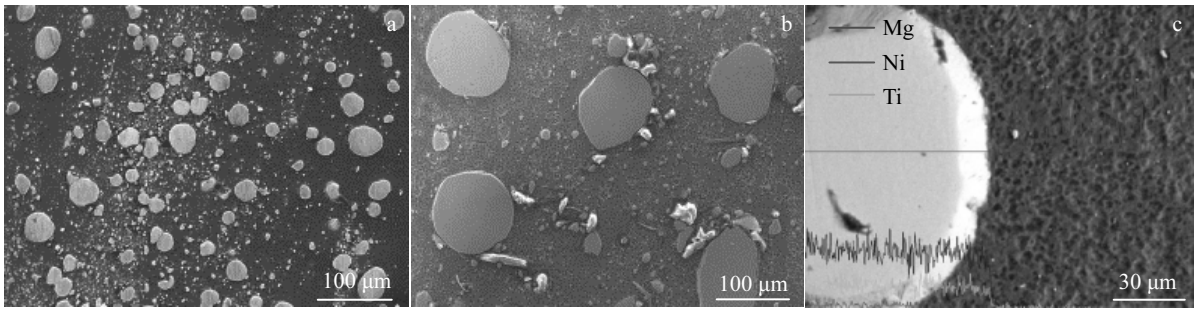

Fig.3 SEM images of the distribution of fine (a) and coarse (b) NiTi particles in $NiTi_p$/WE43 composites; EDS results of the distribution of interfacial element between coarse NiTi particles and Mg matrix (c)

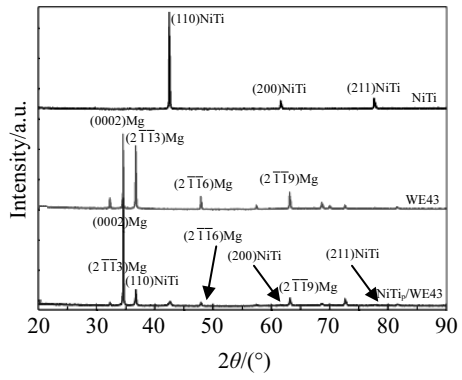

Fig.4 XRD patterns of as-received NiTi particles, WE43 Mg matrix and $NiTi_p$/WE43 composite

2.3 Tensile properties and fractography

Table 1 summarizes the tensile properties of both WE43 Mg matrix and $NiTi_p$/WE43 composite. The yield strength (YS),

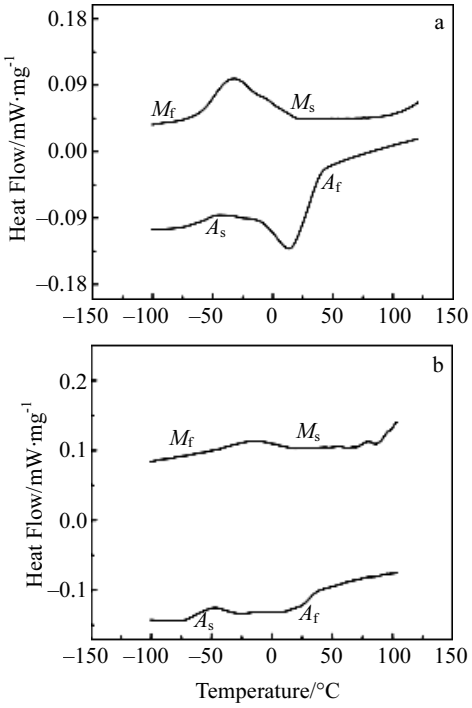

Fig.5 DSC curves of as-received NiTi particles (a) and $NiTi_p$/WE43 composite (b) (A_s and A_f represent austenite start and finish points, respectively; M_s and M_f represent martensite start and finish points, respectively)

ultimate tensile strength (UTS) and elongation (EI) of the $NiTi_p$/WE43 composite decrease, compared with those of the Mg matrix. Even with the addition of fine as-received NiTi particles, the YS, UTS, and EI of the $NiTi_p$/WE43 composite decrease by 33%, 12%, and 18%, respectively. This is mainly because the size of the fine NiTi particles is nonuniform, and the coarse particles with a diameter of 50 μm result in the stress concentration, which decreases the interfacial bonding strength between the particles and Mg matrix. Besides, the previous studies have demonstrated that FSP causes obvious texture softening of the Mg matrix, which ultimately results in the decrease of tensile strength[27]. Accordingly, the strength loss of Mg matrix is another reason responsible for the strength reduction of the $NiTi_p$/WE43 composite.

It is well documented that the size of as-received NiTi particles has a significant enhancement effect on both tensile strength and ductility of the $NiTi_p$/WE43 composite. Compared with the composite with coarse particles, the composite with fine NiTi particles shows much higher YS, UTS and elongation. Therefore, in order to further improve the mechanical properties of the composite, the size and uniformity of as-received particles should be optimized.

Fig.6 shows the fracture surfaces of two kinds of $NiTi_p$/WE43 composites. The fracture surface of WE43 matrix is characterized by the cleavage plane and some dimples. In addition, the pull-out of NiTi particles from Mg matrix is obviously observed. Because stress concentration easily occurs at the interface between NiTi particles and Mg matrix, microcracks are prone to nucleate and then propagate along the interface, as indicated by the arrows in Fig.6a and 6b. Besides, the NiTi particles preferentially fracture under the condition of stress concentration (Fig.6a). Therefore, it can be concluded that the failure mechanism of $NiTi_p$/WE43 composite is interface debonding and the fracture of reinforced particles.

Table 1 Tensile properties of Mg matrix and $NiTi_p$/WE43 composites

Specimen	YS/MPa	UTS/MPa	EI/%
WE43 alloy	119	318	18.5
$NiTi_p$/WE43 composite with fine NiTi particles	79	281	15.2
$NiTi_p$/WE43 composite with coarse NiTi particles	68	165	11.1

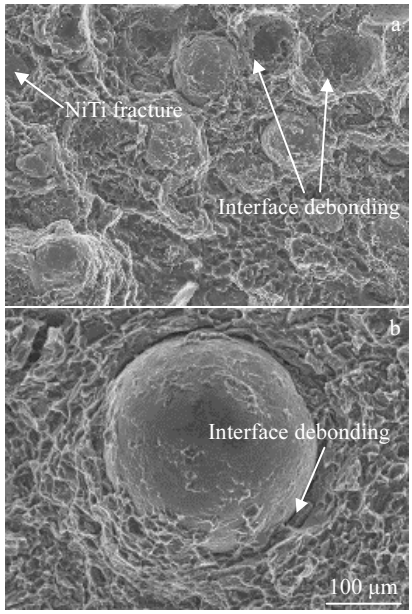

Fig.6 Fracture surfaces of $NiTi_p$/WE43 composite with fine NiTi particles (a) and coarse NiTi particles (b)

3 Conclusions

1) The $NiTi_p$/WE43 composite can be successfully prepared by friction stir processing. The NiTi particles are homogeneously distributed in the Mg matrix, and there is no discernible interfacial reaction between the NiTi particles and Mg matrix. $NiTi_p$/WE43 composites fabricated by FSP possess the shape memory effect (SME).

2) Compared with the Mg matrix, the YS, UTS, and EI of the $NiTi_p$/WE43 composite with fine NiTi particles decreases by 33%, 12%, and 18%, respectively.

3) The failure mechanism of the $NiTi_p$/WE43 composite is interface debonding and the fracture of reinforced particles.

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搅拌摩擦加工制备 $NiTi_p$/WE43 镁基复合材料

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摘　要：提供了一种采用搅拌摩擦加工（FSP）制备 NiTi 颗粒增强 WE43 镁基复合材料的有效方法。采用 SEM 结合 EDS 对 FSP 试样的微观结构进行了研究，并用 XRD 进行了物相分析。结果表明，制备的复合材料具有形状记忆效应。较低的加工温度有效地阻止了 NiTi 颗粒与 Mg 基体在 FSP 过程中的界面反应。无论粒径大小，在 FSP 后，NiTi 颗粒都均匀分布在 Mg 基体中。此外，与 Mg 基体相比，$NiTi_p$/WE43 复合材料的屈服强度、极限拉伸强度和延伸率分别降低了 33%、12%和 18%。随着加入的 NiTi 颗粒尺寸增大，该复合材料拉伸强度和延伸率均降低。复合材料的失效机理是颗粒之间的界面开裂以及增强颗粒的断裂。

关键词：搅拌摩擦加工；NiTi 颗粒；镁基复合材料；微观组织；形状记忆效应

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