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In situ WAl_{12} particle-reinforced Al matrix composites synthesized by combining mechanical alloying and vacuum hot pressing technology

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ABSTRACT

In this work, mechanical alloying (MA) and hot-pressed (HP) sintering have been employed to fabricate a series of Al-based metal matrix composites (MMCs) reinforced with WAl_{12} particles. In particular, mechanical alloying method was used to fabricate $W_{14}Al_{86}$ new alloy. WAl_{12} compound is in situ generated through Al and $W_{14}Al_{86}$ powder blend in order to enhance the mechanical properties. By X-ray diffraction (XRD) and scanning electron microscopy (SEM), it was found that WAl_{12} intermetallic particles were in situ formed in the MMC during hot-pressing Al–$W_{14}Al_{86}$ system. Density measurements using Archimedes' method and microhardness test were carried out for all the samples. By increasing the weight percentage of WAl_{12}, both density and microhardness increased significantly. The tensile strength of samples was up to 487 MPa at room temperature, and up to 248 MPa at 200 °C.

1. Introduction

In recent years, Al-based metal matrix composites (MMCs) have become the subject of numerous research works owing to the need for new structural engineering materials in the areas of aviation, military and transportation industries [1–5]. Among these MMCs, many researchers mainly focus on ceramic phase-reinforced Al-based MMCs since these materials possess better wear resistance, higher specific stiffness and specific strength, etc. compared with the traditional monolithic materials [6–9]. However, the key problem in the fabrication of the MMCs is the poor wettability between the ceramic particles and the Al matrix which will cause weak interface between them. Compared with the ceramics reinforcements, intermetallics can be wetted by corresponding metal matrix easily. In addition, there are some other exceptional merits on intermetallic reinforcements, such as comparable mechanical properties and closer coefficient of thermal expansion with Al matrix [10]. Unfortunately, little attention has been paid on new intermetallic reinforced MMCs systems recently [11–15].

In situ WAl_{12}, as a new intermetallic reinforcement, is a promising candidate owing to its clean grain boundary and favorable compatibility with Al matrix. However, to the best of our knowledge, synthesis of composites with in situ WAl_{12} reinforcement has never been reported except by our group.

Different processing techniques including semi-solid casting, powder metallurgy, hot-pressing (HP) and spray deposition and so on have been applied to fabricate metal matrix composites (MMCs) [16–20]. Among these methods, mechanical alloying (MA) [21–24], a solid-state powder processing technique, was effectively employed to prepare a variety of alloy powders or blend green samples. As a reinforced particle preprocessing, hot-pressing sintering technique has become a highly evolved method for preparing full dense, good matrix-to-reinforcement compatible and microstructural homogeneous materials [25,26].

Herein, we report the preparation of in situ WAl_{12} reinforced Al-based MMC via MA and HP technology using $W_{14}Al_{86}$ alloy, which is of high microhardness, good oxidation resistance and low density, etc. The mechanical properties and the microstructures of the MMC were also investigated as a function of reinforcement content and sintering temperature.

2. Experimental methods

The Al (the mean size approximate to 3.59 μm) with purity of 99.5% and tungsten (the mean size approximate to 0.98 μm) with purity of 99.8% powders were used as received without further purification. The $W_{14}Al_{86}$ powders were prepared in advance using MA technique [23]. Mechanical alloying was performed on a high-energy ball mill with a rotational speed of 580 rpm. The ball-to-powder weight ratio was 15:1. Various contents of Al–$W_{14}Al_{86}$ powders were then blended with 0.2 vol% pure alcohol, as a process control agent, in a stainless steel vial filled with argon atmosphere for 1 h respectively. All the handling was performed under argon atmosphere in a glove box for avoiding oxidation of powders during MA treatment. The blended powders were then cold-pressed in a steel die under a pressure of 250 MPa to form green samples with a dimension of approximately 40 mm × 20 mm × 10 mm.

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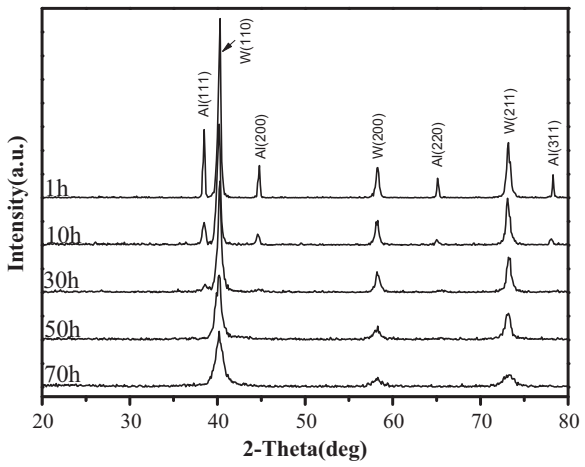

Fig. 1. XRD patterns of the MA-ed $W_{14}Al_{86}$ powders after various milling time.

A TLZK-2001S vacuum hot-press machine was used to consolidate the green samples that have been enclosed in assembling graphite dice under argon atmosphere. The pressure in the die was kept at 25 MPa and the vacuum was 80 Pa in the furnace. Sintering temperature varied from 450 °C to 690 °C and meanwhile, sintering duration changed from 5 min to30 min accordingly.

The structure of in situ phases formed in HPed specimens was investigated by X-ray diffraction (XRD) analyses. The microstructures of fracture surfaces of the composites were observed by scanning electron microscopy (SEM; JSM-5310). XRD analyses were performed on a Rigaku D/max-II B X-ray diffractometer with Cu Kα (λ = 1.5406 Å) radiation, operating at 40 kV and 20 mA. The scanning speed was 0.02° s^{-1}. The Archimedes technique was used to measure the density of each composite sample. Vickers microhardness measurements were carried out on the sintered samples using the microhardness tester FM-700 (FUTURE TECH) with a load of 100 g and dwell time of 15 s. Tensile tests at 25 °C and 200 °C were performed on an Instron model 1125 test machine under 2 mm min^{-1} monotonic loading; the uniform gauge length of tensile test was 10 mm. Tensile specimens were cut from the hot-pressed composites.

3. Results and discussion

3.1. XRD analyses

The typical XRD patterns of mechanical alloyed $W_{14}Al_{86}$ powders as a function of milling time are shown in Fig. 1. In contrast to intensity of W, Al peaks decreased continuously as milling time increased. After 70 h milling, Al peaks disappeared, but W peaks were still visible. It implied that Al was embedded in the tungsten matrix and Al atoms displaced the positions of tungsten during the milling process, indicating that $W_{14}Al_{86}$ alloy was prepared by MA process.

The typical XRD patterns of the HPed Al–5 wt% $W_{14}Al_{86}$ samples at various sintering conditions are presented in Fig. 2. After hot-pressing at 600 °C for 30 min (Fig. 2(a)), the diffraction peaks of the alloy still existed and no WAl_{12} intermetallic phase appeared. However, after sintering at 650 °C for 5 min (Fig. 2(b)), the diffraction peaks of WAl_{12} began to appear. And then the $W_{14}Al_{86}$ peaks completely disappeared with further enhancement of WAl_{12} peaks while the sintering time lasted for 30 min at 650 °C (Fig. 2(c)). Therefore, it can be preliminarily demonstrated that the HPed Al–$W_{14}Al_{86}$ specimens were in situ Al-based MMCs reinforced by intermetallic WAl_{12}.

To validate if intermetallic WAl_{12} was formed directly from the mixture of the two metals, W and Al, in the sintering duration of 30 min, the MA-ed mixture of Al–W was sintered and evaluated at 650 °C. The structure of the HPed Al–3 wt% W sample can be seen from the X-ray diffraction spectra in Fig. 3. The W and Al diffraction peaks are clearly discerned from this figure, but those of WAl_{12} are weak, which indicate that a small number of WAl_{12} reinforcements form in the short sintering duration of 30 min, because the reaction

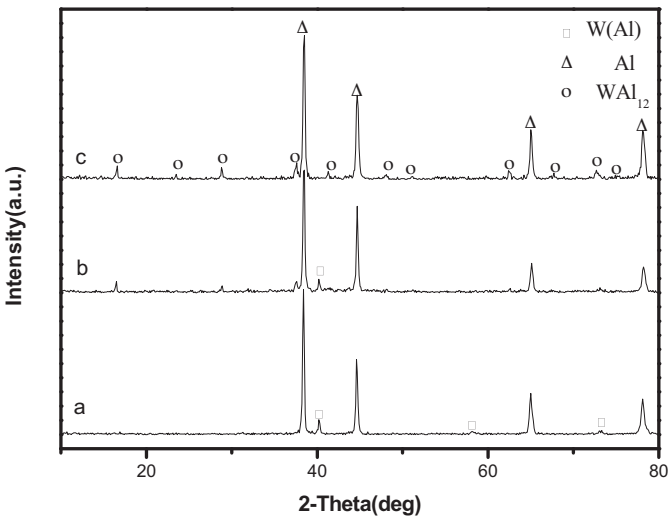

Fig. 2. XRD patterns of Al–5 wt% $W_{14}Al_{86}$ composite obtained at various conditions. (a) 600 °C 30 min, (b) 650 °C 5 min, (c) 650 °C 30 min.

rate of $W_{14}Al_{86}$ alloy with Al is faster than that of W. However, it was necessary to form WAl_{12} reinforcements in short sintering duration in order that the grain size of the reinforcement could be controlled.

3.2. Densities and microhardnesses

Fig. 4 presents the microhardness and density of WAl_{12} reinforced Al-based MMC as a function of weight percentage of WAl_{12}. When the content of WAl_{12} phase changed from 4 wt% to 29 wt%, the microhardness of the MMC raised from 1.25 GPa to 1.80 GPa, while the density only increased from 2.74 g cm^{-3} to 2.96 g cm^{-3}. Cambronero et al. [27] reported the densities and microhardnesses of ceramics, such as Si_3N_4, TiB_2, B_4C, reinforced AA7015 composites, which indicate the microhardnesses of the composites are in the range of 1.0–1.3 GPa, when these densities changed from 2.81 g cm^{-3} to 2.86 g·cm^{-3}, meanwhile the microhardnesses of WAl_{12} reinforced MMCs varied from 1.41 GPa to 1.58 GPa in the approximate density range. Therefore, in the similar density range, compared to ceramic reinforcing phase such as Si_3N_4, TiB_2, B_4C,

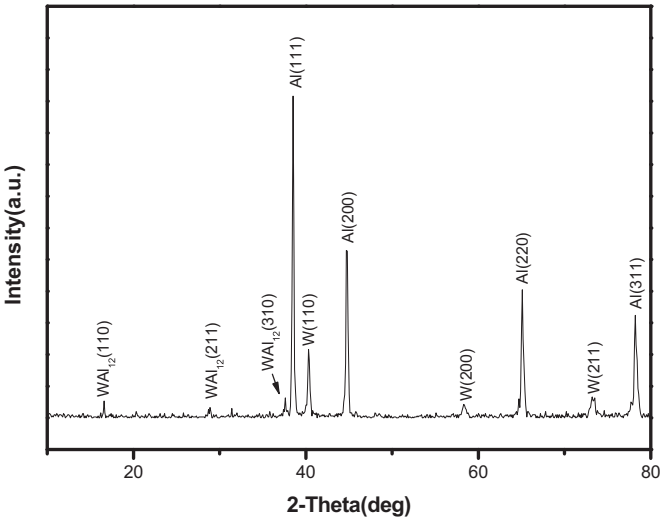

Fig. 3. XRD pattern of Al–3 wt% W composite obtained by sintering at 650 °C for 30 min and a pressure of 25 MPa under vacuum.

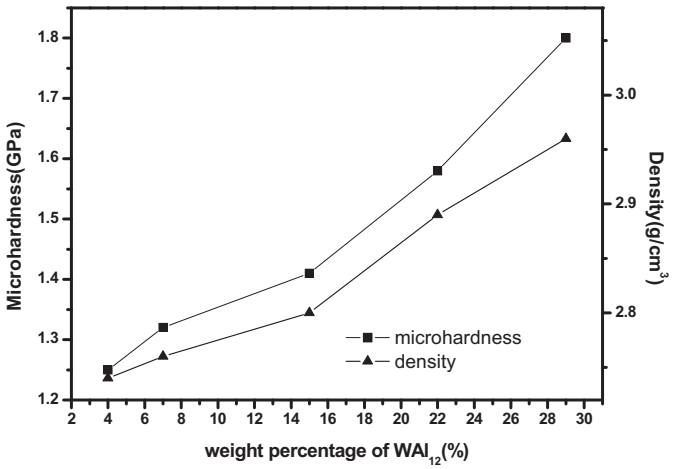

Fig. 4. Microhardness and density of WAl_{12} reinforced Al-based MMC as a function of weight percentage of WAl_{12}.

etc., WAl_{12} intermetallics reinforcing phase can bring much higher microhardness to the materials.

3.3. Mechanical properties

Fig. 5 gives the measurements of the ultimate tensile strength (UTS) and Young's modulus of HP-ed WAl_{12} reinforced Al-based samples as a function of weight percentage of WAl_{12} at room temperature (Fig. 5(a)) and 200 °C (Fig. 5(b)). Two types of UTS and Young's modulus showed a similar changing tendency for both room temperature and 200 °C. As the weight percentage of WAl_{12} increased from 4% to 29%, the UTS at room temperature increased from 410 MPa to 487 MPa, and then dropped to 462 MPa. At the same time, the UTS at 200 °C also reached the maximum strength of 248 MPa with 22 wt% WAl_{12} phase in the sample. The comparable strength value of the specimens can be explained by the formation of reinforcements in the MMCs and the good bonding between Al matrix and reinforcement particle phases due to clean interfaces without contamination among them. However, compared with some ceramics reinforced MMCs [28–30] (Table 1), the strength

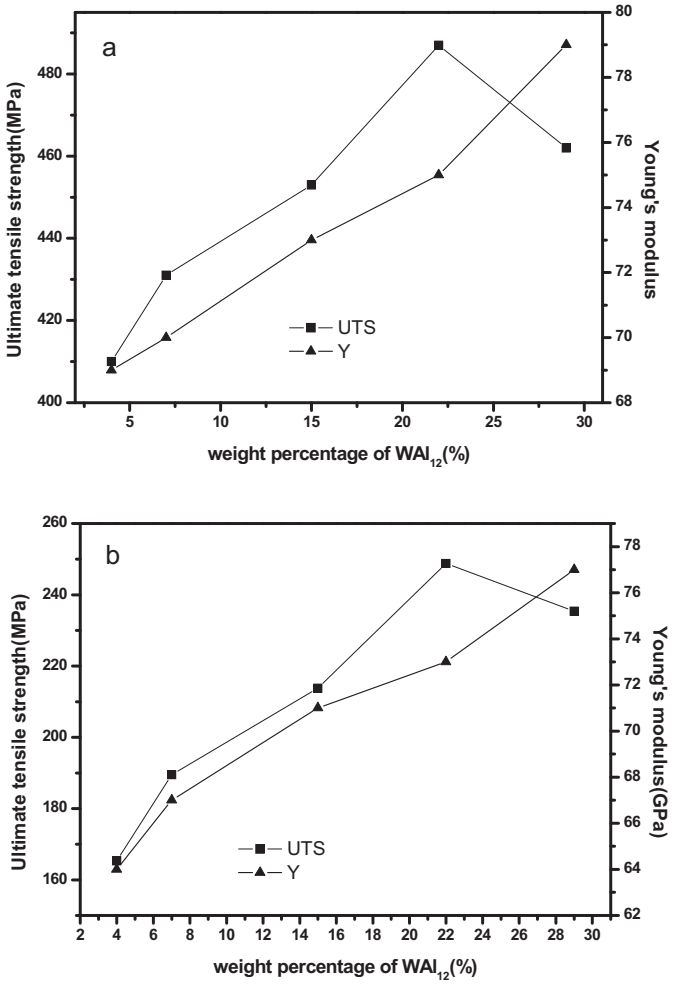

Fig. 5. Mechanical properties of WAl_{12} reinforced Al-based MMC as a function of weight percentage of WAl_{12} at (a) room temperature and (b) 200 °C.

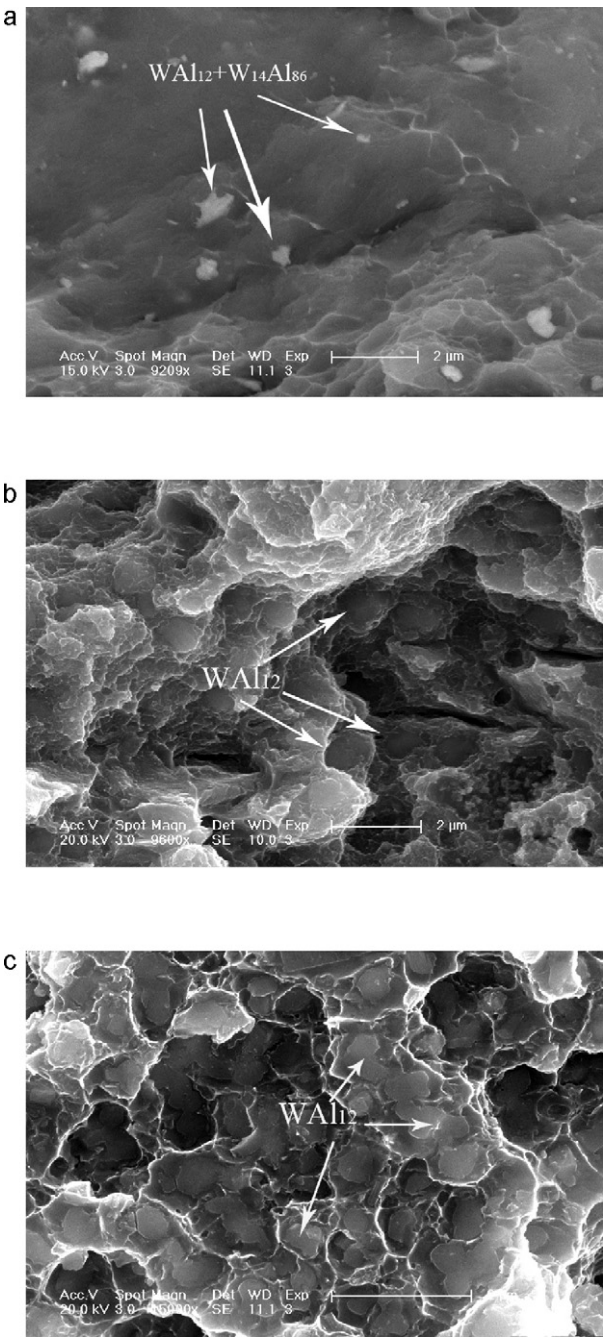

Fig. 6. SEM images of the tensile fracture surface of hot-pressed samples: (a) Al–5 wt% $W_{14}Al_{86}$ system at 650 °C for 5 min; (b) Al–5 wt% $W_{14}Al_{86}$ system at 650 °C for 30 min; (c) Al–15 wt% $W_{14}Al_{86}$ system at 650 °C for 30 min.

Table 1
Ultimate tensile strengths of various MMCs at room temperature and 200 °C compared with those of Al–22 wt% WAl_{12} composite.

Temperature (°C)	Materials	UTS (MPa)
25	5.0 vol% TiB_2/Al–12% Si–1.2% Cu–1% Ni–1% Mg(ZL109) as-cast	289
	15 vol% $K_2O{\cdot}8Ti_{2w}$/ZL109	297
	20 vol% Al_2O_3–SiO_2/ZL109	332
	22 wt% WAl_{12}/Al (16.5 vol% WAl_{12}/Al)	487
200	5.0 vol% TiB_2/ZL109	259
	15 vol% $K_2O{\cdot}8Ti_{2w}$/ZL109	278
	20 vol% Al_2O_3–SiO_2/ZL109	275
	16.5 vol% WAl_{12}/Al	248

loss of WAl_{12} reinforced Al-based MMC was maximal, although its high temperature strength value at 200 °C has nearly the same value with those ceramics candidates. Thus, it seems that WAl_{12} reinforced MMC is not suitable to be used as high temperature structural material.

3.4. Microstructure analyses

Al–7 wt% WAl_{12} composite was fabricated in situ from the mixture of Al–5 wt% $W_{14}Al_{86}$ via hot pressing. Fig. 6(a) and (b) shows the SEM images of the samples of Al–5 wt% $W_{14}Al_{86}$ which were sintered at 650 °C for 5 min and 30 min, respectively. It can be seen from the figures that the reinforced particle in matrix grew from less than 500 nm to approximately 1 μm when the sintering duration ranged from 5 min to 30 min. Fig. 6(c) shows a typical representation of the fracture surface of in situ Al–22 wt% WAl_{12} composite formed via hot pressing at 650 °C for 30 min. As shown in Fig. 6(b), in situ formed WAl_{12} reinforcements were dispersed in the Al matrix. Its fracture surface is of the cleavage type. In Fig. 6(c), the intermetallic phase was dispersed continuously in the matrix. Combined with the mechanical values, it can be concluded that the WAl_{12} intermetallic phase is an effective reinforcement to the matrix.

4. Conclusions

In situ WAl_{12} intermetallic particle reinforced Al-based MMC was fabricated from Al–$W_{14}Al_{86}$ system via mechanical alloying and vacuum hot pressing (HP). The formation of WAl_{12} particulates was confirmed by XRD studies. WAl_{12} particulate, a new intermetallic reinforcement, has a reasonable clean interface with aluminum matrix using the in situ preparation method. The results of tensile test demonstrate that the incorporation of WAl_{12} to the Al matrix is beneficial to enhance the mechanical performance of in situ composite. The optimum content of WAl_{12} phase in the MMC is about 22 wt%. The maximum UTS can reach 487 MPa. But it is not suitable for high temperature structural applications because the loss of the tensile strength under high temperature is more severe than other ceramics reinforced MMCs.

Acknowledgments

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