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Review

Effects of in situ generated ZrB_2 nano-particles on microstructure and tensile properties of 2024Al matrix composites

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ABSTRACT

The main purpose of this paper is to investigate the effects of nano-size ZrB_2 particles on the microstructure and tensile properties of 2024Al matrix composites. The microstructure and tensile properties of the 2024Al matrix composites reinforced by ZrB_2 nano-particles fabricated from 2024Al–K_2ZrF_6–KBF_4 system by direct melt reaction are investigated by XRD, OM, SEM, EDS, TEM and tensile testing. The results reveal that the ZrB_2 nano-particles exhibit rectangular and nearly hexagon shape with the size ranging from 30 to 100 nm, and the reasonable uniform distribution of ZrB_2 nano-particles gives rise to significant grain refinement of 2024Al alloy. Tensile testing results show that the tensile strength and yield stress of the composites increase continuously. However, the elongation of the composites increases almost linearly and declines later as the increasing of ZrB_2 content. The fracture morphologies are in accord with corresponding ductility results. What's more, the strengthening mechanism of ZrB_2 nano-particles is established.

Contents

1. Introduction

Aluminum matrix composites possess many advantages such as low density, high specific stiffness, high specific strength, good thermal stability, electromagnetic shielding capacities and good wear resistance with the development of some non-continuous reinforcement materials, whisker, fibers or particles. In particular, particles reinforced aluminum matrix composites not only have good mechanical and wear properties, but also are economically viable [1–5]. There are many methods for fabrication of particles reinforced aluminum matrix composites such as powder metallurgy [6], squeeze casting [7] and compocasting [8]. Among all the techniques to fabricate particles reinforced aluminum matrix composites, the melt in situ reaction fabrication is one of the most economical and versatile [9–11]. Compared with traditional ex situ composites, the in situ particles reinforced aluminum matrix composites possess more advantages in microstructure such as clean interface, strong interfacial bonding, fine particles and uniform distribution in matrix. However, in situ reaction systems are mainly concentrated on Al–Ti–X, Al–Ti–O, Al–Ti–B and Al–Ti–C. In situ formed reinforcements are only focused on a few particles such as SiC, Al_3Ti, Al_2O_3, TiB_2 and TiC [12–16]. At present the most popular and widely used reinforced particle whose size ranges from 3 to 30 μm leads to a considerable reduction in ductility toughness and ineffective utilization of the strength and stiffness of the reinforcement [17]. It is the result of the easy initiation and propagation of cracks in the ceramic particles or at the interface. In order

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to improve mechanical properties of composites, a proper particle must be used for reinforcement. Zhanget al. [18] fabricated AZ91-based TiB_2 submicron particles reinforced composites and observed that the size of the α-Mg phases and β-$Mg_{17}Al_{12}$ phases are considerably refined compared with the specimen fabricated without the addition of TiB_2 submicron particles. They also reported that the mechanical properties of the as-prepared AZ91 alloy are better than those without TiB_2 submicron particles. Schultz et al. have successfully fabricated Al–Mg based Al_2O_3 nano-particles reinforced composites [19]. However the single ZrB_2 nano-particles reinforced aluminum matrix composite has not been fabricated and the reinforcing mechanism of nano-particles remains further discussion.

In this work, the 2024Al matrix composites reinforced by the ZrB_2 nano-particles were fabricated from 2024Al–K_2ZrF_6–KBF_4 system by melt in situ reaction, the morphologies, sizes and distribution of the in situ particles as well as the microstructure of the composites were investigated by XRD, OM, SEM, EDS and TEM. The tensile properties of the composites were investigated by tensile testing. The effects of the nano-sized ZrB_2 particles on the microstructure and mechanical properties of 2024Al matrix composites were investigated, and the effect mechanisms were discussed systematically.

2. Experimental

The raw materials are commercial 2024Al alloy ingots (shown in Table 1), inorganic salt K_2ZrF_6 and KBF_4 powder. Firstly, the experimental inorganic salt K_2ZrF_6 and KBF_4 powder were pre-heated to dehydrate the bounded water in it at 573 K for 3 h in an electric furnace. Then the dried K_2ZrF_6 and KBF_4 powders were cooled, ground and screened. At the same time, the aluminum ingots were heated in a graphite crucible which was set in a resistance furnace. The dried K_2ZrF_6 and KBF_4 powder were mixed with mass ratio 52:48 to ensure a 20% excess of element B. Then, the mixed salt powder was wrapped by aluminum foil with 30–40 g each package. In order to gain the composites containing 5%, 10%, and 15% (volume fraction) ZrB_2 particles, the mixed salt powder adjusted according to the content of Zr in composites was added with campanulate graphite plunge when the melt was overheated up to 1143 K. Then the in situ reaction between the aluminum alloy melt and added salt causes exquisite vibration instantly. An electromagnetic stirrer (DJMR-1616W) was utilized to increase the mass transfer during the whole in situ reaction. During the melt reaction process, the melt temperature was tested with thermal couple continuously. Optical emission spectrometer (PDA-8000) was used to determine the composition of the composites material. According to the results, some appropriate elements need to be added to adjust the alloy composition to meet the requirements of Table 1. After 30 min, the melt was degassed and refined with C2Cl6, then was poured into a copper module at 983–993 K and cooled to the room temperature. Table 2 lists the spectral analysis of the various alloy samples, which were prepared complying with national standard (HB/Z 208-1991). Based on the fact that in situ reaction could not proceed sufficiently, the yields of Zr and B elements are about 62% and 50% respectively. The actual volume fractions (3.1%, 5.4% and 8.1%) of the composites are less than the calculated theoretical value (5%, 10% and 15%). The specimens were cut into two parts; the small one was used for XRD, SEM and TEM analysis and the large one for tensile properties testing.

X-ray diffraction (DMAX2500PC) using Cu Kα radiation was used to determine phase component of the specimens. OM (LEICA-DM-2500M) and SEM (JSM-7001F) were used to analyze the microstructure of the as-prepared specimens. TEM (JSM2010) was used to observe the morphologies and the interfaces. Tensile properties test of the specimens were carried out at room temperature by a computer-controlled electronic tensile testing machine (DWD-200) at a strain rate $1.67 \times 10^{-4}\ s^{-1}$ according to the ASTM E8 standard. The size of the specimens for mechanical properties testing is shown in Fig. 1.

Table 1
Chemical composition of 2024Al alloy (mass fraction, %).

Si	Fe	Cu	Mn	Mg	Cr	Zn	Al
0.50	0.50	3.8–4.9	0.30–1.0	1.2–1.8	0.10	0.25	Bal.

3. Results and discussion

3.1. Phases of the 2024Al matrix composites reinforced by the ZrB_2 nano-particles

Fig. 2 shows the XRD pattern of the 2024Al matrix composites reinforced by the ZrB_2 nano-particles fabricated from Al–K_2ZrF_6–KBF_4 system. The diffraction peaks of Al, $CuAl_2$ and ZrB_2 phases are observed, respectively. There are no peaks of Al_3Zr phase observed because of the excess of the element B. According to the XRD pattern above and Ref. [20], the possible chemical reactions in the aluminum melt are as follows:

$$2KBF_4 + 3Al = AlB_2 + 2KAlF_4 \quad (1)$$

$$3K_2ZrF_6 + 13Al = 3Al_3Zr + K_3AlF_6 + 3KAlF_4 \quad (2)$$

$$AlB_2 + Al_3Zr = ZrB_2 + 4Al \quad (3)$$

The total reactions can be expressed as:

$$3K_2ZrF_6 + 6KBF_4 + 10Al = 3ZrB_2 + 9KAlF_4 + K_3AlF_6 \quad (4)$$

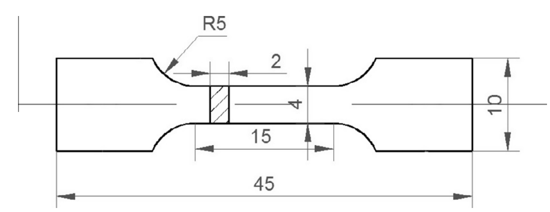

Fig. 1. The size of the specimens for mechanical properties testing (mm).

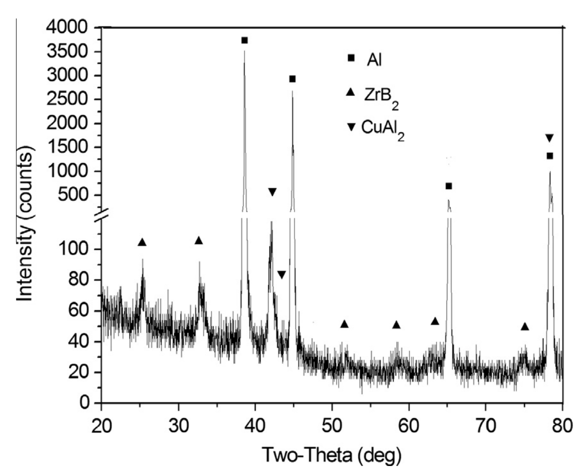

Fig. 2. XRD pattern of the 2024Al matrix composites reinforced by the ZrB_2 nano-particles.

Table 2
Chemical composition of the 2024Al alloy and 2024Al matrix composites reinforced by the ZrB_2 nano-particles (mass fraction, %).

Element	Si	Fe	Cu	Mn	Mg	Cr	Zn	Zr	B	Al
2024Al	0.38	0.42	4.43	0.81	1.43	0.09	0.22	–	–	Bal.
2024Al–5% ZrB_2	0.36	0.43	4.27	0.73	1.42	0.08	0.21	5.38	1.32	Bal.
2024Al–10% ZrB_2	0.41	0.46	4.31	0.76	1.34	0.07	0.23	9.68	2.28	Bal.
2024Al–15% ZrB_2	0.38	0.45	4.34	0.68	1.25	0.08	0.22	14.12	3.46	Bal.