

Influence of TiB₂ particles on aging behavior of in-situ TiB₂/Al-4.5Cu composites

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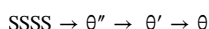
ABSTRACT

In this paper, the aging process of in-situ TiB₂/Al-4.5Cu composites has been investigated carefully, especially the precipitation behavior in the presence of TiB₂ particles. The mechanism of this particle-assisted precipitation in the composites are also studied systematically. Comparisons of the aging behavior between composites and matrix alloy have been made to investigate the effect of TiB₂ particles. Moreover, attention has also been paid to tensile properties and hardness of TiB₂/Al-4.5Cu composites and matrix alloy during aging procedure. Results indicate that TiB₂ particles accelerate the aging processes and also induce inhomogeneous distribution of Al₂Cu precipitates due to the high-density dislocations adjacent to particles. Besides, a rare phenomenon was found that the evolution on mechanical properties of the composites can be divided into two stages and this evolution is in good accordance with the evolution of the Al₂Cu precipitates. Moreover, the time to peak-aged condition of the composites in mechanical properties comes down from 20 h to 8 h.

1. Introduction

Particle reinforced aluminum matrix composites (PRAMCs) are considered as promising materials for various applications such as aerospace, military, transportation and so on, owing to the outstanding mechanical properties they possess [1–7]. Among numerous aluminum alloys, Al-Cu alloys are widely used in the aerospace industry because of their high relatively strength and high hardness, but low yield strength of Al-Cu alloys has greatly inhibited their applications. TiB₂ particle is an excellent reinforcement in aluminum alloys among potential reinforcement particles like Al₂O₃, SiC, B₄C, TiC [8–10], since TiB₂ particles have high hardness, good thermodynamic stability, high corrosion resistance. More importantly, there is no interface reaction between TiB₂ particles and Al alloy melt. In the meantime, in-situ techniques to fabricate TiB₂ particle reinforced metal matrix composites (MMCs) have been greatly improved since the last decade of the 20th century [11,12]. Hence, in-situ TiB₂/Al-Cu MMCs can be easily synthesized without such difficulties as poor wettability, formation of unwanted reaction products, etc., which are common to be seen in the development of ex-situ particulate MMCs [13,14].

Al-Cu alloys are generally strengthened by heat treatment with the aid of aging sequence of θ (Al₂Cu) phase. The decomposition sequence of θ (Al₂Cu) phase is as follows [15]:



However, the addition of particles to the matrix has changed their aging response drastically compared with their matrix alloys, and we can not use the ASTM B917 standard which were designated for the matrix alloys any longer. As a consequence, there is a strong interest in revealing the influence of ceramic particles on aging behavior of composites.

Some studies about the above problem have been made by several studies. Bartels et al. [16] have analyzed the aging response of TiB₂/AA6061 alloy and his results proved the existence of TiB₂ particles enhanced the aging velocity of matrix alloy. Salazar et al. [17] have also found similar enhancement in aging behavior of AA7005 alloy with Al₂O₃ particles introduced to the matrix. Hong et al. [18,19] revealed that the introduction of TiB₂ particles into the AA2009 matrix could not only increase the solution temperature ranges above the eutectic temperature of the matrix alloy but also give rise to two peak behaviors for the hardness of the composites as functions of aging time. Mandala et al. [20] reported TiB₂ particles accelerated the aging process in the Al-Cu alloy and he also found the rate of hardening on peak aging increased with increase of the amount of TiB₂ particles. From the above results, we can come to a preliminary conclusion that the introduction of TiB₂ particles are apparently responsible for a series of transformation. Nevertheless, these previous reports have observed some interesting phenomena, but there still lacked deep investigations into the mechanism of these phenomena. Some of the studies assisted by advanced techniques such as the synchrotron X-ray microdiffraction can

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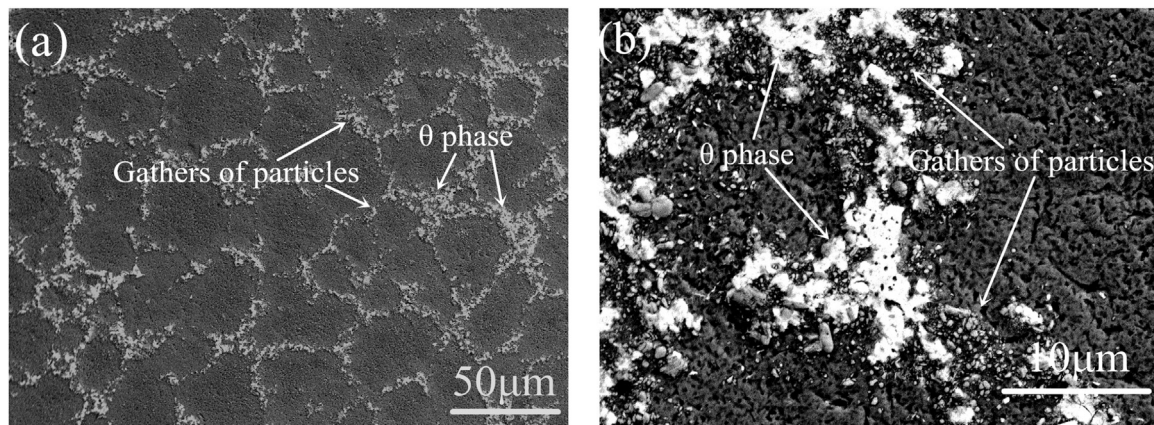


Fig. 1. BSE microstructures of TiB₂/Al-4.5Cu composites in as-cast condition: (a) Low magnification. (b) High magnification.

provide certain reference significance [21,22]. These two references paid the same attention to the microstructure and stress evolution under thermal cycling, and focused on the variation of the dislocation density. Meanwhile, the previous references [16–20] studied mainly on changes of hardness during aging process, and seldom on the mechanical properties, for instance, yield stress, ultimate tensile strength (UTS), which reflect directly on the effect of the heat treatment for the composites.

The objectives of this paper are to clarify the effect of TiB₂ particles on the aging behavior of in-situ TiB₂/Al-4.5Cu composites and obtain relations between aging behavior and mechanical properties of composites. It is the detailed investigation in this direction. Although this study was carried out on TiB₂/Al-4.5Cu composites, the authors believed that these results might be applied to other sorts of metal matrix composites.

2. Experimental procedures

The nominal chemical composition (in wt%) of the matrix alloy is Al-4.5%Cu. The in-situ 5 vol% TiB₂/Al-4.5Cu composites were produced by the addition of preweighted mixture of K₂TiF₆ and KBF₄ salts into the alloy melt at 840 °C through an exothermic reaction. Na₃AlF₆ was also added into reaction system as flux at 10% (in wt%) by the total mass of salts according to the experience. Salt-reaction process last generally for about one hour. After reaction, the slag was skimmed out as completely as possible, and the melt was cooled to 720 °C and cast into a preheated permanent mould to get as-cast ingot.

Belonging to a kind of high volume fraction TiB₂ particles reinforced composites, it is significant to consider the sediment and aggregation effects of the TiB₂ particulate. In accordance with Hashim's study [23], when the size of reinforced particles is below 10 μm, gravitational sediment effect could be negligible. However, according to previous study accomplished by Gao et al. [24], the reinforcements fabricated by the way mentioned above are composed of submicron particles. So, only the agglomerations of TiB₂ particles should be taken into consideration in this study. Gao et al. [25] have also found that ultrasonic vibration treatment for 4 min throughout the molten bulk matrix can not only further refine α-Al grains but also eliminate agglomerations of TiB₂ particles in the liquid. Herein, in order to get a uniform distribution of TiB₂ particles with the liquidoid situation, the same ultrasonic vibration treatments for 4 min with vibration power of 2.8 kW and ultrasound frequency of 20 kHz are applied in the TiB₂/Al-4.5Cu composites. Then the molten composites was immediately cast into a permanent mould preheated at about 200 °C.

After casting, specimens were cut from the composites ingots. The aging behavior of the composites was studied by solution treated at 530 °C for 6 h followed by quenching immediately in 70 °C water and aging at 180 °C for different intervals, i.e. 2 h, 3 h, 4 h, 6 h, 8 h, 10 h,

15 h, 20 h, 30 h, 40 h. During heat treatment, fluctuation of treated temperature was restricted within ± 2 °C. The composites were mechanically polished and then were etched with diluted hydrofluoric acid for 15 s to reveal microstructure morphologies. Microstructure observation for the samples was implemented by scanning electron microscope (FEI Nova-450). A transmission electron microscope (FEI Tecnai-G2) was introduced for TEM observation and identification of precipitates in aging process. TEM thin foils were prepared by the combination use of mechanical technology and ion beam thinning. The hardness of the composites was measured by using a Vickers hardness testing machine with a load of 30 kg load. Finally, room temperature tensile tests for the samples were performed on SHIMADZU AG-100 kN tester with a 1 mm/min crosshead speed. The samples were machined according to ASTM specification B557-14. To ensure the repetitive of composites, tensile data were taken from the average value of 5 specimens.

3. Results and discussions

3.1. Microstructure evolution in aging process

Fig. 1(a) shows that most of the TiB₂ particles are wrapped by bright white Al₂Cu phase in as-cast state, the coarse Al₂Cu phase can be seen at high magnification from Fig. 1(b). When the temperature of the molten matrix drops below the liquidus temperature, solidification starts. Small crystals appear and grow continually as the temperature decrease. Due to the lattice disregistry between TiB₂ ceramics and α-Al grains is far over than 5%, it is quite difficult for TiB₂ particles to be engulfed by the moving liquid/solid interface of the α-Al matrix [26]. This is one of the criteria which cause the TiB₂ particles to be surrounded by the last freezing areas of the molten alloy during solidification process [27]. While the temperature falls below the solid temperature, Al₂Cu phases form along with the grain boundaries in the matrix and adhere to the TiB₂ particles. Backscattered electron (BSE) observation has been introduced to distinguish intermetallic Al₂Cu phase and TiB₂ particles co-existing at grain boundary regions.

In the case of solutionised composites, the coarse Al₂Cu phase can hardly be seen in Fig. 2(a), only the clusters of TiB₂ particles gathering along grain boundaries could be observed in Fig. 2(b). These results indicate that Al₂Cu phase almost dissolved into the matrix of composites and a nearly homogeneous solid solution of the matrix came into being.

After the solution heat treatment, the samples were then artificially aged at 180 °C, and the decomposition of supersaturated solid solution began at this moment. As shown in Fig. 3(a) and (b), few and tiny θ phase precipitates can be clearly seen in the composites. With time going by, there is an obvious increasing tendency for the volume fraction of the precipitates in the composites. The θ (Al₂Cu) phase is a body-

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