Research Article

Yong-Hui Sun, Hong Yan*, and Jun-Jie Xiong

Al₃Ti/ADC12 Composite Synthesized by Ultrasonic Chemistry in Situ Reaction

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Abstract: Al₃Ti/ADC12 composite was synthesized in situ using Al-fluoride potassium titanate (K₂TiF₆) as the reaction system and an ultrasonic assisted direct melt reaction. Results indicate ultrasonic chemistry reactions are both accelerated and more complete compared to traditional in situ reactions. Al₃Ti reinforced particles with a regular shape and size of 1–2 μm were well distributed and as-cast microstructures of composites were superior. Composite particles under ultrasonic assistance were also refined to a greater extent. Tensile strength and elongation rate of the composites reached 255 MPa and 2.2%, an increase of 19.1% and 37.5% respectively to those without ultrasonic aid. Cleavage surface of the composite declined and the number of dimples increased while dimples became smaller and deeper, showing obvious ductile fracture.

Keywords: ultrasonic; in situ synthesis; Al₃Ti/ADC12; microstructures; mechanical properties

1 Introduction

ADC12 is a die casting alloy widely used in automotive and aerospace industries. Augmenting the long, needle-like and coarse microstructure of ADC12 alloy morphology is a principal objective as this will expand its application. The in situ synthesis reaction method is the predominant way to prepare particulate reinforced metal matrix composites. Reinforcements nucleated and grown in the

melt are in thermally stable phases. This means the reinforced phase has favorable wettability and will combine firmly with the matrix interface free from contamination. No harmful reactants are generated during in situ synthesis reactions and there is no need to synthesis, pretreat and add reinforcements [1–4]. Al₃Ti is ideal for the in situ reinforced phase due to the low density, high melting point, high elastic modulus and high temperature oxidation resistance properties of the particle [5, 6]. In situ preparation of Al₃Ti particle reinforced composites has gained considerable attention due to these characteristics [7–9]. The traditional mechanical stirring method requires longer reaction time at an increased temperature. The existing protocol also increases the growth and agglomeration of composites, making it difficult to wet with the melt. In recent years the practice of applying high energy ultrasonic assistance to prepare composite has vastly improved the in situ synthesis reaction and refined particle microstructure [10–14]. Aspects of the composites including tensile strength, yield strength and elongation rate are also superior.

The most effective way to combine casting and ultrasonic technology to prepare composites has become a popular research topic. Sreekumar *et al.* [15] have successfully manufactured Al–MgAl₂O₄ metal matrix composite using the in situ synthesis reaction method with ultrasonication. Results revealed a 10% increase in yield stress and 15% increase in UTS while maintaining ductility similar to the reference alloy. Patel *et al.* [16] reported on Al5083-TiC composites synthesized in situ using a self propagating high temperature synthesis process casting route with and without ultrasonic treatment. Finer TiC particles with an average size of 1–5 μm were dispersed uniformly in Al alloy matrix with ultrasonic aid. It was also found that hardness of the alloy and composites improved. Nano ZrB₂ particle reinforced 2024Al matrix composites synthesized from 2024Al–K₂ZrF₆–KBF₄ system by direct melt reaction were studied by Kai *et al.* [17]. It was demonstrated that high-intensity ultrasonic treatment could improve the nanoparticle uniformity and result in enhanced mechanical properties. Liu, *et al.* [18, 19] used pure Al melt and Ti powder to prepare Al alloy. Microstructure and mechanical properties got improved. In present, there are different ways to

*Corresponding Author: Hong Yan: School of Mechanical Electrical Engineering, Nanchang University, Nanchang 330031, China; Key Laboratory of Light Alloy Preparation & Processing, Nanchang University, Nanchang 330031, China; Email: hyan@ncu.edu.cn; Tel.: +86-791-8396-9633

Yong-Hui Sun, Jun-Jie Xiong: School of Mechanical Electrical Engineering, Nanchang University, Nanchang 330031, China; Key Laboratory of Light Alloy Preparation & Processing, Nanchang University, Nanchang 330031, China

Table 1: Chemical composition of ADC12 wt%

Al	Si	Cu	Mg	Fe	Mn	Zn	Ni	Pb	Sn
Bal.	10.5-11.5	3.0-3.5	≤ 0.3	0.3-0.6	0.3-0.5	0.6-0.9	≤ 0.5	≤ 0.1	≤ 0.1

1. temperature control instrument 2. amplitude transformer 3. crucible
4. thermocouple 5. mobile platform 6. ultrasonic generator 7. resistance furnace 8. ultrasonic controller 9. cooling plate

Figure 1: Schematic diagram of melt ultrasonic device

obtain Al₃Ti. One way is adding Ti powder into Al melt [20, 21], Another is adding K₂TiF₆ powder into Al melt [22]. The first one required higher temperature and longer time. In this paper, in situ Al₃Ti/ADC12 aluminum matrix composite has been synthesized using the direct melt reaction method with ultrasonic assistance in Al-K₂TiF₆ system. The microstructures and properties of in situ composites with and without ultrasonic assistance were compared and the mechanism of reinforcement is discussed.

2 Experimental

Experimental materials employed are ADC12 aluminum alloy (compositions are shown in Table 1) and K₂TiF₆ powder (purity ≥ 99.5%). A schematic diagram of the melt ultrasonic device is shown in Figure 1. The K₂TiF₆ powder was baked at temperatures from 250°C to 300°C for two to three hours. Next ADC12 alloy was overheated to 760°C in a graphite crucible with a heat preservation of 10 min. Following this an ultrasonic amplitude transformer probe was put into the melting furnace to preheat for five to ten minutes. Finally, the ultrasonic amplitude transformer probe was placed in the melt and ultrasonic assistance was applied at 700 W for three minutes at a frequency of 20kHz. Meanwhile 15g of K₂TiF₆ powder packaged in aluminum foil was pressed into the melt at a rate of six times

Figure 2: Tensile specimen geometry

every twenty seconds. (the addition of K₂TiF₆ powder accounts for 10% of the mass of ADC12 melts). The same ultrasonic frequency was reapplied to the melt at 720°C for two minutes at 800W. Refining, drossing and pouring was undertaken immediately after the ultrasonic aid and samples were removed after composites cooled. In situ particulate reinforced aluminum composites were successfully formulated through this process. Composites without ultrasonic assistance were prepared using the same process for comparison. Metallographic specimen sampling from the top, middle, and bottom of the ingot was corroded by 0.5% HF acid solution. Microstructures were examined using an optical microscope (Eclipse MA200, Nikon Metrology, Inc., Brighton, UK). Phase, microstructure and tensile testing was undertaken using X-Ray Diffraction (XRD) techniques and a scanning electron microscope (SEM, VEGA3, TESCAN CHINA, Ltd., Shanghai, China) with an energy dispersive spectrum (EDS). Grain size, dendrite arm spacing and aspect ratio of Si were measured with Image-Pro Plus 6.0 software. The porosity of each material was calculated by dividing the difference value between experimental and theoretical densities by theoretical density. The tensile test (UTM5105, Zhuhai SUST Electrical Equipment Co., Ltd., Zhuhai, China) was performed at room temperature on a universal test machine at a speed of 0.5 mm/min. Five tensile bars per material were tested with tensile specimen geometry illustrated in Figure 2.

3 Results and Discussion

3.1 Phase analysis

The XRD result of composites prepared via ultrasonic chemistry in situ synthesis reaction in the Al-K₂TiF₆ system is shown in Figure 3. In addition to the Al phase,

Figure 3: X-Ray Diffraction (XRD) pattern of $\text{Al}_3\text{Ti}/\text{ADC12}$ composite

Table 2: EDS results of arrow one and two in Figure 4a

Elements	Ti	Si	Al	Total
Arrow 1	21.10%	13.19%	63.88%	100%
Arrow 2	24.76%	10.06%	55.27%	100%

Si phase and CuAl_2 phase, diffraction peaks of the Al_3Ti phase are present. According to the literature [19], the main chemical reaction in the melt is as follows:

The SEM of the composite is shown in Figure 4(a). Observe that some small particles are distributed in the matrix. EDS analysis of arrow one and two in Figure 4(b) demonstrate the particle compositions are Al and Ti. According to the EDS analyses and atomic percentage of Al and Ti in Table 2, it was then determined that the reinforced phase of composites by in situ synthesis reaction could be Al_3Ti .

3.2 Microstructure

3.2.1 Reinforced particles

SEM images of composites prepared with and without high energy ultrasonic assistance are shown in Figure 5. Figure 5(b) is the marked area of Figure 5(a). Figure 5(a) and (b) shows that less reinforced particles of the composite were formulated without ultrasonic assistance and particle agglomeration occurred. The reinforced particle was large with an average size of 7–8 μm and badly-distributed. With ultrasonic aid the occurrence of particle agglomera-

tion gradually disappeared and the number of reinforced particle increased. Morphologies of the reinforced particles were regular and characterized by block and granular shapes as shown in Figure 5(c) and (d). Figure 5(d) is the marked area of Figure 5(c). These particles were smaller with an average size of 1–2 μm and well-distributed in the matrix.

3.2.2 The as-cast microstructures

Figure 6 displays the as-cast microstructure of $\text{Al}_3\text{Ti}/\text{ADC12}$ composite. White particles are Al matrix and the deep black is Si. In Figure 6(a) the particle outline is unclear and the size of the α -Al particle was thick. The outline of the particle became distinct in Figure 6(b) and the α -Al particle was also refined. In Figure 6(c), the α -Al particle grew finer, the outline was more precise and the morphology became increasingly rounded. It can be concluded that K_2TiF_6 enhances α -Al particle refinement. Using K_2TiF_6 with ultrasonic assistance can further refine α -Al. Quantitative analysis including dendrite arm spacing, grain size of Si and aspect ratio of Si were displayed in Figure 6(d). Figure 6(a) also shows the presence of a high number of long acicular-like and strip-like shaped Si phases which are large in size. These Si phases were badly-distributed and harmful to the mechanical properties of the casting. Figure 6(b) shows grains of the composite without ultrasonic assistance were refined to some extent. The Si phases were characterized by short rod-like and particle-like shapes. As shown in Figure 6(d), dendrite arm spacing, the grain size of Si and aspect ratio of Si decreased compared with ADC12 alloy. Figure 6(c) illustrates that composite grains were refined greatly with ultrasonic assistance and the Si phases were well-distributed and characterized by smaller particle-like shapes. The grain size of Si, dendrite arm spacing and aspect ratio of Si got further decreased compared with the composite without ultrasonic assistance. It can be concluded that Si phases were refined by adding K_2TiF_6 and this was accelerated with ultrasonic aid.

With ultrasonic assistance the composites improved in number, size, morphology and grain dispersion. Composite microstructure was refined to some extent compared with the matrix and increased further when assisted by high energy ultrasonic aid. This result is attributed predominantly to the effect of ultrasonic cavitation and acoustic streaming on the propagation of high energy ultrasonic waves in the melt. Ultrasonic cavitation is a series of dynamic processes in the expansion of ‘micro-bubbles’ leading to increased local temperature and pressure. The phys-

Figure 4: (a) SEM image and (b) energy dispersive spectrum (EDS) analysis of Al₃Ti/ADC12 composite

ical or chemical state of the melt was changed so that the in situ reaction was accelerated. According to the following Equation:

$$P_{\max} = P_v \left[\frac{p_m(\gamma - 1)}{P_v} \right]^{\gamma/(\gamma-1)} \quad (2)$$

$$T_{\max} = T_{\min} \left[\frac{p_m(\gamma - 1)}{P_v} \right] \quad (3)$$

In the Equation, P_v is the pressure of the cavitation bubble's initial radius which can be approximated as 0.1 MPa. P_m is the sum of sound pressure amplitude and static pressure and the number of sound pressure amplitude provided by the experimental equipment is 3.19 MPa so that $P_m = 3.29$ MPa. γ is the specific heat ratio of the gases in the bubble and equates to 1.4. It is assumed that intense ultrasonic attenuation does not exist and $P_{\max} = 826.79$ MPa. According to Equation (2), $T_{\min}$ is the heating temperature of the melt and is taken as 760°C at the first instance of ultrasonic aid. It can be calculated that $T_{\max} = 1.3327 \times 10^4$ °C according to Equation (3). When $T_{\min}$ is taken as 750°C in the second instance, it can be calculated that $T_{\min} = 1.2828 \times 10^4$ °C according to Equation (3). Due to the effect of ultrasonic cavitation a local transient high temperature of 10^4 °C level is produced in the melt. This can create an increase in the diffusion coefficient of K₂TiF₆ powder and dramatically improve the reaction process. The process also reduces surface tension of the melt and the wettability of the Al₃Ti refined particle. The aluminum liquid is also superior, creating an increase in the yield of in situ particles. Al₃Ti particles agglomerated at high temperatures

were dispersed by high pressure shock wave produced by the acoustic cavitation effect, becoming refined and evenly distributed throughout the matrix. In addition, a transient cavity was produced by the impact of a strong sound wave on the melt interface which caused an asymmetrical collapse. This promoted the in situ reaction effectively as the K₂TiF₆ powder surface impacted released micro jet. The precipitated α -Al particle was also broken in this process, creating further grain refinement.

The surface energy of melt σ_{LG} was reduced due to instantaneous local high temperatures produced by the ultrasonic cavitation effect. Adsorbed gas and impurities could be eliminated via degassing and slag removal induced by ultrasonic cavitation creating an σ_{SG} increase. Under conditions of high temperature and pressure, the very thin interfacial reaction layer between Al₃Ti particles and the aluminum melt cause a decrease in σ_{SL} . According to Young's Equation [20]:

$$\cos \theta = \frac{\sigma_{SG} - \sigma_{SL}}{\sigma_{LG}} \quad (4)$$

In this Equation, θ is the contact angle, σ_{SG} is the interfacial energy of solid-gas, σ_{SL} is the interfacial energy of solid-liquid and σ_{LG} is the interfacial energy of liquid-gas. According to the Equation, the wettability of the melt and reinforced particles can be improved with the decrease of contact angle θ .

Dendritic crystals as well as long needle and plate-like Si phases are broken up by high pressure shock waves induced by ultrasonic cavitation [21]. This would restrain the growth of grains and refine the structure of matrix.

Figure 5: SEM images of the composites (a) in low magnification without ultrasonic assistance; (b) in high magnification without ultrasonic assistance; (c) in low magnification with ultrasonic assistance; (d) in high magnification with ultrasonic assistance

The orientation relationship [22] between Al_3Ti and $\alpha\text{-Al}$ is as follow: (006) Al_3Ti // (022) Al, (122) Al_3Ti // (110) Al. The lattice constant mismatch between Al_3Ti and $\alpha\text{-Al}$ is only 5.2% which easily leads to the heterogeneous nucleation basement of the $\alpha\text{-Al}$ particle [23]. Additionally, K_2TiF_6 reagent enhanced the microstructure of the ADC12 aluminum alloy to some extent as a kind of refining flux.

It is also possible that a portion of in situ synthesis of Al_3Ti particles contact the Si phase with low energy interface causing their lattices to resist a corresponding relationship. When the mismatch is small enough to satisfy the corresponding conditions of the lattice, Al_3Ti particles can serve as the heterogeneous nucleation core of partial Si phase to refine it.

Figure 6: As-cast microstructures of (a) ADC12; (b) Al₃Ti/ADC12(without ultrasonic); (c) Al₃Ti/ADC12(with ultrasonic); (d) quantitative analysis

The acoustic streaming effect was induced via ultrasonic assistance. Therefore, a certain sound pressure gradient would be generated in the melt leading to melt flow and a strong circulation within the whole melt. The maximum flow rate can be estimated by the following Equation:

$$u = \sqrt{2\pi f A} \quad (5)$$

In this equation, A is the maximum amplitude of the probe and f is the ultrasonic frequency. For this experiment $f = 20$ kHz and $A = 30$ μm so that $u = 2.67$ m/s according to Equation (5). Acoustic streaming caused by ultrasonic aid in the melt can move at a great speed. The flow velocity of acoustic streaming can reach 10 to 10³ times the flow rate of the fluid convection. Acoustic streaming

is attributed to circulation characteristics. The small vortex generated in the melt can cause in situ synthesized particles to roll in cycle so that Al₃Ti particles can diffuse from the surface of K₂TiF₆ quickly. Direct contact area of K₂TiF₆ and liquid aluminum surface is increased creating a more thorough reaction. The flow of finite amplitude acoustic streaming in a viscous melt is created by the turbulent which agitates the particles. The force generated in different sizes and directions for varying positions of particle clusters can force the particle clusters to scatter and restrain the agglomeration of reinforcing particles. The broken crystals present in the non-condensate zone of the melt formed the role of the crystalline nucleus and the matrix structure was refined. Simultaneously, wetting

Figure 7: SEM images of tensile fracture surfaces of composite (a) low magnification without ultrasonic assistance; (b) high magnification without ultrasonic assistance; (c) low magnification with ultrasonic assistance; (d) high magnification with ultrasonic assistance

between the particles and matrix was improved because of the cavitation erosion produced by the ultrasonic. Casting porosity decreased due to degassing and slag which aided in the improvement of the comprehensive properties of composite.

In conclusion, the thermodynamic and dynamic environment of K_2TiF_6 powder in the melt improved with high-

energy ultrasonic assistance. It was found to accelerate the reaction, enhance wetting and dispersion of Al_3Ti particles and matrix as well as improve the yield of Al_3Ti particles. With appropriate ultrasonic assistance it was easy to obtain a finer and more evenly distributed reinforced phase with further refinement in the microstructure of the composite.

Table 3: Mechanical properties of the matrix and composites

Materials	Ultrasonication	Porosity/%	Tensile strength/MPa	Elongation/%
ADC12	without	0.26	186±4	2.4±0.1
ADC12	with	0.30	202±3	2.7±0.1
Al ₃ Ti/ADC12	without	0.32	214±4	1.6±0.1
Al ₃ Ti/ADC12	with	0.35	255±4	2.2±0.1

3.3 The mechanical property

The mechanical properties of the materials were compared in Table 3. As illustrated in the table, tensile strength and elongation of the ADC12 alloy with ultrasonic assistance reached 202 MPa and 2.7%. This marked an increase of 8.6% and 12.5% respectively when compared to those without ultrasonic assistance. Tensile strength and elongation of the Al₃Ti/ADC12 composites synthesized with ultrasonic assistance reached 255 MPa and 2.2%. This marked an increase of 19.1% and 37.5% respectively when compared to those without ultrasonic assistance. With ultrasonic aid the volume fraction of particles in the composites grew, their size decreased, shape became more regular and the particles were dispersed in the matrix. A large amount of dislocations and holes were also produced. When subjected to a single pull, the small, dispersed Al₃Ti can effectively hinder the movement of dislocations and delay the propagation of a crack. The appropriate parameters of high energy ultrasonic assistance can break block-like or strip-like shapes as well as long acicular-like shape Si phases into short rod-like and particle-like shapes. The coarse α -Al dendrite will also be refined to some extent. Microstructure of the composites can also be improved by the secondary effect produced by ultrasonic assistance. K₂TiF₆ powder can be used as a refining flux to improve the microstructure of ADC12 alloy and in situ synthesized Al₃Ti particles can be the core of heterogeneous nucleation of α -Al particle. Some of the Al₃Ti reinforcing particles may also act as heterogeneous nucleation substrates for the Si phase in the matrix, thus playing a role in refining. Performance depends on good structure so the mechanical properties of the composites can be further improved.

3.4 Scanning electron microscope (SEM) images of tensile fracture surfaces

Figure 7 shows the morphology of the tensile fracture at room temperature of composites prepared by the Al-K₂TiF₆ system with or without ultrasonic assistance. Figure 7(b) is the marked area of Figure 7(a). As shown in Fig-

ure 7(a), and (b), the fracture surface of composite without ultrasonic assistance was distributed across a large flat area and a long strip of tear. This would greatly reduce the plasticity of the material. Al₃Ti particles were visible which increased the brittleness of the material and can be attributed as the origin of the fracture which caused the matrix to crack in force. The fracture mode is mainly cleavage fracture. In using high energy ultrasonic aid to process the melt, the ultrasonic cavitation and acoustic streaming effect vastly improved the in situ synthesis reaction leading to a more thorough reaction. Figure 7(d) is the marked area of Figure 7(c). As exhibited in Figure 7(c) and (d), the number of dimples on the fracture surface of the composite increased gradually and the brittle flat area decreased. The dimple became smaller and deeper with fracture characteristics gradually transforming from cleavage fracture into ductile fracture. This further verified that composite performance can be much improved using ultrasonic assistance.

4 Conclusions

1. The number of Al₃Ti particles in the Al₃Ti/ADC12 composite prepared using ultrasonic chemistry in situ synthesis reaction in the Al-K₂TiF₆ system increased. Particle morphology and size were small with an average approximating to 1~2 μ m and Al₃Ti was dispersed in the matrix alloy.
2. The as-cast structure of Al₃Ti/ADC12 composite prepared using the in situ synthesis reaction improved to some extent when compared to ADC12. The outline of the α -Al particle became clearer, the size was gradually refined and the morphology typically was rounded. The morphologies of Si phases transformed gradually from long acicular-like and strip-like shapes into short rod-like and particle-like configurations. In the absence of ultrasonic assistance, crystal grains of the composite were refined to some extent though Si phases of bigger particle-like shape dominated over Si phases of short rod-like shapes.

Crystal grains of the composite with ultrasonic assistance were largely refined and the morphologies of Si phases transformed to smaller particle-like shapes and were even nemaline.

3. Tensile strength and elongation of Al₃Ti/ADC12 composites synthesized by the ultrasonic chemical in situ synthesis reaction in Al-K₂TiF₆ system reach 255 MPa and 2.2%. This presents an increase by 19.1% and 37.5% respectively, compared to those without ultrasonic assistance. The cleavage surfaces were minimized and the quantity of dimples increased. When synthesized with ultrasonic aid dimples also diminished in size and deepened in the composite, performing as a ductile fracture.

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