



Novel application of ultrasonic cavitation for fabrication of TiN/Al composites



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ABSTRACT

A developed approach for a convenient fabrication of titanium nitride (TiN) particulate reinforced commercially pure aluminum (CP Al) composites via ultrasonic cavitation was proposed. The process involved the addition of large size TiN particles (average particle size of 264 μm) into Al melt at 1033 K. In the meantime, ultrasonic vibration was applied into the melt to comminute the large size TiN particles and disperse the produced fine TiN particles simultaneously. Microstructural characterization indicated that the produced TiN particles were much smaller than the starting TiN particles (average particle size of 15 versus 264 μm) and distributed uniformly in the matrix. The fine TiN particles were stable during the process and had a clean interface with the Al matrix. The ultimate tensile strength increased by up to 45% after the formation of fine TiN particles in the commercially pure Al matrix, whilst the hardness increased by a factor of 3. Dry sliding friction test showed that the coefficient of friction of the composites was more stable and lower than the CP Al matrix.

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1. Introduction

Interest in particulate reinforced aluminum metal matrix composites (MMCs) for the use in the automotive industry and other structural applications has increased because of the physical and mechanical properties they possess [1–3]. Aluminum based MMCs with a variety of particulate reinforcements such as Al_2O_3 [4], SiC [5], TiC [6], Ti_2B [7], B_4C [8], TiN [9], Si_3N_4 [10], CNT [11], graphite [12] and industrial waste byproduct [13] have been conceived and developed for various potential applications. However, the extensive applications of particulate reinforced aluminum MMCs have been hindered by the availability of relatively inexpensive, suitable particulate material and high cost of producing complex components [10]. There are two approaches for the fabrication of particulate material: viz bottom-up and top-down. Bottom up approach whether within or outside the matrix involves chemical reactions which make the process more complicated. Top down approach (comminution process) is defined as the mechanical breakdown of solids from the large particle size to a small particle size. It is a relatively simple and low-cost way to produce massive powders.

The technical difficulty of this approach is to ensure all the particles are broken down to the required particle size with low energy consumption. Currently, using low-cost crushed abrasive grade ceramics for the reinforcement of metals is generally done. The one which has received most attention is micron-sized SiC ceramic particles. However, crushed particles always contain internal defects, including cracks or sharp edges along their surfaces. This may cause the stress concentration within the composites when it is stressed, causing the sharp reduction of the ductility [14]. In addition, the particles produced by comminution process may get in contact with air, humidity and other normal contaminants in the atmosphere and the production procedure. It was also suggested that spectacular improvements in composite properties can be further obtained if one uses particles produced by other comminution methods with few internal defects and no sharp angles [15]. In recent years there has been a considerable increase in the use of ultrasound for the comminution of solid materials to produce micron-sized particles [16], even nanoparticles [17]. Ultrasound is considered as a new, powerful and economical ceramic powder fabrication technology, which can prevent contamination of powders with foreign materials (pickup from grinding medium, etc.). But most of the researches were carried out in aqueous media at room temperature. Among the various techniques reported for producing particulate reinforced aluminum MMCs, casting

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processes are being extensively studied due to its capability to produce products with complex shapes and hence minimizes the cost. In general, casting of particulate reinforced MMCs involves the successful introduction of the reinforcement material into the melt.

TiN particulate reinforced aluminum MMCs are very attractive due to its high hardness, high melting point, especially its thermodynamically stability with molten aluminum [9] (e.g. SiC reacts with Al melt). Therefore, TiN/Al composites have been produced by several manufacturing techniques, such as powder metallurgy [9,18], cold spraying [19], in situ technique [20]. However, the production of TiN particulate reinforced aluminum MMCs by casting process is associated with some difficulties due to a number of reasons. The wettability between the Al melt and TiN is poor (the contact angle is 135° at 1173 K), which makes the incorporation and retention of the particles in the liquid extremely difficult. This situation is becoming even worse when the particle size decreases. The practice of addition of surface active elements or coating the particles may affect the Al structure and make the process more complicated. Genma et al. [4] found experimentally that the volume fraction of incorporated Al_2O_3 particles into molten Al–Mg alloy increased with the increase of particle size and application of ultrasonic vibration. It is considered that the application of ultrasonic vibration to melt enhances the wettability between liquid metals and particles, thus making the fabrication of as-cast composites with micro to nano particles successful [5]. However, further research and development are still needed to improve the understanding of the mechanism of the effects of ultrasonic technology. The above review indicates that ultrasound could be utilized to fragment large particles to produce fine particles and simultaneously disperse them uniformly in liquid melts, thus, making the production of particulate and as-cast composites more convenient, energy-saving and feasible. This study is to investigate the feasibility of this concept and the mechanism of ultrasound in liquid melts.

2. Experimental procedure

Commercially pure aluminum (99.6% purity) was selected as the matrix of the composite. Starting TiN powder (average particle size of $264\text{ }\mu\text{m}$) was used as reinforcement material. The starting TiN powders were the raw material to produce fine TiN powders through mechanical comminution process in industry. Al ingot was melted in a graphite crucible in an electrical resistance furnace and superheated by about 100 K. Ultrasonic vibration was transmitted into the melt by a titanium waveguide dipped into the melt. Starting TiN powders ($\sim 15\text{ g}$) were fed into the melt during the ultrasonic vibration from the top of the crucible. The ultrasonic vibration was applied to the aluminum melt for approximately 20 min until the aluminum is solidifying in the furnace. Representative samples for metallographic examinations and various mechanical tests were cut from the solidified TiN/Al composites casting. The microstructure of the polished samples and interface between TiN particles and the matrix were examined by using optical microscope (OM, Olympus-BX51M), scanning electron microscope (SEM, MERLIN, ZEISS) equipped with energy dispersive spectroscopy (EDS) and transmission electron microscope (TEM, JEM-2100). X-ray diffraction (XRD, Bruker D8, 40 kV, 40 mA) using $\text{Si K}\alpha$ radiation with a scan rate of $4^\circ/\text{min}$ was used to determine the phase component and the presence of any reaction product of the starting TiN Powders and TiN/Al composites, respectively. To determine the tensile properties, the cast composites and matrix samples were machined with a rectangular cross-sectional area of $1 \times 3\text{ mm}^2$ and gauge length of 10 mm. The tensile tests were performed by using a universal material testing system (ZWICKZ005). To evaluate the hardness of the matrix and

composites, the test was carried out by using microhardness tester MH-3 with a load of 200 gf for 15 s. To obtain the microhardness on the reinforcement and interfacial zone, a load of 25 gf for 15 s was used. More than 15 values measured on the polished samples were averaged to determine the sample hardness. Dry Sliding Friction tests of the polished CP Al and composites were conducted on a reciprocating ball against the stationary, horizontally mounted sample by UMT-2 tribometer. GCr 15 steel balls of 4 mm with a mirror finished surface and hardness of 60 HRC was used as the sliding partner. The applied normal force was 5 N and the stroke length was 5 mm the oscillation frequency was 10 Hz. The coefficient of friction was determined by dividing the friction force by the normal load. Three replicated tests were conducted for each test condition.

3. Results and discussion

Fig. 1a shows the SEM image of starting TiN powders. The particle size distribution and average particle size of the starting TiN powders are 100–700 μm and 264 μm , respectively. The shape of TiN particles is irregular. Fig. 1b shows the surface structure of a typical TiN particle. It reveals that TiN particles are polycrystalline and there are many tiny pores on the surface of TiN particles. Fig. 1c shows the XRD patterns of the starting TiN powders and synthesized TiN/Al composites. The XRD pattern of the TiN powders conforms with the JCPDS Card 38–1420. The XRD pattern of composites reveals the presence of Al and TiN peaks. Apparently, the TiN particles maintain the crystal structure without any distinct phase changes. No crystalline peaks from other phases can be observed, indicating that TiN particles were stable during the ultrasonic vibration in aluminum melt. This result corroborates the absence of any interfacial reaction between the particles and the Al matrix that is also evident from the SEM and TEM micrographs as shown in Fig. 3a–d. The strong diffraction peaks of TiN indicate the high content of TiN particles in the matrix. This suggests that a mass of TiN particles were successfully incorporated into the matrix. Difficulties, safety risks and wetting problems related to handling and incorporating of fine reinforcement powders were also avoided.

Fig. 2 shows the typical OM micrograph of TiN/Al composites. The microstructure of TiN/Al composites is characterized by a mass of uniformly distributed gold-like particulates. The gold-like particulates are TiN particles as TiN usually reflects in a spectrum similar to elemental gold, which gives it the appearance of gold. In addition, the particles are much smooth and rounded in morphology. The size of most TiN particles (in number) is in the range of $\sim 30\text{ }\mu\text{m}$. The average mass fraction and particle size of the TiN particles are about 12% and 15 μm determined by image analysis system (ImageJ2x software). The average particle size of the produced fine TiN particles is much smaller than the starting TiN of 264 μm . Based on the observation, it can infer that the sharp decrease of the average particle size of TiN particles was attributed to the fragmentation of starting TiN particles induced by the interaction between ultrasound and starting TiN particles. It was known that the effects of ultrasound derive primarily from ultrasonic cavitation: bubble formation and its subsequent collapse in a liquid. It was generally considered that the bubble nuclei stabilized in hydrophobic conical cracks and crevices of solid surfaces in the liquid promoted the cavitation inception process [21]. A step forward was made by Bremond [22], who controlled the position and size of cavitation bubble on a solid wafer by using a hydrophobic surface patterned with microcavities. When the bubble collapses, it drives both shock wave and high-speed microjet into the solid surface. These effects may also lead to the fragmentation of particles. It is obvious in this work that cavitation bubbles tend to occur

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