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Manufacturing of Al-TiB₂ Nanocomposites by Flux-Assisted Liquid State Processing

Abdolreza Javadi^a, Chezheng Cao^b, and Xiaochun Li^{a,b,*}

^aDepartment of Mechanical and Aerospace Engineering, University of California Los Angeles, Los Angeles, California, 90095, United States

^bDepartment of Material Science and Engineering, University of California Los Angeles, Los Angeles, California, 90095, United States

Abstract

Aluminum matrix nanocomposite (AMNC) materials are of great interests for various structural and functional applications for automotive, aerospace, and military. In this study pure aluminum (Al) with one and two volume percent (vol.%) of titanium diboride (TiB₂) nanoparticles were produced via flux-assisted liquid state processing. Adding flux during nanoparticle feeding can significantly improve nanoparticle incorporation into molten Al. TiB₂ nanoparticles as small as 100 nm were successfully incorporated into pure Al using potassium aluminum fluoride (KAlF₄) flux. TiB₂ nanoparticles were fairly distributed and dispersed in the Al-2 vol.% TiB₂ nanocomposite. Vickers hardness of the Al-2 vol.% TiB₂ nanocomposite was higher than the as-received pure Al. While KAlF₄ flux is proven to enhance the nanoparticle incorporation efficiency in Al system, further study is needed to thoroughly remove the flux remnant from Al matrix, to enhance the effect of the TiB₂ nanoparticle reinforcement.

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1. Introduction

One of the great challenges of this century is the high demand for lightweight metals with promising physical, chemical, and mechanical properties for various structural and functional applications [1, 2]. In lightweight metals family, aluminum is one of the most abundant lightweight metals in Earth and is used in many industries such as

* Corresponding author

aerospace, automobile, and naval. Pure Al with low strength and high ductility is less appealing for certain applications where high strength and lightweight are needed. Although alloying pure Al with other metals such as Cu and Zn [3] would result in higher strength, there is a limit to how much these elements can be added. On the other hand, Al matrix nanocomposites can offer excellent properties to compete with high strength and high density metals [4]. AMNCs are a class of materials in which the matrix is pure Al or its alloy and the nano-reinforcement (i.e. nanoparticle) can be represented by different metals, ceramics or organic compounds [5-9]. High performance AMNCs materials have great potentials to improve energy efficiency and system performance in numerous applications. While many researchers strived to develop AMNCs with significant enhanced mechanical property, limited success have been reported to produce bulk AMNCs with uniform dispersion of the nano-reinforcements [10-12].

1.1. Production of Metal Matrix Nanocomposites

There are several approaches for manufacturing of the AMNCs, such as solid state processing (i.e. powder metallurgy), Liquid state methods (i.e. pressure infiltration and squeeze casting), and semi-solid state processing (e.g. semi-solid powder processing). Among the aforementioned processes, liquid state solidification processing is promising for an economical manufacturing of bulk metal matrix nanocomposite specifically for those with crystalline materials as matrix. However, efficient feeding and dispersion of nano-reinforcements in crystalline matrix is still a great challenge [13].

1.2. Nano-reinforcement Feeding and Incorporation

Direct feeding of nano-reinforcements to molten metals has several potential problems such as partial nano-reinforcement burning and loss during feeding [14, 15]. These challenges reduce the incorporation efficiency and therefore less effect on mechanical property improvement. On the other hand, surface oxide formation during liquid state processing at high temperatures poses a barrier for effective incorporation and dispersion of nano-reinforcements due to a reduced wetting between nano-reinforcements and matrix. Using a flux agent, protection gas or vacuum processing can significantly mitigate this barrier and improve nano-reinforcement incorporation efficiency [16]. Achieving a uniform dispersion of nano-reinforcements within crystalline matrix is crucial to enhance the mechanical property of metal matrix nanocomposites [17, 18].

2. Materials and Method

For this study TiB₂ was chosen as the nano-reinforcement since it is an extremely hard ceramic. High purity Al (99.99%) ingot and in-house synthesized TiB₂ nanoparticles with an average size less than 100 nm were used as matrix and nano-reinforcement, respectively. TiB₂ nanoparticles (2 g) and KAlF₄ flux (13 g) were mechanically mixed at solid state for 3 hours. Mixed powders were dehydrated at 120 °C for 1 hours in a vacuum oven. An electrical resistance furnace was used to melt the Al ingots (39 g) at 800 °C under argon (Ar) gas protection. Then, the mixed powders were added to the melt surface and melt was mechanically stirred at 200 rpm for 10 minutes with a one-inch diameter titanium (Ti) mixing blade. The melt was naturally cooled down to room temperature under Ar gas protection. The final product was (in the shape of a disk with 1.5 inches in diameter and 1.0 inch in height) carefully extracted from the graphite crucible. Al-X vol.% TiB₂ (X=1 and 2) nanocomposites were produced (three study samples were prepared from top, middle, and bottom of each nanocomposite sample). Figure 1 shows the schematic of the experimental setup.

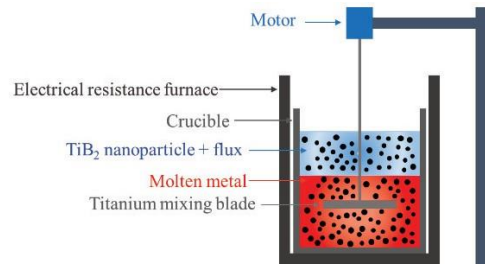


Fig. 1. Schematic of the flux-assisted liquid state processing experimental setup.

3. Results and Discussion

3.1. Microstructure Analysis

Al-X vol.% TiB_2 (X=1 and 2) nanocomposites were produced via the flux-assisted liquid state processing. Al-X vol.% TiB_2 (X=1 and 2) nanocomposites were cut, and three study samples were prepared from top, middle, and bottom part (all from center). The samples were first mounted on graphite-based powder mounting material and then were grinded and polished. The mounted samples were studied by optical microscope (OM) and scanning electron microscope (SEM). The OM and SEM study for Al-1 vol.% TiB_2 samples revealed that most of the TiB_2 nanoparticles remained in the flux zone. It is likely that the Ti blade was not properly positioned inside the Al melt, therefore most of the TiB_2 nanoparticles remained in KAlF_4 flux and only a few percent of TiB_2 nanoparticles were incorporated to the top surface of the Al matrix. In the Al-2 vol.% TiB_2 nanocomposite samples, we observed that the TiB_2 nanoparticles were effectively incorporated in Al matrix. Figure 2A shows the dense TiB_2 nanoparticle area (pseudo-clusters) distributed throughout the Al matrix. Further characterization inside the pseudo-cluster area showed that TiB_2 nanoparticles retain a few nm gap between one another (not sintered). Figure 2B shows a high magnification SEM image taken from inside a pseudo-cluster zone. This can be explained based on the attractive van der Waals forces and a lack of repulsive forces exist between TiB_2 nanoparticles along with relatively poor wettability between Al matrix and TiB_2 nanoparticle at 800 °C. Further improvement is needed to uniformly disperse the nanoparticles, possibly using smaller TiB_2 nanoparticles to enable their self-dispersion in molten Al [17]. Figure 2B was processed by ImageJ software to obtain the smallest and largest nanoparticle. Our study shows that nanoparticle size varies from 100nm to 1Pm.

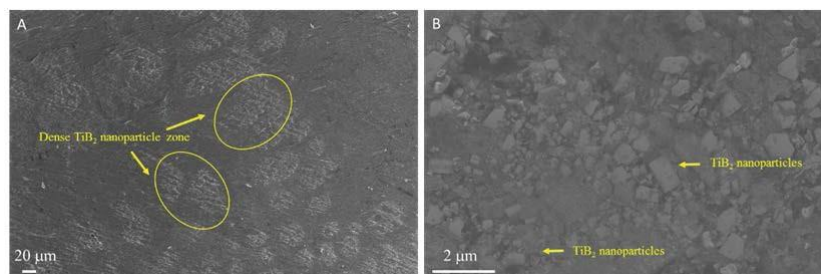


Fig. 2. A) A low magnification SEM image of the Al-2 vol. % TiB_2 study sample illustrating dense TiB_2 nanoparticle zone (pseudo-cluster zone), B) A high magnification SEM image showing inside a typical dense TiB_2 nanoparticle zone where TiB_2 nanoparticles are separated from one another (Al-2 vol. % TiB_2 sample).

In order to confirm the presence of Ti element in the Al matrix, energy dispersive spectroscopic (EDS) was performed on three study samples at different locations. Our EDS study indicated that Ti element along with Al element can be clearly identified in all of the study samples. Figure 3 shows a typical EDS analysis result.

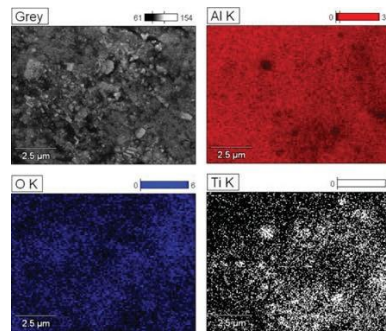


Fig. 3. Energy dispersive spectroscopy results from a study sample (prepared from Al-2 vol. % TiB_2 nanocomposite).

There were two unwanted phases embedded inside the Al-X vol.% TiB_2 (X=1 and 2) nanocomposite samples, KAlF_4 and titanium aluminide (AlTi_3) as shown in figure 4A. It must be noted that these phases only exist at the top surface of the Al nanocomposite (at the interface between flux and Al matrix). AlTi_3 intermetallic phase exist due to the Ti blade used for stirring purpose. A Ti blade can readily react with Al matrix at 800 °C and form eutectic AlTi_3 . To confirm this hypothesis, the Ti blade was examined before and after the experiment. Figure 4B shows a Ti blade before experiment, and Figure 4C shows the same blade after the experiment. It is evident that the blade wore down during the stirring step. Study of the middle and bottom part of the sample did not show similar phenomenon.

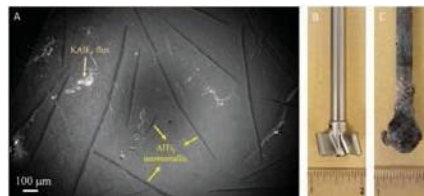


Fig. 4. A) A SEM image of the Al-2 vol.% TiB_2 nanocomposite sample demonstrating the two unwanted phases: AlTi_3 and KAlF_4 , B) An image of the Ti blade before stirring step, C) Ti Blade image after stirring the liquid mixture at 200 rpm for 10 min.

3.2. Vickers Hardness Test

Vickers hardness test was performed on the Al-2 vol.% TiB_2 nanocomposite study samples at random position and the results were compared with as-received pure Al ingot. 32 ± 3 and 18 ± 2 HV were obtained for Al-2 vol.% TiB_2 nanocomposites and pure Al (as-received), respectively (figure 5). The increase in hardness value for Al-2 vol.% TiB_2 nanocomposite is ascribed to the presence of the TiB_2 nanoparticles. However, hardness increase is not that significant yet. This can be attributed to three main reasons. First, the TiB_2 nanoparticles did not dispersed well in Al matrix (formed pseudo-cluster zones), which resulted in low hardness enhancement. The other two reasons are porosity and flux remnant in the Al-2 vol.% TiB_2 nanocomposite. These unwanted phases/defects can adversely reduce the nanoparticle reinforcing effect.

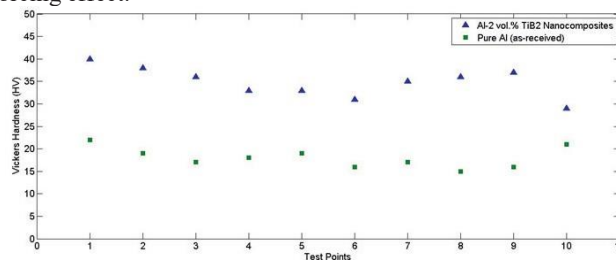


Fig. 5. Vickers hardness test result for Al-2 vol.% TiB_2 nanocomposites and pure Al (as-received).

4. Conclusions

Al-TiB₂ nanocomposites were produced using the flux-assisted liquid processing. In Al-2 vol.% TiB₂ nanocomposite samples, TiB₂ nanoparticles were incorporated into Al matrix successfully in a form of isolated pseudo-cluster zones. Inside the pseudo-cluster zone, TiB₂ nanoparticles maintained a few nm gap from each other, preventing them from sintering and consequently cluster formation. Although a fair dispersion and distribution of the TiB₂ nanoparticles was achieved, the presence of KAlF₄ and AlTi₃ downgraded the TiB₂ nanoparticle reinforcing impact. Al-2 vol.% TiB₂ nanocomposites exhibit hardness enhancement compare to pure Al (as- received). Further improvement in the feeding efficiency is needed to increase the volume percent of nanoparticles embedded in Al matrix. We believe that our manufacturing approach has an advantage of scalability compare to other conventional approaches such as powder metallurgy. By a complete removal of the flux in the final Al-TiB₂ nanocomposites our approach has the potential for industrial production.

Acknowledgements

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