Properties of Al/Al$_2$O$_3$-TiO$_2$ composites prepared by powder metallurgy processing

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Abstract

In this paper, aluminum metal matrix composite (AMCs) containing Al$_2$O$_3$-TiO$_2$ composite powder in different ratios (5, 10, 15, and 20 wt.%) as a reinforcement material was produced using the powder metallurgy method. This paper aims to research the effect of the alumina-titanium oxide powder ratio on Al/Al$_2$O$_3$-TiO$_2$ composite properties. An experimental study has been carried out on density, hardness, tensile strength, phase, and microstructure characterization of aluminum metal matrix composites. The findings of the experimental investigation reveal that the AMCs with 20 wt.% Al$_2$O$_3$-TiO$_2$ exhibited gradually increased mechanical properties, i.e., Vickers hardness of 56 HV and the tensile strength of 150 MPa. It has been concluded that the density of composites improved with the addition of Al$_2$O$_3$-TiO$_2$ reinforcement. Microstructural observation indicates that the distribution of Al$_2$O$_3$-TiO$_2$ particles is usually near-uniform and homogeneous in the aluminum matrix.

Key words: aluminum-based composite, powder metallurgy, Al/Al$_2$O$_3$-TiO$_2$, mechanical behavior

1. Introduction

Similar to polymer and ceramic matrix composites, metal matrix composites (MMCs) take advantage of strong, stiff but brittle reinforcements to improve composite strength and stiffness. The improved properties of metal matrix composites make them an effective alternative to traditional engineering materials. MMCs are comprised of titanium, aluminum, magnesium, copper, and their alloys as a matrix along with a typical reinforcement ceramic material such as carbide, oxide, and boride, in the form of fiber or particles [1]. Particle-reinforced metal matrix composites have paved the way for a unique field of materials science and engineering over the past decade as potential applications for aerospace, automotive, and other structural components. The ceramic reinforcement phase is frequently used in metal matrix composites for improving their mechanical and thermal properties.

Aluminum matrix composites have attracted the attention of materials scientists because of the magnificent combination of properties (such as high strength, low density, improved wear resistance, and thermal properties). Hence, fields such as automotive, aerospace, gas turbine engines, helicopters, military aircraft, etc., have used these materials as lightweight components [2].

Aluminum matrix composites have been under development for many years, with many different types of reinforcement attempted with varying degrees of success. Continuous and short fibers, whiskers, and particulates can be considered among these. Ceramic particle reinforced composites display enhanced strength due to dislocation and dispersion strengthening. Moreover, ceramic particles also provide support for improving the hardness of MMCs [3]. TiB$_2$, ZrB$_2$, TiC, SiC, Al$_2$O$_3$, and B$_4$C can be indicated as commonly used ceramic reinforced phases for aluminum matrix composites (AMMCs). Aluminum oxide (Al$_2$O$_3$) and titanium oxide (TiO$_2$) make up a primarily inherent reinforcement material for aluminum matrix composites since they effectively combine physical and mechanical properties such as high melting point, high hardness, low density, high elastic modulus, and outstanding wear resistance [4–7].

Many processing methods have been developed...
for Al metal matrix composites. Previous works have focused primarily on Al metal matrix composites (AMMC) fabricated by stirring casting [8, 9], melt infiltration [10], powder metallurgy [11, 12], and spray deposition [12, 13]. Uniform distribution of reinforcement particles in the matrix, higher mechanical properties, minimum undesirable interfacial reactions, and the low-cost processing method can be attained through powder metallurgy (PM) [11]. The PM approach has the benefit of reducing the interaction among the ceramic reinforcement and the matrix through semisolid and/or solid-state processing. It also has various other advantages: its capacity to combine various matrix reinforcements in the same composite and achieve high reinforcement volume fractions [14].

PM was used in the present study to synthesize Al metal matrix composite (AMMC) using Al₂O₃-TiO₂ reinforcement, after which the impacts of the Al₂O₃-TiO₂ content on structural and mechanical properties were examined. The objective of the present study was to establish a basis for the processing of high-performance AMMC reinforced by ceramic particles using powder metallurgy.

## 2. Experimental

Aluminum (Al) with a particle size smaller than 40 μm (99.99 % purity, Acros Organics) and Al₂O₃-TiO₂ composite powder with a particle size of 30.6 μm (99.99 % purity, Metco) were used in the study as starting materials. The powders of aluminum (Al) and alumina-titanium oxide (Al₂O₃-TiO₂) were first characterized for compositions using wavelength dispersive X-ray fluorescence (XRF) (Panalytical/Axios MAX), the results of which are presented in Table 1. Composite samples were produced with different weight contents of Al₂O₃-TiO₂: 5, 10, 15, and 20 wt.%. Required amounts of Al and Al₂O₃-TiO₂ powders were mixed and ball milled for 1 h in alumina pot using alumina ball as grinding media at the speed of 300 rpm min⁻¹ to achieve a specific weight content of a composite sample. During the mixing process, ~35 wt.% ethanol was added to decrease the possibility of agglomerate formation. The dried powder was pressed in a die under a pressure of 2 tons for preparing the pellets. The pressed samples were sintered further in a tube furnace at 800 °C in an argon atmosphere. The samples were heated from room temperature up to 800 °C at a rate of 5 °C min⁻¹, then sintered at that temperature for 4 h, and cooled back naturally to room temperature.

Archimedes’ method was used for determining the density of sintered composites. Phase characterization was performed via X-ray diffraction (XRD) using a Panalytical, empyrean brand model X-ray diffractometer with a Cu Kα radiation source operating at 40 kV and 40 mA. The microstructures and phase compositions were examined via scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) (FEI Nova NanoSEM 650). Vickers hardness experiments were performed to determine the hardness of the sintered samples. Sintered pure Al and composite samples were placed inside a mold with epoxy resin and sanded with SiC paper. They were polished with a diamond paste to obtain a correct measurement of the indentation length created before the hardness test. The tensile properties of the composites at room temperature were determined using an Instron material-testing machine (INSTRON, 50 kN) at a cross-head speed of 0.5 mm s⁻¹. The ultimate tensile strength of the sample was calculated using three bars test samples 75 mm × 5 mm × 55 mm in size.

## 3. Results and discussion

### 3.1. Structural analysis

The aluminum powder used has high purity as shown by X-ray fluorescence (XRF) data (Table 1) and X-ray diffraction (XRD) peak (Fig. 1) exhibited at 2θ values of 38.3°, 44.7°, 65.0°, and 78.1° corresponding to d₁₁₁, d₂₀₀, d₂₂₀, and d₃₁₁ planes of aluminum, respectively. Figure 1 shows no peak from other elements, which shows the purity of aluminum and alumina-titanium oxide powder. XRD of alumina displayed peaks at 2θ angles of 25.6°, 36.2°, 43.2°, 52.5°, 57.4°, 66.2°, and 68.1°. XRD of titanium oxide depicted peaks at 2θ angles of 25.4°, 37.6°, 49.3°, 53.6°, 54.5°, 59.6°, 62.4°, 63.4°, and 65.9°.

XRD spectra illustrate that the peak intensity

| Table 1. Composition of Al and Al₂O₃-TiO₂ powder from XRF data (wt.%) |
|-------------------|---|---|---|---|---|---|---|---|---|
| Aluminum powder   | Al | C  | Fe | Si | Cu | S  | Co | Zn | Mn | Loss of temp. |
| Al₂O₃-TiO₂ powder | Na₂O| MgO| Al₂O₃ | SiO₂ | SO₃ | K₂O| TiO₂| Fe₂O₃| ZrO₂| Loss of temp. |
|                   | 99.99| 0.01| 84.00| 0.21| 0.05| 0.02| 14.15| 0.20| 0.46| 0.38 |

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corresponding to aluminum (Al) decreases while the corresponding intensities of the peak associated with 
$\text{Al}_2\text{O}_3$ and TiO$_2$ increase slightly with the decrease of the weight fraction of aluminum. XRD of alumina depicted major peaks at 2$\theta$ angles of 25.6°, 36.2°, 43.2°, and 57.4°. JCPDS card no. of this phase is 98-0006-5594. Alumina phase has been observed in similar studies in the literature involving Al/$\text{Al}_2\text{O}_3$ [15]. XRD of titanium oxide depicted major peaks at 2$\theta$ angles of 25.4°, 53.6°, and 54.5°. The experimental XRD pattern is in accordance with the JCPDS card no. 98-000-9852 (Anatase, TiO$_2$) and the 2$\theta$ at peak 25.4° affirms the TiO$_2$ anatase structure [14]. XRD of the compound containing aluminum titanium intermediate phase (Al$_5$Ti$_2$) depicted major peaks at 2$\theta$ angles of 39.2° and 46.8°. JCPDS card no. of this phase is 98-010-6255. It is observed in studies involving Al/$\text{Al}_2\text{O}_3$-TiO$_2$ in the literature that intermediate phases containing aluminum titanium have been formed [16].

In our work, after the treatment of the sample at 800°C, growth mechanism of the intermetallic zone in Al-TiO$_2$ liquid-solid interface changes following the
phase transition of Ti, from being dominated by the dissolution rate of TiO₂ in molten Al to that by the chemical reaction speed of the atoms of Al and Ti. It has been proven that among all Ti-Al compounds, Al₁₃Ti, Al-Ti, and Al₅Ti₃ can be directly formed by the reaction between Ti and Al, while Al₂Ti and Al₅Ti₂ can be formed only through a series of intermediate reactions [15]. Researchers investigated the phase diagram of the Al-Ti-O system. Among all the reported isothermal sections of the Ti-Al-O system, reported phase diagram evaluations (isothermal sections) have usually been carried out at 945, 900, 871, 800, 700, and 500°C [17–20]. Usually, ceramic particles are difficult to react with molten Al. Maity et al. [15] reported that though the reactive oxide particles’ wetting should be better, up to only 3 wt.% TiO₂ and 20 vol.% Al₂O₃ could be incorporated into the molten Al. When we look at stoichiometry, approximately the same amounts of TiO₂ and Al₂O₃ were used in our study.

SEM images of composite samples of Al/Al₂O₃-TiO₂ at different compositions display the varying weight percentage of Al₂O₃-TiO₂ particles (Fig. 2). The presence of ceramic particles inside the aluminum metal matrix is proven with the micro-level examination. The microstructure of all composites consists of rounded Al₂O₃ and TiO₂ grains, scattered both intragranularly and intergranularly. Intergranular Al₂O₃ and TiO₂ grains also appeared as agglomerates with pores between them.

Figure 2 displays the even distribution of reinforcing particles (Al₂O₃ and TiO₂) within the Al matrix, which was discerned using a scanning electron microscope when the weight percentage of the particles increased in Al/Al₂O₃-TiO₂. It is apparent that some visible clusters are found. Moreover, particle distribution is found to be almost homogeneous. As mentioned in similar previous works by the authors, the formation of agglomeration can be ascribed to the mixing conditions and/or sintering process [14, 22]. Kang and Chan [21] put forth that agglomeration can contribute to the composites’ strengthening if the agglomeration appears to be well bonded to the matrix.

The weight percentage and distribution of ceramic reinforcement in the metallic matrix signifi-

![Fig. 3. SEM image of synthesized Al-20 wt.% Al₂O₃-TiO₂ composite (a) and EDS mapping of titanium (b), aluminum (c), and oxygen (d).](image-url)
Fig. 4. EDX spectrum and elemental mapping of Al-20 wt.% Al₂O₃-TiO₂ composite.
Figure 5 presents the density (theoretical and experimental) values for different compositions of Al/Al₂O₃-TiO₂. It can be stated based on the studies in the literature that Al₂O₃ and TiO₂ reinforcements have lower density due to the previously indicated cluster formation during pressing and the need for extra pressure to enable softer metal particles for filling the pores between hard ceramic particles [23]. It can be stated based on Fig. 5 that both theoretical and experimental densities are in good agreement with each other, thus affirming the suitability powder metallurgy technique.

### 3.2. Mechanical characterization

Vickers hardness values of the sintered pure Al and Al/Al₂O₃-TiO₂ composite samples were calculated using at least five successive indentations for each sample by a Duromine-M Hardness Tester under a load of 1 kg for 15 s.

Vickers hardness for pure aluminum (Al) and the variation of Vickers hardness for Al/Al₂O₃-TiO₂ composites are shown in Fig. 6. It has also been noted that when wt.% of Al₂O₃-TiO₂ increases up to 10 wt.%, there is a slight increase in hardness value of Al/Al₂O₃-TiO₂ composite. This low hardness value is due to the near-uniform dispersion of reinforcement in the metal matrix phase and the cluster formation, which leads to porosity (Fig. 2). It can be seen that the hardness of Al/Al₂O₃-TiO₂ composite increases with an increase in the number of Al₂O₃-TiO₂ particles; maximum hardness was 56 HV, and it was due to the sample containing 20 wt.% of Al₂O₃-TiO₂ particles. This may be related to particles hindering matrix dislocation movement in the composite, thus leading to increased strength and hardness. Maity et al. [15] suggested that the presence of alumina particles can increase the hardness of the Al alloy. Shelvavit et al. [23] have stated that reinforcing particles play an important role by sharing a main part of the contact load and support contact stresses for preventing plastic deformation, subsequently increasing the hardness of the composite. Das et al. [25] reported that the hardness of aluminum-based composites increased with increasing weight fraction reinforcements. With the increase in reinforcement weight content, the reinforcement-to-matrix ratio becomes richer, which provides increased hardness to the composite.

Figure 7 shows the tensile stress-strain curves for Al and Al/Al₂O₃-TiO₂ composites with 5, 10, 15, and 20 wt.% of alumina-titanium oxide. Adding Al₂O₃ and TiO₂ reinforcing particles increases the tensile strength of aluminum matrix composite and keeps growing due to dispersion hardening. The composite strength is increased further by enhancing the bonding or relationship of liquid aluminum with Al₂O₃ and TiO₂ particles. Tensile strength increased significantly...
Table 2. Mechanical properties of various Al-MMC<sub>4</sub> composites

| Reinforcement materials | Sintering temperature (°C) | Hardness | Strength σ (MPa) | UTS (MPa) | References |
|-------------------------|-----------------------------|----------|-----------------|-----------|------------|
| Al/Fly ash, Al/aloe, and Al | 720, stir casting method | 28.2 and 33.8 BHN | 53.36 and 62.97 (yield strength) | 104.21 and 119.83 | [2] |
| 7075Al/TiB<sub>2</sub> | LMD process | 1.97 GPA nanohardness | 734 (comp. strength) | – | [3] |
| Al/Fe-based metallic glass | 450/640 MPa via hot pressing | – | 218 (yield strength) | 374 | [10] |
| Al/Al<sub>2</sub>O<sub>3</sub> | 620 (sintered) | 45.6 HRC (%1) | ≥ 130 (%1) | – | [21] |
| | 420 (extruded) | 68.4 HRC (%7) | ≥ 180 (%7) (yield strength) | – | |
| | 350 (annealed) | – | – | – | |
| TiB<sub>2</sub> | 650, muffle furnace | ≥ 70 HV (%5) | – | – | [23] |
| Al/graphene | 1400 | 57 HV | – | ~ 120 | [27] |
| Al/TiO<sub>2</sub> | 700, stir casting method | 104.6 BHN (%2), 124 BHN (%8) | – | ≥ 175 (%2), and ≥ 225 (%8) tensile strength | [28] |
| Al/VC | 600 (conventional sintering) | 232 ± 16 HV | 295 (bending strength) | – | [29] |
| | 600 (microwave sintering) | – | – | – | |
| | 450 (SPS) | – | – | – | |

following the addition of 20 wt.% Al<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub>. A comparison was made between the mechanical properties of the composites obtained and those found from the related literature for dense Al/Al<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub> materials obtained through different processes (Table 2). This finding is in good agreement with previously published results [3, 8, 12, 21, 23–25]. Also, the reasons for lower values of hardness and tensile strength can be explained as follows:

The micrograph of some clusters in the Al matrix and porosity due to cluster formation indicates that the TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> particles are surrounded by aluminum particles. This is related to the near-uniform dispersion of reinforcement in the metal matrix phase or the tendency of smaller particles to form cluster pressing, which overall contributes to increased material porosity and poor mechanical properties. Also, the lack of interfacial strength, partial wetting of particles, and the presence of constituent particles at the interface adversely affect its mechanical properties [15, 21].

Figure 7 shows that the higher the weight percentage of reinforcements in the matrix, the lower the toughness value is. This is due to obstacles to the motion of dislocation. Micron-sized particles are generally used for enhancing the metal tensile properties [25]. However, increasing ceramic reinforcement weight fractions lead to a significant decrease in the ductility of the composite.

It was observed that satisfactory results obtained mechanical properties (Figs. 6 and 7) of Al/Al<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub> composites produced via powder metallurgy specimens sintered at 800°C compared with those of the samples from similar studies in the literature (Table 2). The values we have achieved are close to those of similar composite materials studied in the literature.

Ghasali et al. [24] demonstrated that the formation of Al<sub>3</sub>Ti phases positively impacts the final mechanical properties of the aluminum matrix composite. Milman et al. [26] pointed out that Al<sub>3</sub>Ti compounds act as an useful reinforcement for aluminum alloys due to significant properties such as low density, high hardness, good high-temperature properties. Based on
phase analysis, it seems that a partial reaction occurs between Al and TiO\textsubscript{2} resulting in the formation of a small amount of Al\textsubscript{5}Ti\textsubscript{2}. Hence, the previously mentioned phase is considered to have the same effect as the Al\textsubscript{5}Ti phase.

4. Conclusions

Al\textsubscript{2}O\textsubscript{3}-TiO\textsubscript{2} composite samples used in the present study were fabricated with 5–20 wt.% of Al\textsubscript{2}O\textsubscript{3}-TiO\textsubscript{2} through milling, compacting, and sintering processes. Afterward, the structural (phase composition, microstructure, relative density), mechanical (tensile strength, hardness) properties of the composites were characterized. Aluminum matrix composite reinforced with alumina-titanium oxide (Al\textsubscript{2}O\textsubscript{3}-TiO\textsubscript{2}) was produced successfully during the current study by implementing powder metallurgy processing methods. The following conclusions were achieved:

- The diffraction peaks of Al, Al\textsubscript{2}O\textsubscript{3}, and TiO\textsubscript{2} phases were observed. The Al phase in the XRD patterns was represented by sharp peaks, and minor peaks indicated the presence of Al\textsubscript{2}O\textsubscript{3} and TiO\textsubscript{2} particles. The XRD results were also in accordance with the EDS results, which attests to the fabrication of phase pure different ceramic-reinforced Al-composites.
- The densities of the developed composite were in accordance with the theoretical values and displayed a near-uniform distribution of reinforcement particles. Samples with 20 wt.% of Al\textsubscript{2}O\textsubscript{3}-TiO\textsubscript{2} particles had a higher relative density of Al/Al\textsubscript{2}O\textsubscript{3}-TiO\textsubscript{2} composite. The highest relative density of 96% was observed in specimens sintered at 800°C.
- The tensile strength of composites at room temperature increases along with an increase in the Al\textsubscript{2}O\textsubscript{3}-TiO\textsubscript{2} composite powder’s weight fraction from 5 to 20 wt.% in the Al matrix. Low inherent ductility of the ceramic particles used as reinforcement resulted in low ductility in the produced Al/Al\textsubscript{2}O\textsubscript{3}-TiO\textsubscript{2} composites compared to pure Al. Minimum and maximum tensile strength values for the composite samples were observed as 84 and 150 MPa, respectively. The low porosity level and the good distribution of ceramic particulates led to a good enhancement in tensile strength.
- The weight ratio of reinforcement-to-matrix increased further with increased reinforcement content resulting in increased hardness in the composite.
- Microstructure characterization and SEM micrographs of the composites revealed a fairly near-uniform distribution of Al\textsubscript{2}O\textsubscript{3} and TiO\textsubscript{2} particulates. Some unreacted TiO\textsubscript{2} clusters and discrete Al\textsubscript{2}O\textsubscript{3} particles were observed in the matrix.

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