



# Effective two-step method for producing Ti–6Al–4V alloy particles with various morphologies



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## ABSTRACT

Magnesium was used as a reductant for the direct combustion synthesis of titanium alloy particles (Ti–6Al–4V) from titanium oxide (TiO<sub>2</sub>). The feed material (TiO<sub>2</sub>) and reductant (Mg) were mixed with Al (or AlCl<sub>3</sub> · 6H<sub>2</sub>O), vanadium pentoxide (V<sub>2</sub>O<sub>5</sub>), and calcium hydroxide (Ca(OH)<sub>2</sub>), and the reaction mixture was combusted under pressurized argon. Temperature–time profiles were intermittently measured during combustion to analyze the combustion temperatures and wave velocities. The combustion product was subsequently acid-leached to eliminate secondary phases (MgO, MgCl<sub>2</sub>, CaO) and produce Ti–6Al–4V alloy particles whose oxygen content was ~1.3%. Further the content of oxygen was decreased to 0.2 wt.% by deoxidizing the Ti–6Al–4V alloy particles with calcium in the range 850–900 °C. The AlCl<sub>3</sub> · 6H<sub>2</sub>O affected the particle morphology more than the Al metal did; i.e. the Al-doped alloy particles were porous and consisted of relatively small agglomerated particles whereas using AlCl<sub>3</sub> · 6H<sub>2</sub>O produced dense, round particles. Pilot-scale production experiments revealed that Ti–6Al–4V alloy particles can be efficiently produced using our method.

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## 1. Introduction

Titanium-based materials are widely used in aerospace, automotive, biomedical, and marine applications because of their superior properties, such as light weight, excellent mechanical strength, and good corrosion resistance. Ti–6Al–4V (Ti64) is the alloy most commonly applied in many industrial sectors, but its application is limited because of the high material and processing costs.

There are two basic methods of producing titanium alloy particles: the blended elemental method and the prealloyed method [1–5]. The former essentially involves the use of pure titanium and a master alloy (e.g., 60:40 Al:V) to produce alloys such as Ti64. In the prealloyed method, the alloying additives are already in the material before the alloy particles are produced. Alloy particles produced using the blended elemental method are cheaper than those produced using the prealloyed method but have inferior mechanical properties. In contrast, alloy particles produced using the prealloyed method are relatively expensive but show superior mechanical properties and hence can be readily cast and wrought (such as in ingot metallurgy) [1,2].

The main source of the titanium powder used in blended elemental powder metallurgy (P/M) is titanium fines [1–5], a byproduct of the Kroll process [6–8]. However, titanium sponge fines typically contain 0.12–0.15 wt.% chloride, which prevents the production of dense

compacts of 100% Ti. Commercially pure titanium powders whose chloride contents are <10 ppm can be obtained by crushing and subsequently dehydrogenating (i.e., hydrogenating–dehydrogenating; HDH) hydrogenated Ti ingots. Prealloyed powders can be produced from alloys such as Ti64 by melting prealloyed stock and rotating an electrode (i.e., the plasma rotating-electrode process) or by gas atomization [1–5,8].

The aim of this paper is presentation of the process of forming Ti–6Al–4V alloy particles with various morphologies by a solid combustion process. We used TiO<sub>2</sub> as the source material, Mg as the reductant, and Al (or AlCl<sub>3</sub> · 6H<sub>2</sub>O) and V<sub>2</sub>O<sub>5</sub> as dopants. Ca(OH)<sub>2</sub> was experimentally selected as an additive to effectively decrease the content of oxygen in the alloy powder. Our method is simpler and faster than conventional commercial methods such as the prealloyed method, so it shows promise to decrease production costs and can be applied to produce a wide range of titanium alloy particles.

## 2. Experimental

Titanium dioxide rutile powder (TiO<sub>2</sub>, 99% pure, particle diameter: 0.1–0.3 μm, Millennium, USA), magnesium powder (Mg, 99% pure, particle diameter: 50–300 μm, Samchun Pure Chemical Co., Ltd., South Korea), aluminum powder (Al, 99.5% pure, particle diameter: 7–15 μm, Alfa Aesar, USA), aluminum chloride hexahydrate (AlCl<sub>3</sub> · 6H<sub>2</sub>O, chemical-grade powder), vanadium pentoxide powder (V<sub>2</sub>O<sub>5</sub>, 98.5% pure, particle diameter: 1–10 μm, Chameleon Chemical Reagent, Japan), calcium hydroxide powder (Ca(OH)<sub>2</sub>, 98% pure,

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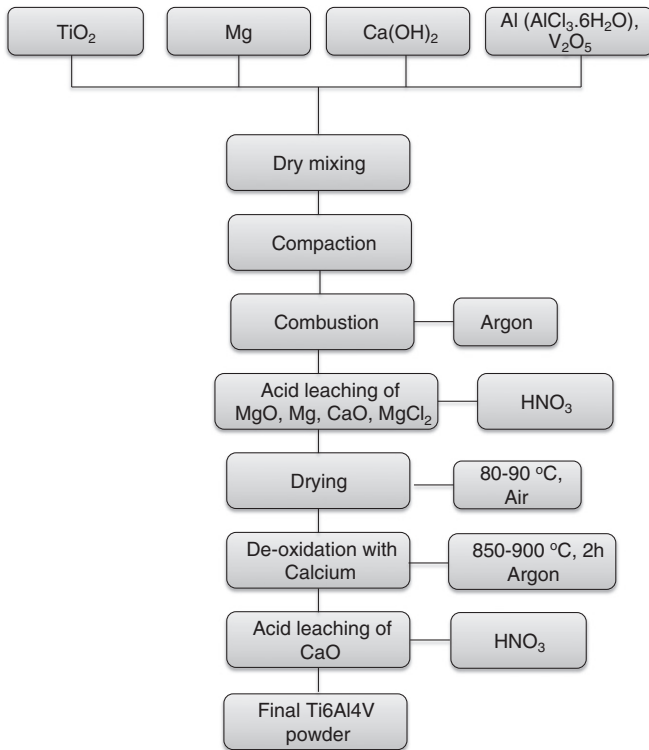
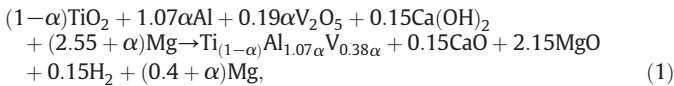


Fig. 1. Process flow diagram.

particle diameter: <math><5\text{--}50\ \mu\text{m}</math>, Samchun Pure Chemical Co., Ltd.), and granular calcium (Ca, 99.5% pure, granule diameter: 2–5 mm, Junsei Chemical Co., Ltd., Japan) were used for the combustion synthesis of Ti64 alloy particles with various morphologies.

Ti64 alloy particles were prepared by a two-stage process involving combustion synthesis of Ti64 powders and subsequent deoxidation with calcium at a high temperature. The chemical equation for synthesizing Ti alloy particles is as follows:



where  $\alpha$  is a coefficient that shows the change in the concentration of dopants (Al and  $\text{V}_2\text{O}_5$ ) and Mg in moles. According to the given equation, the pure titanium, Ti64 alloy and the Ti12Al8V alloy can be

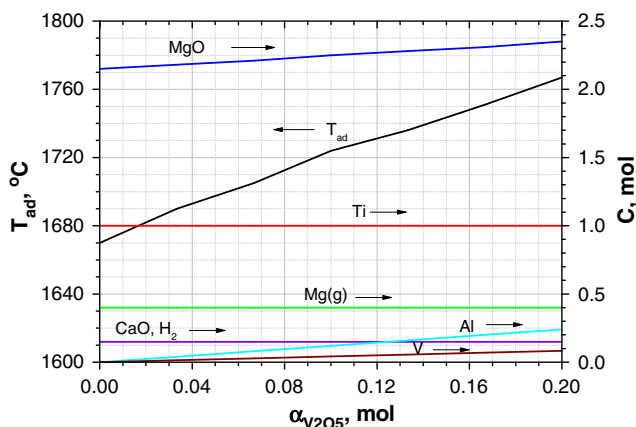


Fig. 2. Adiabatic combustion temperature and equilibrium phases in the  $(1-\alpha)\text{TiO}_2 + 1.07\alpha\text{Al} + 0.19\alpha\text{V}_2\text{O}_5 + 0.15\text{Ca}(\text{OH})_2 + (2.55 + \alpha)\text{Mg}$  system.

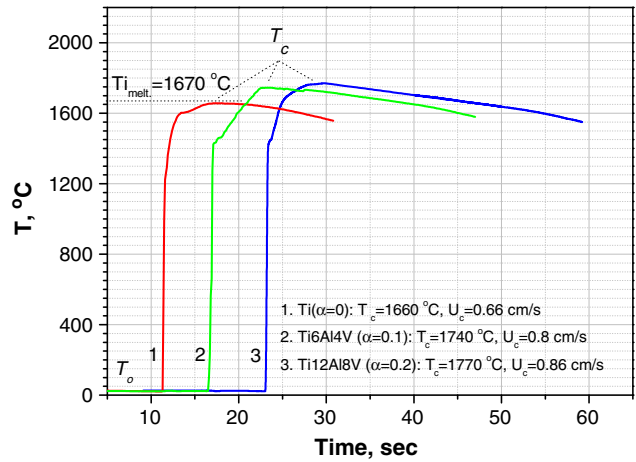


Fig. 3. Temperature–time profiles for the  $(1-\alpha)\text{TiO}_2 + 1.07\alpha\text{Al} + 0.19\alpha\text{V}_2\text{O}_5 + 0.15\text{Ca}(\text{OH})_2 + (2.55 + \alpha)\text{Mg}$  system: (1)  $\alpha = 0$ ; (2)  $\alpha = 0.1$ ; and (3)  $\alpha = 0.2$ .

synthesized at  $\alpha = 0, 0.1$ , and  $0.2$ , respectively. The optimal  $\text{Ca}(\text{OH})_2$  concentration was experimentally determined as  $0.15\ \text{mol}$ . The Al powder was replaced with  $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$  in some experiments to demonstrate the effect of Al source on the morphology of the Ti64 particles.

The flow diagram for the entire process is shown in Fig. 1. Approximately 280–300 g of the mixture was hand-compacted into a 6.0-cm-diameter, 10–12-cm-high stainless steel cup for the combustion experiment. Two 50- $\mu\text{m}$ -diameter,  $\Lambda$ -shaped tungsten–rhenium thermocouples (WR-26/WR-5) were placed inside the sample near the center during compaction. The thermocouples had been precoated with a thin layer of  $\text{Al}_2\text{O}_3$  and were placed into thin ceramic pipes to increase their resistance to oxidation and to prevent any reaction between the thermocouples and the powder bed at elevated temperatures. Approximately 3–5 g of an exothermic mixture composed of  $\text{Ti} + 0.9\text{C}$  (black soot) +  $0.1[(\text{C}_2\text{F}_4)_n\text{-Teflon}^\circledast]$  was used as an agent to easily ignite the reaction sample and was placed on top of it. The cup containing the reaction mixture and thermocouples was subsequently placed under a nickel/chromium coil in a combustion chamber. The chamber was then evacuated and filled with argon to 2.0 MPa. A nickel–chromium filament electrically heated to 900–1000 °C was used to locally ignite the reaction sample within 1–2 s. A computer-assisted data logger (GL100A, Graphtec Co., Japan) continuously recorded the temperature–time history of the process at 10 Hz. The combustion velocity was calculated as  $U_c = x/t$ , where  $x$  represents the physical distance between the thermocouples, and  $t$

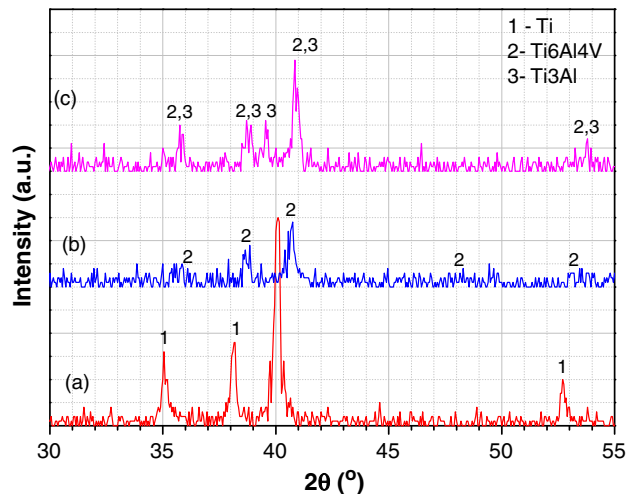


Fig. 4. XRD patterns of acid-leached products produced for  $\alpha$ : (a) 0; (b) 0.1; (c) 0.2.

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