***In situ* Al₃Ti–Al₂O₃ intermetallic matrix composite: Synthesis, microstructure, and compressive behavior**

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A fully dense *in situ* Al₃Ti–Al₂O₃ intermetallic matrix composite containing about 30 vol% Al₂O₃ particles was prepared by combining squeeze casting with combustion synthesis using the chemical reaction between TiO₂ and Al. The microstructure of the *in situ* composite was examined using x-ray diffraction, scanning electron microscopy, and transmission electron microscopy techniques. Compressive behavior of the composite was investigated in the temperature range of 25–600 °C and compared with that of the as-cast Al₃Ti alloy. The *in situ* formed spherical α -Al₂O₃ particles with a size of 0.2–1 μm were uniformly distributed in the Al₃Ti matrix. The grain size of the Al₃Ti matrix containing a small amount of Al₂Ti precipitate was 2–10 μm . The compressive strength of the *in situ* composite was about 6–9 times that of the as-cast monolithic Al₃Ti alloy and could be maintained at temperatures up to 600 °C. This was mainly attributed to the fine grain size of Al₃Ti matrix and the rule of mixture strengthening of Al₂O₃ particles. The existence of Al₂Ti phase and high dislocation density in the matrix also contributed positively to the composite strength.

I. INTRODUCTION

Much attention has been paid to the development and application of metal-matrix composites and intermetallic matrix composites.¹ Traditionally, they have been produced by such processing techniques as powder metallurgy,² rapid solidification,³ and diverse casting techniques (e.g., squeeze casting).¹ In all these techniques, the reinforcing phase (Al₂O₃ particulate for instance) is first mixed with the matrix materials. The scale of the reinforcing phase is consequently constrained by the starting powder size, which is typically of the order of a few microns to a few tens of microns and rarely below 1 μm .¹

In the last ten years, new *in situ* processing technologies for fabricating metal and ceramic composites have emerged. *In situ* techniques use chemical reactions for the formation of the reinforcement. These technologies include self-propagating high-temperature synthesis (SHS), direct metal oxidation method (DIMOXTM), exothermic dispersion (XDTM), mechanical alloying,^{4–9} and reactive infiltration^{10–13} or reaction powder metallurgy.^{14,15} Because of the fineness and thermodynamic stability of the reinforcing phase, it is expected that *in situ* composites should offer excellent dispersion of

fine reinforcing particles and nascent interface, resulting in better mechanical properties and high-temperature performance.

Another advantage of *in situ* techniques is their capability for processing materials or composites that could be difficult to obtain by other conventional methods, such as Al–Ti-based composites.^{11,16} Al₃Ti has a lower density and better oxidation resistance than other Al–Ti intermetallic compounds. Such attractive characteristics also make Al₃Ti a potential candidate for low-density, high-temperature structural materials. But due to its low cleavage strength, its application is so far limited. One way to improve the strength is to introduce a reinforcing phase into the Al₃Ti matrix. Unfortunately, there have been few reports of success.¹¹

Fukunaga and Wang¹⁰ fabricated *in situ* composites by reactive melt infiltration of TiO₂ whisker preform with molten Al. However, the microstructure of the composite was nonuniform and the Vickers hardness varied between 30 and 1050 in the composites. Wagner *et al.*¹¹ reported an interpenetrating Al₂O₃–Al₃Ti composite by squeeze casting of Al into TiO₂-containing Al₂O₃ preforms and subsequent annealing for 4 h in air at 640 °C to achieve complete reaction. Wang *et al.*¹⁶ prepared

TiO₂/Al mixtures by squeeze casting and subsequent sintering at 810 and 910 °C to produce Al₂O₃/Ti_xAl_y *in situ* composites. An incomplete reaction was experienced in these works.

In a related field, the conventional combustion synthesis or SHS method can produce high-purity products but with high porosity.¹⁷ The relatively high porosity (30 ~ 70%) in the products is mainly due to (i) the low green density of the reactant mixture, and (ii) the intrinsic volume changes between the reactants and products in the combustion synthesis reaction. If the green density of the reactant mixture is increased, the relative density of the products is expected to improve. But for the powder-SHS process, it is difficult to achieve full density in the reactant mixture. Therefore, consolidation techniques, such as hot isostatic pressing (HIP) and hot extrusion, have to be employed to increase the relative density of the final products. Although these techniques can provide high relative density, they do incur an economic penalty.¹⁷

In this paper, a fully dense Al₃Ti–Al₂O₃ *in situ* composite was synthesized by reaction between TiO₂ powder and molten Al via a squeeze casting route. The method combines the combustion synthesis and the consolidation steps into a one-step process.

II. EXPERIMENTAL DETAILS

A. Materials synthesis

The anatase TiO₂ powder (manufactured by Shenyang Fifth Chemicals Products Ltd., Shenyang, China) with an average diameter of 0.8 μm and pure Al (supplied by Northern-East Light-Alloys Ltd., Harbin, China) with a purity of 99.7% were selected as the raw materials. The maximum contents of impurities in the as-received TiO₂ powder are listed in Table I.

TiO₂/Al billets with controlled volume fraction of TiO₂ (determined according to the reaction formula presented in Sec. III. A) were prepared by a squeeze casting method.¹⁰ The dried and dispersed TiO₂ powder was directly filled into the steel die with an inner diameter of 80 mm to which a graphite disk with good permeability was tightly fitted. Then the die together with the TiO₂ powder were shaken to make the preform with predetermined volume fraction while a steel ram was placed on the top of the powder. The molten aluminium was then

introduced onto the preheated preform through a channel. A ram was activated by a 100-ton hydraulic press to force the melt into the preform. After the infiltration process a maximum pressure of 55 MPa was maintained until the die cooled and the aluminium phase was solidified. The typical temperatures of molten aluminium (T_m), preform (T_p), and die (T_d) were 820, 630 and 630 °C, respectively.

Subsequently, the squeeze-cast TiO₂/Al samples were heated to about 820 °C in a proper melt bath in a furnace to avoid the possible negative effects of the air to produce the final Al₃Ti–Al₂O₃ composite. The reaction process was monitored through an observation window purposely designed on the furnace. Once the reaction finished, the sample was taken out and cooled to room temperature.

In order to judge the heat-treatment schedules and characterize the thermal characteristic of the reaction between TiO₂ and Al, differential thermal analysis (DTA) was conducted on as-infiltrated samples. A Jeol TAS-100 (Japan) analyzer was used with a heating rate of 10 °C/min.

B. Microstructural characterization

The phase composition of the reaction product was examined by x-ray diffraction (XRD). Using scanning electron microscopy (SEM) and transmission electron microscopy (TEM) technique, the microstructure of the composites was investigated in detail. The TEM foils were prepared by a conventional procedure, which involved cutting disks of 3 mm in diameter, mechanically polishing them to 50 μm, dimpling to 20 μm, and finally ion-beam milling to performance. SEM and TEM observations were carried out on Philips S-570 and EM-420 devices, respectively. Phases are identified by selected area diffraction technique combining the sample tilt.

C. Hardness and compression tests

The hardness of the composite was measured using an Hv-50 microindenter with a load of 5 kg and a dwell time of 10 s. Compression tests were conducted on a Gleeble 1500 test machine at a strain rate of $4.2 \times 10^{-4} \text{ s}^{-1}$ to failure in most cases. The specimens with a diameter of 9 mm and length of 20 mm were prepared by spark machining and subsequently ground to ensure parallel surfaces. Each data point is an average of at least three samples. The composite density was determined by a water immersion technique.

III. RESULTS AND DISCUSSION

A. Thermodynamic analysis

The standard free energy of formation (ΔG_T) of TiO₂ and Al₂O₃ were obtained from thermodynamic data available in the literature¹⁸ and were used to determine

TABLE I. Maximum contents of impurities in as-received TiO₂ powder.

Impurity	Maximum content (wt%)
Nondissolved in HCl	0.3
Sulphate	0.2
Fe	0.01
Heavy metals	0.005
As	0.0007

the free-energy change of the reaction between TiO₂ and Al to form Al₂O₃. The calculated results indicate that the following reaction is possible in a large range of temperatures:

In addition, the thermodynamic analysis also revealed that in the presence of excess Al, the following reaction is possible:

This gives the following overall reaction:

and ΔG for Reaction (3) is negative, $\Delta G < 0$.

Based on the above reaction formulae, the enthalpy of Reactions (1) and (3) can be calculated as follows:

$$\Delta H_T = \Delta H_{298} + \sum n_i (H_T - H_{298})_{Pi} - \sum n_i (H_T - H_{298})_{Ri} \quad (4)$$

where the subscripts *P* and *R* represent product and reactant, respectively. The values of enthalpy *H* at different temperatures are available in the literature,¹⁸ which already include the phase transformation enthalpy. The calculated enthalpies (ΔH_T) of Reaction (1) and Reaction (3) at different temperatures are shown in Table II, which indicate that Reactions (1) and (3) are exothermic. The theoretical adiabatic temperatures (*Tad*) of these reactions can also be calculated by assuming the whole system is adiabatic. Supposing the reactants are at a preheating temperature *T*₀, *Tad* can be worked out from the following equation:

$$\sum n_i (H_{Tad} - H_{298})_{Pi} = \sum n_i (H_{T_0} - H_{298})_{Ri} - \Delta H_{298} \quad (5)$$

In the case of an initial temperature *T*₀ = 1200 K, the calculated theoretical adiabatic temperature of Reaction (1) (*Tad*) is 2300 K. This temperature is slightly lower than the melting point of Al₂O₃ (2303 K), but much higher than those of Al (932 K) and Ti (1933 K). The calculation of *Tad* for Reaction (3) is limited by the availability of thermochemical data of Al₃Ti at different temperatures, which are listed only up to 1613 K (the melting point of Al₃Ti).¹⁸ However, the calculation by

TABLE II. The calculated enthalpies (ΔH_T) of Reactions (1) and (3) at different temperatures (*T*).

<i>T</i> (K)	ΔH_T (kcal/mol)	
	Reaction (1)	Reaction (3)
800	−44.28	−78.43
900	−44.35	−78.70
1000	−47.87	−90.23
1100	−47.85	−90.40
1200	−46.86	−90.52

error method indicated that the *Tad* for reaction (3) is much higher than 1613 K. Under some conditions, when *Tad* is relatively lower, the reaction is liable to be incomplete and thus is commonly activated by thermal means; for example, by preheating the whole sample.¹⁷ Therefore, it is concluded that it is feasible to fabricate Al₃Ti–Al₂O₃ composite through combustion synthesis techniques.

B. The squeeze casting process and synthesis of the composite

In order to obtain Al₃Ti–Al₂O₃ composite, according to the stoichiometry of Reaction (3), the volume ratio of TiO₂ to Al is worked out to be 47:100 from which the volume fraction of TiO₂ (*f*_{TiO₂}) powder was predetermined to be 33–34 vol% during the squeeze casting process. The measured *f*_{TiO₂} was 35 vol% after casting. No chemical reaction occurred throughout the squeeze process in the present work as indicated by the XRD results presented later. According to the reaction kinetics, the reaction can be achieved by increasing the processing temperature, but this is limited by the squeeze casting technique.

Subsequently, DTA was carried out in order to characterize the thermal characteristic of the reaction between TiO₂ and Al. The result is shown in Fig. 1, which indicated that an exothermic reaction occurred in the temperature range of 750–820 °C after aluminium phase

FIG. 1. DTA curve showing the exothermic reaction between TiO₂ and Al.

in the cast block had melted in the temperature range of 660–670 °C. This is in agreement with the previous work.^{16,19} It is important to note that the reaction between TiO₂ and Al did not take place immediately after the melting of aluminium as shown in Fig. 1. Based on these results, the reaction between TiO₂ and Al was achieved by heat treatment.

The squeeze-cast TiO₂/Al blocks were heated in an aluminium bath in a furnace. When the temperature reached 800 °C indicated by the thermocouple, the exothermic reaction usually occurred simultaneously in the whole sample and instantaneously (a few seconds). This is termed the “thermal explosion” mode in the combustion synthesis.¹⁷ The highly exothermic reaction results in high brightness of the composite blocks. Therefore, it is easy to monitor the reaction through an observation window. In fact, no reaction was observed when an as-infiltrated TiO₂/Al sample was kept at a temperature below 700 °C. This is in agreement with the DTA results. After the reaction was completed, the sample was taken out and cooled. Occasionally, the reaction started locally at one side of the sample before propagating through the whole block in a short time. This is similar to the propagation of the combustion wave in SHS mode.^{17,19}

FIG. 2. XRD traces of (a) as-cast TiO₂/Al billet and (b) the final composite.

Figure 2 compares the XRD trace of the as-cast TiO₂/Al block with that of the synthesized composite. No chemical reaction occurred between TiO₂ and Al during the squeeze casting process within the limit of the detection of XRD [Fig. 2(a)]. There were only α -Al₂O₃ and Al₃Ti peaks in the XRD trace of the synthesized composite. Neither Al nor TiO₂ was detected [Fig. 2(b)]. This indicates that the chemical reaction between Al and TiO₂ is complete. According to stoichiometry of Reaction (3) and the densities of Al₂O₃ and Al₃Ti, the volume ratio of α -Al₂O₃ to Al₃Ti in a completely transformed composite will be 1:2.246. So the produced composite is an Al₂O₃-reinforced Al₃Ti matrix composite in which the volume fraction of α -Al₂O₃ is about 0.31.

C. Microstructure and its formation mechanisms

A typical SEM micrograph of the produced composite is shown in Fig. 3. The bright phase is Al₂O₃ particles and the dark phase is Al₃Ti matrix as identified by energy dispersive spectroscopy analysis. The α -Al₂O₃ particles have a fine particle size, usually less than 1 μ m, and a uniform distribution in the Al₃Ti matrix. SEM observations also revealed that the produced composite was very dense, and porosity was not found. The measured density of the composites was 3.56 g/cm³. If the theoretical densities of Al₂O₃ and Al₃Ti are taken as 3.99 g/cm³ and 3.37 g/cm³, respectively, the measured density of the composite corresponds to 99% of the theoretical density.

Figure 4 shows the TEM bright-field images of the synthesized composite. Al₃Ti phase had an equiaxed morphology and a particle size ranging from 2 to 10 μ m, which is much smaller than the grain size of the cast Al₃Ti alloy, which is about 1 mm.²⁰ Some Al₂O₃ particles were dispersed randomly within the Al₃Ti phase

FIG. 3. SEM micrograph showing the typical microstructure of the *in situ* composite.

FIG. 4. TEM bright-field images showing the morphology and locations of Al₂O₃ particles and the dislocations in the synthesized composite.

[Figs. 4(b) and 4(c)]. A high density of dislocations was also observed in the Al₃Ti phase, as shown in Figs. 4(c) and 4(d). These immobile dislocations can result in a higher strength of the matrix. The TEM observations also revealed that the interfaces in the composite are clean and free from any reaction product.

In addition, a phase with plate morphology was found in the Al₃Ti matrix as shown in Fig. 5. Figure 5(b) is an image of Fig. 5(a) after tilting a given angle. This phase was identified as Al₂Ti by selected-area diffraction. This is in good agreement with Wu and Pope's work²¹ where Al₂Ti phase was found to appear in Al–Ti binary systems when the atomic percent of Ti exceeds 26 ~ 27 at%.

In the present work, the measured volume fraction of TiO₂ in the preform is 35%. According to Reaction (1), the 35% TiO₂ together with about 23% Al were consumed to produce the Al₂O₃ and Ti, and about 42% Al left. According to the thermodynamic analysis and the very short duration time of the reaction, both Ti and Al are likely in liquid states to form an Al–Ti binary system in which the content of Ti displaced from Reaction (1) was worked out to be about 30 at%. This gave rise to the existence of the Al₂Ti phase in the Al₃Ti matrix. But due to the relatively small volume fraction, Al₂Ti was not detected by XRD as shown in Fig. 2(b). Investigation of the monolithic Al₂Ti compound indicated that it has a high compressive strength at elevated temperature.²² It is expected that the existence of Al₂Ti phase in Al₃Ti matrix should make a positive contribution to the high compressive strength and high hardness at elevated temperature, which will be discussed in Sec. III. D.

Based on the thermodynamic analysis and the experimental results presented above, we propose the following mechanisms for the reaction synthesis of Al₃Ti–Al₂O₃ *in situ* composite. The reaction between TiO₂ and Al will

FIG. 5. TEM bright-field images of Al₂Ti precipitates; (b) is the bright-field image of (a) after tilting.

produce Al₂O₃ particles and titanium according to Reaction (1). Due to the instant reaction, the temperature of the sample is expected to be near the adiabatic temperature, which is much higher than the melting point of Al₃Ti (1613 K) for either Reaction (1) or Reaction (3). Then, Ti as a reaction product will dissolve into the excess liquid Al to form an Al–Ti solution. During the subsequent cooling process of the Al–Ti solution, Al₃Ti matrix is formed while the Al₂Ti phase forms from the residual liquid phase as suggested by the equilibrium Al–Ti binary diagram.^{23,24} During this process, the distribution of Al₂O₃ particles was established by Reaction (1) and might be altered during the solidification of the Al₃Ti matrix.

D. Compressive behavior and strengthening mechanisms

Figure 6 shows the changes of the microhardness across the transition between the unreacted area (TiO₂/Al) and the reacted area (Al₃Ti–Al₂O₃) in an incomplete SHS sample. To obtain such a sample, a rectangular bar with size of 10 × 15 × 60 mm³ was machined from the cast billet. The reaction was then set in the SHS mode and terminated half way along the sample length by controlling the preheating temperature. The sample was then polished for the hardness measurements. It can be determined that the size of the transition region was about 1 mm. The increase in microhardness is more than H_v 600, indicating that the material was strengthened substantially after the reaction.

Compressive load-displacement curves of the *in situ* composites tested at different temperatures are shown in Fig. 7. The curve of the TiO₂/Al bulk materials in the as-cast conditions is also shown for comparison. In the temperature range explored, the load-displacement curves have a common feature: after yielding, compressive load began to decrease and soon the curves were terminated by fracture. Fracture often preceded yielding

FIG. 6. Microhardness changes from unreacted area to reacted area of the incomplete SHS sample.

FIG. 7. Load versus displacement curves for the composite compressed at different temperatures.

except in the case of TiO₂/Al, as seen in Fig. 7. The observation of such a small fracture strain means that the composite exhibits very low ductility in this temperature range. This is mainly due to the inherent brittleness of Al₃Ti with a DO₂₂-type structure and the presence of a large amount of Al₂O₃ particles. Compression tests showed that a monolithic Al₃Ti alloy has a very small fracture strain, although intensely deformed regions in a microscale were observed in compressed Al₃Ti at room temperature.²⁰

At ambient temperature, there is only one slip system in Al₃Ti along the basal plane²⁰ and this is responsible for the observed low ductility. Study of the compressive deformation of Al₃Ti at 25–860 °C indicated that the main deformation mechanism at low temperatures is twinning of the type (111)[11 $\bar{2}$], which does not disturb the symmetry of the DO₂₂ lattice, resulting in very limited plastic deformation.²⁰ However, at high temperatures (>620 °C), plastic deformation becomes pronounced for a multicrystalline Al₃Ti sample due to the four available (111)[11 $\bar{2}$]-type twinning systems, which are augmented by the slip of the types [110], [010], and [110]. In the present study, due to the existence of a large number of fine Al₂O₃ particles in the Al₃Ti matrix, the slip could be hindered and hence very small fracture strain of the composites were observed.

The compressive yield strength of the synthesized composite is plotted in Fig. 8 as a function of the testing temperatures, and that of the as-cast monolithic Al₃Ti,²⁰ cast Al₂Ti, and powder-processed (PP) Al₂Ti²² are also shown for comparison. The yield stress of Al₃Ti–Al₂O₃ composite increases gradually from 1200 to 1560 MPa with increasing temperature up to about 300 °C and then decreases until about 600 °C. However, the compressive strength is maintained at 645 MPa at 600 °C, which is

FIG. 8. Compressive strength of the composite as a function of temperature. The compressive yield strength of cast Al₃Ti,²⁰ cast Al₂Ti,²² and PP Al₂Ti²² are also shown for comparison.

still much higher than that of the as-cast monolithic Al₃Ti and Al₂Ti alloy. Generally, the strength of the Al₃Ti–Al₂O₃ composite is about six to nine times greater than that of as-cast Al₃Ti alloy and is about twice that of cast Al₂Ti in the temperature range tested. However, the cast Al₂Ti and PP Al₂Ti do possess significantly higher strength than cast Al₃Ti, as shown in Fig. 8. At room temperature, the yield strength of cast Al₂Ti and PP Al₂Ti are 710 and 1360 MPa, respectively, whereas the yield strength of cast Al₃Ti is about 170 MPa. The high strength of Al₂Ti phase may contribute positively to the compressive strength of the composite due to its existence in the Al₃Ti matrix as observed by TEM (Fig. 5).

Based on the microstructural examination and compressive testing results, it is proposed that the high compressive strength of the composite relative to monolithic Al₃Ti is due to a combination of (i) small grain size of Al₃Ti matrix (<10 µm in composite as compared to 1 mm in the cast Al₃Ti), (ii) rule of mixture strengthening introduced by the inherently high compressive strength of alumina, and (iii) the existence of Al₂Ti phase.

IV. CONCLUDING REMARKS

(1) By combining squeeze casting with combustion synthesis, fully dense Al₃Ti–Al₂O₃ *in situ* composite has been fabricated.

(2) The DTA of squeeze-cast TiO₂/Al block indicated that an exothermic reaction occurred at 750 °C. The reaction between TiO₂ and Al is highly exothermic. Based on x-ray analysis, the following reaction occurred to form the final composite during heat treatment of the as-cast TiO₂/Al samples:

(3) Very fine scale and uniform dispersion of the reinforcing Al₂O₃ phase with a particle size of 0.2–1.0 µm was obtained in the Al₃Ti matrix. Al₂Ti precipitates

and high dislocation density were observed in the Al₃Ti matrix.

(4) The *in situ* composite possesses high compressive strength relative to the cast monolithic Al₃Ti at explored temperature range up to 600 °C.

(5) The strengthening mechanisms are a combination of (i) fine grain size of Al₃Ti matrix, (ii) the rule of mixture strengthening, and (iii) the existence of Al₂Ti phase in the Al₃Ti matrix.

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