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Combustion Synthesis of FeAl-based Composites from Thermitic and Intermetallic Reactions

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Abstract: Combustion syntheses involving intermetallic and thermitic reactions were conducted to fabricate FeAl–TiB₂–Al₂O₃ composites. Two combustion systems consisting of Fe, Al, Ti, Fe₂O₃ and B₂O₃ were studied for formation of $x\text{FeAl}-y\text{TiB}_2-\text{Al}_2\text{O}_3$ composites with $x = 1.5-3.5$ and $y = 0.5-0.8$. In the reaction series, thermitic reduction of Fe₂O₃ and B₂O₃ by Al thermally activated the reaction between Fe and Al. As a result, the combustion wave of the synthesis reaction was sufficiently exothermic to propagate in a self-sustaining manner. With an increase in TiB₂ and FeAl of the composites, the decrease of reaction exothermicity resulted in a decline of the combustion wave velocity and reaction temperature. The activation energy $E_a = 88.92$ kJ/mol was deduced for the synergetic combustion reaction. Based on XRD analysis, a thorough phase conversion was achieved and composites composed of FeAl, TiB₂, and Al₂O₃ with different contents were obtained. The SEM micrograph showed the FeAl-based composite with a dense and connecting morphology.

Keywords: combustion synthesis; intermetallic reaction; thermitic reaction; FeAl-based composites; activation energy

1. Introduction

Aluminides of transition metals such as titanium, iron, nickel, and niobium are recognized by their excellent physical and mechanical properties [1–3]. Among these compounds, iron aluminides (FeAl and Fe₃Al) are of considerable interest for high-temperature structural applications, due to their inexpensive raw materials, high melting point, low density, and outstanding resistance to corrosion at elevated temperatures under oxidizing, sulfidizing, and carburizing atmospheres [1,4–6]. Moreover, FeAl exhibits high electrical resistivity, which renders it suitable as a heating component [7]. Porous FeAl intermetallic foams are promising in a hot and aggressive environment like the industrial chimney, where FeAl foams work as a corrosion-resistant gas filter to reduce the emission of smog-forming particles generated from coal and hydrocarbon combustion [8–10]. However, the main drawbacks of iron aluminides are their low ductility and brittle fracture at room temperature and poor wear resistance [4,5]. The addition of ceramic components, such as TiB₂, TiN, TiC, WC, Al₂O₃, and ZrO₂, as reinforcement has proven to effectively improve the room-temperature mechanical properties in nickel and iron aluminides, their wear resistance and high-temperature strength without degrading the inherent oxidation resistance [11–15].

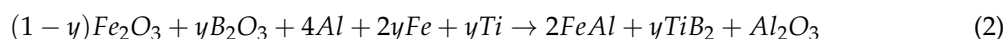
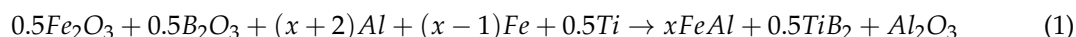
A number of manufacturing methods have been applied to produce ceramics-iron aluminide composites, including mechanochemical synthesis [14], spark plasma sintering [15], hot pressing [16], liquid-phase sintering [17], thermal explosion [18] and self-propagating high-temperature synthesis (SHS) [19,20]. Combustion synthesis in the SHS sense is recognized by its high energy effectiveness, rapid reaction process, easy operation, diversity of products, and in situ formation of composite components [21–23]. When incorporated with a thermitic reaction using Al as the reducing agent,

the thermite-based SHS method is an in situ production route to fabricate Al_2O_3 -reinforced ceramics and intermetallics [24,25]. Moreover, aluminothermic reduction of metal oxides is advantageous to the SHS process in thermodynamic aspects. According to Wang et al. [24], a variety of composite materials with uniformly distributed phases have been simultaneously produced by thermite-based combustion synthesis. The materials include not only ceramic-metal composites (e.g., Al_2O_3 -Cr and Al_2O_3 -Fe), but also Al_2O_3 -added borides (e.g., TiB_2 , VB_2 , MoB , and WB), carbides (e.g., TiC , SiC , B_4C , Cr_3C_2 , and WC) and silicides (e.g., MoSi_2 , ZrSi_2 , and WSi_2).

By means of the classical SHS technique, aluminides of nickel, titanium, and cobalt (e.g., NiAl , Ni_3Al , TiAl , and CoAl) have been produced from compacted metal powders of corresponding stoichiometries [26–28]. However, formation of FeAl and Fe_3Al from direct intermetallic reaction in the Fe–Al system is not feasible, owing to their low enthalpies of formation (i.e., FeAl with $\Delta H_f = -50.2$ kJ/mol and Fe_3Al with $\Delta H_f = -67$ kJ/mol) [1]. In contrast, the thermite reaction of Fe_2O_3 with Al is extremely exothermic (i.e., the heat of reaction $\Delta H = -852.3$ kJ per unit mole of Al_2O_3 and adiabatic temperature $T_{\text{ad}} = 3622$ K) [24]. Therefore, this study aims to take advantage of the thermite-based SHS method to fabricate $\text{FeAl-TiB}_2\text{-Al}_2\text{O}_3$ in situ composites. The reaction systems involve the intermetallic reaction of Fe and Al, as well as aluminothermic reduction of Fe_2O_3 and B_2O_3 . Composites with different contents of FeAl and TiB_2 were synthesized. The activation energy of the thermite-based SHS reaction was determined from the measured reaction temperature and combustion front velocity. In addition, the constituents and fracture surface microstructures of as-synthesized composites were investigated.

2. Materials and Methods

The starting materials included Fe_2O_3 (Alfa Aesar Co., <45 μm , 99.5% Ward Hill, MA, USA), B_2O_3 (Strem Chemicals, 1–2 μm , 99.6%, Newburyport, MA, USA), Al (Showa Chemical Co., <45 μm , 99.9%, Tokyo, Japan), Fe (Alfa Aesar Co., <45 μm , 99.5%), and Ti (Alfa Aesar Co., <45 μm , 99.5%). Two oxide powders, Fe_2O_3 and B_2O_3 , were employed as thermite reagents. Similar to Fe_2O_3 , the thermite reaction of B_2O_3 with Al is exothermic and has $\Delta H = -403.8$ kJ/mol of Al_2O_3 and $T_{\text{ad}} = 2315$ K [24], which is also advantageous to the SHS process. Moreover, instead of expensive elemental boron, B_2O_3 serves as the source of boron for the formation of TiB_2 . Two combustion systems of different stoichiometries were formulated for synthesis of $\text{FeAl-TiB}_2\text{-Al}_2\text{O}_3$ composites and expressed as Reactions (1) and (2).



where the stoichiometric coefficients, x and y , denote the numbers of mole of FeAl and TiB_2 formed respectively from Reactions (1) and (2) in the $\text{FeAl-TiB}_2\text{-Al}_2\text{O}_3$ composites.

As can be seen, Reaction (1) is formulated by fixed amounts of Fe_2O_3 , B_2O_3 , and Ti to keep the quantities of TiB_2 and Al_2O_3 constant in the products. With the increase of x , the product of Reaction (1) contains a higher content of FeAl . On the other hand, Reaction (2) has variable contents of Fe_2O_3 and B_2O_3 . A larger y in Reaction (2) signifies an increase in B_2O_3 and Ti to yield TiB_2 at a larger amount. In Reaction (2), the decrease of Fe_2O_3 as y increases is balanced off by extra Fe powders to keep the same number of mole of FeAl formed in the products. Test matrices investigated by the present work are $1.5 \leq x \leq 3.5$ for Reaction (1) and $0.5 \leq y \leq 0.8$ for Reaction (2), within which the combustion wave propagates in a stable and self-sustaining manner.

The powders of raw materials were thoroughly mixed in a ball mill and then compressed into cylindrical test specimens with a diameter of 7 mm, a length of 12 mm, and a relative density of 60%. The SHS experiments were conducted in a stainless-steel chamber equipped with viewing windows under a high-purity argon environment (99.99%). The combustion wave propagation velocity (V_f) was determined from the time series of recorded images. The reaction temperature was measured by a 125 μm R-type thermocouple (Pt/Pt-13%Rh) attached on the sample surface. Phase analyses of

product constituents were performed by an X-ray diffractometer (Bruker D2, Bruker Co., Billerica, MA, USA) using $\text{CuK}\alpha$ radiation. The microstructure and atomic percentage of elements were examined under a scanning electron microscope (Hitachi, S3000H, Tokyo, Japan) coupled with energy dispersive spectroscopy (EDS). Details of the experimental setup and method were previously reported [29].

3. Results and Discussion

Self-Propagating Combustion Wave Kinetics

Figure 1 illustrates a typical series of the combustion images recorded from Reaction (1) with $x = 3.0$. It is apparent that a well-defined combustion wave develops upon ignition and traverses the entire sample in a self-sustaining manner. The synthesis reaction required no additional heat input. The combustion wave spent about $t = 4.8$ s to reach the end of the sample.

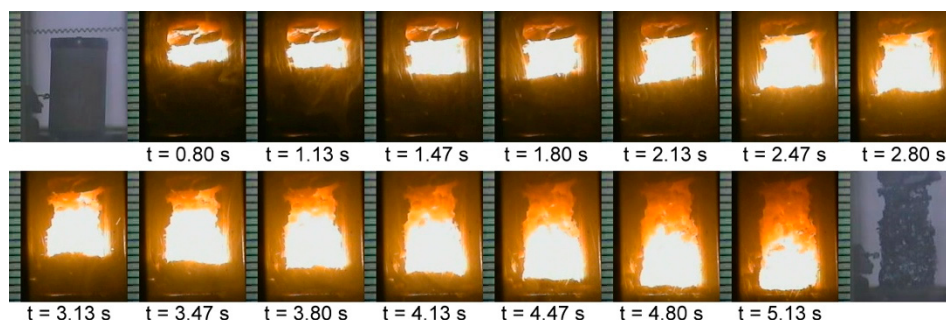


Figure 1. A time sequence of sample images recoded from Reaction (1) with $x = 3.0$ illustrating self-propagating combustion wave.

It is useful to note that in Reaction (1), the increase of Fe and Al (i.e., the coefficient x) for increasing FeAl imposes a dilution effect on combustion due to the low formation enthalpy of FeAl. Experimental observations indicated a combustibility limit for Reaction (1) at $x = 3.5$, beyond which the reaction ceased to proceed after ignition. However, for Reaction (1) with $x < 1.5$, a violent and hasty reaction accompanying substantial sample melting occurred, which led to incomplete phase conversion as a result of insufficient reaction times. This explains the range of $1.5 \leq x \leq 3.5$ for Reaction (1) in this study. For Reaction (2), since the thermite reaction of B_2O_3 is less energetic than that of Fe_2O_3 , the exothermicity of whole aluminothermic reduction declines with the increase of y and it was found that the reaction was extinct in Reaction (2) at $y > 0.8$.

The effect of sample stoichiometries on combustion front velocities of Reactions (1) and (2) is presented in Figure 2. The wave speed of Reaction (1) falls from 3.17 to 1.68 mm/s with increasing x from 1.5 to 3.5. The increase of x in Reaction (1) increases FeAl formed in the composite by having additional Fe and Al, but makes no change in the amount of Ti and the thermite composition. Because the increase of Fe and Al in the reactant mixture had a cooling effect, the combustion wave of Reaction (1) slowed down as FeAl augmented. Figure 2 also reveals a decline in the flame-front velocity from 2.84 to 1.83 mm/s for Reaction (2) as y augments from 0.5 to 0.8. The increase of y in Reaction (2) is to increase TiB_2 but maintains constant mole numbers of FeAl and Al_2O_3 . Therefore, the decrease of overall reaction exothermicity of Reaction (2) with increasing y could be responsible for deceleration of the combustion wave.

Figure 3 depicts typical combustion temperature profiles of Reactions (1) and (2). The profiles are characterized by an abrupt rise (i.e., a high temperature gradient), signifying rapid arrival of the combustion front, a peak value representing the combustion front temperature (T_c), and a significant descent behind the combustion wave (i.e., a rapid cooling rate). Such a unique synthesis condition leads to a uniform microstructure and phase distribution for the products [23]. Profiles #1, #2, and #3 in Figure 3 are recorded from Reaction (1) with $x = 1.5$, 2.0, and 3.0, respectively. The peak temperature

decreases from 1318 °C for $x = 1.5$ to 1269 °C for $x = 2.0$ and 1162 °C for $x = 3.0$, which confirms the decrease of reaction exothermicity with increasing FeAl. Moreover, a comparison between profiles #2 ($T_c = 1269$ °C at $y = 0.5$) and #4 ($T_c = 1141$ °C at $y = 0.8$) substantiates a decline of reaction exothermicity for Reaction (2) as the content of TiB_2 augments. It is useful to note that the composition dependence of flame-front velocities of Reactions (1) and (2) is consistent with that of the reaction temperature.

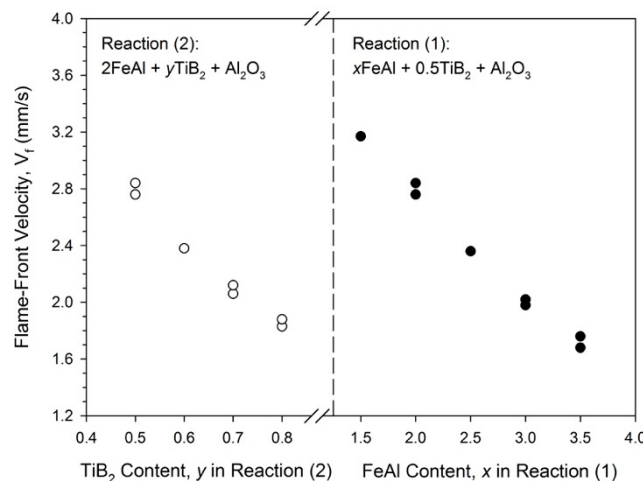


Figure 2. Variations of flame-front velocities of Reactions (1) and (2) with stoichiometric coefficients x and y .

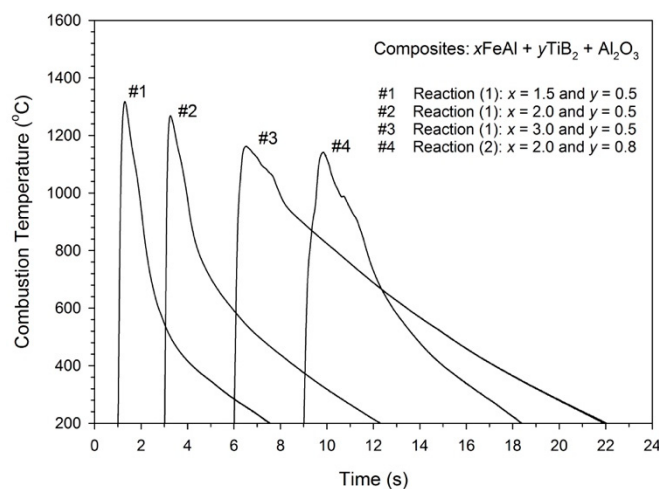


Figure 3. Variations of combustion temperatures of Reactions (1) and (2) with different contents of FeAl and TiB_2 .

The combustion front propagation velocity and reaction temperature are essential for the kinetics analysis of the combustion wave and determination of the apparent activation energy (E_a) of a solid-state combustion reaction. The dependence of the combustion wave velocity on reaction temperature plotted in Figure 4 correlates the relationship between $\ln(V_f/T_c)^2$ and $1/T_c$ [30]. Based upon the slope of a best-fitted linear line, $E_a = 88.92$ kJ/mol was obtained for the synergetic combustion reaction. It is useful to note that the Fe_2O_3 –Al thermitic reaction has E_a in a range between 82.3 and 97.0 kJ/mol [31]. This implies that once the kinetic barrier of reduction of Fe_2O_3 by Al is overcome, the synthesis sequences of Reactions (1) and (2) are able to proceed.

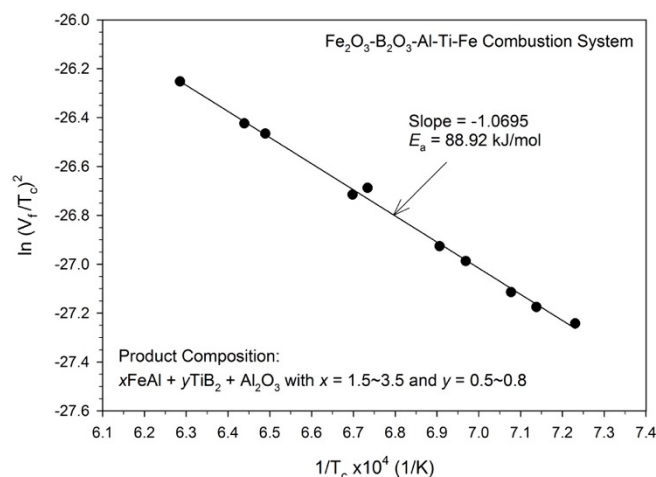


Figure 4. Correlation of reaction temperature (T_c) and combustion wave velocity (V_f) for determination of activation energy (E_a).

Figure 5 depicts the XRD patterns of as-synthesized $x\text{FeAl}$ - $y\text{TiB}_2$ - Al_2O_3 composites of different compositions. Apparently, the constituents comprise no other than FeAl, TiB_2 , and Al_2O_3 , indicative of a complete phase conversion from the reactants to final products. The XRD spectra of Figure 5a,b are correspondingly associated with Reaction (1), with $x = 2.0$ and 3.0 . The increase of FeAl is confirmed by FeAl signatures with stronger peak intensity in Figure 5b than Figure 5a. Figure 5c represents the composite with a content of TiB_2 at $y = 0.8$ obtained from Reaction (2). It is evident that the signature peaks of TiB_2 are obviously intensified in Figure 5c when compared to those in Figure 5a,b, both of which have a TiB_2 content of $y = 0.5$.

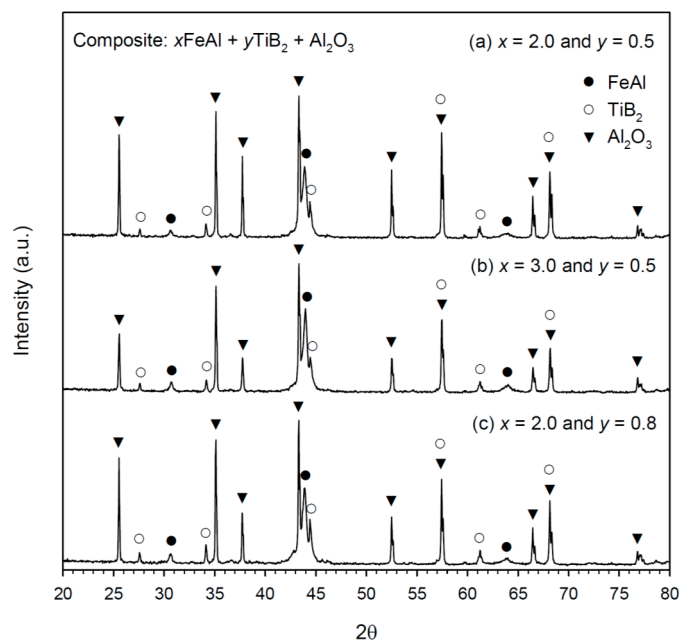


Figure 5. XRD patterns of self-propagating high-temperature synthesis (SHS)-derived products with (a) $x = 2.0$ and $y = 0.5$, (b) $x = 3.0$ and $y = 0.5$, and (c) $x = 2.0$ and $y = 0.8$.

Figure 6 exhibits the microstructure of the fracture surface of the $x\text{FeAl}$ - $y\text{TiB}_2$ - Al_2O_3 composite with $x = 3.0$ and $y = 0.5$. The morphology of the product is dense and connecting. Most of the TiB_2 and Al_2O_3 grains are embedded in the FeAl-based agglomerates, which have a size about $20\text{--}30\text{ }\mu\text{m}$. The EDS spectrum S1 specifies an atomic proportion of $\text{Al}:\text{O} = 38:62$, which certainly signifies Al_2O_3 .

The average size of Al_2O_3 grains is about 5–10 μm and some Al_2O_3 crystals are randomly distributed over the fracture surface. The spectrum S2 detects four elements Fe, Al, Ti, and B. The atomic ratio of Fe:Al is equal to 51:49, which matches well with FeAl. Based upon the elemental proportion of Ti:B = 34:66, the other component is identified as TiB_2 which has a short-rod shape around 3 μm .

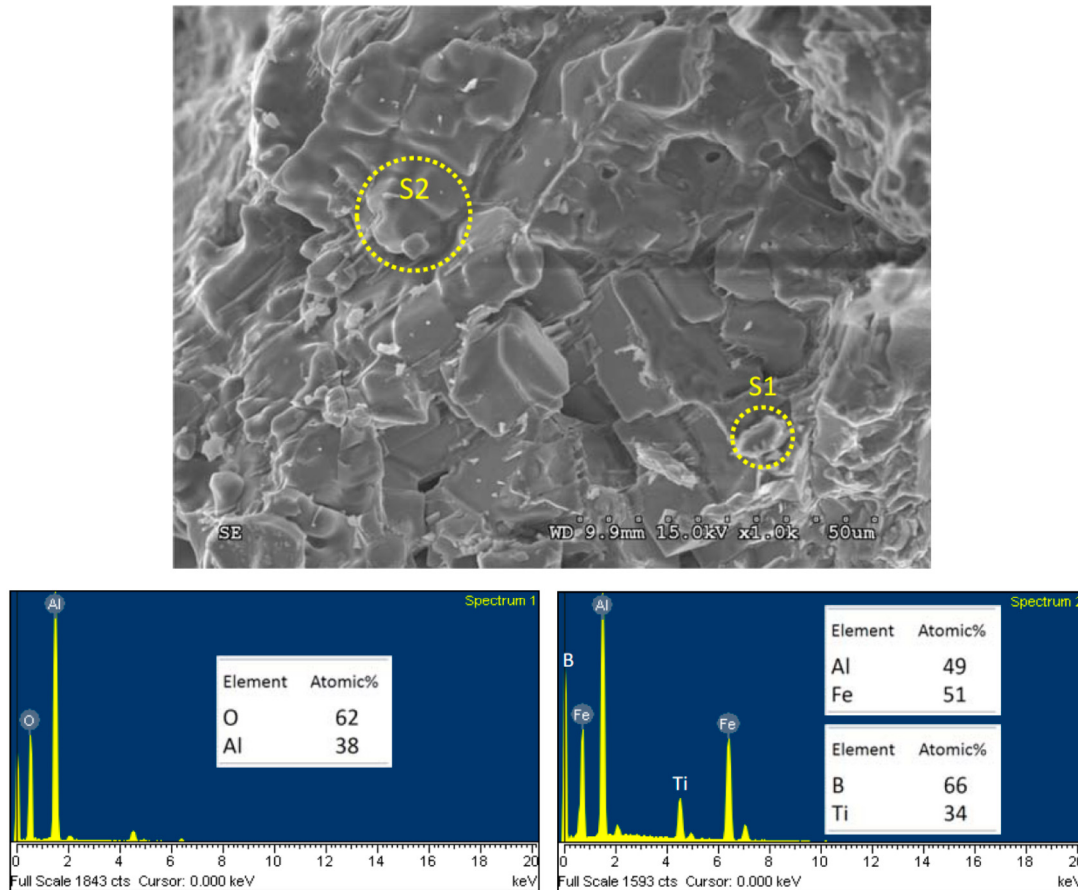


Figure 6. SEM micrograph and EDS spectra of $x\text{FeAl}-y\text{TiB}_2-\text{Al}_2\text{O}_3$ composite produced from Reaction (1) with $x = 3.0$ and $y = 0.5$.

4. Conclusions

Combustion syntheses involving intermetallic reaction of Fe with Al and thermite reduction of Fe_2O_3 and B_2O_3 were conducted to produce $\text{FeAl}-\text{TiB}_2-\text{Al}_2\text{O}_3$ composites with a broad composition. Two combustion systems, consisting of Fe, Al, Ti, Fe_2O_3 and B_2O_3 at different stoichiometries, were studied for the synthesis of $x\text{FeAl}-y\text{TiB}_2-\text{Al}_2\text{O}_3$ composites with $x = 1.5-3.5$ and $y = 0.5-0.8$. For the samples formulated within the test matrix, the combustion wave propagates in a self-sustaining manner. With an increase in FeAl and TiB_2 , the combustion wave velocity and reaction temperature decreased from 3.17 to 1.68 mm/s and 1318 °C to 1141 °C, respectively. This was attributed to the low enthalpy of formation for FeAl and a decrease in heat release of thermite reactions. Based on a correlation between combustion front velocity and reaction temperature, the activation energy $E_a = 88.92$ kJ/mol was deduced for the combined metallic and thermite reaction. According to the XRD analysis, a complete phase conversion was achieved, and synthesized composites were composed of FeAl, TiB_2 , and Al_2O_3 . The SEM micrograph showed the FeAl-based composite with a dense and connecting morphology. Three constituents were further confirmed by the EDS element ratios.

Author Contributions: Conceptualization, C.-L.Y. and C.-Y.K.; methodology, C.-L.Y. and C.-Y.K.; software, C.-L.Y.; validation, C.-L.Y. and C.-Y.K.; formal analysis, C.-L.Y. and C.-Y.K.; investigation, C.-L.Y. and C.-Y.K.; resources, C.-L.Y.; data curation, C.-L.Y. and C.-Y.K.; writing—original draft preparation, C.-L.Y.; writing—review and editing, C.-L.Y. and C.-Y.K.; visualization, C.-L.Y.; supervision, C.-L.Y.; project administration, C.-L.Y.; funding acquisition, C.-L.Y.

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Conflicts of Interest: The authors declare no conflict of interest.

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