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Formation of Ta–Al intermetallics by combustion synthesis involving Al-based thermite reactions

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ABSTRACT

Thermite-based combustion synthesis was conducted to investigate in situ formation of tantalum aluminides (Ta_2Al , TaAl, $TaAl_2$, and $TaAl_3$) and Al_2O_3 from the powder compacts of Al and Ta_2O_5 under a molar ratio ranging from Al/Ta₂O₅ = 13/3 to 28/3. Effects of the sample stoichiometry on the combustion behavior and degree of phase conversion were studied. Experimental evidence showed that upon ignition, the synthesis reaction proceeded in a self-sustaining manner featuring a planar combustion front or two localized reaction zones traveling spirally. The combustion front temperature varies between 1135 and 1460 °C. In agreement with composition dependence of the reaction exothermicity, the reaction front velocity in the axial direction increased from 5.8 to 7.2 mm/s with molar ratio of Al/Ta₂O₅ from 13/3 to 16/3, beyond which the flame velocity decreased substantially. The slowest reaction front with an axial velocity of 1.15 mm/s was observed in the sample of Al/Ta₂O₅ = 28/3. Based upon the XRD analysis, in situ formation of the composites composed of Al₂O₃ and tantalum aluminides was confirmed. For the samples with Al contents of Al/Ta₂O₅ = 13/3-18/3, an increase in the Al/Ta₂O₅ ratio improved the production of tantalum aluminides and caused the change in the dominant aluminide phase from Ta₂Al. TaAl, to TaAl₂. Due to lack of adequate reaction time, the phase conversion was not stoichiometrically achieved in the samples of $AI/Ta_2O_5 = 13/3 - 18/3$. However, it was found that the slower reaction front provided longer reaction time to complete the phase conversion almost stoichiometrically in the samples of Al/Ta₂O₅ = 22/3-28/3, which yielded the Al-rich phases TaAl₂ and/or TaAl₃ in the final products.

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1. Introduction

Intermetallic compounds based upon aluminides of several transition metals such as iron, nickel, titanium, cobalt, niobium, and tantalum have been recognized as potential candidates for the high-temperature structural applications [1–5]. Many fabrication routes for aluminides of the transition metals have been developed; for instance, mechanical alloying, hot pressing, reaction synthesis, melting, casting, etc. [6,7]. With the merits of time and energy savings, combustion synthesis particularly in the mode of self-propagating high-temperature synthesis (SHS) represents a promising alternative to produce a variety of refractory materials, such as borides, carbides, nitrides, carbontrides, silicides, aluminides and other intermetallics [8-10]. Most of the research effort in the preparation of aluminide compounds by SHS from metallic powders of their constituents has been devoted to the aluminides of nickel (NiAl and Ni₃Al) [11-14], titanium (TiAl, Ti₃Al, and TiAl₃) [15–18], cobalt (CoAl) [19,20], and niobium (NbAl₃) [20–22].

Because the reactions between metallic reactants are normally less exothermic than those between metals and non-metal elements (e.g., B, C, and N), the SHS technique applied to the preparation of aluminides often requires additional treatments or modifications, such as prior heating [13,18,19], electric-field activation [15,17], microwave assistance [16], and high-energy mechanical milling [21,22].

On the other hand, when compared with the conventional SHS process using elemental powder compacts, formation of transition metal aluminides by combustion synthesis of the thermite type is generally more energetic and starts with less expensive metal oxides as the source of transition metals [23]. For example, by means of the thermite-based SHS process with Al and Nb₂O₅ as the reactants, Yeh and Wang [24] successfully fabricated two Nb-rich aluminides Nb₃Al and Nb₂Al, both of which cannot be formed through the reaction between Nb and Al particles due to weak exothermicity. Moreover, the integration of combustion synthesis with Al-based thermite reactions leads to in situ formation of the aluminide matrix composite reinforced by Al₂O₃ [25-27]. Many composites comprising an aluminide matrix (such as FeAl, TiAl, NiAl, and NbAl₃) and a reinforcing phase of Al₂O₃ have been shown to possess improved high-temperature strength and creep resistance [28-31].

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Among various aluminides of transition metals, the compounds in the Ta-Al system have been of great interest for their excellent mechanical properties and high degree of structural complexity [32,33]. The Ta-Al phase diagram depicted in Fig. 1 shows the existence of four aluminides, including Ta₂Al, TaAl, TaAl₂, and TaAl₃ [34]. However, the enthalpy of formation (ΔH_f) associated with each tantalum aluminide is relatively low and varies between -22 and -30 kJ/mol [35], which is responsible for the unfeasibility of formation of the tantalum aluminide by SHS from the elemental powder compact. Therefore, instead of using Ta and Al powders, this study employs tantalum oxide (Ta_2O_5) and Al as the starting materials to prepare tantalum aluminides through thermite-based combustion synthesis. Such a process essentially combines two consecutive stages: the first one produces elemental Ta and byproduct Al₂O₃ via the thermite reaction that concurrently liberates substantial reaction heat to initiate the second stage, which yields aluminide compounds from the interaction between the reduced Ta and AL

In this study, the Al_2O_3 -reinforced tantalum aluminides were obtained through the SHS process of the thermite type and the aluminide phase formed in the product was investigated with respect to the initial composition of the reactant compact (i.e., the molar ratio of Al to Ta₂O₅). Additionally, the effect of sample stoichiometry was studied on the combustion temperature as well as the spreading mode and propagation rate of the combustion wave.

2. Experimental methods of approach

The present study adopts tantalum oxide Ta_2O_5 (Strem Chemicals, 99.8% purity) and elemental Al (Showa Chemical Co., <40 μ m, 99% purity) powders as the raw materials. Reactions (1) through (4) correspond to stoichiometric formation of four different tantalum aluminides, Ta₂Al, TaAl, TaAl₂, and TaAl₃, with Al₂O₃.

$13AI + 3Ia_2O_5 \rightarrow 3Ia_2AI + 5AI_2O_3 \tag{1}$	1)	
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 $16\text{Al} + 3\text{Ta}_2\text{O}_5 \rightarrow 6\text{Ta}\text{Al} + 5\text{Al}_2\text{O}_3 \tag{2}$

 $22Al + 3Ta_2O_5 \rightarrow 6TaAl_2 + 5Al_2O_3 \tag{3}$

 $28AI + 3Ta_2O_5 \rightarrow 6TaAl_3 + 5Al_2O_3 \tag{4}$

According to reactions (1)–(4), the starting powder mixtures were formulated with a molar ratio of Al/Ta₂O₅ ranging from 4.33 (13/3) to 9.33 (28/3). Moreover, the standard reaction enthalpy (Δ H) of reactions (1)–(4) was calculated with thermodynamic data of Refs. [35,36] and presented in Table 1 under unit mole of the product (including the aluminide phase and Al₂O₃). Due to an appreciable amount of heat generated from the production of Al₂O₃, reactions (1)–(4) are about 10-fold more exothermic than the formation of tantalum aluminides from their constituent elements (i.e., their enthalpies of formation).

Under the prescribed stoichiometry, the reactant powders were dry mixed in a ball mill and then cold-pressed into cylindrical test specimens with a diameter of 7 mm, a height of 12 mm, and a compaction density of 50% relative to the theoretical maximum density (TMD). The SHS experiment was conducted in a stainless-steel windowed combustion chamber under an atmosphere of high-purity argon (99.99%). The ignition was accomplished by a heated tungsten coil with a voltage of 60 V and a current of 1.5 A. Details of the experimental setup and methods of measurement approach were previously reported [13,19].

3. Results and discussion

3.1. Observation of combustion characteristics

Two typical combustion sequences in association with the reactant compacts of different starting compositions of Ta_2O_5 :Al = 3:16 and 3:22 are respectively illustrated in Fig. 2(a) and (b). As shown in Fig. 2(a), upon ignition a nearly planar combustion front forms and traverses the entire sample self-sustainingly in about 1.6 s. However, Fig. 2(b) reveals that shortly after initiation the planar front is transformed into two localized combustion zones moving along a spiral trajectory on the sample surface. In the recorded image

Table 1

Standard reaction enthalpy (ΔH) per unit mole of product for reactions of Al with Ta_2O_5 under different stoichiometries.

Thermite-based combustion synthesis reactions	$\Delta H (kJ/mol)$
$\frac{13}{8}\text{Al}+\frac{3}{8}\text{Ta}_2\text{O}_5 \rightarrow \frac{3}{8}\text{Ta}_2\text{Al}+\frac{5}{8}\text{Al}_2\text{O}_3$	-288
$\tfrac{16}{11}\text{Al} + \tfrac{3}{11}\text{Ta}_2\text{O}_5 \rightarrow \tfrac{6}{11}\text{Ta}\text{Al} + \tfrac{5}{11}\text{Al}_2\text{O}_3$	-217
$\frac{22}{11}Al+\frac{3}{11}Ta_2O_5\rightarrow \frac{6}{11}TaAl_2+\frac{5}{11}Al_2O_3$	-218
$\tfrac{28}{11}\text{Al} + \tfrac{3}{11}\text{Ta}_2\text{O}_5 \rightarrow \tfrac{6}{11}\text{Ta}\text{Al}_3 + \tfrac{5}{11}\text{Al}_2\text{O}_3$	-220



Fig. 2. Recorded SHS sequences recorded from powder compacts with starting stoichiometric ratios of Ta₂O₅:Al = (a) 3:16 and (b) 3:22. (A scale with a unit of mm is optically superimposed on the left-hand side of each image.).



Fig. 3. Photographs of synthesized products obtained from powder compacts with starting stoichiometric ratios of Ta₂O₅:Al = (a) 3:16 and (b) 3:22.



Fig. 4. Effect of molar ratio of Al/Ta₂O₅ of reactant compacts on flame-front propagation velocity of SHS processes.

at t = 1.60 s of Fig. 2(b), two combustion zones are circled. Obviously, it took longer time of approximately 2.9s for the spinning combustion wave to arrive at the bottom of the sample. According to Ivleva and Merzhanov [37], once the heat flux liberated from self-sustaining combustion is no longer sufficient to maintain the steady propagation of a planar front, the combustion front forms one or several localized reaction zones. In view of the comparable reaction enthalpies reported in Table 1 for the reactions with powder compacts of Ta_2O_5 : Al = 3:16 and 3:22, the appearance of the spinning combustion wave in the sample of Ta_2O_5 :Al = 3:22 is essentially attributed to consumption of more reaction enthalpy as the latent heat of Al (28.7 kJ/mol) by the sample with a higher content of Al. As a result, the experimental observations of this study indicated that for the powder compacts with a molar ratio of Al/Ta₂O₅ \ge 22/3, the combustion front was restricted typically within two localized regions propagating in a spinning manner, similar to that of Fig. 2(b).

Fig. 3(a) and (b) shows two photographs of the synthesized products respectively obtained from the sample compacts with starting stoichiometric ratios of Ta_2O_5 :Al = 3:16 and 3:22. The product in Fig. 3(a) subjected to the propagation of a planar combustion front exhibits a porous appearance and a metallic shine. For the synthesized product experiencing a spinning combustion wave, Fig. 3(b) displays spiral marks on the rugged exterior of the product.

3.2. Measurement of flame-front propagation velocity

The flame-front propagation velocity (V_f) in the axial direction was determined from the recorded combustion images and plotted in Fig. 4 as a function of the starting stoichiometry of the reactant compact. The flame-front velocity was found to increase from 5.8 to 7.2 mm/s with increasing molar ratio of Al/Ta₂O₅ from 4.33 (13/3) to 5.33 (16/3), beyond which the flame velocity decreased significantly with Al content and arrived at a minimum of 1.15 mm/s in the sample of Al/Ta₂O₅ = 28/3. The increase of combustion velocity observed in Fig. 4 is mainly due to an improvement in the degree of aluminide formation, which leads to a higher reaction temperature and hence a faster combustion front. This is confirmed by the fact that the amount of elemental Ta left unreacted in the final product was reduced by increasing the Al content in the sample. On the other hand, as mentioned above, the increase of the ratio of Al/Ta₂O₅ in a powder compact means more reaction heat consumed by Al as its latent heat, which lowers the overall reaction exothermicity and accounts for the decrease of the flame speed shown in Fig. 4. In addition, the low axial velocity of the reaction front for the powder compacts with Al/Ta₂O₅ \geq 22/3 is also a consequence of the spiral propagation of the reaction zone, under which the combustion wave travels not only longitudinally but also transversely.

3.3. Measurement of combustion temperature

Six combustion temperature profiles recorded from the Ta₂O₅–Al powder compacts with different initial stoichiometries are depicted in Fig. 5, where the abrupt rise in temperature signifies rapid arrival of the combustion front and the peak value represents the reaction front temperature. It is evident that the temperatures curves of two different shapes are seen. One features a single peak corresponding to the longitudinal progression of a planar front (for Al/Ta₂O₅ = 13/3, 16/3, 18/3, and 20/3 in Fig. 5). The other accounting for the spinning motion of two consecutive reaction zones is characterized by double spikes (for $Al/Ta_2O_5 = 22/3$ and 26/3 in Fig. 5). The combustion front temperatures presented in Fig. 5 range between 1135 and 1460 °C, which are lower than the melting points of tantalum aluminides in Fig. 1 and Al₂O₃ (2054 °C). This explains that the burned sample almost retains its original shape after combustion. In agreement with the composition dependence of combustion velocity, the combustion front temperature increases with Al content, reaches a maximum at $Al/Ta_2O_5 = 16/3$, and then decreases with further increase of Al.

3.4. Phase constituent of SHS products

The XRD patterns of the products synthesized from the reactant compacts of Ta_2O_5 :Al = 3:13 and 3:14 are depicted respectively in Fig. 6(a) and (b), which confirm in situ formation of tantalum aluminides and Al₂O₃. Despite the production of two kinds of aluminides (Ta₂Al and TaAl), Fig. 6(a) and (b) indicates a considerable amount of elemental Ta left unreacted in the final products. The remnant Ta was diminished by introducing more Al, which also led to the appearance of another intermetallic TaAl₂ when the Al content reached the proportion of Al/Ta₂O₅ = 16/3, as shown in Fig. 6(c). Furthermore, Fig. 6(d) reveals that for the powder compact of Ta₂O₅:Al = 3:18, TaAl₂ becomes the dominant aluminide. In view of reactions (1) and (2), the presence of multiple aluminide phases and free Ta in Fig. 6(a) and (c) signifies incomplete conversion, which is presumably due to lack of sufficient reaction time caused by rapid propagation of the reaction front.



Fig. 5. Dependence of combustion temperature on Al content of Ta_2O_5 -Al powder compacts.



Fig. 6. XRD patterns of SHS products obtained from powder compacts with $Ta_2O_5:AI$ (a) 3:13, (b) 3:14, (c) 3:16, and (d) 3:18.

On the other hand, Fig. 7(a) shows in situ formation of Al_2O_3 and $TaAl_2$ from the sample of Ta_2O_5 :Al = 3:22, which represents complete conversion according to reaction (3). With a larger molar ratio of Al/Ta₂O₅, as shown in Fig. 7(b)–(d), the other Al-rich com-



Fig. 7. XRD patterns of SHS products obtained from powder compacts with Ta_2O_5 :Al = (a) 3:22, (b) 3:24, (c) 3:26, and (d) 3:28.

Table 2

Summary of phase composition of SHS products with respect to their initial stoichiometries.

Stoichiometry Ta ₂ O ₅ :Al	Phase composition of SHS products			
	Dominant aluminide(s)	Secondary aluminide(s)	Others	
3:13	Ta ₂ Al	TaAl	Ta, Al ₂ O ₃	
3:14	Ta ₂ Al	TaAl	Ta, Al ₂ O ₃	
3:15	Ta ₂ Al	TaAl, TaAl ₂	Ta, Al ₂ O ₃	
3:16	TaAl	Ta ₂ Al, TaAl ₂	Ta, Al ₂ O ₃	
3:18	TaAl ₂	TaAl, Ta ₂ Al	Al_2O_3	
3:20	TaAl ₂	Ta ₂ Al	Al_2O_3	
3:22	TaAl ₂		Al_2O_3	
3:24	TaAl ₂	TaAl ₃	Al_2O_3	
3:26	TaAl ₃	TaAl ₂	Al_2O_3	
3:28	TaAl ₃	TaAl ₂	Al_2O_3	

pound TaAl₃ is identified along with TaAl₂. Because TaAl₂ is trivial in Fig. 7(d), the product obtained from the sample of Ta₂O₅:Al = 3:28 is practically a composite of TaAl₃ and Al₂O₃, as described by reaction (4). Fig. 7(a) and (d) means that in contrast to reactions (1) and (2), the complete conversion of reactions (3) and (4) is verified. Better phase transformation achieved in the powder compacts of Al/Ta₂O₅ \geq 22/3 is most likely attributed to the longer reaction time provided by slower propagation of the reaction front.

A summary of the product composition with respect to the starting stoichiometry of the Ta_2O_5 -Al sample is given in Table 2. It was found that an increase in the molar ratio of Al/Ta₂O₅ not only improved the aluminide formation, but also altered the dominant aluminide phase. No elemental Ta was observed in the final products until the molar ratio of the sample reached Al/Ta₂O₅ = 18/3 and above. The Ta-rich phase Ta₂Al was identified as the dominant aluminide for the initial compositions of Al/Ta₂O₅ = 13/3-15/3, while the Al-rich phase TaAl₂ was recognized for Al/Ta₂O₅ = 18/3-24/3 and TaAl₃ for Al/Ta₂O₅ = 26/3 and 28/3. Besides, due to the complexity of the aluminide TaAl is granted to be the major aluminide according to reaction (2).

4. Conclusions

This study presents a comprehensive investigation of the thermite-based SHS process associated with in situ formation of tantalum aluminides and Al₂O₃. The experiments were conducted with Al-Ta₂O₅ powder compacts under a molar ratio of Al/Ta₂O₅ between 13/3 and 28/3. For the samples of $Al/Ta_2O_5 = 13/3 - 21/3$, the SHS process featured a planar combustion front. However, due to a decrease in the overall reaction exothermicity, the reaction in the samples of Al/Ta₂O₅ = 22/3-28/3 was confined to two localized areas propagating spirally. The increase of the Al content in the sample contributed to an improvement in the formation of tantalum aluminides, but it enlarged the fraction of the reaction heat consumed as the latent heat of Al. Therefore, the combustion temperature as well as the reaction front velocity exhibited an increase with molar ratio of Al/Ta₂O₅, approached their maxima at $AI/Ta_2O_5 = 16/3$, and then decreased constantly with further increase in Al.

For the samples with Al contents of $Al/Ta_2O_5 = 13/3-16/3$, the final products contained free Ta and aluminide compounds like Ta₂Al, TaAl, and TaAl₂. As the molar ratio of Al/Ta_2O_5 increased up to 18/3 and above, the elemental Ta reduced from Ta_2O_5 was fully converted into TaAl₂ and/or TaAl₃. Moreover, the phase conversion was stoichiometrically achieved in the samples of Ta_2O_5 : Al = 3:22 and 3:28, from which in situ composites of TaAl₂-Al₂O₃ and TaAl₃-Al₂O₃ were produced, respectively.

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