

Combustion synthesis of $(\text{Ti}_{1-x}\text{Nb}_x)_2\text{AlC}$ solid solutions from elemental and $\text{Nb}_2\text{O}_5/\text{Al}_4\text{C}_3$ -containing powder compacts

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Abstract

Preparation of the $(\text{Ti}_{1-x}\text{Nb}_x)_2\text{AlC}$ solid solution (formed from the $\text{M}_{n+1}\text{AX}_n$ or MAX carbides, where $n = 1, 2$, or 3 , M is an early transition metal, A is an A-group element, and X is C) with $x = 0.2$ – 0.8 was investigated by self-propagating high-temperature synthesis (SHS). Nearly single-phase $(\text{Ti,Nb})_2\text{AlC}$ was produced through direct combustion of constituent elements. Due to the decrease of reaction exothermicity, the combustion temperature and reaction front velocity decreased with increasing Nb content of $(\text{Ti}_{1-x}\text{Nb}_x)_2\text{AlC}$ formed from the elemental powder compacts. In addition, the samples composed of Ti , Al , Nb_2O_5 , and Al_4C_3 were adopted for the in situ formation of Al_2O_3 -added $(\text{Ti,Nb})_2\text{AlC}$. The SHS process of the $\text{Nb}_2\text{O}_5/\text{Al}_4\text{C}_3$ -containing sample involved aluminothermic reduction of Nb_2O_5 , which not only enhanced the reaction exothermicity but also facilitated the evolution of $(\text{Ti,Nb})_2\text{AlC}$. Based upon the XRD analysis, two intermediates, TiC and Nb_2Al , were detected in the $(\text{Ti,Nb})_2\text{AlC}/\text{Al}_2\text{O}_3$ composite and their amounts were reduced by increasing the extent of thermite reduction involved in the SHS process. The laminated microstructure characteristic of the MAX carbide was observed for both monolithic and Al_2O_3 -added $(\text{Ti,Nb})_2\text{AlC}$ solid solutions synthesized in this study.

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1. Introduction

Layered ternary compounds $\text{M}_{n+1}\text{AX}_n$ (also called the MAX phase), where $n = 1, 2$, or 3 , M is an early transition metal, A is an A-group (mostly IIIA and IVA) element, and X is either C or N , are a new class of materials featuring crystal structures of the hexagonal symmetry with the space group $P6_3/mmc$ [1–4]. The major difference between M_2AX ($2\ 1\ 1$), M_3AX_2 ($3\ 1\ 2$), and M_4AX_3 ($4\ 1\ 3$) compounds is the number of M – X layers separating each A layer; i.e., there are 2, 3, and 4 M – X layers for $2\ 1\ 1$, $3\ 1\ 2$, and $4\ 1\ 3$ phases, respectively. Representative MAX phases, such as Ti_3SiC_2 , Ti_3AlC_2 , Ti_2AlC , Cr_2AlC , and Ti_2AlN , have been extensively studied [3–9]. Due to their unique properties combining the characteristics of metals and ceramics, MAX carbides and nitrides have attracted considerable attention. In general, they are relatively soft, readily machinable, thermally and electrically conductive, elastically

stiff, damage tolerant, and highly resistant to thermal shock, oxidation, and corrosion [1–9]. These properties stem from an inherently laminated crystal structure with M_{n+1}X_n slabs intercalated with pure A-element layers. Comprehensive reviews contributed by Refs. [10–13] summarize recent achievements on the processing methods, microstructural characterizations, formation mechanisms, and mechanical, physical, and electrical properties of various MAX phases.

Moreover, solid solutions based on the MAX phases can be formed by substitutions on the M , A , or X site, which renders great potential for tailoring and/or optimizing the properties of such machinable ceramics. $(\text{Ti,V})_2\text{AlC}$, $(\text{Ti,Nb})_2\text{AlC}$, and $(\text{Cr,V})_2\text{AlC}$ are typical M-site solid solutions [14–19]. Excellent miscibility of M_2AlC ($\text{M} = \text{Ti}$, V , Nb , and Cr) was confirmed [14,15]. For the $(\text{Ti}_{0.8}\text{V}_{0.2})_2\text{AlC}$ solid solution produced by the in situ hot pressing/solid–liquid reaction [16], the Vickers hardness, flexural strength, and shear strength were enhanced when compared to those of Ti_2AlC . By pulse discharge sintering (PDS), Tian et al. [19] prepared single-phase $(\text{Cr,V})_2\text{AlC}$ and reported an increase in the Vickers hardness with increasing V content. Considerable studies were

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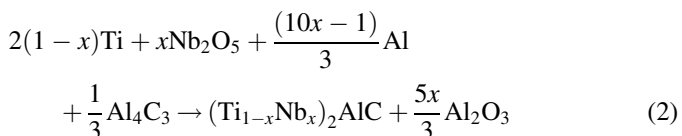
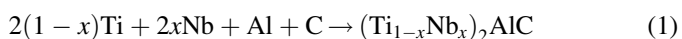
also performed on numerous A-site solid solutions, such as $\text{Ti}_3(\text{Si},\text{Al})\text{C}_2$, $\text{Ti}_3(\text{Si},\text{Ge})\text{C}_2$, and $\text{Ti}_3(\text{Sn},\text{Al})\text{C}_2$ [20–23]. A significant strengthening effect on the physical properties was observed for $\text{Ti}_3\text{Al}_{1-x}\text{Si}_x\text{C}_2$ with x greater than 0.15 [20], but little was contributed by the Ge substitution on $\text{Ti}_3(\text{Si},\text{Ge})\text{C}_2$ [22]. For the MAX solid solution on the X site, the carbonitride $\text{Ti}_2\text{Al}(\text{C},\text{N})$ is the representative and the associated fabrication routes are hot isostatic pressing (HIP) [24] and combustion synthesis in gaseous nitrogen [25,26].

Of particular interest for this work is the M-site solid solution $(\text{Ti},\text{Nb})_2\text{AlC}$. Thus far, $(\text{Ti},\text{Nb})_2\text{AlC}$ has been synthesized from the powder mixtures of Ti, Nb, graphite, and Al_4C_3 by HIP at 1600 °C and 100 MPa for 8 h [27,28]. With the advantages of time and energy savings, combustion synthesis particularly in the mode of self-propagating high-temperature synthesis (SHS) represents an attractive alternative of producing advanced materials including borides, carbides, nitrides, silicides, carbonitrides, intermetallics, etc. [29–31]. Moreover, a number of MAX carbides, such as Ti_3AlC_2 [32], Ti_2AlC [33], Nb_2AlC [34], and Cr_2AlC [35], have been successfully produced by the SHS process.

As the first attempt, this study aims to prepare $(\text{Ti}_{1-x}\text{Nb}_x)_2\text{AlC}$ solid solutions with a broad range of the Nb/Ti proportion by the SHS process. The reactant compacts adopted in this study have two kinds: one comprises constituent elements for the synthesis of monolithic $(\text{Ti},\text{Nb})_2\text{AlC}$, and the other consists of Ti, Nb_2O_5 , Al, and Al_4C_3 for the in situ formation of Al_2O_3 -added $(\text{Ti},\text{Nb})_2\text{AlC}$. According to the previous studies [36–38], the addition of Al_2O_3 to the MAX carbides like Ti_2AlC , Ti_3AlC_2 , and Ti_3SiC_2 substantially improved many of their mechanical properties. In this study, the influence of starting stoichiometry of the sample is investigated on the phase constituent and morphology of the final product, as well as on the combustion temperature and propagation velocity of the reaction front.

2. Experimental methods of approach

The starting materials for the synthesis of monolithic $(\text{Ti},\text{Nb})_2\text{AlC}$ shown in Reaction (1) include four elemental powders: Ti (Strem Chemicals, $\leq 45 \mu\text{m}$, 99%), Nb (Strem Chemicals, $\leq 45 \mu\text{m}$, 99.8%), Al (Showa Chemical Co., $\leq 40 \mu\text{m}$, 99%), and carbon black (Showa Chemical Co.). For the formation of the $(\text{Ti},\text{Nb})_2\text{AlC}/\text{Al}_2\text{O}_3$ composite, Reaction (2) indicates that the reactant mixture is composed of Ti, Al, Nb_2O_5 (Strem Chemicals, 99.9%), and Al_4C_3 (Strem Chemicals, $\leq 45 \mu\text{m}$, 98%) powders.



where the stoichiometric parameter x represents the Nb content of $(\text{Ti}_{1-x}\text{Nb}_x)_2\text{AlC}$. It should be noted that Reaction (2) involves thermite reduction of Nb_2O_5 by Al. The thermite reaction based upon Al as the reducing agent is recognized by the release of a

large amount of reaction enthalpy and formation of a stable oxide Al_2O_3 [39]. Reactant powders according to Reactions (1) and (2) were prepared with x varying from 0.2 to 0.8. Powder mixtures were cold-pressed into the cylindrical compact with a diameter of 7 mm, a height of 12 mm, and a compaction density of 55% relative to the theoretical maximum density (TMD). The SHS experiment was performed in a stainless-steel windowed chamber under an atmosphere of high purity argon (99.99%). Details of the experimental setup and measurement approach were reported elsewhere [40].

3. Results and discussion

3.1. Observation of combustion characteristics

Fig. 1(a) and (b) illustrates two SHS sequences associated with the elemental powder compact and $\text{Nb}_2\text{O}_5/\text{Al}_4\text{C}_3$ -containing sample, respectively. As shown in Fig. 1(a), the direct combustion of constituent elements under Reaction (1) with $x = 0.2$ features a distinct reaction front traversing the entire sample in a self-sustaining manner. For the $\text{Nb}_2\text{O}_5/\text{Al}_4\text{C}_3$ -containing sample based on Reaction (2) of $x = 0.5$, the combustion process involving thermite reduction of Nb_2O_5 by Al is presented in Fig. 1(b). When compared with the direct reaction of elemental reagents, the thermite-incorporated combustion appears to be more violent, causing a significant melting of the sample during combustion. This might imply higher reaction exothermicity for the sample composed of Ti, Al, Nb_2O_5 , and Al_4C_3 than the elemental powder compact.

3.2. Measurement of flame-front propagation velocity and combustion temperature

Fig. 2 presents the variation of the flame-front propagation velocity (V_f) with Nb content of $(\text{Ti},\text{Nb})_2\text{AlC}$ formed from samples of Reactions (1) and (2). The reaction front velocity of the elemental powder compact, as shown in Fig. 2, decreases from 7.6 to 3.6 mm/s upon the formation of $(\text{Ti}_{1-x}\text{Nb}_x)_2\text{AlC}$ with increasing Nb proportion from $x = 0.2$ to 0.8. On the other hand, the flame-front velocity of the $\text{Nb}_2\text{O}_5/\text{Al}_4\text{C}_3$ -containing sample increases from 4.1 to 11.4 mm/s as the stoichiometric parameter x of Reaction (2) rises from $x = 0.3$ to 0.8. It should be noted that, due probably to lack of sufficient reaction enthalpy, combustion was extinguished after ignition for the powder compact of Reaction (2) with $x = 0.2$.

Based upon the reaction mechanism of Ti_2AlC [41,42], it is believed that for the sample of Reaction (1) the thermal energy released from the reaction of Ti with carbon plays a critical role in sustaining the combustion wave. The increase of Nb means a corresponding decrease in Ti for the reactant mixture of Reaction (1), which reduces the reaction exothermicity and thus decelerates the reaction front. For the combustion system of Reaction (2), the thermite reduction of Nb_2O_5 by Al is considered as the most important energy contributor to trigger and sustain the reaction. The increase of combustion velocity of the $\text{Nb}_2\text{O}_5/\text{Al}_4\text{C}_3$ -containing sample is attributed to the increase

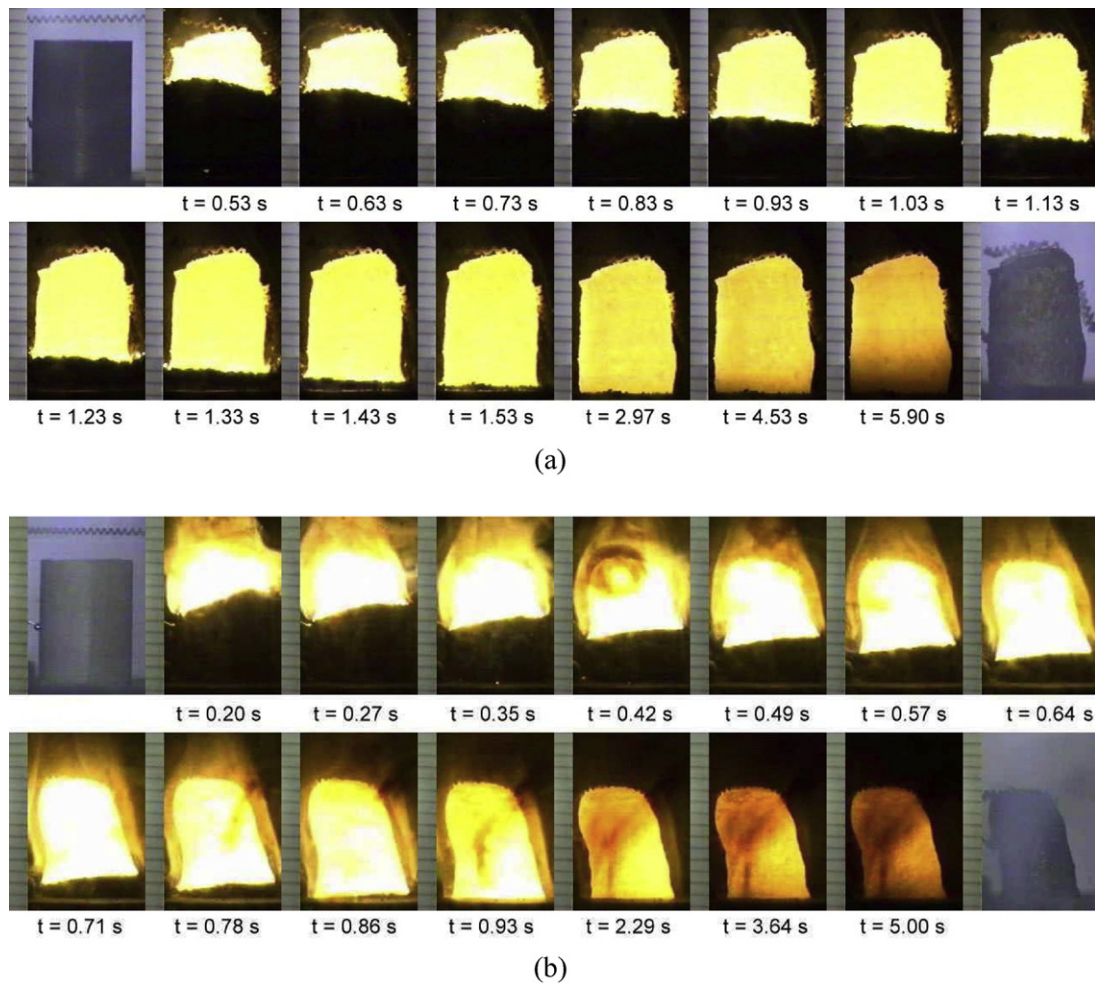


Fig. 1. Recorded images illustrating self-propagating combustion along (a) an elemental powder compact with $x = 0.2$ and (b) a $\text{Nb}_2\text{O}_5/\text{Al}_4\text{C}_3$ -containing sample with $x = 0.5$.

of thermite reagents in the reactant mixture. This also explains the quench of combustion for the sample of Reaction (2) with $x = 0.2$, under which only small amounts of Nb_2O_5 and Al are included.

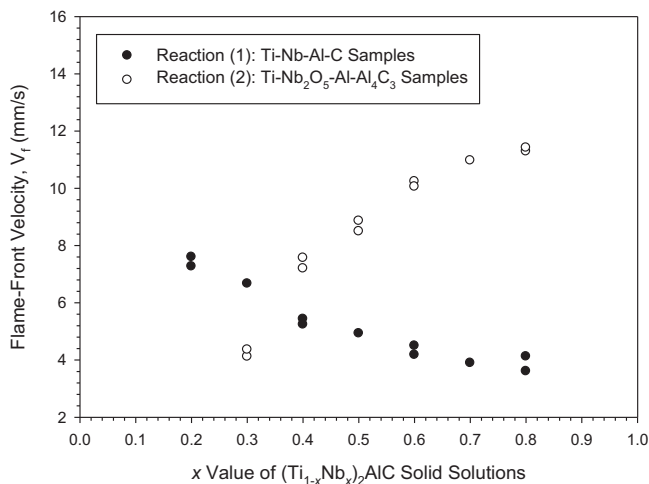


Fig. 2. Variations of flame-front propagation velocity with Nb content of $(\text{Ti}_{1-x}\text{Nb}_x)_2\text{AlC}$ solid solutions synthesized from samples composed of different reactant mixtures.

Fig. 2 also indicates that except for the cases of $x = 0.2$ and 0.3 , the flame-front velocity of the $\text{Nb}_2\text{O}_5/\text{Al}_4\text{C}_3$ -containing sample is higher than that of the elemental powder compact and the gap between them is widened for the formation of $(\text{Ti}_{1-x}\text{Nb}_x)_2\text{AlC}$ with a higher proportion of Nb. This is because the reaction enthalpy generated from aluminothermic reduction of Nb_2O_5 to produce 1 mole of Al_2O_3 is 536 kJ, which is much larger than the heat of formation (ΔH_f) of TiC (-184.1 kJ/mol) [43]. When $(\text{Ti}_{1-x}\text{Nb}_x)_2\text{AlC}$ with a higher content of Nb is produced, more thermite reagents are included in the sample of Reaction (2) while a lesser amount of Ti is adopted in that of Reaction (1). This could be responsible for the enlarged difference in the reaction velocity between Reactions (1) and (2) with $x \geq 4$.

Fig. 3 plots measured combustion temperature profiles from samples of different powder mixtures. As shown in Fig. 3, the abrupt rise in temperature signifies the rapid arrival of the combustion wave and the peak value corresponds to the combustion front temperature. After the passage of the reaction front, an appreciable decrease in temperature is a consequence of the heat loss to the surroundings. Fig. 3 indicates a decrease in the reaction front temperature from about 1200 to 1057 °C as the stoichiometric parameter x of Reaction (1) increases from

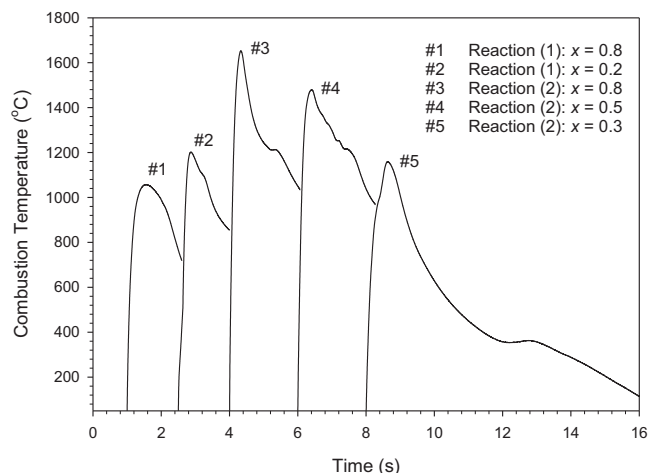


Fig. 3. Dependence of combustion temperature on Nb content of $(\text{Ti}_{1-x}\text{Nb}_x)_2\text{AlC}$ solid solutions synthesized from samples composed of different reactant mixtures.

0.2 to 0.8, confirming the reduction of reaction exothermicity with increasing metallic Nb. However, the peak combustion temperature of Reaction (2) increases from 1160 to 1652 °C with increasing x value from 0.3 to 0.8. This suggests a boost of reaction exothermicity by increasing the thermite reagents, Nb_2O_5 and Al, in Reaction (2). Most importantly, the composition dependence of the combustion temperature for the samples of Reactions (1) and (2) is in a manner consistent with that of the reaction front velocity.

3.3. Composition and morphology analysis of combustion products

Fig. 4(a)–(c) depicts typical XRD patterns of the synthesized products from two different combustion patterns. As revealed in Fig. 4(a), the $(\text{Ti,Nb})_2\text{AlC}$ solid solution dominates the resulting product of an elemental powder compact and the minor phase TiC is trivial. As reported previously, TiC is a key intermediate in the formation of Ti_2AlC and Ti_3AlC_2 [41,42] and often exists as a secondary phase in the final product [32,33].

For the powder compacts composed of Ti, Nb_2O_5 , Al, and Al_4C_3 at $x=0.4$ and 0.7, the phase constituents of their respective products are unfolded in Fig. 4(b) and (c), both of which verify simultaneous formation of $(\text{Ti,Nb})_2\text{AlC}$ and Al_2O_3 via solid state combustion involving reduction of Nb_2O_5 by Al. The XRD analysis also detected two minor phases, TiC and Nb_2Al . The intermetallic compound Nb_2Al , an intermediate phase in the evolution of Nb_2AlC [34], could be

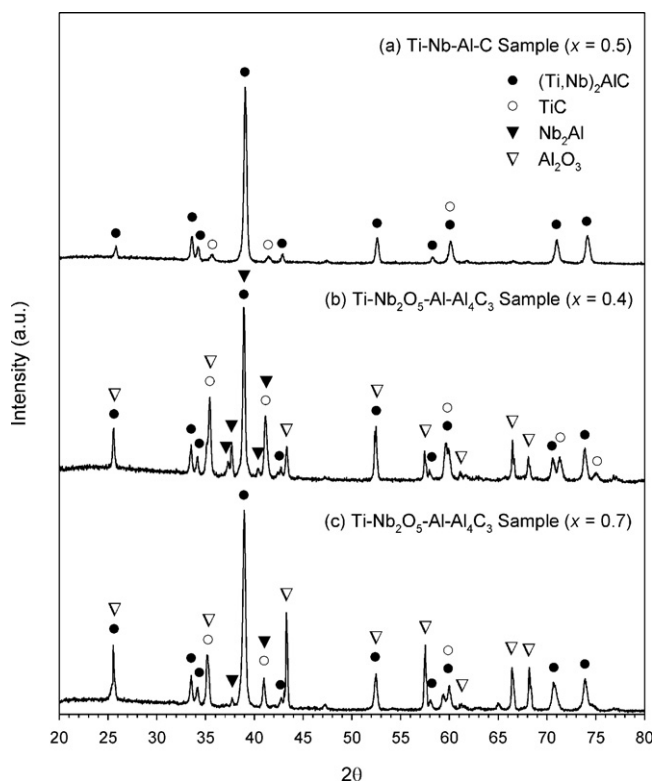
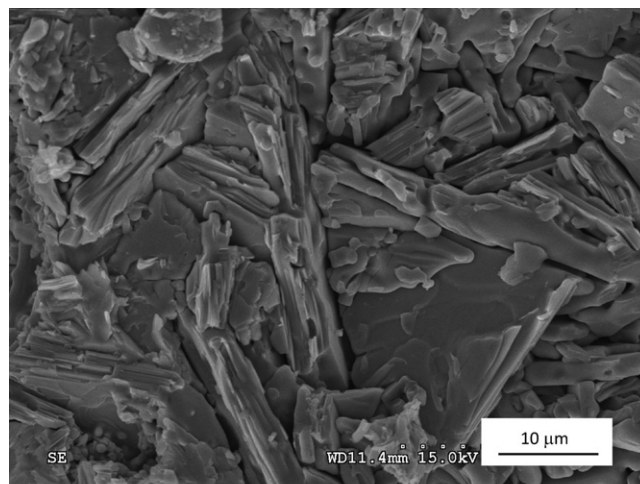
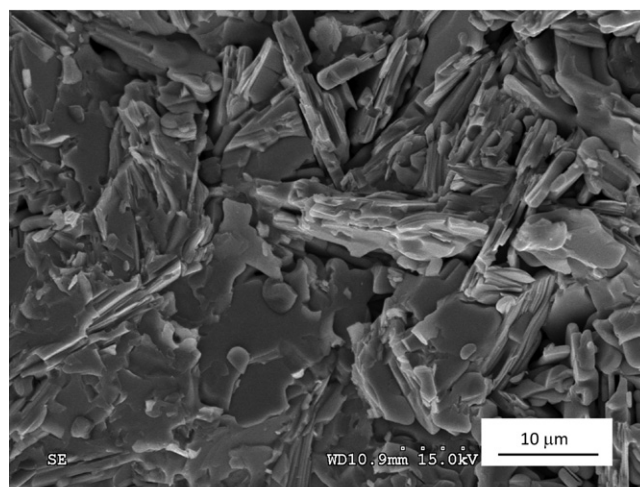


Fig. 4. XRD patterns of synthesized products from (a) Ti–Nb–Al–C sample with $x = 0.5$, (b) Ti– Nb_2O_5 –Al– Al_4C_3 sample with $x = 0.4$, and (c) Ti– Nb_2O_5 –Al– Al_4C_3 sample with $x = 0.7$.



(a)



(b)

Fig. 5. SEM micrographs illustrating fracture surfaces of products synthesized from (a) Ti–Nb–Al–C sample with $x = 0.5$ and (b) Ti– Nb_2O_5 –Al– Al_4C_3 sample with $x = 0.5$.

yielded from the interaction of reduced Nb with Al and/or Al_4C_3 . With the increase of thermite reagents in the reactant mixture, as indicated in Fig. 4(c), both TiC and Nb_2Al present in the final product were diminished and a better degree of $(\text{Ti,Nb})_2\text{AlC}$ formation was achieved. This is most likely attributed to the enhanced reaction exothermicity stemming from more thermite reactions involved in the combustion synthesis.

Typical microstructures of the products synthesized from the elemental powder compact and $\text{Nb}_2\text{O}_5/\text{Al}_4\text{C}_3$ -containing sample are presented in Fig. 5(a) and (b), respectively. Fig. 5(a) shows that $(\text{Ti,Nb})_2\text{AlC}$ grains in the shape of thin plates are closely stacked into a laminated structure. The layered structure characteristic of the MAX ternary carbide is also evident in Fig. 5(b), which is associated with a $(\text{Ti,Nb})_2\text{AlC}/\text{Al}_2\text{O}_3$ composite.

The density of monolithic $(\text{Ti,Nb})_2\text{AlC}$ solid solutions obtained in this study increases with Nb content and varies from 3.4 to 4.2 g/cm³. When compared with those of bulk Ti_2AlC (4.11 g/cm³) and Nb_2AlC (6.37 g/cm³), the densities are relatively low because of the porous structure inherent in the SHS-derived products. For the in situ formation of Al_2O_3 -added $(\text{Ti,Nb})_2\text{AlC}$, the melting of the sample during combustion contributes to densification of the final product, and therefore, the density increases up to 4.3–5.6 g/cm³.

4. Conclusions

Preparation of monolithic and Al_2O_3 -added $(\text{Ti}_{1-x}\text{Nb}_x)_2\text{AlC}$ solid solutions with $x = 0.2$ – 0.8 was conducted by combustion synthesis in the SHS mode. The yield of nearly single-phase $(\text{Ti,Nb})_2\text{AlC}$ was achieved by direct combustion of constituent elements. The as-synthesized product exhibits lamellate grains typical of the MAX carbide. It was found that the reaction exothermicity decreased with increasing Nb content of $(\text{Ti,Nb})_2\text{AlC}$, resulting in the decrease of both combustion temperature and reaction front velocity.

For the in situ formation of $(\text{Ti,Nb})_2\text{AlC}/\text{Al}_2\text{O}_3$ composites, the sample compacts were composed of Ti, Al, Nb_2O_5 , and Al_4C_3 powders and the corresponding combustion involved aluminothermic reduction of Nb_2O_5 . The increase of thermite reagents, Nb_2O_5 and Al, in the combustion system increased not only the content of Nb in $(\text{Ti}_{1-x}\text{Nb}_x)_2\text{AlC}$, but also the mole fraction of Al_2O_3 in the final composite. Due to the highly exothermic nature for the thermite reduction of Nb_2O_5 by Al, the combustion temperature and reaction front velocity increased with increasing thermite reagents. In addition to $(\text{Ti,Nb})_2\text{AlC}$ and Al_2O_3 , the as-synthesized composite contains two intermediates TiC and Nb_2Al . Similarly, the $(\text{Ti,Nb})_2\text{AlC}/\text{Al}_2\text{O}_3$ composite is overwhelmed by a laminated microstructure.

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