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Preparation of borides in Nb–B and Cr–B systems by combustion synthesis involving borothermic reduction of Nb₂O₅ and Cr₂O₃

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ABSTRACT

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Keywords: Ceramics Solid-state reactions X-ray diffraction Combustion synthesis Transition metal borides An experimental study on the preparation of metal borides in the Nb-B and Cr-B systems was conducted by self-propagating high-temperature synthesis (SHS) involving the reduction of Nb₂O₅ and Cr₂O₃ by amorphous boron. The starting stoichiometry of the reactant compact was shown to make a great impact on the combustion behavior and the phase composition of the final product. For the powder compacts of Nb₂O₅ and boron, self-sustaining combustion was performed under a molar ratio of B/Nb₂O₅ between 5 and 10, but complete reduction of Nb₂O₅ was achieved when $B/Nb_2O_5 \ge 8$. Partial reduction of Nb₂O₅ caused a decrease in the combustion temperature and velocity, and was responsible for the presence of NbO₂ in the final products. For the samples with stoichiometry of $6 \le B/Nb_2O_5 \le 8$, three boride phases NbB, Nb₃B₄, and NbB₂ were synthesized. An increase in the boron content up to B/Nb₂O₅ = 8.5-10 resulted in not only full reduction of Nb₂O₅, but also formation of single-phase NbB₂. On the other hand, the SHS process involving Cr_2O_3 and boron was feasible for the powder compacts of $4 \le B/Cr_2O_3 \le 9$, wherein the highest combustion temperature and the fastest reaction front were observed in the compact with $B/Cr_2O_3 = 6$. During combustion Cr_2O_3 was fully reduced, leading to the formation of three borides Cr_5B_3 , CrB, and CrB₂ in either monolithic or composite form. With a boron content more than the stoichiometric amount, the powder compacts of $B/Cr_2O_3 = 4$, 5, and 9 yielded single-phase Cr_5B_3 , CrB, and CrB₂, respectively.

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1. Introduction

Borides of the transition metals are very attractive materials, due to a unique combination of favorable properties like high melting point, high hardness, high mechanical strength, high electrical conductivity, good chemical stability, and excellent wear and corrosion resistance [1–3]. Most transition metal borides feature many stoichiometric compositions. For example, five niobium borides (Nb₃B₂, NbB, Nb₅B₆, Nb₃B₄, and NbB₂) and six chromium borides (Cr₂B, Cr₅B₃, CrB, Cr₃B₄, CrB₂, and CrB₄) have been reported in the Nb–B and Cr–B systems, respectively [4]. Among them, NbB₂ is promising for high-temperature structural applications [5] and has been recognized as a superconductor [6,7]. CrB₂ shows potential not only as a high-temperature structural material [3], but also as a hard coating or a protective layer on tools and materials exposed to wear and corrosion [8,9].

Many transition metal borides have been fabricated by the solidstate reaction between boron and metal powders [10-13], and by the reduction of metal oxides with boron [14,15] or boron carbide [16] as the reducing agent. Matsudaira et al. [10] produced NbB and NbB₂ by heating amorphous boron and niobium powders of their equivalent compositions at 1000 °C for 60 min and obtained Nb₃B₄ under a higher temperature of 1800 °C. According to Iizumi et al. [12,13], both CrB and CrB₂ have been synthesized in pure form by milling chromium and boron powders in a planetary ball mill for 20–40 h, followed by annealing at 900–1000 °C. Peshev et al. [14,15] performed a series of experiments on the borothermic reduction of metal oxides between 1000 and 1750 °C to prepare the corresponding metal borides, including CrB₂, Mo₂B₅, W₂B₅, VB₂, NbB₂, and TaB₂. In addition, Sonber et al. [16] fabricated CrB₂ through the reaction of Cr₂O₃ with boron carbide at 1500–1700 °C in the presence of carbon.

In contrast to the aforementioned manufacturing routes that are generally time-consuming and energy-intensive, combustion synthesis in the mode of self-propagating high-temperature synthesis (SHS) takes advantage of the self-sustaining merit from highly exothermic reactions and hence has the potential of time and energy savings [17–19]. The SHS technique has been effectively applied to produce a variety of advanced materials, including borides, carbides, nitrides, hydrides, silicides, and intermetallics, as well as composites on their bases [17–19]. Production of the transition metal borides by SHS is typically through the direct reaction of mixed constituent elements in a sample compact, by which numerous borides such as TiB₂, ZrB₂, NbB₂, TaB₂, HfB₂, NbB, MoB, and

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Fig. 1. Recorded combustion images illustrating SHS processes of powder compacts with stoichiometric ratios of (a) Nb₂O₅:B=1:8.5 and (b) Cr₂O₃:B=1:6.

TaB have been obtained [20–25]. However, when direct combustion between the metal and boron is not feasible, the SHS process involving borothermic reduction of a metal oxide has been considered as an alternative of preparing the metal boride. For example, three borides of molybdenum (Mo₂B, MoB₂, and Mo₂B₅) were produced by SHS from the reactant compacts composed of MoO₃, Mo, and B powders under different stoichiometries [26].

The objective of this study is to investigate the production of metal borides in the Nb–B and Cr–B systems through the reaction of metal oxides (Nb₂O₅ and Cr₂O₃) with amorphous boron in the form of self-propagating combustion. In contrast to niobium borides which have been produced by SHS from elemental powder compacts [24], direct combustion between Cr and boron is not feasible. However, formation of borides of niobium and chromium has not been well characterized by SHS involving borothermic reduction of the relevant metal oxides. For both combustion systems, this study gives emphasis to establish the relation between the boride phase formed and the initial sample composition (i.e., the molar ratios of B/Nb₂O₅ and B/Cr₂O₃). In addition, the effects of sample stoichiometry were explored on sustainability of the combustion process, propagation velocity of the combustion front, and combustion temperature.

2. Experimental methods of approach

Niobium oxide Nb₂O₅ (Strem Chemicals, 99.9% purity), chromium oxide Cr₂O₃ (Showa Chemical Co., 99% purity) and amorphous boron (Noah Technologies Corp., 92% purity) were employed as the starting materials. Similar to typical amorphous boron, the major impurities in the boron powders include magnesium (Mg) about 5.0%, water soluble boron (0.50%) and moisture (0.50%). In view of five boride phases

 $(Nb_3B_2, NbB, Nb_5B_6, Nb_3B_4, and NbB_2)$ in the Nb–B system, the powder mixtures of Nb_2O_5 and boron were formulated with proportions of a broad range. As expressed in reactions (1) and (2), Nb_3B_2 and NbB_2 can be stoichiometrically formed from the samples with a molar ratio of $B/Nb_2O_5 = 14/3$ and 22/3, respectively.

$$3Nb_2O_5 + 14B \rightarrow 2Nb_3B_2 + 5B_2O_3$$
 (1)

$$3Nb_2O_5 + 22B \rightarrow 6NbB_2 + 5B_2O_3$$
 (2)

In order to cover the scope denoted by reactions (1) and (2), this study takes on a span of the stoichiometric ratio from $B/Nb_2O_5 = 4$ to 10 for the synthesis of various niobium borides. In the case of preparing chromium borides, the existence of six different phases (Cr₂B, Cr₅B₃, CrB, Cr₃B₄, CrB₂, and CrB₄) in the Cr–B system signifies a requisite of the proportion of B/Cr_2O_3 from 3 to 10, as described in reactions (3) and (4).

$Cr_2O_3 + 3B \rightarrow$	$Cr_2B + B_2O_3$	(3)	1

$$Cr_2O_3 + 10B \rightarrow 2CrB_4 + B_2O_3 \tag{4}$$

The reactant powders with a prescribed composition were dry mixed in a ball mill and then cold-pressed into cylindrical test specimens with a diameter of 7 mm and a height of 12 mm. The compaction density was 50% relative to the theoretical maximum density (TMD) for the Nb₂O₅-B samples and 45% TMD for the Cr₂O₃-B compacts. The SHS experiment was conducted in a stainless-steel windowed chamber under an atmosphere of high-purity argon (99.99%). The sample holder is equipped with a 600 W cartridge heater used to raise the initial temperature of the sample prior to ignition. It was found that a preheating temperature of 300 °C was required for both types of the samples to assure self-sustaining combustion. Moreover, a Ti-C pellet made up of the powder blend with an atomic ratio of Ti:C=1:1 was placed on the top of the test specimens to serve as an ignition enhancer which was triggered by a heated tungsten coil. Details of the experimental setup and measurement approach were reported elsewhere [27]. It should be noted that according to Ref. [26], the by-product B2O3 yielded from the displacement reaction is voluntarily expelled in the form of very tiny liquid droplets from the porous sample during the SHS process, thus resulting in the absence of B₂O₃ in the final products.



Fig. 2. Effect of sample stoichiometry on flame-front propagation velocity of $Nb_2O_5\text{--}B$ powder compacts.

3. Results and discussion

3.1. Observation of combustion characteristics

Fig. 1(a) and (b) illustrates typical SHS sequences associated with solid-state combustion of the Nb₂O₅–B and Cr₂O₃–B powder compacts, respectively. It is evident in Fig. 1(a) and (b) that a distinct combustion front forms upon initiation and propagates along the sample in a self-sustaining and nearly parallel manner. Moreover, a brighter burning glow and a faster combustion front are observed for the Nb₂O₅–B powder compact, implying a more exothermic reaction between Nb₂O₅ and boron when compared to that of Cr₂O₃ with boron.

However, the experimental observations indicated that the reaction was quenched shortly after the ignition for the powder compacts with $B/Nb_2O_5 = 4.0$ and 4.67. This is due most likely to a lack of sufficient reaction heat to sustain the combustion for the samples of such low boron contents. On the other hand, the Cr_2O_3 -B samples were shown to possess a flammability limit of self-sustaining combustion residing in $4 \le B/Cr_2O_3 \le 9$, beyond which combustion ceased to propagate.

3.2. Measurement of flame-front propagation velocity

The propagation velocity (V_f) of the combustion front was determined from the recorded SHS images. It was found for both types of the powder compacts that the combustion velocity was significantly affected by the reactant composition. As shown in Fig. 2, the reaction front velocity of the Nb₂O₅-B compact first increases with increasing B/Nb₂O₅ ratio, approaches to a maximum about 3.9 mm/s at $B/Nb_2O_5 = 8.5$, and then decreases with further increase of the boron content in the reactant mixture. It is believed that for the samples of Nb₂O₅ and boron, the extent of borothermic reduction of Nb₂O₅ is responsible for the variation of combustion velocity with sample stoichiometry. Based upon the XRD analysis of synthesized products to be presented later, an incomplete reduction of Nb₂O₅ occurred in the samples of B/Nb₂O₅ < 8. Partial reduction of Nb₂O₅ yields another metal oxide NbO₂ and liberates less reaction heat, which causes a decrease in the propagation rate of the combustion wave. The degree of borothermic reduction of Nb₂O₅ was enhanced by increasing the proportion of boron to Nb_2O_5 in the powder mixture. This accounts for an increase in the flame-front velocity with increasing B/Nb₂O₅ ratio from 5.0 to 8.5. However, a moderate decrease in the combustion velocity is observed in Fig. 2



Fig. 3. Effect of sample stoichiometry on flame-front propagation velocity of $C_{\Gamma_2}O_3$ -B powder compacts.

for the reactant compacts containing boron higher than the proportion of $B/Nb_2O_5 = 8.5$. Such a decrease is believed to be a result of the dilution effect produced by an excessive amount of boron.

The influence of sample stoichiometry on the flame-front velocity of the Cr_2O_3 -B powder compact is presented in Fig. 3. With the increase of the molar ratio of B/Cr₂O₃ from 4 to 9, the combustion wave velocity increases from 0.86 mm/s to a peak value of 2.56 mm/s at B/Cr₂O₃ = 6 and decreases afterward to about 1.25 mm/s. No combustion was achieved for the samples with compositions out of this range. Unlike the reaction of Nb₂O₅ with boron, Cr₂O₃ was completely reduced by boron during combustion. Therefore, the composition dependence of combustion front velocity of the Cr₂O₃-B sample is attributed primarily to an alteration in the reaction exothermicity associated with formation of different borides of chromium in the final products.

3.3. Measurement of combustion temperature

Five temperature profiles recorded from solid-state combustion of the Nb₂O₅–B powder compacts with different initial stoichiometries are depicted in Fig. 4, where the abrupt rise in temperature signifies rapid arrival of the combustion front and the peak value represents the reaction front temperature. As shown in Fig. 4, the combustion front temperature increases from 1048 to 1380 °C with



Fig. 4. Measured combustion temperature profiles of Nb₂O₅–B powder compacts with different stoichiometric ratios.



Fig. 5. Measured combustion temperature profiles of Cr₂O₃–B powder compacts with different stoichiometric ratios.

increasing B/Nb_2O_5 ratio from 5.0 to 8.5, but declines slightly to 1343 °C as the stoichiometric ratio further increases to 9.0. As mentioned above, the rise is due to an improvement in the borothermic reduction of Nb_2O_5 and the fall is caused by the dilution effect of excessive boron.

When compared with those of the Nb₂O₅–B samples, Fig. 5 reveals considerably lower flame-front temperatures varying between 675 and 830 °C for the Cr_2O_3 –B samples. This confirms that the reaction of Cr_2O_3 with boron is less exothermic than that of Nb₂O₅ with boron. In agreement with the composition dependence of combustion velocity, the combustion front temperature of the Cr_2O_3 –B sample increases with boron concentration to a maximum at B/Cr₂O₃ = 6 and then decreases with further increase of boron.

3.4. Composition and morphology analysis of combustion products

Fig. 6(a)-(c) plots XRD spectra of the products obtained from the reactant compacts of Nb₂O₅:B = 1:5, 1:7, and 1:9. The presence of NbO₂ in Fig. 6(a) and (b) signifies partial reduction of Nb₂O₅. A noticeable decrease in the peak intensity of NbO₂ is observed in Fig. 6(b), implying a substantial improvement in the reduction of niobium oxides by the sample with a larger amount of boron. In addition, as shown in Fig. 6(a), NbB is the only boride phase formed from combustion. Fig. 6(b) indicates the formation of multiple boride compounds, including the dominant phase NbB₂ and two secondary phases NbB and Nb₃B₄. For the sample of Nb₂O₅:B = 1:9, Fig. 6(c) shows not only a full reduction of the metal oxide, but also a complete conversion to single-phase NbB₂.

A summary of the product composition with respect to the reactant stoichiometry for the Nb₂O₅–B samples is given in Table 1. An increase in the molar ratio of B/Nb₂O₅ was found to enhance the borothermic reduction of Nb₂O₅, but a complete reduction was not observed until the stoichiometric ratio reached up to B/Nb₂O₅ = 8. For the samples with a low boron content like Nb₂O₅:B = 1:5 and 3:16, NbB is the only boride formed along with a significant amount of NbO₂. Within a composition range of B/Nb₂O₅ = 6–8, the synthesized products consist of three boride compounds (NbB₂, Nb₃B₄, and NbB) and the dominant phase changes from NbB to NbB₂ as the ratio of B/Nb₂O₅ = 8.5–10 led to formation of single-phase NbB₂. It is interesting to note that in this study the lowest amount of boron required for the production of pure NbB₂ is B/Nb₂O₅ = 8.5, which is greater than the stoichiometric quantity of B/Nb₂O₅ = 7.33 (22/3)



Fig. 6. XRD patterns of products synthesized from solid-state combustion of powder compacts with molar ratios of Nb_2O_5 :B = (a) 1:5, (b) 1:7, and (c) 1:9.

given in reaction (2). The need of an excessive amount of boron was partly to compensate the relatively low purity of amorphous boron about 92%, and in part to account for the loss of boron due to evaporation during the SHS process.

For the solid-state combustion involving Cr_2O_3 and boron, Fig. 7(a), (b), and (e) indicates that single-phase Cr_5B_3 , CrB, and CrB₂ were practically produced from the powder compacts of B/Cr₂O₃ = 4, 5, and 9, respectively. In agreement with that observed in the synthesis of NbB₂, an additional amount of boron was required for the production of pure Cr_5B_3 , CrB, and CrB₂ when compared to the corresponding stoichiometric quantity. In addition, Fig. 7(c) and (d) reveals that the reactant compacts of B/Cr₂O₃ = 6 and 7 yielded the mixtures of CrB and CrB₂.

As summarized in Table 2, the SHS process of the Cr_2O_3 –B samples produces three boride phases, Cr_5B_3 , CrB, and CrB_2 , and each of them can be obtained in pure form as mentioned above. It is useful to note that the sample of Cr_2O_3 :B=3:14 produces a CrB– Cr_5B_3 composite rather than single-phase Cr_3B_4 that represents the

Table 1

Summary of phase composition of synthesized products with respect to their initial molar ratio of Nb_2O_5 to B.

StoichiometryNb ₂ O ₅ :B	Phase composition of synthesized products		
	Dominant boride	Secondary boride(s)	Oxide
1:5	NbB		NbO ₂
3:16	NbB		NbO ₂
1:6	NbB	Nb_3B_4 , NbB_2	NbO ₂
1:7	NbB ₂	Nb ₃ B ₄ , NbB	NbO ₂
3:22	NbB ₂	Nb ₃ B ₄ , NbB	NbO ₂
1:8	NbB ₂	Nb ₃ B ₄ , NbB	
1:8.5	NbB ₂		
1:9	NbB ₂		
1:10	NbB ₂		



Fig. 7. XRD patterns of products synthesized from solid-state combustion of powder compacts with molar ratios of Cr_2O_3 :B=(a) 1:4, (b) 1:5, (c) 1:6, (d) 1:7, and (e) 1:9.

Table 2

Summary of phase composition of synthesized products with respect to their initial molar ratio of Cr₂O₃ to B.

Stoichiometry Cr ₂ O ₃ :B	Phase composition of synthesized products		
	Dominant boride	Secondary boride	
1:4	Cr ₅ B ₃		
3:14	CrB	Cr ₅ B ₃	
1:5	CrB		
1:6	CrB	CrB ₂	
1:7	CrB ₂	CrB	
1:8	CrB ₂	CrB	
1:9	CrB ₂		



Fig. 8. A typical SEM micrograph showing morphology of niobium borides synthesized from a sample compact of Nb_2O_5 :B = 1:9.

boride phase based upon stoichiometric balance under such a starting composition. For the powder compacts of $B/Cr_2O_3 = 6$, 7, and 8, the other composite product composed of CrB and CrB₂ was obtained.

A typical SEM micrograph is illustrated in Fig. 8, which reveals the morphology of niobium borides synthesized from a sample compact of Nb₂O₅:B=1:9. As displayed in Fig. 8, the product is porous and consists of softly agglomerated boride grains with a small particle size about $1-3 \mu$ m. The morphology of chromium borides obtained from the Cr₂O₃-B samples is very similar to that shown in Fig. 8.

4. Conclusions

This study presents an experimental investigation on the preparation of borides of niobium and chromium through combustion synthesis involving borothermic reduction of Nb_2O_5 and Cr_2O_3 . The starting stoichiometry of the reactant compact was shown to considerably affect the combustion behavior and the phase composition of the final product.

For the production of niobium borides, the SHS process was conducted with Nb₂O₅–B powder compacts formulated by a molar ratio of B/Nb₂O₅ between 5 and 10. The degree of borothermic reduction of Nb₂O₅ was improved by increasing the boron content in the reactant mixture, which enhanced the reaction exothermicity and hence accelerated the combustion wave. Complete reduction of Nb₂O₅ was achieved in the samples of B/Nb₂O₅ \geq 8, below which the partial reduction yielded another oxide NbO₂ in the final products. Upon a full reduction of the niobium oxide, the solid-state combustion of the sample with B/Nb₂O₅ = 8 resulted in formation of an NbB₂-dominated product with trivial amounts of NbB and Nb₃B₄. Moreover, single-phase NbB₂ was obtained from the powder compacts with proportions of B/Nb₂O₅ = 8.5, 9, and 10.

For the powder compacts of Cr_2O_3 and boron, self-sustaining combustion was feasible within a stoichiometry range from $B/Cr_2O_3 = 4$ to 9. Both combustion front temperature and velocity increased with boron content, reached their maxima at $B/Cr_2O_3 = 6$, and decreased with further increase of the B/Cr_2O_3 ratio. During the SHS process, Cr_2O_3 was fully reduced by boron and three boride phases Cr_5B_3 , CrB, and CrB_2 were produced in either monolithic or composite form, depending upon the initial composition of the sample. Based upon the XRD analysis, the powder compacts of $B/Cr_2O_3 = 4$, 5, and 9 yielded single-phase Cr_5B_3 , CrB, and CrB_2 , respectively. Two types of the composite products, $CrB-Cr_5B_3$ and $CrB-CrB_2$, were obtained from the samples of other stoichiometries.

This study demonstrates the feasibility of producing niobium and chromium borides through the SHS process involving borothermic reduction of the relevant metal oxides. This approach is especially appropriate for the reaction system, in which formation of the metal borides from direct combustion between metallic elements and boron is not feasible. In the future, this method can be worthily applied to prepare the borides of iron, nickel, tungsten, and manganese from the borothermic reduction of Fe₂O₃, NiO, WO₃, and MnO₂, respectively.

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