# MICROWAVE SYNTHESIS OF $B_4C$–$Al_2O_3$ COMPOSITE IN A MECHANICALLY ACTIVATED Al/$B_2O_3$/C POWDER MIXTURE

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***Abstract:*** *$B_4C$–$Al_2O_3$ composite powder was produced by aluminothermic reduction in Al/$B_2O_3$/C system. In this research, microwave heating technique was used to synthesize desired composite. The ball milling of powder mixtures was performed in order to study the effect of mechanical activation on the synthesis process. The synthesis mechanism in this system was investigated by examining the corresponding binary sub-reactions. The self-sustaining reduction of boron oxide by Al was recognized to be the triggering step in overall reaction.*

***Keywords:*** *Microwave, Composite, Combustion, Mechanial Activation*

## 1. INTRODUCTION

Boron carbide ($B_4C$) belongs to the hard materials class and is described as the hardest material after diamond and cubic boron nitride (C-BN) [1, 2]. $B_4C$ is considered to be suitable for use in various industries because of its excellent properties such as high melting point (2723 K), low density (2.52 g/cm$^3$), high elastic modulus, good chemical stability and high neutron absorption [1, 3, 4]. This carbide is a high potential candidate for several applications including cutting tools, wear resistant materials and neutron absorbers in nuclear reactors. Moreover, it can be used as a second phase (reinforcement) in ceramic and metal matrix composites [1]. For instance, it has been shown that the addition of 10-20 volume percent of $B_4C$ to alumina increases the toughness considerably [3]. High hardness and wear resistance of $B_4C$–$Al_2O_3$ composites make them appropriate selections for use in cutting tools and blasting nozzles [3, 5].

Several methods have been employed for boron carbide synthesis. Commercial $B_4C$ is generally produced by carbothermic reduction of boron oxide or boric acid in an electric arc furnace in a controlled atmosphere condition. This process requires very high temperature and long time because the reaction is very endothermic [1, 6]. In order to prepare a product using inexpensive initial materials and techniques, other methods such as vapor phase reactions, sol-gel and self-propagating high-temperature synthesis (SHS) have been therefore employed [7-10].

The self-propagating high-temperature synthesis (SHS) method is a synthesis technique which exploits the released heat of a highly exothermic reaction as a resource of energy for the progression of the reaction front. The reaction occurs between powder components to produce a large range of unique materials by controlling high-temperature combustion. The combustion Initiates either by a chemical route or an electrically heated element. Once initiated, the combustion is self-sustaining and proceeds by a combustion wave sweeping through the compacted material from the initiation side to the opposite side and will be completed in a time of a few seconds to a few minutes. With regard to the empirical Merzhanov's criterion, a reaction is considered to occur in an SHS manner when its adiabatic temperature (Tad) is higher than 1800 K [11-13]. SHS synthesis has several advantages including low fabrication cost, high efficiency, low energy consumption, short production time and feasibility of in-situ synthesis of ceramic composites, although the control of microstructure

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and properties of products is rather difficult [14]. Several researchers have used microwave heating source for synthesis of their desired compounds in a combustive manner [15, 16].

Combustion synthesis of $B_4C–Al_2O_3$ composite has been studied by a number of researchers. Won et al. [17] reported the production of $Al–Al_2O_3–B_4C$ composite layer by a combustion reaction between $B_2O_3$, carbon and different amounts of Al. The SHS reaction in the $Al–B_2O_3–C$ system was investigated by Yi et al. [18] with the aim of studying the effect of the density of initial compact on the porosity and mechanical properties of $B_4C–Al_2O_3$ composite. Liu et al. [19] discussed about the formation of aluminum borate ($9Al_2O_3.2B_2O_3$) as a by-product of combustion reaction during heating of the $4Al:2B_2O_3:C$ system. The main reason of the formation of this borate phase was explained to be the reaction between unreacted $B_2O_3$ and the produced $Al_2O_3$. The effect of mechanical activation on the combustion synthesis of $B_4C–Al_2O_3$ composite has been examined by Kahrizsangi and his co-worker [3] via using DTA analysis. The results showed that the onset temperature of the aluminothermic reaction decreased with increasing the milling time. Furthermore, once the particle size of $B_2O_3$ reduced and complete mixing of reacting powders during milling happened, the reaction kinetic was enhanced and the borate phase formation decreased.

In previous studies on the self-sustaining synthesis of $B_4C–Al_2O_3$ composite, the techniques used for initiating the combustion reactions have been either the heating in a furnace or inducing local ignition via electric arc. The use of microwave energy for heating materials and especially initiating combustion reactions has been suggested in recent years. Microwave heating has several advantages over other conventional heating methods, including very high heating rate, volumetric and homogeneous heating, selective heating and higher energy efficiency [20, 21]. Therefore, the goal of this work was to study the feasibility of SHS synthesis of $B_4C–Al_2O_3$ ceramic composite by means of microwave heating of the $Al–B_2O_3–C$ system which, to the author's knowledge, has not been reported so far. Furthermore, the effect of mechanical activation on stimulating the SHS reactions was examined.

## 2. EXPERIMENTAL

The starting materials used in this work were $B_2O_3$ (98%, Fluka, USA), Al (99%, MPY, Iran) and graphite (99%, Fluka, USA). The general web site of FactSage software (www.factsage.com) was used to obtain thermodynamic data. In some test runs, the blends of required materials were hand-ground in an agate mortar while the raw materials in other tests were mechanically mixed and activated by a

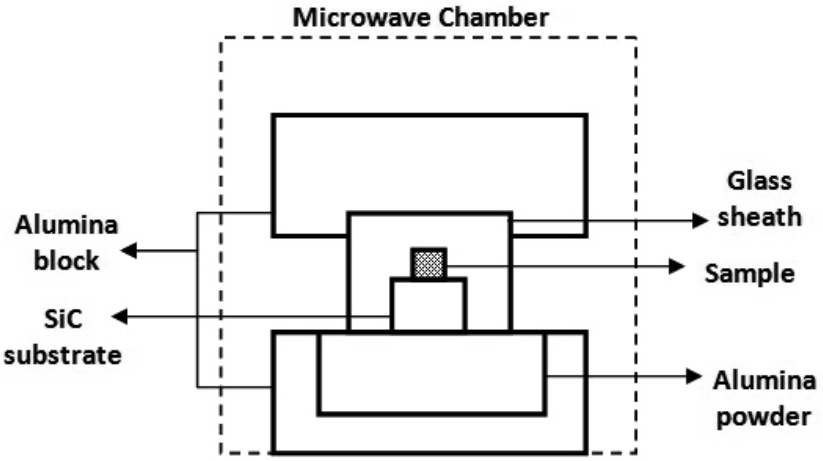


**Fig. 1.** Schematic representation of microwave setup.

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planetary ball mill. Ball milling was carried out under air atmosphere for up to 180 min at 250 rpm using a ball-to-powder mass ratio of 20:1. A uniaxial hydraulic press with a pressure of 500 psi was used to shape the mixtures of reagents into a disk of 10 mm in diameter and 5 mm in height. Microwave heating was carried out by a microwave oven (Samsung, model: GE2370G) with an external power of 850 W.

Figure 1 shows the schematic diagram of the synthesis setup used in this study. Because the initial materials had a low ability to absorb electromagnetic energy, i.e. microwave susceptibility, an SiC substrate was placed beneath the green compacts as a microwave susceptor. Due to the microwave absorption, the temperature of the SiC block increases quickly and hence, rapid heating of the specimen occurs. To restrict the presence of air during the combustion, a glass cover was used over the specimen. Alumina blocks were also utilized as holders. Ignition could be visually recognized by a brilliant combustion occurred inside the setup. To characterize the specimens, XRD (Philips X'Pert PW1800) and FTIR (Shimadzu 8400S) analyses were performed.

## 3. RESULTS AND DISCUSSION

Table 1 shows a detailed list of the experiments performed in this work. The observations of ternary system and its binary sub-systems are discussed in the following sections. Then, the effect of milling on the formation of desired product in these systems will be explained.

### 3. 1. Microwave Synthesis

#### *3. 1. 1. $B_2O_3$–C–Al System*

To synthesize the $B_4C$-$Al_2O_3$ composite, the following reaction was considered:

$$2\ B_2O_3 + C + 4\ Al = B_4C + 2\ Al_2O_3 \qquad (1)$$

$\Delta G^\circ_{298} = -837$ kJ, $\Delta H^\circ_{298} = -866$ kJ, $T_{ad} \approx 2330$ K

According to the calculated thermodynamic parameters, the above reaction shows a great

**Table 1.** Details of experimental tests performed on various systems.

| Specimen code | System | Mechanical activation duration (min) | Microwave heating duration (min) | Visual observation |
|---|---|---|---|---|
| X1 | $2B_2O_3$:C:4Al | 0 | 15 | No ignition |
| X2 | $2B_2O_3$:7C | 0 | 15 | No ignition |
| X3 | C:4Al | 0 | 15 | No ignition |
| X4 | $B_2O_3$:2Al | 0 | 15 | No ignition |
| X5 | $2B_2O_3$:C:4Al | 60 | 15 | No ignition |
| X6 | $2B_2O_3$:C:4Al | 120 | Until ignition moment | Incomplete propagation of combustion wave |
| X7 | $2B_2O_3$:C:4Al | 180 | Until ignition moment | Complete ignition |
| X8 | $2B_2O_3$:7C | 180 | 15 | No ignition |
| X9 | C:4Al | 180 | 15 | No ignition |
| X10 | $B_2O_3$:2Al | 180 | Until ignition moment | Complete ignition |
| X11 | $2B_2O_3$:C:4.4Al | 180 | Until ignition moment | Complete ignition |

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exothermicity and high tendency to occur. With regard to the Merzhanov's criterion for self-propagating reactions (Tad > 1800 K) [6], this reaction is expected to proceed in an SHS manner. A homogeneous mixture of starting materials was subjected to the microwave heating, as described before. Figure 2 shows the XRD pattern of the sample X1 after 15 min microwave heating. It can be observed that there was no chemical reaction between initial materials which had been confirmed by visual observations (Table 1). To investigate the reason for the lack of reaction during heating of Al–$B_2O_3$–C system despite the thermodynamic predictions, binary sub-systems including $B_2O_3$–C, C–Al and $B_2O_3$–Al were examined.

### *3. 1. 2. $B_2O_3$–C System*

The probable carbothermic reactions in this system are as follows:

$$2\ B_2O_3 + 7\ C = B_4C + 6\ CO\ (g) \quad (2)$$
$$\Delta G^\circ_{298} = 1504\ kJ,\ \Delta H^\circ_{298} = 1822\ kJ$$

$$2\ B_2O_3 + 4\ C = B_4C + 3\ CO_2\ (g) \quad (3)$$
$$\Delta G^\circ_{298} = 1144\ kJ,\ \Delta H^\circ_{298} = 1304\ kJ$$

Regarding to the above Gibbs energy and enthalpy values, these reactions are highly endothermic and have an extremely low possibility for occurring at room temperature. The XRD pattern of sample X2 after 15 minutes of microwave heating (Fig. 3) shows carbon and boron oxide leaving un-reacted, which is in conformity with visual observation. This is reasonable in accordance with the above thermodynamic predictions, which can be confirmed by the necessity of usage of very high temperatures (above 1500°C) and long times in the industrial processes for $B_4C$ production from $B_2O_3$/C mixture. It seems the temperature does not reach to such a high level inside the setup used in this work.

### *3. 1. 3. Al–C System*

The possible reaction between these two elements is as follows:

$$4\ Al + 3\ C = Al_4C_3 \quad (4)$$
$$\Delta G^\circ_{298} = -194\ kJ,\ \Delta H^\circ_{298} = -207\ kJ,\ T_{ad} = 1510\ K$$

The above reaction has a high tendency to occur. However, the adiabatic temperature of above reaction does not satisfy the Merzhanov's

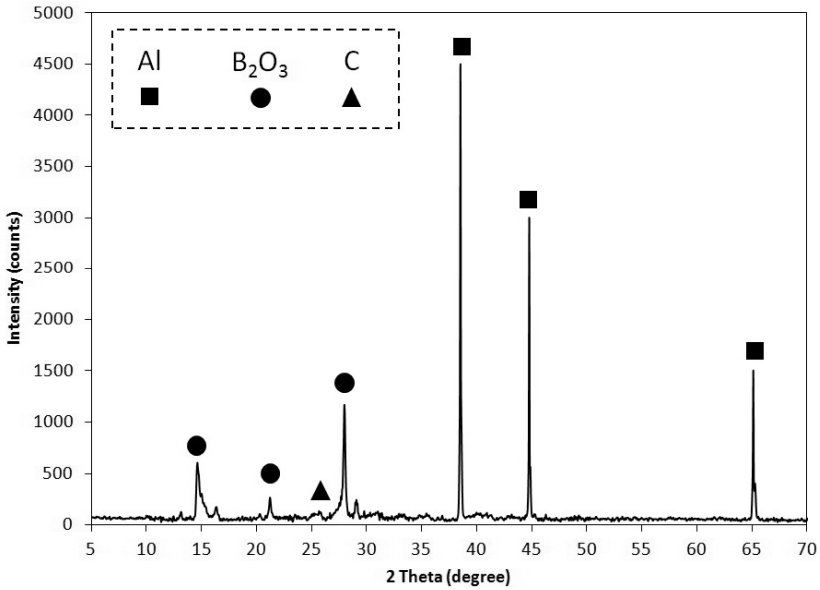


**Fig. 2.** X-ray diffraction pattern of the sample X1 after microwave heating for 15 min.

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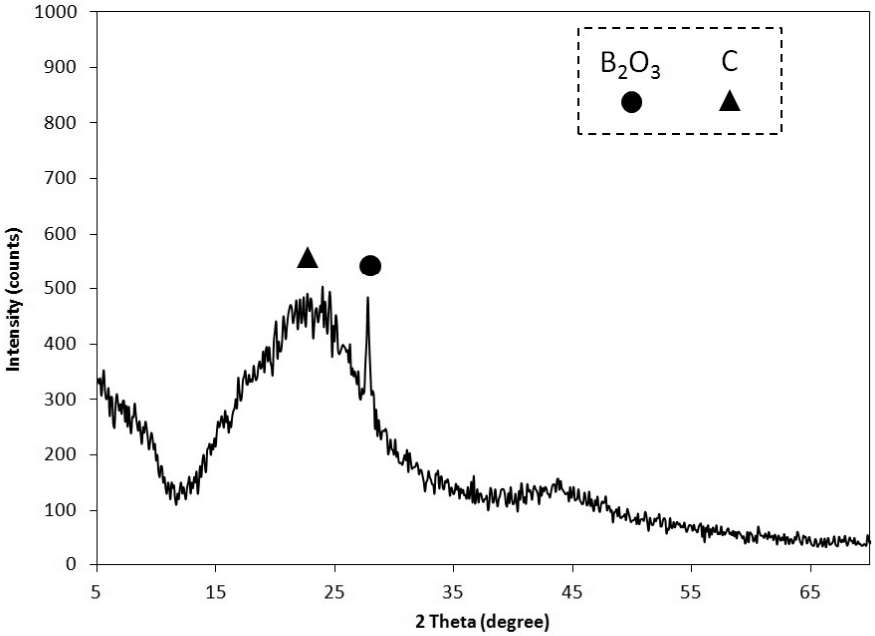


**Fig. 3.** X-ray diffraction pattern of the sample X2 after microwave heating for 15 min.

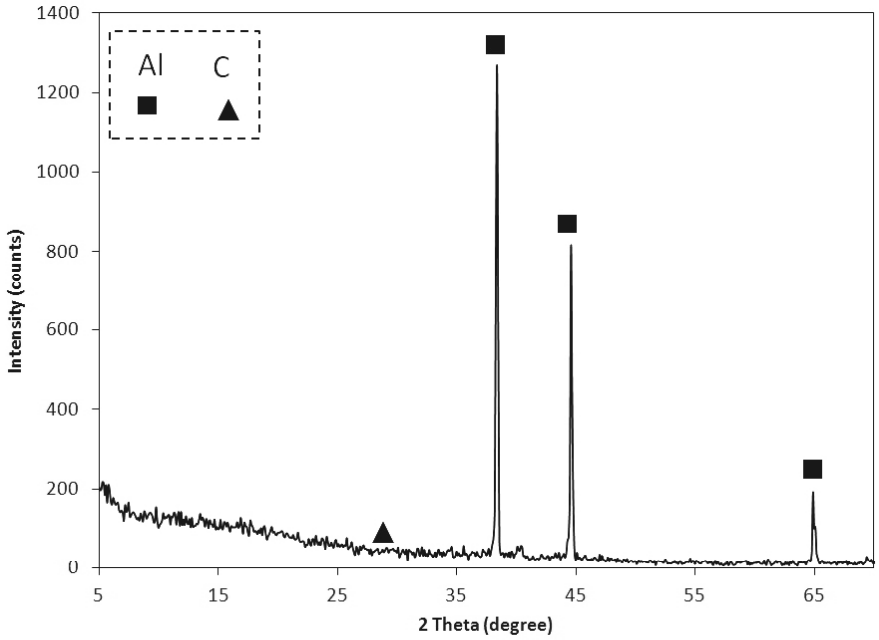


**Fig. 4.** X-ray diffraction pattern of the sample X3 after microwave heating for 15 min.

criterion for SHS synthesis and hence, this reaction is expected to be an ordinary, gradual one. A mixture (sample X3) with the same mole fraction used in the sample X1 (i.e. C:4Al) was subjected to the microwave heating. The XRD pattern taken after 15 min of microwave heating of the sample X3 (Fig. 4) shows un-reacted aluminum and carbon, revealing no reaction occurred which was also confirmed by our visual observation.

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### *3. 1. 4. $B_2O_3$–Al System*

The reaction of aluminothermic reduction of boron oxide is as follows:

$$B_2O_3 + 2\,Al = 2\,B + Al_2O_3 \quad (5)$$

$\Delta G^\circ_{298}$= -388 kJ, $\Delta H^\circ_{298}$= -402 kJ, $T_{ad}$ = 2327 KJ

Highly exothermicity of this reaction and its very high adiabatic temperature (Tad>1800 K) imply that the above reaction is expected to occur in an SHS manner. However, no ignition was visually observed after 15 min microwave heating of sample X4 (see Table 1), which is in conformity with the XRD pattern of this sample shown in Fig. 5. The lack of chemical reaction can be attributed to the low contact between reacting materials due to the coarseness of particles and low homogeneity of the mixture as well as the failure to provide enough activation energy during microwave heating.

### 3. 2. The Effect of Mechanical Activation

Mechanical milling is considered as a beneficial method which brings about the fragmentation of particles, preparation of a more homogeneous mixture and provision of an external energy which can be stored in the initial materials and increases the energy level of the system. These phenomena would result in the materials to be activated and facilitate the occurrence of the desired reaction.

### *3. 2. 1. $B_2O_3$–Al–C System*

To study the effect of mechanical activation of Al–$B_2O_3$–C system on the SHS synthesis during microwave heating, the samples milled for 60, 120 and 180 min (X5 to X7) were examined. As can be seen in Fig. 6, there was no reaction after 15 min microwave heating of initial materials milled for 60 min (sample X5). By increasing the milling time up to 120 min (sample X6), the reaction could proceed partially. Visual observation (Table 1) indicated that although the ignition occurred after 110 s microwave heating, combustion wave progressed incompletely and could not sweep the whole of the sample. The XRD pattern of the sample X6 (Fig. 6) shows that a fraction of starting aluminum remained in the sample un-reacted just after partial ignition moment, confirming the incompleteness of the synthesis process. It can be concluded that increasing the milling time gives rise to an increase in reacting particles contacts and can make the reaction begin. However up to 120 min of milling, initial materials did not activate to

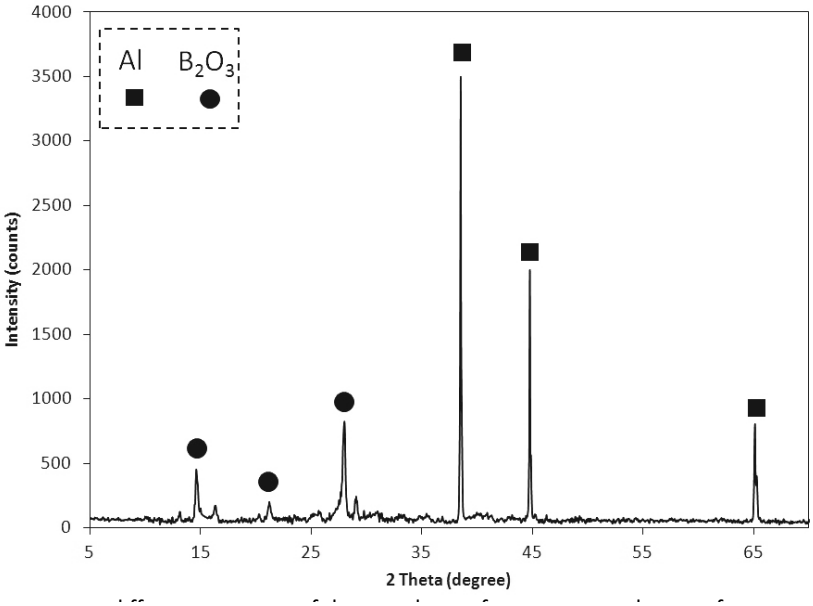


**Fig. 5.** X-ray diffraction pattern of the sample X4 after microwave heating for 15 min.

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such a level which could cause the reaction to occur completely. Therefore, the milling time was increased so that the desired reaction became complete. When the initial materials milled for 180 min (sample X7), an ignition occurred at 120 s of microwave heating and a complete combustion wave coverage was visually observed. Figure 6 shows the XRD pattern for the sample X7 which includes only alumina and boron carbide peaks, indicating the reaction (1) to occur completely with no remaining initial materials. The lower intensity of $B_4C$ peaks compared to $Al_2O_3$ ones can be attributed to the great difference of mass absorption coefficient (MAC) of these materials in the X-ray technique (2.75 and 30.91 $cm^2/g$ for $B_4C$ and $Al_2O_3$, respectively). The presence of a very small peak related to boron oxide in this XRD pattern can be an evidence for the deficiency of reducing agent (Al) in the mixture. Regarding to the high-temperature nature of the ignition, a slight fraction of Al may react with entrapped air and be oxidized into alumina inside the setup and hence, the reaction can encounter with an aluminum loss.

Infrared (IR) spectroscopy was used to identify the chemical bands of phases existed in the synthesized product to introduce another evidence for production of boron carbide together with alumina. Figure 7 shows the room temperature IR spectrum of the sample after microwave synthesis in the range of 350 to 3000 $cm^{-1}$. The sharp bands located at the range of 450–800 cm-1 are attributed to Al–O vibration in $Al_2O_3$ [3, 22-24], while the broad peak at around 1050 cm-1 is due to the infrared vibration of the B–C bond in boron carbide [3, 25-26]. The FTIR result, therefore, confirmed the synthesis of boron carbide–alumina composite after microwave processing.

The effect of increasing the milling time on the progression and completion of the reaction can be explained by an increase in structural defects and lattice strain as well as particle size reduction of reagents leading to the increase of contact areas.

### *3. 2. 2. Binary Sub-Systems*

The effect of milling was also studied on the binary systems of $B_2O_3$–Al, $B_2O_3$–C and Al–C. No major reaction was observed in the $B_2O_3$–C

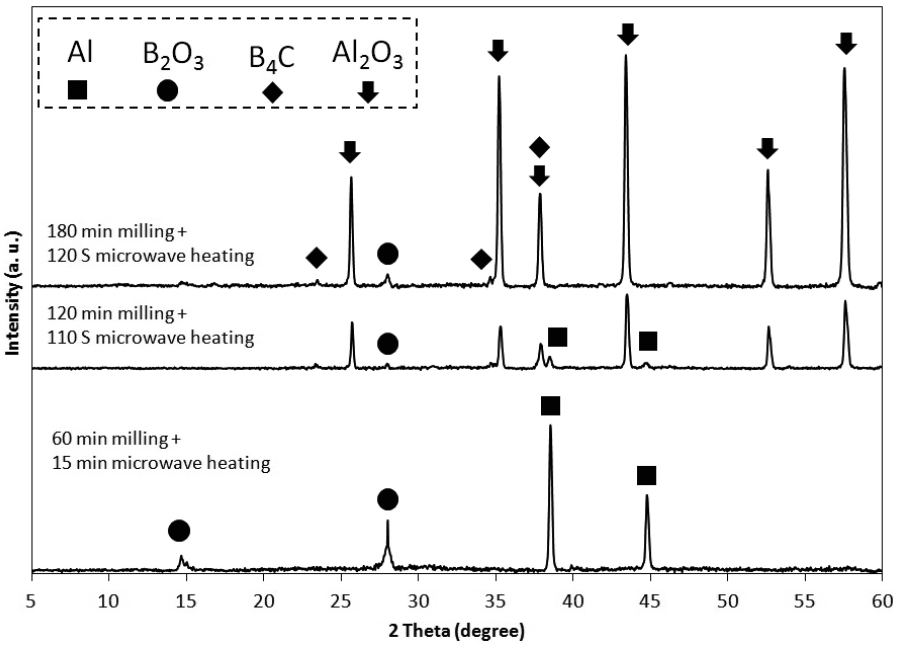


**Fig. 6.** X-ray diffraction patterns of microwave-heated samples X5, X6 and X7 after different hours of milling.

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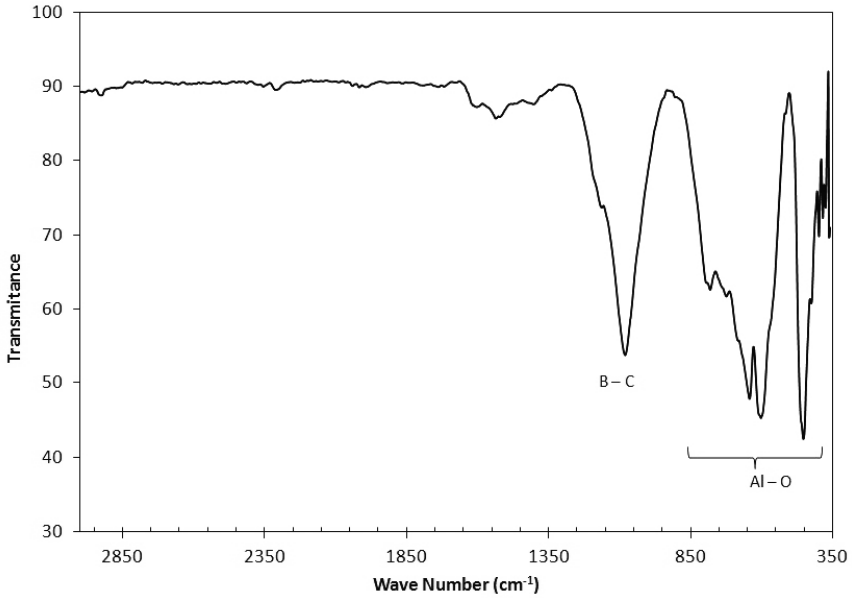


**Fig. 7.** FTIR spectrum of the sample X7 after 3h of ball milling followed by microwave synthesis.

sub-system (samples X8) after a milling for 3h followed by microwave heating for 15 min. In the case of Al–C sub-system (sample X9), a little aluminum carbide ($Al_4C_3$) was formed together with remaining initial materials during the same processing (3h milling, 15 min microwave heating). Although a slight amount of $Al_4C_3$ could be formed in the binary system, this phase was not observed in the ternary system (Al–$B_2O_3$–C) after ignition. This is because that from thermodynamic standpoint, the formation of $Al_2O_3$ is much more favorable than that of $Al_4C_3$ (see Fig. 8). However, an ignition was visually observed after 180 s microwave heating of the $B_2O_3$–Al sub-system milled for 3h (sample X10). Figure 9 shows the XRD pattern for this sample just after ignition moment. The presence of $Al_2O_3$ phase among product confirms the occurrence of aluminothermic reaction (reaction 5). Elemental boron cannot be detected in the XRD pattern mostly because of its amorphous nature due to the rapid cooling after ignition. As pointed out before, the aluminum loss during microwave process causes a slight amount of $B_2O_3$ to be remained un-reacted as can be seen in Fig. 9.

Fig. 9. X-ray diffraction pattern of 3h-milled sample X10 just after ignition during microwave heating.

Consequently, the sub-reaction between boron oxide and aluminum can be considered as the triggering step in the overall reaction 1. When Al, $B_2O_3$, and C are all present in one system and necessary energy for activation is provided (for example by mechanical milling), Al reduces boron oxide in a self-sustaining manner to yield elemental boron and alumina together with the release of a great deal of heat. The ignition that rise the system temperature can favor the reaction between boron and carbon to form boron carbide.

### 3. 3. The Effect of Additional Aluminum

To compensate for the aluminum loss occurred inside the setup during the ignition, some experiments were performed to investigate the effect of extra amount of aluminum on the completion of boron oxide reduction. It was observed that the addition of extra Al can reduce the amount of residual (i.e. un-reacted) boron oxide. The optimal amount of additional Al was found to be 10 molar percent. The XRD pattern of the sample X11 (Fig. 10) which relates to a

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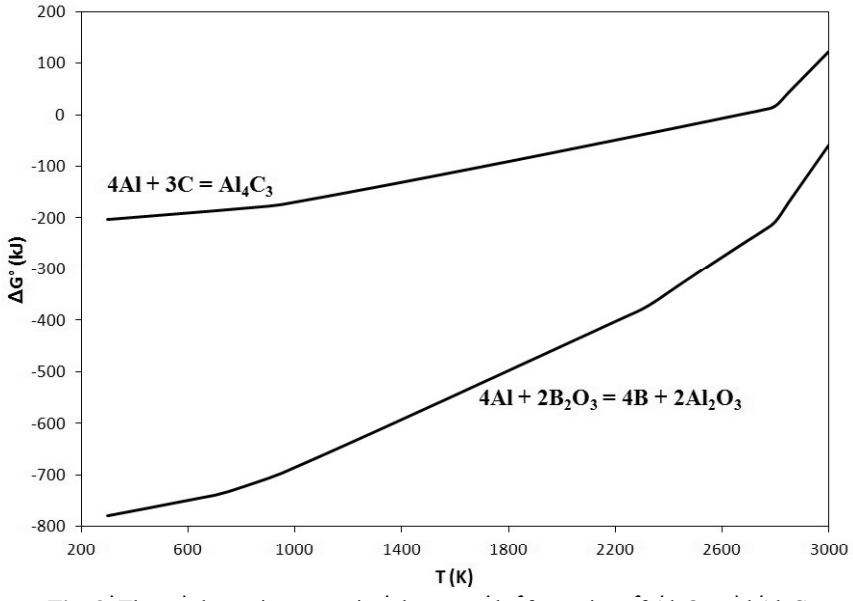


**Fig. 8.** Thermodynamic comparison between the formation of $Al_2O_3$ and $Al_4C_3$.

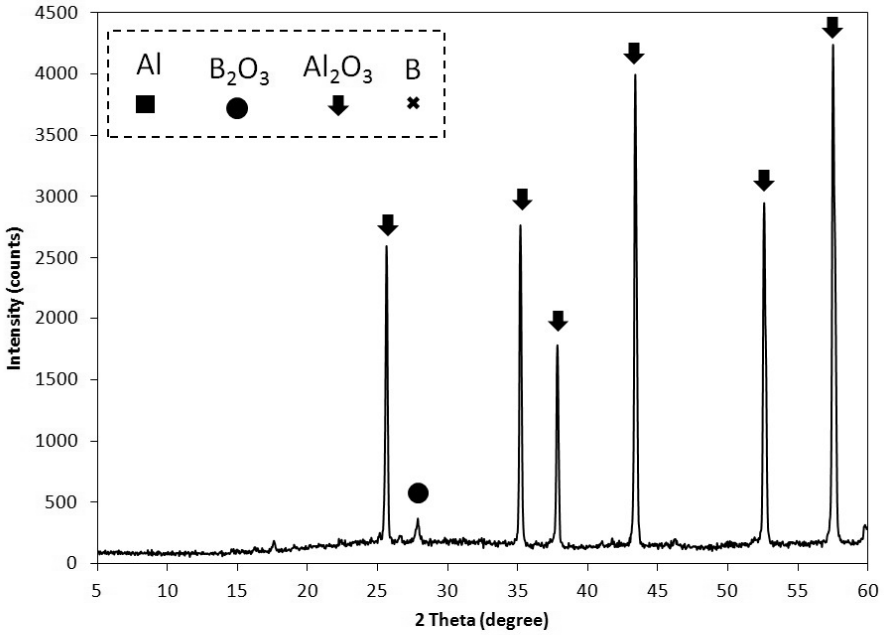


Fig. 9. X-ray diffraction pattern of 3h-milled sample X10 just after ignition during microwave heating.

**Fig. 8.** Thermodynamic comparison between the formation of $Al_2O_3$ and $Al_4C_3$.

ternary system with 10 % extra Al after 3h of milling followed by 120 s microwave heating shows that there is no longer residual boron oxide. Therefore, the remaining $B_2O_3$ was definitely due to the aluminum loss which can be overcome by adding extra reducing agent (Al) to the initial mixture.

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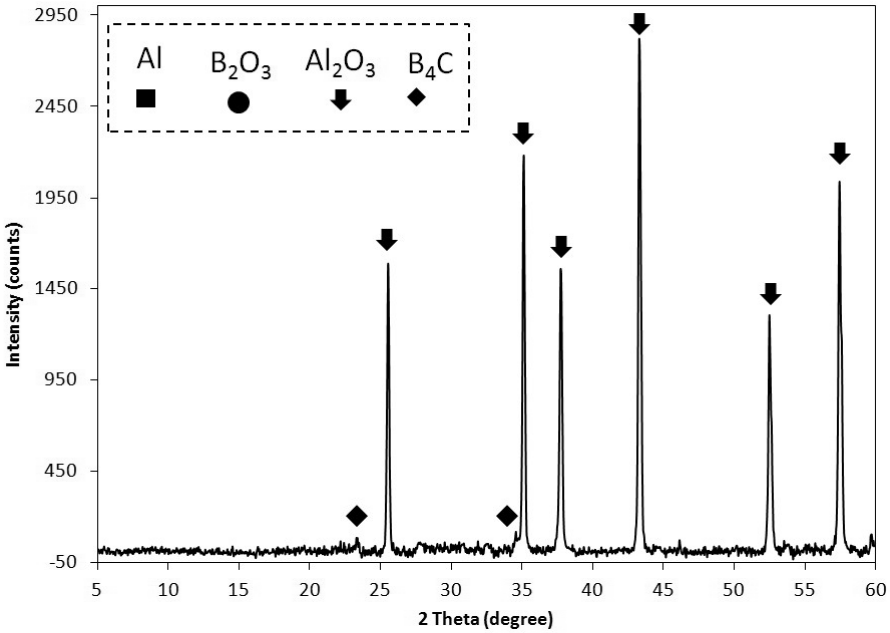


**Fig. 10.** X-ray diffraction pattern of 3h-milled sample X11 just after ignition during microwave heating.

## 4. CONCLUSION

Microwave heating technique was not by-itself able to initiate an SHS reaction in the $Al/B_2O_3/C$ system to produce boron carbide-alumina composite. However, the self-sustaining reaction occurred during microwave processing with an ignition time of a few seconds, when the initial mixture was mechanically activated for enough time (3 hours). Examination of the sub-reactions for understanding the governing mechanism revealed that boron oxide is preferably reduced by Al and hence, this sub-reaction can be considered as the triggering step in overall reaction. It was concluded that 10% extra Al is necessary to compensate for aluminum loss during the high-temperature ignition.

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