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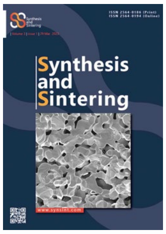

Research article

Influences of mechanical activation and tartaric acid addition on the efficiency of B_4C synthesis

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ABSTRACT

In this paper, mechanical activation and tartaric acid addition were employed to reduce the residual carbon and intensify the efficiency of B_4C synthesis using glucose and boric acid as starting materials. To investigate the role of mechanical activation on synthesis performance, one sample was subjected to high-energy ball milling before pyrolysis and the other after pyrolysis. To study the role of additives, in the precursor production stage, on synthesis efficiency and residual carbon reduction, different amounts of tartaric acid (0, 5, 10, 25, and 50 wt%) were tested. FT-IR and XRD analyses were used to characterize the bonds created in the precursors and the phases formed during the pyrolysis and synthesis steps, respectively. The results confirmed that mechanical activation before synthesis can improve the synthesis efficiency, but ball milling before pyrolysis did not significantly affect the final synthesis product. The addition of tartaric acid enhanced the formation of B–C bonds; hence, it increased the efficiency of B_4C synthesis. The optimum additive amount was 25 wt% and higher amounts weakened the synthesis performance.

KEYWORDS

B_4C
Synthesis efficiency
Mechanical activation
Tartaric acid addition
Residual carbon

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1. Introduction

Nowadays, there is a growing request for promising carbide ceramics, especially boron carbide (B_4C) owing to the collection of its unrivaled characteristics like low specific weight, extreme hardness, high melting point, supreme chemical stability, and outstanding Young's modulus [1–3]. These mentioned excellent properties have made B_4C a perfect choice for adaptable usages in the military, cutting and abrasive tools, aerospace, nuclear, and electronic industries [4, 5]. Recently, the applications expansion of B_4C in various industries has caused researchers to develop different methods for the synthesis of this material such as sol-gel [6], magnesiothermic reduction [7, 8], carbothermal reduction [9–11], microwave synthesis [12], co-reduction in the autoclave [13], and the elemental reaction of boron and carbon [14, 15], all of which are designed to achieve a wide range of particle sizes for prepared B_4C powder. Hence, controlling the morphology, size, and purity of the final product are some of the challenges of these techniques [16, 17]. On one hand, researchers try to reduce the synthesis temperature of B_4C, which leads to the degradation of mechanical properties and high energy consumption. On the other hand, the synthesis of high-purity B_4C is also another challenging problem with conventional methods. Hence, Optimizing the synthesis temperature and finding the appropriate processing method are the concerns of scientists [18–21].

Gubernat et al. [22] utilized fine amorphous boron and expanded graphite powders with the B to C in a mass ratio of 10:1 for the production of B_4C powder via direct synthesis. The powder with a crystallite size of 20–40 nm was attained. Indeed, the penetration of carbon in the form of CO toward boron was known as the mechanism of B_4C synthesis. Karaahmet et al. [23] obtained a reactant including of B_2O_3 and carbon via calcination of polymeric precursor, which was synthesized from industrial raw components through the sol-gel process. The mechanical modification process of the reactant reduced its particle size and enhanced its crystallinity. Heat treatment of

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reactant and mechanically modified carbon-based reactant (MMCBR) for 5 h at 1400–1700 °C under an argon atmosphere led to achieving a non-uniform powder morphology with rod-shaped B_4C particles. The B_4C powder prepared from the unmodified reactant had agglomerated particles, while no such problem was observed in the powder produced from MMCBR. More free carbon was seen in the B_4C obtained from MMCBR.

Kakiage [24] obtained crystalline B_4C powder using low-temperature synthesis by carbothermal reduction at a temperature of 1200 °C in Ar atmosphere. He formed B_2O_3/carbon structure using the thermal decomposition of a condensed H_3BO_3 and a polyol in air. Synthesis of B_4C with low residual carbon was accelerated by the improvement of homogeneity and dispersibility of the B_2O_3/carbon microstructure. Kakiage and his coauthors [25] have also successfully synthesized crystalline B_4C particles with no free carbon using a condensed H_3BO_3–glycerin by a carbothermal reduction method for 5 h at 1250 °C under Ar circulation. Dehydration of directly mixed H_3BO_3 and glycerin was utilized to produce such condensed precursor followed by air pyrolysis to achieve a product with no extra carbon.

Rafi-ud-din et al. [26] investigated the effects of ethylene glycol addition on the synthesis of B_4C powders via carbothermal reduction of borate citrate precursors. Using this additive leads to the facilitation of B_4C synthesis at a needed temperature of 1350 °C, which is 100–300 °C lower than that required for the synthesis of the additive-free sample. They also reported that increasing the ethylene glycol content to 20% resulted in a 4% reduction in residual carbon. A low-temperature production method for the synthesis of rod-like crystalline B_4C with minimized content of unfavorable carbon using high char yield and aromatic polymeric precursor was found by Wang et al. [27]. Increasing the contact surface between B_2O_3 and carbon as a result of controlling the carbon amount by pyrolysis temperature led to a reduction of synthesis temperature to 600 °C.

Baklanova et al. [5] used mechanical activation of carbon black in the synthesis of Mo_2C. Carbon black was soaked with aqueous ammonium heptamolybdate solution by using the incipient wetness method in an inert media at a milling speed of 100 g for 30 min. Calcination of the prepared carbide-containing composites was carried out at 800 °C in an inert medium. They showed that mechanically activated material includes iron oxide, iron carbide, and molybdenum carbide. The molybdenum carbonization degree was measured to be 50% of the theoretical value by X-ray analysis.

Recently, it was displayed that using glucose (as a carbon source) plays an effective role in the synthesis of B_4C through reaction with boric acid (as the boron source) by our group [28]. Next, the investigation of the influence of glucose pretreatment and the using further boric acid on the synthesis of B_4C was also accomplished [29]. Besides, the study and optimization of pyrolysis parameters like temperature, time, and atmosphere were also performed [30]. In continuation of those research works, the parameters of the synthesis procedure including the time, the atmosphere, and the temperature were optimized [31]. In the present research, the impacts of the addition of tartaric acid and mechanical activation on the performance of B_4C synthesis are studied.

2. Materials and methods

After examining the effective conditions and variables in the boron carbide synthesis process, the results of which have been published in the previous articles of our group [28–31], in this research, various methods were investigated in order to reduce the residual carbon in the final product. For this purpose, two common and effective methods for polymer materials were used.

2.1. Mechanical activation to reduce residual carbon

In order to evaluate the effect of mechanical activation on the efficiency of the synthesis reaction as well as the reduction of residual carbon, the method of mechanical activation of the precursor prepared at two stages of the general synthesis process was used. For this purpose, in the first sample, the prepared precursor was subjected to high-energy ball milling for 3 hours before being placed in the pyrolysis furnace, and then the material was sent to the pyrolysis stage. In the second sample, the material obtained from the pyrolysis stage was ball milled for 3 hours. Then the powder obtained from this operation was used for pellet preparation and synthesis operation. In the following, these two samples were subjected to the synthesis process in optimal conditions and were compared with the base sample that did not undergo any mechanical activation operation on it. In order to make a quantitative and qualitative comparison of the samples, X-ray diffraction (XRD) analysis was performed.

2.2. Adding tartaric acid to reduce the remaining carbon

In order to investigate the effect of adding an organic additive to the precursor to increase the efficiency of the boron carbide synthesis and reduce the residual carbon percentage, tartaric acid was used as an additive in the precursor production stage. This organic acid is one of the types of organic materials, which has a chemical composition of $C_4H_6O_6$ and a molar weight of 150 g/mol. To observe the effect of adding tartaric acid on the efficiency of boron carbide synthesis, five different proportions of tartaric acid were used in raw materials. For this purpose, the acid in the amounts of 0, 5, 10, 25, and 50 wt% of the polymer was added to the mixture of caramelized glucose and boric acid at the ratio of 8.6 g of polymer to 13 g of acid. In order to keep the amount of incoming carbon constant, the amount of polymer was reduced by the amount of added tartaric acid, and then the preparation of the precursor was performed. To check the bonds created in the precursors, FT-IR analysis was taken from the materials. Finally, the samples were subjected to pyrolysis and synthesis, and the final products were characterized by the XRD method.

3. Results and discussion

3.1. Effect of mechanical activation

The results of the XRD analysis of the samples activated by mechanical energy are shown in Fig. 1 and compared with the base sample. These findings show that the use of a high-energy ball mill for precursor pyrolysis has caused an approximate increase in the intensity of the main peak of B_4C at 38 degrees and also has weakened the peaks of carbon impurity materials at 16 and 32 degrees. In addition, in the pre-synthesis activated sample, due to the fact that the peaks of hydrocarbon materials have been reduced, one of the characteristic peaks of B_4C located at 32 degrees has been revealed. In fact, mechanical activation before the synthesis process has improved the efficiency of the synthesis process.

Moreover, the results show that the use of high-energy ball milling for the precursor before pyrolysis does not have much effect on the final

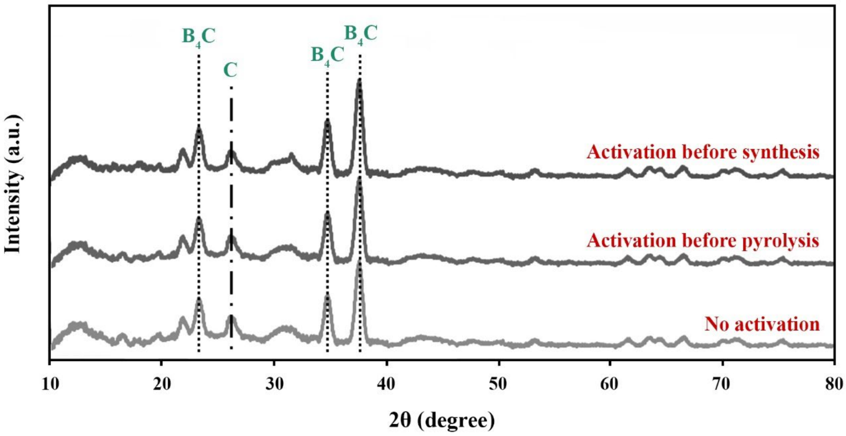

Fig. 1. The XRD results related to the effect of mechanical activation.

result of the synthesis process, and no effect of reducing impurities is observed. This can be deduced from the slight increase in the intensity of the main peak of B_4C at 38 degrees. The only thing that happened in this sample was a slight shift in the location of a number of peaks of the polymer material and a negligible change in their intensity.

The raw materials used in the synthesis reaction included caramelized polymer and boric acid. As a result of heat treatment, the polymer has undergone minor changes in bonds and the arrangement of atoms. Therefore, the bonds that exist in this polymer are generally weak covalent and van der Waals bonds. Boric acid also has a similar structure. Therefore, if these materials are subjected to high-energy ball mill operations, the strong blows from the steel balls and the rotational movement of the mill set cause the bonds in the polymer and boric acid to change greatly [32, 33]. Relatively weak material bonds are broken or deformed a lot during this process [34–36].

The energy entered into the molecules in the form of temporary shape change is stored as potential energy in the structure of the material to be used in the next step. Since the energy stored in the material can be released in relatively high-temperature conditions, it is consumed in the pyrolysis process and does not contribute much to the synthesis process. Therefore, performing mechanical activation before pyrolysis does not have much effect on the synthesis reaction efficiency. It should be noted that the reactions that occur in the pyrolysis stage are mostly thermal decomposition [37, 38], and on the other hand, the energy released in the pyrolysis stage does not have much effect on the final product of the pyrolysis process [39, 40].

If the mechanical activation and introduction of potential energy to the particles take place before the final synthesis operation, it can be expected that the energy stored in the material will be released during the synthesis process, causing the reaction to be carried out faster and improving the synthesis efficiency. In fact, the mechanism of reaction in the synthesis step is diffusion. The rate of diffusion has a direct relationship with the size of the particles participating in the reaction and increases as the particle size decreases [41–44]. Since the high-energy ball milling causes the particles to break and get smaller due to successive collisions, it can be expected that such an operation has a favorable effect on the rate and efficiency of the synthesis reaction [36, 45, 46]. Additionally, as the activation energy required for the B_4C formation reaction is very high, the use of this method provides a part of the energy needed for the reaction through the energy storage in the molecules in the form of distortion and temporary deformation, and accordingly, the starting temperature of the reaction is lowered. In other words, if the reactions are carried out at the same temperature, the synthesis efficiency will be increased, and the residual carbon content will be reduced.

3.2. Effect of tartaric acid additive

Tartaric acid was used in order to investigate the effect of adding an auxiliary substance to the raw materials on the efficiency of the synthesis process and obtaining pure B_4C with minimal impurities. The results of the FT-IR analysis of the samples are shown in Fig. 2. The outcomes display that with the addition of tartaric acid to the raw materials, a characteristic peak at the wave number of 1750 cm^{-1} has appeared, which has become more intense with the increase in the amount of the additive. This peak corresponds to the carbon-carbon double bonds present in tartaric acid. In fact, the detection of this peak reveals the presence of acid in the substance. Moreover, with the addition of tartaric acid, peaks appeared in the wave numbers 1050 cm^{-1} to 1150 cm^{-1}, whose intensity is directly related to the percentage of tartaric acid. These peaks are related to C–O bonds. Such variations in the curves somehow indicate the presence of more hydrocarbons in the raw materials, which is caused by the tartaric acid increase as an organic material. The peaks related to O–H bonds in the range of wave numbers from 3100 cm^{-1} to 3300 cm^{-1} have also intensified with the addition of tartaric acid.

Introducing an additive, in addition to the changes mentioned above, which only indicates the presence of a new hydrocarbon substance in the raw materials, also leads to other results. The characteristic peak related to the B–C bond, which appears at the wave number of 1190 cm^{-1}, is longer and more intense in the samples containing tartaric acid than in the sample without acid. Therefore, it can be concluded that the presence of tartaric acid has increased the tendency of boron to form bonds with carbon, strengthened the bonds of B with C, and increased the formation of B–C bonds. The increase in the peak intensity of O–H and C–O bonds as well as B–C all indicate the formation of borate ester during these reactions and the formation of B–O–C bonds. The reason for this can be the dehydration of polymer compounds and boric acid and the reaction between them. In fact, the presence of tartaric acid helps in the formation of borates and has a useful function in creating crosslinks between boric acid and polymer.

From the above results, it can be deduced that the presence of tartaric acid in the composition of raw materials is useful for increasing the

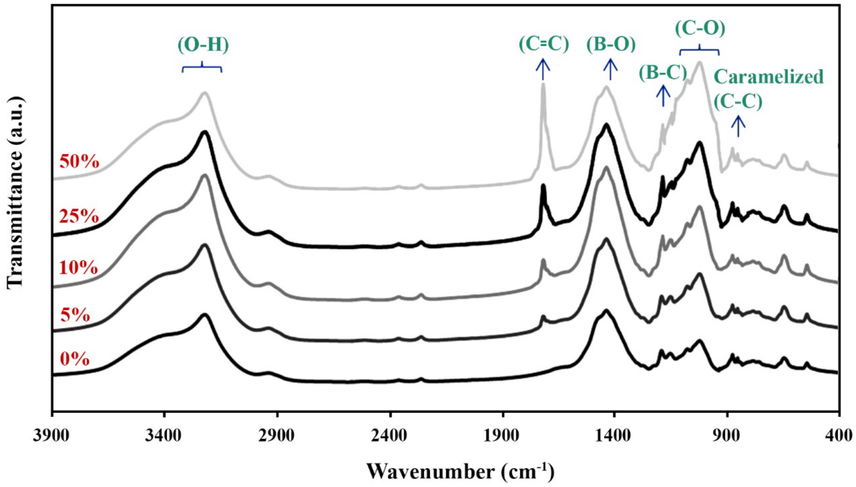

Fig. 2. The FT-IR spectra related to the influence of tartaric acid addition on the precursor structure.

efficiency of B_4C synthesis and increasing the strength and intensity of bonds between materials and boron. In order to achieve the optimal percentage of tartaric acid in the composition of materials and to investigate the effect of this additive on the quality of B_4C synthesized in the final stage, all samples were subjected to pyrolysis and synthesis under optimal conditions. The results of the XRD analysis are displayed in Fig. 3, according to which, the addition of tartaric acid to the mixture of raw materials has increased the intensity of the peaks related to B_4C, and decreased, and even disappeared in some cases, the peaks related to carbon impurities.

According to Fig. 3, the group of peaks that are in the range of 10 to 20 degrees generally indicates the presence of heavy hydrocarbon impurities in the final product. Also, in the region of 30 to 33 degrees, hill-like peaks can be seen, which are related to the remaining carbonaceous materials in the samples containing tartaric acid additives. Due to the reduction of the remaining carbon percentage in the samples with a higher percentage of additive, the B_4C peaks have appeared more prominently.

The mentioned cases indicate that by increasing the percentage of tartaric acid, the peak intensity of the unreacted carbon material decreased, which increases the purity of the final B_4C and subsequently boosts the synthesis efficiency. Tartaric acid is an additive of the family of carboxylic acids with relatively high acidity among organic acids, which can effectively react with polymers and change small and simple structures such as glucose and sucrose [47–49]. Also, one of the most important features of this acid is the ability to chelate metal ions in carbohydrates and create ionic complex structures [50]. This characteristic of tartaric acid can improve boron bonds with polymer matrix. In fact, the presence of this acid helps more boron to be placed in the polymer matrix and become chelated. Therefore, improving the synthesis efficiency, increasing the intensity of B_4C peaks, and reducing the peaks of carbon impurities can be attributed to the ability of tartaric acid to chelate boron in the polymer matrix and to make changes in the structure of the polymer in order to increase the suitable positions for bonding with boron.

In fact, by introducing boron into the polymer structure, tartaric acid increases the possibility of the formation of borate ester bonds (B–O–C). These bonds increase the probability of boron and carbon atoms being placed together and at appropriate distances to carry out the diffusion reaction and synthesize B_4C. This has caused carbon peaks with less intensity to be seen in the samples containing tartaric acid. With the increase in the percentage of tartaric acid, the amount of impurity was reduced, and a purer final product was obtained, specifically no traces of carbon peaks were seen in the sample with

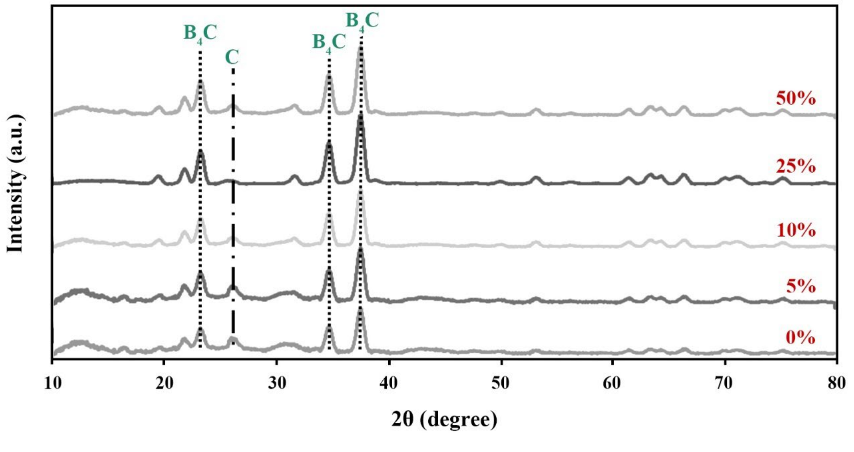

Fig. 3. The XRD patterns related to the effect of tartaric acid addition on the synthesized B_4C.

25 wt% additive. However, with the addition of more tartaric acid up to 50 wt%, the carbon peaks reappeared and intensified. This trend shows that the addition of tartaric acid is effective up to a certain limit and adding too much will decrease the synthesis efficiency. Such a phenomenon is probably due to the fact that the amount of polymer in the precursor has decreased with the excessive increase of tartaric acid, and the decrease of polymer means the reduction of carbon available for the reaction. This problem also shows that tartaric acid cannot be used as the only carbon source, and it can have a positive effect on the synthesis efficiency with another polymer. According to the obtained results, it was found that the addition of 25 wt% tartaric acid has optimal synthesis efficiency.

4. Conclusions

The roles of tartaric acid addition and mechanical activation on the efficiency of B_4C synthesis (employing glucose and boric acid as starting materials) and the reduction of residual carbon were investigated. To study the effect of mechanical activation, two samples were subjected to high-energy ball milling: one sample before pyrolysis and the other after pyrolysis. Moreover, tartaric acid with various contents (0, 5, 10, 25, and 50 wt%) was used as an additive in the precursor production step to boost the synthesis efficiency. Mechanical activation after pyrolysis enhanced the B_4C synthesis efficiency, but ball milling before pyrolysis was not effective. The addition of tartaric acid, with an optimum amount of 25 wt%, boosted the efficiency of the synthesis process.

CRediT authorship contribution statement

Seyed Faridaddin Feiz: Methodology, Writing – original draft.
Leila Nikzad: Conceptualization, Supervision, Resources.
Hudsa Majidian: Project administration, Funding acquisition, Writing – review & editing.
Esmaeil Salahi: Supervision, Project administration, Funding acquisition.

Data availability

The data underlying this article will be shared on reasonable request to the corresponding author.

Declaration of competing interest

The authors declare no competing interests.

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