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Research Article

# Preparation of Aluminium dodecaboride ($AlB_{12}$) powder by Self-propagating High-temperature Synthesis (SHS)

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## Abstract

Self-propagating High-temperature Synthesis (SHS) process is used to prepare Aluminium dodecaboride ($AlB_{12}$). The phase analysis results of preparing $AlB_{12}$ with Al and $B_2O_3$ as raw materials show that under air and argon conditions, the self-propagating and acid-washed self-propagating powders all have α-$Al_2O_3$ impurities when Mg, Al and $B_2O_3$ are used as raw materials. The phase analysis results of the preparation of $AlB_{12}$ show that under argon conditions, the self-propagating and acid-washed, self-propagating powder has un-removable $MgAl_2O_4$ impurities, and the root cause of the low purity of $AlB_{12}$ prepared by the self-propagating method is the presence of un-removable impurities.

## Introduction

Most of the borides are crystals with high hardness and melting point [1-4]. Stable chemical properties and a wide range of applications make it widely used in composite materials, semiconductors, and in various areas of national defense, such as radiation protection [5-8]. Among them, $AlB_{12}$ has a special electronic structure and bonding characteristics [9,10]. It can effectively adjust the conductivity of semiconductor materials, and thus is extensively employed in conductors and semiconductor materials. In addition to the above characteristics, the content of boron in $AlB_{12}$ is extremely high, reaching 82.8%, which is very promising as neutron shielding material [11-13].

Ceramic powders are usually synthesized by traditional sintering methods [14-16]. However, the use of this method to synthesize ceramic powder takes a long time, consumes a great deal of energy and pollution [17]. Self-propagating high-temperature synthesis (SHS) is a unique technique for synthesizing materials by self-heating and self-conduction of high chemical reaction heat between reactants. This technology was first discovered by Merzhanov et al., in their research on the combustion of solid propellants in rockets and was announced in 1967. Compared with the conventional sintering method, the advantages of the SHS method can be summarized as follows: (1) It is time saving and makes full use of energy [18]. (2) It requires only simple equipment and processes [19]. (3) The high product purity and product conversion rate are close to 100% [20]. (4) It can not only produce ceramic powder, but if the proper amount of pressure is applied at the same time, high-density combustion products can also be produced [21]. (5) High output [22].

In previous studies, $AlB_{12}$ powder was fabricated by using the SHS method [23-25]. The calculation results of preparing

$AlB_{12}$ with Mg, $Al_2O_3$ and $B_2O_3$ as raw materials show that the adiabatic temperature of the system is 2789.5K, which satisfies the self-propagating reaction conditions. Further, the phase analysis results show that there is no matter in air or argon, the self-propagating powder and the acid-washed self-propagating powder all have $Mg_{0.4}Al_{2.4}O_4$ impurities, and the purity of the prepared $AlB_{12}$ is not high.

Although $AlB_{12}$ is produced, $Mg_{0.4}Al_{2.4}O_4$ has not been removed and still exists. In this work, Al, $B_2O_3$ and Mg, Al, $B_2O_3$ were used as raw materials to conduct experimental studies on self-propagating synthesis of $AlB_{12}$.

## Experimental procedure

The starting materials used in this research were Al powder (purity>99% Al, average particle size 50 μm, provided by Dandong Chemical Research Institute, Dandong, China), B2O3 powder (purity>99%, average particle size 96 μm, provided by Dandong Chemical Research Institute, Dandong, China), and Mg powder (purity>98%, average particle size 100 μm, provided by Dandong Chemical Research Institute, Dandong, China).

The steps used in the self-propagating process to synthesize $AlB_{12}$ ceramic powder are as follows: (1) Weigh a certain amount (in proportion to the reaction equation) of the original material powder, place it in the ball milling tank, and mix the ball mill for 2 hours. (2) Intercept the resistance wire and connect it to the two poles of the self-propagating device and place the material in the atmosphere with one end close to the resistance wire. (3) Start the ignition device and slowly increase the current. When the pointer fluctuates sharply, reduce the current and keep the current increasing steadily. Finally, the resistance wire will reach a molten state when the material is induced to burn, and the current is turned off. (4) The reaction product is pulverized and sieved with 160 meshes, and samples under the sieve are sampled for detection and analysis.

The phase analysis of the synthesized powder was carried out using an X-ray diffractometer (XRD, X'Pert Pro MRD, Netherlands) with a Philips diffractometer using Cu Ka. The microstructure of powders and elements analysis were investigated using a scanning electron microscope with EDS detector (SEM, S-3400N, Japan).

This article focuses on the study of two reaction systems, system 1: Al and $B_2O_3$, and system 2: Mg, Al, and $B_2O_3$. Two experimental atmospheres are used in both systems (Table 1).

In the Al-$B_2O_3$ system, the following chemical reactions mainly occur:

$$13Al+6B_2O_3 \rightarrow AlB_{12}+6Al_2O_3 \tag{1}$$

In the Mg-Al-$B_2O_3$ system, the following chemical reactions mainly occur:

$$3Mg+B_2O_3 \rightarrow 2B+3MgO \tag{2}$$

$$Al+12B \rightarrow AlB_{12} \tag{3}$$

$$18Mg+Al+6B_2O_3 \rightarrow AlB_{12}+18MgO \tag{4}$$

After the combustion synthesis, the extraneous components were leached out from the synthesized powder with 60° C in diluted HCl (pH value is 3).

## Results and discussion

Figure 1 is the X-ray diffraction pattern of Al and $B_2O_3$ prepared under both air conditions (before and after pickling) and argon conditions (before and after pickling) respectively. It can be seen from the figure that in either air or argon conditions, irremovable $Al_2O$ is found in the bottom. Analysis of its crystal

**Table 1:** The experimental scheme of tests.

| Serial condition | Reactant | Atmosphere | Pickling |
|---|---|---|---|
| S1 | $Al+B_2O_3$ | Air | Before pickling |
| S2 | $Al+B_2O_3$ | Air | After pickling |
| S3 | $Al+B_2O_3$ | Ar | Before pickling |
| S4 | $Al+B_2O_3$ | Ar | After pickling |
| S5 | $Mg+Al+B_2O_3$ | Ar | Before pickling |
| S6 | $Mg+Al+B_2O_3$ | Ar | After pickling |

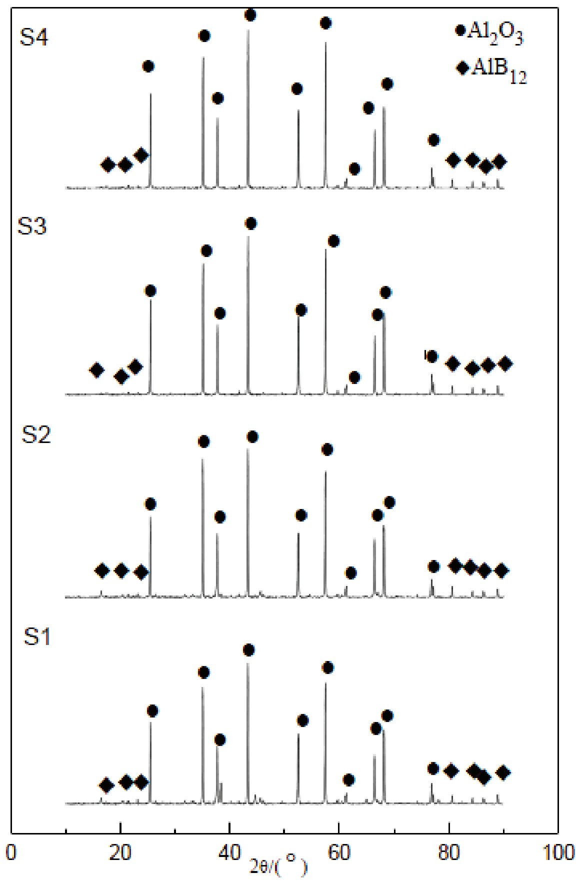


**Figure 1:** XRD patterns of fabrication $AlB_{12}$ from Al and $B_2O_3$.



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structure revealed that the $\alpha$-$Al_2O_3$ is corundum, an extremely stable substance that is difficult to remove through physical and chemical reactions. Therefore, $AlB_{12}$ prepared from Al and $B_2O_3$, contains a large amount of inseparable corundum, which contributes to the failure of the self-propagating preparation of $AlB_{12}$ using Al and $B_2O_3$ as raw materials.

Figure 2 is the X-ray diffraction pattern of powder prepared with Mg, Al and $B_2O_3$ under argon conditions (before pickling). From the results of phase analysis, the main components of the coarse powder before pickling are MgO, $Mg_3B_2O_6$, $MgAl_2O_4$, and $AlB_{12}$, while in the powder after pickling, when MgO and $Mg_3B_2O_6$ are removed, the main impurity is $MgAl_2O_4$. This shows that the purity of $AlB_{12}$ is not high when prepared by self-propagating, self-propagation when the raw materials used are Mg, Al and $B_2O_3$.

Figure 3 shows the microscopic morphology of powder prepared through use of the self-propagating method under argon conditions with Al and $B_2O_3$ as raw materials after pickling. From an analysis of the energy spectrum results, the A particles – with obvious layering phenomenon on the left are $Al_2O_3$ particles – while the B particles – with more obvious granular shape on the right – are $AlB_{12}$. This situation shows that despite the pickling treatment, $Al_2O_3$ is still untreated. It also shows that the unremovable by-product $Al_2O_3$ uses Al and $B_2O_3$ as raw materials and is the biggest obstacle to self-propagating preparation of $AlB_{12}$.

Table 2 shows the elemental analysis results of EDS analysis after pickling of Al and $B_2O_3$ as raw materials and self-propagating preparation of $AlB_{12}$ with Mg, Al, and $B_2O_3$ as raw materials. From the results in the table, the acid wash product prepared with the use of Al and $B_2O_3$ as raw materials has the highest content of O element, followed by B and Al. Observing the test samples, there are still insoluble substances, so the test results are also relatively incomplete. This shows that under argon conditions, using Al and $B_2O_3$ as raw materials to prepare $AlB_{12}$, and using the self-propagating method, the purity of $AlB_{12}$ in the prepared product is low, and the B content is insufficient.

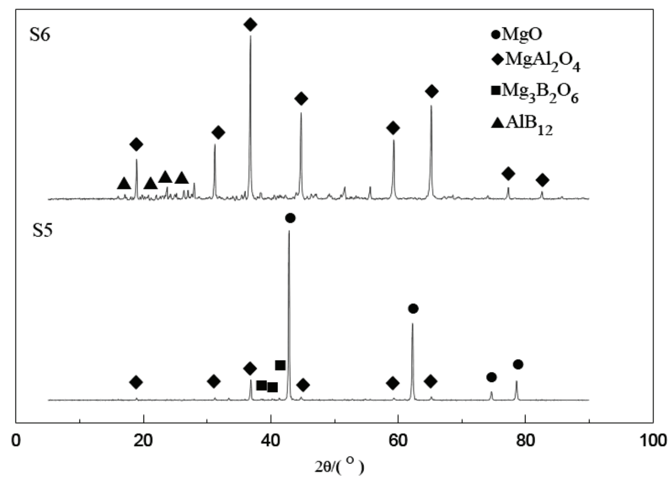


**Figure 2:** XRD patterns of fabrication $AlB_{12}$ from Mg, $B_2O_3$ and $Al_2O_3$.

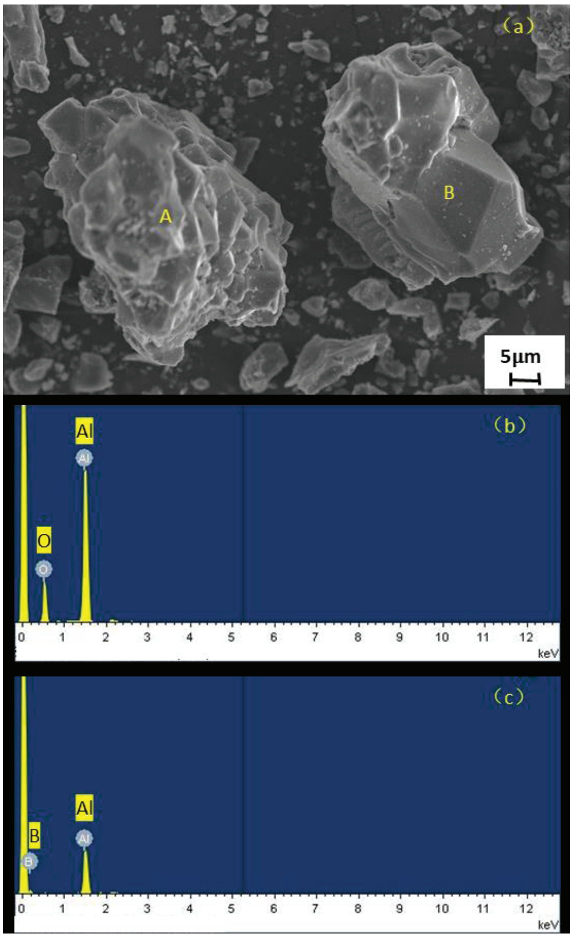


**Figure 3:** (a): Microstructure photographs of powder by SHS; (b): EDS analysis of A area; (c): EDS analysis of B area.

**Table 2:** The results of elementary analysis (mass fraction, %)

| Serial | B | Mg | Al | O |
|---|---|---|---|---|
| S4 | 12.6 | – | 2.21 | 85.19 |
| S6 | 58.5 | 9.62 | 3.43 | 28.45 |

## Summary

The phase analysis results of preparing $AlB_{12}$ using Al and $B_2O_3$ as raw materials shows that there are $\alpha$-$Al_2O_3$ impurities in the self-propagating powder regardless of either the air condition or the argon condition, and it cannot be removed. Consequently, the purity of the prepared $AlB_{12}$ is not high. The phase analysis results of preparing $AlB_{12}$ using Mg, Al and $B_2O_3$ as raw materials indicates that the self-propagating and acid-washed self-propagating powder has unremovable $MgAl_2O_4$ impurities under argon conditions, and the purity of the prepared $AlB_{12}$ is not high, causing self-propagation. The fundamental reason for the low purity of $AlB_{12}$ prepared by this method is the existence of impurities that cannot be removed. For future research work, it may be very promising to consume $Al_2O_3$ through aluminum electrolysis.

## Notes

The authors declare that they have no competing financial interest.



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