

## The Impact of Mechanical Effects on Granulometric Composition of TiB<sub>2</sub> – Based Materials

P.M. Bazhin, A.M. Stolin\*, A.S. Konstantinov

*Institute of Structural Macrokinetics and Materials Science, Russian Academy of Sciences,  
8, Academician Osipyan St., Chernogolovka, Moscow Region, 142432, Russia*

\* Corresponding author: Tel. +7 496 524 63 76. E-mail: amstolin@ism.ac.ru

### Abstract

The article explores the synthesis of refractory TiB<sub>2</sub>-based materials under self-propagating high-temperature synthesis (SHS) and mechanical effects. Using the experimental data, the role of mechanical effects on SHS products is shown. Scanning electron microscopy and X-ray phase analysis were used to study the resulting powder materials.

### Keywords

Self-propagating high-temperature synthesis; SHS; mechanical effects; refractory materia; titanium diboride.

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### Introduction

Titanium diboride has a set of unique physical and mechanical properties. These are, above all, a high melting point, high hardness, low electrical resistance, high thermal conductivity, resistance to abrasive wear and corrosive environment, relatively low density [1, 2]. Because of this, titanium diboride is widely used in modern technology as part of wear-resistant and erosion-resistant materials, in the manufacture of lightweight ceramic armor, crucibles and boats for vacuum evaporation of metals with subsequent metallization, parts of chemical equipment, and cermets for nuclear power engineering [3–5].

One of the promising methods for preparation of refractory materials is a self-propagating high-temperature synthesis (SHS) method. The SHS process is based on chemical interaction of the starting reagents in the solid phase in the form of high-temperature combustion [6, 7]. The combustion is here an exothermic reaction of powdered reactants reacting with one another or with liquid or gaseous reactants to form solid chemical compounds. Quite often, these are refractory inorganic compounds (carbides, nitrides, borides, etc.) that do not degrade in combustion, as well as materials based on them.

One of the specific examples of the SHS process under mechanical impact is the new technological method for producing powders of refractory compounds, called SHS-grinding [8]. The features of the method consist in the combination of mechanical effects with combustion in the SHS mode in one installation, in which the deformation parameters have a strong influence on the combustion and structure formation of synthesized materials [9].

The present study focuses on the influence of mechanical effects on the granulometric composition of TiB<sub>2</sub>-based materials, their morphology and structure. It is shown that the deformation parameters, and, first of all, the deformation rate, have a strong effect on the change in size of the synthesized particles. Changing the parameters of mechanical effects – the rotor speed, the delay time, one can change the quality of the resulting powder.

### Research methods

The experiments were carried out in a closed reactor without rotation of the rotor at rotational speeds of 120 and 240 rpm [9]. The rotor was in the form of a cone with an apex angle of 140°. For the experiments, a mixture of titanium powder of PTM brand (the basic substance content was 99.1 %, the

particle size of main fraction was 45  $\mu\text{m}$ ) and B-99B boron (the basic substance content was 99 %, the particle size of the main fraction was 5  $\mu\text{m}$ ) was taken for the stoichiometric synthesis of titanium diboride. The backfill mass in the rotor was 10 g. After passing the combustion wave (2–4 s), the rotor with a specified frequency of rotation was lowered to the lower base of the reactor for 20–30 s. During this time, the synthesized material completely underwent phase and structural transformations. The rotating rotor at SHS-grinding provides equalization of the concentration of reactants in the whole volume of the reactor, and involves the unreacted initial reagents that are observed at the reactor walls in the chemical synthesis. After cooling, the deformed powder was sieved through sieves with mesh sizes of 200, 400, 500, 630, 1000  $\mu\text{m}$ .

To perform material science investigations of the synthesized powder materials, the equipment of the Distributed Center for Collective Use of ISMAN was used: the Carl Zeiss Ultraplus high-resolution field-scanning electron microscope (Germany), the ARL X'TRA powder X-ray diffractometer and other certified methods and techniques.

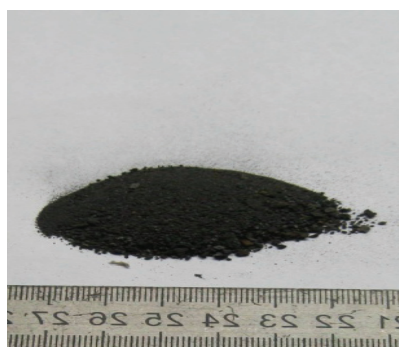
### Results and discussion

In the synthesis of powders of refractory compounds by the conventional SHS method, there is a technological problem of grinding products after synthesis, which are usually obtained in the form of a sintered mass having a high strength. In this connection, it is necessary to add an additional technological operation of its grinding, which is carried out in various mechanical grinders, ball mills and attritors. In such a case, due to high hardness of titanium diboride, the grinding requires great efforts and the powder is contaminated with lining and grinding material. Fig. 1 shows the typical form of the titanium diboride powder obtained by SHS at a bulk density of the initial components. As the bulk density of the initial reagents was 40–50 %, then during the synthesis, the particles were located at great distances from each other, as well as gases that were in the pores of the initial charge prevented the formation of a continuous sinter. However, the size of the macroparticles was 0.5–1.5 cm. Mechanical effects after SHS contributed to the destruction of the agglomerated synthesized particulates and substantially reduced their size, which became less than 1–2 mm.

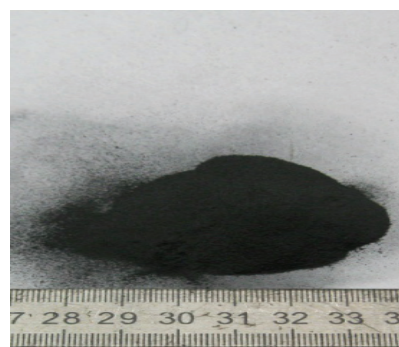
In the experiments, it was found that deformation parameters, and, first of all, the deformation rate, have a strong effect on the change in the size of the synthesized particles. By changing the parameters of



a)



b)

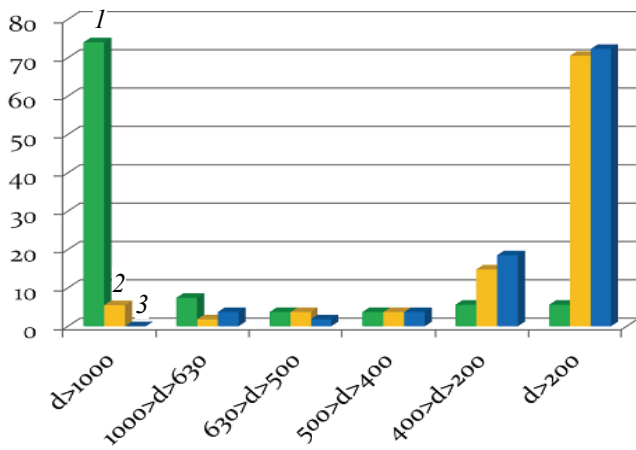


c)

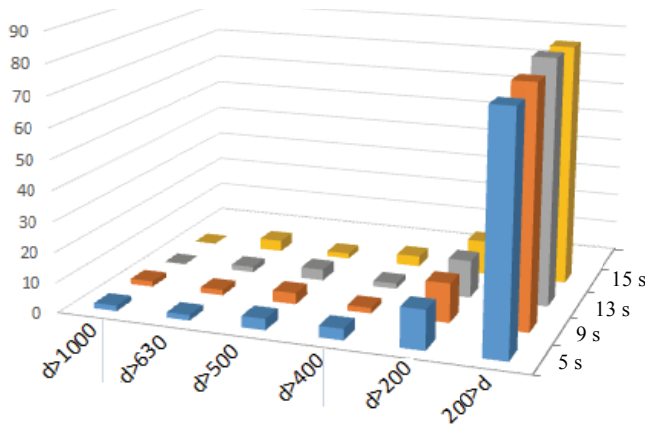
**Fig. 1. A general view of the synthesized powder:**  
a – SHS; b – SHS with mechanical effects (120 rpm);  
c – SHS with mechanical influences (240 rpm)

mechanical effects, such as the rotor speed and the delay time, one can change the quality of the powder obtained and its morphology. Using the obtained data, a comparative diagram of the product yield, depending on the rotor speed and on the delay time before the application of mechanical influences, was compiled (Fig. 2). When applying mechanical effects, the majority of titanium diboride particles with a dispersion of less than 200  $\mu\text{m}$  were more than 70 wt. %. At the same time, an increase in the rotor speed slightly increased their share.

Fig. 3 shows a diagram for a synthesized powder subject to mechanical effects at a rotor speed of 240 rpm, and with different delay times before applying mechanical loads: 5, 9, 13, 15 s, respectively.



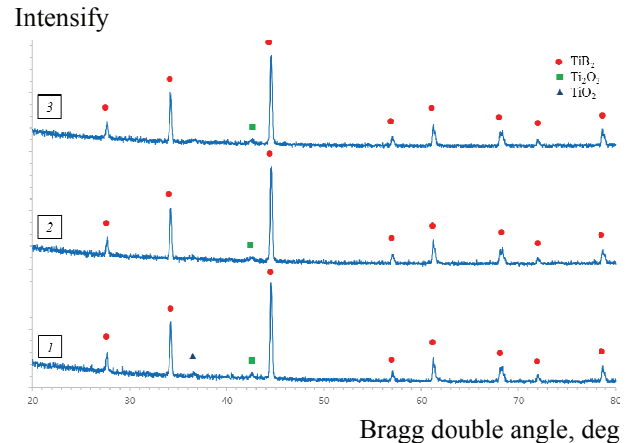
**Fig. 2. A comparative diagram of the particle size distribution of  $\text{TiB}_2$  by the production method:**  
 1 – without mechanical impact; 2 – delay 5 s, 120 rpm;  
 3 – delay 5 s, 240 rpm



**Fig. 3. A comparative diagram of  $\text{TiB}_2$  particle size distribution under mechanical effects with rotor speed of 240 rpm for different delay times**

It was found that the main part consisted of particles with a dispersion of less than 200  $\mu\text{m}$  (more than 70 mass. %), the delay time before application of mechanical effects slightly affected the dispersion of the particles. With an increase in the delay time up to more than 15 s, the material cooled down and the synthesized particles consolidated with each other, which required greater efforts for their dispersion. The results of investigations of the main powder fraction of less than 200  $\mu\text{m}$  on the Microsizer 201 laser analyzer showed that the powder obtained under mechanical effects had a narrow particle distribution in the range of 65–150  $\mu\text{m}$ .

The X-ray phase analysis (Fig. 4) revealed that in the SHS of bulk density batch mixtures without mechanical effects, titanium oxides  $\text{TiO}_3$  and  $\text{Ti}_2\text{O}_3$  were formed, apart from the main phase of titanium diboride. The obtained powders by the SHS-grinding method had much less oxide inclusions, and  $\text{TiO}_3$  was absent in them. In the SHS-grinding, mechanical effects



**Fig. 4. The results of XFA synthesized powder:**  
 1 – SHS without mechanical effects; 2 – SHS with mechanical influences (120 rpm, delay time 5 s); 3 – SHS with mechanical effects (240 rpm, delay time 5 s)

prevented the penetration of oxygen into the synthesis zone of the powder material. The synthesized powder by the conventional SHS method and the proposed one allowed obtaining titanium diboride with practically identical parameters of the hexagonal crystal lattice,  $\text{\AA}$ :  $a = 3.02621$  and  $c = 3.22812$  (for SHS without mechanical effects),  $a = 3.02666$  and  $c = 3.22684$  (for SHS with mechanical effects at a rotor speed of 120 rpm and a delay time of 5 s),  $a = 3.02681$  and  $c = 3.22867$  (for SHS with mechanical effects at a rotor speed of 240 rpm and delay time of 5 s).

The studies of the microstructure of the synthesized powder showed that grains of titanium diboride obtained under SHS conditions without mechanical effects had a structure similar to whiskers with sizes from 1–3  $\mu\text{m}$  to 20–30  $\mu\text{m}$ . The morphology of the particles obtained under SHS-grinding conditions became closer to a rounded form with an increase in rotor speed. In this case, the dimensions of titanium diboride grains reduced to 1–10 microns (Fig. 5). Thus, mechanical effects during the synthesis and subsequent cooling of the material prevented the sintering and agglomeration of the formed particles with each other, and led to the destruction of the sintered titanium diboride particles among themselves. Intensive mixing of the powder, due to rotation of the rotor, prevented the enlargement of  $\text{TiB}_2$  particles during the cooling of the synthesized material.

## Conclusions

Mechanical effects, realized immediately after the passage of the combustion wave in the SHS mode, contribute to the grinding of hot synthesis products directly upon cooling of the powder. Due to the applied effects, the particles of synthesized material do not consolidate among themselves, as it is typical of

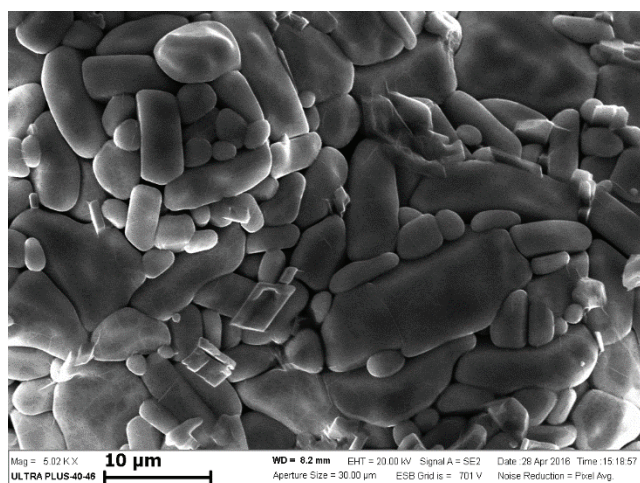


## Acknowledgements

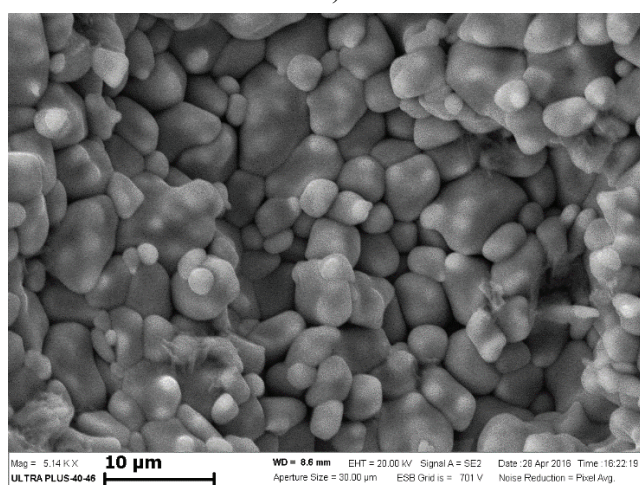
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a)



b)

**Fig. 5. A typical view of synthesized particles of titanium diboride:**  
a – SHS; b – SHS with subsequent mechanical effects  
at a rotor speed of 240 rpm

the traditional SHS. The titanium diboride powder obtained under SHS-grinding conditions consists of more than 70 % by weight of particles with dimensions in the range of 65–150 μm.

The intensity of mechanical effects after SHS significantly influences the morphology of the powder and the dimensions of its structural components. Thus, by changing the technological parameters of the SHS-grinding process, it is possible to obtain a titanium diboride powder with a given structure and properties in one technological stage and on the same equipment.