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The Specifics of Synthesis and Structure of Titanium Carbide Powder Produced by the SHS-Grinding Method in a Closed Reactor

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Abstract

The paper describes the research into the synthesis of titanium carbide under combined processes of combustion and shear deformation in a closed reactor. The SHS-grinding is one of the new processes of obtaining magnetic-abrasive materials based on Fe-TiC system. Synthesis of powder materials by SHS-grinding allows varying deformation parameters (deformation rate, external pressure), which have a significant effect on the formation of the material structure. The experimental data has shown that with the change of process parameters of synthesis and deformation it is possible to change the quality of the produced titanium carbide powder, namely, the grain size, its shape and morphology. Scanning electron microscopy and X-ray phase analysis were used to study the resulting powder materials. A comparative analysis of powders produced by the SHS-grinding and by the conventional SHS without application of external loads has been made.

Keywords

Self-propagating high-temperature synthesis (SHS); SHS-grinding, mechanical effects; titanium carbide.

Introduction

Magnetic abrasive polishing is a new method of finishing machining of various parts widely used in aviation, machine building, radio engineering and other industries for finishing of various products [1, 2]. Magnetic abrasive polishing involves placing the workpiece in a magnetic field, the energy of which is used both for forming the working tool and for abrasive action on the workpiece. Gliding along the surface to be treated, a magnetic abrasive powder entrained by a magnetic field polishes the surface of the part to the desired purity. The main magnetic abrasive materials include powders that have magnetic properties and a sufficiently high hardness for abrasive cutting (ferrites, alsiphores, etc.). Promising abrasive powders include composite materials that significantly exceed uniform in abrasive powders [3]. Composite powders consist of ferromagnetic and abrasive constituents. Iron is used as the first constituent, while titanium carbide and other carbide materials are used as the second constituent.

The main industrial method for production of titanium carbide is the carbon-thermal reduction of its compounds in bulk or in the compacted form in a non-oxidizing atmosphere (nitrogen, hydrogen, vacuum, inert gas) [4]. This method has high energy consumption both at the stage of long-term synthesis in furnaces, and at the stage of grinding of sintered pellets of titanium carbide in grinding plants. Promising methods for the production of titanium carbide include the technology of self-propagating high-temperature synthesis (SHS) based on the nonfiltration combustion of the charge – the initial mixture of titanium and soot powders – in bulk or in compressed pellets in a closed reactor [5, 6]. As a result of the synthesis, there is a significant increase in pressure in the reactor, resulting in production of highly sintered titanium carbide, which is difficult to crush and grind to the desired dispersion. To prevent sintering in agglomerates, the SHS-grinding method was developed; it combines the processes of synthesis and subsequent grinding of the synthesis products in one plant and in one technological cycle [7, 8].

The proposed research explores the features of the synthesis and the structure of titanium carbide powders produced by the SHS-grinding method in a closed-type reactor. The comparative analysis of powder produced by the traditional SHS without application of external loads has been made. It is shown that due to mechanical influences with a specified rotor speed and delay time, it is possible to produce powder with the necessary dispersion and morphology before applying an external pressure.

Experimental

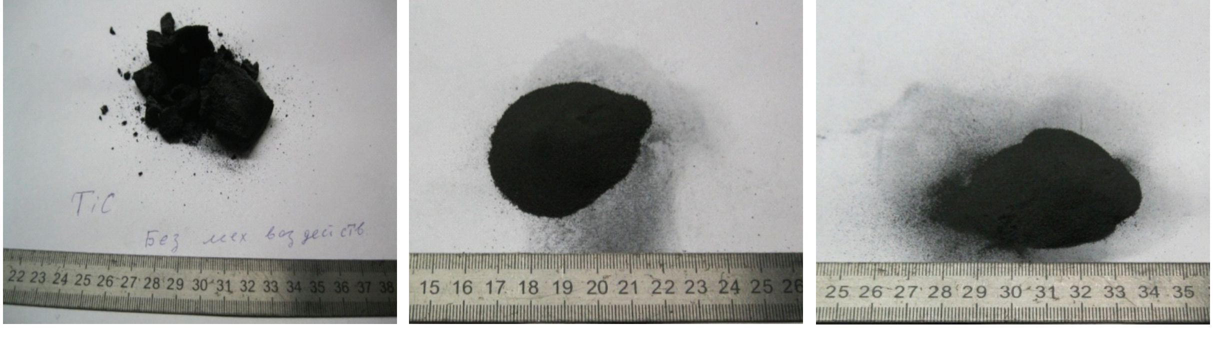
The experiments were carried out in a closed reactor without rotating the rotor and at rotational speeds of 120 and 240 rpm [6]. The rotor was taken in the form of a cone with an angle at the apex of 160°. For the experiments, a mixture of titanium powder (the content of the main substance was 99.1 %, the particle size of the main fraction was 45 μm) and PM-15TS soot (the content of the main substance was 99.1 %, the particle size of the main fraction was 1 μm) was calculated as in the synthesis of stoichiometric titanium carbide. The backfill mass in the rotor was 20 g. After passing the combustion wave at a delay time of 5 and 9 s, the rotor with a predetermined rotational speed dropped 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 provided equalization of the concentration of reacting substances in the whole volume of the reactor, and involved in the chemical synthesis the unreacted initial reagents that were observed at the reactor's walls. After cooling, the deformed powder was sieved through sieves with mesh sizes of 200, 400, 500, 630, 1000 μm.

The material science research into synthesized powder materials was performed on the equipment of the SMAN Shared Knowledge Center: the CarlZeissUltraplus high-resolution field-scanning electron microscope (made in Germany), the ARL X'TRA powder X-ray diffractometer, and other certified methods and techniques.

Results and discussion

Using the conducted experiments, it has been established that the deformation parameters, and, first of all, the deformation rate, have a strong influence on the formation of the material structure: the change in the size of the synthesized particles. By changing the synthesis parameters – the rotor speed, the delay time before applying the load – it is possible to change the quality of the powder obtained and its morphology. Fig. 1 shows the images of synthesized powders. As can be seen from the Figures, in the case of synthesis without the application of external loads, the powder presents sintered agglomerates of more than 1 to 3 cm in size; consequently, their subsequent grinding will require a lot of effort. When mechanical effects are applied, the particle sizes decrease and become less than 1 mm, and a greater uniformity in size is achieved (Table 1).

From the data presented, it can be seen that the powder produced by the conventional SHS method has particles larger than 1 mm (more than 70 wt%). Mechanical effects in SHS-grinding lead to grinding of the synthesized powder, and the main part of the carbide particles has sizes less than 200 μm (more than 70–80 wt%). At a rotor speed of 120 rpm with an increase in the delay time before applying the load, the fraction of larger particles increases insignificantly due to a longer cooling of the synthesis products. When the frequency increases to 240 rpm due to more intensive mechanical action on the synthesized material, the fraction of particles with dimensions less than 400 μm increases.



a) *b*) *c*)

Fig. 1. General view of synthesized titanium carbide powder produced by:
a – SHS; *b* – SHS-grinding (120 rpm); *c* – SHS-grinding (240 rpm)

Table 1

Results of granulometric analysis of the synthesized powder at different rotational speeds of the rotor and the delay time before applying the load

Method of synthesis	Particles with dimensions: $d > 1000$ μm	Particles with dimensions: $1000 > d > 630$ μm	Particles with dimensions: $630 > d > 500$ μm	Particles with dimensions: $500 > d > 400$ μm,	Particles with dimensions: $400 > d > 200$ μm,	Particles with dimensions: $200 > d$ μm
SHS		7.4			5.5	
SHS-grinding: $t3 = 5$ s; $n = 120$ rpm	5.5	1.8	3.7		12.9	72.2
SHS-grinding: $t3 = 9$ s; $n = 120$ rpm	9.2	5.5	5.5		9.6	62.9
SHS-grinding: $t3 = 5$ s; $n = 240$ rpm			3.7		12.9	74.7
SHS-grinding: $t3 = 9$ s; $n = 240$ rpm		1.8	3.7	1.8	12.9	76.9

As a rule, some time after the beginning of synthesis and grinding, the SHS product reacts with shear loads, the grinding rate decreases, and after a while, it is impossible to grind the residue within the limits of the plant possibilities. For the investigated rotational speeds, this residue for different delay times was 5–10 wt%. Thus, by setting the process and design parameters of the SHS-grinding (rotor speed, rotor type, delay time before applying the load, thermal insulation of the synthesized SHS material, etc.), it is possible to produce powder with the necessary dispersion.

The X-ray phase analysis of the produced powder by the SHS and SHC-grinding methods show that all characteristic peaks overlap, which indicates the production of a single-phase product with identical parameters of the titanium carbide crystal lattice. Thus, mechanical effects during SHS-grinding at different frequencies do not lead to the formation of new phases, do not distort the parameters of the crystal lattice, which is cubic with $a = 0.4327$ nm (Fig. 2).

The titanium carbide powder produced by the SHS method is a sintered mass and large agglomerates consisting of characteristic roundish and fragmented particles with sizes up to 60 μm (Fig. 3, *a*). In addition, there are particles of TiC spongy type 20–80 μm in size with pores, releasing gas during synthesis. Titanium carbide produced by the SHS-grinding method, is represented by macroparticles with sizes of 20–40 μm, consisting of individual rounded grains with dimensions of less than 1 μm (Fig. 3, *b*). At the same time, fragmented titanium carbide particles do not occur. Thus, mechanical effects during SHS-grinding prevent sintering and agglomeration of titanium carbide particles with each other, and lead to the destruction of the sintered particles among themselves.

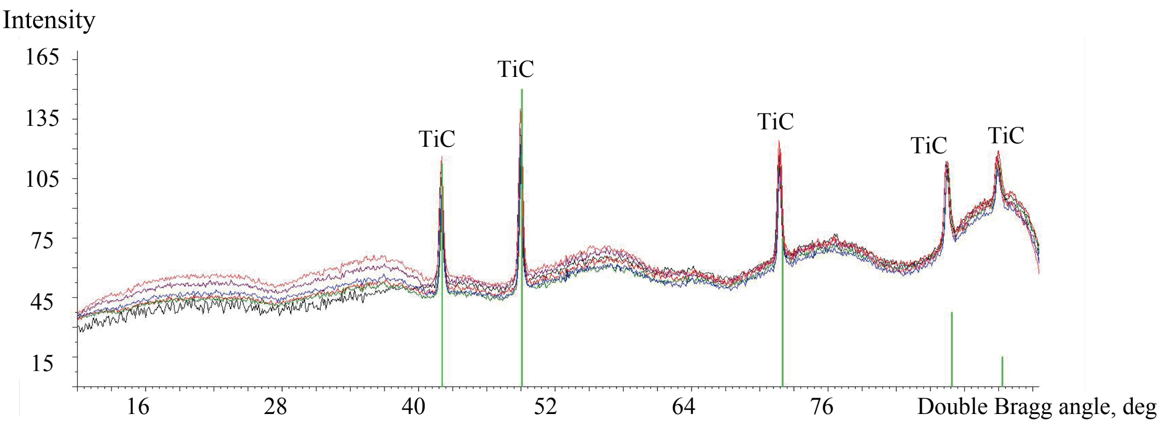
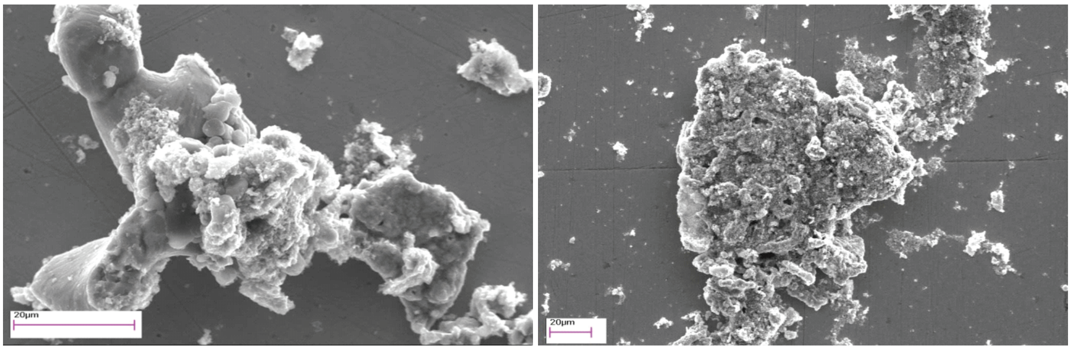


Fig. 2. Results of X-ray phase analysis of powders produced by the SHS and SHS-grinding methods at rotor speeds of 120 and 240 rpm



a) *b*)

Fig. 3. Types of powder synthesized by:
a – SHS; *b* – SHC-grinding at a rotor speed of 240 rpm

Intensive mixing of the synthesized powder by the rotor prevents the enlargement of TiC particles during cooling. As a result, the effect of quenching of the initial morphology of synthesized grains takes place, and in the future, when they cool down, they partially coalesce with each other, but without coarsening in size.

Conclusion

By the SHS-grinding method, a titanium carbide powder with a characteristic crystal lattice with a = 0.4327 nm was obtained. It is shown that mechanical effects lead to grinding of the synthesized powder, with the majority of carbide particles having a size of less than 200 μm (more than 70 to 80 wt%).

An increase in rotor speed caused by intensive mechanical effects on the synthesized material increases the proportion of carbide particles with dimensions of less than 400 μm. At the same time, the fraction of particles with dimensions greater than 400 μm for different delay times is not more than 10 wt%.

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