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DEPENDENCE OF THE PRODUCT'S PHASE COMPOSITION ON THE RATIO OF PRECURSORS IN PLASMODYNAMIC SYNTHESIS OF TITANIUM DIBORIDE

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In present time materials with high physic-mechanical properties are in demand. Materials based on nanostructured powder show higher properties than coarse-grained analogs. Titanium diboride (TiB_2) is an excellent powder for functional ceramics, which needs in a lot of application in industry, for example, metallurgy, mechanical engineering [1]. It can be used as surface coatings on iron, steels, refractory metals, because TiB_2 has high hardness, good wear and corrosion resistance [2].

In this paper presents one of a possible way of obtaining TiB_2 – plasmodynamic synthesis using coaxial magnetoplasma accelerator (CMPA). The aim of this work was to find the optimal ratio of precursors to synthesize the phase of titanium diboride hexagonal syngony with spatial group P6/mmm. The possibility of synthesis nanopowders was shown in the work [3].

According to the aim series of experiments with different mass ratio of Ti:B precursors were implemented (26.7 : 73.3; 45.5 : 54.5; 61.1 : 38.9; 86.4 : 13.6).

Synthesized powder products without any pretreatment were analyzed by X-ray diffraction (XRD), transmission electron microscopy (TEM). Quantitative X-ray analysis was performed using the PowderCell program. The highest yield of the titanium diboride phase (96.2 %) was achieved in the experiment with a Ti : B ratio of 45.5 : 54.5, bur the average size of particles is 67.8 nm. The experiment with a boron percentage of 73 % was implemented, which from the Ti-B state diagram is the region for the production of titanium diboride. The yield of TiB_2 is 93.2 % and the particle size is the smallest in comparison with the other experiments – 56.1 nm.

The analysis of the crystal forms of the product was carried out on the basis of a set of light-field TEM and HRTEM images. There is typical bright-field image of transmission electron microscopy in figure 1. It can be seen that the product basically includes at least two types of particles. The first type is prismatic crystals of a hexagonal and dihexagonal forms, which looks like a circle in a plan. This form corresponds to the spatial group P6/mmm of the hexagonal system. The habit of a crystal can exist in several simple forms: hexagonal and dihexagonal prisms and pyramids. In other areas they can be related to the second type – crystals in the form of a cube that corresponds to the spatial group Fm3m for the corresponding phase of titanium diboride TiB cubic syngony. This product consists only of particles of two fractions: cubes and hexagons with a narrow width distribution up to 100 nm in size.

Thus, plasmodynamic synthesis of nanocrystalline particles of titanium diboride with hexagonal syngony with the spatial group P6/mmm was car-

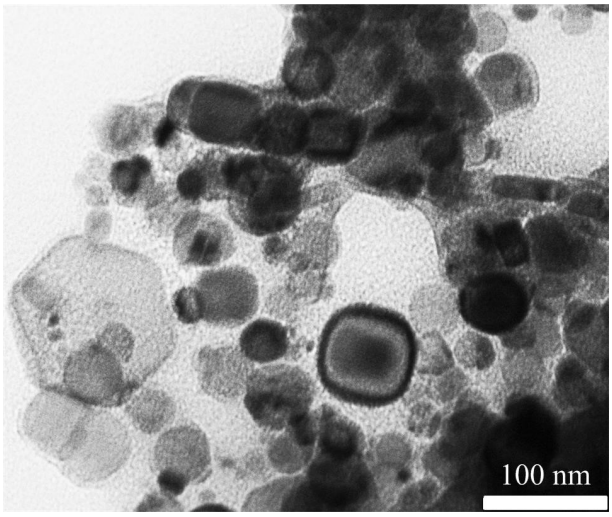

Fig. 1. *Typical bright-field image of transmission electron microscopy*

ried out. The optimal ratio of precursors Ti:B with the smallest particles 56.1 nm is 26.7 : 73.3. In the synthesized product, the predominant content of hexagonal and cubic titanium diboride particles was found.

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FLUSHING CATHODE DEPOSIT FROM SODIUM, POTASSIUM AND LITHIUM FLUORIDES

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As a result of electrolysis of UF_4 in the molten salt FLiNaK (the composition is given in Table 1), metallic uranium is obtained [1].

In order to obtain pure uranium powder, it is necessary to separate it from fluoride salts of electrolyte (LiF, NaF, KF), complex uranium-containing salts, micro impurities that are contained because of the primary components, and also because of corrosion processes.

FLiNaK melt salts must be extracted from an economic point of view. They can be returned to the cycle, thereby reducing the cost of the resulting metallic uranium.

To solve these problems, we can offer several methods of flushing:

1) "Aqueous" way of separating the cathode deposit.

It is necessary to thoroughly reground the preformed precipitate to create a larger reaction surface.

The quantity of substance is mixed with distilled water upon the application of heat. After filtration, the residue is mixed with HCl and $AlCl_3$. The resulting solution will contain fluoride salts.

After this, it is necessary to evaporate the water in order to separate the salts. The costs of heating and evaporation of water significantly increase the cost of uranium. Therefore, it is necessary to find another, more cost-effective way of flushing the cathode deposit.

Moreover, the difficulty of this method is that it is possible to form an oxide film on metallic uranium. It is necessary to take additional measures to destroy it.

2) Flushing the cathode deposit with ethyl hydroxide.

The cathode deposit is comminuted. Quantity

Table 1. FLiNaK salt composition

Fluoride	Content, % wt.
LiF	29.21
NaF	11.7
KF	59.09

Table 2. Mass values for salt dissolution in ethanol at room temperature

Fluoride	Weight of the sample before dissolution, g	Weight after dissolution in ethanol, g
KF	0.4	0.39
LiF	0.4	0.38
NaF	0.4	0.15

Table 3. Mass values for salt dissolution in ethanol upon the application of heat

Fluoride	Weight of the sample before dissolution, g	Weight after dissolution in ethanol, g
KF	0.2	0.2
LiF	0.2	0.18
NaF	0.2	0.06