SH-SYNTHESIS OF POWDERS BASED ON TRANSITION METAL BORIDES

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New materials creation with different properties and level of quality based on widely used raw materials, including man-made, is currently determined by the tasks of scientific and technological progress. One of the main sources of boron ores in Kazakhstan are borates from the Inder deposit. These wide available raw materials can be used to obtain boron-containing refractory powder materials [1–3]. Popular methods of producing such materials are characterized by high energy consumption and high labor intensity. The use of self-propagating high-temperature synthesis (SHS) is currently one of the most effective approaches for creating new materials. An important role in obtaining materials in SHS mode is played by preliminary mechanochemical activation (MA), which allows achieving a high degree of particle dispersion and changing the structure, energy intensity, and high reactivity of the material [4].

To obtain samples of refractory powders of transition metal borides were prepared from a mixture containing powdered titanium dioxide, chromium oxide, enriched borate ore from the Inder deposit (boron oxide content up to 40%), and magnesium powder. Preliminary mechanical activation of samples was carried out in a Pulverisette 5 high-energy planetary-centrifugal mill. Green mixtures were prepared with a stoichiometric ratio of components.

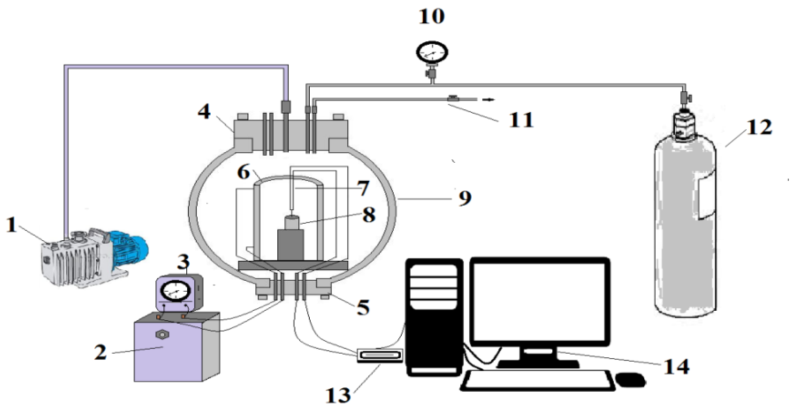


Fig. 1. High pressure reactor: *1* vacuum pump, *2* transformer, *3* ammeter, *4* upper reactor lid, *5* lower reactor lid, *6* tubular heating furnace, *7* thermocouple, *8* sample, *9* reactor vessel, *10* pressure gauge, *11* exhaust valves, *12* balloon with argon, *13* data acquisition system block LTR U 1, *14* computer.

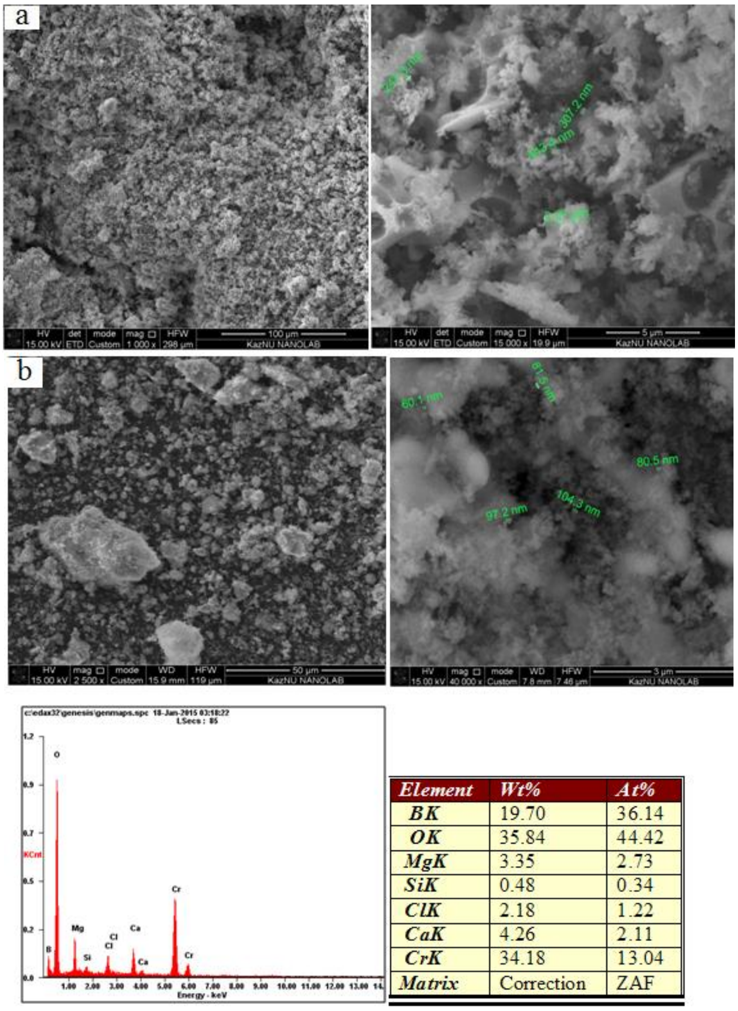
To carry out SH-synthesis in order to obtain refractory powders of titanium and chromium borides, the systems TiO_2–B_2O_3(ore)–Mg and Cr_2O_3–B_2O_3(ore)–Mg were selected. Experiments were performed in a high-pressure reactor (Fig. 1). After SH-synthesis, the obtained powders were leached with hydrochloric acid and washed with distilled water. X-ray phase analysis of samples was performed by a DRON-4M diffractometer using Co*K* radiation in the interval $2\theta = 10°–70°$. The morphology of the obtained samples was studied by scanning electron microscopy (QUANTA 3D 200i, FEI, USA).

The synthesis of titanium and chromium diborides took place in the following reactions in the combustion wave:

$$TiO_2 + B_2O_3 + 5Mg = TiB_2 + 5MgO \quad (1)$$

$$Cr_2O_3 + 2B_2O_3 + 9Mg = 2CrB_2 + 9MgO \quad (2)$$

The preliminary MA mixture was used. It is known that MA in a high-energy planetary mill contributes to a decrease in the powder particle size and an increase in the reactivity of components during SHS [3]. The phase composition of titanium products and chromiferous systems after SHS process and treatment with hydrochloric acid was investigated.



Element	Wt%	At%
BK	19.70	36.14
OK	35.84	44.42
MgK	3.35	2.73
SiK	0.48	0.34
ClK	2.18	1.22
CaK	4.26	2.11
CrK	34.18	13.04
Matrix	Correction	ZAF

Fig. 2. Microstructure of SHS products of the Cr_2O_3–B_2O_3(ore)–Mg system: (a) without MA, (b) MA for 5 min.

SEM studies showed the formation of crystal phases of combustion products of the Cr_2O_3–B_2O_3–Mg system (Fig. 2) without and with MA (nanoparicles). Similar results were obtained for the system TiO_2 + B_2O_3(ore) + Mg.

Thus, it is shown that the combined use of MA and SHS makes it possible to obtain submicron and nano-sized powder of titanium diboride (up to 98.2%), chromium diboride (up to 98.6%).

It is established that the use of MA contributes to the formation of nanoscale particles of refractory powders of titanium and chromium borides.

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