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COMBUSTION PROCESSES

INVESTIGATING THE COMBUSTION OF ENERGETIC MATERIALS WITH A BORON ADDITIVE BY THE METHOD OF SMALL-ANGLE X-RAY SCATTERING

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The authors have presented results of investigation into the process of combustion of solid specimens consisting of ammonium perchlorate and a binder with boron and aluminum-dodecaboride additives by the method of small-angle x-ray scattering with a high time resolution using high-speed optical photography. An analysis has been made of the particles preserved after the combustion. It has been shown that the procedure of small-angle x-ray scattering permits recording particles of the B and AlB_{12} additive directly in the combustion wave of the compound.

Keywords: *small-angle x-ray scattering, synchrotron radiation, combustion of boron.*

Introduction. The influence of adding aluminum and boron to energetic materials on the parameters of their combustion is actively investigated by theoretical and experimental methods [1–4]. One experimental problem is the complexity of analyzing the degree of reaction of the additive directly in a combustion wave. The particle's shape in the combustion wave can be determined by the method of small-angle x-ray scattering (SAXS).

In the present work, a study has been made of the combustion of cylindrical specimens of ammonium perchlorate with the boron (B) and aluminum-dodecaboride (AlB_{12}) particles of diameter and length about 1 cm in an air atmosphere at normal pressure. Basic characteristics of the specimens under study are given in Table 1.

Experimental Station and Experimental Procedure. Experiments on measuring small-angle x-ray scattering were conducted at the Experimental Station "Extremum State of a Substance" of the Center for Collective Use "Siberian Center of Synchrotron and Terohertz Radiation" [5] with a VÉPP-4M accelerator complex of the Institute of Nuclear Physics of the Siberian Branch of the Russian Academy of Sciences. For dynamic experiments, use was made of the measuring diagram presented in Fig. 1.

A synchrotron-radiation (SRS) beam was formed by a Kratky collimator consisting of two knives K1 and K2 in front of the explosive chamber, and also by horizontal slots and had the dimension 0.5 × 0.5 mm. A straight beam was cut by the K3 knife located in front of the detector. Scattered radiation was recorded by a DIMEX detector [6] with an angular resolution of $3 \cdot 10^{-5}$ rad. The detector permitted recording 100 frames with a minimum time between the frames of 100 ns. The periodicity of SP pulses in the experiments was 611 ns and was determined by the operating regime of the accelerator complex. The specimen under study was placed at the center of the explosive chamber at a distance of 3432 mm to the detector. The experimental procedure was described in [7–9] in detail. The rate of combustion of the specimens under study was about 1 mm/s, which enabled us to increase the time between the frames to 0.175 s, and the exposure time, to 61 μs (scattered radiation from 100 electron bunches was integrated).

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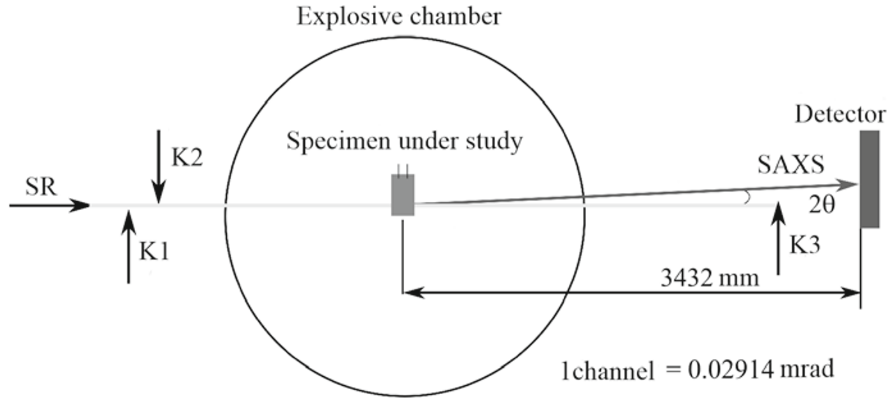

Fig. 1. Scheme of experiments on measuring small-angle x-ray scattering.

TABLE 1. Basic Characteristics of the Specimens Under Study

Fuel	Mass fraction, %				v, mm/s
	Binder	AlB_{12}	B	Ammonium perchlorate	
Compound with an aluminum-dodecaboride additive	19.7	0	15.7	64.6	1.2 ± 0.1
Compound with an boron-dodecaboride additive	19.7	15.7	0	64.6	0.8 ± 0.1

TABLE 2. Composition of the Original Additives and Solid Combustion Products of the Specimens under Study

Element	Atomic fraction, %			
	B		AlB_{12}	
	Original additive	Solid combustion products	Original additive	Solid combustion products
Boron	98.27	75.19	90.66	75.02
Aluminum	0	0	7.54	5.59
Carbon	0	8.09	1.8	16.02
Nitrogen	0	6.85	0	0
Oxygen	1.73	9.88	1.8	3.37

Plots of the intensity of radiation from the combustion of compounds with a boron and aluminum-dodecaboride additive versus the vector of scattering are shown in Fig. 2. Figure 3 gives the integral signal of small-angle x-ray scattering. The radiation intensity decreases proportionally to the scattering vector: $I = 1/q^4$, which corresponds to the scattering on homogeneous particles. The exponent of the power dependence changes with increase in the signal intensity with variation in the conditions of scattering in the combustion wave only slightly. Scattering on the reaction products upon the traversal of the combustion wave is also described by the power dependence $I = 1/q^3$. The slight change in the dependence of the scattered radiation on the angle of scattering speaks of the insignificant change in the shape of boron-additive particles in the combustion wave. The integral signal of small-angle x-ray scattering points to the presence of nanosize inhomogeneities, and its dynamics permits determining the kinetics of these inhomogeneities. For the specimens under study, the signal of small-angle x-ray scattering increases for approximately 1 s, and thereafter decreases approximately

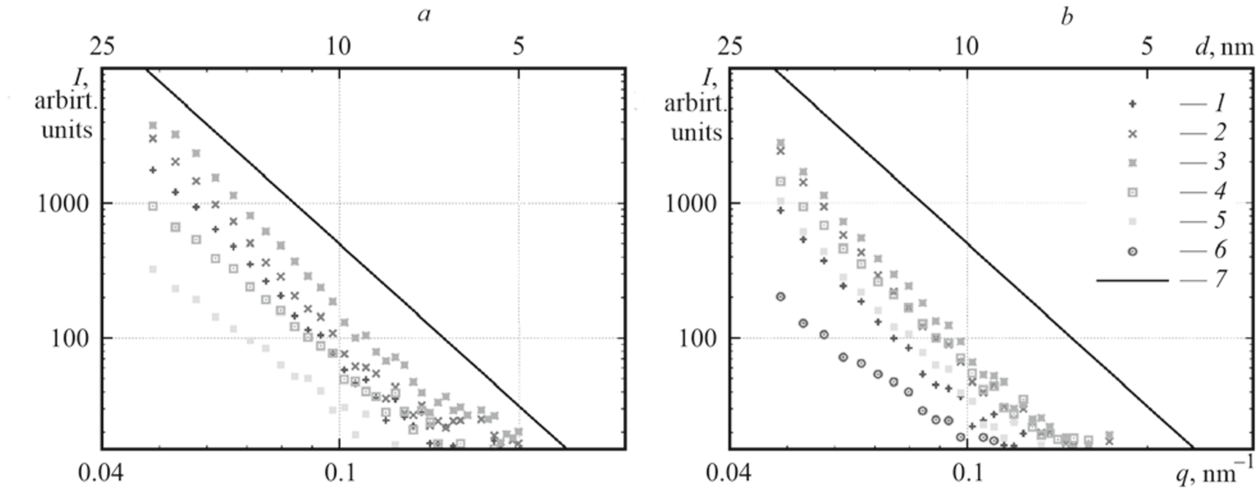

Fig. 2. Experimental dependences of the intensity of radiation from the combustion of the compound with boron (a) and aluminum-dodecaboride additives (b) on the scattering vector at the instants of time t = 0 (1), 0.5 (2), 1.0 (3), 1.5 (4), 2.0 (5), and 2.3 s (6): 7) q^{-4}.

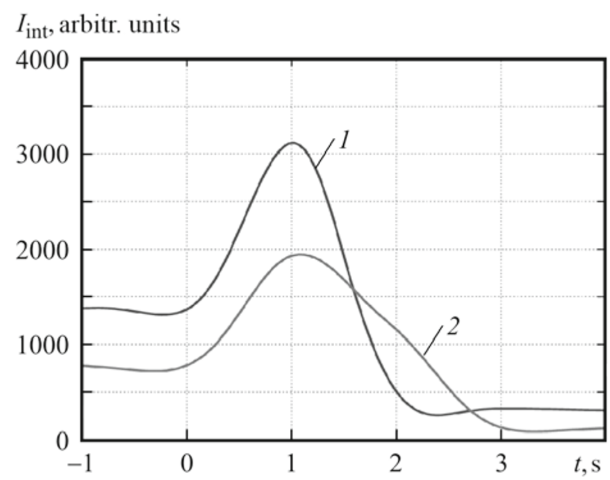

Fig. 3. Experimental dependences of the integral radiation intensity on time in combustion of compounds with boron (1) and aluminum-dodecaboride additives (2).

over the same period for pure boron, and in the case of boron dodecaboride, the time of decay in the intensity of the signal of small-angle x-ray scattering is about 2 s. Growth in the small-angle x-ray scattering signal is due to the increase in the contrast of scattering particles of the additive in the combustion wave [10, 11]. Before the arrival of the combustion wave, the particles are in the volume of the dense specimen, and in the process of combustion, they turn out to be surrounded by the gaseous rarefied reaction products; this tends to increase the intensity of scattered radiation. The decay in the small-angle x-ray scattering signal is associated with the escape of the particles from the region of observation.

Studying the Preserved Residue by the Scanning Electron Microscopy Method. The original particles of the boron additive and the products collected after the combustion were investigated with an electron microscope (EM) (Figs. 4 and 5). A comparison of the obtained images confirms the weak degree of reaction of the boron additive and insignificant change in the particle's shape in the combustion wave. There is also no oxygen in the elemental composition of the condensed residue preserved after the combustion, which confirms the weak degree of reaction of boron additives (Table 2).

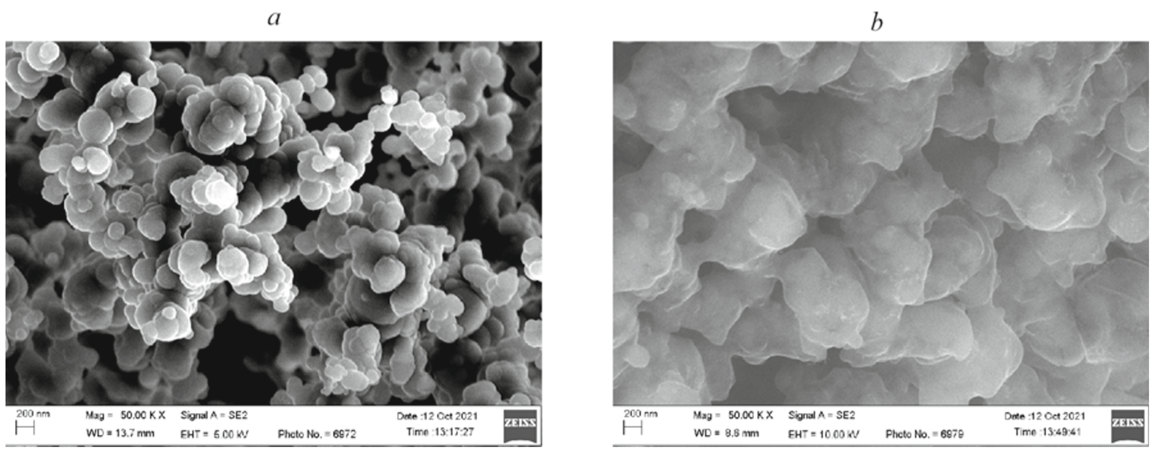

Fig. 4. EM images of the original particles of the additive of boron (a) and the products of its combustion (b).

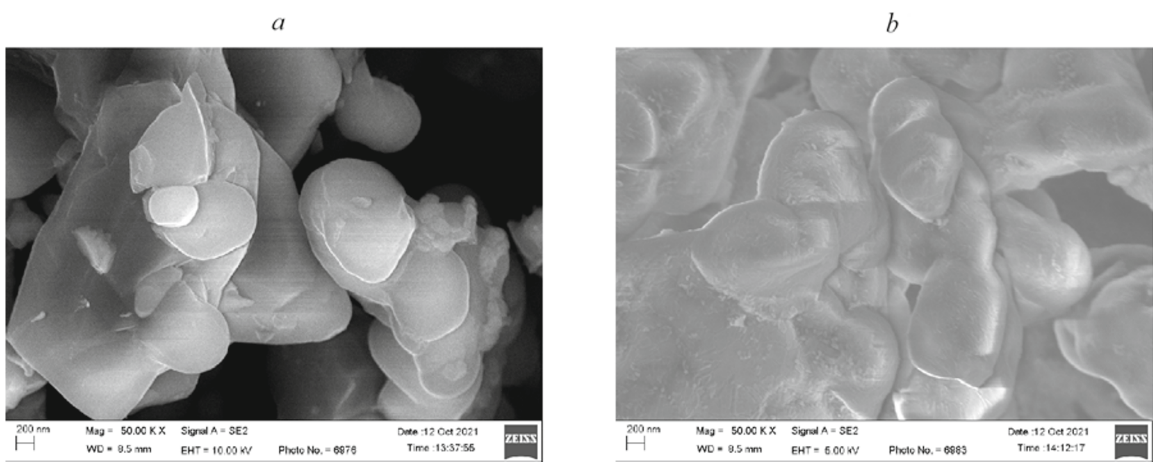

Fig. 5. EM images of the original particles of the additive of aluminum dodecaboride (a) and the products of its combustion (b).

Conclusions. The method of small-angle x-ray scattering permits determining B and AlB_{12} additives in the original specimen and directly in the process of combustion. There is a fundamental possibility of investigating the dynamics of transformation of metal to oxide in the torch of a composite fuel with a high time and space resolution. Changes in the metallic particles in the torch of the investigated specimen of the composite fuel turned out to be slight: in actual fact, the metal goes into the gas phase in nearly constant form and the degree of its transformation to oxide is small.

To obtain information on the combustion of boron-containing fuels, better conditions should be ensured for the combustion of the metal, namely, pressure should be increased and the torch of the specimen be encased in a protective tube.

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NOTATION

d, diameter of particles, mm; I and I_{int}, intensity and the integral intensity of radiation, rel. units; q, radiation-scattering vector; t, time, s; v, combustion-wave velocity, mm/s; 2θ, angle of scattering. Subscripts: int, integral.

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