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Pecyliarities of initiation by high-current electron beam of energy composites based on porous silicon with niobium boride and graphene additives

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The paper presented the results of experiments on ignition of samples of energy-saturated composites (ESCs) based on porous silicon (fuel) and sodium perchlorate (oxidizer) containing niobium boride and graphene powders by high-current electron beam. The addition of niobium boride, as compared to the addition of graphene, has an ambiguous effect on the flammability of ESC‘s samples.

Keywords: energy-saturated composite, porous silicon, niobium boride, graphene, high-current electron beam, ignition.

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Introduction

Energy-saturated composites (ENC) based on porous silicon (*por*-Si) with various oxidizing agents, since the beginning of the XXI century, have aroused increased interest among researchers in many foreign countries, including Russia [1–4].

Calculated and experimental values of the specific heat of explosive conversion Q (explosive conversion means burning, explosion, detonation [5]) for ENC based on *por*-Si exceed the corresponding values of specific heat Q both of individual secondary explosives and explosive compositions [3,5–7]. And, although, the detonation rate D is proportional to the square root of Q: $D \sim \sqrt{Q}$ [5], nevertheless, the measured values of the process speeds do not exceed 4.5 km/s [1,2,4,6–11]. It should be noted that the value of the reaction rate of 10 km/s over the sample surface, provided in [11], can hardly be considered as the detonation rate. Velocity maxima of 5000 and 6710 m/s were recorded at 67% silicon porosity in [10]. However, it was reported that these high velocity values were measured only during one or two frames of high-speed video, where the reaction front „jumps“ by 2 cm between frames in a video with 250,000fps. Low values of D, despite the high values of Q, naturally affect the explosive characteristics of these ENC (initiating and detonation abilities), which hinders their practical application.

At the same time, it is known [12] that the maximum heat of explosion $Q_{\max}$ (a measure of the maximum capability of releasing explosive energy in the process of conversion of chemical energy into thermal energy during explosive conversion (burning, explosion, detonation), which correlates with Q, is a quantitative indicator of operational safety (a set of indicators for mechanical and thermal sensitivity) of classical explosives of $C_aH_bN_cO_d$ type (i.e. secondary (disruptive) explosives) and can be used for their classification according to the hazard degree. This is probably also true for porous silicon-based ENC. For example, studies conducted in [13] showed that the impact and friction sensitivity of binary mixtures (*por*-Si + perchlorates) which have higher explosion heat than classical secondary explosives, is very high and is similar to the sensitivity level of classical primary (initiating) explosives.

Therefore, it is necessary to solve the problem of increasing Q with the concurrent reduction of sensitivity, at least to mechanical impacts, which will increase the operational safety of products with ENC (*por*-Si + oxidizer). The reduction of the sensitivity of explosives and compounds to mechanical impacts is known to be carried out either by phlegmatization or by diluting them with inert additives [14].

Powdered boron is often offered as an additive with high heat of combustion to increase the energy intensity of various energy-saturated materials (ESM), including rocket fuels and pyrotechnic compositions [15,16]. But along with this, boron has a number of constraining factors of its application: high ignition temperature ($\sim 1900\,K$), large values of ignition delay time and characteristic burning time. Therefore, aluminum diborides (AlB_2), titanium (TiB_2) and magnesium polyboride (MgB_{12}) [1,15–17] are proposed to replace it, which reduce the negative factors of boron and also increase energy the characteristics of these ESM due to their increased heat of explosive conversion (combustion heat) [18,19]. However, it should be noted that the introduction of inert powdered high-density metal additives (steel, tungsten) into individu secondary always reduces the detonation rate [20]. There is no data about the impact of high-density metal additives on composite ESM.

In light of what has been said, niobium boride (NbB) may be one of possible additives for ESM. Unfortunately, there is no data in the literature about its calorific value. But it can be assumed that it is much higher than calorific value of aluminum diborides (AlB_2), titanium (TiB_2) and magnesium polyboride (MgB_{12}), since the calorific value of niobium is much higher than the calorific value of aluminum, titanium and magnesium. In addition, the advantage of niobium boride is that it has a significantly higher density compared to boron and aluminum and titanium diborides ($7.5 g/cm^3$ vs. $2.4 g/cm^3$, $3.2 g/cm^3$, $4.52\,g/cm^3$ respectively). This can results in a more significant increase of charge density with its additives. As a result, there is a possibility of an increase of detonation pressure during the explosive conversion (EC) under the detonation conditions provided that niobium boride is completely oxidized [5].

However, it is not clear which concentrations of niobium boride additives in porous silicon-based ENC charges are optimal in terms of the ratio between energy intensity and sensitivity. Moreover, high concentration values may result in high energy intensity values, but also in low sensitivity which will create the problem of initiation of such charges. However, it is possible to assume that the above-mentioned ENC charges with niobium boride additives have a good application potential from the point of view of initiation by electrophysical impacts (electric discharge, exposure to a nanosecond high-current electron beam (HCEB), etc.).

Therefore, this paper studies the impact of niobium boride additives in ENC charges based on porous silicon (as a fuel) and sodium perchlorate (as an oxidizer) on their flammability in case of the exposure to nanosecond high-current electron beam. The electron-beam ignition initiation of priming (ignition) was chosen because nanosecond HCEB having a three-factor effect (thermal, mechanical and shock wave) on ESM [21] acts more efficiently on compositions containing particles of increased electrical conductivity and density [22].

1. Energy-saturated composites and experimental methods

por-Si$^+$ sodium perchlorate ($NaClO_4$) ENC in the ratio $50:50$ (mass fractions) was taken as the main energy-saturated composite. Porous silicon with a porosity of $\sim 66-68\%$ was obtained from $\sim 700-800\,\mu m$ thick plates of monocrystalline silicon doped with boron (grade KDB-100). The technology for producing porous silicon wafers, powder (powder particle size $35-45\,\mu m$) from them and an energy-saturated composite based on *por*-Si is described in [23–25].

Sodium perchlorate was chosen as an oxidizer because it is the most optimal (both in terms of its solubility and in terms of the resulting energy characteristics of composites) for ENC based on *por*-Si along with calcium perchlorate according to a number of researchers [1]. A solution of sodium perchlorate in ethyl alcohol and porous silicon powder was treated in 50 W ultrasonic bath „Sapphire" for 30 min at room temperature. As a result, $NaClO_4$ was adsorbed on the pore walls and evenly distributed in the mass of porous silicon.

Powdered niobium boride (particle size $20-30\,\mu m$) and multilayer (2–5 layers) graphene (Gr) containing hydroxyl groups OH^- (Fig. 1), were used as additives and as a result such graphene contains $\sim 9\%$ oxygen [26,27].

The graphene is chosen because it is an effective sensitizing additive in ESM when exposed to HCEB [28]. The concentration of niobium boride additives was 5, 10, 12.5, 15 and 20% by mass (in excess of 100% in basic ENC). The concentration of Gr additives in *por*-Si + $NaClO_4$ + NbB composite (5 mass.%) was 5 mass.% (over 100% relative to the main ESC) and 10 mass.% over 100% in *por*-Si + $NaClO_4$ composite (10 mass.%). Graphene and niobium boride were introduced into *por*-Si + $NaClO_4$ subsample in dry form which was mixed to homogeneous powder for obtaining *por*-Si + $NaClO_4$ + NbB or *por*-Si+ $NaClO_4$+NbB+Gr compositions.

All ENC were pressed into caps with an inner diameter of 4.55mm to a height of $\sim 1.1-1.2$mm (full cap depth 2.0mm). The mass of all pressed ENC was 32–44 mg, density was $\rho_c \approx 1.64-2.57\,g/cm^3$. It can be noted that the addition of graphene resulted in a deterioration of the conditions of pressing of the charge in the cap, and no

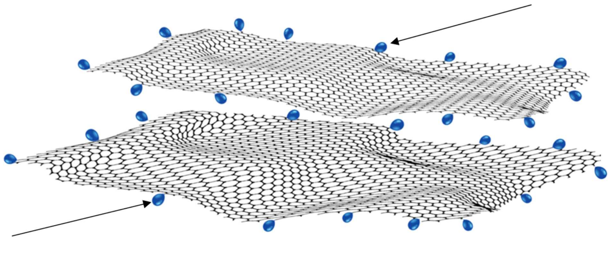

Figure 1. Two-layer graphene containing hydroxyl groups (indicated by arrows), open valences in graphene are closed with hydrogen.

Results of experiments on the initiation of ENCs

№	ENC	Density, g/cm^3	Result	Note
1	*por*-Si+$NaClO_4$	1.64–1.79	+	
2	*por*-Si+$NaClO_4$+NbB (5 mass.%)	1.83–2.0	±	Partial burnout 4–5 mg ENC
3	*por*-Si+$NaClO_4$+NbB (10 mass.%)	2.35–2.57	–	Surface darkening
4	*por*-Si+$NaClO_4$+NbB (12.5 mass.%)	2.4–2.6	+	$\sim$ 3 mg ENC was ejected by a wave of reflections into a vacuum chamber
5	*por*-Si+$NaClO_4$+NbB (15 mass.%)	2.25–2.46	+	
6	*por*-Si+$NaClO_4$+NbB (20 mass.%)	2.3–2.5	+	Two positive results and two negative results were obtained (incomplete burnout compositions)
7	*por*-Si+$NaClO_4$+NbB (5 mass.%)+Gr (5 mass.%)	1.84–2.01	+	
8	*por*-Si+$NaClO_4$+NbB (10 mass.%)+Gr (5 mass.%)	2.3–2.5	+	

Note. The results of three experiments are shown in every line (except the line 6), four experiments are shown in line 6.

increase of charge density was observed (see table, lines 6 and 7), and in some cases this resulted in a decrease of density relative to charges without graphene (see table, line 7).

The scheme of irradiation of an ENC sample in a nanosecond HCEB (30–40 ns at half height) cap (diameter 8 mm) is in principle similar to the scheme given in [29]. The average energy of the electrons in the beam was 250 keV. The experiments were carried out in a vacuum chamber with residual air pressure 10^{-2} Pa.

2. Experimental results and discussion thereof

The results of the experiments are listed in the table. The complete absence of an ENC charge in the cap was considered as a positive result of the explosive conversion that took place. Fig. 2 shows an example of an incomplete explosive conversion of a charge.

The type and conditions of explosive conversion remains unknown. The analysis of the results listed in the table showed the following.

Firstly, the results provided in the 4th line of the table indicate that the rate of explosive conversion of ENC (*por*-Si+$NaClO_4$+12.5% NbB) is comparable to the velocity of propagation of elastic waves in this composite. This conclusion is based on the fact that the shock wave initiated by the HCEB shock wave factor, passing through the ENC sample, partially reflected from the bottom of the cap and ejected a very small amount of composite (the rest of the composite burnt out as a result of the explosive conversion).

It can be assumed that the velocity of elastic wave in the composite is in the conditional range 3.5–4.5 km/s [30,31]. Therefore, the rates of explosive conversion are obviously in the same range in all cases of a complete burnout of the ENC. Can these values be considered as detonation rates? The question remains open, and additional study is required to answer it.

Secondly, a complete burnout takes place in the charges of ENC without additives and with graphene additives.

Thirdly, a complete burnout takes place at certain concentrations of NbB in ENC charges with niobium boride additives (without graphene additives). What can such results be related to?

Currently, the theory of „hot spots“ [5] has become the most widespread and developed in case of exposure of ESM (explosives) to energy pulses resulting in explosive conversions. The heating of explosives occurs in local sites (which are called „hotspots“) according to this theory. The heating of local sites to a critical temperature (T_{cr}) (flash point) results in a rapid exothermic reaction in them, i.e. to explosive conversion (burning). If concentration of burning spots is high enough, their combination results in the burning of a large charge surface, which as a result can lead to a higher explosive conversion mode (low-velocity or normal detonation).

The energy composition is close to an individual explosive in case of ENC charges without additives, in which the fuel and the oxidizer are in the same molecule. Then, the beam energy is released on almost every explosive particle or ENC pore filled with sodium perchlorate, together with the pore walls when an electron beam acts on a explosive charge or on a *por*-Si+$NaClO_4$ charge, i.e. the entire energy release region of the electron beam should be considered as a „hotspot“ or the initiation site, having, in the first approximation, the beam diameter and thickness equal to the extrapolated electron path [21]. Therefore, the beam energy is sufficient to create a „hot spot “ in almost every particle of the charge and thereby excite EC in the charge when the ENC without additives is exposed to HCEB.

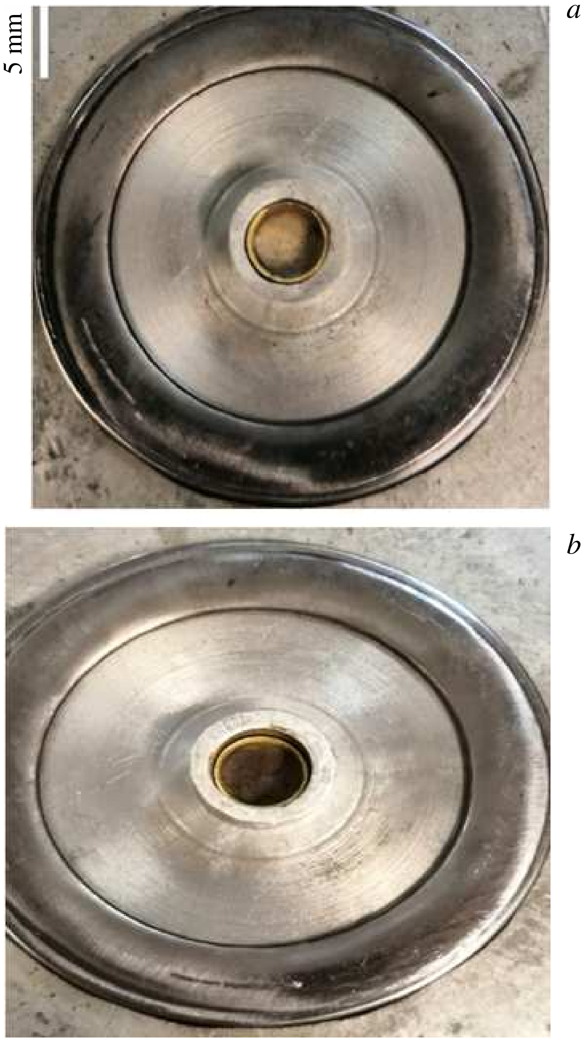

Figure 2. General view of samples after exposure to nanosecond HCEB: *a* — complete burnout of the ENC charge in the cap; *b* — incomplete burnout of the ENC charge in the cap.

However, the heating process can occur in another more complex scenario in case of exposure of ENC charges with NbB additives to HCEB. Since complete explosive transformation does not occur in the samples even with a relatively small concentration of niobium boride (5 and 10%), it is reasonable to assume that ignition sites are not formed in the main composite or their concentration is very low, i.e. the role of local heating sites in this case is performed by inert niobium boride particles, which are areas of increased density (together with nearby ENC particles). Such regions either attract electrons to a greater extent in accordance with general physical principles, thereby increasing the probability of their collision with NbB particles, or are absorbed by these particles passing through lighter particles of the main ENC. The inert niobium boride particles are accelerated as a result of such multiple collisions and are more intensively heated up to the melting point ($T_f = 2545\,\mathrm{K}$). The possibility of achieving such temperatures is evidenced by long-known methods of quenching various metals and alloys with a high-current electron beam of various durations [32]. It should also be taken into account that the specific heat capacity of niobium boride is two times lower than the specific heat capacity of the main composition ($c \approx 300-400\,\mathrm{J/(kg{\cdot}K)}$ for NbB vs.$700-750\,\mathrm{J/(kg{\cdot}K)}$ for *por*-Si+$NaClO_4$). Therefore, niobium boride particles can be heated according to the ratio (1) to higher temperatures even with the same radiation dose:

$$\Delta T = \frac{D_e}{C_1}, \tag{1}$$

where E_e — the amount of radiation energy ΔE absorbed per unit mass m of the irradiated substance (absorbed radiation dose): $D_e = \Delta E/m$.

NbB particles heated to high temperatures (for example, melting or close to it) actually heat up the nearby regions of the ENC to critical temperatures, causing thermal decomposition and rapid exothermic reactions (burning) in them. The combination of burning sites results in the combustion of most of the area of the ENC charge (excluding the area occupied by niobium boride particles), which can cause one of the modes of explosive transformation such as the low-velocity or normal detonation. The high temperature of the EC ($3500-4000\,\mathrm{K}$) of the main composition can result in complete decomposition of niobium boride particles, enhancing the full temperature effect of the EC of the entire composition and possibly its detonation parameters.

Obviously, a certain critical value n_{cr} of concentration of burning sites is a necessary condition for their complete fusion [33]. It is easy to explain the results of EC in samples with niobium boride (without graphene additives) based on this condition.

Their quantity is clearly insufficient in case of 5% concentration of NbB particles. Therefore, the particles of the main ENC act as hot spots, but the burning extinguishes since the number of such spots is also insignificant (because a large number of electrons fall on the particles of niobium boride). Their quantity is also insufficient in case of 10% concentration of NbB particles, but in this case the radiation dose attributable to the particles of the main ENC is very insignificant and therefore there are either no ignition sites in *por*-Si+$NaClO_4$ or their number is very small and the burning does not occur in the charge because of this. Finally, 12.5% concentration is a critical value starting from which the charge completely burns. But the energy of the active part of the charge (*por*-Si+$NaClO_4$) begins to be insufficient for the burning of its inert part (NbB) already at 20% niobium boride concentration which results in an incomplete explosive conversion of the charge in some cases.

The explosive conversion of ENC charges with graphene additives is probably explained by the following reasons. Firstly, as mentioned above, the graphene used contained additional oxygen located on the periphery of graphene sheets. The amount of oxygen in such graphene sheets reaches $\sim 9\%$. That is, the applied samples of ENC additionally contained 0.45% (mass.) (5% Gr) and 0.9% (mass.) (10% Gr) of oxygen, which is an active oxidizer. Secondly, graphene has (at least theoretically) high electrical conductivity. Moreover, it has an abnormally high thermal conductivity coefficient $\sim 5 \cdot 10^3$ W/(m·K) [34], which may result in a strong heat removal and a wide heating zone of the main ENC material adjacent to graphene particles, which can be heated by the action of an electron flow to very high temperatures, thus being a kind of hot spots. It should be noted that the melting point of graphene, according to various estimates, is in the range $T_{mel} = 3400-5000$ K [35,36]. However, there are studies [37], which show that thermal degradation processes result in the loss of approximately 20% of the mass of the bilayer graphene sample in an inert atmosphere when heated to 600°C, the graphene residue will disintegrate in an inert atmosphere at a higher temperature. Considering our results (the experiments were carried out in a vacuum and graphene was completely burned), it is possible to assume that graphene T_{mel} is still closer to the value of $T_{mel} = 3400$ K.

Conclusion

It was shown in this paper that niobium boride particles and graphene sheets can be used as active additional components of energy-saturated composites based on porous silicon. Both niobium boride particles and bilayer graphene particles play the role of hot spots, heating adjacent particles of the main ENC to their flash point. Moreover, niobium boride particles increase the charge density and thus can contribute to an increase of pressure in explosive transformation products.

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Conflict of interest

The authors declare that they have no conflict of interest.

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