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## Features and mechanism of combustion of energetic materials based on tungsten and polytetrafluoroethylene

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Abstract: Multifunctional energetic materials (reactive materials) under normal conditions are inert mixtures, but under strong mechanical or thermal impact they are capable of intensive exothermic reactions. For the development of reactive materials and optimization of their composition it is necessary to take into account the sequences of chemical reactions. The features of combustion in powder reactive material based on tungsten, polytetrafluoroethylene and aluminum have been investigated. Since the metal components react with the decomposition products of polytetrafluoroethylene, this stage was studied in separate experiments on combustion of metals with  $CF_4$  tetrafluoromethane. The use of tetrafluoromethane instead of polytetrafluoroethylene made it possible to determine that in the Al–CF4 system the reaction starts at 900–1000 °C, and in the W–Al system at 700 °C. Intensive evaporation of polytetrafluoroethylene in the W–PTFE–Al system prevents the reaction of tungsten with aluminum, which leads to ignition at 900–1000 °C. Combustion of two-component systems W–Al and Al–CF4 showed that the heat released during their interaction is insufficient for self-sustaining synthesis. Combustion of W–Al samples in tetrafluoromethane environment is realized in self-sustained mode. The reaction of tungsten with aluminum gives the initial impulse, and the main heat source is the interaction of aluminum with tetrafluoromethane.

Keywords: ignition; combustion; reactive materials; reaction mechanism; energy materials.

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# Особенности и механизм горения энергетических материалов на основе вольфрамаи политетрафторэтилена

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Аннотация: Многофункциональные энергетические материалы (реакционные материалы) при нормальных условиях являются инертными смесями, а при сильном механическом или тепловом воздействии способны к интенсивной экзотермической реакции. Для разработки реакционных материалов и оптимизации их состава необходимо учитывать последовательности химических реакций. Исследованы особенности горения в порошковом реакционном материале на основе вольфрама, политетрафторэтилена и алюминия. Поскольку металлические компоненты должны вступать в реакцию с продуктами разложения политетрафторэтилена, эта стадия выделена в отдельные эксперименты по горению металлов с тетрафторметаном CF4. Использование тетрафторутилена в место политетрафторутилена позволило установить, что в системе Al–CF4 реакция начинается при 900...1000 °C, а в системе W–Al при 700 °C. Интенсивное испарение политетрафторутилена в системе W–ПТФЭ–Al препятствует реагированию вольфрама с алюминием, что приводит к воспламенению при 900...1000 °C. Горение двухкомпонентных систем W–Al и Al–CF4 показало, что выделяющееся при их

взаимодействии тепло недостаточно для самоподдерживающегося синтеза. Горение образцов W–Al в среде тетрафторметана реализуется в самоподдерживающемся режиме. Реакция вольфрама с алюминием дает начальный импульс, а основным источником тепла является взаимодействие алюминия с тетрафторметаном.

**Ключевые слова:** воспламенение; горение; реакционные материалы; механизм реакции; энергетические материалы.

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## 1. Introduction

Multifunctional reaction materials (RM) under normal conditions are inert mixtures, but under strong mechanical or thermal impact they are capable of intense exothermic reaction [1]. There are three main types of RMs: thermite [2–4], intermetallic [5–7] and metal-polymer mixtures [8–12]. Metal-polymer RMs typically consist of polytetrafluoroethylene (PTFE) and one or more metals. This is a relatively new class of materials that differs from traditional energetic compositions (pyrotechnic compositions, rocket fuel, explosives) by high energy density and safety under normal conditions. High-velocity impact initiates an intense redox reaction (up to deflagration and explosion), releasing a large amount of thermal energy [13].

PTFE, being a source of strong oxidizers (carbon, fluorine), enables oxidation of metals with a high thermal effect (about 8 MJ·kg<sup>-1</sup> for the Al–PTFE system), which is significantly higher than that of TNT [14, 15]. In addition, PTFE is a technological additive - it improves the compactibility of powder compositions to a virtually pore-free state and acts as a protector from external influences (moisture, atmosphere). A metal-polymer mixture can combine sufficient strength, electrical and thermal conductivity of metal with high antifriction properties and chemical resistance of PTFE [16].

Despite its obvious advantages, Al–PTFE RM pressed from powders and sintered has low density and strength [17]. To improve the characteristics of RM, high-density additives in the form of tungsten, nickel, etc. powder are used [16, 18]. In [18], it was shown that with an increase in the content of tungsten or nickel in the Al–PTFE mixture, the strength and density of the composite increases, however, this leads to a decrease in the thermal effect of the reaction and sensitivity to impact initiation.

In addition, an urgent task is to increase the completeness of the reaction: during combustion of RM based on PTFE and high-density additives (W, Hf), the products contain a significant amount of unreacted metal.

A promising RM is a composition based on tungsten and PTFE. Calculations show that the optimal composition is a mixture with a tungsten content of 80 and 20 wt. % PTFE, which demonstrates a high combustion temperature (2115 °C) and a share of condensed products [19]. The main calculated products of this composition are condensed tungsten carbide W<sub>2</sub>C (53 wt. %) and graphite (3 wt. %), as well as gaseous tungsten fluorides WF<sub>6</sub> (14 wt. %), WF<sub>5</sub> (20 wt. %), WF<sub>4</sub> (9 wt. %). However, experiments have shown that binary W-PTFE systems are difficult to initiate by shock-wave loading and activating additives are required to increase the reactivity. Such an additive can be aluminum powder, which allows increasing the reactivity of difficult-to-initiate RMs [20, 21]. The completeness of the reaction and the level of energy release of such multicomponent RMs are difficult to predict; they depend heavily, among other things, on the initiation method (heating, impact).

Thus, to develop energy compositions and optimize their composition, it is necessary to take into account the sequences of chemical reactions. Since combustion is a chemical process, it is necessary to take into account the chain nature of PTFE combustion: thermal destruction occurs with a fast stage of chain decomposition of the polymer into Therefore, for further monomer links [22]. improvement of the compositions of new reaction mixtures, an integrated approach to solving the problem is required: the use of specific patterns of chain processes that contribute to an increase in the efficiency of additives. Since the metal components of the RM must react with the decomposition products of PTFE, this stage can be separated into separate experiments on the combustion of metals with the final product of PTFE decomposition tetrafluoromethane  $CF_4$  (TFM). The aim of the paper is to study the features of combustion and the reaction mechanism in RM based on tungsten and PTFE.

## 2. Materials and Methods

As a result of the calculation in the ISMAN-THERMO program [23], a powder mixture based on tungsten, PTFE, and aluminum with sufficient exothermic effect and forming solid end products with high specific gravity was taken as an object of study (Table 1). The maximum adiabatic combustion temperature ( $T_{ad}$ ) of 2866 °C corresponds to the composition with 10 wt. % Al with a high proportion of condensed products.

The initial components for RMs were tungsten powders PV-2 (particle size  $\leq 50 \,\mu$ m), polytetrafluoroethylene Fluralite ( $\leq 5 \,\mu$ m) and aluminum ASD-1 ( $\leq 50 \,\mu$ m). Dry mixing of the powders was carried out in a "drunken barrel" mixer for 3 hours with a drum rotation speed of 30 rpm with a ball to powder mass ratio of 5 : 1.

To measure the combustion rate, parallelepipedshaped samples with dimensions of  $5 \times 5 \times 20$  mm and a relative density of 0.7–0.8 were prepared using a manual hydraulic press (PGR-10, Lab Tools, St. Petersburg, Russia). Two blind holes with a diameter of 1 mm and a depth of 2 mm were drilled on the side surface of the samples for thermocouples (BP5/20 with a diameter of 100 µm), the distance between the holes was 14–15 mm (Fig. 1*a*). The samples were installed in a 20-liter reactor [24]. Air was pumped out of the reactor to a value of  $2 \times 10^4$  Pa, and then either an inert gas (argon) or tetrafluoromethane (CF<sub>4</sub>) was pumped in. The sample was initiated from the side end with a graphite plate by passing an electric current (Fig. 1*b*). The distance from the initiation point to the first thermocouple was about 3–4 mm. Signals from thermocouples were recorded at a frequency of 250 Hz via a QMBox analog-to-digital converter (R-Technology, Moscow, Russia). The X-ray phase analysis (XPA) was performed using DRON-3M. Samples were scanned in the range from 20° to 80° (2 $\Theta$ ) with a step of 0.02°. The quantitative analysis was performed using the corundum number method.

The ignition temperature was measured using the method described in [25, 26] on samples with a diameter of 5 mm and a height of 1.5-2.0 mm. The samples were ignited in a reactor (Fig. 2). The samples were placed on a 50 µm thick VR5/20 thermocouple in a boron nitride crucible and heated at a rate of 70–100 °C·s<sup>-1</sup> using a graphite plate to achieve thermal explosion conditions.

 
 Table 1. Calculated adiabatic combustion temperature and fraction of condensed products of W-PTFE-Al

Composition, wt. %	$T_{\rm ad}$ , °C	Condensed product, wt. %	Gas product, wt. %
76W-19PTFE-5A1	2776	C – 1 W <sub>2</sub> C – 77	$AlF_3 - 16; C_2F_2 - 1 CF_2 - 1; WF_4 - 3 WF_5 - 1$
72W-18PTFE-10A1	2866	$\begin{array}{c} C-2\\ W_2C-74 \end{array}$	$AlF - 7; AlF_2 - 5$ $AlF_3 - 11$
64W-16PTFE-20A1	1710	$W_2C - 66$ $Al_4C_3 - 4$	AlF – 29
56W-14PTFE-30A1	1382	AlF – 23	$W_{2}C - 57$



**Fig. 1.** Combustion rate measurement: *a*) sample; *b*) combustion rate measurement scheme: *I* – graphite heater, *2* – sample, *3* – thermocouples BP5/20, *4* – refractory substrate



**Fig. 2.** Measurement of the ignition temperature: *1* – graphite heater; *2* – thermocouple; *3* – boron nitride crucible; *4* – sample

#### 3. Results and Discussion

Under normal conditions, PTFE is inert because the chain of carbon atoms is surrounded by fluorine atoms, which block access to possible oxidants. However, when PTFE  $(-(CF_2-CF_2)_n-)$  is heated, thermal degradation of the polymer occurs, which begins with free-radical initiation. This process is followed by a rapid stage of chain rupture into monomer units from the free-radical end of the polymer chain. Thus, the rupture of the polymer chain leads to the formation of a large number of different free radicals. The main one is  $(R-CF_2)$ difluorocarbene radical [22].

$$-(CF_2-CF_2)_n \rightarrow nCF_3-CF_2^{\bullet}; \qquad (1)$$

$$2CF_2 = CF_2 \rightarrow C_4F_8 \bullet or cyclo-C_4F_8;$$
 (2)

$$CF_2 = CF_2 + CF_2 \bullet \to CF_2 = CF - CF_2 \bullet.$$
(3)

The final product of PTFE decomposition is the monomer tetrafluoroethylene (TFE):

$$-(CF_2 - CF_2)_n - \rightarrow nCF_2 = CF_2.$$
(4)

Since the system is constantly heated, TFE also undergoes decomposition. The final products of TFE decomposition are carbon and tetrafluoromethane CF<sub>4</sub> [27]:

$$CF_2 = CF_2 \rightarrow C + CF_4.$$
 (5)

Thus, aluminum and tungsten react with the decomposition products of PTFE:

$$4Al + 3CF_4 \rightarrow 4AlF_3 + 3C. \tag{6}$$

$$W + C \rightarrow WC. \tag{7}$$

With excess W content:

$$WC + W \rightarrow W_2C.$$
 (8)

The results of calculating the adiabatic combustion temperature and the proportion of products formed when replacing PTFE with TFM showed that the W–CF<sub>4</sub> composition behaves similarly to W-PTFE and demonstrates a relatively low combustion temperature (1464 °C) (Table 2). The main calculated products of this composition are condensed tungsten carbides, as well as gaseous tungsten fluorides (Table 2). The calculations showed that an increase in the aluminum content leads to a sharp decrease in the adiabatic combustion temperature (Fig. 3).

Ignition in the W–CF<sub>4</sub>–Al-based system began with the melting of aluminum and its interaction with tungsten to a temperature of 1200-1300 °C, regardless of the aluminum content (Fig. 4*a*). Then, a drop in temperature to 1100-1150 °C was recorded. A further increase in temperature to 1200-1400 °C was associated with the combustion of aluminum with TFM. An increase in the aluminum content increased the rate and duration of the reaction. Ignition of the W-PTFE-Al system began at a temperature of 800-1000 °C, since at this temperature intense evaporation of PTFE and the formation of TFM, which interacted with aluminum, begin (Fig. 4*b*).

 Table 2. Calculated values of the adiabatic combustion temperature and the proportion of condensed products of the W–CF<sub>4</sub>–Al

Composition, wt. %	$T_{ad}$ , °C	Condensed product, wt. %	Gas product, wt. %
80W-20CF <sub>4</sub> -0A1	1464	$WC - 32; W_2C - 23$	$WF_5 - 3; WF_6 - 41$
76W-19CF <sub>4</sub> -5Al	2416	$W_2C - 67$	$A1F_3 - 16; WF_4 - 8WF_5 - 7; WF_6 - 1$
72W-18CF <sub>4</sub> -10A1	2777	W <sub>2</sub> C - 74	$AIF - 5; AIF_2 - 5$ $AIF_3 - 16$
68W–17CF <sub>4</sub> –15Al	1777	W <sub>2</sub> C - 70	$AIF - 20; AIF_2 - 2$ $AIF_3 - 8$
64W-16 CF <sub>4</sub> -20Al	1277	$W_2C - 66; Al - 4$	AlF – 24; AlF <sub>3</sub> – 5

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**Fig. 3.** The adiabatic combustion temperature and the proportion of condensed products in the W–CF<sub>4</sub>–Al system depending on the aluminum content

According to the thermograms of the studied mixtures, it is evident that when PTFE is replaced by TFM, the combustion intensity increases (Fig. 4*a*).

This is manifested in an increase in the slopes and height of the peaks of the curves, as well as in a decrease in the ignition temperature. The lower intensity of ignition of the mixture with PTFE is associated with the process of polymer decomposition and the removal of decomposition products from the reaction zone.

To confirm the above mechanisms and the role of TFM in increasing the ignition intensity, tungsten and aluminum were heated both together and separately (Fig. 5*a*). When tungsten was heated in TFM, ignition did not occur, and the temperature increased only due to continuous heating. When aluminum was heated in TFM, ignition began at a temperature of 900 °C and reached 1300 °C, and then the temperature dropped to 1100 °C with a subsequent increase to 1300 °C. The main ignition products in the Al–CF<sub>4</sub> system were AlF<sub>3</sub> and Al<sub>4</sub>C<sub>3</sub> (Fig. 5*b*). The Al–W and Al–W–CF<sub>4</sub> systems ignited at 600–700 °C when heated.



Fig. 4. Thermograms of ignition of system samples: a) W-CF<sub>4</sub>-Al, b) W-PTFE-Al



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The main product in the mixture without TFM was the intermetallic  $Al_4W$ , and when TFM was used, tungsten carbides were formed (Fig. 5b). The conducted ignition experiments show that the intense evaporation of PTFE in the W–PTFE–Al system prevents the interaction of tungsten with aluminum. Thus, the interaction of aluminum with TFM provides enough heat to form tungsten carbides and aluminides.

The experimental results showed that the Al–CF<sub>4</sub>, 72 wt. % W – 10 wt. % Al systems are capable of ignition when heated, unlike W–CF<sub>4</sub> (Fig. 6*a*). As can be seen from the thermograms, the reactions of tungsten with aluminum and aluminum with TFM do not occur in a self-sustaining mode. High temperatures are not required for ignition of the W-Al system, but due to the low exothermicity of this reaction, the synthesis is not realized in a self-sustaining mode. To implement the self-sustaining mode in the Al–CF<sub>4</sub> system, a temperature significantly exceeding the melting point of aluminum is required; such conditions are created only near the heating element.

The experimental results on combustion of the system based on W–CF<sub>4</sub>–Al with different aluminum content show the possibility of implementing a self-sustaining reaction with the participation of three components (Fig. 6*b*). The reaction was initiated by the interaction of tungsten with aluminum, and the self-sustaining synthesis mode occurs due to the reaction of aluminum with TFM with a combustion temperature of about 1200–1300 °C (Fig. 6*a*). The combustion rate is minimal for the composition with 5 wt. % Al – about 0.3 mm·s<sup>-1</sup>. The maximum combustion rate for the composition with 20 wt. % Al was 1.4 mm·s<sup>-1</sup>, while for the compositions with 10 and 15 wt. % Al they were 0.5 and 0.6 mm·s<sup>-1</sup>, respectively.

The sample of the W–CF<sub>4</sub>–Al system with a content of 5 wt. % Al combusted partially, and the unreacted part showed tarnish colors (Fig. 7*a*). The samples with a content of 10, 15, and 20 wt. % Al combusted completely, forming a white coating on the surface, and the samples elongated from 20 to 30 mm (Fig. 7*b*).



Fig. 6. Combustion thermograms of the system: a) W-CF<sub>4</sub>, Al-CF<sub>4</sub>, W-10Al, W-CF<sub>4</sub>-10Al; b) W-CF<sub>4</sub>-5/10/15/20Al



Fig. 7. Samples after combustion W–CF<sub>4</sub>–Al: a) 5 % Al, b) 20 % Al



Fig. 8. XRD of the inner (a) and outer (b) parts of the W–CF<sub>4</sub>–Al samples separately

		Phase content, wt. %				
Sample	W Cubic Im-3m	W <sub>2</sub> C Hexagonal P-31m	WC Hexagonal P-6m2	AlF <sub>3</sub> Rhombohedral R-3c	Al <sub>4</sub> W Monoclinic Cm	
5 % Al outer	57	16	5	23	_	
5 % Al inner	79	16	1	4	_	
10 % Al outer	46	20	4	23	7	
10 % Al inner	55	16	2	15	12	
15 % Al outer	22	18	12	48	_	
15 % Al inner	34	20	4	25	17	
20 % Al outer	8	6	10	75	_	
20 % Al inner	40	40	15	5	-	

Table 3. Quantitative analysis of combustion products of the W–CF<sub>4</sub>–Al system

Due to the nature of combustion of the samples, X-ray fluorescence analysis was carried out separately for their external and internal parts (Fig. 8). Based on the intensity of the peaks of the formed products, it can be concluded that increasing the aluminum content reduces the amount of unreacted components both outside and inside the sample (Fig. 8).

The quantitative analysis of the outer part of the sample showed that an increase in the aluminum content leads to an increase in the proportion of AlF<sub>3</sub> from 5 to 75 wt. %. The proportion of WC increased from 4–5 to 10–12 wt. %, and the proportion of W<sub>2</sub>C in the compositions with 5, 10 and 15 wt. % Al was from 16 to 20 wt. %. In the composition with 20 wt. % Al, the proportion of W<sub>2</sub>C decreased to 6 wt. %. The quantitative analysis of the inner part of the sample shows that at 5 and 20 wt. % Al, the proportion of AlF<sub>3</sub> was about 5 wt. %. This fact indicates that aluminum fluoride is formed mainly on

the surface of the sample. An increase in the Al content in the sample leads to an increase in the formation of Al<sub>4</sub>W, WC and W<sub>2</sub>C to 17, 15 and 40 wt. %, respectively. As expected, the proportion of unreacted components is higher inside the sample (Table 3).

## 4. Conclusion

The combustion features and reaction mechanism in a reactive material based on tungsten with aluminum and polytetrafluoroethylene are investigated. At high temperatures, PTFE decomposes by a radical-chain mechanism, where the main decomposition product is the tetrafluoroethylene monomer. With further heating of tetrafluoroethylene, it decomposes to carbon and TFM. When PTFE is replaced by TFM, the ignition of the W–CF<sub>4</sub>–Al system begins with the interaction of tungsten with aluminum at 700 °C with a temperature increase to

1300 °C, and at 900–1100 °C, aluminum burns in TFM with an increase to 1300 °C. In the W–PTFE–Al system, ignition begins only at 900 °C due to intense evaporation of PTFE and loosening of the sample, which prevents the interaction of tungsten with aluminum.

Combustion of two-component W–Al and Al–CF<sub>4</sub> systems showed that the heat released during their interaction is insufficient for self-sustaining synthesis. Combustion of W-Al samples in the TFM environment is realized in a self-sustaining mode. The reaction of tungsten with aluminum gives the initial impulse, and the main source of heat is the combustion of aluminum in the TFM, which provides conditions for the formation of tungsten carbides. Increasing the aluminum content in the W–CF<sub>4</sub>–Al system increases the completeness of synthesis and the combustion rate from 0.3 to 1.4 mm·s<sup>-1</sup>.

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## 6. Conflict of interests

The authors declare no conflict of interests.

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