# Investigation of the metal powders activity based on aluminum, boron and titanium

Alexander Korotkikh
School of Energy & Power Engineering
Tomsk Polytechnic University
Tomsk, Russia
korotkikh@tpu.ru

Ivan Sorokin
School of Energy & Power Engineering
Tomsk Polytechnic University
Tomsk, Russia
ivans3485@gmail.com

***Abstract*—The high heat of oxidation makes boron and borides attractive as a metal fuels additive in composite solid propellants. Amorphous boron ignites at the temperature of more than 800 ℃. The formed $B_2O_3$ oxide layer during the combustion of boron prevents complete combustion and results in a higher burning time of the particles. Aluminum borides are characterized by a high melting point and when heated in an oxidizing environment they are oxidized to form $Al_2O_3$ and $B_2O_3$ oxides. This study investigates the metal powders activity based on aluminum, boron and titanium, used in composite solid propellant as additives. The paper presents data of metal powders activity: the onset temperatures of oxidation and the intense oxidation temperatures of the powders, the conversion coefficient and the weight gain of the samples in the temperature range of 400 – 1200 ℃.**

***Keywords—aluminium; amorphous boron; aluminum diboride; conversion coefficient; oxidation temperatures.***

## I. INTRODUCTION

Boron and boride powders are one of the most promising components of solid and hybrid rocket fuel due to their high specific energy during oxidation during its combustion. Among the usual solid and liquid fuels for the aerospace industry, boron has the highest calorific value (137.7 kJ/cm$^3$) [1, 2].

Boron is difficult to ignite and burn, it burns irregularly due to the high oxygen demand and high melting and boiling points [1, 3]. However, its use is greatly complicated by the fact that an inert oxide layer of $B_2O_3$ forms on the surface of boron particles during storage and combustion, which has a low melting point (450 °C) and a high boiling point (1860 °C) [1-4].

The additives of metallic powders and their oxides, such as magnesium, aluminum, titanium, zirconium, cerium, iron, copper, are used in composite propellant to improve their combustion parameters [5–9].

The borides of aluminum, magnesium, titanium and zirconium are the most promising additives. The use of metal borides can be widely used in high-energy materials since the oxidation state of $AlB_2$ is ~80%, the oxidation state of the initial boron powders is ~20%, and aluminum is 67% [10]. The combustion heat of these borides is much higher than the combustion heat of the corresponding metals. It is also worth noting that the interaction of Ti and Al with boron produces a large amount of energy, providing the basis for the process of self-propagating high-temperature synthesis (SHS) [10-11].

The SHS technology makes it possible to obtain inorganic compounds of various classes (carbides, borides, nitrides, hydrides, silicides, oxides, intermetallic compounds, etc.), both as individual compounds and more complex in composition. The advantages of SHS are low power consumption, ease of the process organization and purity of the resulting product. The principle of the SHS method consists of the exothermic interaction of the initial mixture components, proceeding in a specially organized mode of directional combustion. Due to the high reaction temperatures, the final synthesis products are obtained in the form of a cake that retains the original form of the mixture [10–11].

## II. TEST SAMPLES

To study the main parameters of the powders activity, we used aluminum powder grade ASD-4 with the mean diameter of $d_{43}$ = 10.8 μm (Fig. 1); ultrafine powder (UFP) of aluminum Alex ($d_{43}$ = 0.18 μm) obtained in argon using the technology of electric explosion of conductors; amorphous boron powder B ($d_{43}$ = 2.0 μm); aluminum diboride AlB2 ($d_{43}$ = 6.2 μm); aluminum dodecaboride $AlB_{12}$ ($d_{43}$ = 2.3 μm) and titanium boride $TiB_2$ ($d_{43}$ =54.5 μm). The metal powders $AlB_2$, $AlB_{12}$ and $TiB_2$ are obtained by self-propagating high-temperature synthesis (SHS-method). The content of active metal in powders is 98.5 wt. % for aluminum ASD-4, 85 wt. % for UFP Alex and 99 wt. % for amorphous boron. For powders $AlB_2$ and $AlB_{12}$ the ratio of aluminum and boron is 55.5 wt. % Al and 44.5 wt. % B for aluminum boride and 17.2 wt. % Al and 82.8 wt. % B for dodecaboride aluminum.

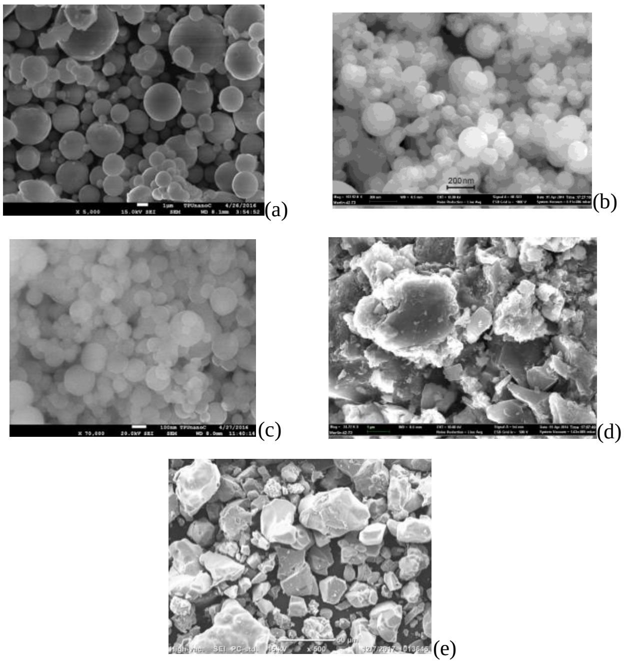

Fig. 1. SEM images ASD-4 (a), Alex (b), B (c), $AlB_2$ (d) and $TiB_2$ (e)

## III. RESULTS AND DISCUSSION

We performed a series of TG and DSC measurements on the heating of the studied powders. The study of the thermal oxidation process of powders was carried out using a combined analyzer Netzsch STA 449 F3 Jupiter in argon at a heating rate of 10 °C/min. The samples weighing ~10 mg were used for TG and DSC analysis, which were placed in a platinum crucible and then placed in a heating furnace. For the studied samples containing various metallic fuel TG and DSC data were obtained, the lines of which are presented in Fig. 2.

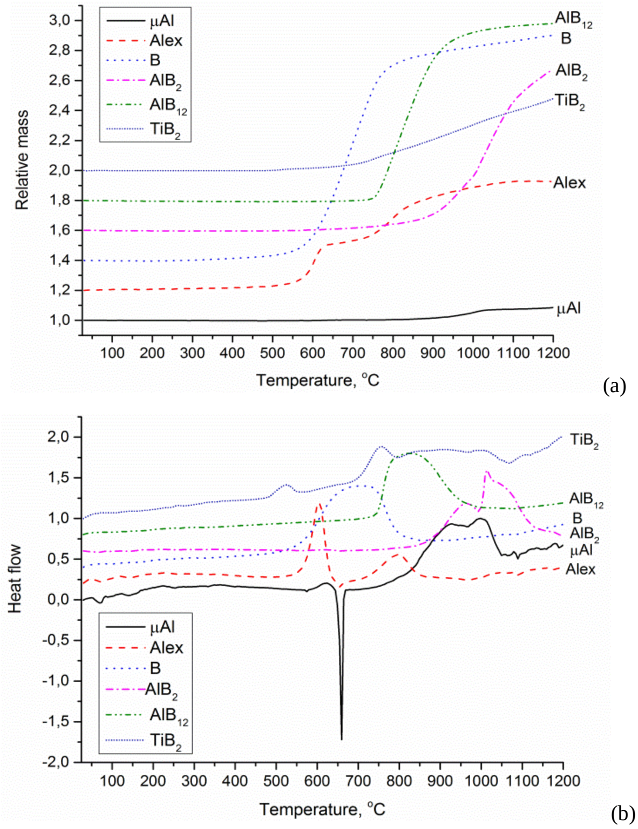

Fig. 2. Fig. 2. TG (a) and DSC (b) data of the powders ASD-4, Alex, B, $AlB_2$, $AlB_{12}$ and $TiB_2$

The particles oxidation of UFP aluminum Alex and micro-sized powder ASD-4 is carried out at different temperatures and exothermic specific heat flux. The oxidation of Alex powder begins at a temperature of ~570 °C with maximum heat release at 600 °C. The endothermic melting of aluminum occurs at a temperature of 660 °C with a sharp increase in the mass of the Alex sample due to the formation of aluminum oxide $Al_2O_3$. There is an insignificant heat release due to the oxidation of ASD-4 particles in the temperature range 580 – 640 °C, which then passes into the endothermic process of aluminum melting at a temperature of ~ 660 °C. Upon subsequent heating to 1200 °C, a slight increase in the sample mass is observed due to the formation of $Al_2O_3$.

The oxidation of amorphous boron powder begins at a temperature of 560 °C and is accompanied by intense heat release. A decrease in the mass of boron sample is observed upon heating above 800 °C. The samples $AlB_2$ and $AlB_{12}$ oxidation begin at temperatures of 880 and 750 °C, respectively. Aluminum diboride $AlB_2$ shows a sharp increase in the sample mass to a temperature of 1200 °C (Fig. 2). The oxidation of titanium boride is accompanied by a gradual increase in the sample mass up to a temperature of 1200 °C compared with powders of aluminum boride and boron.

We determined the values of the onset and intensive oxidation temperatures, the weight gain, the conversion coefficient and the oxidation rate of the studied powders. The activity parameters of the powders are presented in Table 1 and Table 2.

TABLE I. THE ACTIVITY PARAMETERS OF THE POWDERS: ONSET AND INTENSE OXIDATION TEMPERATURES, WEIGHT GAIN

| **Sample** | **$T_{on}$, °C** | **$T_{int}$, °C** | **Weight gain Δ*m*, %** | | |
|---|---|---|---|---|---|
| | | | ***400 – 660 °C*** | ***660 – 1200 °C*** | ***400 – 1200 °C*** |
| ASD-4 | 830 | 960 | 0.40 | 8.31 | 8.71 |
| Alex | 570 | 604 | 29.5 | 41.5 | 71.0 |
| B | 559 | 708 | 47.8 | 101.0 | 148.7 |
| $AlB_2$ | 876 | 1020 | 1.30 | 106.6 | 107.9 |
| $AlB_{12}$ | 747 | 826 | 0.60 | 118.3 | 118.9 |
| $TiB_2$ | 482 | 825 | 2.97 | 45.1 | 48.1 |

TABLE II. THE ACTIVITY PARAMETERS OF THE POWDERS: THE CONVERSION COEFFICIENT AND THE OXIDATION RATE

| **Sample** | **Conversion coefficient α, %** | | | **Maximum oxidation rate, $v_{max}$, mg/s (at T range, °C)** |
|---|---|---|---|---|
| | ***400 – 660 °C*** | ***660 – 1200 °C*** | ***400 – 1200 °C*** | |
| ASD-4 | 0.45 | 9.48 | 9.94 | 0.00045<br>(978 – 1017) |
| Alex | 39.0 | 54.9 | 93.9 | 0.0014<br>(589 – 617) |
| B | 21.7 | 45.96 | 67.7 | 0.0089<br>(651 – 748) |
| $AlB_2$ (Al/B) | 0.8/0.26 | 66.5/21.4 | 67.3/21.6 | 0.0051<br>(1006 – 1077) |
| $AlB_{12}$ (Al/B) | 0.12/0.22 | 22.9/44.1 | 23.0/44.3 | 0.010<br>(777 – 870) |
| $TiB_2$ | - | - | - | 0.0012<br>(734 – 787) |

The value of the conversion coefficient can be determined by the following formula:

$$\alpha = +\Delta m/\,(C_{Me} \cdot k)\cdot 100\%, \qquad (1)$$

where +Δm, % is weight gain; $C_{Me}$, wt. % is the content of the active metal in the sample; *k* is the oxidation level in the case of incomplete oxidation, $C_{Al} = 0.89$, $C_B = 2.22$.

According to Table 2 micro-sized aluminum powder ASD-4 has a lower conversion coefficient compared to Alex UFP in the temperature range of 400–1200 °C. The aluminum dodecaboride powder $AlB_{12}$ has a maximum oxidation rate of 0.01 mg/s, the minimum oxidation rate (0.00045 mg/s) is observed for the aluminum powder ASD-4.

## IV. CONCLUSION

The experimental and calculated studies of the powders activity were carried out for powder of aluminum, boron, borides of aluminum and titanium. The data of thermal oxidation of the powders are presented using a combined Netzsch STA 449 F3 Jupiter analyzer in argon at a heating rate of 10 °C/min. The using a combined TG and DSC analyzer, it was found that the onset temperature of powders intense oxidation varies from 600 to 960 °C and depends on the dispersion and type of metal powder.

We established the onset temperatures of oxidation and the intense oxidation temperatures of the powders, the conversion coefficient and the weight gain of the samples in the temperature range of 400 – 1200 °C. The maximum oxidation rate of powders was determined: the aluminum dodecaboride $AlB_{12}$ (0.01 mg/s) has the highest value, the smallest is the micro-sized aluminum powder ASD-4 (0.00045 mg/s).

## ACKNOWLEDGEMENT

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