Study of the free falling particles trajectory at the burning monolithic titanium particles

N S Belousova, O G Glotov, A V Guskov
Novosibirsk State Technical University (NSTU), 20 Karl Marx street, Novosibirsk, 630073, Russia

E-mail: nata.bel.94@mail.ru

Abstract. The technique for producing the large titanium monolithic burning particles with a diameter of 250-550 microns is developed. The combustion of titanium particles in free fall in air was investigated. The characteristic times for the following events – beginning of fragmentation, end of fragmentation, end of burning, as well as the particle’s motion law (including the coordinate and the velocity at the moment when the fragmentation process starts) are defined using the video recording. The size of particle at which the fragmentation picture changes from "star" to "spruce branch" is estimated. The combustion condensed products of particle are sampled and investigated. Three types of products are found. First are objects with aerogel structure with overall dimensions up to thousand microns consisting of the oxide spherules chains with an arithmetic mean diameter of spherules of 85 nanometers. Second are spherical oxide particles with diameter of units–tens of microns. Third are the spherical residues of mother particles with sizes up to hundreds microns (in the case of fragmentation in spruce branch mode).

1. Introduction
Titanium is fourth prevalence of structural materials after Al, Fe, Mg. On the one hand, Ti is a light, high-strength and corrosion-resistant structural material used in extreme conditions (in rocket-space and aviation equipment [1], in chemical reactors, etc.). On the other hand, Ti is pyrophoric metallic fuel. In this capacity, Ti is used as part of pyrotechnic compositions [2] and in compositions of technological combustion [3]. The possibilities of using Ti in rocket fuel [4] and in explosive mixtures of technological purpose are studied. The fundamental studies of Ti combustion were carried out at the time of its introduction into technology as a structural material in the 1960–1970s. The works were aimed at the determination of practically demanded characteristics of ignition and combustion of titanium samples (not particles). Using of powder (that is, particles) of Ti as a metallic fuel in pyrotechnics and technological combustion systems did not require a detailed understanding of combustion mechanism of particles. However, using of Ti particles burning in the air to create a cloud of photocatalytically active TiO₂ particles (TiO₂ - the main product of titanium burning) was proposed in 2005 [5] to deactivate harmful or hazardous substances trapped in atmospheric air as a result of a man-made accident or terrorist act. The study of the combustion mechanism of Ti particles is aimed at solving specific problems, namely, the study of the influence of the size of parent Ti particles and the conditions of their burning on the burning time and on the characteristics of the TiO₂ particles formed. In the future, it is required to establish how to organize the combustion process for the effective conversion of metal to oxide with the required properties. In the end, we can think about development of a technical device that implements of titanium particles burned in the air to generate photocatalytic titanium dioxide “on request”, that is, in the right place at the right time. It should be noted that the
The latter requirement is extremely difficult to implement on the basis of the gas-dispersed flare principle [6], [7], used as a method for the production of oxide nanoparticles.

Let's summarize the results of previous studies.

In the combustion wave of mixed system, including powdered solid oxidizer, polymer binder and powdered titanium with a particle size of the order from units to tens of microns as a metal fuel, metal agglomeration takes place. As a result, agglomerates with characteristic sizes of the order of hundreds of microns instead of the original titanium particles, fly out from the burning surface [8]. Complete combustion of such particles with a significant yield of finely dispersed oxide is almost impossible. Therefore, the approach has been proposed in [9] allows us to produce monodisperse burning titanium agglomerates. In [10], empirical information about large particles combustion of agglomeration origin with a diameter of 300, 390, and 480 µm in free fall in air is presented. The characteristic times of the onset of fragmentation, the end of fragmentation, the end of combustion, as well as the velocity and coordinate of the particle at the corresponding time moments were determined through video filming.

The regularities of the burning particles motion, the remnants of combustion of parent particles, the products of combustion in micron [10] and nanometer ranges were investigated.

The peculiarity of burning titanium particles and titanium agglomerates is the phenomenon of fragmentation, which can occur in two modes, namely, as a single explosion of a burning particle with a star-like scatter of fragments, or as a set of small particles-fragments stretched out in time from the parent particle with preservation the last. In this mode, the photographic image of the burning particle track with flying off fragments resembles a spruce branch. The “spruce branch” mode was observed in [10] for agglomerates with a diameter of 300, 390 and 480 µm. The "star" mode was observed for particles of 240 and 280 microns in [11] and particles in the range of 20–125 microns in [12].

Obviously, the phenomenon of fragmentation reduces the burning time and contributes to the efficiency of original metal transformation into a highly dispersed oxide, and the “star” mode is preferable. It follows from the mentioned above that the determination of particle size, at which the fragmentation regime changes, as well as the establishment of quantitative fragmentation parameters and response features of agglomerates and monolithic particles is a relevant research direction which important for increasing the combustion efficiency. In this work, the approach [9, 10], developed for agglomerates with a diameter of 300–480 µm, was adapted and used for monolithic particles with a diameter of 250–550 µm, which made it possible to correctly compare a number of parameters.

The objectives of the work is development of "technology" to create burning monolithic particles of a given size, establishing the times of fragmentation and burning of these particles, determination of granulometric characteristics of particle-products of combustion, including in the nanoscale range.

2. Experimental methods

The essence of the original approach [9], designed for creating the objects of study - burning particles with given parameters is to follow. A sample is used consisting of metal-free fuel matrix, in which a certain number of identical miniature metalized inclusions are placed. Inclusions can be pieces made from metalized mixed fuel [12] or spherical metal particles [13]. In the first embodiment, in the burning wave of the matrix, each inclusion turns into a burning agglomerate particle. The mass, composition and structure of the agglomerates were predetermined by the recipe and the size of inclusions. In the second variant, the size of the burning particle is initially set. In all cases, the metalless matrix is ignited and ejects particles. Depending on the experiment formulation, the further combustion of the particles occurs either in the air [9, 10] or in the combustion products of the matrix [13, 14].

The sample is a strip of metal-free matrix with embedded inclusions, deposited on a quartz tube as it is shown in Figure 1.
Figure 1. Photograph of the finished sample.

The tube with a diameter of 12 mm plays the role of the sample holder and is used repeatedly. Composition-matrix has the consistency of clay, easily formed and holds the shape. Its composition was given in [10]. Typical stripe dimensions W × L × H (width × length × height) are about 3 × 30 × 2 mm. In the matrix strip, cylindrical holes with a diameter of 1.2 mm and a depth of about 1 mm were made, in which titanium inclusions are placed. Unlike experiments [9, 10] with titanium agglomerates, in the present work the inclusions were monolithic miniature flat pieces of metallic titanium. Inclusions were made as follows. A piece of metal with size of 2-3 mm was cut off from an ingot of metallic titanium (purity 99.38%). The fragment was flattened on a titanium anvil with a titanium hammer then rolled on steel rollers to a thickness of 50–80 µm. As a result, a titanium “petal” was obtained, (Figure 2), from which the required number of pieces (usually 10) were cut off by scissors to place them in one sample (in the wells on the strip of the matrix). Figure 2 shows that the size of the pieces of inclusions about 1 mm. Experiments with different shapes of pieces (triangle, square, rectangle with L/W ≥ 3) did not reveal significant differences in the measured parameters. Orientation of titanium pieces in the hole is important at the laying. It was established that orientation the plane of the pieces perpendicular to the plane of the strip of the matrix and parallel to the axis of the tube is better contributes to ignition of the pieces.

Figure 2. Titanium “petal” and particles of inclusion cut off from it for one experiment.

Petal weight is 0.01–0.04 g, typical weight of one inclusion is about 0.0002 g. Direct weighing of inclusions on ordinary analytical scales with the required accuracy is impossible. The problem is solved by replacing the weighting of real objects with the weighting of their “models” cut from photographs printed on thick paper. The weight of a metal inclusion is easily recalculated from the
weight of its paper model, using the weights ratio of the whole metal petal and its paper model. Knowing the inclusion weight, the initial particle diameter after melting and tightening into a sphere can be calculated by the formula:

\[ D_i = \frac{\sqrt{6 m_i}}{\pi \rho_{Ti}} \]  

(1)

Where \( \rho_{Ti} = 4.5 \text{ g/cm}^3 \) is titanium density and \( m_{Ti} \) is titanium mass. The procedure described ensures the accuracy of determining the calculated diameter of 1–3 \( \mu \text{m} \) at hundreds of microns level (at the condition that the petal and the pieces cut off from it have the same thickness).

Experiments were performed as follows. The sample quartz holder tube was fixed horizontally at a height of 2–3 m so that the matrix strip was on the underside of the tube (Figure 3).

A strip of the matrix was set on fire from the end protruding beyond the end of the tube-holder. As the combustion wave passes through the strip of the matrix, the inclusions ignite and are ejected from the matrix. The escaping particles turn into burning spheres falling freely in the air. In the course of the experiment, shooting of burning particles was made by digital camera (Canon EOS 650D) at the shooting speed of 25 frames per second. In the experiments performed, forced extinguishing of particles was not used, and a high fall height (up to 3 m) ensured the combustion of particles of the sizes studied in flight. Frame-by-frame processing of video recordings was performed as described in [14]. For each particle, the particle coordinate was measured (current location relative to the sample holder tube), and characteristic events were recorded: the beginning of fragmentation, the end of fragmentation, and the end of burning. The definition of these events is shown on Figure 4. The burning time \( t \) corresponds to the moment when the particle and/or its fragments cease to glow and be visible.

Collection of combustion residues was carried out in one of two ways. First variant (Figure 3a) is using a large (\( \approx 80 \times 100 \text{ cm} \)) pallet of coated paper of A0 format which was used to collect predominantly combustion residues of the parent particles in case of their fall from a height of more than 2 m. Remnants of burning were taken from the pallet immediately after the experiment. In second

![Figure 3. The scheme of the experiment and two options (a, b) for the collection of combustion residues. 1 sample, 2 pallet, 3 camera, 4 Petri dish.](image)
variant (Figure 3b), the sample was located above the open end of a large quartz tube mounted vertically on a Petri dish. The tube had a diameter of 9 cm and a height of 220 cm. The burning and falling of particles occurred inside the tube. After experiment, the top of the tube was closed with a lid and left alone for a period more than three days, during this time the combustion products gradually settled on the dish. In this case, particles of combustion products of all types are fairly fully selected. Of course, there is a possibility of a certain number of particles sedimentation on the tube walls. All selected particles were subjected to particle size analysis using suitable visualization techniques. Use were made of macro photography, optical and electron microscopy.

![Image](image-url)

**Figure 4.** Videogram - a sequence of frames of burning particles Ti in free fall in the air.

3. **Experimental results**

Figures 5a and 5b, as an example, present the results of particle trajectory measurements in free fall. In the future, a set of such data will be used to determine the aerodynamic resistance coefficient of burning particles.

![Image](image-url)

**Figure 5.** Particle trajectory measurements: particle trajectory (a); particle velocity (b).

Three morphological types were distinguished among the selected products:
- spherical remnants of parent particles combustion. Its diameter is comparable to the original diameter of particles and amounts to hundreds of microns;
- spherical oxide particles with diameters of the order from units to tens of microns. These are the remnants of the combustion fragments;
objects with an airgel structure with dimensions of up to one thousand microns, consisting of primary oxide nanoparticles chains (so-called spherules) with diameters of tens nanometers.

The three main morphological types of combustion products are presented in Figures 6 and 7.

The residues diameter of the first type is hundreds of microns (Figure 6a), which is comparable with the initial diameter of the parent particles. After destruction it can be seen that the remains are hollow, and the shell thickness is about 20 microns (Figure 6b).

![Figure 6. First morphological type of combustion products under an optical (a) and electron (b) microscopes.](image)

Particles of the second type have a distributed electrical charge and can jump on each other and then remain in this position (Figure 7a). An airgel "cloud" of frozen smoke (third type), as well as several spherical particles, similar to that shown in Figure 7a, can be seen in Figure 7b. In this case, the airgel formation has an overall size of about 300 micrometers. In other cases, the airgel formations reached sizes of several millimeters, and often had an elongated shape similar to the shape of a smoke plume of a burning particle.

In the future we plan to conduct a detailed particle size analysis of selected particles.
4. Conclusion
Information about characteristic burning times and fragmentation characteristics of monolithic mother titanium particles with a diameter of 300–500 µm was obtained. The planned task is the quantitative characterization of highly dispersed combustion products and fragmentation of parent particles, including particle-products in the nanoscale range. In experiments, it is necessary to ensure the most complete capture of particle-products so that in the long term perspective, at least semi-quantitatively estimate the yield of certain products from a single parent particle-source.

Nomenclature
W (mm) – width of composition-matrix.
L (mm) – length of composition-matrix.
H (mm) – height of composition-matrix.
D_{Ti} (cm) – initial particle diameter.
ρ_{Ti} (g/cm³) – titanium density.
m_{Ti} (g) – titanium mass.
S (cm) – distance traveled by the particle.
V (cm/s) – particle velocity.
t (s) – time of particle falling.

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