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Macrokinetics of Combustion of Powder and Granular Titanium Mixtures with Different Allotropic Forms of Carbon

B. S. Seplyarskii[a,*], **R. A. Kochetkov**[a],
T. G. Lisina[a], **and N. I. Abzalov**[a]

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Abstract: Even a slight change in the content of impurity gases during a self-propagating high-temperature synthesis can lead to a change in the combustion regime and the characteristics of the target products. In this work, the dependence of the burning rate of Ti + C granular mixtures on a titanium particle size is determined for the first time, and the effect of impurity gas evolution when using various allotropic modifications of carbon (graphite/soot) is studied. Experimental results are analyzed using the convective–conductive combustion model, which explains the strong influence of impurity gas release on the front velocity. Interaction rate of the components becomes a key factor for granular mixtures in which the influence of impurity gases is leveled. Experiments show that the burning rates of granular mixtures of titanium with soot are noticeably higher than the burning rates of a mixture of titanium with graphite. The curves approximating the dependence of the burning rate of a granular mixture of titanium and graphite on the size of titanium particles correspond to the linear law of interaction of the initial components. The interaction in a mixture of titanium and soot occurs according to the parabolic law.

Keywords: SHS, macrokinetics, burning rate, powder mixtures, granules, impurity gas release, titanium particle size, soot, graphite.

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INTRODUCTION

The stability of the properties of cermet materials obtained by high-temperature self-propagating synthesis (SHS) [1] depends on the reproducibility of combustion parameters. At combustion temperatures of the order of several thousand degrees, impurity gases from powders of one or more components are often released [1–3]. Even a slight change in the impurity gas content can lead to a change in the combustion regime and the target product characteristics. In order to explain the strong effect of impurity gas release (IGR) on the front velocity, a convective–conductive combustion model (CCCM) is developed [4]. Such methods for stabilizing SHS processes, namely thermal vacuum treatment of initial samples, are rather long and energy consuming [5, 6]. Another approach is to granulate the initial powder mixture [7]. Granular mixtures are distinguished for the structure of the porous medium, which is different from that in powder mixtures. In accordance with the CCCM, this makes it possible to explain the leveling of the effect of the IGR on the burning rate and phase composition of the synthesis products [4, 7–9].

It is shown by the studies based on Ti–C (Ti + C + TiC, Ti + xC, Ti–C–Ni, or Ti–C–Cr) with carbon in the form of soot [8, 10–12] that the burning rate of granular mixtures is usually higher than the burning rate of powder mixtures of the same composition.

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[a] Merzhanov Institute of Structural Macrokinetics and Materials Science, Russian Academy of Sciences, Chernogolovka, 142432 Russia; *seplb1@mail.ru.

Table 1. Characteristics of initial components

Component	Brand	Particle size, μm	
		up to 50% (wt.)	up to 90% (wt.)
Titanium	PTM	<34	<54
	PTM	<105	<169
Carbon (soot)	P-803	<1	<2
Carbon (graphite)	Aldrich	<5	<14
Polyvinyl butyral (PVB)			
95% industrial alcohol			

Soot is a good adsorbent due to its high specific surface area. As hydrogen dissolved in titanium and gases adsorbed on the soot surface contribute in the IGR in the heating zone of the combustion wave, it is impossible to separate the effect of IGR from titanium on the burning rate. It is known from [2] that the evolution of gases from a Ti + 2B mixture with amorphous boron does not depend on the size of titanium particles, and the replacement of amorphous boron with crystalline boron makes the contribution of boron to the IGR insignificant. It is assumed in [2] that the most important question is why do impurity gases increase the burning rate of SHS mixtures in some cases, but not in others?

The purpose of this work is to determine how the replacement of amorphous soot with crystalline graphite affects the change in the burning rate upon transition from powder to granular mixtures of Ti + C with differently sized titanium particles. Another goal is to reveal the nature of such a change from the standpoint of the CCCM and establish the dependence of the burning rate of granular mixtures on the titanium particle sizes for various allotropic modifications of carbon.

MATERIALS AND METHODS OF EXPERIMENTS

The initial substances used in this work and their brief characteristics are given in Table 1. Two sizes of the PTM titanium powders are used in the experiments. A mixture with graphite is prepared by sieving narrow fractions from coarse grain titanium having particle sizes $d < 40$ μm, 40–80, 90–125, and 125–200 μm. A mixture with soot is prepared by sieving narrow fractions with particle sizes $d < 63$ μm, 63–90, 90–125, and 125–160 μm. The powder mixture densities ρ with different fractions of titanium are given in Table 2.

Table 2. Initial data and the parameter calculation results L, t, and $t_{\rm h}$ for the Ti + C powder mixture (soot/graphite)

d, μm (Ti)	ρ, g/cm^3	u, mm/s	$t_{\rm h}$, μs (Ti)	L, μm	t, ms
Ti + C (soot)					
31.5	1.10	12	31	83	7
76.5	1.11	8	183	125	6
107.5	1.08	7	361	143	20
120	**1.15**	**11**	**450**	**91**	**8**
142.5	1.10	5	635	200	40
Ti + C (graphite)					
20	1.25	25	13	40	1.6
35	**1.22**	**20**	**38**	**50**	**2.5**
60	1.20	10	113	100	10
107.5	1.3	7.5	361	133	18
120	**1.20**	**10**	**450**	**100**	**10**
162	1.3	6	820	167	28

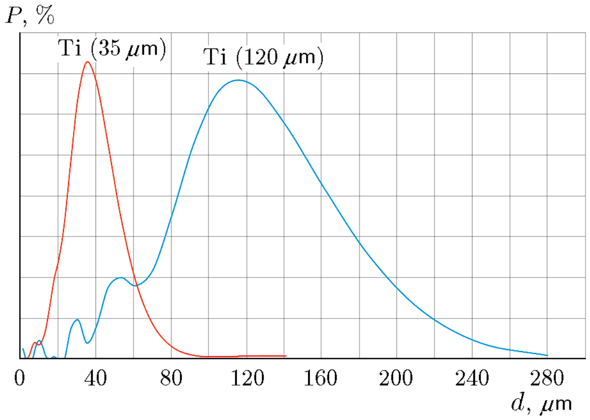

Fig. 1. Distribution of initial fractions by the titanium particle size.

The characteristic sizes d of Ti particles of the initial powders in the calculations are the values of the maximum size distribution: $d = 35$ μm for fine grain titanium and $d = 120$ μm for coarse grain titanium. Figure 1 shows the size distribution of titanium particles of the initial fractions. The characteristic particle sizes of narrow fractions are determined as the arithmetic mean of the upper and lower bounds of the fraction.

The experimental device and the method for granulating mixtures are described in [11]. In this work, granules with a fraction of 0.6–1.6 mm are used,

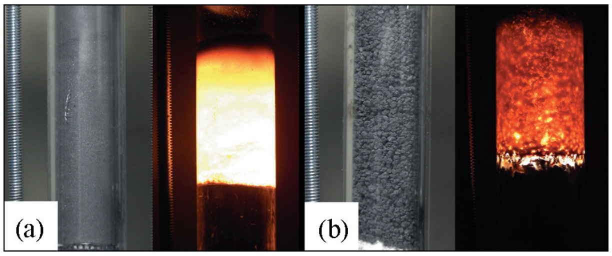

Fig. 2. Appearance of the initial mixtures and the images of the combustion of the powder (a) and granular (b) Ti + C (graphite) charge.

and the density of granular mixtures with graphite is 0.7–0.73 g/cm^3 and that with soot is 0.7–0.8 g/cm^3.

The particle size distribution of the components is determined on a Microsizer-201C laser analyzer. The combustion is recorded on video using a SONY FDR AX-700 video camera (a shooting speed of 100–250 fps). The phase composition of the final product is studied using a DRON-3M diffractometer using monochromatic Cu K_α radiation. X-ray diffraction patterns are obtained via step scanning in an angle range $2\theta = 20$–$80°$ with a shooting step of 0.2°. The resulting data are analyzed using the PDF-2 database.

RESULTS AND DISCUSSION

The video recording shows that the combustion of both Ti + C (soot/graphite) powder and granular mixtures is stationary. The combustion front is flat, which makes it possible to reliably determine the burning rate. Figure 2 shows the images of the combustion of the Ti + C (graphite) powder and granular charge. The combustion wave front of the Ti + C mixture (soot) looks similarly. The video camera settings are changed in the course of experiments with different batches to obtain the clearest image, so the difference in the brightness of samples in the photographs in this case does not indicate a different combustion temperature.

Figures 3 and 4 show the experimental values of the burning rate of the Ti + C (soot) and Ti + C (graphite) powder and granular mixtures as a function of the average size of titanium particles and the approximating curves based on data for narrow fractions. The deviation of the experimental data from the average values does not exceed 10%.

When analyzing the data presented in Figs. 3 and 4, one first draws attention to the fact that the burning rates of granular compositions exceed the burning rates of powder mixtures in the entire range of titanium particle sizes. The observed results are explained using the CCCM [4]. In accordance with this model, the combustion wave front propagation in a powder mixture is the movement of a melt of a fusible component under the action of capillary forces and a pressure difference of impurity gases ahead of and behind the melt layer. An increase in the pressure of impurity gases ahead of and behind the reaction front leads to a decrease and increase in the burning rate, respectively. Therefore, it is important to determine whether the particles of the initial components have time to warm up and release impurity gases ahead of the combustion front.

It is assumed that the particles of the initial components in the powder mixture have enough time to

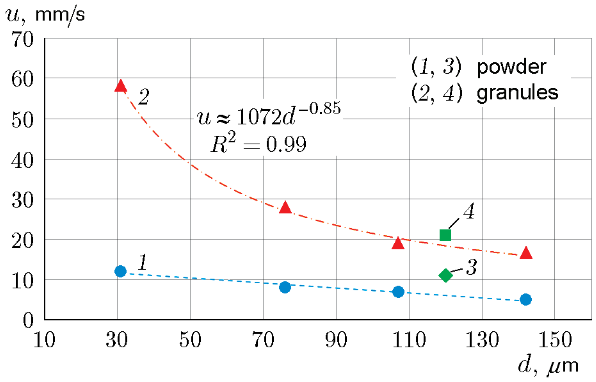

Fig. 3. Burning rate of the powder (*1* and *3*) and granular (*2* and *4*) Ti + C (soot) mixtures versus the particle size of titanium: the approximating curves *1* and *2* are plotted for the burning rate of narrow fractions of titanium, and points *3* and *4* refer to the velocity values for an initial wide fraction $d = 120$ μm.

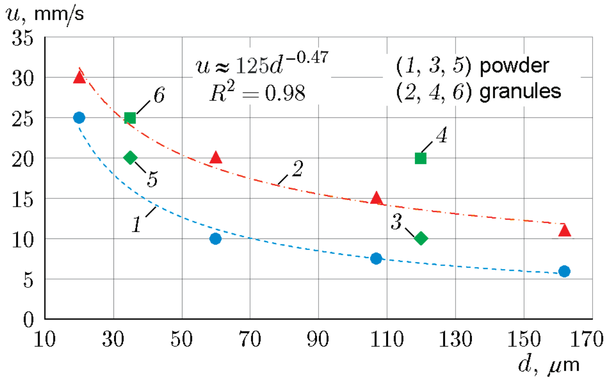

Fig. 4. Burning rate of the powder (*1*, *3*, and *5*) and granular (*2*, *4*, and *6*) Ti + C (graphite) mixtures versus the particle size of titanium: the approximating curves *1* and *2* are plotted for the burning rate of narrow fractions of titanium, points *3* and *4* refer to the velocity values for an initial fraction $d = 120$ μm, and points *5* and *6* refer to the velocity values for a fraction $d = 35$ μm.

warm up ahead of the combustion front when two conditions are simultaneously fulfilled. First, the characteristic particle size d should be smaller than the heating zone width $L = a_c/u$ (u is the experimental burning rate and a_c is the thermal diffusivity of the heterogeneous powder mixture):

$$d < L. \quad (1)$$

Second, the thermal relaxation time of the particle $t_h = d^2/4a$ (a is the thermal diffusivity of the particle substance) should be shorter than the characteristic residence time of the particle in the heating zone $t = L/u = a_c/u^2$ [1]:

$$t_h < t. \quad (2)$$

The following values of the thermophysical characteristics of the components are used in the calculations: $a_c = 10^{-6}$ m^2/s [13], a (Ti) $= 8 \cdot 10^{-6}$ m^2/s [14], a (graphite) $= 10^{-4}$ m^2/s [15], and a (soot) $= 2 \cdot 10^{-7}$ m^2/s [16].

Computational results for L, t, and t_h for the case of a powder mixture of titanium of different fractions with soot and graphite are given in Table 2. Data for the mixture with the initial titanium powder (wide fraction) is given in bold in the table. It can be seen that condition (1) is not fulfilled only for mixtures with titanium of the initial wide fraction $d = 120$ μm. This may indicate that using the characteristic size d, determined from the maximum of the size distribution of particles of a wide fraction, is incorrect, and it is confirmed by the experimental data and calculations below. Therefore, only data for narrow fractions are used for correctly comparing the dependences of the burning rates of granular and powder mixtures when constructing approximating curves (Figs. 3 and 4). Condition (2) is fulfilled for all mixtures (Table 2): the heating time of titanium particles of all fractions (t_h) is much shorter than their residence time in the heating zone of a combustion wave (t).

For carbon (soot and graphite), the heating condition (1) is fulfilled in all the mixtures studied. Indeed, in these mixtures, the particle sizes of soot (1–2 μm) and graphite (5 μm) are much smaller than the heating zone width L (Table 2). The thermal relaxation time of soot particles does not exceed $5 \cdot 10^{-6}$ s and that of graphite particles is $3.1 \cdot 10^{-5}$ s. It is shown by comparing these values of t_h with the computational results for time t, given in Table 2, that condition (2) for carbon is also always fulfilled. Thus, the necessary and sufficient conditions for the release of impurity gases from these components ahead of the reaction front are satisfied for all the powder mixtures. According to the CCCM, this reduces the combustion front propagation velocity.

It is known that the granulation of the SHS powder compositions contributes to eliminating the effect of impurity gases on the combustion process [7]. In such mixtures, the structure of the porous medium is artificially changed and the melt flow is limited by the size of individual granules. The combustion time of granular mixtures is determined both by the burnout time of individual granules and the heat transfer time from one granule to another, which depends on the contact area and the efficiency of conductive heat transfer between granules. As the granule size is much larger than the sizes of the initial components used in the experiment, the combustion of an individual granule can be considered similar to the combustion of a powder mixture. However, the characteristic size of granules is only 1 mm, so the conditions created in the granule for gas removal from the combustion zone are better than those in the powder filling. This reduces the IGR effect on the combustion of the granules themselves [7]. If there is no IGR in the heating zone of powder mixtures, granulation reduces the burning rate of the sample, which is associated with the presence of the stage of combustion transfer from one granule to another. For the studied compositions in which the IGR is present in the heating zone, granulation, on the contrary, should increase the burning rate, which is confirmed experimentally.

An increase in the burning rate upon transition from the Ti + C (soot) powder mixture to a granular one with initial titanium of a size $d = 120$ μm [the titanium heating condition (1) in the powder mixture is not satisfied] indicates noticeable IGR from the soot in the heating zone.

Aside from the IGR, the titanium–carbon interaction kinetics has a strong influence on the burning rate. The interaction rate of the components becomes the determining factor in granular mixtures because the effect of IGR in them is leveled. Therefore, an increase in the burning rate of the granular mixtures with a decrease in the Ti particle size results from the increasing interaction rate between the components. It follows from comparing the data for granular mixtures in Figs. 3 and 4 that the burning rates in the Ti + C (soot) mixture are noticeably higher than in the Ti + C (graphite) mixture. This indicates a higher interaction rate between titanium and soot.

The burning rate of the granular mixture is approximated by the dependence on the titanium particle sizes: $u \sim d^{-0.5}$ for Ti + C (graphite) and $u \sim d^{-0.9}$ for Ti + C (soot) (Figs. 3 and 4).

The resulting analytical dependences make it possible to use a different method for determining the characteristic particle size d of the initial titanium powders, which is used in calculating time t_h and in studying dependences $u(d)$. It is assumed that the experimental value of the burning rate of granular mixtures with titanium of wide fractions u should correspond to the approximation curve obtained using narrow fractions. The titanium particle sizes determined from the analytical dependence is denoted as d^*. It is revealed that, for the Ti + C (soot) granular mixture, $u = 21$ mm/s at $d = 120$ μm [$d^* = 102$ μm for a point of 21 mm/s on the approximation curve (Fig. 3)]. It is obtained that, for the Ti + C (graphite) granular mixture, $u = 25$ mm/s at $d = 35$ μm [$d^* = 32$ μm for a point of 25 mm/s on the analytical curve (Fig. 4)]. It is determined that, for the Ti + C (graphite) granular mixture, $u = 20$ mm/s at $d = 120$ μm [$d^* = 52$ μm for a point of 20 mm/s on the analytical curve (Fig. 4)]. Thus, it is shown on the example of Ti mixtures with carbon of various allotropic modifications that the dependence between a burning rate and a particle size can be determined correctly only by using narrow fractions of powders.

Approximating dependences (determination coefficient close to unity) are reliable if they are constructed using the experimental burning rates of narrow fractions of powders in a wide range of sizes.

The laws of the flame front propagation in a heterogeneous condensed medium are usually explained using a theory that is based on the conductive heat transfer mechanism in a combustion wave and that ignores the effects of melting and spreading of reagents [17]. The assumptions made in [17] in the formulation of the model are actually implemented in the combustion of granular mixtures. Indeed, in this case, the structure of the porous medium remains the same [8, 10–12], granules of similar size act as reaction cells, and the spreading of the melt is limited by the granule size. In addition, impurity gases have little effect on combustion, and heat transfer between granules is determined by conduction [18]. In accordance with [17], the dependence of the burning rate on the titanium particle sizes $u \sim d^{-0.5}$, observed for a Ti + C (graphite) mixture, corresponds to the linear law of interaction of the initial components, when the reaction rate does not depend on the thickness of the product layer formed. For the Ti + C (soot) granular mixture, the burning rate is described by dependence $u \sim d^{-0.9}$, which is close to the parabolic law $u \sim d^{-1}$ of interaction of components.

As for the analysis of the combustion of the Ti + C powder mixtures (Figs. 3 and 4), it is suggested by comparing the burning rates of granular mixtures (the burning rate of a titanium–soot mixture is noticeably higher than that of a titanium–graphite mixture) that a powder mixture of Ti with soot burns faster than a mixture of Ti with graphite in the absence of the decelerating effect of IGR. However, it is obvious from the experimental results shown in Figs. 3 and 4 that this is not the case. Thus, in a powder mixture, the heating zone pressure created by the IGR from graphite is lower than that created by soot.

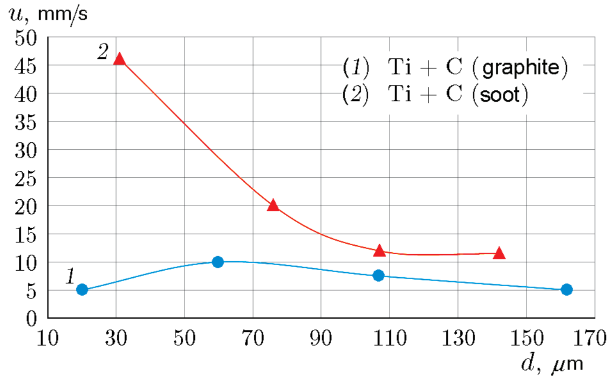

Fig. 5. Burning rates of the granular and powder mixtures versus the titanium particle size.

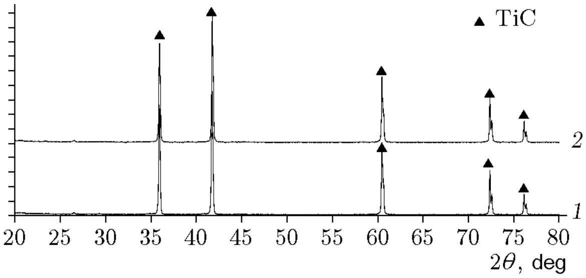

Fig. 6. X-ray diffraction analysis data for the combustion products of the powder (*1*) and granular (*2*) Ti + C (graphite) mixtures.

Figure 5 shows the dependences of the difference in the burning rates of granular and powder mixtures of titanium (narrow fractions) with soot and graphite. It can be seen that, as the titanium particle size drops, the difference in the burning rate increases in mixtures of titanium with soot. This means that, a decrease in the Ti particle size increases the amount of hydrogen released from titanium in the heating zone, which decelerates the combustion wave in powder mixtures. For a mixture with graphite, a decrease in the Ti particle size does not lead to a significant increase in the difference between the burning rates of powder and granular mixtures. As the same titanium powders are used in the experiments, it is only logical that this result is due to a decrease in the IGR from graphite ahead of the front, which compensates for the decelerating effect of the IGR from titanium. Indeed, the amount of gas released ahead of and behind the combustion front depends on the residence time of graphite in the heating zone, which, according to [1], is inversely proportional to the square of the burning rate: $t = a/u^2$. Therefore, the main gas release in fast-burning compositions, in which the degree of graphite gasification ahead of the melt layer is smaller, occurs behind the melt layer, thereby increasing the burning rate.

Despite the fundamental differences in the mechanism and patterns of combustion of powder and granular mixtures, the X-ray diffraction analysis suggests that the phase composition of the synthesis products is identical, independent of the titanium particle size, and represents a single phase of TiC. Figure 6 shows the X-ray diffraction patterns of the combustion products of the Ti + C (graphite) powder and granular charges. The data of X-ray diffraction analysis of the combustion products of the Ti + C mixture (soot) have the same form.

Consequently, different dependences of the burning rates of the Ti + C mixtures on the titanium particle sizes and on the allotropic form of carbon are not associated with different completeness of conversion of the initial reagents in the combustion wave, but are explained by the influence of the IGR and the change in the interaction rate of the initial components.

CONCLUSIONS

The dependence of the burning rate of the Ti + C granular mixtures on the particle size of titanium is determined for the first time for various allotropic modifications of carbon (graphite/soot).

An increase in the burning rate during the transition from powder to granular mixtures is explained from the standpoint of the convective–conductive combustion model. It is shown that, with the same-sized titanium particles, the burning rate of granular mixtures with soot is higher than that of mixtures with graphite, and the difference between the values increases with a decrease in the titanium particle size.

The approximation dependences of the burning rate of granular mixtures on the titanium particle size are obtained: $u \sim d^{-0.5}$ for Ti + C (graphite) and $u \sim d^{-0.9}$ for Ti + C (soot), which correspond to the linear and parabolic laws of interaction of the initial reagents according to the theory of the flame front propagation in a condensed heterogeneous medium.

It is established that different dependences of the burning rate on the sizes of components for various allotropic modifications of carbon in both powder and granular mixtures are not related to the difference in the phase composition of the condensed products and the completeness of conversion of the initial reagents.

It is shown that, as the titanium particle size decreases, the burning rate of the Ti + C (graphite) powder mixtures increases because of the action of multidirectional factors: a decrease in the impurity gas release from graphite in the heating zone of the combustion wave and an increase in the impurity gas release from titanium.

It is shown that the interaction kinetics between the components becomes the determining factor influencing the burning rate in granular mixtures in which the effect of impurity gas release is leveled.

It is revealed that, for the initial wide fractions of titanium, the characteristic particle size of the mixture should be determined using the data of the approximation dependences of the burning rate of this mixture on the particle size of the narrow fractions of titanium.

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