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The effect of degassing modes on the self-propagating reactions for the SiO_2–Al–C system in the presence of reaction promoters

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Abstract

The combustion synthesis process activated by organic polymers (Teflon and polystyrene) in the $SiO_2/Al/C$ system is investigated under the counter-current and co-current degassing conditions. It is found that the combustion parameters, i.e. wave velocity and temperature, and final product microstructure are influenced by the degassing mode considered as well as the type and amount of promoters added to the starting mixture. In particular, the combustion wave results to accelerate during its propagation when adopting the forward infiltration configuration and in absence of polystyrene. In addition, under the same degassing condition, the produced gases are found to provide a specific orientation on the growth direction of thread-like SiC crystals during final product formation.

Keywords: $SiO_2/Al/C$ system; Combustion synthesis; Reaction promoters; Degassing modes

1. Introduction

Combustion wave velocity represents one of the most important parameters which characterizes a self-propagating high-temperature synthesis (SHS) process of either a solid–solid or gas–solid type [1,2]. This parameter typically depends on several factors such as the chemical nature of initial reagents, the composition of the starting mixture, powder particles size, initial temperature, green density, etc. In addition, when gas–solid systems are considered, the permeability of the gaseous reactant which strongly affects its availability in the combustion zone also influences combustion front propagation [3].

Processes involving gas–solid systems are typically described in the literature using the well known filtration combustion theory [4,5], where the velocity of gas filtration is given by the Darcy law: $u = -K\frac{\partial P}{\partial x}$, K being the permeability coefficient, P the gas pressure, and x the coordinate of the direction of front propagation. In particular, when the process is not permeation-controlled, i.e. without filtration limitations, some analytical expressions for velocity of the combustion front were also recently developed in the literature [6].

It is worth mentioning that the combustion of the majority of the so-called gasless systems always involve a certain amount of gases, which may also affect process dynamics. This phenomenon is due to the gases adsorbed on the surface of initial powders, the low-volatile impurities present in the starting mixture and the gas-phase products of the oxide films typically covering the surface of chemical elements all of which are released during combustion [3]. Moreover, organic and inorganic additives used as promoters (hydrocarbonaceous and halogen-containing polymers, nitrogen-containing substances, hydrides of metals, etc.) to chemically stimulate the combustion reactions lead to the formation of gaseous species which influence combustion process evolution [7–13].

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It is worth noting that gas evolution during SHS processes has some practical advantages. For instance, it is known that the presence of gas typically gives rise to an increase in the brittleness of target product. Therefore, this aspect can be exploited in order to facilitate further grinding of the obtained product, when required. Furthermore, it has been shown [7] that replacement of a part of soot in Me-C (where Me is Ti, Zr, Nb, etc.) systems by polystyrene results in decreasing of carbide particles size.

However, it should be noted that gas evolution during combustion synthesis typically gives rise to an increase in pressure. This may cause a change in process dynamics and, more important, a decrease in the degree of reaction conversion, due for instance to the expulsion of the reactant mixture during synthesis. Therefore, the behavior of combustion synthesis systems involving gas evolution represents an important issue to be investigated in order to identify the optimal operating conditions in terms of product quality as well as process safety.

The influence of the mode of degassing, i.e. if the gas flows in the same (co-current flow) or in the opposite direction (counter-current flow) as that of combustion wave propagation, was recently investigated in the literature in the combustion synthesis of Ti–C and Ta–C systems [14]. It was found that the combustion process in the Ta–C system result was not affected by the direction of gas evolution. On the contrary, when the Ti–C system is considered, combustion wave velocity grew appreciably when passing from the countercurrent flow to the co-current configuration. The explanation given in the paper to justify such different behavior in the two systems is based on the fact that, while the Ta–C synthesis reaction proceeds through a "true" solid–solid mechanism the combustion temperature being lower than both reactants and product melting points, in the combustion synthesis of Ti–C, titanium melts before reacting with carbon. Therefore, the evolution of gases in the region beyond the combustion wave promotes spreading of titanium around carbon particles thus favouring the progress of combustion process. Of course, such effect is not possible if only solid phases are involved during combustion, as in the case of the Ta–C system.

Studies along this direction and related to the combustion synthesis in the Ti–C system when a portion of soot was replaced by powdered polystyrene were recently performed [9]. It was found that for the system $Ti+0.9C$ (soot)$+0.1C$ (polystyrene) the combustion velocity resulted significantly higher when gas filtration and combustion wave proceeded in the same direction relatively to the other configurations considered, i.e. counter-flow and bilateral. However, it was found that the increase of combustion velocity corresponds to a decrease of final product quality. In fact, the smallest degree of conversion was obtained in the forward filtration, being the largest one reached in the bilateral one.

In the present work, the effect of gas filtration during the combustion of the $3SiO_2+4Al+3C$ system, where a portion of carbon (as soot) is replaced by organic polymers (polytetrafluoroethylene and polystyrene), is investigated. In particular, the dynamics of combustion wave, i.e. front velocity and combustion temperature, and final products morphology obtained at different polystyrene content under the conditions of counter- and forward-filtration are examined and compared.

2. Experimental

Aluminum powder ASD-4 (St.-Petersburg, Russia) with particle size less than 10 μm, natural quartz sand (Rostov-on-Don, Russia) grinded up to particle size less than 15 μm (with 99% SiO_2 content), granulated carbon black P803 (VNIITU, Russia) with particle size less than 1 μm, powdered polytetrafluoroethylene or Teflon (FT-4) and polystyrene (PSE) with particle size less than 10 μm (Kuskovsk's Chemical Factory, Russia) were used in the experiments. The powders were first mixed in the desired stoichiometric ratios and then pressed manually into a metallic cylinder (3.0 cm in diameter and 4.0–4.5 cm in height).

As schematically shown in Fig. 1, the counter-flow (a) and forward-flow (b) configurations are taken into account in this work. The counter mode of gas filtration (cf. Fig. 1a) was ensured by initiating the reaction mixture in the open end of the cylinder. In fact, in this case the produced gases filtrate mainly through the final product, i.e. their transport occurs in the opposite direction of combustion wave propagation. On the other hand, for the co-flow mode of filtration the initiation was performed in the closed end of the cylinder, as illustrated in Fig. 1b. In this case, most of the degassing phenomena take place through the initial mixture, i.e. in the same direction of the combustion front. In both cases, the reaction was initiated from the upper end of the sample using $Ti+C$ burning mixture.

Tungsten-rhenium thermocouples 0.1 mm in diameter were places into four holes spaced at 1 cm intervals from the top to the bottom of the sample. Average values of combustion wave velocity and its variation along the height of the sample were calculated from signals of the four thermocouples. Combustion experiments were carried out in a constant pressure reactor under nitrogen medium at a pressure level of 0.5 MPa. Experiments control and the recording of thermocouple signals were performed using a personal computer.

The burning down samples were exposed to X-ray analysis on a DRON-3.0 diffractometer. The study of sample microstructure of the initial and final product was carried out using a BS-300 scanning electron microscope.

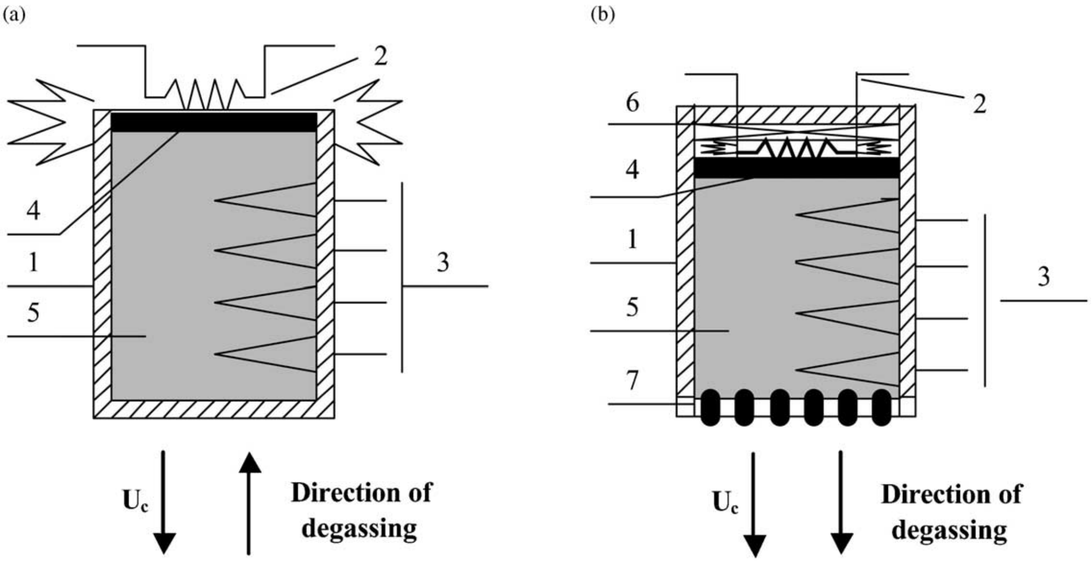


Fig. 1. Schematic representation of the two types, i.e. counter (a) and forward (b), of degassing configurations investigated: (1) metallic cylinder, (2) tungsten spiral, (3) thermocouples, (4) combustion front, (5) initial mixture, (6) high-temperature insulator, and (7) graphite grid.

3. Results and discussion

3.1. Influence of degassing modes on combustion parameters

The investigation of the effect of the degassing mode on the combustion temperature and wave velocity was carried out by assuming the following stoichiometry:

$$3SiO_2 + 4Al + (3 - x - y)C(soot) + xC(FT - 4) + yC(PSE) \rightarrow 3SiC + 2Al_2O_3 \quad (1)$$

where "x" and "y" represent the number of carbon moles added to the initial mixture as polytetrafluoroethylene and polystyrene, respectively. In particular, in this work the "x" value was kept constant ($x=0.26$) in all experiments while "y" was changed in the range 0÷1.1.

The dependencies of combustion parameters on polystyrene content in the starting mixture are illustrated in Fig. 2, where T_c^c, and U_c^c represent the combustion temperature and the average value of wave velocity for the condition of counter infiltration configuration, while T_c^f, and U_c^f are the same parameters when using the forward infiltration mode.

In the first case, where the gases produced during combustion filtrate mainly through the final product, it is seen that an increase of the amount of polystyrene added to the initial mixture leads to a decrease of the combustion velocity. Specifically, the value of U_c^c decreases from about 0.22 cm/s (at $y=0$) up to about 0.12 cm/s (at $y=1.1$), while the combustion temperature remains almost constant at about 2200 K, at least within the investigated interval of "y".

The U_c^c drop observed when increasing the polystyrene content may be related to the degassing conditions. In fact, under the counter-current flow, the gases initially allocated at the combustion front leave the burning sample moving in the opposite direction to that of the wave propagation. This leads to an increase of the heat transfer resistance between the hot portion of the pellet already reacted and the non reacted mixture. As a consequence, the front reaction decelerates during its propagation. On the other hand, the reason why the corresponding maximum combustion temperature remains approximately constant when varying polystyrene content may be due to the fact that the degree of conversion, and consequently the heat liberated during reaction, does not change significantly in the range of "y" considered.

As shown in Fig. 2, when the forward-flow configuration is taken into account, the dependence of the combustion parameters on the polystyrene contents resulted different from the case previously considered. In particular, the maximum temperature is observed to decrease from about 2200 to approximately 2000 K when increasing the amount of polystyrene from 0.135 to 1.1 mols. Correspondingly, wave propagation velocity decreases although with a much slower rate if compared to the counter-flow mode. The temperature behavior may be justified by the fact that in the forward-flow mode the gases formed are forced to filter through the unreacted mixture thus changing pellet compactness and, therefore, the contact among reactants particles. As a consequence, the conversion degree resulted decreasing when the amount of gas formed is augmented, i.e. if polystyrene content in the mixture is higher. However, the fact that the produced gas travels in the direction of the combustion wave, i.e. without

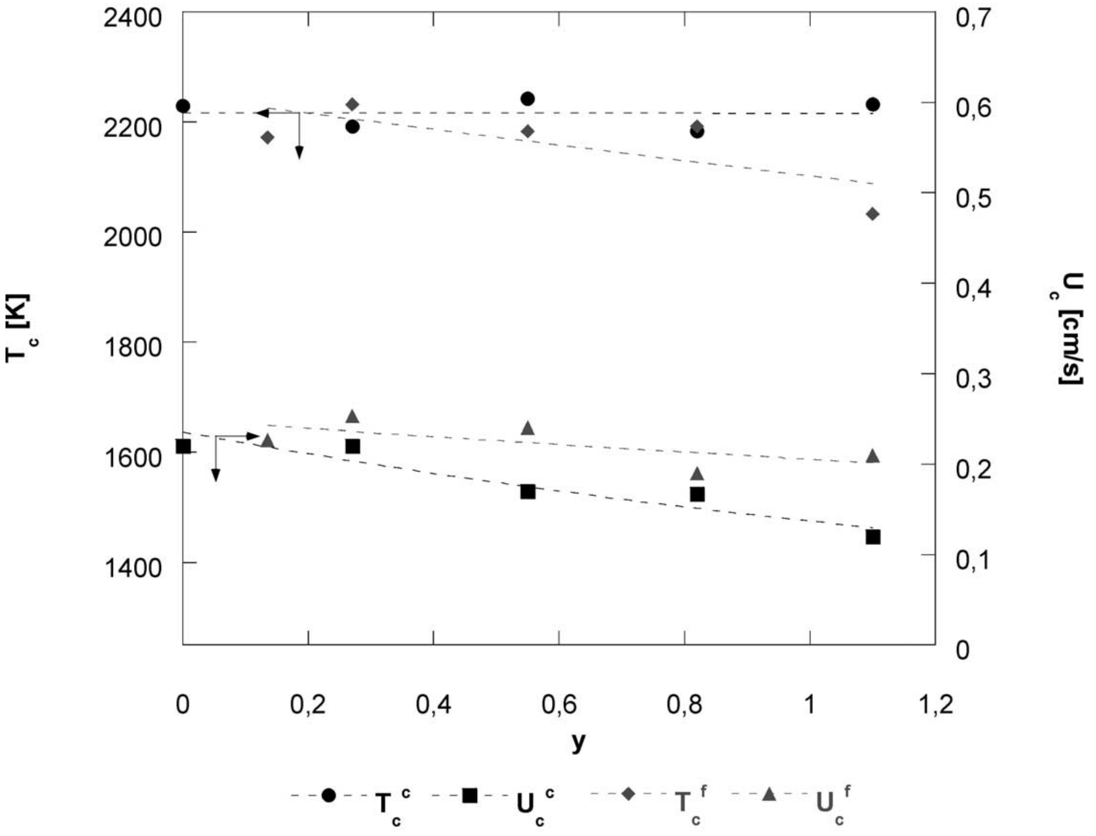


Fig. 2. Effect of polystyrene content in the starting mixture (y) on the combustion temperature and average wave velocity in the combustion of $3SiO_2+4Al+(3-x-y)C(soot)+xC(FT\text{-}4)+yC(PSE)$ system ($x=0.26$), under counter- (U_c^c, T_c^c) and forward-flow (U_c^f, T_c^f) conditions.

specific interaction with the latter one, makes the average value of reaction front velocity only slightly sensitive to the polystyrene addition.

3.2. *Effect of degassing modes on combustion wave dynamics*

Results related to the influence of the degassing mode on the dynamics of wave velocity in the combustion of the $3SiO_2+4Al+(3-x-y)C(soot)+xC(FT\text{-}4)+yC(PSE)$ system are shown in Figs. 3 and 4, for the counter and forward flows, respectively. Here, wave velocity values (U_c) at various distances (l) from the ignition sample end are reported. In particular, when the counter infiltration mode is considered, it is shown that in absence of polystyrene, i.e. $y=0$, the value of U_c^c is practically constant at about 0.22 cm/s (cf. curve 1 in Fig. 3) throughout the entire sample length.

On the contrary, when polystyrene is added to the initial mixture, in the first portion of the sample the combustion velocity still remains approximately constant at relatively low polystyrene content, i.e. $y=0.27$, or slightly increases when the amount of this polymer is augmented, i.e. $y=0.82$. However, in the second part of the pellet, the combustion front appears in both cases to decelerate significantly during its propagation. The possible justification of this behavior is discussed below.

As already mentioned in the previous section, when the counter-current configuration is taken into account, wave propagation is affected by the presence of gases generated by the polystyrene decomposition which gives rise to an additional resistance to heat transfer between the hot sample portion and the unreacted powder mixture. In addition, since the possibility of gas escaping becomes more difficult as the wave propagates through the sample due to the augmenting distance between the region where the gas is formed and the only location where it can escape, this may explain the decreasing character of front velocity vs. sample length. In particular, on the basis of the results reported in Fig. 3, this effect seems to be negligible in the first part of the sample where other factors, such as preheating, may play a major role, while it becomes predominant in the second portion of the sample.

A completely different behavior was observed under forward-flow conditions. In fact, as shown in Fig. 4, when the initial mixture is polystyrene free (i.e., $y=0$), wave velocity, whose initial value is about 0.3 cm/s, is found to sharply increase up to about 2.7 cm/s when the location of 1.6 cm from the ignition point is reached. It

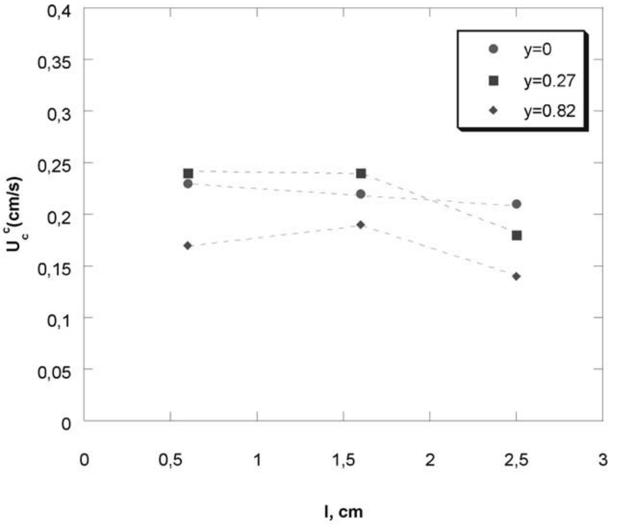


Fig. 3. Influence of polystyrene content on the dynamics of wave velocity, in the combustion of the $3SiO_2+4Al+(3-x-y)C(soot)+xC(FT\text{-}4)+yC(PSE)$ system ($x=0.26$) under counter-infiltration condition.

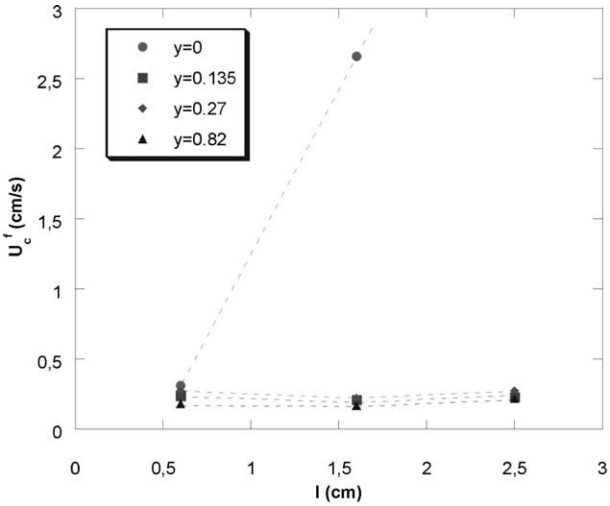


Fig. 4. Influence of polystyrene content on the dynamics of wave velocity, in the combustion of the $3SiO_2+4Al+(3-x-y)C(soot)+xC(FT\text{-}4)+yC(PSE)$ system ($x=0.26$) under forward-infiltration.

is worth noting that further measurements of combustion velocity at distances larger than about 2 cm from the ignition side of the sample resulted very difficult using the applied technique, since the signals obtained from the thermocouples were almost overlapped. However, although not reported in Fig. 4, a rough estimation of wave velocity from experimental observation gives about 10 cm/s at 2.5 cm from the ignition sample end. Thus, the front seems to accelerate during its propagation in exponential manner.

The system displayed a different behavior when introducing certain amounts of polystyrene in the initial mixture. In fact, under these conditions the stabilization of the combustion front propagation, i.e. front velocity resulted quite constant throughout the sample length, is observed as shown in Fig. 4 for the case of $y=0.135$, 0.27, and 0.82, respectively. In particular, it was found that the reaction front proceeds through the mixture with a velocity equal to about 0.23, 0.25, and 0.19 cm/s, when $y=0.135$, 0.27, and 0.82, respectively. It is worth noting that range of these values is rather close to the one observed when considering the counter-flow configuration (cf. Fig. 3).

On the basis of the results shown above, different combustion wave dynamics are obtained when the counter- and forward-flow configurations are compared. Specifically, if we consider the case of $y=0$, i.e. in absence of polystyrene, a constant wave velocity was found in the counter-flow mode, while the reaction front accelerates during its progress when considering the forward-flow one. These differences may be explained as follows.

Teflon decomposition occurring during reaction evolution causes a considerable change between the gas permeability of non reacted mixture (K_{mix}) and the one corresponding to the combustion product (K_{prod}), i.e., $K_{prod} >> K_{mix}$. If the counter-flow configuration is taken into account, the produced gases leave the burning sample moving through the reaction product, i.e., in a relatively easily manner because of the increased gas permeability. Therefore, the gases produced during the process freely leave through formed pores, so they do not influence significantly the combustion velocity dynamics.

On the contrary, when the forward-flow infiltration is considered, the produced gases can only leave the sample permeating through the green mixture where, due its relatively lower permeability value if compared to that one of the reaction products, they can be liberated with more difficulty as for the other configuration here taken into account. Consistently, the gases result to be mainly accumulated in front of the combustion wave region thus giving rise to an increase of the pressure level. The latter one leads to an improvement of capillary spreading of the liquid phase formed during process evolution, such as Al, AlF_3, Si, SiO_2, and, consequently, to the acceleration of front propagation along the sample.

The effect of the addition of polystyrene in the starting mixture on combustion process dynamics will be now discussed. As it will be confirmed later, it is possible to assume that the polystyrene introduced in the mixture decomposes before being reached by the combustion front. Consequently, the gases produced, which are mainly constituted by hydrogen [7,9], give rise to an augment of the porosity of the sample portion corresponding to reactants. Therefore, the gases subsequently obtained during the combustion process can escape

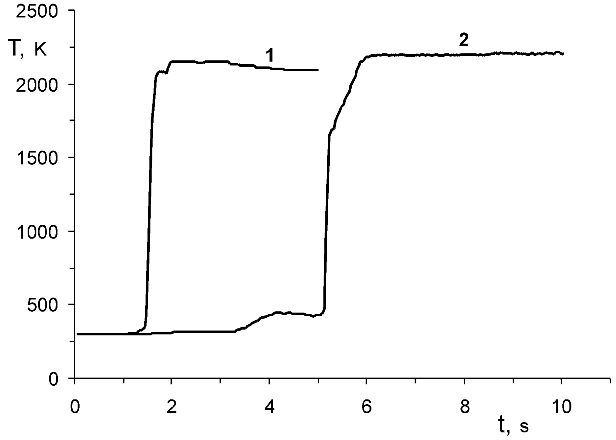


Fig. 5. Effect of the presence of polystyrene in the starting mixture on thermograms obtained during the combustion of the $3SiO_2+4Al+(3-x-y)C(soot)+xC(FT\text{-}4)+yC(PSE)$ system ($x=0.26$) under the condition of forward infiltration: 1–($y=0$); 2–($y=0.27$).

more easily through the formed pores, thus without practically influencing combustion wave dynamics.

The results discussed above are consistent with the thermograms shown in Fig. 5 which are related to the forward degassing configuration for the case of polystyrene-free mixture (cf. curve 1) or in the presence of this polymer (cf. curve 2). In fact, as shown in curve 1, the temperature does not change from the room value (about 300 K) until the combustion wave reaches the point of the sample where the thermocouple is embedded. On the contrary, the thermogram represented in curve 2 and related to the case when polystyrene was introduced in the mixture, shows a temperature change (from 300 to about 450 K) before the combustion stage takes place. Therefore, according to the discussion above, the temperature change revealed before combustion may be a consequence of the occurrence of the convective heat transfer caused by gas permeation through the initial mixture, which is enhanced because of the additional porosity produced by the polystyrene decomposition.

3.3. Combustion products morphology

According to reaction (1), the final products obtained for the chemically activated $3SiO_2+4Al+3C$ system are constituted only by Al_2O_3 and SiC (both the α and the β phases), as revealed by the XRD analysis results shown in a previously published paper [13]. It should be noted that this result was obtained either when the reaction was performed under argon atmosphere or at relatively low nitrogen pressure, i.e. $P_{N_2} < 1.5$ MPa, which is satisfied by the experimental conditions adopted in this work. Other phases, including nitrogen compounds, were also found at higher N_2 pressure [13]. This has been confirmed in the present work, where the phase composition of end-products was found not to be affected by the infiltration mode adopted.

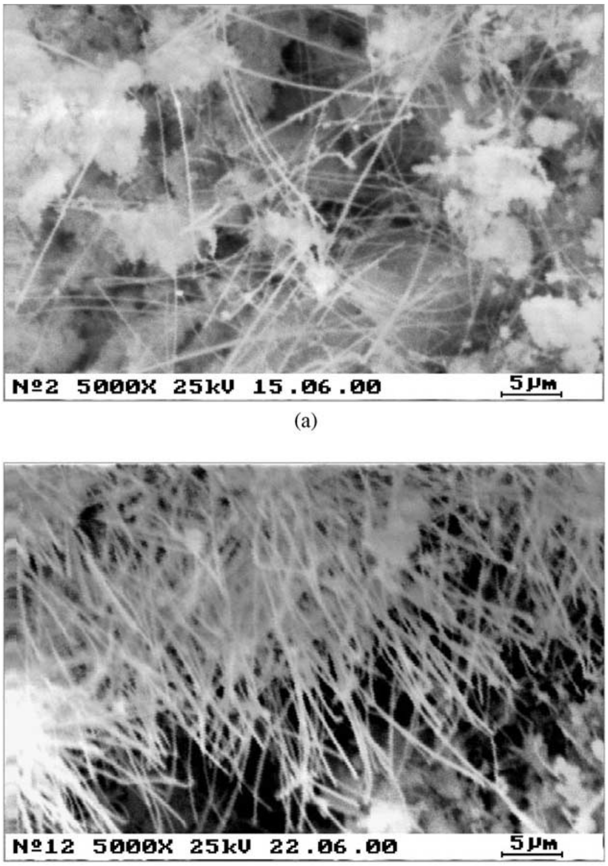
It is worth noting that the use of polymer additives in the combustion of the $3SiO_2+4Al+3C$ system produces not only the chemical activation of the reaction, but also leads to the formation of products characterized by peculiar microstructures. For instance, it was found that the obtained silicon carbide is in the form of thread-like crystals [13]. In addition, it was established that the gases deriving by organic polymer decomposition play an important role in the mechanism of end-product formation. Therefore, it is expected that the direction of gas infiltration may also influence the spatial distribution of these crystals and, thus, product morphology.

This aspect is confirmed by the experimental observations performed in this work, which indicate that characteristics of samples change depending on the infiltration mode considered. In particular, it was found that a non reacted outer layer is present in both cases. This may be related to the fact that the reacting mixture is in contact with the metallic cylinder, where heat losses from the lateral surface of the pellet are augmented.

However, the thickness of the non reacted layer is 1–2 mm smaller under the counter infiltration mode as compared to forward infiltration conditions. The central part of the sample obtained in the latter case is more easily separated from the non reacted outer layer. Note, that the samples burned down under the counter infiltration mode were more brittle. Moreover, the micropores and macropores formed, as well as the silicon carbide thread-like crystals, are more uniformly distributed throughout the sample when using the forward-flow mode.

An example of typical microstructure of samples combusted under the counter infiltration conditions is shown in Fig. 6a. It may be seen that, since gases leave the specimen through the final product, the SiC thread-like crystals are non-uniformly allocated in the sample, and, in addition, they do not show a specific orientation. Moreover, they are found mainly localized in free volumes of the sample, i.e. pores and cracks, as well as accumulated on the sample surface, where gaseous flows are more intensive.

A different product morphology is observed when considering the forward-flow mode as illustrated in the typical sample micrograph reported in Fig. 6b. In fact, the additional porosity generated in the reacting mixture by polystyrene decomposition, before the arrival of the combustion front, stimulates the direct formation of SiC thread-like crystals. In addition, such formation is also probably promoted by the leaving gases flows, which contain intermediate compounds mainly constituted by silicon fluorides and fluorocarbons.



(a)

(b)

Fig. 6. Distribution of SiC thread-like crystals in the final product obtained in the combustion of the $3SiO_2+4Al+(3-x-y)C(soot)+xC(FT\text{-}4)+yC(PSE)$ system ($x=0.26$ and $y=1.1$) under the conditions of counter (a) and forward (b) infiltration.

4. Concluding remarks

Although its relatively higher combustion temperature ($T_{ad}=2050$ °C), the reaction:

$$3SiO_2+4Al+3C \rightarrow 3SiC+2Al_2O_3$$

does not proceed under the self-propagating mode due to the presence of the oxide layer on aluminum particles surface which determine a barrier for diffusion processes. It has been recently demonstrated [10,13], that the reaction above can be activated by the addition to the starting mixture of a relatively small amount of powdered organic polymers. In particular, the mechanism of initiation of the reactive process proposed when using Teflon as a promoter is based on the destruction of the oxide film of aluminum by the molecular fluorine liberated by polymer decomposition [10]. In addition, it was found that combustion parameters as well as product microstructure depend on the amount of additive, specifically Teflon [10,13]. For instance, it was observed that wave velocity increases monotonically with Teflon content, while the combustion temperature displays a maximum. However, it is worth noting that in the previously cited investigations, the experiments were carried out using typical pellets so that there are no specific constrains to the degassing process of gases liberated during reaction evolution.

In this work, the influence of polystyrene content on the combustion parameters and product morphology in the combustion of the $3SiO_2+4Al+(3-x-y)C(soot)+xC(FT\text{-}4)+yC(PSE)$ system is investigated under the counter- and forward-infiltration modes.

Generally, it was observed that combustion synthesis parameters and products microstructure depend on the degassing mode considered as well as the amount of polystyrene added to the starting mixture. Moreover, it is postulated that this behavior may be related to the structural changes taking place in the reacting sample during process evolution and generated by polymer decomposition.

Firstly, it was found that the combustion temperature and average wave velocity results to be influenced by the degassing mode as well as the polystyrene content. Specifically, if the counter-infiltration mode is adopted, while the combustion temperature does not change significantly when increasing the polystyrene content in the starting mixture, front velocity decreases monotonically. On the contrary, when considering the forward-infiltration configuration, only slight changes were observed in the wave velocity, while the combustion temperature decreases with the amount of polystyrene. For the case of counter-current mode, gas evolution occurs in the opposite direction to that of the combustion wave, thus making the propagation of the latter one more difficult, i.e. causing a decrease of combustion front velocity when augmenting the polystyrene content. However, this does not lead to a change in the degree of conversion, and therefore the heat liberated during reaction as well as the maximum temperature does not vary significantly. On the contrary, when the co-flow configuration is adopted, the gas and the combustion front travel in the same direction so that they interfere much less, thus the front velocity does not change significantly when "y" varies. Correspondingly, the decrease of the combustion temperature occurs because of the pellet compactness changes provoked by the gas when moving across the unreacted mixture, before the latter one being interested by the combustion process.

The degassing mode adopted and the use of polystyrene was found to affect also combustion front dynamics. In fact, it is seen that when polystyrene-free systems ($y=0$) are considered, the combustion wave propagates with a constant velocity under the conditions of counter infiltration, while under forward infiltration a strong increase of the combustion wave velocity takes place during its progress. It is proposed that this is a consequence of the high pressure level arising in front of the combustion wave due to gas evolution which promotes the spreading of molten reactants and, therefore, combustion process propagation.

The addition of polystyrene to the initial mixture ($y > 0$) was found to produce a stabilization of combustion front propagation. It is proposed that this effect is connected with the formation of additional porosity caused by polystyrene decomposition which makes easier the degassing process through the reacting mixture.

It is observed that the infiltration modes also influence combustion products morphology. In particular, it is found that under the condition of forward-infiltration, the additional porosity formed by polystyrene decomposition promotes the directed growth of SiC thread-like crystals on certain sections of the sample.

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