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Chrono-topographic analysis of the fire focus dynamics in the SHS wave

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Abstract. The paper presents the means for measuring the spatial and temporal coherence of temperature in the micro regions of a wave of self-propagating high-temperature synthesis (SHS) and revealing the relationship between the events of ignition of individual foci. The method for determining the parameters of the thermal wave structure of the synthesis is based on the chronographical and topographical representation of micro thermal data in the form of maps that visualize the ergodicity of the SHS process and facilitate the recognition of individual foci of burning on thermal imaging images. The ergodicity of the phenomenon is used in the method to determine the time of induction of the combustion sites, the time of their growth, the growth rate of the foci tangentially to the front of the SHS wave, the size of the foci in the direction of the normal to the front. The error in measuring the velocity of the SHS front of the wave with the pro-posed technique was 0.05%.

1. Introduction

Self-propagating high-temperature synthesis of materials from powder mixtures in the mode of wave combustion is an exothermic process in which the patterns of product structure and phase formation are closely related to heat and mass transfer in a discrete medium and the chemical kinetics of reactions [1, 2]. In the synthesis wave, the products are heated to temperatures of 1200-4500 K, which makes it possible to investigate this phenomenon by means of optical measurements operating at effective wavelengths of 400-1000 nm [3-5]. High-speed thermal imaging of the SHS process through an optical microscope (micro thermal imaging survey) shows that the combustion wave consists of separate foci that flash through a quasi-regular time interval and have characteristic dimensions that depend on the parameters of the charge and the conditions of heat removal into the environment. Most modern means of observing the SHS phenomenon allow us to control only two parameters of the thermal wave of the reaction – the average velocity of its propagation and temperature [6]. This satisfies the needs of the fundamental mathematical models of the phenomenon, but it is not enough for its detailed analysis and identification of conformities in the thermal structure of the combustion wave. The present work was aimed at creating tools for measuring the spatial and temporal coherence of temperature in the micro regions of the process, and to identify the relationship between the events of their ignition.

2. Optical micro thermal imaging system

To observe the fast flowing process of the SHS wave propagation, an original micro thermal imaging system. It is made up of automated sample positioning, a high-speed digital monochrome camera



operating at 725 nm and an MBS-10 microscope providing spatial resolution at 2.4, 5.8, and 17.6 μm . The camera allows you to adjust the spatial resolution in the range from 10 μs to 500 ms in 2 μs increments (figure 1) [7].

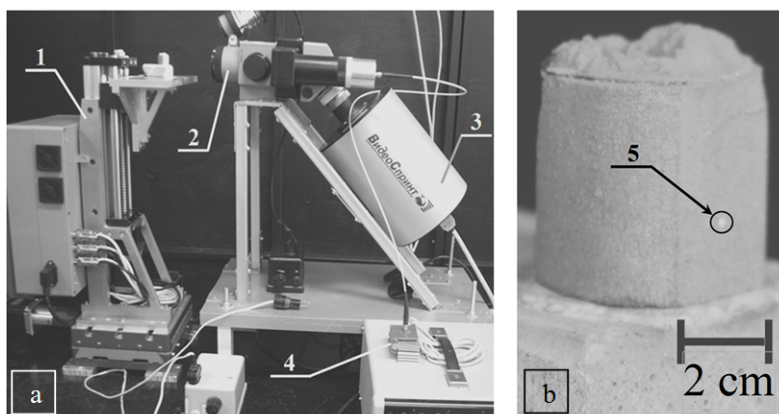


Figure 1. Micro thermal imaging measuring system (a) and experimental sample with spotlight (b) (1 – 3D positioning system, 2 – MBS-10 microscope, 3 – VideoSprint digital camera, 4 – aiming illuminator, 5 – spotlight spot on the experimental sample).

In the process of data registration, the parameters of the optoelectronic tract of the micro thermal imaging system are fixed. Therefore, the signal of the photocell depends only on the temperature of the observed object. Calibration of a digital thermal imaging system using a model temperature lamp TRU-1100-2350 made it possible to establish a correspondence between the measured signal levels and the reference temperature. This relational connection was used to determine the temperature of the foci of combustion in the SHS wave. As a result of calibration, the full range of temperatures measured by the complex was from 1200 to 4500 K with an error of 1.6 to 0.1%, respectively [8].

The original technique for analyzing the data of a micro-thermal imaging survey is based on the fact that different elementary volumes of charge are equal to the initial conditions for ignition of the mixture, and the physicochemical processes in them develop in the same way. Then the duration of heating of the substance from the conditional auto ignition temperature to an arbitrary level (in the temperature growth region) is the same for different elements of the sample surface. Thus, the temperature level measured at the heating site corresponds to a certain stage of the development of ignition (figure 2).

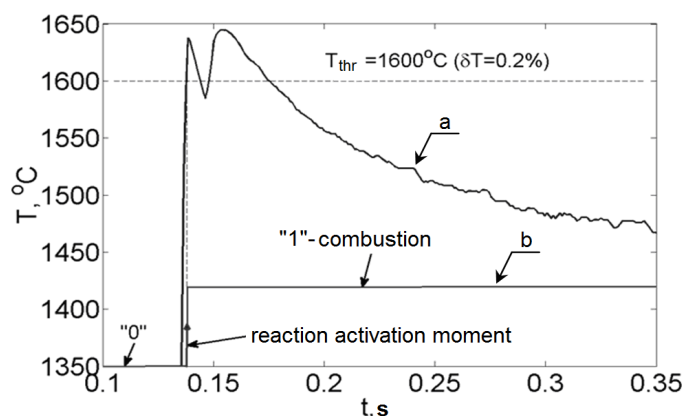


Figure 2. The thermogram of the SHS process (a) and the activation function of the reaction (b) at the point (x, y) on the sample surface.

Let us take as the local event the instant of time when the ignition process reaches a certain stage of development and the temperature of the elementary charge site becomes equal to the given level (figure 2b). A set of similar events, found from the data of a micro thermal imaging survey (figure 3) and presented in the space-time plane (figure 4), visualizes the magnitude of the shift of the combustion reaction front at different instants of time.

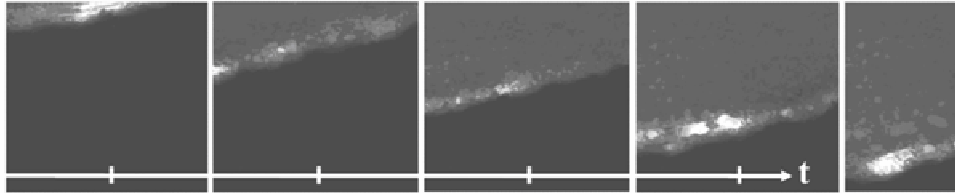


Figure 3. Micro thermal imaging of the SHS process with a spatial and temporal resolution of $5.8\ \mu\text{m}$ and $2\ \text{ms}$, respectively.

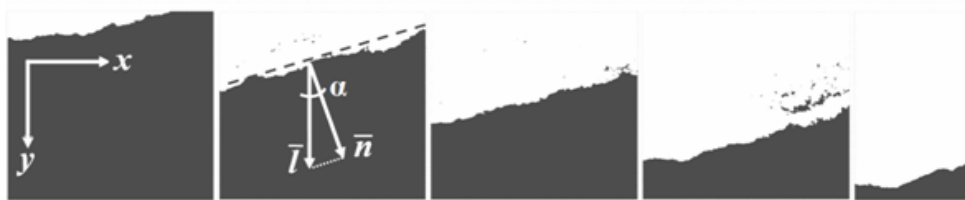


Figure 4. Event presentation of the SHS process.

Such a chronographic representation of thermal information allows us to divide in time and space the region of coherent combustion. The integral form of the chronogram of the combustion process reveals the ergodicity of the SHS process in the wave regime and allows to determine with accuracy up to 0.05% the propagation velocity of the reaction front (figure 5).

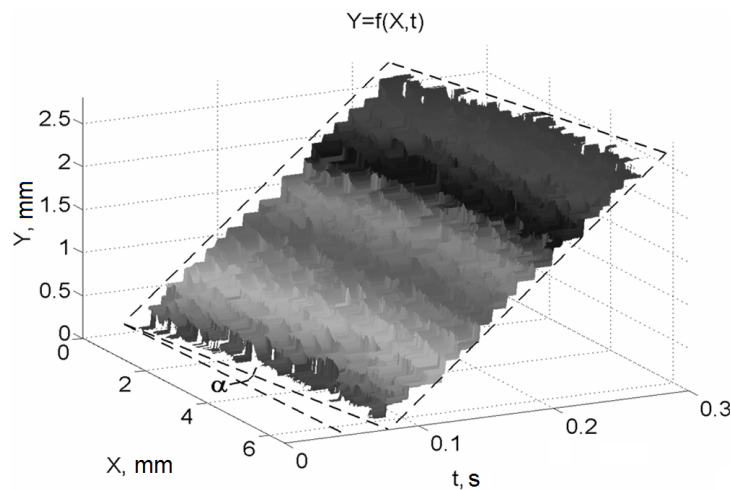


Figure 5. Visualization of ergodicity of SHS process.

On the basis of the differential format of the combustion chronogram, taking into account the ergodicity of the high-temperature synthesis, it is possible to determine such characteristics of the structure of the thermal wave of the reaction as the width of the front, the time of thermo chemical induction, and the time during which the growth of the dimensions of the focus is observed.

Chronographic analysis allows us to determine the spatial boundaries of individual foci in the combustion wave, construct a topographic map of the foci in the observation area, perform selective focal treatment of thermal imaging data, track the dynamics of temperature distribution in each source, and establish the direction and speed of foci development along the combustion wave front. The method of chrono-topographic analysis (HTA) of micro thermal data is implemented in the original software using modern image processing technology and parallel computing [9, 10].

3. Experimental results and discussion

Below are some examples of the application of the micro thermal imaging measuring system for the investigation of the kinetics of the SHS process and the dynamics of temperature and focal boundaries in the combustion wave.

3.1. Investigation of combustion kinetics in the Ni-Al-NiAl system

Approbation of the HTA technique was performed during experimental studies of the NiAl synthesis process in the wave combustion mode with variation of the proportion of the inert additive. The initial components were nickel powders PNA-UT3 with a dispersion of 15 μm and aluminum PA-4 with a dispersion of 50 μm . To carry out the experiment, three fractions of the inert additive NiAl were used, which were obtained in the preliminary stage by means of SHS, grinding on a spherical planetary activator and a screen analyzer (figure 6).

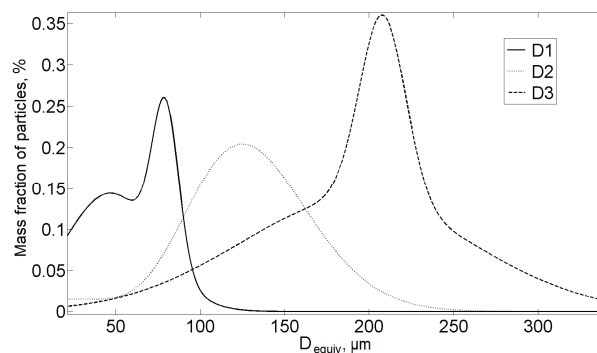


Figure 6. Granulometric composition of fractions of an inert additive.

The mass fraction of the inert additive of each fraction varied from 0 to 30% in increments of 2.5%. The apparent density of the charge was 2.6 g/cm³, and the initial temperature was maintained at 25°C. The SHS process was registered with a spatial resolution of 5.9 μm , and the frame rate was 500 Hz. As a result of the processing of micro thermal imaging data, the following dependences are determined: the propagation velocity of the combustion wave (V_n), the mean time of thermochemical induction between the ignition of the foci of the reaction (t_{ind}), the mean time for widening the focal boundaries (t_{exp}), the size of the foci in the direction of the normal to the front (L_n) by mass fraction of the inert additive (g) (figure 7).

3.2. Investigation of the kinetics and dynamics of foci of combustion in unstable modes of the SHS process

During the experiments, Ni₃Al was synthesized at an initial charge temperature of 25°C. The apparent density of the charge varied from 2.2 to 3 g/cm³. The studies were aimed at revealing in unstable burning regimes of qualitative and quantitative features of the SHS kinetics, the dynamics of the boundaries of individual foci, and the distribution of temperature in them.

Figure 8 presents a chronogram of the process by which it is possible to determine at what speed one of the combustion sites propagated at different instants of time, both in the direction normal to the front of the SHS wave, and parallel to it. According to the chronological map of the SHS process, it is

found that the development of foci along the wave front is similar to the propagation of the very front of the reaction - there is a stage of rapid displacement of the boundary of the fire focus with a sharp increase in temperature in it (flash stage), and the stage of inhibition, accompanied by a decrease in temperature. Moreover, the transition from the stage of inhibition to a flash causes the creation of a secondary fire focus, which propagates along the front of the SHS wave, but in the opposite direction with respect to the primary fire focus. The spread of secondary foci is unstable and ceases after a while. Figure 9 shows the dynamics of the temperature distribution of the combustion site in accordance with the chronogram of figure 8.

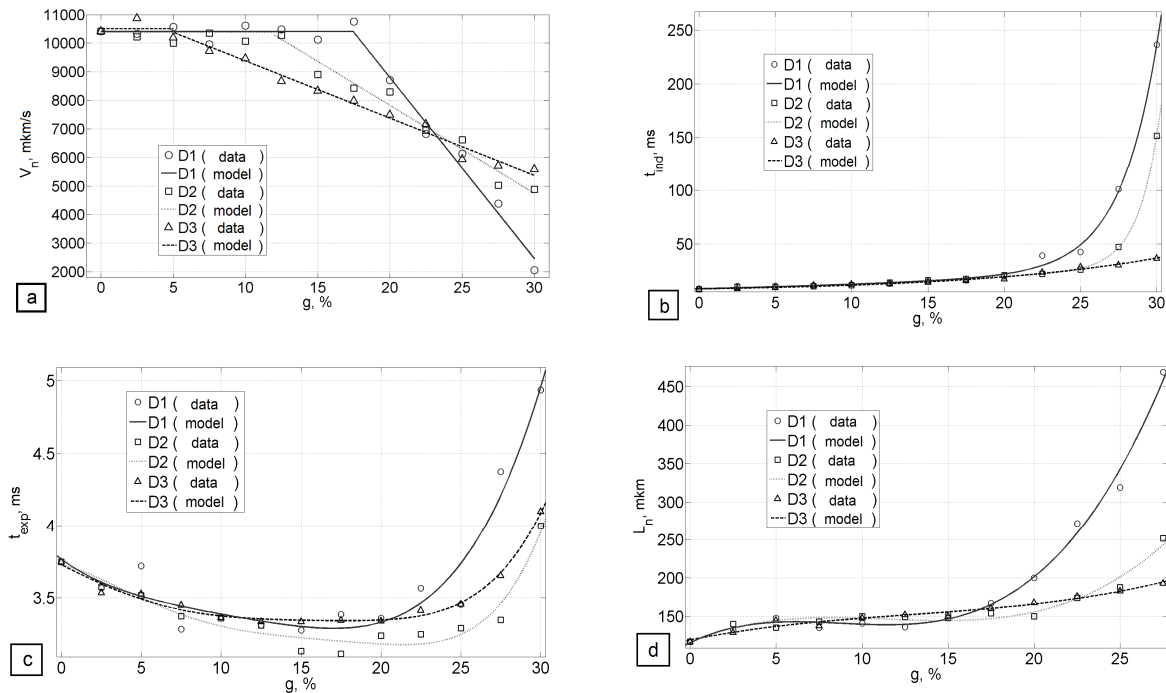


Figure 7. Families of the kinetic characteristics of the SHS process in the Ni-Al-NiAl system.

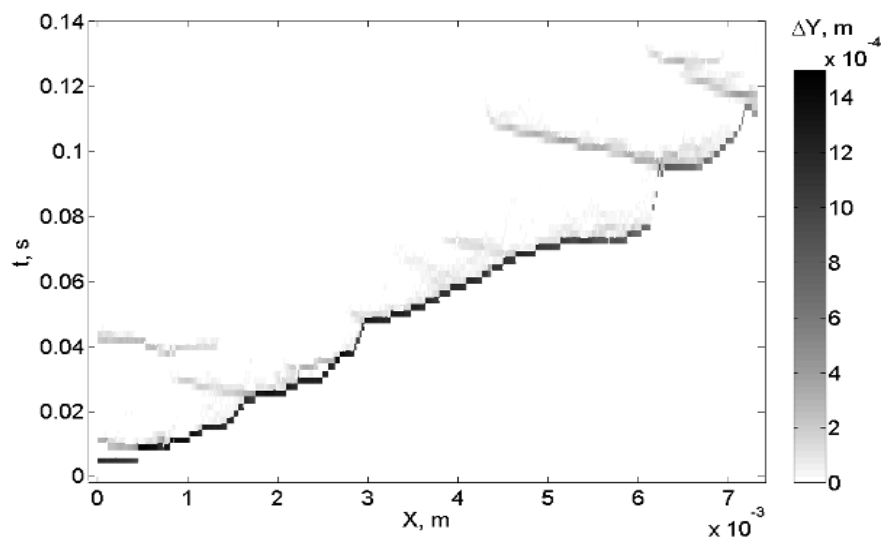


Figure 8. Differential chronogram of the combustion source in the SHS wave.

Experiments have shown that the dependence of the maximum temperature of the combustion sites on time is periodic. Comparison of the ignition time of foci located on the same normal to the wave front shows that the process of chemical transformation in unstable combustion regimes is rapidly "frozen" and re-triggered upon ignition of neighboring foci. An analysis of the entire set of foci of combustion presented on the topographic map of the SHS process shows the correlation of the renewal of combustion in the source with the ignition of at least 5 subsequent foci located in the direction of the normal to the wave front.

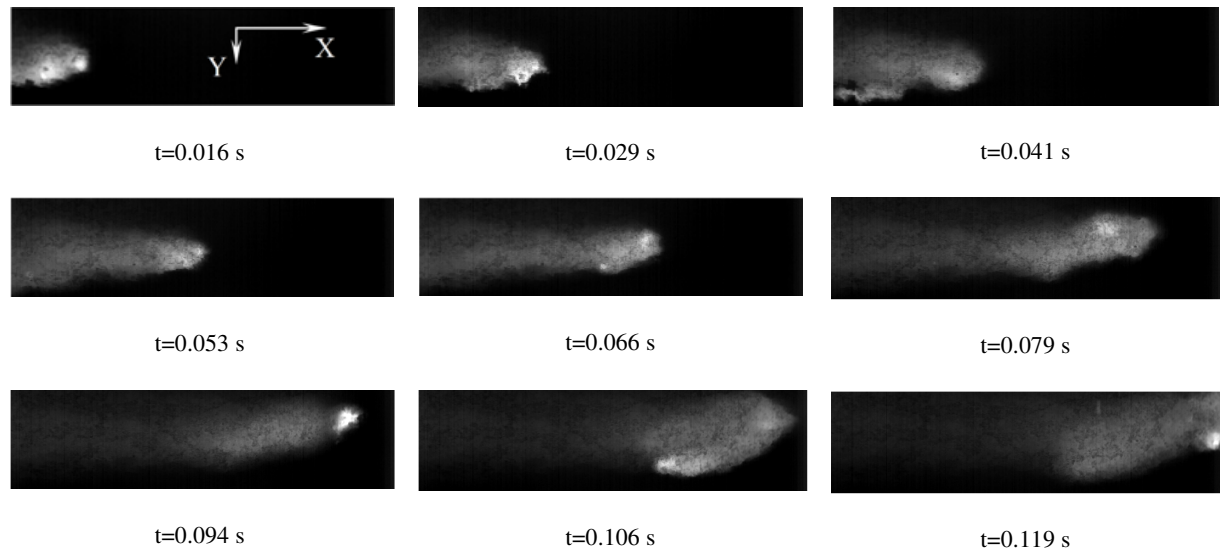


Figure 9. Dynamics of temperature distribution in the focus of combustion of the SHS wave.

4. Conclusion

The discreteness of the SHS process limits the accuracy of measuring the propagation velocity of the reaction wave. Overcoming this barrier can be achieved through the use of statistical indicators and a significant increase in the volume of information analyzed. The proposed chrono-topographical approach makes it possible to visualize the ergodicity of the SHS process. Conducting micro thermal imaging studies with modern measuring instruments within the framework of the year-old model reduces the confidence interval of statistical estimates up to 100 times and makes them sensitive to small variations in the parameters of the charge. In the technological aspect, this opens the possibility of controlling the quality of the initial mixture and predicting the actual properties of the product at the stage of its synthesis. Thus, the proposed method for determining the normal velocity of the SHS wave front allows one to control the process with a spatial and temporal resolution of up to 1 μm and up to 10 μs , respectively, and the measurement error reaches 0.05%. In this case, the chronographic and topographic maps of the SHS process contribute to the recognition of individual elements of the thermal structure of the SHS wave, their classification and characterization.

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