Trace-Analysis of Images of the Differential Chronogram of the Combustion Wave for Recognition of Transitional Modes of SHS

Pavel Gulyaev[1] and Vladimir Jordan[2]

[1] Ugra State University, Khanty-Mansiysk, Russia
P_Gulyaev@ugrasu.ru

[2] Altai State University, Barnaul, Russia
Jordan@phys.asu.ru

Abstract. In this article the method of visualization and recognition of the modes of propagation of the combustion wave in the additive technology of selective thermal sintering (SHS) for 3d printing of porous materials from cermets and oxides is considered. The effectiveness of compression of video recording by differential chronoscopy methods (DCS) and presentation of results in the form of 2d chronogram without loss of informative signs of the SHS combustion mode is shown. On the basis of Fourier and Trace transforms, an analysis of the SHS-chronogram in the Ni-Al system with the additive admixtures of an inert powder from 1 to 15% was carried out. The criteria for recognition of combustion modes based on physical invariants of the combustion wave motion are proposed (of spatial and temporal coherence). Using the example of the SHS of complex oxide bronzes, the possibility of the mode identifying of the stationary and relay-race combustion has been demonstrated. The results of the work are planned for implementation in 3D-printers control systems.

Keywords: Trace transform, SHS, additive manufacturing control, chronogram, combustion wave velocity.

1 Introduction

Self-propagating high-temperature synthesis (SHS) is the basis for the development of additive technologies for the production of 3D materials from cermets [1, 2]. For the formation of functional properties, inert additives in the form of low-melting salts [3] or compound oxides are introduced into the composition of the main exothermic mixture of Ni and Al powders [4].

The visualization of the SHS combustion wave and the control of the stability of technological process parameters are carried out using high-speed video cameras of the nanosecond resolution [5]. The synthesis temperature is measured by high-speed brightness micropyrometry methods [6, 7].

Theoretically, in accordance with the Hume-Rothery criterion, it is permissible to incorporate up to 15% of inert additives into the composition of the synthesis starting materials, which do not change the trajectory of the chemical combustion reaction and give the final product the necessary functional properties. In practice, the previously unknown phenomenon of discretization of the SHS combustion wave on the scales of microheterogeneity [8], which leads to a theoretically unpredictable change in the combustion modes (velocity and temperature) is discovered, as a result of which the phase and structural composition of the final products of synthesis is changed [9]. The solution to this problem is seen in the development of rapid methods for the recognition of critical and transient SHS modes by Fourier methods and Trace-analysis of video recording of the combustion wave propagation process, which have proved themselves in the analysis of fast physical processes in plasma powder metallurgy [10].

2 Experimental Technique

To study the unstable and transient modes of SHS, video recording of the propagation of the combustion wave in chemical reactions of a one-stage and two-stage synthesis was performed. In the first case, a combustion reaction was used in a Ni-Al system with an inert additive:

$$3Ni + Al + x(Ni_3Al) = Ni_3Al + Q\,(1-x), \tag{1}$$

where x is the mass fraction of the inert additive, Q is the thermal effect of the reaction. The transition to the unstable combustion mode was provided by an internal heat sink to an inert additive, the share of which increased from 5 to 30%. The two-stage reaction of synthesis of oxide bronzes includes the endothermic process of intercalation of Na into titanium dioxide TiO_2:

$$2CuO + Ti \rightarrow 2Cu + TiO_2 + Q \qquad \text{exothermic} \tag{2}$$

$$2TiO_2 + 2xNaI \rightarrow 2Na_xTiO_2 + x\,I_2 \quad \text{endothermic} \tag{3}$$

where x ($0{,}25 \leq x \leq 0{,}50$) is the stoichiometric coefficient of Na in the oxide bronze, which likewise determines the internal heat sink in the second stage of the endothermic reaction. As can be seen from equation (3), in this reaction the coefficient x determines the fraction of convective heat transfer through the gas phase of I_2, which considerably complicates the problem.

Fig.1 demonstrates serial frames of records of the solid state TiO_2 burning process, registered by Streak-camera "VS-NanoGate" (Videoscan Ltd, Russia). The main stages of the process can be described as follows.

The warming up of the lowest cold layer is presented in zone 1. Here we do not observe any chemical reactions. In zone 2 the rapid ignition and exothermic combustion reaction as local thermal explosion take place [11]. The effective thermal width X_T varies from 0.15 to 1 mm.

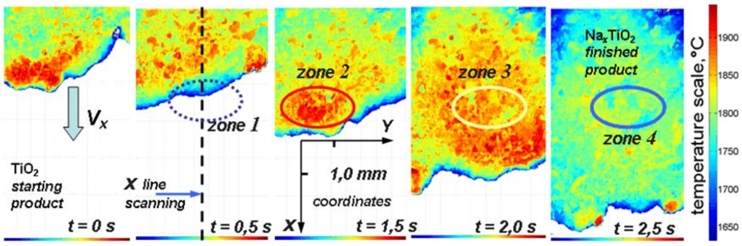

Fig. 1. Serial frames of high-speed video (1000 fps) of combustion wave TiO_2

However the necessary structural and phase changes of the crystal lattice still do not have time to occur. Zone 3 presents the disintegration process of high-temperature site to the small ones due to internal heat outflow. The new sites form the extensive "thermal cloud" where the temperature approaches the adiabatic value and promotes the intercalation of Na atoms into the octahedral lattice of TiO_2 crystals. The next stage is presented in zone 4. The endothermic process of the bronze Na_xTiO_2 formation decrease the temperature of the final product and the needed stoichiometric proportion established. The micropyrometry measurements are presented in Fig.2 as the 1D scanning thermal chronoscope along the dashed line of the heat monitoring photo-matrix. The thermal emission time in zone 2, the time of heat induction in zone 3 and the constant of the heat outflow time in zone 4 can be measured.

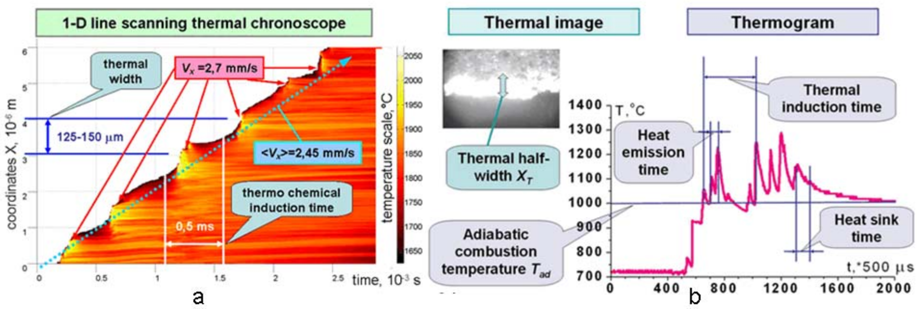

Fig. 2. SHT-synthesis of metal oxides: thermogram of the process of solid state burning

The method of technological control of the values is the preliminary mechanical activation and grinding of the initial metal oxide powders in the mill under the controllable energy stress loading of the mill, time of activation and the specific surface of the powder [1]. Thus, the high reproducibility of the synthesized metal oxide bronzes can be achieved.

3 Method of Differential Chronoscopy

The authors developed a method of differential spatial chronoscopy (DCS) of the SHS combustion front, which makes it possible to objectively control the stability of the SHS wave and the heat release modes in classical terms of the spatial and temporal coherence of the wave process. DCS-method of two-dimensional visualization of the video data stream binary with respect to a given brightness threshold, in which one of the spatial coordinates is replaced by a time coordinate, and the time derivative of the given coordinate plays the role of the brightness of the video data stream. The use of multi-exposure modes and the "global shutter" greatly expand the possibilities of studying the rapid combustion processes of SHS, due to the multiple recording of the image of a moving object with nanosecond delays between frames [7].

Earlier in Fig. 1 gives an example of micro-video recording of the 2D-field $T(x, y)$ of the temperatures of the combustion wave at time t, and in Fig. 2 and 3 show the principle of measuring the average velocity of the combustion wave $<Vx>$ along the selected row with the Y coordinate. High spatial resolution (1200x800 pxl), short multi-exposure time (30 ns) and interframe interval (Δt≤1 ms) provide a detailed analysis of the evolution of the fine thermal structure of the SHS combustion wave [5].

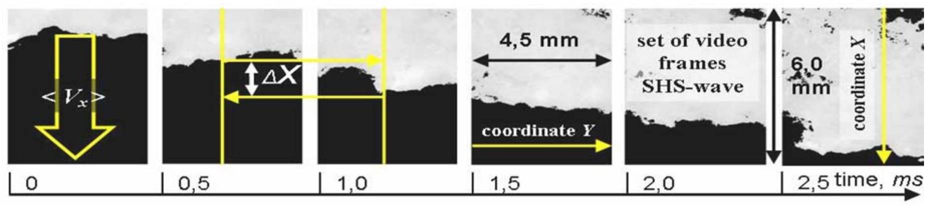

Fig. 3. The principle of the interframe difference for determining the velocity of the SHS front coordinates

The final result of the 2D-visualization of the heat transfer of the SHS wave for the two-stage combustion reaction (1) - (2) is shown in Fig. 4.

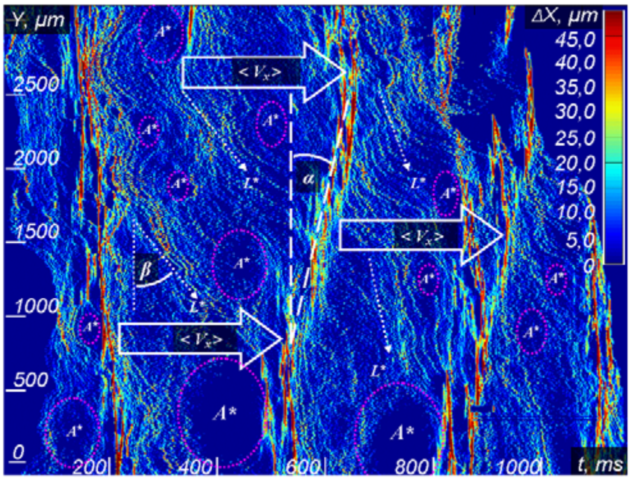

Fig. 4. The DCS-map for the heat transfer velocities $V(x, y) = \Delta X/\Delta t$ in the SHS wave

The result is obtained by calculating the interframe coordinate difference $\Delta X(t, y) = X(t, y)-X(t-\Delta t, y)$ of the wave front over the entire series of thermal imaging video frames. With the change of variables in equality $x=<Vx>t$, the chronogram becomes the velocity field $V(x,y)$. On the DCS-map of the combustion wave propagation of the two-step SHS reaction, three characteristic elements of the thermal structure are clearly visible: F_i* - discrete heat release front; L* - lines of the relay-race combustion; A_i* - area of diffusional alignment; $sin(\alpha)F_i$* и $sin(\beta)L$* - irregularity of coherence of the wave (time and space of localization). The observed structure of the wave can be explained by the competition between heat-mass transfer mechanisms and the nonlinear character of the change in the coefficients of thermal conductivity or diffusion.

Maps of the propagation of the single-stage reaction wave (1) in the Ni-Al system, shown in Fig. 5 for different values x of the dilution factor by an inert additive, were obtained in a similar manner.

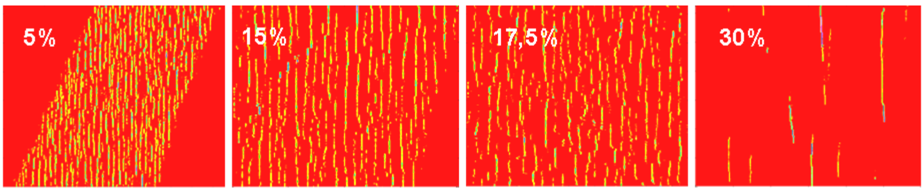

Fig. 5. DCS-map of the combustion wave in the Ni-Al system with a change in the proportion of the inert additive

4 Methods of Chronogram Analysis

Two-dimensional maps DCS of the combustion waves $NaxTiO_2$ and Ni_3Al were analyzed by Fourier methods (Fig. 6) and Trace transforms (Fig. 7).

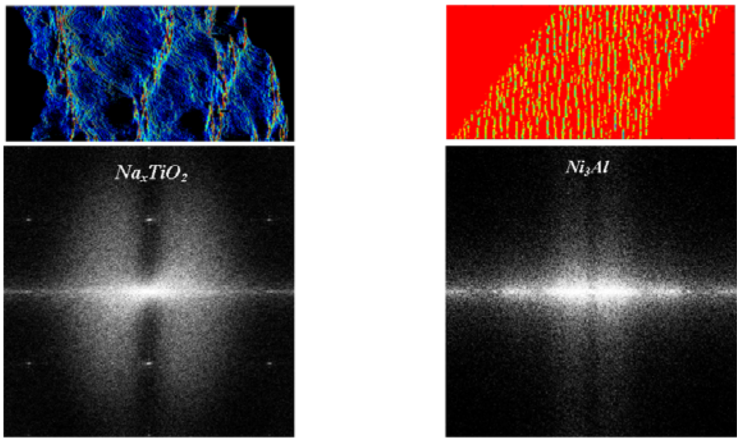

Fig. 6. The Fourier spectra of the combustion wave in a two-stage and one-stage SHS reaction

A comparison of the spatial-frequency Fourier spectra is shown in Fig. 6.

Trace transform was used to analyze the most complex case of combustion in a two-stage SHS reaction with the gas transmission mechanism of convective heat transfer. In this case, from the differential chronogram of the combustion wave shown in Fig. 4, four rectangular zones were identified: zone SHS.01: from 0.5 to 0.9 s; zone SHS.02: from 0.9 to 1.2 s; zone SHS.03: from 1.2 to 1.5 s; zone SHS.04: from 1.5 to 1.8 s. Trace-images are obtained for the Radon type conversion (T1) and P. Finsler's metric (T3), which are clearly different and are shown in Fig. 7.

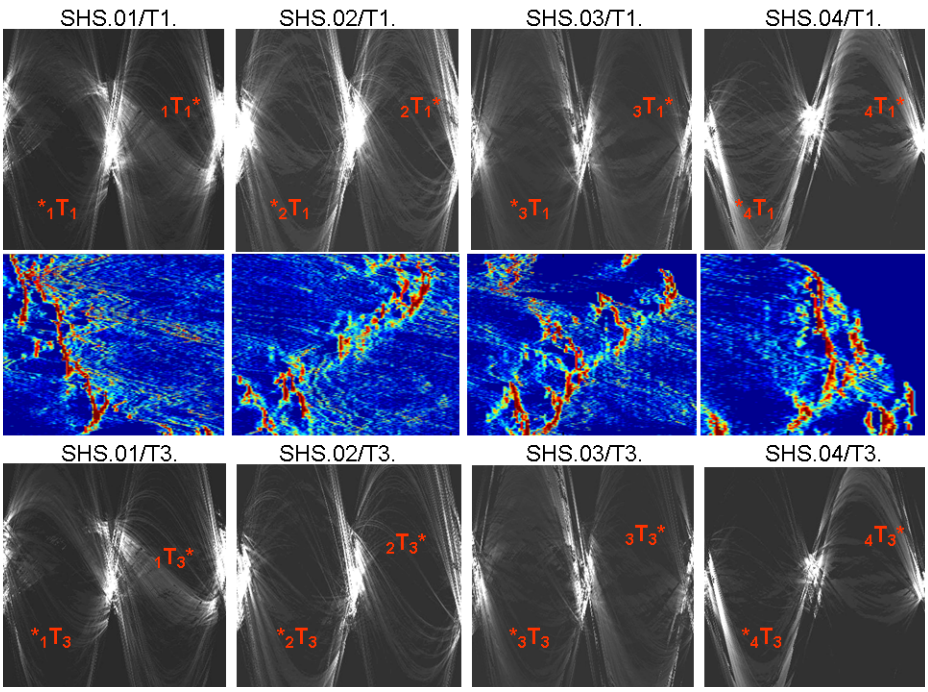

Fig. 7. Trace-images: zone SHS.01 – coherent combustion; zone SHS.02 – relay-race combustion; zone SHS.03 – thermal explosion; zone SHS.04 – unstable decay of combustion wave

5 Results and discussion

A comparison of the results obtained in Fig.4 and Fig.5 allows us to notice a significant difference between the DCS-maps of the combustion wave of a one-stage and a two-stage SHS reactions. This is further supported by the results of Fourier analysis in Fig. 6. As can be seen in Fig. 5, the only indication of the transition mode of combustion of a single-stage reaction is an increase in the time interval of thermochemical induction. Thus, for the single-stage SHS reactions, the usual Fourier analysis is quite sufficient, which allows one to determine the limiting fraction of the additives introduced into the composition at according to the changing the frequency of occurrence of local combustion sites [12].

Trace-transforms, as can be seen from Fig. 7, very well determine the transition from the stationary (coherent) combustion regime to others, but further it is required to select the selectively sensitive Trace-T parameters and the type of Trace transform core.

6 Conclusions

1. 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.

2. The proposed chronographic approach allows us to compactly visualize the propagation of SHS in the wave combustion mode and to carry out the ergodicity analysis by Fourier methods and trace-transformations.

3. The revealed differences of the thermal explosion from the coherent and relay-race propagation of the wave make it possible to create a database on the Trace-transform invariants corresponding to the 3-fold combustion modes in the SHS process.

7 References

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