A NEW HIGH-SPEED BRIGHTNESS PYROMETRY METHOD TO INVESTIGATE SELF-PROPAGATING HIGH-TEMPERATURE SYNTHESIS

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A new method of high-speed brightness pyrometry is presented which allows self-propagating high-temperature synthesis to be studied with a time resolution on the order of 1 μsec. Test of the method showed that the high time resolution can reveal previously undocumented details of the fine structure of combustion waves.

Self-propagating high-temperature synthesis is a method to obtain desired products during an exothermal reaction (or usually a combination of reactions) which propagates as a wave or in bulk through a heterogeneous condensed system of dispersed components [1]. The complex physicochemical nature of this synthesis has required development of a set of experimental and theoretical methods to study the reaction conditions and to establish laws and mechanisms for the formation of various classes of products. Historically, study of self-propagating high-temperature synthesis has been dominated by a phenomenological approach which emphasized the set of thermokinetic parameters which are sufficient for a reaction to occur based on the construction of formal combustion models.

However, interaction laws in real dispersed systems do not follow classical homogeneous kinetics, because both the heterogeneity of the material and the high melt temperatures of the starting and reaction products prevent premixing and a subsequent homogeneous reaction. The reagents must diffuse into each other for a reaction to occur. Also, reactions in powder mixtures do not produce heat uniformly over the whole reaction space, but locally at the contact surfaces [2].

The following phase-formation mechanisms occur in self-propagating high-temperature synthesis [3, 4]:

1) A new phase layer forms on the surface separating the reaction components.
2) A product layer grows within one of the components.
3) The product layer is dissolved and the melt-solution of the reaction zone and is saturated with one of the components.
4) The nucleus of a new phase forms on a fragment of the crystalline structure of one of the reagents within the melt.
5) Ordered compounds form from metal-chemical reactions during crystallization.

The heat of reaction must be favorable for any of these mechanisms to be realized. Thus, information relating the thermal structure of the combustion waves to phase formation is one of the most important sources for establishing a means to control the phase composition of the end product and to optimize its properties.

Research methods which can observe thermal transformation dynamics and can record the phases and products the moment they form, i.e., on a real time scale, are severely limited for reactions in heterogeneous condensed systems [5, 6]. Thermocouple methods [7], which have been widely for recording temperature profiles in combustion zones are not completely adequate for the requirements of experimental combustion physics, mainly because these methods have poor time resolution which exceeds characteristic elementary reaction times.

Today the electron microscope is one of the most informative methods for investigating processes which model self-propagating high-temperature synthesis [5]. However, even this method has its disadvantages, which make it impossible to transfer the results to real systems, because it does not give information on the characteristic times and temperatures at which transformations occur.
Synchrotron radiation [6] provides important information on the features of component reactions in combustion waves and on the dynamics of phase formation. This method has been used in experimental research on rapid, high-temperature processes, including gasless combustion of condensed systems [6] in combination with the formation dynamics of product phases; however, it cannot relate the phase dynamics to the temperature intervals at which transformations occur. Therefore, in order to create an exhaustive picture of the reaction, it is useful to augment this method with a method fast enough to record the temperature profile of the combustion wave.

**EXPERIMENTAL METHOD**

In order to develop a rapid method for recording the temperature and rate characteristics of self-propagating high-temperature synthesis reactions, the Department of Applied Optoelectronics of the Institute of Semiconductor Physics, Siberian Branch, Russian Academy of Sciences, along with the Applied Scientific Research Laboratory of Self-Propagating High-Temperature Synthesis Materials of the Altai State Technical University, developed and produced the "LISTIK" device for monitoring the temperature and rate characteristics of self-propagating high-temperature synthesis processes (Fig. 1, 1).

The device is based on a two-barrel optical CCD camera 4, which contains two transducers 3 rigidly fastened some distance apart along the path of the wave front. During the test stage, we investigated the possibility of using various solid-state photosensors for the transducers: LFD-1A avalanche photodiodes (100 × 100 μm sensing element), four-quadrant silicon photodiodes (4 × 4 mm), and FD-3A discrete photodiodes (2 × 2 mm). The spatial resolution of the device at the hot sample (60 μm to 5 mm) depended on the spectral filtering of the object image and on the dimensions of the replaceable diaphragms (slit and circular), which were mounted at the surface of the photodetector and on the optical focusing system. We used ÉOP-66 and ÉOP-72 telescopic pyrometer systems and a UM-2 dispersion monochrometer system for filtering. The spatial resolution was determined from the magnitude of the video signal as a 100-μm wide horizontal slit diaphragm was displaced vertically in the object plane by means of a micrometer; the time resolution was specified by a 2-MHz quartz oscillator and a KR580V153 programmable timer. The radiant flux in the plane of the photosensor was monitored through the telescope of an ÉOP-66 optical pyrometer by a calibrated small-format TV camera which used a MF-14 MDP photodiode matrix. The measured radiation flux varied from 0.015 to 1.0 mW/cm² at a wavelength of 0.63 μm.

The photosensors were compared and the equipment spread functions were determined in order to select the simplest camera design: FD-3A silicon photodiodes and Zenith-3E mirror optics with a PZF telescopic adapter and a Jupiter-37A objective. The attainable spatial resolution (on the order of 100 μm) at the hot sample was limited by practical considerations; further enhancement of the resolution would not significantly change the form of the video signal recording the combustion front, while diffraction distortion would increase. The resolution is sufficient for evaluating the integral characteristics of the temperature and rate parameters.

The sensitivity of the silicon photosensor had a marked nonlinear dependence on the wavelength, with a maximum in the range of 0.8-0.95 μm. This maximum was one of the reasons for using selective light filters in the optical system of the camera. The photodiodes, equipped with 100 μm masks to minimize possible distortions introduced by the optical system, transform the radiation from the combustion wave, which is formed in the self-propagating high-temperature