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ANALYSIS ON A PROMISING RESEARCH TOOL FOR CHEMICAL PHYSICS AND PHYSICAL CHEMISTRY

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ABSTRACT.

A hardware-software measuring infrared thermography-based complex was developed, allowing semi-automatic experimental exploration of sorption dynamics and low-temperature catalytic processes during the interaction of various gases and their mixes with solid-state highly dispersed materials. The complex includes an optical-mechanical component as well as a WiFi-controlled automated gas supply system. Using the new-generation infrared thermography methods, the latent kinetics of phase transformations on the surface of highly dispersed samples with catalytic and non-catalytic properties has been experimentally investigated. The quantitative characteristics of fine-grained materials have been experimentally studied. Using the described methodology, a number of interesting phenomena taking place on the surface of highly dispersed media were experimentally revealed.

Keywords: Infrared Thermography, Thermal Imaging, Adsorption, Desorption, Catalysis

1. INTRODUCTION

The study of the basic mechanisms and main regularities of gaseous molecules interaction with a solid surface of media containing micro- and nano-sized inclusions, pores, and other heterogeneities is of considerable scientific interest. The physical and chemical properties of such surfaces are substantially influenced by adsorption-desorption and chemisorption processes, including catalytic reactions. The fundamental physicochemical surface properties of highly dispersed (powdered) materials are still little studied in this field of knowledge and are subject to the comprehensive research, which, in turn, makes the progress and development of new experimental research methods relevant.

It is known that adsorption and desorption are accompanied by heat release and absorption, respectively. These thermal processes are usually analyzed by calorimetric and some other methods, which inherent feature is a rather long duration of the measuring procedure. Similar measurement methods are also used in the analysis of chemical reactions in heterogeneous catalysis. As shown in [1], [2], [3], a highly promising approach in this field is the use of modern infrared thermography (IRT) [4] that allows studying the fast kinetics of thermal processes accompanying gas molecules interaction with a solid.

The rapid development of focal plane array (FPA)-based infrared cameras production technology that ensured wide availability of these devices for scientific research facilitates, in particular, the experimental studies of exothermic and endothermic processes occurring on solid surfaces [5]. The ability of IRT to extract in real time from the objects under investigation the thermodynamic data in the form of two-dimensional temperature maps allows you to quantitatively analyze the effects reflecting the reaction spatial heterogeneity and kinetics. It was used, in particular, to observe a thermal adsorption-desorption waves propagation along with an extended adsorbent layer [3]. The IRT method is endowed by wide observability that is successfully used for the quantitative analysis of sample libraries. It allows the thermal processes occurring in many cells simultaneously to be compared by studying them synchronously in the same experimental conditions [6]. In [7], [8], when studying sorption processes on a solid surface, the thermal imaging method was combined with

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the ellipsometrymethod.

The use of thermal imaging for controlling various catalytic reactions is described in [1], [9]. Itshould be noted that catalytic and non-catalytic reactions are usually studied using thermal imaging at a sufficiently high temperature of the chemical process reaching 100-200 □C and higher [10], [11]. In the present work, the IRT-based investigations of sorption and catalytic processes were carried out at room temperature. Under such conditions, the thermal effects associated with the physical adsorption and desorption of gas molecules make a significant contribution to the heating and cooling of solid-state samples and are well recorded by an infrared camera.

2. SUBJECT AND METHODS

The new-generation FPA-based IRT is the only experimental tool today that allows a quantitative and precise study of the rapid evolution of temperature transformations accompanying the sorption-catalytic processes without direct contact with the object of interest. It makes it easier to reveal the real origin of these processes and understand the physical and chemical nature of the latter. The estimates made in [4] showed that the sensitivity of the order of 0.01 □C anda frame rate of about 100 frames per second inherent to the new-generation FPA-based infrared camerasallow us to register temperature effects at the level of surface concentrations equal to only a fraction of a monolayer. The use of this contactless, remote, fast- acting and highly sensitive quantitative experimental methodology, making it possible for taking a measurement from the outside of the environment in which the process itself develops, is a distinctive feature of the research presented in this paper.

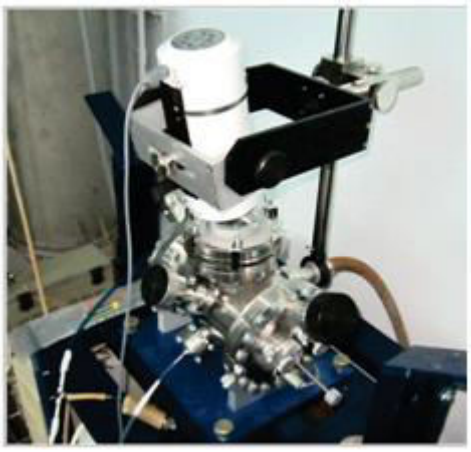

Fig. 1. The optical-mechanical part of the hardware-software measuring thermal imaging complex.

In the experiments, a TKVr-IFP/SVIT thermal imager with a cooled detector based on InAs semiconductor was used. This infrared camera allows recording, in the spectral range of 2.5-
3.05 □m with the temperature sensitivity of about 0.03 □C, and storing in the computer memory the continuous thermo-films with an acquisition rate of 100 thermograms per second. For protecting the reaction zone from the external environment, as well as for the calibrated supply of gas reagents into this zone, etc., we created a semi-automated hardware-software thermal imaging-based complex that allowed, in particular, studying the sorption and catalytic processes developed on highly dispersed material surfaces.

A photograph of the optical-mechanical assembly of this complex is shown in Fig. 1. The support, on which the infrared camera is mounted, is precisely adjusted to the reaction chamber and allows a reproducible return of the thermal imager to the initial position after the reaction chamber reload or any other manipulation. The experimental samples, or a four (or more)- sample library, were placed into the chamber through the loading gate.

In addition to the thermal imager, the complex includes a Wi-Fi-controlled electronic system providing a possibility for a

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gas mixture of a given composition to be supplied into the reaction chamber [12]. The thermal imaging data and gas stream protocols are stored synchronously in the memory of the respective computers.

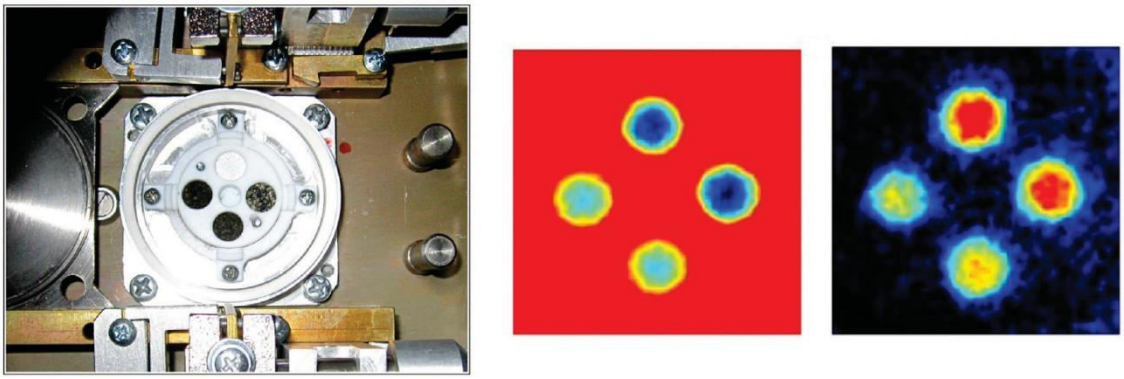

Fig. 2. The library (left) and its two IRT thermograms (right) of highly dispersed samples of aluminosilicatescontaining different percentages of cobalt and cerium. The library is measured in desorption (left) and adsorption (right) modes, respectively. Despite different colors displayed in a visible-light diapason, these samples, in the infrared spectral range, are identical in their emissivity (this fact is verified experimentally).

3. RESULTS

A photograph of one of the numerous libraries of highly dispersed samples examined by us is shown in Fig. 2 (left). In the right part of the same figure, shown are the thermograms of this library measured using IRT in the atmospheric-air water vapor desorption (left) and adsorption (right) modes. It is clearly seen that the thermal imaging method allows one to successfully register the difference in the development of thermal processes in the structures of this kind.

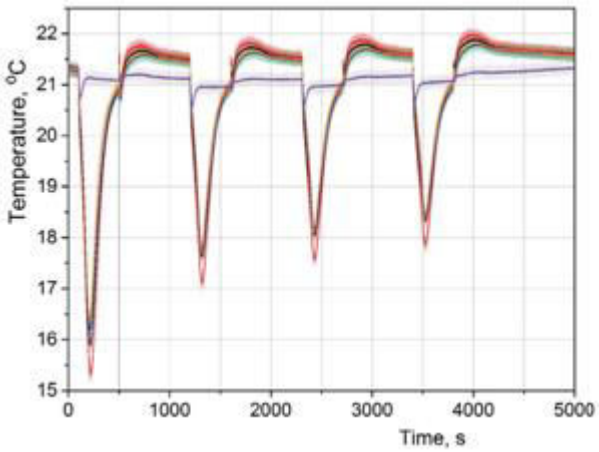

Fig. 3. Degradation of the desorption temperature dip inthe repetitive "atmospheric air inflow–pumping out" cycle realized with the sample library shown in Fig. 2.

The curve that the most weakly reacts to the "gas inflow – pumping out" cycles, is given for comparison and corresponds to the element of the surrounding background (rubber sealant) with no relation to the testsamples.

The quantitative characteristic measured in the course of the cyclical repetition of the same process of gas supply into the chamber and pumping out of it is shown in Fig. 3. It is rather difficult to obtain the presented result using other research methods, but it may be easily revealed using IRT.

4. DISCUSSION

As an interesting phenomenon, that is clearly seen in Fig. 3, is a quantitative degradation of the desorption cooling of the investigated structures subjected to the regime of repetitive "gas inflow – pumping out" cycles. In addition to the above-mentioned effect, itmay be clearly determined from Fig. 3 that the investigated samples exhibit different activities with respect

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to both adsorption anddesorption. Namely, the temperature curves are reliably separated in the same conditionsof the gas environment.

5. CONCLUSIONS

Up-to-date infrared thermography is a promising research tool in chemical physics and physical chemistry. With its help, it is possible to extract new information about the sorption and catalytic properties of a solid surfacesubjected to a gas molecules attack.

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