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AN EXPERIMENTAL-COMPUTATIONAL SYSTEM FOR THE DETERMINATION OF THERMAL PROPERTIES OF MATERIALS. I. EQUIPMENT, INSTRUMENTATION AND METHODICAL SUPPORT OF THERMAL TESTING

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Abstract
An experimental-computational system is presented for investigating the thermophysical properties of composite materials by methods of inverse heat transfer problems and which is developed at the Thermal Laboratory of Department of Space Systems Engineering, Moscow Aviation Institute (MAI). The system is aimed at investigating the materials in conditions of unsteady contact and/or radiation heating over a wide range of temperature changes and heating rates in a vacuum, air and inert gas medium. The paper considers the hardware components of the system, including the experiment facility and the automated system of control, measurement, data acquisition and processing, as well as the aspects of methodical support of thermal tests.

1.INTRODUCTION

Over many years when developing new engineering prototypes the attention was concentrated on the composite materials of the structural application. At present interest is shown on composites of other functional applications, such as acoustic, optical, electromagnetic, heat engineering. Modern materials of heat engineering applications are widely used in different structures operating in conditions of extreme thermal loads, such as, for example, in pressurised spacecrafts, thermal and nuclear power technology, ferrous and nonferrous metallurgy, chemical industry, engine manufacturing, in building material production, etc.

Of great interest in solving heat engineering problems are: high-density composite materials such as carbon-carbon, carbon-ceramic, carbon-glass; high-temperature ceramic and metal-ceramics; glass-, asbestos- and carbon-fiber plastics; high-porosity heat-insulating materials based on ultra thin fibers and microspheres; light-weight foam carbon materials; flexible woven and non-woven materials based on thermostable fibers; as well as different thermoprotective and heat-insulating coatings based on the above-said materials.

The development and application of these materials is impossible if there is no positive information about their thermophysical properties. At present to define the thermophysical properties of materials, especially those of a complex structure, a methodology of inverse heat transfer problems (IHTP) is effectively used [1,2]. This approach has a high information content, allowing observed data of the characteristics of the material's specimen thermal state to define its thermophysical properties with regard to real unsteady and nonlinear effects of heat transfer processes in conditions approaching those of full-scale. So, a successful implementation of IHTP methodology is possible only by a rational combination of physical simulation of heat transfer processes in the specimen, exact measurements of initial thermal parameters and correct mathematical processing of experimental data based on the solution of the IHTP.

As an effective way to solving the given problem is to design a problem-oriented scientific research system interconnecting the specially developed experimental equipment, computer-aided research system and the methodical support of the experiment based on IHTP. In addition, the actual task of IHTP methodology implementation in scientific and engineer practice of investigations of the thermophysical properties of materials is solved. The key principles of developing such systems were considered in [3].

The experimental-computational system described in the present paper is the result of further development of investigations carried out in MAI and connected with the elaboration of the computer-aided research system of the thermal processes based on IHTP methodology [3,4]. The system is designed for laboratory and semi full-scale thermal testing in order to determine and investigate thermophysical, radiation-optical and physical-chemical properties of composite materials and members of structures designed on their basis. Testing can be performed in conditions of unsteady contact and/or radiation heating at a rate up to100°C/s in the range of temperatures from a room temperature to 1600 °C in air or in the inert gas medium at pressure up to 1.6 bar or in a vacuum as low as 2.5×10^{-8} bar.

In addition, the system permits the following investigations to be conducted: selection testing for perspective thermal materials (flexible and multilayer, inclusive); design and development of new experimental-computational methods for identification of unsteady heat transfer processes in materials and structures; design and development of highly accurate thermal sensors and instrumentation.

The experimental-computational system

The hardware equipment system includes: a high-temperature thermovacuum stand TVS-1M; special experimental modules (EM) for realization of preset heat transfer models in the testing of specimens; an automated system (AS) of heating control, experimental information measurement, collection and processing based on PC and PXI module system (National Instruments Co.); hardware of local network providing interconnection of the system with distant workbenches of research-engineers and Internet; as well as special technological equipment for the preparation of experimental specimens, the manufacture of the thermosensors and their installation in polymerizing and high-porous composite materials.

Methodical support of experimental investigations include: the methods for preparation and conduction of thermal testing and experiment design [2]; an applied software for processing and interpretating experimental data based on the IHTP solution [1,5]; a special software of the information-measurement subsystem (IMS) based on integrated software LabVIEW of the National Instruments Co. A configuration of the system is shown in Figure 1. The main technical data and parameters of the system are given in Table 1.

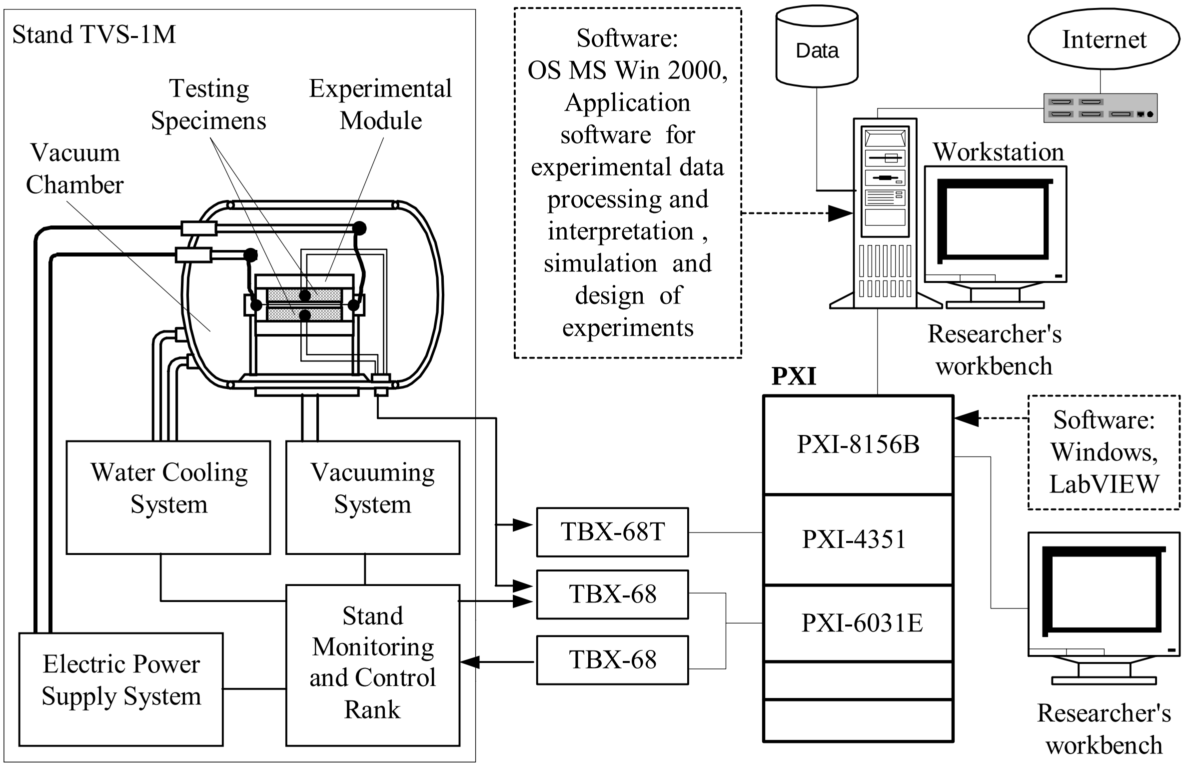

Figure 1. Experimental/computational system configuration.

The main components of the system are the following:

Thermovacuum stand TVS-1M

The experimental system has been developed based on a high-temperature thermovacuum stand TVS-1M. This stand is an essentially modernized version of thermovacuum stand TVS-1 developed in MAI [1]. The general view of TVS-1M stand is shown in Figure 2. The stand includes a horizontal water-cooled vacuum chamber and the following systems: vacuuming, water cooling, electric power, control and monitoring. Figure 3 illustrates a photo and Figure 4 – a drawing of the vacuum chamber with the installed experimental module.

Case 3, covers 1, 6 and the workbench, 5, of the chamber are water-cooler. On the back fixed cover, 1, there are pressurized inputs of water-cooled current terminals, 2, of the electric power supply system of the experimental module heating element. On the case there are sealed connectors of cables of the control and measurement subsystem, as well as a mounting flange of the chamber vacuum gate. An easy-to-open front cover, 6, helps the installation on the chamber workbench of the experimental module, 4. A vacuuming system includes roughing-down and diffusion oil-vapor pumps, vacuum gate of the chamber, vacuum mainlines, valves and instrumentations. A water-cooling system provides the given thermal conditions of the chamber, diffusion pump and current terminals of the electric power system.

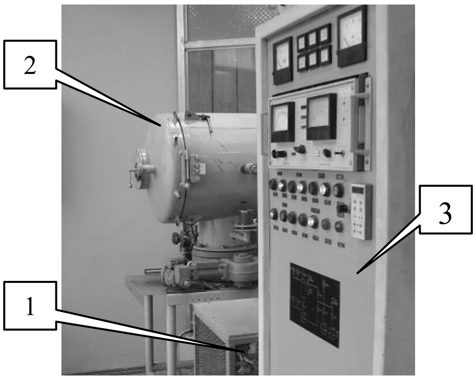

Figure 2. General view of TVS-1M stand:
1 – power transformer; 2 – vacuum chamber;
3 –monitoring and control rack.

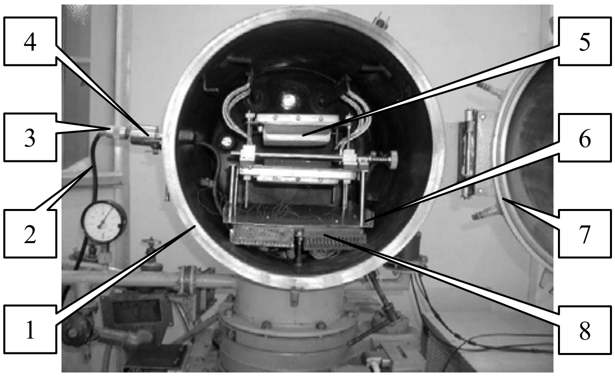

Figure 3. Vacuum chamber of TVS-1M stand:
1 – vacuum chamber; 2 – communication cable with AS;
3 – connector; 4 – seal lead; 5 –experimental module;
6 – chamber's workbench; 7 – cover of vacuum chamber;
8 – contact plate.

The electric power supply system of the heating element includes a 40 KW step-down power transformer (current in the heating element circuit up to 3800 A), a thyristor regulator and instrumentations.
The control of heating is performed from AS by a specified program. As a feedback during the procedure there is an indication of the thermocouple mounted on the heating element or on the specimen's heated up face. The control devices and instrumentations of the stand are arranged in the monitoring and control rack.

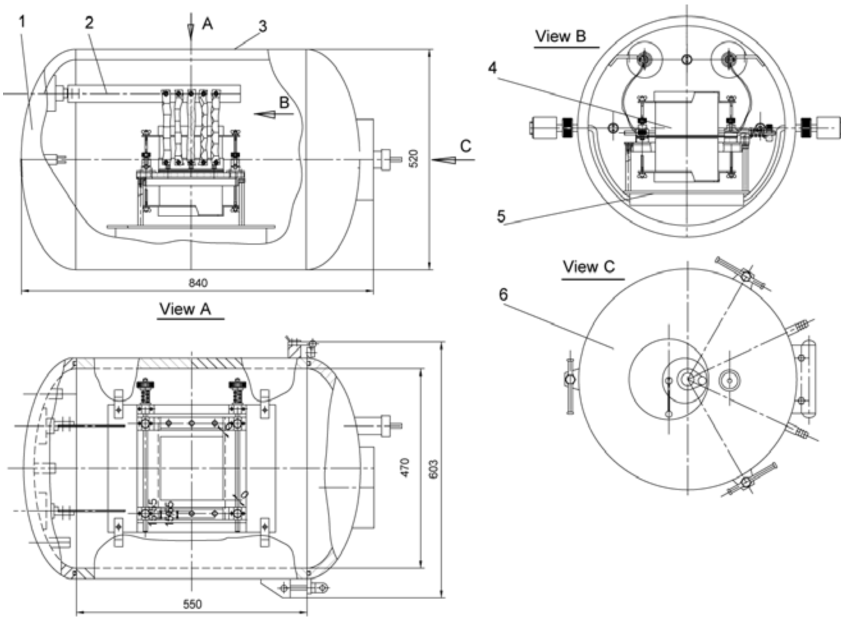

Figure 4. An assembly drawing of the vacuum chamber with the experimental module: 1 – chamber's back cover; 2 – current lead; 3 – case; 4 – experimental module; 5 – chamber's workbench; 6 – chamber's front cover.

The experimental module EM-2 and its modification

The experimental module is among the main assemblies of the experimental system. Module EM-2 is designed for: arranging the specimens of the materials under investigation in the stand's vacuum chamber; to provide the given thermal conditions for specimens during thermal testing (for realization of the given heat transfer model) including a heating by the program and the sustaining of a thermal condition at the specimen's boundaries. Figure 5 gives a drawing of the EM-2 module. In the base of 1 there is the attachment and tension point of the flat heating element (HE) 6. The assembly consists of fixed, 2, and movable, 7, hold-down straps of the heating element, of two guide bushes, of two guide rods, 3, springs and adjusting nuts. The hold-down strap, 2, and guide bushes are attached to the base through the electric insulating plates made of thermoresistant material. The hold-down strap, 7, is also electrically insulated from the base. It is fixed on guide rods by lockscrews. It can move by the action of springs relative to the strap, 2, providing constant tension of the heating element fixed by straps. A tension stress is controlled by special adjusting nuts in the range of 0.1 to 20 kg. This allows reduced deformation of the heating element at heating. Two suspension points, 4 (the upper and the lower) are for fixing the specimens of material, 5, arranged in the heat-insulating mandrels. Each such point consists of a setting frame, which is attached to the locating pins. The pins are attached to hold-down straps, 2 and 7. A structure of the suspension points helps to compensate for the translation of the moving strap under the action of springs providing accurate latching of specimens and gap adjustment between the faces of specimens and the heating element in the range of 0 to 10 mm. Flexible current inputs of the module are fixed by screws to the hold-down straps and current terminals of the vacuum chamber, see Fig. 4.

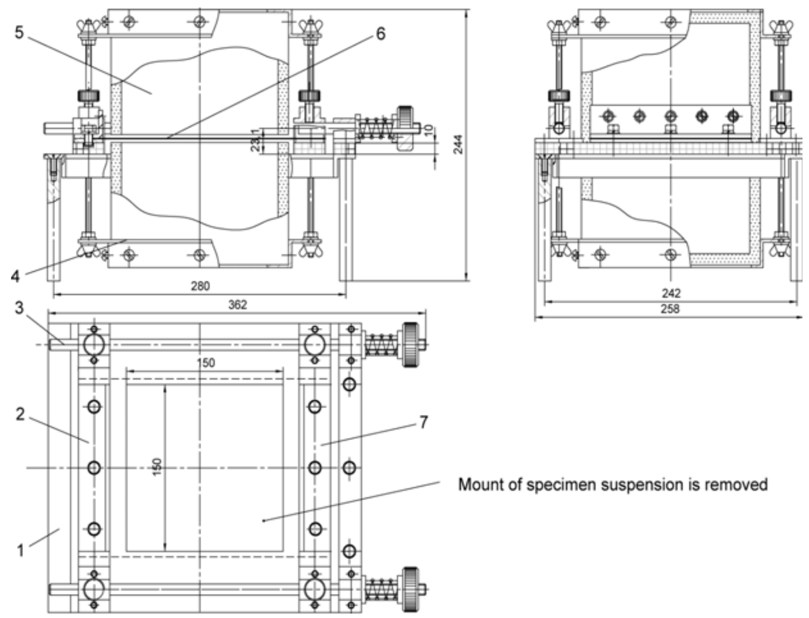

Figure 5. The EM-2 module assembly drawing: 1 – basis; 2 – immobile hold-down strap; 3 – guide rod; 4 – specimen's suspension unit; 5 – specimen in heat-insulating holder; 6 – heating element; 7 – mobile hold-down strap.

The main structural members of the module EM-2 are made of thermoresistant stainless steel, allowing service to it over a wide range of temperature change and heating-up time without cooling the hold-down straps of the heating element. The absence of such cooling helps to reduce the temperature field's distortion in the heating element. Heat-insulating holders of the specimen are made of thermoresistant ceramic materials with low heat conduction and are used for the protection of the lateral and the back surfaces of the specimens during testing.

For replaceable ohmic type heating sources in the module flat heating elements in the form of foil or grid with thickness 0.05 to 0.5 mm, width 30 to 150 mm and length 100 to 180 mm made of refractory metals and alloys (thermoresistant stainless steel, nichrome, Ti, Nb, Ta, Mo, W) are used. Also linear heating elements in the form of fibers with diameter 0.05 to 0.5 mm made of the similar materials are used. Choice of the type of the heating element and material, as a rule, depends on the required thermal conditions and on those of testing such as the peak temperature value on the specimen's surface, heating time, medium and pressure in the vacuum chamber. The size of the heating element depends on the specimen's size.

A number of tests have been conducted during the development of the experimental module. In particular, to estimate the heating element deformation in the process of heating there were conducted the tests of a 150 × 180 mm heating element made of thermoresistant stainless steel in the form of a foil of different thickness and of grids with different wire diameters and cell size. Test data showed that in the temperature range from 500 °C to 800 °C there is observed a wave-shaped deformation of the heating element, especially in areas adjacent to the hold-down straps. Minimum deformation was observed on the heating element, a twill-woven grid with wire diameter 0.12 mm and cell size 100 μк for which the maximum wave amplitude was 0.2 mm. At temperatures higher than 800 °C a deformation of the heating element was practically absent except for areas of 5 mm depth near the hold-down straps.

To estimate the level of temperature field non-uniformity the temperature values have been taken by means of thermocouples at some points on the surface of the heating elements. For all the heating elements considered the temperature values at points located in the central region of the 70 × 70 mm element differed by less than 4°C. Temperature values in the region of a 5 mm width located near the hold-down straps differed from values in the central point of the heating element by 10%. Figures 6 and 7 illustrate the photos of tests of two heating elements.

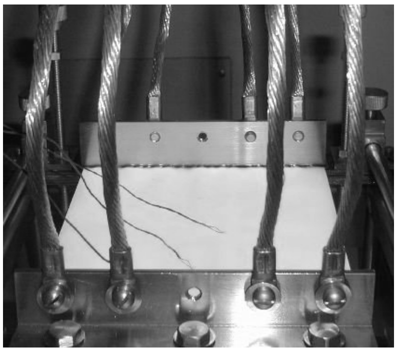

Figure 6. Heating element from stainless steel – 0.12 mm wire size grid and cell size 100 μk, temperature 800°C.

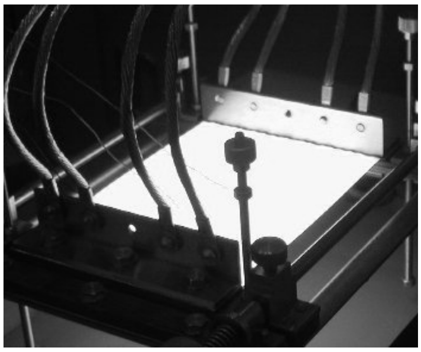

Figure 7. Heating element from heat-resistant stainless steel – 0.1 mm thick foil, temperature 1200°C.

The structure of the module EM-2 allowed tests to be made simultaneously. One or two (by a symmetrical heating mode - Figure 5) with specimens in the shape of rectangular parallelepiped with a size (length × width × height) in mm from 10 × 10 × (≈ 0) to 150 × 150 × 100. Figure 8 shows a photo of module EM-2 without flexible current inputs and lower setting frame.
With regard to the results of the experimental development and the experimental operation of the module EM-2, its modification EM-2A appeared allowing the use of heat-insulating holders of different size and heating elements from 20 mm to 180 mm in length. This is necessary in conducting the tests. A photo of module EM-2A is shown on Figure 9. When thermal tests are carried out at marginal allowable heating operation (peak temperatures from 1000 °C to 1700 °C, heating time from 5 minutes to several hours, tests in air, including those at over-pressure) based on module EM-2, a module EM-2B with water cooling of hold-down straps of the heating element has been developed, allowing one to avoid overheating and the destruction of module's structural members and uncontrolled heating of heat-insulating surfaces of specimens and thermocouple conductors of temperature sensors. Figure 10 shows a photo of module EM-2B with the specimens arranged in the vacuum chamber. Module EM-2A, if necessary, can be also equipped with a cooling system (modification EM-2A/2B).

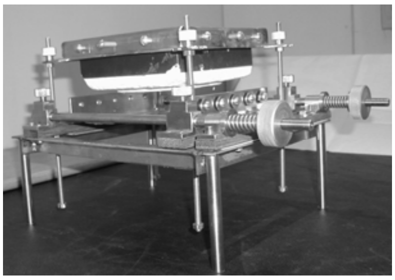

Figure 8. Experimental module EM-2 (lower setting frame and flexible current leads are removed).

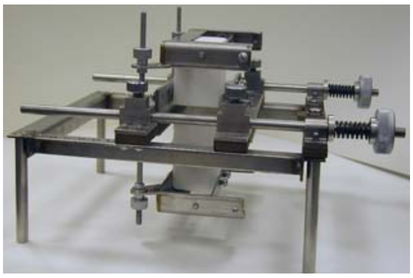

Figure 9. Experimental module EM-2A..

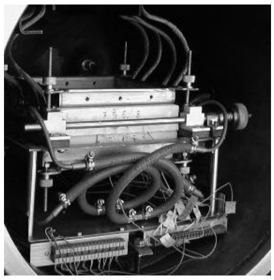

Figure 10. Experimental module EM-2B.

The information-measurement subsystem
The information- measurement subsystem (IMS) has been carried out using the software-hardware tools of the PXI family of National Instruments Co. A PXI is a free industry standard oriented to a solution of problems of experimental measurements and construction of automated systems for scientific research. The construction of the IMS facility based on PXI bus-module standard allows the use a reliable compact equipment providing high metrodological characteristics of the system and ease of configuration of all equipment under OC Windows control.

The computer architecture of systems in PXI standard includes three main components: Chassis, System controller, Functional modules and Software.
IMS general view and its configuration are shown in Figs. 11 and 12, respectively.

The measuring lines from the sensors of the physical values are linked to those of the connectors, then signals run to the inputs of multi-channel multi-functional measuring modules.

Figure 11. IMS general view.

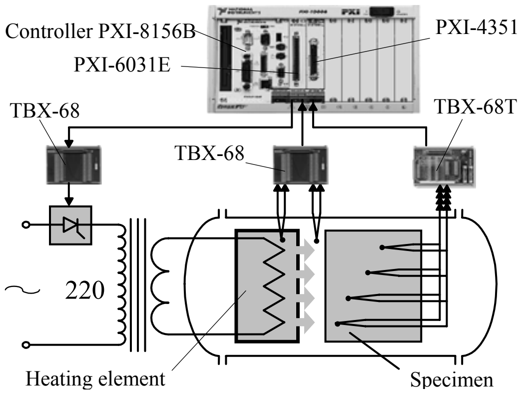

Figure 12. IMS configuration.

The IMS components.
The chassis PXI – 1000B, being a compact, universal and flexible platform, has 8 slots for installing functional modules of 3U standard. The first slot is for installing the crate's controller (the industrial computer). The remaining 7 slots are used for installing functional modules.
The integrated controller NI-8156B, based on a 233MHz Intel Pentium MMX processor, is a control tool for up to 7 functional modules. The controller NI-8156B, on the one side, provides efficiency sufficient for conducting the experiment and, on the other side, allows optimal use of the resources of power and ventilation, thus increasing the reliability of the whole IMS.
The multifunctional input/output control module NI-6031E involves: analog input channels-32 differential, analog-digital word length, bits-16, discrete frequency, k/word/s- up to 100, channels of analog output-2, analog-digital word length, bits-16. The module NI-6031E provides high-effective and reliable techniques for data acquisition: control when synchronization is required between the analog input and analog output, signal conditioning. NI6031E has a protection which reduces the ambient air temperature effect. A temperature drift on the equipment is less than 0.0006% C, providing accuracy in all media. The given module is used for recording parameters and "TBC-1" facility control.
For power control of the facility heater there is a thyristor drive in the system. The control analog signal is sent from the measuring-control module PXI-6031E to the thyristor drive. The size and form of the control signal of module's analog output are presented by a program.
The high-accuracy module PXI-4531 for voltage and temperature measurements includes: channels of analog input- 14 thermocouples; temperature measurement error (C) - 0.42 thermocouple'J'', analog-digital word length, bits- 24, discrete frequency, meas./sec-2 – 60, compensation of cold junction, automatic zero calibration, detection of thermocouples break. The module is used for precision temperature measurement. The module allows the operation with sensors of different types (thermocouples, resistance thermometers, theristors). It also allows one to use both standard and individual calibration of sensors (realized by program).
Software can be divided in to the following groups: measuring and control drivers and services; software tools; software of the upper (user's) level.
The program input/output of information between the computer and the measuring control equipment is provided by the software tools which include the input/output libraries and hardware drivers. This software provides support for the input/output equipment at the level of the operating system and also interfaces for programming system LabVIEW. Libraries of the input/output programs are used for programming the input/output operations oriented to a particular type of equipment:
LabVIEW is a high-effective medium for programming combining the intuitionism and ease of graphic approach with the flexibility of powerful programming language. Any program of LabVIEW is a virtual instrument and has a pictorial user interface. It is possible to connect LabVIEW with the program modules formed in other media of programming, for example, C++.

The main advantage of National Instruments technologies is that the software and hardware NI amount to a standard de facto. At the same time the software NI LabVIEW is open to the maximum and allows practical work with any hardware including those of Russian origin.

The software of the upper (user's) level consists of a testing program and a set of test programs. The software allows one to afford the following IMS modes of operation: configuration of functional modules; debugging and calibrating tests, individual calibration of each parameter; on-line monitoring of chosen parameters, conversion of chosen parameters, conversion of electrical values into physical ones; graphical and tabular display in the rate of the experiment with the possibility of archival recording of results on hard disks; translation of observed data into a local computer network.
The IMS admits simple enough and quick updating for the optimal solution of wanted problems. Such updating can be performed both by changing the software and by resupplying the functional modules and other hardware tools.
The testing program is a file at which the starting of the computer screen opens a virtual instrument panel which takes up the control. The IMS main virtual panel is shown in Figure 13.

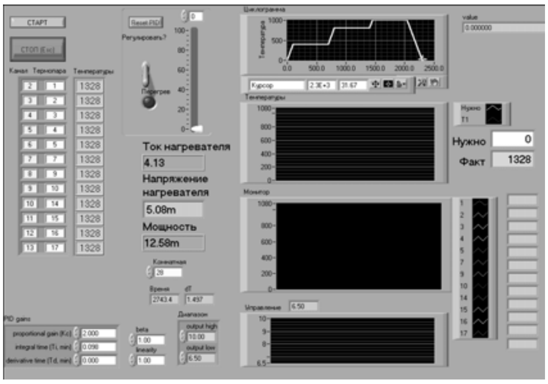

Figure 13. The main virtual panel of the IMS.

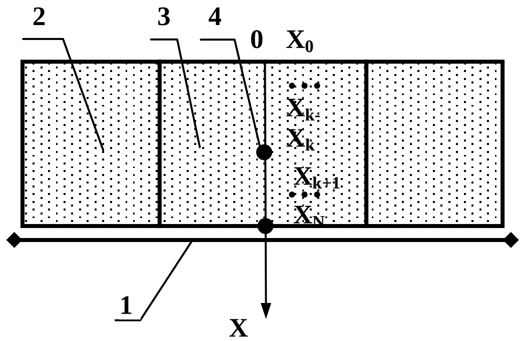

Figure 14. Temperature measurement circuit in specimen: 1 – heating element; 2 – specimen; 3 – specimen's sensing element; 4 – setting points of thermal sensors with coordinates X_k, k=1,…,N.

In the area of the main panel there are physical property indicators and function buttons. The buttons are responsible for the program modes of operation and open other panels.
A virtual panel allows the opening of the data file – the experiment protocol – where all necessary measurements will be filed in the course of the experiment.
In the vacuum chamber 1, see Figure 3, on the work bench, 6, there is an experiment module, 5, with test pieces. Thermocouples mounted on the heater and in specimens are lead to a contact plate, 8, together with thermocouples which measure temperature inside the chamber and that of input terminals. By means of a thermal resistant cable the contact plate is connected via the pressure seal, 4, with the outlet connector, 3, which by cable, 2, is connected with the terminal units TBX-68 and TBX-68T.

Preparation and thermal testing technique
In the preparation of the thermal tests carried out for determining the thermophysical properties of materials it is necessary to account for the requirements of the physical statement of research work, for the features of the IHTP methodology, in particular, special requirements to model measurements, for the characteristics of used thermosensors, for the initial conditions and to those of heat loading at the boundaries of the specimen under investigation, for the potentials of the available hardware equipment.
The main task of such tests, in most cases, is to measure the temperatures $T_k(\tau),\ \tau_0 \le \tau \le \tau_n$ at given points of the specimen with coordinates $X_k,\ k = 0,\dots,N$, see Figure 14, where $\tau_0,\ \tau_n$ are the times of measurement initiation and termination, respectively. In doing so, it is necessary to realize the required heat transfer conditions at the boundaries of the specimen, or these boundaries conditions must be determined in the process of testing. To measure the temperature in the specimen, thermocouples of different types (WRe(5%)–WRe(20%), PtRh(30%)-PtRh(6%), PtRh(10%)–Pt, Chromel-Alumel, Chromel-Copel, Cu-Copel), with diameters 0.05 to 0.1 mm. are used. Thermoelectrodes of thermocouples are connected by a butt welding method, allowing reduction of the distortions introduced by thermocouples to the temperature field of the specimen.
The dimensions of the experimental specimens are defined by the accepted physical heat transfer model and the material's assumed thermophysical properties. For example, to realize a one-dimensional model it is necessary to

provide a certain ratio of thickness and two other dimensions of the specimen. The use of the above-described experimental module allows the avoidance of rigid requirements to the specimen's size on the part of the experimental equipment. Possible dimensions of specimens are given in Table 1.

Selection of the technique of thermocouple installation in the specimen depends on the measurement model and on the size, structure and mechanical properties of the material. In some cases it is possible to arrange thermocouples directly during the material manufacture. Sometimes, by technological considerations, the thermocouples can be arranged in the so-called sensing element of the specimen, see Figure 14. The sensing element is made out of the material under investigation and it serves for the thermocouples location. Then this element is densely installed or glued in a specially made hole in the specimen. It is required to provide good thermal contact between the sensing element and the specimen. Each of the known techniques of the thermocouple arrangement has its own advantages and disadvantages which might be connected with the accuracy of thermocouple arrangement relative to a hot face, with a distortion of the temperature field, with the provision of thermal contact with the material, with manufacturing problems, etc.

The number and places of thermocouple location (temperature measurement model) depends on the IHTP to be solved and is defined from a solution of the corresponding task of the experiment design [2]. The accuracy of IHTP solving depends essentially on the accuracy of the thermocouple placement. So the important task is to control the position of the thermocouples. Such control is performed in the process of specimen manufacture and after by X-ray methods or tomography.

To provide thermal conditions on specimen's back and lateral faces there are special heat-insulating holders made of ceramic materials with operating temperature 1000 to 1750 °C and coefficient of thermal conductivity 0.03 to 0.11 W/(m × °C). The distance from the heating element to the specimen surface is sampled for each particular test and can vary from 0 (at contact heating) to 10 mm (at radiation heating). For testing the electrically conducting materials there is a circuit with radiation heating or an electric-insulation coating of the heating element based on thermoresistant ceramic.

Heating control is made by the temperature of the hot face of the specimen or the heating element. Use of the symmetrical heating method for two identical specimens, see Figure 5, and the measurement of the electrical characteristics of the heating element (root-mean square voltage $U(\tau)$, root-mean square current $I(\tau)$) allows one to estimate accurately the heat flux which is put into the specimen: $q_2(\tau) = U(\tau) \times I(\tau)/(2\times S)$, $0\leq \tau \leq \tau_n$, S – square of the working area of the heating element. This allows one to acquire in one test the data necessary for unique definition from a solution of IHTP of a complex of thermophysical characteristics of the material, for example, temperature dependences of the coefficient of thermal conductivity and volumetric heat capacity [5,6].

Heating control, temperature measurements in specimens and the heating element, as well as measuring of electric characteristics of the heating element and recording the measurement data is performed using the information-measurement subsystem, which also provides collection and preliminary processing of the experimental information, formation of test protocol and report. Test protocols are transferred into a database of the AS, where data is archived and stored. AS provides secondary processing, analysis and interpretation of the experimental data based on the solution of the corresponding IHTP using the application software [5] and a solution of problems of heat transfer process modeling and experiment design at the stage of the preparation of the experimental investigations.

Conclusions

The experimental-computational system described here includes the experimental equipment, instrumentation and automation facilities, hardware, methodical support and application software, modern methodology of identification of the unsteady heat transfer processes in materials and structures based on IHTP techniques.

Development of such systems is a perspective direction for implementing the inverse methodology in scientific and engineering practice of thermophysical investigations and thermal tests of promising composite materials for aerospace engineering, thermal and nuclear power, ferrous and nonferrous metallurgy, chemical industry, engine technology, etc.

The system can be effectively used in research, design and testing laboratories engaged in the development and investigation of new composite heat-engineering materials and structures.

Acknowledgement

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Table 1
The main technical data and parameters of the experimental-computational system

Characteristic, parameter	Value
Vacuum chamber volume [m^3]	0.1
Vacuum chamber internal size [m]: • Diameter • Length	 0.47 0.60
Medium in chamber	Vacuum, air, N_2, inert gases
Ultimate vacuum in cool state [bar]	2.5×10^{-8}
Maximum overpressure in chambers [bar]	1.6
Water coolant pressure [atm]	2 to 2.5
Water coolant temperature [°C]	5 to 45
Heating source power [Kw]	up to 40
Peak temperature of specimen surface [°C]	1600
Rate of heating [°C/s]	up to100
Change of heating temperature	by program from AS
Models of experimental modules: • With uncooled current terminals • With adjustable size of setting frame • With cooled current terminals of heating element • With adjustable size of setting frame and cooled current terminals of heating element	 EM-2 EM-2A EM-2B EM-2A/2B
Number of simultaneously tested specimens	1 or 2
Size of investigated specimens (length × width × thickness) • Minimum [mm] • Maximum [mm]	 10 × 10 × (≈0) 150 × 150 × 100
Number of input channels AS	32
Number of output channels AS	2
Number of temperature measurement channels	14
Temperature [°C]	0.5
Temperature dependences of materials properties (in the range of room temperature up to 1600 °C)	Thermal conductivity and/or volumetric heat capacity and/or emissivity

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