A03

AN EXPERIMENTAL-COMPUTATIONAL SYSTEM FOR THE DETERMINATION OF THERMAL PROPERTIES OF MATERIALS. III. APPLICATION FOR SPACECRAFT STRUCTURES TESTING

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Abstract - In this part the results of the practical implementation of hardware and software, presented in the previous two parts for the estimating the thermal properties of thermal-insulating materials [1], are given. The main purpose of this study was: to confirm operability and effectiveness of the methods developed and hardware equipment for determining thermal properties (TP) of particular modern high porous materials on the basis of the solution of the inverse heat conduction problem (IHCP) using stand TVS-1 in MAI [2]. The physical and mathematical model of the heat transfer processes in the experimental specimens of thermal materials are given. Mathematical formulations of the corresponding IHCP are also presented. The requirements are formulated to experimental specimens, parameters and testing conditions. A scheme of thermal tests and a test technique are developed. The results of specimens' thermal tests for determining TP of the light-weight thermal-insulating material of ETTI-CF-ULT type and ETTI-CF-ERG type thermal conductivity and volumetric heat capacity in the temperature range 20…1000°C are given. The material is delivered for study by ASTRIUM GmbH upon agreement with ESTEC/ESA. Thermal tests of specimens' material were carried out using a specially designed and manufactured experiment module EM-2 at the thermo vacuum stand TVS-1 in MAI for four specimens: two specimens (1a, 1b) of the material ETTI-CF-ULT and two specimens (2a, 2b) of the material ETTI-CF-ERG.

1. INTRODUCTION

In this section the physical and mathematical model of the heat transfer process in the specimen installed in the experimental module EM-2 [2] is considered.

The experimental specimen is a plate from the material under study in the form of a rectangular parallelepiped with the big edge thickness-to-length ratio not less than 1:5. Such a ratio of the specimen's dimensions, as well as the using of a symmetrical heating scheme of two identical specimens and the corresponding test procedure [2] provide in the course of testing a formation in the specimen a temperature distribution close to one-dimensional. Initially a uniform temperature distribution is realized in the specimen.

To execute thermal tests of specimens in order to study their thermal statement it is expedient to select the specimen's thickness close to that of the submitted plates of the material. The input data for identification of the thermal properties of indestructible thermal-insulating materials are developed based on the observed data and include boundary conditions (first or second kind) and time temperature dependences at some internal points of the specimen. The type of boundary conditions and the number of temperature measurement points should satisfy the conditions of uniqueness of the inverse problem solution [3]. The conditions of uniqueness usually determine a minimum required number of measurements to be made in one experiment. For simultaneous determination of temperature dependences on the coefficient of thermal conductivity and volumetric heat capacity it is necessary firstly to measure a heat flux passing through the specimen which differs from zero at least at one boundary and secondary to measure temperature not less than in two additional points (internal or on the surface with boundary conditions of second kind). Selection of a temperature-measuring scheme (location of thermocouples in specimen) is conditioned by the material features. A lightweight heat-insulating material is high-porous, brittle enough and electroconductive. This considerably complicates the internal installation of thermocouples in the specimen and prevents temperature measurements from being accurate by such installation. With regard to these features of the material under study it is of little sense to install thermocouples inside the specimen.

Should one of the temperature measurements on the heating or back surface of the specimen be used as a boundary condition, the condition of uniqueness will be violated. It follows that it is of sense to provide experimental determination of the heat flux density on the back face and use it as a boundary condition, together with temperature measurements on both surfaces of the plate as complementary information for solving the inverse problem. For the described scheme of experimentation and measurements, the data obtained in one test will be sufficient to determine two properties: coefficient of thermal conductivity $\lambda(T)$ and volumetric heat capacity $C(T)$. Finally, a scheme of temperature measurements has been chosen with thermosensors installed only on the heating and back surfaces of the specimen [2].
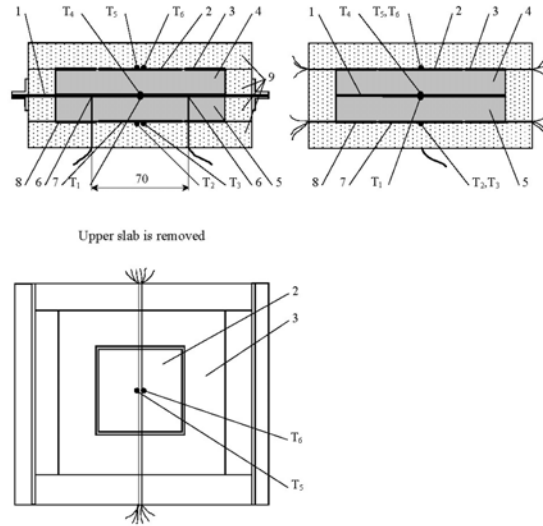
Figure 1. A testing scheme for specimens: 1 – heating element of module EM-2; 2 – calorimeter on the upper specimen (1a/2a); 3 – mask of the upper calorimeter; 4 – upper specimen (1a/2a); 5 – lower specimen (1a/2b); 6– voltage measuring points on the heating element; 7 – calorimeter on lower specimen (1b/2b); 8 – mask of the lower calorimeter; T_1, T_2, T_3 - thermocouples on the lower specimen (1b/2b); T_4, T_5, T_6 - thermocouples on the upper specimen (1a/2a).

Based on the given physical model, a mathematical model of heat transfer process in the material's specimen (infinite slabted plate of known thickness) can be presented as follows [4]:

$$C(T)\frac{\partial T}{\partial \tau} = \frac{\partial}{\partial x}\left(\lambda(T)\frac{\partial T}{\partial x}\right), \quad x \in (X_0, X_1), \quad \tau \in (\tau_{\min}, \tau_{\max}] \tag{1}$$

$$T(x, \tau_{\min}) = T_0(x), \quad x \in [X_0, X_1] \tag{2}$$

$$-\lambda(T)\frac{\partial T(X_0, \tau)}{\partial x} = q_1(\tau), \quad \tau \in (\tau_{\min}, \tau_{\max}] \tag{3}$$

$$-\lambda(T)\frac{\partial T(X_1, \tau)}{\partial x} = q_2(\tau), \quad \tau \in (\tau_{\min}, \tau_{\max}] \tag{4}$$

Data on the material known properties are presented in Table 1.

Table 1: Known properties of material.

Type of material	Type of materials	Density, kg/m^3	Emissivity	Destruction temperature, K
1	ETTI-CF-ULT	42	0.9	3000
2	ETTI-CF-ERG	48	0.9	3000

Conditions and modes of specimen's heating during thermal tests offered by ESTEC/ESA specialists:
- Heating of specimens in a vacuum with pressure $p \leq 1 \times 10^{-7}$ bars.
- Prior to the tests, the specimens should be heated up to 700 K to remove the absorbed water.
- Initial temperature of the specimens is $T_0 = 293K$.
- The behaviour of the temperature change of the heating surface $T_w(\tau), 0 \leq \tau \leq \tau_n$ is trapezoidal. The first segment – heating by a linear law of temperature change at a rate of 2 to 8 K per second from temperature T_0 to $T_{\max}$. The second segment – with uniform temperature $T_w(\tau) = T_{\max} = constant$ during 1200 s. The third segment – cooling by a linear law of temperature change at a rate of 2 to 8 K per second from $T_{\max}$ to T_0. The peak temperature of the specimen's heating surface from 570 K till 1270 K.

It should be noted that on trials using the stand TVS-1 there is no way to simulate the specimen cooling mode by the form suggested here. A possible approach is to switch off the heating at the end of a section with uniform temperature and to continue natural cooling of the specimen heated surface in conditions of cooling system operation of the experiment module and vacuum chamber. The results of tuned-up tests of the material under study are shown that with such a scheme the specimen cooling to the initial temperature takes several hours.

Temperature measurement schemes chosen with regard to requirements of the IHCP statement, properties of the material under investigation and rules of thermal test procedure on stand TVS-1 [2] are presented in Tables 2 and 3. The coordinates of the measuring points $X_m, m = 0,...,M$ are read from the specimen's back face, see Figure 1.

Thermocouples are installed in specimens 1a/2a:

- T_1 at the heating surface;

- T_2 and T_3 on the copper calorimeter located on the back surface (T_3 - stand-by thermocouple).

- Thermocouples are installed in specimens 1b/2b:
- T_4 at the heating surface;

- T_5 and T_6 on the copper calorimeter located on the back face (T_6 - stand-by thermocouple).

With consideration for the requirements presented by ESTEC/ESA, the requirements to the IHCP statement, the test procedure on stand TVS-1 and the results of preliminary studies, the following final requirements have been formulated for the specimens, tests and parameters:

- The experimental prototypes have the shape of rectangular parallelepipeds with a thickness – big rib length ratio not less than 1:5.
- The specimen thickness should be close to that of material's reference plate.
- Temperature sensors are installed only on the heating and back surfaces of the material specimens.
- The heat flux definition on the heating and internal faces of specimens should be provided.
- The thermocouples wire diameter should not exceed 0.2 mm.
- The specimen heating runs in a vacuum with pressure $p \leq 1 \times 10^{-7}$ bars.
- Prior to testing the material specimens they should be warmed until a temperature of about 430 °C.
- Measurements and record of thermal and electrical parameters in each test are conducted for 3000s.
- Thermal contact between the thermocouples and material specimens should be close to ideal.

2. DEVELOPMENT AND MANUFACTURE OF SPECIMENS

The material under study was delivered in the shape of 4 round plates of 200 mm diameter and about 25÷26 mm thickness. Two plates were from the material under number 1 (ETTI-CF-ULT, plates 1a and 1b) and two plates - from the material under number 2 (ETTI-CF-ERG, plates 2a and 2b).

The experimental specimens were developed and manufactured with regard to the requirements formulated in subsection 1. The specimens are rectangular parallelepipeds of the sizes given in Tables 4 and 5.

As temperature sensors Chromel-Alumel with thermocouple wire of 0.1 mm in diameter and butt-welded are used. Such thermocouples T_1 and T_4 are installed on the specimen's heating surfaces.

To determine the heat flux on the specimens' back faces, the calorimeters are installed there in the form of thin copper plates of 60×60×0.3 mm in size with guard masks (frames) from the same material, see Figure 1.

Two thermocouples T_2 are installed on each calorimeter, T_3 - on the lower calorimeter and T_5, T_6 - on the upper calorimeter. Thermocouples T_3 and T_6 are stand-by ones. These thermocouples are used for temperature measurements on the back surfaces of specimens. They also are made using Chromel-Alumel thermoconductors of 0.1 mm in diameter but spot-welded. The points of installation of all thermocouples are shown in Figure 1. The coordinates of the measuring points are given in Tables 2 and 3.

A special thermoresistant coating is used to provide the electric insulation of thermocouples to be installed on the heating surfaces of specimens from the material of the specimen and the heating element. Thermocouple conductors installed on the calorimeters are electrically insulated from the specimen from the calorimeter surface. A thin boron nitride layer is applied on the heating surfaces of the specimens in order to electrically insulate against the heating element.

Table 2: Temperature measurement schemes in the material specimens ETTI-CF-ULT.

Specimen	X_0, mm Thermocouples numbers	X_1, mm Thermocouples numbers	Note
1a	0,0 T$_4$	26,0 T$_5$, T$_6$	Thermocouple T$_6$ (stand-by)
1b	0,0 T$_1$	26,0 T$_2$, T$_3$	Thermocouple T$_3$ (stand-by)

Table 3: Temperature measurement schemes in the material specimens ETTI-CF- ERG.

Specimen	X_0, mm ———— Thermocouples	X_1, mm ———— Thermocouples	Note
2a	0,0 ———— T_4	24,8 ———— T_5, T_6	Thermocouple T_6 (standby)
2b	0,0 ———— T_1	24,7 ———— T_2, T_3	Thermocouple T_3 (standby)

Two identical specimens have been made for testing: 1a, 1b of the material ETTI-CF-ULT and 2a, 2b of the material ETTI-CF-ERG. Specimens of regular shape by geometry have been manufactured, see Tables 4 and 5, with consideration the requirements formulated above. At this stage the specimens were weighed, their volume was defined and the reference material density was specified, see Tables 4 and 5. A photograph of one of the specimens prior to installation of thermocouples and calorimeters is presented in Figure 2.

Table 4: Parameters of the material specimens ETTI-CF-ULT (prior to testing).

Specimen	Size, m	Volume, m^3	Weight, kg	Density, kg/m^3
1a	0.138 x 0.138 x 0.026	0.000495	0.02662	53.78
1b	0.138 x 0.138 x 0.026	0.000495	0.02938	59.35

Table 5: Parameters of the material specimens ETTI-CF-ERG (prior to testing).

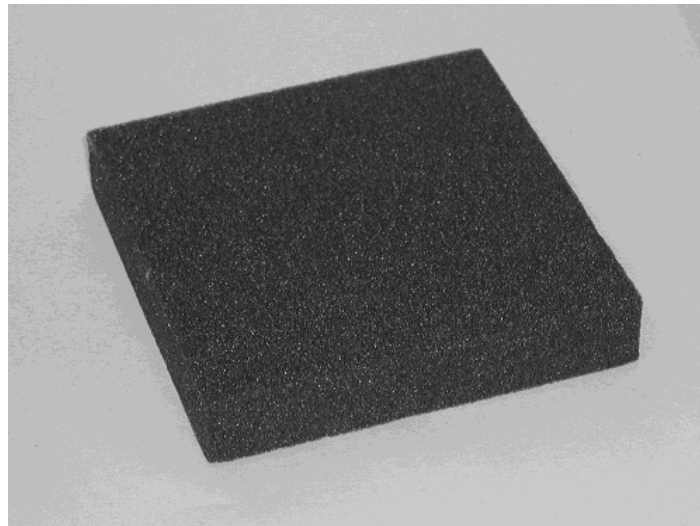| Specimen	Size, m	Volume, m^3	Weight, kg	Density, kg/m^3
2a	0.138 x 0.138 x 0.0248	0.0004722	0.02695	57.07
2b	0.138 x 0.138 x 0.0247	0.0004703	0.02760	58.69

Figure 2. A specimen of material.

When installed on the specimen's heating surfaces, the thermocouples were fixed on the surface by means of a special thermoresistant compound. The thermocouple junction relative to the specimen's surface was hence under control. The calorimeters with guard masks (frames) installed on the back faces of the specimens were also fixed on specimen's surface by means of a special thermoresistant compound. Prior to the thermocouple installation, the calibration and functional test was made.

A picture of the experimental module with installed specimens and removed upper plate of the heat-insulating holder is shown in Figure 3, and a picture of the experiment module prepared for testing is shown in Figure 4.

The assembled experiment module was installed in the vacuum chamber of stand TVS-1.
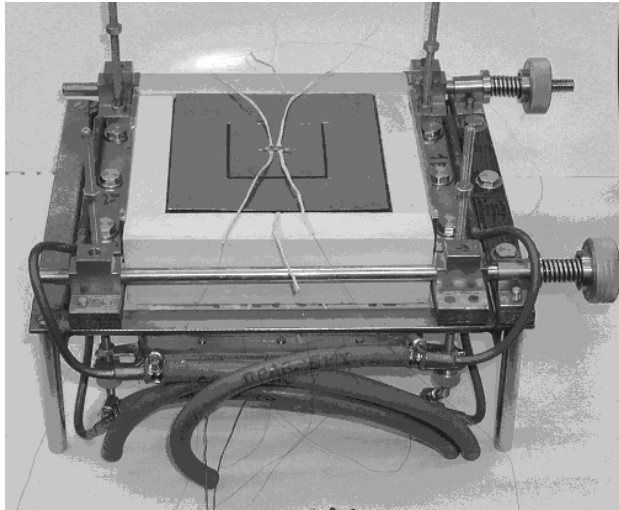
Figure 3. The experiment module with installed specimens and removed upper plate of the heat-insulating holder.
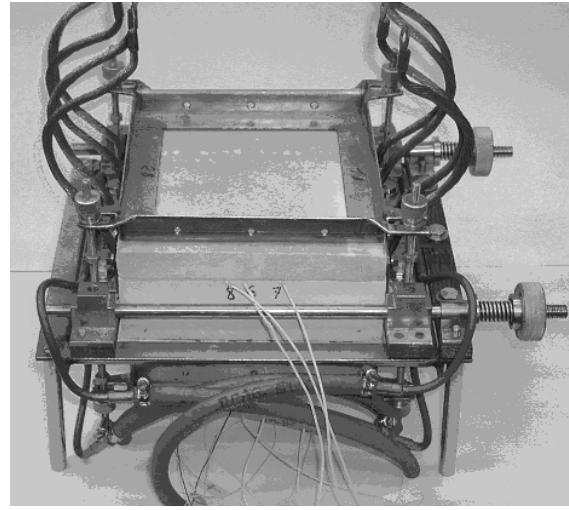

Figure 4. The experiment module prepared for testing.

3. TESTING

The purpose of the present thermal tests was the measurement of the following functions of the thermal statement of the experimental specimens of a lightweight heat-insulating material in the process of their non-steady heating:

- temperatures $T_m(\tau)$, $0 \leq \tau \leq \tau_n$, $m = 0,1$ on the front and back faces of specimens, see Tables 2 and 3;
- heat flux $q_2(\tau)$, $0 \leq \tau \leq \tau_m$ on the front (heating) surfaces of the specimens;
- heat flux $q_1(\tau)$, $0 \leq \tau \leq \tau_m$ on the back surfaces of the specimens.

Since the number of specimens was limited (two specimens from material ETTI-CF-ULT and two from ETTI-CF-ERG), testing of each pair of specimens was conducted by the following program:

- a preliminary test with the pre-heating mode № 0 in the conditions of a vacuum when the material was warmed up in order to remove the absorbed water. Such heating conditions were allowed to perform heating of the material specimens along the whole thickness for a temperature not less than 430 °C. The consequent conservation of specimens installed in the experimental module until the next test was made in conditions of vacuum (the experiment module with specimens remained in the vacuum chamber with a vacuum level $\leq 1 \times 10^{-5}$ bars). The heating mode № 0 (600 °C):
    - the first segment: heating by a linear law at a rate of 4 °C/s from $T_o = 15\ °C$ to $T_{max} = 600°C$;
    - the second segment: with uniform temperature $T(\tau) = 600\ °C$ for 1800 s;
    - the third segment: natural cooling from $T(\tau) = 600\ °C$.
- a test with the heating mode № 1 in conditions of a vacuum $\leq 1 \times 10^{-7}$ bars. The conservation of specimens in conditions of a vacuum $\leq 1 \times 10^{-5}$ bars until the next test. The heating mode № 1 (300):
    - the first segment: heating by a linear law at a rate of 4 °C/s from $T_o = 18\ °C$ to $T_{max} = 300\ °C$;
    - the second segment: with uniform temperature $T(\tau) = 290\ °C$ for 1200 s;
    - the third segment: natural cooling from $T = 290\ °C$.
- a test with the heating mode № 2 in conditions of a vacuum $\leq 1 \times 10^{-5}$ bars. The conservation of specimens in conditions of vacuum $\leq 1 \times 10^{-5}$ until the next test. The heating mode № 2 (550):
    - the first segment: heating by a linear law at a rate of 4 °C/s from $T = 290\ °C$ to $T_{max} = 550\ °C$;
    - the second segment: with uniform temperature $T(\tau) = 550\ °C$ for 1200 s;
    - the third segment: natural cooling from $T = 550\ °C$.
- a test with the heating mode № 3 in conditions of a vacuum $\leq 1 \times 10^{-5}$ bars. The conservation of specimens in conditions of vacuum $\leq 1 \times 10^{-5}$ until the next test. The heating mode № 3 (800):
    - the first segment: heating by a linear law at a rate of 4 °C/s from $T_o = 12,2\ °C$ to 800 °C;
    - the second segment: with uniform temperature $T(\tau) = 800\ °C$ for 1200 s;
    - the third segment: natural cooling from $T = 800\ °C$.
- a test with the heating mode № 4. The heating mode № 4 (1000):
    - the first segment: heating by a linear law at a rate of 4 °C/s from $T_o = 14\ °C$ to $T_{max} = 1000\ °C$;
    - the second segment: with uniform temperature $T(\tau) = 1000\ °C$ for 1200 s;

- the third segment: natural cooling from $T = 1000\ {}^{o}C$

On completion of the thermal tests, a disassembly of the experiment module and specimens was made. The specimens were released from complimentary materials, measured and weighed in order to estimate the material density after testing. The corresponding results are given in Table 6.

Table 6: Parameters of the material specimensETTI-CF-ULT (after testing).

Specimen	Size, m	Volume, m^3	Weight, kg	Density, kg/m^3
1a	0.138 x 0.138 x 0.026	0.000495	0.02385	48.18
1b	0.138 x 0.138 x 0.026	0.000495	0.02664	53.82
2a	0.138 x 0.138 x 0.0248	0.0004731	0.023265	49.18
2b	0.138 x 0.138 x 0.0247	0.0004717	0.023535	49.89

The experimental data of specimens 1a and 1b of the material ETTI-CF-ULT and 2a and 2b of the material ETTI-CF-ERG as $T_m\ (\tau)$, $0 \le \tau \le 3000$ s (for the heating modes №2 and №3 only) are presented in Figures 5-8. Since indications of the main thermocouple T_2 actually coincided with those of the standby thermocouple T_3, there are shown only the readings of the T_2. This remark is true also for the thermocouples T_5 and T_6.

Tests carried out have shown that:

- After tests, the experiment module, including the heat-insulating holder, shows no signs of heating.
- After a series of tests under different conditions, the material specimen surfaces have no observable signs of heating or destruction. The material only became more rigid (brittle) and easy to crumble.
- Density of specimens 1a, 1b, 2a and 2b, see Tables 4,5 and Figure 9, determined before testing differed considerably (22%, 29.2%, 15.9% and 18.2%, respectively) from the value given in Table 1. The possibility must not be ruled out that the material density given in the run of heat treatment at 700 K. In our case, however, under estimation at this stage was the density of basic materials. However the density of material specimens 1a and 1b after tests, see Table 6, also was considerably different (12.8% and 22%, respectively) from the value given in Table 1. The density of specimens 2a and 2b which, by initial data, are made out of the same material differed not scientifically (2.8%). Comparison of the results in determining the density of the material specimens after tests showed that specimens 2a and 2b have a density close to a value given in Table 1 for the material ETTI-CF-ERG (the density after tests, see Table 6, was different at 2.4% and 3.8%, respectively). Therefore its density corresponds with the tabular value of density for material ETTI-CF-ERG, within the accuracy of measurements.
- At all heating conditions, a more rapid cooling of the specimen's heating surfaces as against their back surfaces was observed. This can be explained by the cooling of the heating element depending on the cooling system operation of the experiment module.
- Peak temperature values on the heating surfaces of the specimens 1a and 1b measured in the course of the tests under prescribed heating conditions differ insignificantly from each other (up to 3%) (for specimens 2a and 2b from 0.2% at № 3(1000) and № 4(1000) up to 5% at others). By this, the temperature on the surface of the specimen 1a was higher than that on the face of specimen 1b (apart from conditions № 4 (1000) and temperature on the surface of the specimen 2a was higher than that on the surface of the specimen 2b (apart from modes № 4 (1000)).
- Peak temperature values on the back surfaces of specimens 1a and 1b realized in the course of the tests under prescribed heating conditions differ insignificantly (from 4.3% in № 1 (290) to 0.4% in № 4 (1000), as well as 2a and 2b). This together, with a previous remark taken into account, was allowed to count in favor of sufficient heating symmetry of specimens in the realized scheme of tests.
- At all heating conditions a peak temperature on the back face of the specimen 1b was higher than that on the back face of specimen 1a. Perhaps, this is because of a difference in the material thermal properties of specimens 1a and 1b (particularly, specimen 1b has a higher density).
- At all heating conditions for specimens 1a and 1b the difference in values of the peak temperature on the specimens' back surface and the peak temperature on the specimens' hot surface was within the limits of 128 °C for № 1 (290) until 163 °C for № 2 (550). A mean value of this difference for all conditions was about 143 °C. At all heating conditions for specimens 2a and 2b the difference in values of the peak temperature on specimens' back surface and peak temperature on specimens' heating surface was within the interval of 202.4 °C for № 2 (550) till 216.5 °C for № 4 (1000). A mean value of this difference for all conditions was about 209 °C.
- Comparisons of the heating merits of specimens 2a and 2b of ETTI-CF-ERG and specimens 1a and 1b of ETTI-CF-ULR have shown, that for the same heating specimens of ETTI-CF-ULR there is significantly higher response at the back surfaces (about 50 °C or 25%).
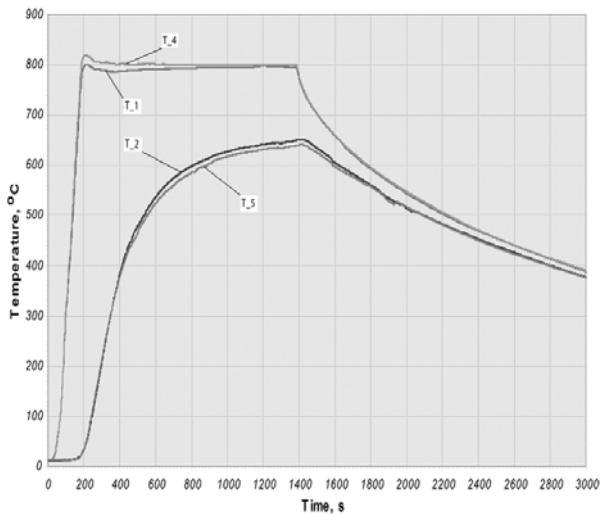
Figure 5. The temperature measurements in specimens 1a and 1b of material ETTI-CF-ULT under testing at the heating mode № 3 (800).
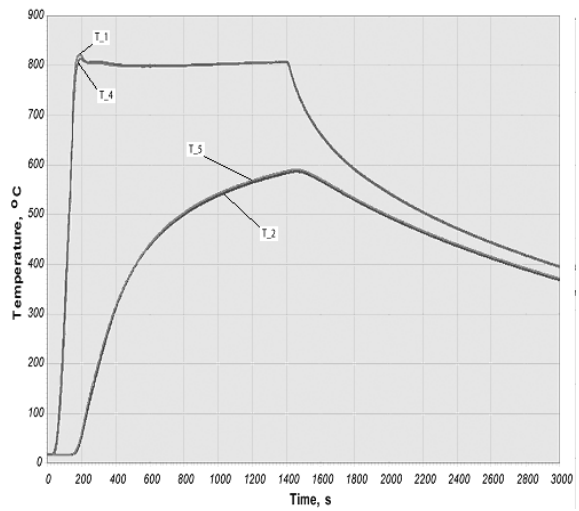

Figure 6. The of temperature measurements in specimens 2a and 2b of material ETTI-CF-ERG under testing at the heating mode № 3 (800).
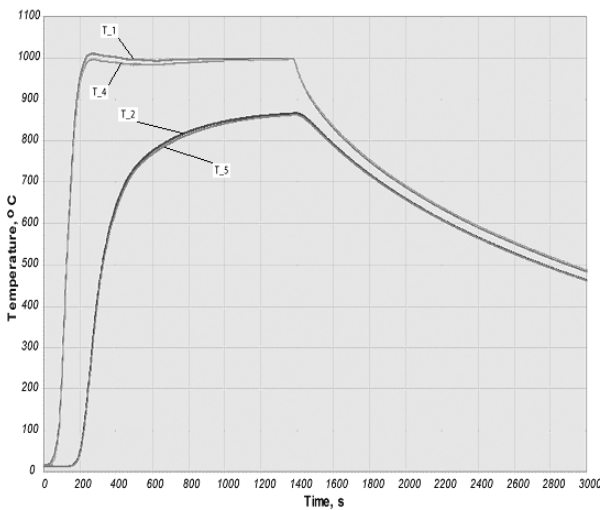

Figure 7. The temperature measurements in specimens 1a and 1b of material ETTI-CF-ULT under testing at the heating mode № 4 (1000).
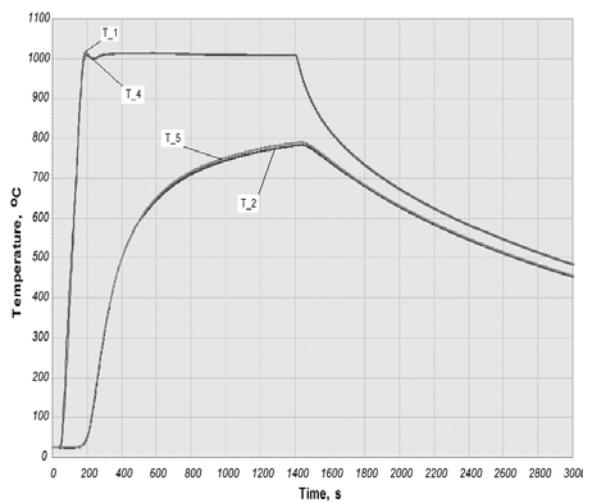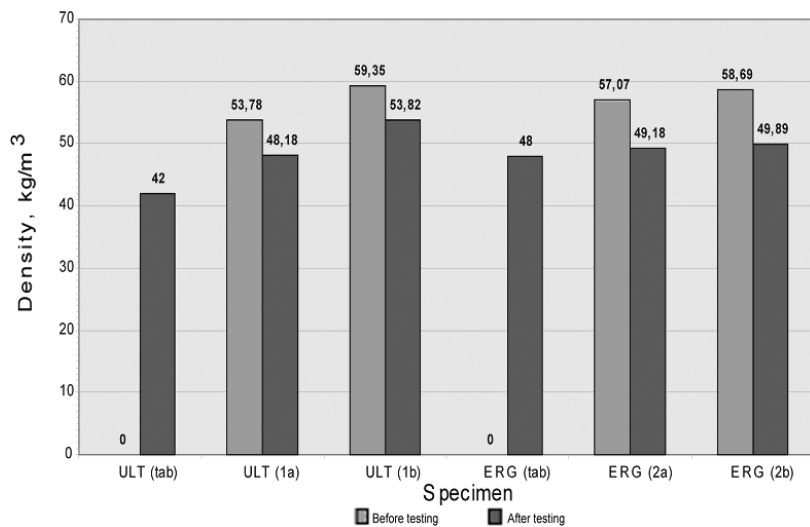

Figure 8. The temperature measurements in specimens 2a and 2b of material ETTI-CF-ERG under testing at the heating mode № 4 (1000).

Figure 9. Variations of the density of the specimens of material ETTI-CF-ERG and ETTI-CF-ULT.

4. ESTIMATION OF the THERMAL PROPERTIES BY AN INVERSE METHOD

In models (1) – (4) the coefficients $C(T)$ and $\lambda(T)$ are unknown. The complimentary information needed for solving the inverse problem prescribed are the results of the temperature measurements

$$T^{\exp}(x_m,\tau) = f_m(\tau), \quad m = \overline{1,2}, \quad x_1 = X_0, \ x_2 = X_1 \tag{5}$$

In the inverse problem (1) – (5) it is essential above all to indicate the domain of definition of the unknown functions in the form of the temperature interval, common for all experiments, $[T_{\min}, T_{\max}]$ where the inverse problem under analysis has a unique solution. The initial temperature minimum value is taken for $T_{\min}$, the temperature maximum value obtained from the thermocouple located on the heating surface - for $T_{\max}$. A least-square residual of computational and measured temperatures at points of the thermocouple installation is given by [5]:

$$J(C(T),\lambda(T)) = \sum_{m=1}^{2} \int_{\tau_{\min}}^{\tau_{\max}} (T(x_m,\tau) - f_m(\tau))^2 \, d\tau , \tag{6}$$

where $T(x_m,\tau)$ is defined from a solution of the boundary-value problem (1) – (4).

The heat flux density on the heating surfaces $q_2(\tau)$, $0 \le \tau \le \tau_n$ can be defined by electric power $W(\tau)$ of the heating element based on the measurements of the active voltage $U(\tau)$ at the boundaries of the heating element operating zone with area A and active current intensity $I(\tau)$ in the scheme of the heating element:

$q_2(\tau) = W(\tau) / (2 * A), \quad 0 \le \tau \le \tau_m$ (7)

where τ - time, τ_n - measurement termination time.

For the given tests a heating element was used in the experiment module which was in the form of a refractory stainless steel foil of 180 mm in length, 140 mm in width and 0.1 mm in thickness. A domain 140×70 mm in size was considered for the operating zone of the heating element for the determination of q_2.

It is possible to estimate and record heat losses at the instants of time τ_j depending on the heat capacity of the heating element from stainless steel.

$q_2^*(\tau) = q_2(\tau) - q_c(\tau)$ - heat flux on the heating surface of specimen considering heat losses;

$$q_c(\tau_i) = \rho_h \, \delta_h \, c_h \, \frac{\partial T(X_o,\tau)}{\partial \tau} \tag{8}$$

where $q_c(\tau_i)$ are losses of heat flux depending on the heat capacity of the heating element, $\rho_h = 7900$ kg / m^3 - stainless steel density; $\delta_h = 0{,}0001$ mm -heating element thickness; $c_h = (450 + 0.57\,T)$ J / (kg*grade) - specific heat capacity of stainless steel.

To calculate derivative $\partial T(X_o,\tau)/\partial \tau$ the interpolation of the obtained experimental data has been performed at the nodes of uniform grid by means of the second-order smoothing polynomial defined at the six nodes of the initial non-uniform grid and positioned on both sides of the node under consideration. It was calculated by the temperature values on a new grid as a derivative of the function containing random errors

$$\frac{\partial T}{\partial \tau} = \frac{1}{10\Delta\tau}\left(-2T_{j-2} - T_{j-1} + T_{j+1} + 2T_{j-2}\right) \tag{9}$$

The heat flux on the specimens' back surfaces $q_1(\tau)$, $0 \le \tau \le \tau_n$ at instants of time τ_i was defined by means of thin copper calorimeters of thickness δ, with density ρ and heat capacity c.

$$q_1(\tau) = \rho * \delta * c * \frac{\partial T(X_1,\tau)}{\partial \tau} \tag{10}$$

where $\rho = 8930$ kg/m^3 - copper density at 20 °C; $c = (389 + 0.042 \text{ x·}T)$ J/(kg*grade) - copper specific heat capacity at 20 °C; $\delta = 0.0003$m - calorimeter thickness.

The experimental data obtained from testing, presented in the previous subsection has been used to determine the thermal properties of the analyzed materials. For this, the results of the tests on a uniform time grid should be submitted as required by the software of inverse problem solution. Heat flux values on the specimen's surfaces must be also calculated as heat passes from the heater on the heating surface and penetrates into a copper calorimetric plate at the back surface. The analysis of experimental data has shown that the thin cooper calorimeter is not enough to estimate the heat flux at the back surface of the specimen during all time duration of experiment. (It can be used only during 300-400 initial seconds). To overcome this problem, the using of estimated heat flux into the thermal-insulated holder was suggested. (The thermal properties of thermal insulator are known to a very high accuracy). Therefore the back heat flux $q_1(\tau)$ was calculated using the temperature measurements on the surface of the thermal-insulated holder. The released heat flux on the back surface is determined by the sum of heat fluxes accumulated by the calorimetric plate - q_{cal} and penetrated into thermal-insulating slab - q_{ins}.

$$q_1(\tau) = q_{cal}(\tau) + q_{ins}(\tau) \tag{13}$$

where $q_{cal}(\tau)$ is calculated using (12) and $q_{ins}(\tau)$ (that penetrated into the insulation) – from a solution of the direct problem for the insulated slab of 30 mm – thickness with a temperature boundary condition on the heating surface defined by temperature on the copper calorimeter and by heat balance equation at the back (cold) surface of the insulator.

To solve the inverse problem in the presented version the software developed and presented above was used. In solving the inverse problems the initial values of unknown functions the carbon heat capacity dependence on temperature and the corresponded thermal conductivity was taken, see Table7.

Table 7: Initial values.

Temperature, °C	0	200	400	600	800	1000
Heat capacity J/(kg grade)	730	1172	1486	1717	1821	1905
Thermal conductivity, W/(m grade)	0.06	0.09	0.12	0.15	0.19	0.30

Comparisons of the calculated and measured temperatures on the specimens' surfaces for materials ETTI-CF-ULT and ETTI-CF-ERG are presented in Figures 10 and 11. The result of estimating the functions $\lambda(T)$ and $c(T)$ for material ETTI-CF-ULT and ETTI-CF-ERG are presented in Figures 12 and 13.
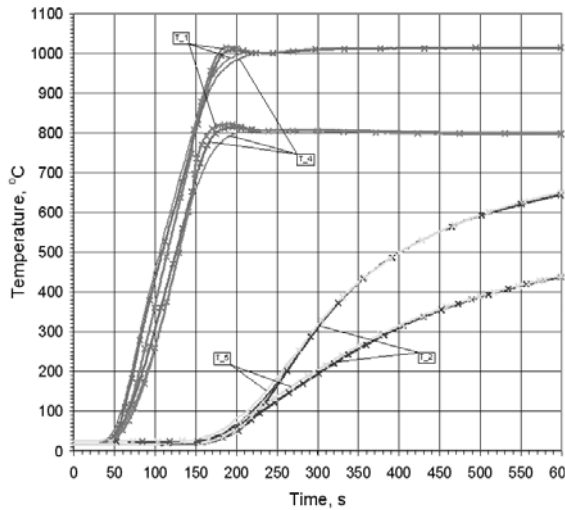
Figures 10. Comparing the calculated and measured temperatures for material ETTI-CF-ULT
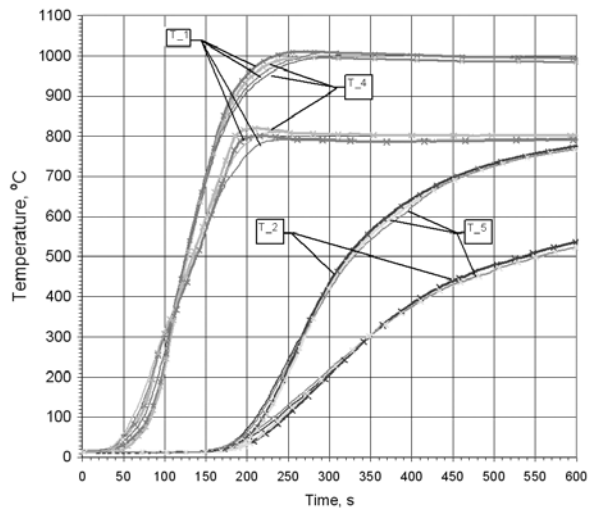
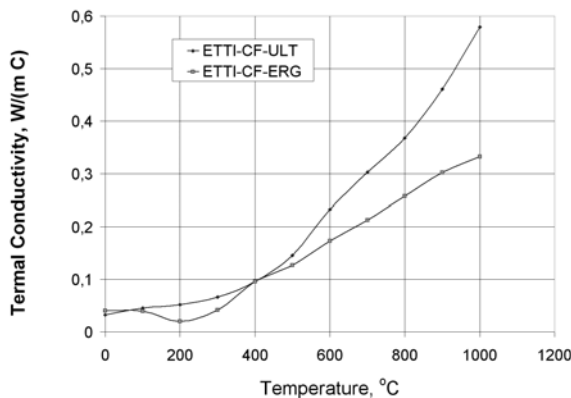Figures 11. Comparing the calculated and measured temperatures for material ETTI-CF-ERG

Figure 12. Estimated value of the thermal conductivity for material ETTI-CF-ERG and ETTI-CF-ULT.
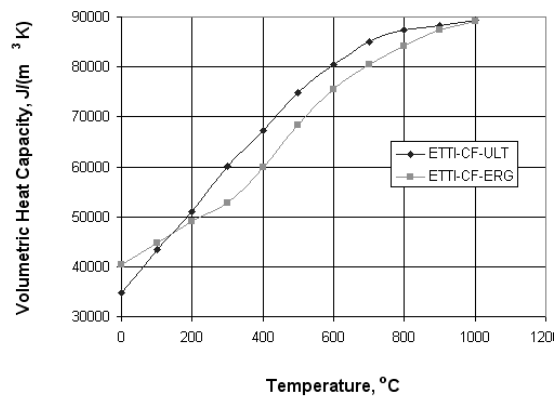

Figure 13. Estimated value of the volumetric heat capacity for material ETTI-CF-ERG and ETTI-CF-ULT.

Table 8 includes the obtained values of the least squares and the maximum deviation of the calculated temperatures from that measured in the experiments.

Table 8: The deviation of the calculated temperatures.

Material	Least-squares temperature deviation (K)	Maximum temperature deviation (K)
ETTI-CF-ULT	5.64	17.5
ETTI-CF-ERG	11.35	32.8

5. CONCLUSIONS

The executed thermal testing provided the solving of the above formulated problems to study the heat transfer in the specimens and to estimate the thermal properties of the lightweight porous materials ETTI-CF-ULT and ETTI-CF-ERG.

The operating opportunity and effectiveness of the developed methodology for preparing and carrying out the thermal testing of high porous, electrical conductive thermal insulating materials on the high temperature vacuum stand TVS-1 was approved (including the technology of specimen and thermosensors manufacture and installation).

The operating opportunity and effectiveness of developed hardware for the thermal testing of the above-mentioned materials was approved (including experimental module EM-2 and automatic system for heating, control, experimental data collection and processing).

The deviations of the calculated temperatures (using thermal property estimations) from the temperature measured in the experiments are insignificant showing sufficient accuracy in the estimations of thermal properties of analyzed materials.

The main point of discussion is the using of the one-dimensional mathematical model of heat transfer. The very low value of the thermal conductivity of analyzed materials did not agree with the assumption of thermal-insulating boundary conditions at the left and right sides of the specimen. Therefore the next stage is to considered a three-dimensional mathematical model of heat transfer for materials with similar properties.

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