REVIEW

THERMOPHYSICAL INSTRUMENTS

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Thermophysical measurements are typified by an enormous multivariety of techniques and, as a rule, relatively low precision. This state of affairs can be traced to many objective causes, particularly to the fact that, unlike other areas of measurement engineering, almost all thermophysical investigations are carried out by means of individually constructed laboratory facilities. No exception is to be found even in the most tried and true problem areas, where the measurement objectives are purely technical and conceptual difficulties no longer arise.

Another characteristic aspect of thermophysical measurements is the length and breadth of research occasioned by the burgeoning interest of scientific and industrial organizations over the last twenty years in the thermophysical properties of materials. Their interest has been stimulated primarily by the growing severity of the operating regimes and conditions of objects in terms of temperature and pressure ranges, heat-flux densities, and other factors, as well as the discovery and application of a host of new materials in engineering. In chemistry alone there are now reckoned to be approximately two and a half million substances and about a hundred thousand new ones are emerging annually [2]; very often the potential applications of these materials are determined precisely by their thermophysical properties.

The indicated trends in the evolution of science and engineering appear very likely to continue for the next ten or fifteen years [1]. Naturally, in the face of such a plethora of new materials and diversity of experimental and theoretical problems individualized laboratory facilities cannot be expected to ensure the necessary volume of information. The major problem here must be solved by a broad-based diversified approach, including: 1) the organization of industrial manufacture of various types of thermophysical instruments; 2) the development of theoretical research in those branches of physics in which it is possible, quantitatively as well as qualitatively, to predict with satisfactory accuracy the physical properties of both individual substances and all their possible chemical compounds, mixtures, composite materials, etc.; 3) the organization of an appropriate metrological service under the aegis of the State Standards Committee of the USSR (Gosstandart) with responsibility for testing equipment, standard specimens, and the attestation of pure and technologically stable materials.

The present review only grazes the surface of the problem and is limited to instruments for the measurement of thermal conductivity, specific heat, and thermal diffusivity.

The need for the industrial manufacture of thermophysical instruments has been emphatically reiterated at various thermophysical conferences over the last fifteen years. The following articles were adopted, in particular, in the resolutions of the Fourth All-Union Conference on Heat and Mass Transfer (Minsk, 1972) [21].

1. Work shall be continued in the development of theoretical and experimental methods for the investigation of heat and mass transfer with the extensive use of modern computer engineering and contactless measurement techniques, as well as in the development and application of complex automated data-gathering and processing systems to be used in experimental research.

2. Redoubled effort shall be spent in seeking the most general, most reliable, and fastest methods for determining the thermophysical, mass-transfer, rheological, electrical, mag-

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netic, and other properties of materials under diverse conditions. It is deemed in the best interest to unify those methods and to ensure regular production of the necessary instruments.

The resolutions of conferences and the activities of the specific Council on Heat and Mass Transfer in Technological Processes of the State Science and Technology Committee have had a very positive effect. Work has commenced in a number of organizations throughout the Soviet Union on the design of thermophysical instruments, some of which have begun to be manufactured in small commercial lots, while other individual prototypes have already passed metrological testing in various agencies of Gosstandart.

Realistic proposals have been drawn up at the present time for organizing the industrial manufacture of thermophysical instruments designed for both engineering and scientific-research applications. These proposals were motivated by the founding in 1972 of the State Special Design Bureau for Thermophysical Instrumentation (GSKB TFP) and the special training of engineers and thermophysicists with instrument-design orientation at the Leningrad Institute of Precision Mechanics and Optics (LITMO).

The foregoing events and considerations have necessitated a state-of-the-art study of work in the field of thermophysical instrumentation.

FUNDAMENTAL REQUIREMENTS FOR THERMOPHYSICAL INSTRUMENTATION IN ENGINEERING APPLICATIONS

The information presented below was acquired through a survey of the literature and visits to facilities with concrete developments. Before this kind of study can be undertaken, however, it is necessary to develop certain general requirements for thermophysical instrumentation designed for engineering applications, to define the concept of "instrument," and to formulate general criteria by which an instrument is to be distinguished from experimental apparatus used for scientific research.

The All-Union State Standard GOST 16263-70 classifies measuring instruments as measurement means designed for the acquisition of measurement information about a quantity to be measured, in a suitably perceptible form. This definition, of course, applies equally to instruments for engineering applications ("technical" instruments) and to laboratory apparatus and, unfortunately, lacks specificity.

In our opinion, an instrument must differ from laboratory apparatus, not only in terms of assiduous theoretical, methodological, and experimental refinement of the measurement technique, but also in its more sophisticated construction, enhanced reliability, higher efficiency, and greater simplicity and ease of operation. An instrument must have clear-cut limits of applicability with respect to temperature, pressure, and various external influences. The principal measurement error must be a function of the instrument and not of the experimenter.

The specification of these requirements eliminates from the analysis a vast group of assorted laboratory apparatuses stocked by the leading thermophysical laboratories of the country. These apparatuses are designed for scientific research; many of them are charcterized by universality and high precision and with appropriate modification could be reclassified as instruments.

Domestic Soviet instruments complying with the majority of the above-stated requirements are listed in Table 1. All of the instruments included in the table can be classified into two groups: 1) room-temperature instruments; 2) instruments for test in a wide temperature range.

ROOM-TEMPERATURE INSTRUMENTS

The great majority of thermophysical investigations are conducted at room temperature. It is only natural, therefore, that the greatest number of instrument designs should be concentrated here. By far the bulk of the research has been applied to be group of thermal insulating materials (solid, particulate, and fibrous) with thermal conductivities ranging from 0.1 to 2.0 W/m°K; semiconductors and metals tend to receive much less attention.

The operating principles of the given group of instruments are based on extremely diverse thermal testing regimes: steady-state; regular regime of the first kind; initial stage of the thermal process; and quasisteady-state.

TABLE 1. Instruments Developed in the USSR

Instrument type and function	Measurement range	Specimen shape and size, mm	Temperature range; error	Measurement technique; developer
1. Instrument for combined measurement of λ, a, and c of liquids and particulate solids	λ = 0.1-1.5 W/m°K	Disk ϕ50, H = 1-6	20°C, 3-5%	Initial stage of thermal process (constant-temperature source); Lensovet LTI
2. Instrument for measuring λ of solids	λ = 0.2-100 W/m°K	Plate 10 × 10, H = 1-10	−100 to 20°C, 3-5%	Regular regime of first kind; AFI
3. Lambda instrument for measuring λ of solids	λ = 0.15-1.5 W/m°K	Disk ϕ 15, H = 4	20°C, 7-8%	Steady-state, comparative; VNIIM
4. Set of four RK-20 instruments	λ = 0.1-100 W/m°K, 200 kg/m³	Disk ϕ10, H = 20	20°C, 3-5%	Regular regime of first kind; LITMO
5. Instrument for measuring λ of solids	λ = 0.2-100 W/m°K	Plate 10 × 10, H = 1-10	−195 to 20°C, 3-5%	Regular regime of first kind; AFI
6. TP-5-66 instrument for measuring λ of construction materials	λ = 0.03-1.5 W/m°K	Two plates 100 × 100, H = 50	20°C, 10%	Initial stage; VNIISMI
7. Instrument for measuring thermophysical properties of construction materials	λ = 0.03-1.5 W/m°K	100 × 100 × 100	20°C, 3-5%	Initial stage, cylindrical probe; LISI
8. TMP-1 instrument	λ > 0.1 W/m°K	ϕ50 or more	20°C, 10%	Constant power source with standard; Sevastopol Instrument-Construction Institute
9. Instrument for measuring thermal activity of rocks	b = 28-2800 J/m³·°K)	Bulk	−20 to 40°C, 10%	Initial stage; NIISMI (Kiev)
10. BP-66 instrument for measuring λ of construction materials	λ = 0.2-2 W/m°K	Plate ϕ160, H = 10-40	20°C, 6%	Regular regime of first kind; LITMO
11. Instrument for combined measurement of λ, c, and a of solids	λ = 0.2-5 W/m°K	Two disks ϕ 40, H = 5-20	50°C, 5-7%	Monotonic warmup; VNIIFTRI
12. Instrument for measuring thermal conductivity	λ = 0.2-5 W/m°K	Plate 70 × 70, H = 1-10	20-100°C, 5%	Steady-state; ITTF AN UkrSSR
13. Instrument for measuring λ of fabrics	λ = 0.01-5 W/m°K	Disk ϕ50, H = 1-6	20°C, 3%	Regular regime of first kind; LITMO
14. Instrument for measuring λ of solids	λ = 0.2-200 W/m°K	Plate 10 × 10, H = 1-10	20-400°C, 3-7%	Monotonic heating plus regular regime of first kind; AFI
15. Instrument for measuring λ of thermal insulators	λ = 0.2-5 W/m°K	Two plates 100 × 100, H = 5	20-600°C, 5-8%	Quasisteady-state heating; TIKhM
16. DK-aλ-400 dynamic calorimeter	λ = 0.1-5 W/m°K	Disk ϕ15, H = 0.5-5	−80 to 400°C, 3-5%	Monotonic heating; LITMO
17. DK-aλ-400 dynamic calorimeter	λ = 5-80 W/m°K	Cylinder ϕ15, H = 30	−80 to 400°C, 3-5%	Monotonic heating; LITMO
18. UDK-acλ-400 dynamic calorimeter	λ = 0.1-5 W/m°K	Disk ϕ15, H = 1-6	−150 to 400°C 3-5%	Monotonic heating; LITMO
19. DK-ac-900 dynamic calorimeter	λ = 5-40 W/m°K	Cylinder ϕ25, H = 40	50-900°C, 5-8%	Monotonic heating; LITMO
20. KDM-ac-900 dynamic calorimeter	λ = 15-150 W/m°K	Cylinder ϕ20, H = 30	50-900°C, 5-7%	Monotonic heating; LITMO
21. Instrument for measuring c of solids	λ > 5 W/m°K	Cylinder ϕ15, H = 15	50-950°C, 1-3%	Dynamic pulsed heating; LITMO
22. Apparatus for combined analysis of λ, a, and c of thermal insulators	λ = 0.1-2 W/m°K	ϕ40, H = 120 or 70 × 70 × 10	4-400K, 5-8%	Quasisteady-state regime; ITMO AN BSSR
23. Apparatus for thermophysical measurements	λ = 0.1-5 W/m°K	—	4-400K, 1-3%	Steady-state, adiabatic, pulsed; VNIIFTRI-Khabarovsk
24. Instrument for measuring c of materials	—	ϕ30, H = 60	20-700°C, 10%	Continuous adiabatic warmup; Krasnodar Polytech. Inst.
25. Instrument for measuring λ of polymers	λ = 0.1-2 W/m°K	Two samples ϕ50, l = 2-25	40-250°C, 3%	Steady-state twin-plate scheme; IONKh AN SSSR

An instrument has been developed at the Lensovet Leningrad Technological Institute (LTI) for the combined measurement of thermal conductivity λ, thermal diffusivity a, and specific heat c. It operates on the principle of the initial stage of the thermal process [20]. The test specimen is placed in contact with a standard body, and both are heated by a constant-temperature source. The time variation of the temperature at the interface between the bodies is measured with a differential thermocouple and a mirror galvanometer. The instrument is a compact table model and can be used for the investigation of solid, liquid, and particulate materials with thermal conductivities λ = 0.1 to 1.5 W/m°K. Liquids and particulate solids are placed in a small container. The specimens have a diameter of 50 mm and thickness of 2 to 6 mm. The measurement error is 3 to 5% (all instrument errors are quoted from the authors' estimates). The heat source is a metal plate, whose temperature is automatically stabilized by a special mechanism. The time intervals are automatically recorded by means of photoresistances mounted on the galvanometer scale. The calculations are minimized through the use of special tables.

An instrument for investigating the thermal conductivity of solid thermal insulation materials and semiconductors with λ = 0.2 to 100 W/m°K was developed in the early sixties at the Agrophysics Institute (AFI) [4]. The instrument operates on the double-calorimeter principle in the regular regime of the first kind. The tests are conducted on rods with a cross section of approximately 2 cm^2 and heights of 1 to 10 mm. The specimen is set up inside a large metal chamber, which is thermostatically regulated by circulating water, and a heated metal rod with a known heat capacity (standard) is placed on the specimen. The standard rod is cooled mainly through the specimen. The heat lost by the standard through the air space to the chamber walls is corrected experimentally. The regular cooling rate is recorded by means of an ÉPP-09 multipoint electronic potentiometer. The measurement error is 3 to 5%. Of course, for materials with $\lambda > 10$ W/m°K such figures elicit serious doubts.

The D. I. Mendeleev All-Union Scientific-Research Institute of Metrology (VNIIM) has developed the lambda comparative steady-state instrument for measuring the thermal conductivity of solid materials with λ = 0.15 to 1.5 W/m°K [5]. The investigated specimens are disks with a diameter of 10 mm and thickness of 4 mm. The instrument scale is read directly in units of thermal conductivity. Standard substances are used for calibration: fused quartz and polymethyl methacrylate (PMMA). The instrumental error was determined at 7-8%. The instrument has a compact reference apparatus equipped for technical documents. At present this instrument is being readied by GSKB TEP for commercial production.

A set of instruments has been developed at the Leningrad Institute of Precision Mechanics and Optics (LITMO) for investigating the thermophysical characteristics λ, a, and c of solid, particulate, and liquid materials in small specimens [6]. All the instruments operate in the regular regime of the first kind, are distinguished by the simplicity of their measurement circuits, and incorporate a modular construction. Each instrument configuration includes a device for heating of the specimens, a mirror galvanometer, and a timer.

For measurements of the thermal diffusivity of samples having a thickness of 5 to 10 mm and diameter of 20 mm (with a hole for a thermocouple at the mid-thickness plane) the end surfaces are placed in contact with two metal slabs, which serve as a constant-temperature reservoir. A sheathed thermocouple placed in the hole measures the regular cooling rate of the sample. The heat transfer between the side surface of the specimen and the slabs does not exceed 7% and is corrected analytically. The thermal diffusivity measurement error for samples with λ = 0.1 to 5 W/m°K lies between 3 and 5%.

For the measurement of thermal conductivity with the same instrument a set of three disks is placed between the metal slabs; the metal middle disk has a known heat capacity, and the two equal-thickness outer disks are the specimens. The cooling rate of the middle disk is determined by the thermal resistance of the specimens. The specimens are made with thicknesses between 1 and 3 mm. The measurement error for materials with $\lambda > 0.1$ is 3 to 5%.

Finally, the same instrument can be used to measure the heat capacity of solid materials with $\lambda > 0.2$ W/m°K with samples in the shape of a disk of the same diameter and thicknesses of 3 to 5 mm. The regular cooling rate of the specimen is measured with a thermocouple placed in the hole. The tested disk is placed inside the cavity formed by the metal slabs, on three slender thermally nonconducting pins, and are cooled through the surrounding air space. The heat-transfer coefficient of the air space is determined from a calibration test on a known specimen. For the investigation of materials with $\gamma > 500$ kg/m^3 the heat capacity measurement error is not greater than 3 to 5%.

Another instrument, the RK-c-20, operates on an analogous scheme with a cylindrical specimen having a diameter of 10 mm and height of 30 mm. The specimen is jammed onto a needle with a thermocouple and is cooled in a cylindrical cavity in a large metal slab. For the investigation of particulate, fibrous, and liquid media the latter are placed in a vessel. The method is relative, and the instrument constant is determined in an experiment with a copper specimen. For the investigation of materials with $\gamma > 500$ kg/m^3 the measurement error is 2 to 3%. The instrument can also be used to analyze lightweight materials (fabrics and highly porous powders) with $\gamma \geqslant 200$ kg/m^3, but the attendant error increases to 7 to 8%.

The RK-ac-20 instrument is designed for investigations of the thermal diffusivity and heat capacity of metals and dense oxides. A cylindrical specimen with a diameter of 15 mm and height of 30 mm and having two side holes for thermocouples is placed inside a metal slab, which functions as a constant-temperature reservoir. The preheated specimen is set with its end on a thin insulation layer, through which it is cooled. The side heat transfer through the exposed side surface does not exceed 3% and is corrected analytically. In the actual experiment the cooling rate of the specimen and the temperature lag at two points along its height are measured. The measurement error for a and c in materials with a = (4 to 20)$\cdot 10^{-6}$ m^2/sec is 3 to 5%.

All three instruments passed metrological testing in 1971.

The All-Union Scientific-Research Institute of Physicotechnical and Radio-Engineering Measurements (VNIIFTRI) has developed an instrument for the combined measurement of the characteristics λ, a, and c in a single specimen. The tests are conducted in the monotonic warm-up regime [7]. A rod-shaped specimen with a diameter of 40 mm and height of 10 to 40 mm is placed in a metal slab and warmed up together with the latter by means of a constant-power heater. Temperature measurements are performed with three thermocouples. Two of them are situated on the end-on metal contact plates, and the third in the mid-thickness plane of the specimen. The temperature lag of the midpoint of the specimen relative to outermost points and the heat flux from the ends of the specimen are measured by means of heat-flowmeters. All signals are recorded by a mirror galvanometer, which is built into the common control panel. For materials with λ = 0.2 to 5 W/m°K the error is 5 to 7%.

A distinguishing aspect of the foregoing instruments is the small dimensions of the test specimens. The instruments are used to investigate precious materials, which are not obtainable in large quantities, as well as materials having a homogeneous structure (plastics, semiconductors, glasses, dense ceramics, and metals).

The instruments used to investigate structural and thermal insulation materials are designed for work with large specimens.

A representative instrument is the BP-66 twin-plate calorimeter developed by LITMO [8]. It operates in the regular regime of the first kind and can be used to measure the thermal conductivity of solid and particulate materials with λ = 0.2 to 2 W/m°K. The tests require two identical specimens with a diameter of 160 mm and thicknesses of 10 to 40 mm. The regular cooling rate is recorded by means of a mirror galvanometer and timer. The instrument has a compact construction. Thermostatic regulation of the jacket requires a liquid thermostat. The measurement error is 6%.

Two instruments for measurements of the thermophysical characteristics of structural materials have been developed by the Kiev Scientific-Research Institute of Structural Materials and Products (NIISMI) [9]. One of them, the TP-5-66, is designed to measure the thermal conductivity of materials with λ = 0.03 to 2.5 W/m°K. The instrument operates in the initial stage of the thermal process. A constant-power linear heater made of manganese wire 0.15 mm in diameter with a copper resistance thermometer wound on it is placed between two plates with dimensions $100 \times 100 \times 50$ mm. An EPL electronic bridge automatically sets the power and pulsewidth and measures the heater temperature. The instrument scale is calibrated in values of λ. The measurement error is 10%.

The second instrument (TP-1-63) is designed for measurements of the thermal activity b of rock. The basis for the determination of the coefficient b is the solution of the problem of the temperature distribution in two abutting semi-infinite rods with a plane heat source between them. Materials with b = 28 to 2800 J(m$^3\cdot$sec$\cdot$°K)$^{-1/2}$ are investigated. The estimated measurement error is 10%. The instrument is portable and compact, having a weight of 4.5 kg.

The Sevastopol Instrument-Construction Institute and the Dnepropetrovsk Mining Institute have collaborated in the development of several instruments for determining the thermophysical characteristics of rock under field conditions [24]. They differ from the two preceding instruments only in their measurement circuits. One of them uses pneumatic servo elements, while the other has a circuit geared for the measurement of temperature by means of semiconductor thermistors. The quantity measured directly in testing is the thermal activity, and the thermal conductivity is calculated from the known specific heat at constant volume. In one study the authors have extended the domain of application of the TPM-1 instrument to the investigation of the thermal and electrical conductivities of metals. However, the basis for this modification is not too sound. In their published papers [24] the authors do not analyze the errors or limits of applicability of the instruments.

It is appropriate to conclude the survey of room-temperature instruments with a description of an instrument developed by the Leningrad Structural Engineering Institute (LISI). They have engineered two modifications of a thermal conductivity instrument based on the probe method. One of them is designed for laboratory work, and the second for field operations. The sensor is a probe in the shape of a needle 2 to 3 mm in diameter with a length of 10 to 90 mm. Inside the needle is wound a helical heating coil and semiconductor thermistor, which is connected into a bridge-type measurement circuit. The signal is recorded by an ÉPPV-26 potentiometer in the laboratory version and by a dial-reading instrument in the field version. In the field measurements the probe is inserted into the investigated block of material. The laboratory measurements are conducted on bulk specimens with an opening approximately 90 mm deep for the needle probe. The measurement time does not exceed a few minutes. Special tables have been compiled to simplify data processing. The instrument is used extensively in construction organizations.

WIDE-TEMPERATURE-RANGE INSTRUMENTS

The overwhelming majority of instruments in the second group operate in the quasisteady-state and monotonic regimes, whereby the temperature dependence of the investigated parameters is obtained in a single experiment. Two exceptions are the instruments developed by the Institute of Technical Thermophysics of the Academy of Sciences of the Ukrainian SSR (ITTF AN UkrSSR) [10] and the Institute of General and Inorganic Chemistry (IONKh AN SSSR) [25] for the determination of the thermal conductivity λ. The first of these instruments operates in the steady state and is designed for the investigation of solid, liquid, and gaseous substances with $\lambda = 0.02$ to $5\ W/m°K$ in the temperature interval from 20 to 100°C under atmospheric conditions. It has the unique capability of measuring the heat flux through a specimen by means of plate heat-flowmeter situated in the central zone on the cold side of the specimen. The use of a heat-flowmeter greatly simplifies the experimental technique and eliminates the usual guard accessories. The recording device is a PPTV potentiometer. The instrument comes as a table model. The specimens are prepared in the form of plates with a cross section of 70×70 mm and a thickness of 1 to 10 mm. The time to settle into the steady state is about one hour. The measurement error is approximately 5%.

The instrument designed by IONKh AN SSSR also operates in the steady state and is designed for measurements of the thermal conductivity of polymers and other materials with relatively low values of the latter: 0.1 to 2.0 W/m°K, in the temperature interval from −40 to 250°C. Testing requires two identical specimens 50 mm in diameter with a thickness of 2 to 25 mm. The measurement error is about 3%.

An apparatus has been designed at the Tambov Institute of Chemical Machinery (TIKhM) for measuring the thermal diffusivity of material with $\lambda = 0.2$ to $5\ W/m°K$ in the temperature interval from 20 to 600°C [11]. The tests are conducted in an evacuated medium in the linear warmup regime. Two plate specimens with dimensions $100 \times 100 \times 5\ mm^3$ are used for the measurements. A linear warmup program is ensured at one point of the sample by means of a special automatic regulator. The apparatus as a whole is in the finished design stage, but its main component, the calorimetric device, has not been adequately developed insofar as the installation of the thermocouples is awkward and the interchange of specimens is complicated. An important feature of the apparatus is the automated computation and recording of the measured parameter. Lately TIKhM has placed considerable emphasis on the automation of thermophysical experiments. Systems of converters and ancillary equipment have been developed for interfacing thermophysical instruments with computers [26].

A set of instruments has been developed at LITMO for research in the temperature range from −100 to 900°C [27]. The tests are conducted in the monotonic warmup regime on small specimens. The calorimetric subsystems of all the instruments are made of reproducible metal parts and have a permanent configuration for the installation of thermocouples and thermometers.

One of the instruments in the set, the DK-$a\lambda$-400 dynamic $a\lambda$-calorimeter [12], is designed for measurements of the thermal conductivity and thermal diffusivity of solid materials with λ = 0.1 to 5 W/m°K in the temperature range from −100 to 400°C. The tests are conducted on specimens 15 mm in diameter. The specimen thickness is selected in the interval from 0.5 to 5 mm for thermal conductivity measurements and in the interval from 4 to 8 mm for thermal diffusivity measurements. The instrument is a table model and comprises two independent calorimeters (λ-calorimeter and a-calorimeter), a measurement control panel, and a power supply section. The instrument kit includes an M195/1 mirror galvanometer. The total time for an experiment plus data processing over the entire temperature range does not exceed two hours. The DK-$a\lambda$-400 instrument passed metrological testing in 1967.

The second instrument in the set, the DK-ac-400 dynamic calorimeter [13], is designed for the concurrent measurement in a single experiment of the thermal diffusivity and true specific heat of solid materials with λ = 5 to 80 W/m°K (metals, semiconductors, and alloys) in the temperature range from −100 to 400°C. The investigations are carried out with cylindrical specimens 15 mm in diameter with a thickness of 15 to 30 mm. Two radial holes 1 mm in diameter are drilled near the ends of the specimen for insertion of the thermocouples. A distinctive feature of the instrument is it capability of measuring the heat flux by means of a metal heat-flowmeter [14]. The instrument is a table model and consolidates the calorimeter, measurement panel, and supply components into a single unit housing. The instrument kit includes a PPTN-1 potentiometer and an M195/1 galvanometer.

The third LITMO instrument is the DK-ac-900 dynamic calorimeter [15], which is designed for measurements of the thermal diffusivity and true specific heat of materials with λ = 5 to 50 W/m°K in the temperature range from 50 to 900°C. The investigations are carried out in an inert medium with preliminary evacuation. The test specimens are cylinders 25 mm in diameter with a height of 40 to 50 mm and three axial holes drilled half-way through their height. It has a unique facility for measuring heat losses by means of radiation thermometers [16]. The only objects in the warmup zone are an instant tubular heater, the specimen, and three thermocouples. The instrument configuration comprises a vacuum chamber with built-in calorimeter, electrical power supply components, and inert-gas purge and fill lines. The instrument kit includes an ÉPP-09 electronic potentiometer [28].

In the period from 1969 through 1973 a new set of instruments was developed at LITMO for research in the temperature range from −150 to 900°C [29-30]. Like their predecessors, these instruments operate on the monotonic warmup principle and can be used to obtain the temperature dependence of the investigated parameters in a single experiment. All the instruments have a permanent thermocouple installation scheme and table-model construction.

The UDK-$ac\lambda$-400 instrument [29] makes it possible to investigate the thermal conductivity of materials with λ = 0.1 to 5 W/m°K in disk specimens 15 mm in diameter with a thickness of 0.5 to 5 mm, the specific heat of substances with $c\gamma > 10^6$ J/m$^3\cdot$°K in specimens 7 mm in diameter with a thickness of 25 mm, and the thermal diffusivity of materials with a = (0.1 to 1)$\cdot 10^{-6}$ m^2/sec in samples 15 mm in diameter with a thickness of 6 to 10 mm. Each parameter is investigated in its own calorimetric accessory, which differs from the others only in the construction of its components for measurement of the heat content. All other components of the instrument (measurement circuit, power supply system, water and liquid-nitrogen cooling systems) are common. The calorimeters are situated on a panel with a unified control system. The measurement errors do not exceed 3 to 5% over the entire temperature range for any of the three parameters. The measurements are conducted manually by means of a galvanometer and timer.

The KDM-ac-900 dynamic calorimeter [30] is used to investigate the specific heat and thermal diffusivity in the temperature range from 50 to 900°C for materials with λ = 15 to 150 W/m°K (alloys, carbides, borides, silicides, hydrides, and other intermetallic compounds). The measurements are conducted in an inert medium with preliminary evacuation. The test specimens are rods with a diameter of 20 mm and height of 30 mm. Two radial holes 1.5 mm in diameter are drilled into the specimen near the ends. The time for a single experiment, including purging and filling with the inert gas, does not exceed one hour, and the data-processing time is from one to one and a half hours. The measurements are conducted manually by means of a galvanometer, PPTV potentiometer, and timer. The error does not exceed 5 to 8%.

The basic construction of the KDM-c-900 dynamic pulsed calorimeter is the same as for the preceding instrument, differing only in the actual measuring device. The instrument is used to investigate the true specific heat of materials with $\lambda > 5$ W/m°K for cylindrical specimens 15 mm in diameter with a height of 15 mm. The specimens must be provided with two holes 2.6 and 1.6 mm in diameter for insertion of the thermocouple and heater. The measurements are conducted in the dynamic pulsed regime at an average rate of 0.1 °K/sec. The measurement period is about four hours, and data processing takes one and a half to two hours. The error is 1 to 3%. It is possible to investigate the specific heat near phase transitions.

In 1969-71 the Agrophysics Institute (AFI) of the V. I. Lenin All-Union Academy of Agricultural Sciences (VASKhNIL) in Leningrad developed two instruments for measurements of the thermal conductivity, electrical conductivity, and thermo-emf of semiconductors. One of the instruments spans the temperature range from −190 to 20°C, and the other from 20 to 400°C. The specimens are plates with a cross section of 10 × 10 mm and thickness of 1.0 to 10 mm. The experimental data are processed on a computer of the Promin' or BÉSM type during the measurement operations. The thermal conductivity measurement error is 3 to 7% by the authors' estimates. In our opinion, however, such an estimate appears highly optimistic for materials with a high thermal conductivity ($\lambda > 5$ W/m°K), because the thermocouples are not inserted into the specimen and the thermal resistance of the specimen turns out to be commensurate with the contact resistance.

An apparatus has been developed at the Institute of Heat and Mass Transfer of the Academy of Sciences of the Belorussian SSR (ITMO AN BSSR) for the combined measurement of the thermophysical properties of poor heat conductors with $\lambda = 0.1$ to 2 W/m°K in the temperature interval from 4 to 400°K. The investigations are carried out in the quasisteady state on cylindrical specimens 40 mm in diameter with a height of 120 mm and with several holes for the insertion of thermocouples and heaters. The apparatus is designed for individual specimen mounting. The duration of one experiment plus data processing is about forty hours. The duration of one experiment plus data processing is about forty hours. The measurement error is 5 to 8%.

The Khabarovsk Branch of the All-Union Scientific-Research Institute of Physicotechnical and Radio-Engineering Measurements (VNIIFTRI-Khabarovsk) has developed an apparatus for determining the specific heat and thermal conductivity of various substances in the same temperature range as the preceding instrument, 4 to 400°K. The specific heat is determined in the steady-state pulsed regime by means of an adiabatic calorimeter, and the thermal conductivity by the steady-state axial method. The measurement errors are 1 to 3%. The instrument design has been completed and passed metrological testing in 1970-72; it can also be used for the investigation of other thermophysical parameters.

Work has been done in recent years at the Georgian Polytechnic Institue on the design of an instrument for measuring the thermal and electrical conductivities of metals and alloys in the temperature range from 100 to 700°K. The investigations are carried out in vacuum on rod samples with cross sections of 8 to 32 mm^2 and a length of about 130 mm. The steady-state axial rod method with individual heater and system for adiabatic regulation of the side surface is used to determine the thermal conductivity, and the electrical conductivity is determined by a null method on the same sample. The test period is about eight hours in steps of 50°K each. The measurement error is 5% for the thermal and 2% for the electrical conductivity.

INSTRUMENTS OUTSIDE THE SOVIET UNION

The data presented below on the developments of non-Soviet companies are incomplete. They have been obtained from brochures of foreign exhibits, foreign manufacturers' catalogs, and journal articles. The available information is summarized in Table 2.

One of the earliest instruments was developed and manufactured commercially in the nineteen-fifties by Feutron in East Germany (GDR) for measuring the thermal conductivity of construction materials. The instrument operated on the steady-state twin-plate principle in application to specimens with dimensions 200 × 200 × 25 mm. The hot-face temperature did not exceed 100°C in testing. The measurement error was 5%. In 1958-1960 the Soviet Union purchased a quantity of these instruments, some of which are still being used in various construction organizations. Their main shortcoming is a rather excessive testing time. A single measurement requires four or five hours.

TABLE 2. Non-Soviet Instruments

Instrument type and function	Specimen size; measured parameter	Temperature range; error	Measurement principle; company, country
1. Thermal conductivity thermal instruments (thermal comparator)	$\lambda > 0.2$ W/m°K	20°C, 1% reproducibility	Quick-test method; Kubelik Karlsruhe, TRAC, GFR
2. TC-1 instrument for measuring λ	Disk ϕ = 65 mm, H = 10 mm, λ = 0.1-5 W/m°K	20°C, 5%	Steady-state; Stanton Instruments Ltd., England
3. ADL-6 instrument for measurement in powders, solids and porous materials	$\lambda < 2$ W/m°K	20°C, 5%	Steady-states; USA
4. Instrument for measuring λ of construction materials	200 × 200 × 25 mm^3, λ = 0.1-5 W/m°K	(20-100)°C, 5%	Steady-state plate method; Feutron, GDR
5. DSK-1500 calorimeter for for measuring c	Specimen volume 1.7 cm^3	20-1500°C	Continuous heating at rates to 0.14 °K/sec; Setaram, France
6. CRMT calorimeter for measuring c	15 and 100 cm^3 cells	20-80°C	Setaram, France
7. Model SH-2B apparatus for measuring c	ϕ18 mm, H = 32 mm	20-1000°C, 1%	Sykes continuous heating principle; Shimadzu, Japan
8. Thermotest instrument for measuring λ of liquids	Layer thickness 0.01 m for 0.5°K drop, λ = 0.1-1 W/m°K		Steady-state plate method, Brno, Czechoslovakia
9. HC-21 instrument for measuring λ	λ = 0.2-10 W/m°K	20-800°C, 7%	Principle undisclosed; Eiko, Japan
10. DSK-1B differential scanning calorimeter	ϕ10 mm, H = 2-6 mm	−100-500°C	Continuous warmup at 0.625-80 °K/min; Perkin-Elmer, USA
11. TC-200 instrument for measuring λ, model ZM	ϕ25mm, H = 25 mm or cube 25 × 25 × 25 mm^3, $\lambda > 0.2$ W/m°K	25-1200°C, 5%	Steady-state, comparative; Dynatech, USA
12. Model QTA No. 7 quantitative adiabatic calorimeter for measurement of c and differential thermal analysis	25-100 g "c"	−190-400°C, 2-5%	Continuous adiabatic heating; Dynatech, USA
13. Model TCFCM instrument for measuring λ	$\lambda > 0.2$ W/m°K	−190-1100°C, 5-10%	Dynatech, USA
14. Models TCAGM-10 and TCAGM-20 instruments for measuring λ	ϕ9-12 mm, l = 125 mm, $\lambda > 10$ W/m°K	−185-510°C −185-1100°C 2-5%	Steady-state axial, with autonomous heater; Dynatech, USA
15. Models TCFGM No. 4 and No. 18 instruments for measuring λ of heat insulators	2 specimens ϕ200 mm, l = 6-30 mm, λ = 0.02-2W/(m°K)	−185-200°C −185-1000°C 2-5%	Steady-state plate method; Dynatech, USA
16. Series TCHM instrument for measuring λ (high-speed λ-tester)	ϕ100 mm, λ = 0.02-2 W/m°K	−20-200°C, 5%	Steady state, with automatic regulation; Dynatech, USA
17. Mixing calorimeter for measuring c, Models SHDW No. 20, No. 30; R20 No. 20; R30 No. 30	25-100 g "c"	−150-150°C, 150-1100°C, 150-1700°, 3-5%	Mixing principle; Dynatech, USA

An instrument called a thermal comparator is described in one of the journals [31]. It is used for rapid thermal conductivity assessments of a wide variety of materials ranging from metals to polymer films. Its outstanding attribute is its speed; no more than a few minutes are required to complete a measurement, which is done without calorimetric measurements or the insertion of heat sensors in the specimen. The instrument comprises a calorimeter, a reading or recording section, and a set of six standard specimens. The reproducibility is good to at least 1%. The article does not state the specimen dimensions or the measurement principle. An accompanying photograph indicates that the instrument has passed the final design stage.

Another journal article [33] describes the improved ADL-6 guarded-cold-plate thermal conductivity apparatus, which is used for low-thermal-conductivity powdered, porous, particulate, and solid heat-insulation materials. The measurements are conducted near room temperature with 5% error.

Stanton Instruments Ltd. manufactures the TC-1 instrument [32] for measuring the thermal conductivity of polymers, rubbers, glasses, and insulation and construction materials. The measurement error is 5%, and the testing regime is steady-state. The specimens are 65 mm in diameter and up to 10 mm thick.

The product line of the well-known Setaram firm in France includes two instruments used to investigate the true specific heat of materials over a wide range of temperatures. The DSK-1500°C calorimeter measures the specific heat at temperatures from 20 to 1500°C in the continuous warmup regime at rates up to 0.14 °K/sec. The volume of the test specimen is 1.7 cm^3. The second instrument is the model CRMT with 15 and 100 cm^3 cells, which can be used to determine the specific heat in the temperature interval from 20 to 80°C. The instruments are of excellent design quality and come furnished with a control and recording instrument package, which makes it possible to vary the warmup rate considerably.

The Chepos firm in the city of Brno, Czechoslovakia, manufactures the Thermotest apparatus for measuring the thermal conductivity of liquids. This apparatus, which operates on a comparative steady-state plane-layer principle, is capable of measuring the λ of liquids in the temperature range from −10 to 100°C with a temperature differential of 0.5°K across a working layer 10 mm thick. The standard of comparison is distilled water. The hot- and cold-face temperatures of the specimen are maintained by means of a system of liquid thermostats.

At an instrument exhibition held in 1968 the Japanese firm, Eiko Seiki Sanyo, demonstrated the HC-21 instrument for measuring the thermal conductivity of ceramics, glasses, and polymers in the temperature range from 20 to 800°C. The measurement error is 7%. The measurement principle was not disclosed.

The catalog of the Japanese firm, Shimadzu Corp., which specializes in equipment for differential thermal analysis, lists the SH-2B instrument designed for measurement of the true specific heat of materials in the temperature range from 20 to 1000°C. The instrument operates on the familiar principle of continuous adiabatic warmup. The test specimen, which is 18 mm in diameter and 32 mm thick, is warmed by an internal heater under adiabatic conditions established by means of a double adiabatic-envelope system. The estimated measurement error is 1%. The measurements are conducted in vacuum at a pressure of 10^{-2} Pa. The instrument is made in two sections. One houses the instrumentation and regulating apparatus, and the other contains the actual measuring device, evacuation system, and control system.

The Perkin-Elmer Corporation in the United States specializes in the design and manufacture of instruments for thermal and thermogravimetric analysis, dilatometry, etc. One of their instruments is the DSK-1B differential scanning calorimeter, which measures the true specific heat of substances in the temperature interval from −100 to 500°C. It operates on a comparative continuous warmup principle at rates of 0.01 to 1.5 °K/sec. The specimens are fairly small: 10 mm in diameter and 2 to 6 mm thick. The instrument is characterized by simplicity of design and ease of servicing. One version of the instrument can be used to record directly the dependence of the specific heat on the temperature of the test specimen.

A set of instruments for thermal conductivity and specific heat measurements has been developed by Dynatech Corporation in the United States. They are distinguished by the liberal use of comparative steady-state methods. In addition to supplying equipment, Dynatech also furnishes a set of standard specimens spanning a wide range of thermal conductivities, from 0.02 to 200 W/m°K. The development of instruments for the measurement of thermophysical properties — thermal conductivity and specific heat in particular — is the responsibility of a special in-house facility, the Thermatest Department. The Dynatech TC-200 instrument (model 3M) can be used to determine the thermal conductivity of metals, ceramics, plastics, glasses, and rubbers with values from 0.2 to 220 W/m°K in the temperature interval from 25 to 1200°C. The test specimens are cylinders 25 mm in diameter and 25 mm in height or cubes 25 mm on a side. The measurements are conducted in vacuum or in any inert medium. The measurement principle is based on the steady-state axial method using a standard specimen. The test specimen is placed between two standards. A system of seven guard heaters maintains side adiabaticity of the specimen. The test regime is stabilized by means of an automatic control system. The measurement error is 5%. The instrument is furnished with six standard specimens covering the entire range of measurable values of λ.

The Dynatech model TCFCM instruments are designed for large-scale thermal conductivity measurements. The first instrument operates in the temperature range from −180 to 250°C with 5% error, and the second in the range from −180 to 1100°C with 10% error. The test materials range from plastics to metals. The instruments are equipped with a precision temperature regulating and automatic monitoring system.

The models TCAGM-10 and 20 instruments are designed for measurements of the thermal conductivity of metals with $\lambda > 10$ W/m°K. They operate in the steady-state axial mode with an individual heater. The specimens are rods 9 to 12 mm in diameter and 125 m long; they are pressed between a hot slab and a cold slab. The general temperature level is set by means of a space heater. Model No. 10 was developed for the temperature interval from −185 to 510°C with 2% measurement error, and model No. 20 for temperatures from −185 to 1100°C with 5% error. The investigations are carried out in vacuum or any inert medium. The instruments are equipped with automation, evacuation (purging), and precision measurement systems.

For investigations of the thermal conductivity of construction and other thermal insulation materials Dynatech has developed two groups of model TCFGM instruments (No. 4 for temperatures from −185 to 200°C and No. 18 from −185 to 1000°C) and the TCHM series (λ-tester for the interval from −20 to 200°C). The first model is designed for tests on two specimens 200 mm in diameter and 6 to 30 mm thick with λ = 0.02 to 2 W/m°K, and the second model (TCHM series) for a sample 100 mm in diameter and 5 to 15 mm thick. The errors are 2 to 5%. A distinctive feature of the instruments is their facility for rapid specimen interchange. The specimen thickness is measured directly in the instrument. The TCHM series is particularly fast in testing; two measurements per hour are possible.

Dynatech also proposes to manufacture two instruments for specific heat measurements; one, the model QTA No. 7, can be used for investigations of the true specific heat in the temperature range from −190 to 400°C. The instrument operates on the continuous adiabatic warmup principle. The specimens have a mass of 25 to 100 g, and the measurement error is 2 to 5%. The second instrument is the SH-100, which is capable of measuring mean specific heat. It uses the classical principle of mixing with a water or heavy-duty copper calorimeter. The specimens again weight 25 to 100 g. The SH-100 instrument comes in three models:

1) SHDW No. 20 for the interval −150 to 150°C, 5% error;
2) R20 No. 20 for the interval 150 to 1100°C, 3% error;
3) R30 No. 30 for the interval 150 to 1700°C, 3% error.

GENERAL STATE OF THE ART; FUNDAMENTAL PROBLEMS

Several conclusions about the present state of the art of thermophysical instrumentation can be drawn from the foregoing analysis of the nomenclature and technical specifications of instruments recently developed by various organizations in the Soviet Union for the determination of thermal conductivity, specific heat, and thermal diffusivity and from a comparison of those instruments with their foreign counterparts.

1. Clearly, the development of Soviet instruments to date has been concentrated in organizations of the Academies of Sciences of the USSR, Ukrainian SSR, and Belorussian SSR, Gosstandart, and MVSSO SSSR, i.e., organizations not directly concerned with the industrial manufacture of thermophysical apparatus. The work of these agencies has been carried on without mutual coordination. Almost every instrument prototype has an individualized design configuration. Some of them duplicate the technical specifications of others and in a number of instances fail to meet the present-day level of measurement engineering. In other countries we find that the manufacturers of such instruments are specialized firms geared to the development and small-scale production of equipment. Each firm is oriented toward a

relatively restricted nomenclature applicable to instruments similar in their operating principles, equipping them with unified modules and components.

2. Thermal conductivity measurements outside the Soviet Union are almost exclusively limited to comparative methods in the steady-state thermal regime. The only possible exceptions are the developments of Eiko Seiki Sanyo in Japan and TRAC in West Germany (GFR). Soviet developments utilize all main thermal testing regimes. In particular, they are based on steady-state laws, linear and monotonic warmup of the specimen, the regular regime, the initial stage of the thermal process, and several others. This consideration clearly mirrors the deeper interest of our thermal measurement laboratories in advanced nonsteady-state thermophysical measurement techniques. As a result, a great many Soviet instruments have a significantly higher performance rating than their foreign counterparts.

3. The picture changes with respect to instruments for the measurement of specific heat. Foreign countries make extensive use of differential-type instruments, instruments with automatic adiabatization of the calorimetric vessel, dynamic pulsed calorimeters, mixing calorimeters, and a number of others. The Soviet instruments operate mainly on the principles of the regular regime and comparative versions of dynamic warmup, the entire group of specific heat instruments being represented by just a few prototypes. Of course, the table does not include the large group of Soviet laboratory apparatuses currently in service for reliable specific heat measurements but not manufactured on a mass-production scale.

4. Instruments for thermal diffusivity measurements are represented by relatively few models in the Soviet Union and do not seem to have been developed at all abroad. This situation is mainly attributable to the fact that the thermal diffusivity can be calculated from the measured values of the thermal conductivity and specific heat. It is important to acknowledge, however, the growing demand in recent years for direct measurements of thermal diffusivity.

5. The tables reflect preponderantly the range of standard physical conditions (room temperature; open medium at atmospheric pressure). There are instrument prototypes designed for the investigation of solid materials in the temperature intervals from −100 to 400°C and from 20 to 1000°C. Materials research in more extreme physical states (high and low temperatures, high pressures, the liquid state, near phase transitions, various force fields, etc.) is still carried out on individualized experimental apparatus.

6. An important characteristic of an instrument is its guaranteed measurement error. Experience has shown that the main factor contributing to the total error in thermophysical measurements is the disregarded systematic error inherent in a particular measurement technique. This consideration poses the problem of calibrating every instrument prototype separately [23].

The comparative measurement principle is used extensively in foreign designs, so that the instruments are calibrated by and large against standard substances furnished to companies by the appropriate metrological agencies. According to the available information American companies have at their disposal a large number (more than ten) standard thermal conductivity specimens attested over a wide range of temperature variation.

In the Soviet Union the service responsible for supplying organizations with standard thermal conductivity and specific heat specimens has branched out considerably, but a sizable portion of the developed thermophysical instruments are still based on absolute measurement principles. This situation results in an unwarranted complication of measurement procedures and the absence of a reliably guaranteed error for the majority of instruments.

7. A trend toward automation of the measurement process has been noted in the instrumentation industry in recent years. In direct-reading instruments the measurement result is read out directly in the form of a number. Indirect-measurement instruments are equipped with special-purpose computing accessories for the automation of data processing.

In the stated respect, with the exception of the NIISMI and Lambda instruments, thermophysical instruments are designed for manual processing of the measurement results. Successful efforts toward the automation of thermophysical measurements are currently being made at a number of scientific-research institutions in the Soviet Union [26].

The foregoing analysis suggests that the present state of the art in measurement engineering and the widespread scale of thermophysical research should be channeled into the centralized manufacture of thermophysical apparatus under the auspices of the Ministry of Instrument Construction, Automation Means, and Control Systems and that specific developers must be urged to come up with a qualitatively new approach to the stated problem.

It is unrealistic to expect to provide the multitude of problems confronting experimental heat physics and the constant proliferation of new trends in research with specific instrument developments in every single case. In our opinion, therefore, present-day thermophysical apparatus must be designed on modular principles with the extensive use of unified modular subsystems: calorimetric measurement cells; heating, cooling, thermometric, and heat-flux measuring modules; power-supply sections and sections for stabilization of the measurement regime; computer accessories and other devices interfaced according to a unified system of communication signals. With this design approach the researcher decides how to assemble measuring equipment from the indicated modules and subsystems, having to be concerned only with the design of specialized calorimetric cells. The same approach is also viable in the design of general-purpose multiple-user instruments, but their design configuration must clearly be governed by cost-engineering considerations.

The present survey has dealt primarily with instruments for general use. However, the problems of thermophysical instrumentation go beyond this limitation. As more scientific and design experience is gained it will be possible to implement the centralized development of individualized thermophysical apparatus for vital scientific research in the presence of various field effects (magnetic, electric, gravitational, etc.) at high (above 1000°C) and low (down to 4°K) temperatures, near phase transitions, in critical states, etc.

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