COMMERCIAL THERMOPHYSICAL INSTRUMENTS (STATE OF THE ART AND PROBLEMS)

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In the review in [1], the status of thermophysical instrument making in our country and abroad for the period up to 1975 was examined and the characteristic tendencies for the development of this branch of instrument making in the immediate years ahead were revealed.

The years 1972-1975 saw the beginning of thermophysical instrument making in this country. In 1972, the State Special Design Office for Thermophysical Instrument Making (GSKB TFP), the head organization for developing commercial samples of instruments, was created. During those years, substantial results were achieved: The first instruments in this country completed the state certification tests and were included in the Gosreestr of the USSR.

Commercial instruments differ from the experimental laboratory type setups by more careful theoretical, methodological, and experimental analysis, a more complete design, higher reliability, high productivity, simplicity and convenience in servicing. They have clearly defined limits of applicability in a range of measured characteristics, regions of operating temperatures, pressures, external actions and corresponding to these limitations basic instrumental error.

Commercial instruments must be technologically efficiently manufactured, easily calibrated under assembly line production conditions and have metrological provisions in the form of a collection of standards. In order to satisfy all these requirements, it is necessary to solve different scientific-technical problems both of an organizational nature in carrying out scientific research and experimental design work as well in managing commercial production. The instruments must have a high level of standardization of design elements and, therefore, their list must consist of a standardized series of instruments based on a reference model.

At the first stage of managing the commercial production of thermophysical instruments, their list was determined by objective factors: necessity for providing guaranteed supply, existence of metrological provisions for determining the range of characteristics and temperatures, the potential of making new samples based on the reference model.

Taking into account the considerations enumerated above, two series of thermophysical instruments were chosen: one for making measurements for room temperatures and a second (wide temperature range instruments) for making measurements from -100 to $+400°C$.

Table 1 shows the characteristics of commercial thermophysical instruments made in this country. The first four concern instruments for room temperature and the rest concern wide temperature range instruments.

The IT-20 instrument is intended for measuring the thermal conductivity of solid materials with $\lambda = 0.2$-1.5 W/m·K for specimens in the form of discs with fixed dimensions (diameter 12 mm, height 4 mm). Its operation is based on the comparative stationary method [2]. Standard substances are used to calibrate the instrument: melted quartz, polymethyl methacrylate. The time necessary for establishing a stationary state is 50 min, the measurement is manual, the scale of the instrument is labeled in units of thermal conductivity. The error of the instrument is $\varepsilon_\lambda = 8\%$.

The ITÉM-1 instrument is intended for rapid measurements of thermal conductivity of solid materials with $\lambda = 0.2$-80 W/(m·K) and is based on the comparative stationary method for measurements [3]. One of the following standard substances is used for calibrating the instrument: fused quartz, polymethyl methacrylate, 1Kh18N10T steel, Armco iron. In studying materials with $\lambda \leq 5$ W/m·K, thermocouples are not mounted into the specimen, while for higher thermal conductivities reinforced thermocouples are placed in two

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TABLE 1. Commercial Thermophysical Instruments

No. in order	Instrument purpose, type	Measurement range	Dimensions and shape of samples, mm	Temperature range, °C	Measurement error, %	Measurement method	Test duration	Measurement form, instruments	Development state
1	For measuring the thermal conductivity λ of solid materials, IT-20	$\lambda=0{,}2-1{,}5$ W/m·K	Disk ⌀1, $H=4$	20	$\varepsilon_\lambda=8$	Stationary, comparative [2]	50 min	Direct, constant-current potentiometer, galvanometer	Passed the State Design Institute incl. in Gosreestr
2	For rapid measurements of λ for solid materials, ITEM-1	$\lambda=0{,}2-80$ W/m·K	Disk, cylinder ⌀15, $H=1-30$	20	$\varepsilon_\lambda=10$	Same [3]	5 min	Indirect, M195/1 galvanometer	Same
3	For rapid measurements of λ for solid materials with digital reading, ITEM-1M	$\lambda=0{,}1-80$ W/m·K	Disk, cylinder ⌀15, $H=1-30$	20	$\varepsilon_\lambda=8$	—	5 min	Direct, with digital indication, electronic circuit	Working project
4	For comprehensive rapid measurements of heat capacity c and thermal conductivity λ of solid materials with digital reading	$\lambda=0{,}1-80$ W/m·K	Disk, cylinder ⌀15, $H=5-30$	20	$\varepsilon_\lambda=8$, $\varepsilon_c=3$	Initial stage of thermal process, stationary, comparative	5 min	Same	Scientific-research work
5	For measuring λ for solid materials, IT-λ-400	$\lambda=0{,}2-5$ W/m·K	Disk ⌀15, $H=0{,}5-5$	From −100 to +400	$\varepsilon_\lambda=10$	Monotonic heating [4]	1,5 h	Indirect, M195/1 galvanometer	Passed the State Design Institute incl. in Gosreestr
6	For measuring c of solid materials, IT-c-400	$c\gamma\geqslant1\cdot10^6$ J/m³·K	Cylinder ⌀15, $H=10$	Same	$\varepsilon_c=10$	Same	1,5 h	Same	Same
7	For measuring the thermal diffusivity a of solid materials, IT-a-400	$a=(0{,}1-1)\times10^{-6}$ m²/sec	Cylinder ⌀15, $H=6-10$	"	$\varepsilon_a=10$	"	1,5 h	"	"
8	For comprehensive measurement of a and c of solid materials, ITS-400	$\lambda=5-80$ W/m·K	Cylinder ⌀15, $H=20-35$	"	$\varepsilon_a=10$, $\varepsilon_c=10$	"	1,5 h	"	"
9	For comprehensive measurement of λ and c of solid materials, ITS-2-400	$\lambda=0{,}2-5$ W/m·K	Cylinder ⌀20, $H=6-10$	"	$\varepsilon_\lambda=10$, $\varepsilon_c=10$	"	1,5 h	"	Technical project

radial openings with a diameter of 1 mm near the end faces of the specimen. The sample is shaped like a disc or cylinder with a diameter of 15 mm and a height of 1-30 mm. The measurement time is 5 min and the error is 10%. Both instruments at the present time are in a commercial assimilation stage.

The instrument ITÉM-1M represents an updating of ITÉM-1 and performs the same functions, but differs from it by the presence of direct digital reading. It is one of the new examples of thermophysical instruments, which fully satisfies modern requirements. The measurement process is automated by using modern electronic technology.

The new examples of instruments also include an automated comprehensive rapid measurement device for measuring thermal conductivity and heat capacity of solid materials with $\lambda = 0.1\text{-}80$ W/m·K. The thermal conductivity is measured according to the method presented in [3], and in order to determine the heat capacity, the initial stage of the process is used. The samples are shaped like cylinders with a diameter of 15 mm and a height of 5-30 mm. In materials with $\lambda \geq 5$ W/m·K, thermocouples are mounted in openings in the specimen. An electronic circuit permits obtaining directly the results of the measurements of thermal conductivity, heat capacity, and thermal diffusivity of a material in digital form.

Wide temperature range instruments are represented in Table 1 by five types, based on the monotonic heating regime [4]. All of them represent a standard series, based on the use of a single reference model. Four of the five instruments have passed the state instrument tests and are included in Gosreestr of the USSR. The IT-λ-400, IT-a-400, and IT-c-400 instruments are at the present time in a stage of commercial assimilation and are included in GOST 236301-79, 236302-79, 236303-79 "Plastics. Methods for determining the thermophysical characteristics in the temperature range from −100 to +400°C."

The first four wide temperature range instruments are described in detail in [4], and their technical characteristics are presented in Table 1, so that we will only describe the ITS-2-400 instrument for comprehensive measurements of thermal conductivity and heat capacity of solid materials with $\lambda = 0.2\text{-}5$ W/m·K. In the process of monotonic heating of a flat specimen, the heat flux is measured on two of its opposite boundaries, as well as the rate of heating. The specimen is made in the shape of a cylinder with a diameter of 20 mm and a height of 6-10 mm, and thermocouples are not mounted in it.

As can be seen from Table 1, the error in the measurements in all instruments is 5-10%. The magnitude indicated determines the basic error for the worst measurement conditions and, in addition, there is a technical reserve of about 20% for the error of a commercial instrument. Indirect measurements are conducted on all of the enumerated wide temperature range instruments. Direct measurements of quantities determined in a test are performed by a specialized built-in constant current potentiometer and a mirror type M195/1 galvanometer or a digital Shch68 000 type voltmeter.

In comparing the technical level of commercial instruments, included in Gosreestr (Table 1), with instruments manufactured by foreign firms, it is evident that the foreign instruments are better equipped with electronic and computing technology. Modern thermophysical instruments of leading firms, such as Setaram (France), Sinku-Rico (Japan), Mettler (Switzerland), Kuoto Electronics (Japan), have digital reading of measured parameters. They are equipped with precision analog–digital converters, programmed blocks for regulating the temperature, systems for collecting and processing data, and digital computers. Extensive use of automation, electronics, and computing technology is characteristic of modern instrument making. For this reason, the urgency of work on automation of commercial thermophysical instruments is obvious.

The fact that the level of automation of instruments made in this country lags behind the foreign-made instruments is explained by many reasons. The main reason is the absence of recent commercial production of thermophysical instruments in our country. In developing apparatus for their needs, thermophysicists have been mainly concerned with developing a method and creating heat measurement units, without considering automation. Another important reason is the low level of signals from measuring converters. An instrument, permitting recording and processing signals with an error of 0.1-1 μV, has appeared only in recent years. And finally, the method of investigating thermophysical properties has itself created its own difficulties. The measurements are always indirect and the computational formulas are relatively complicated, including different types of corrections and sometimes special functions.

At the present time, all of these problems have been overcome: A special organization, GSKB, for thermophysical instrument making is in operation, and commercial series of instruments are being assimilated at factories. Analog–digital converters, systems for collecting and processing information, and computing systems made in this country have appeared. In addition, the presence of standardized and specialized control computers permits researchers to solve any computational problems, make experiments and computational relations more complicated and to use a computer for solving inverse problems of heat conduction in investigating thermophysical properties.

In our view, modern automated instruments must meet two basic requirements. First, they must automatically set the test regime and give results in a form useful for comprehension and use: numbers in a table, tables of values of measured quantities, either recording data on a punched tape or a magnetic tape. Second, the information must be processed during the course of a test and the final result must be recorded on a TsPM or a plotter.

In creating automated instruments, two approaches are possible: the first consists of creating automated instruments with a specialized system for processing results and the second consists of creating measuring complexes, constructed with an aggregate block principle.

Automatic instruments must satisfy the requirements for the highest quantities of thermophysical measurements, they must be simple and reliable in use, relatively inexpensive, with high standardization of units and parts based on the use of a small list of reference models.

Measuring complexes can be more effectively used in carrying out scientific studies, for servicing completely equipped thermophysical laboratories intended for different purposes, as well as for calibrating and checking commercial instruments.

In creating equipped thermophysical laboratories for the purpose of further expanding the list of measurement cells, the role of the aggregate block principle for constructing instruments becomes much more significant and it becomes economically justified, since a more complete utilization of excess possibilities of separate functional blocks, specialized and universal computers will be ensured.

Studies with the use of different approaches to the automated thermophysical experiment are carried out at the Moscow Energy Institute, Moscow Aviation Institute, Institute for Heat and Mass Exchange, Academy of Sciences of the Belorussian SSR, Leningrad Institute for Precision Mechanics and Optics, TIKhM and other organizations. However, in most cases, they are limited to creating a specific measuring system or instrument, andgeneral principles for constructing schemes for obtaining direct readout in automated instruments are not brought out in them. Such schemes must be distinguished by universal application, a high level of standardization of electronic circuit modules, high precision, and low cost. At the same time, many essential difficulties arise, especially for the wide temperature range instruments. The most important are: low level of input signals, nonlinear temperature dependence of most of the parameters, long duration of experiment, complexity of calibration. The technical–economical analysis that has been conducted showed that automation of autonomous commercial instruments should be done by developing standardized analog–digital and digital electronic blocks, as well as control blocks with rigorous logic. Then, automation of any new instrument will reduce to merely changing the logic of the control block.

The development of thermophysical instrument making is closely related to metrological provisions for the measurements. The list of available standard samples does not cover the entire range of measurement characteristics, in particular, it does not include the high temperature range up to 1000°C for thermal conductivity. Therefore, the work of metrological organizations and GSKB TFP must be closely coordinated.

New series of thermophysical instruments can form instruments for nondestructive monitoring of the thermophysical properties of materials both in production as well as under field conditions and instruments for high temperatures in the range 900-2000°C. In the high temperature range, it is desirable to use the well-developed temperature wave, modulation, and pulsed methods (work of Moscow State University, Institute of High Temperatures of the Academy of Sciences of the USSR, Ural Polytechnical Institute, and others).

In assimilating the commercial output of instruments, especially wide temperature range instruments, great difficulties arise in tuning, calibrating, and checking instruments. The operations themselves require repeated repetition and take up a great deal of time. In this connection, it is necessary to search for self-checking methods, constructed in a testing and processing regime such that the calibration is done automatically and the characteristic systematic errors are taken into account automatically as well.

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