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Instrumentation complex for measuring the thermophysical properties of rocks in natural occurrence temperature conditions

A A Kalyak, S A Kazantsev, V M Gruznov* and I I Zasypkina

Trofimuk Institute of Petroleum Geology and Geophysics Siberian Branch of the Russian Academy of Sciences, Novosibirsk, Russian Federation

E-mail: gruznovvm@ipgg.sbras.ru

Abstract. A schematic diagram and an operation algorithm of an instrumentation complex for measuring the thermal conductivity and thermal diffusivity of rocks by the non-stationary method of a linear source under natural occurrence temperature conditions up to 200 °C are proposed. Thermocouples are used to measure the temperature of the probes. The complex contains two linear (needle) probes, which are placed in a rock sample at a certain distance r from each other. One probe, containing a linear heater and a thermocouple, radiates heat, the other one, containing a thermocouple, is a measuring probe. The cold junction temperature of thermocouples is measured by a semiconductor sensor. To calculate the voltage of the cold junction from the value of its temperature, a direct conversion of thermoEMF by a power polynomial is used. To calculate the temperature of the hot junction of the probe, an inverse transformation by a power polynomial of the voltage values of the hot junction of thermocouples into temperature was applied. In direct and inverse transformations, the degree of polynomials is 10. To reduce the temperature measurement error, digital filtering of signals from thermocouples was used. A digital-to-analogue converter is included in the heater circuit of the radiating probe for flexible heating control. The results of measuring the thermal conductivity and thermal diffusivity of dry and wet sand samples are consistent with the literature data on the temperature dependence.

1. Introduction

Heat transfer by geological media plays an important role in a large variety of different geological processes: the formation of geological structures, mineral deposits, the relief of the Earth's daily surface, etc.

The study of thermophysical properties of extracted rocks is important both for the theory of heat transfer and for the practice of their technical application. The study of these properties provides valuable information that allows to deepen and expand theoretical ideas about the processes of heat propagation and dissipation in complex multicomponent geological media [1]. During core sampling and removal to the surface, the properties of rocks and the fluid saturating them noticeably change, so the results of core analysis in normal laboratory conditions do not provide a complete picture of the thermal properties of rocks at a depth corresponding to their natural occurrence [2]. On average, the geothermal gradient is about 3°C per 100 m of depth and the pressure gradient is about 0.1 MPa per 10 m of depth. For example, at a depth of 5 km in a bed of rock strata, for example, the temperature can be as high as 150°C and the pressure can be as high as 50 MPa.

Based on the relevance of knowledge of the thermophysical properties of rocks in real conditions of their occurrence, there is a need for experimental studies of the thermophysical properties of rocks in laboratory-defined conditions of high temperatures (150 °C and above). Such studies allow to estimate the thermal properties of rocks and heat flux density at different depths of the Earth's crust and obtain new data on the patterns of these characteristics of rocks under natural conditions.

There are several methods for determining the thermophysical properties of rocks, among which we distinguish between stationary and non-stationary [3]. In the stationary split bar method, a disc-shaped sample is placed between two discs of reference material of known thermal conductivity, the temperature of which is kept constant. After reaching a steady state, the thermal conductivity of the sample is determined by comparing the temperature drop across its height with the drop in the reference material. The method is sufficiently reliable and is a standard of accuracy, but it requires time-consuming sample preparation in advance and a lot of time when determining only one parameter - thermal conductivity.

Non-stationary non-contact optical scanning method allows determining thermal conductivity and thermal diffusivity by heating a small area of the sample with a laser and measuring the temperature with infrared sensors on the surface of the samples. This method is highly productive and accurate (up to 350-400 samples per day with an error of about 5%), but requires sample preparation and is applicable only for measurements at room temperature and atmospheric pressure.

The non-stationary method of linear source measurement of thermal conductivity and thermal diffusivity of rocks [4] uses two linear probes placed in a rock sample at a distance *r* from each other. A schematic of the method is shown in figure 1.

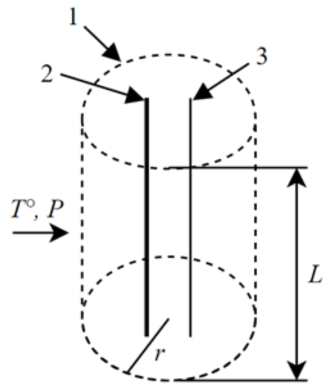

Figure 1. Schematic diagram of the linear source method: 1 - rock sample under study; 2 - emitting probe with a linear heat source and a temperature sensor; 3 - receiving probe with a temperature sensor.

One probe (figure 1, item 2) is emitting heat by a linear heater and with temperature sensor S1, the other probe (item 3) is receiving probe with temperature sensor S2. To determine thermal conductivity, a short calibrated voltage pulse is applied to the heater of the emitting probe, creating a heat wave. With the beginning of the pulse, sensors S1 and S2 register the temperature changes of the probes. According to these values the time τ_{max} of the thermal wave maximum passage from the radiating probe to the measuring probe is determined. The temperature conductivity a is determined by the formula:

$$a = \frac{r^2}{4 \cdot \tau_{max}} \tag{1}$$

To determine the thermal conductivity, a voltage is applied to the heater of the radiation probe for a longer time (about 2-3 minutes). With the beginning of heating the temperature registration by the temperature sensor S1 begins. Then, in the linear section of the temperature growth of the radiating probe, the times τ_0, τ_1 and their corresponding temperatures T_0, T_1 are recorded. Using the obtained data, the thermal conductivity λ is determined by the formula:

$$\lambda = \frac{q}{4 \cdot \pi \cdot (T_1 - T_0)} \cdot ln \frac{\tau_1}{\tau_0} \quad (2)$$

where q is specific heat flow per unit length of radiating heat probe, T_0 is the temperature corresponding to time τ0 after turning on the heater, T_1 is the temperature of the probe at time τ_1 ($\tau_1 > \tau_0$).

The advantages of the method lie in the fact that there is no need to establish thermal equilibrium, typical of the divided rod method, due to which the result is achieved faster, and the presence of a thermostat in the scheme allows working with the temperatures corresponding to the conditions of the natural occurrence of rocks.

Scheme of the method with two probes, shown in figure 1, is implemented in [5]. In this paper, semiconductor thermoresistors are used as temperature sensors in probes which are limited in maximum measured temperature to +125°C, which makes it impossible to use them when measuring thermophysical properties of rocks in cases of modeling conditions of their natural occurrence, namely measurements at high temperatures (+200°C and above).

The purpose of the study is to justify the basic electronic scheme and algorithm of the hardware complex to measure the thermal conductivity and thermal conductivity of rocks at temperatures up to 200 ° C and to improve the accuracy of measuring thermal physical characteristics.

2. Principle diagram and algorithm of the complex operation

Semiconductor thermoresistors in needle probes limit measurements of thermophysical properties of rocks at temperatures above +125°C. The use of chromel-alumelium thermocouples has extended the temperature range of measurements to at least 200°C.

Figure 2 shows a schematic diagram with designations of elements of the developed hardware complex for measuring the thermal conductivity and thermal conductivity of weakly cemented rocks at temperatures up to 200 ° C. The main units: thermostat (figure 2, item 1) with the cell (item 2) with rock sample and two probes (items 3 and 4), electronic temperature meter (item 6) and personal computer (PC). Thermocouples with the ability to measure temperature up to 200 °C are used as temperature sensors in the probes.

Operating principle of the complex: at a steady temperature set in the thermostat, the power level and duration of the probe heater 3 is set from a personal computer. These data are sent to the microcontroller (MC) of the electronic meter. A signal from digital-to-analog converter (DAC) output or microcontroller's pulse-width modulation (PWM) is sent to the heater's power switch (PS), which determines heater's power level and duration of its operation.

Temperature measurement with thermocouples is performed as follows. Using a semiconductor temperature sensor, located in direct contact with the cold junctions of thermocouples, their temperature T_{cold} is recorded. The value of this temperature is necessary to compensate for the cold junction of thermocouples when calculating the temperature T_{hot} of hot junctions of thermocouples located in the probes. To calculate the cold junction voltage E_{cold} by its temperature value T_{cold}, a direct transformation of the thermoelectromotive force by a power polynomial of the form [6] is used:

$$E_{cold} = d_0 + d_1 \cdot T_{cold} + d_2 \cdot (T_{cold})^2 + d_n \cdot (T_{cold})^n \quad (3)$$

where d_n are the coefficients of the direct thermoelectric EMF conversion (order of polynomial n = 10).

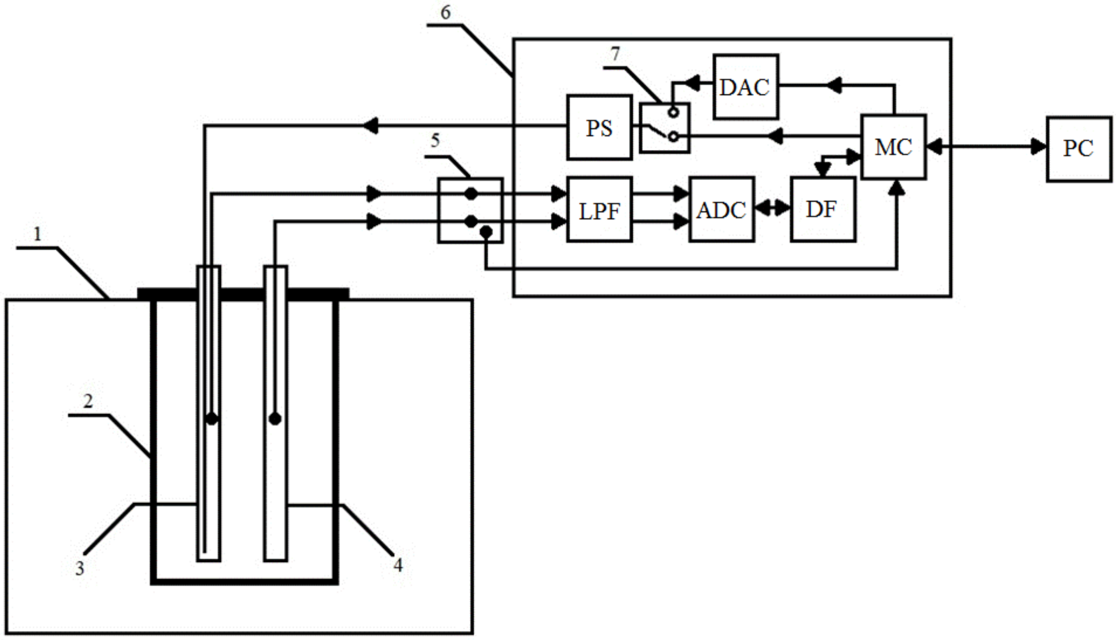

Figure 2. Schematic of hardware complex: 1 - thermostat; 2 - sample cell; 3 - probe with heater and thermocouple; 4 - probe with thermocouple; 5 - cold junctions of thermocouples with semiconductor temperature sensor (cold junction temperature control); 6 - electronic temperature meter; 7 - heater power switch control method switch (external DAC, or integrated PWM microcontroller); PS - heater control power switch; LPF - low-pass filter; DAC - digital-to-analog converter; ADC - analog-to-digital converter; DF - digital filter; MC - microcontroller; PC - personal computer.

The thermocouple signals through a low-pass filter (LPF) are fed to the input of the analog-to-digital converter (ADC). In order to get the E_{hot} value at the hot junction of the thermocouple, the ΔE value at the ADC input and the calculated Ecold value of the cold junction are summed:

$$E_{hot} = E_{cold} + \Delta E \tag{4}$$

Then, the inverse conversion of hot junction thermocouple voltages to temperature is done:

$$T_{hot} = k_0 + k_1 \cdot E_{hot} + k_2 \cdot (E_{hot})^2 + k_n (E_{hot})^n \tag{5}$$

where the order of the polynomial is $n = 10$, k_n are the coefficients of the inverse thermoelectric EMF conversion.

Together with the digital filtering of the signal (through a DF unit) the error of the applied polynomial approximation of the thermocouple signal is from ±0.02°C to ±0.08°C, in contrast to the traditional linear [6], where the error may be 0.5°C and more.

Values of obtained probe temperatures and times are fed to MC and sent to a personal computer to calculate thermophysical parameters of samples.

Temperature conductivity a of the studied rock samples is determined by the formula (1) [5, 7]. Measuring range a: $(0.1 - 15)\times10^{-7}$ m^2/s.

Thermal conductivity λ is calculated by formula (2) [4, 8]. The specific heat flux per unit length of the heater q is calculated as follows:

$$q = \frac{P}{L}, \tag{6}$$

where P is heater power, L is heater length.

The times τ_1 and τ_0 are determined by a timer built into the microcontroller. Thermal conductivity measurement range: 0.1 - 6.5 W/(m×K).

3. Results and discussion

The sensitivity coefficient of the thermocouple depends on the temperature of its working junction. Figure 3 shows experimental data of the deviation of the measured thermocouple temperature by the value of ΔT up or down from the one actually set in the thermostat T_{set}. The graph shows that when the thermocouple EMF is converted linearly to temperature using the formula:

$$T_{hot} = (\Delta E + S \times T_{cold})/S, \tag{7}$$

in which the dependence of the coefficient S on temperature is not taken into account, the measurement error reaches more than 0.5°C.

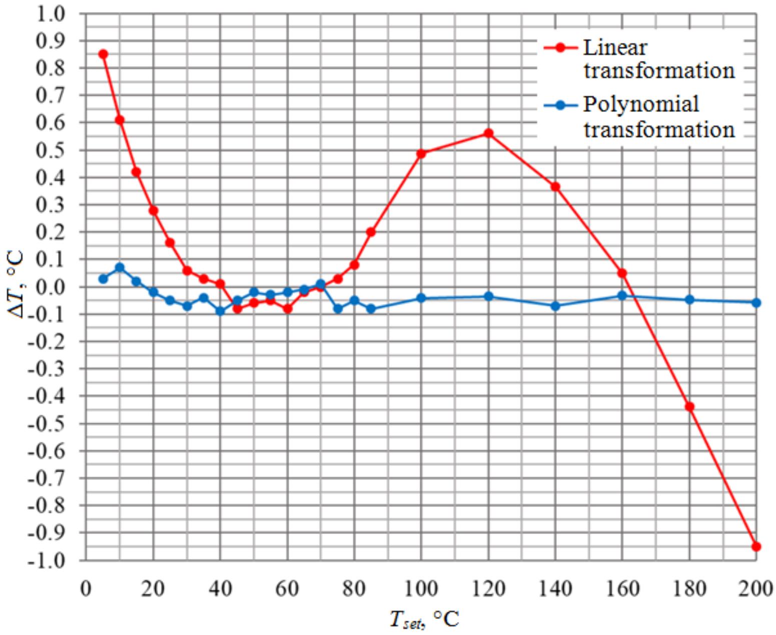

Figure 3. Temperature measurement error depending on the type of thermocouple EMF-to-temperature conversion.

Increasing the accuracy of temperature measurement is achieved by taking into account the dependence of the sensitivity coefficient of the thermocouple on the temperature. The dependence of the temperature of the hot junction on the EMF in this case is represented by a power polynomial (5). The error of temperature determination at polynomial transformation in conjunction with digital signal filtering is no more than 0.08°C, which is demonstrated by the graph in figure 3.

The workability of the complex was tested by measuring the thermal conductivity and thermal conductivity of dry sand (humidity not more than 0.1%) and moisture-saturated (humidity 25%). The average size of sand grains was 0.4 mm, specific gravity 1.7 g/cm3.

Figure 4 shows a series of measurements of the thermal conductivity of dry sand under normal climatic conditions. The measurement error of the thermal conductivity values was no more than 8%.

Compared to the dry sand sample, the measured thermal conductivity of the moisture-saturated sample was expected to increase about 9 times. The measurement error was no more than 5%.

Figure 5 shows the results of measuring the thermal diffusivity of dry and moisture saturated sieved sand. The measurement error of the thermal conductivity varies from 5 to 10%.

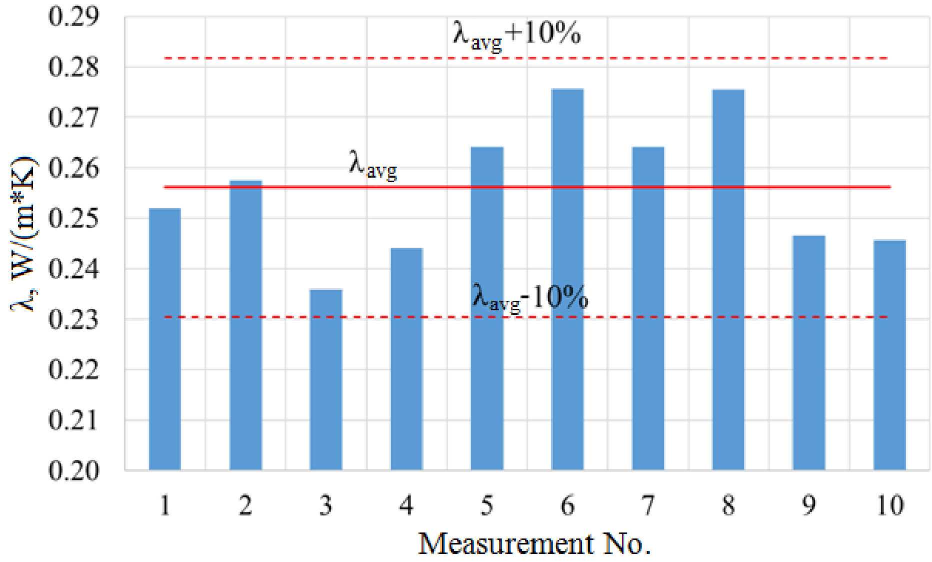

Figure 4. Series of measurements of the thermal conductivity of dry sand.

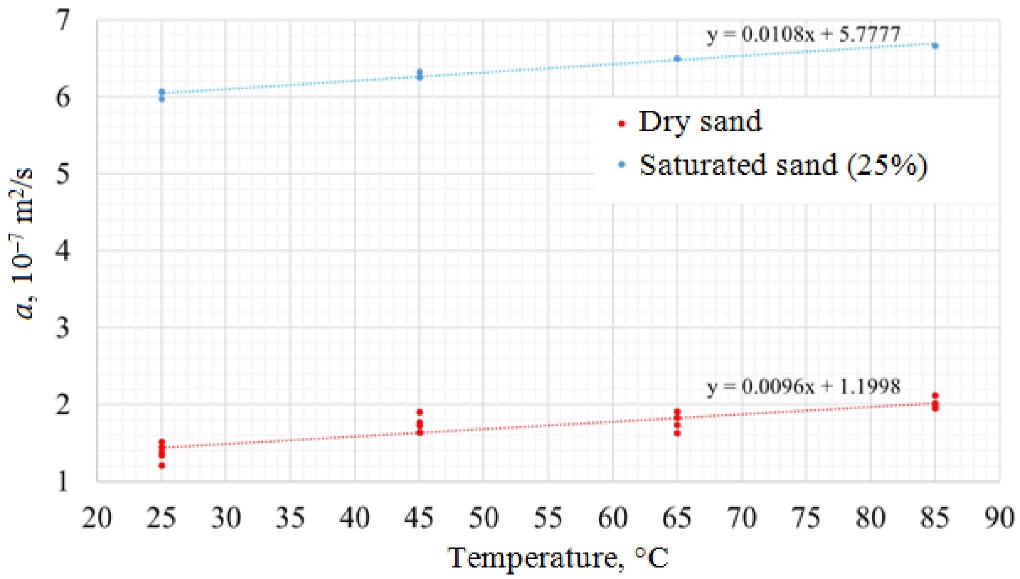

Figure 5. Measurement of the thermal diffusivity of dry and moisture-saturated sieved sand as a function of temperature.

The trend of change in thermal diffusivity depending on temperature is similar to the trend of change in thermal conductivity and also corresponds to the data presented in [9].

The measurement error of 5-10% is due to the following: in non-stationary methods analytical expressions describing time-varying temperature fields are used to determine thermophysical properties.

This leads to cumbersome calculation formulas derived from the solution of the boundary value problem, and when they are simplified into formulas (1) and (2), the deviation of the model from the real object inevitably increases, which leads to an increase in the methodological error [1].

The main source of errors in measurement of thermophysical properties are temperature measurement errors. Application of digital filtering in combination with polynomial approximation of thermocouple signal provided an increase of temperature measurement accuracy up to 0.08°C.

At the same time, the measurement error of thermal conductivity and thermal diffusivity was 5-10 %, which is typical for the non-stationary method of the linear source and is acceptable for estimation of thermal properties of rocks at depths of 4-5 km.

4. Conclusion

The basic scheme and algorithm of the hardware complex for measuring the thermal properties of rocks by the linear source method in the temperature conditions of natural occurrence up to 200 °C have been improved in comparison with [5]. Such temperature range is provided by application of thermocouples for probe temperature measurements.

Application of power polynomials for direct and inverse transformations when calculating thermocouple temperatures in combination with digital filtering of signals from thermocouples ensured an error of measurements of thermophysical characteristics within 5-10 %. Such measurement results were achieved with unconsolidated sand samples. The applicability of this method is also possible for consolidated rock samples.

The use of digital-to-analog converter in the circuit of the heating element of the radiating probe provides flexible control of the probe heating modes, which significantly expands the possibilities of selecting optimal levels of the radiating probe heating power for rock samples with different thermal-physical characteristics.

Acknowledgements

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