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AN EQUIPMENT FOR MEASURING THERMOPHYSICAL QUANTITIES BY MEANS OF HEAT—PULSE METHODS IN THE TEMPERATURE REGION BETWEEN 20—300 °C

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The present paper describes an equipment for measuring the thermal conductivity, thermal diffusivity and specific heat by means of the heat-pulse method with a plane heat source in the temperature region between 20—300 °C. The equipment can be used for measurements by the pulse method with a line and point source. An analysis of the influence of the temperature stability of the sample on the accuracy of the measured characteristics is made.

I. INTRODUCTION

In recent years heat-pulse methods have begun to be used for the measuring of the thermal conductivity λ, the thermal difusivity k and the specific heat c. The heat-pulse methods can be divided with respect to the heat source into methods with point, line and plane sources [1, 2]. Some authors suggested an experimental arrangement for the measurement of c, k, λ using pulse methods. Krempaský [1, 2] described some modifications. Kulakov [3] described an arrangement with a plane source.

The present paper describes an equipment for measuring the thermophysical quantities c, k, λ by means of the heat-pulse method with a plane heat source in the temperature region between 20—300 °C. The equipment was used for measuring c, k, λ of an $NaNO_3$ single crystal. The measured characteristics are published in papers [5, 6].

II. GENERAL CHARACTERISTICS OF THE PULSE-METHOD WITH A PLANE SOURCE

From [1] it follows that for the thermophysical quantities measured by the pulse-method with a plane source the following relations must be used:

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$$k = x^2/2t_m, \qquad c = Q/(2\,\pi\,e)^{1/2}\gamma T_m, \qquad \lambda = kc\gamma,$$

where: x — distance between the plane source and an indicator of the temperature rise T_m [cm]; t_m — time of maximum T_m at the distance x [sec]; Q — amount of heat generated in the plane source [cal/cm²]; γ — specific mass [g/cm³].

The relations were found for an infinite medium, for heat sources with no heat capacity and no contact resistance (the thermal resistance of the contact between the heat source and the sample).

In practice the measurements can be realized only with the contact resistance and the heat source with its heat capacity The size of the measured sample is definite. **Kulakov** [3] found the condition for the heat capacity of the plane source. Kubičár and Krempaský [4] analyzed the influence of the sample and the source geometry, the heat transfer from the surface of the sample and the contact resistance. An analysis gives the correction factors which are used in practical cases.

An equipment for measuring c, k, λ in the temperature region between 20—300 °C consists of two parts. The first part consists of an electrical circuit for the realization of the heat-pulse and a circuit for the registration of the rise of temperature T_m. The other part consists of devices for heating the sample and devices for measuring the sample temperature and the temperature stability of the sample.

The heat-pulse is generated in the plane source through the current-pulse. Then the amount of the Joule-heat Q generated in the plane source can be expressed as:

$$Q = RI^2\Delta t,$$

where: R — resistance of the plane source [Ω/cm₂]; Δt — width of the current-pulse [sec]; I — intensity of the current [A].

The circuit for the registration of T_m consists of a differential thermocouple which measures the rise of temperature T_m and a galvanometer. Then T_m can be expressed as:

$$T_m = \frac{(R_G + R_T)G_i d}{\alpha},$$

where: R_G — resistance of the galvanometer [Ω]; R_T — resistance of the differential thermocouple [Ω]; G_i, d are the sensitivity and the deflection of the galvanometer [mm], respectively; α — thermopower of the differential thermocouple [V/°C]. The accuracy of measurements of c, k, λ depends apart from the above mentioned factors on estimating the parameters of electrical circuits and the temperature stability of the sample. We must consider that circuit parameters vary with varying temperature of the sample.

The temperature stability of the sample plays the most important role as regards the determination of the value of t_m. We consider the constant temperature rate b of the furnace or the sample, then the temperature ΔT registered by the differential thermocouple can be expressed as

$$\Delta T = \Delta T' + bt,$$

where

$$\Delta T' = \frac{Q}{c\gamma}\,\frac{1}{(\pi\,kt)^{1/2}}\exp\left(-\frac{x^2}{4kt}\right)$$

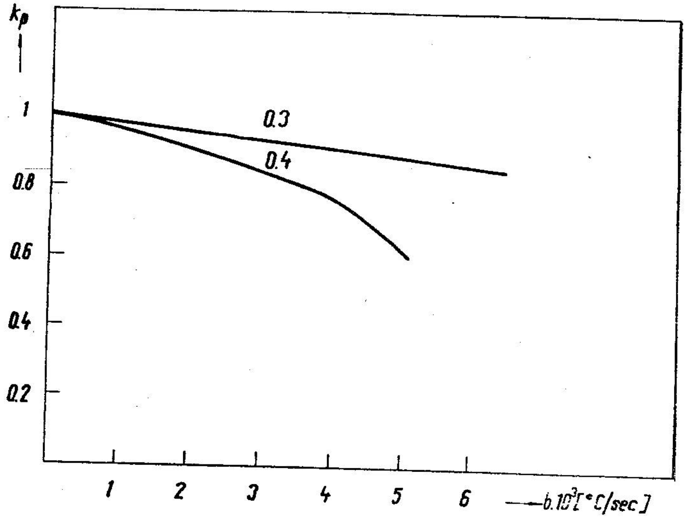

Fig. 1. The correction factor k_p;x — parameter.

and the condition for the maximum of the local rise of temperature $\partial\Delta T/\partial t =$ $= 0$ at $t = t_m$ leads to the equation:

$$\frac{x^2}{2kt_m} = 1 - \frac{b}{\dfrac{Q}{c\gamma}\,\dfrac{1}{2t_m(\pi\,kt_m)^{1/2}}\exp\left(-\dfrac{x^2}{4kt_m}\right)}.$$

We denote k_p as

$$k_p = 1 - \frac{b}{\dfrac{Q}{c\gamma}\,\dfrac{1}{2t_m(\pi\,kt_m)^{1/2}}\exp\left(-\dfrac{x^2}{4kt_m}\right)}$$

then in the case of $NaNO_3$ ($c = 0.24$ cal/g °C, $\gamma = 1.915$ g/cm^3, $k = 7 \times 10^{-3}$ cm^2/sec, $x = 0.3$ cm) the correction factor k_p is represented in Fig. 1. If we suppose the accuracy of estimating k to be better than 10 % and both welds of the differential thermocouple to be in the furnace, then the temperature rate between the welds must be less than 0.005 °C/sec.

III. DEVICES FOR MEASURING c, k, γ

The devices (Fig. 2) consist of two electrical circuits. The first circuit for generating the heat-pulse consists of the current pulse cource (SP), the plane resistance (PS) and an apparatus for measuring the intensity of the current-pulse as well as an apparatus for measuring the value of the plane resistance.

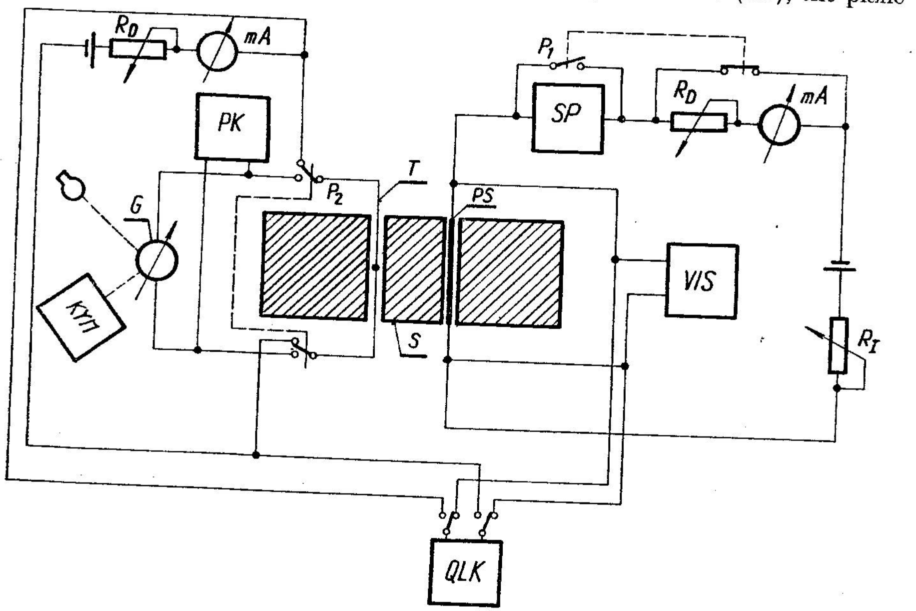

Fig. 2. Schematic arrangement for the pulse method with a plane source. KYM — Kymograf; G — galvanometer; PK, QLK — compensator; P₁, P₂ — switches; T — thermocouple; S — sample; PS — plane source; SP — pulse source; VIS — Visicorder; R$_I$ — slide rheostat for regulating the intensity of the current pulse; R$_D$ — resistance box for regulating the current through R$_V$ and R$_T$, when the value R$_V$, R$_T$ is measured.

The intensity of the current pulse can be recorded by a Honywell Visicorder (VIS). When the switch P₁ is switched over, then the value of R$_V$ (plane resistance) can be determined by measuring the voltage on R$_V$ and the current through R$_V$. The source of the current-pulse (SP) is a switch circuit which is coupled with a flip-flop circuit. The electrical circuit diagram of the pulse source is in Fig. 4. Applying two suitable trigger pulses to the flip-flop circuit, we can obtain the current-pulse. The space between the trigger pulses determines the width of the current-pulse. Suitable trigger pulses are given by the Tesla Electronic Chronometer, type NTZ 616. The current-pulse source has the following parameters: $t = 0.05, 0.1, 0.2, 0.5, 1, 2, 5, 20$ sec; $I = 0.1-15$ A.

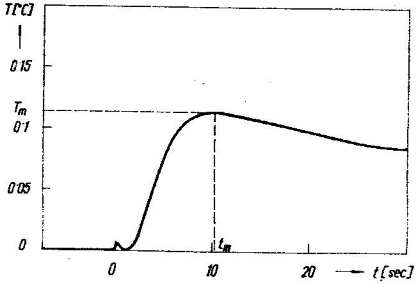

Fig. 3. The record of the rise of T_m; $T_m = 0.11$ °C, $t_m = 11$ sec.

The second circuit for the recording of the temperature rise T_m consists of a compensator (PK), a galvanometer (G), a differential thermocouple (T) and devices for measuring the resistance of the differential thermocouple.

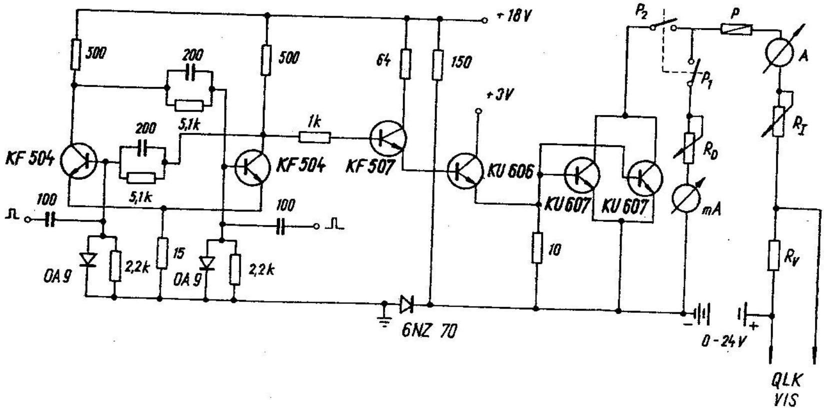

Fig. 4. Electrical circuit diagram of the pulse source.

The thermovoltage of the differential thermocouple which measures the temperature rise T_m is recorded by a Kipp galvanometer, type A 82. A light beam from the galvanometer is passed through the Kymograf (KYM), the recording photo-paper of which moves at a constant speed. Any temperature gradient between the welds of the differential thermocouple is compensated by a compensator (PK). The suitable position of the light beam in the Kymograf can be regulated by the same compensator. When the switch P_2 is switched over, the value of R_T can be determined in the same way as the value of R_V.

The differential thermocouple was made of Cu-konst wire of 0.1 mm diameter.

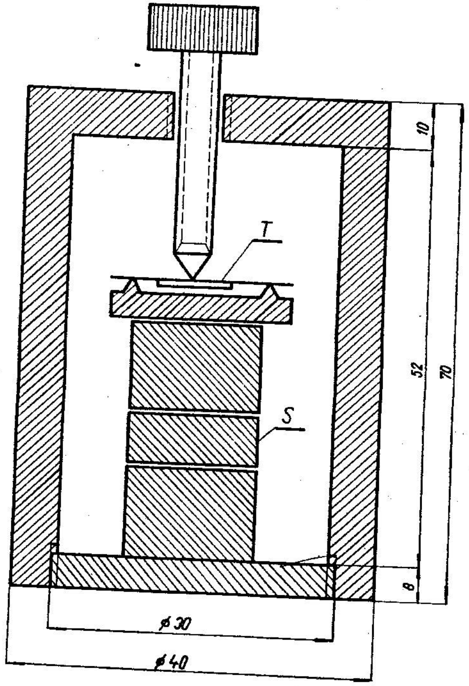

Fig. 5. The holder of the sample. T — tensometer; S — sample; material: brass.

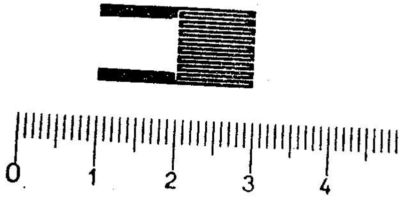

Fig. 6. The plane resistance.

Fig. 3 presents the record of the galvanometer. The parameters of measuring were: temperature of the sample $T = 50\,°\mathrm{C}$, $x = 0.3$ cm, $Q = 0.3$ cal/cm^2.

The construction of a holder is in Fig. 5. A tensometer measures the pressure applied to the sample. The plane resistance (Fig. 6) was made of a thin foil (40 μ) of beryllium bronze. The plane resistance with dimensions of 1 × 1 cm has the value $R_V = 0.85\,\Omega$.

IV. DEVICES FOR MEASURING THE TEMPERATURE STABILITY OF THE SAMPLE

A schematic arrangement of the furnace, of the device for measuring the temperature of the sample and of the apparatus for measuring the temperature stability of the sample is shown in Fig. 7.

The temperature of the sample is measured by a Tesla compensator, type QLK. A MAW recorder BKT1 records the temperature of the sample and of the furnace. The temperature stability of the sample is recorded by a Labora recorder, type EZ4. All thermocouples are made of Ni-Nichrome. Fig. 8 presents typical courses of the temperature rate of the sample and of the furnace, which were recorded by BT1 and EZ4. The courses have 3 regions. The first presents the temperature rate of the sample, the second presents the stabilization of the temperature and the last one presents the measuring region. At every stable temperature several measurements were made. The heating current of the furnace has to be regulated in a suitable way so that we may obtain the courses of the temperature mentioned above.

V. CONCLUSION

The present paper describes the experimental arrangement used for measuring the thermophysical quantities c, k, λ of an $NaNO_3$ single crystal [5, 6].

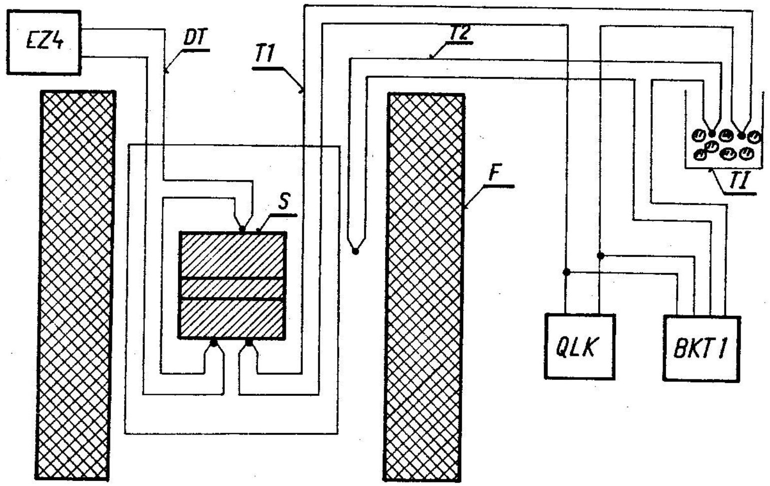

Fig. 7. The control unit of the sample temperature, schematic assembly. EZ4, BKT1 — recorders; DT, T1, T2 — thermocouples; S — sample, F — furnace; QLK — compensator; TI — thermos flask.

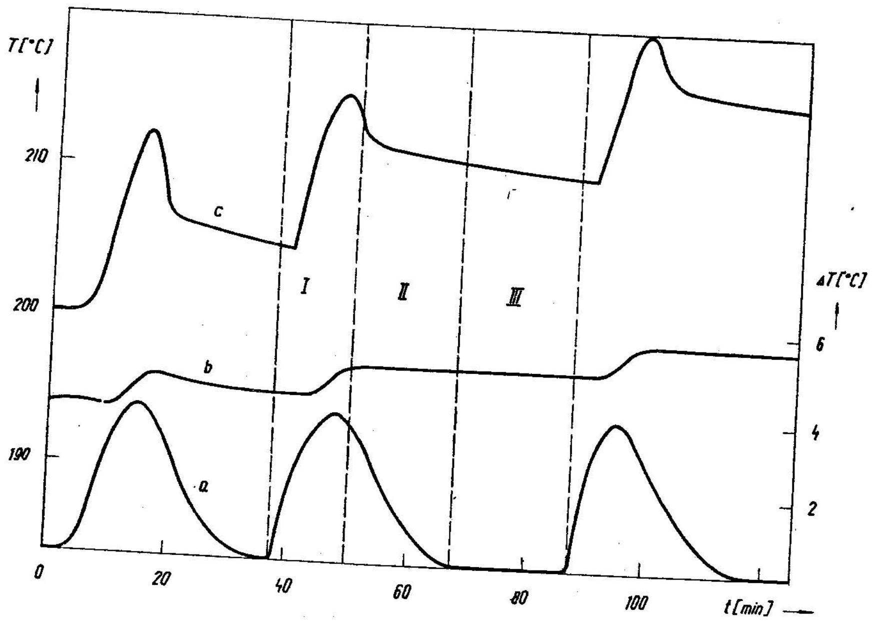

Fig. 8. The courses of the rise and the stabilization of the temperature. a — ΔT recorded by DT and EZ4; b, c — T recorded by T1, T2 and BKT1 (Fig. 6); I — the region of the temperature rate; II — the region of the stabilization of the temperature; III — the measuring region.

The equipment can be used for measuring c, k, λ by the pulse method with a point, a line or a plane heat source. Some parts of the equipment can be used for measuring c, k, λ in the temperature region between −180—0 °C. The described equipment is suitable also for the measurements of c, k, λ in critical regions (phase transformations).

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REFERENCES

[1] Krempaský J., *Meranie termofyzikálnych veličin.* Vydavateľstvo SAV, Bratislava 1969.
[2] Krempaský J., Čs. čas. fys. *A 16* (1966), 136.
[3] Кулаков В. М., *Тепло и массоперенос.* Издавательство Акад. наук БССР, Минск 1962.
[4] Kubičár L., Krempaský J., Phys. Stat. Sol. *2* (1970), 739.
[5] Kubičár L., Fyz. čas. SAV *18* (1968), 58.
[6] Kubičár L., Mariani E., Fyz. čas. SAV *18* (1968), 251.

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