Digital device for thermophysical measurements by wire probe

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Abstract. The paper is devoted to development of a digital device for rapid measurements over a wide temperature range, including superheated states of a substance. The design of the device enables automation of the measurement procedure with a minimum number of preset settings and real-time output of information. Single- and double-pulse heating modes are discussed.

1. Introduction

A methodology for studying the properties of liquids in not fully stable states is based on the development of pulsed techniques for local heating of a substance using miniature probes [1-3]. The use of modern elemental base and special signal processing procedures makes it possible to diversify the choice of measurement modes and to obtain a priory unknown results in the temperature range inaccessible to quasi-static methods. The report discusses the principles of constructing a digital device for short-term measurements on a wire probe – resistance thermometer. The device action is aimed at ensuring the accuracy of reproducing specified heating modes and the speed of recording response signals appropriate to the conditions of the task.

2. Device

A digital device was developed; its block diagram is shown in Figure 1. The operation of the device is controlled by a microcontroller unit (MCU). MCU executes the probe heating with a current pulse and simultaneously measures its temperature. The magnitude of the current is adjusted to the properties of the measured sample. The setting is implemented by a controlled current regulator that receives the current value in digital form from the MCU and stabilizes it in the course of the pulse. Measurement of the “instant” probe temperature during pulses is carried out by a measuring block. Adjustment of the measuring circuit to a predetermined part of the temperature range of measurements is performed by a measuring block using a digital-to-analog converter (DAC). The measuring block converts the voltage drop on the probe to a signal that goes to analog-to-digital converter (ADC) of the MCU. These data are recalculated in a change in the probe resistance related to a change in its average-mass temperature. Temperature curves are stored in the flash memory unit and sent to the PC via USB interface.
3. Double pulse heating technique

As an example of using the device for thermophysical measurements, consider a technique of double pulse heating of a thin wire probe (resistance thermometer) placed in a liquid. The first pulse heats the probe to a predetermined temperature $T_1$. The second pulse of an order of magnitude lower power keeps the probe in a heated state near $T_1$ in the time interval required for reheating to this value (Figure 2). The $T_1$ value can be chosen in such a way that the liquid will spontaneously boil up during the second pulse. Boiling-up is accompanied by a sharp increase in the heat flux from the probe ($S^* > S$) and a decrease in its temperature. From Figure 2 it can be seen that in this case, the length of the second pulse will stepwise increase up to the limiting pulse length $t_{lim}$. This provides a basis for unambiguous detection of the spontaneous boiling-up moment in the course of an increase in the $T_1$ value. The attained temperature value is taken as the temperature of thermal instability onset under given heating conditions $T_1^*$, which serves as individual characteristic of the liquid at a given pressure.

![Figure 1. Schematic diagram of the experimental setup.](image1)

![Figure 2. The essence of double pulse technique: current across the probe and its temperature versus time. Characteristic probe heating curves at the conditions of $t_2 < t_{lim}$ (left) and $t_2 > t_{lim}$ (right), where $t_{lim}$ is the limiting pulse length. Characteristic time of heating section ($t_1 - t_0$) and measuring one ($t_2 - t_1$) is of the order of $10^{-4}$ s and $10^{-3}$ s, correspondingly.](image2)
Approach (from below) the temperature $T_1^*$ was carried out in small steps of (1–5)°C in a certain temperature range characteristic of this sample (Figure 3). It was revealed in experiments that this heat release mode was extremely sensitive to the appearance of volatile impurities in the initially “pure” liquid. An increase in the content of volatile impurities led to a decrease in the temperature $T_1^*$. The corresponding mode setting allows one to detect the presence of impurities at the level of traces, i.e., units of grams of impurity per ton (ppm) [4]. As an example, Figure 4 shows the results of calibration of transformer oil by moisture content. The $T_1^*$ value for the oil is plotted on the ordinate; moisture content $c$ in oil in ppm, on the abscissa. The dependence is monotonous and close to linear in the range relevant for transformers. This made it possible to apply this technique in devices for express control of the actual moisture content in oils directly in the operating equipment [5]. The observed effect was manifested solely at temperatures close to the value of boiling-up temperature of watered sample.

4. Constant power technique

Due to the possibility of programming the heating current, it is possible to realize thermal conductivity measurements of a liquid based on the THW-technique [6]. The essence of the implementation lies in the step-by-step selection of the current so that the power of heat release (product of square of the current and the probe resistance) become constant in time:

$$P(t)R(t) = \text{const.} \quad (1)$$

The measurement consists of several cycles. In the first cycle, heating the probe with a constant current of a selected duration occurs, see Figure 5, dashed line. Taking into account the evolution of the probe temperature $T(t)$ calculated from the experimental dependence $R(t)$, in the subsequent heating cycles, the selection of the dependence $I(t)$ occurs in such a way as to satisfy condition (1), see Figure 5, solid line. Recording the evolution of temperature $T(t)$ at a constant heat release power and the known probe size is sufficient to calculate the thermal conductivity coefficient [7]. Figure 6 shows the temperature dependence of thermal conductivity of water in the temperature range from -150 to 25°C, which was calculated from the primary data measured by this device. At a temperature of 0°C, the expected phase transition has been observed, which is characterized by a surge in thermal conductivity values.
5. Evaluation of PMMA thermophysical properties

The experimental setup has a typical structure for pulse measurements on a wire probe and contains a powerful key transistor to supply a heating pulse with specified parameters, see details in [8, 9]. The temperature-sensitive resistance of the probe varies with a change in its temperature according to the known dependence. Resistance is measured in the bridge circuit with necessary amplification and conversion into a digital form. The equivalent of resistance variations measured in the experiment is the voltage drop on the probe and standard resistor. The final results are processed on the computer by calculating the time evolutions of the probe heating power and probe temperature based on the primary experimental data (Figure 7). These results serve as input data for modelling the transient heat transfer in the experimental cell [9].

Figure 5. Raw signals of a current in the probe circuit in the course of first (dashed line) and final cycle (solid line) of measurement.

Figure 6. Thermal conductivity of water versus temperature obtained in the mode of constant temperature increase in the cell.

Figure 7. Time evolution of the heating power and probe temperature obtained from the primary data. The higher is the heating curve for a given power value (see insert), the lower the corresponding value of heat conduction for a sample: 1 – tetradecane, 2 – hexadecane, 3 – benzaldehyde, 4 – benzaldehyde/ethanol mixture, 5 – aniline, 6 – ethanol.
Experiments on PMMA (polymethyl methacrylate, the only polymer obtained by polymerization of the monomer directly [8]) samples have been carried out according to the procedure described above. During the pulse experiment, the weight-averaged probe temperature was increased from $T_0 \approx 295$ K up to the value of 675 K. Heating pulse length was 3 ms. Evaluation of the thickness of the heated polymer layer is shown in Figure 8. The probe radius and the heating parameters are taken from the real experiment.

![Figure 8](image.png)

**Figure 8.** Calculation of the temperature field in a direction normal to the probe surface ($r = 0$): polymer temperature rise reduced to temperature rise at the probe surface versus distance to the probe surface ($r$). The pulse length serves as a parameter: $t = 1$ ms (curve 1); 2 ms (2); 3 ms (3).

PMMA properties were calculated with varying coefficients of polynomial functions of the second order describing the expected temperature dependencies of thermal conductivity and volumetric heat capacity. Some results of this calculation are presented in Figure 9. One can see that difference between the recovered values for the properties increases with increasing the probe temperature. The recommended $\lambda(T)_h$ dependence was found by formal averaging over the ensemble of the solutions obtained. The corresponding values of $\rho c_p(T)_h$ were found taking into account just the previous solution for $\lambda(T)_h$. The recommended dependencies are written as follows:

$$\lambda(T)_h = -6.89586 \cdot 10^{-8} \cdot T^2 + 4.93216 \cdot 10^{-5} \cdot T + 0.18683 \text{ [W/(m·K)]},$$

$$\rho c_p(T)_h = -8.01129 \cdot T^2 + 4210.97331 \cdot T + 1.51604 \cdot 10^6 \text{ [J/(m^3·K)].}$$

The behavior of the thermophysical properties of polymers under the conditions of pulse local heating in a wide range of heat release power is almost unknown [10]. The known data were obtained in a relatively narrow range of temperature. Moreover, they have shown an essential scatter in absolute values and, paradoxically, may differ in the sign of temperature dependency. This makes attempts to extrapolate the data into the elevated-temperatures region extremely unreliable. The presented above approach with short-term experiment on a miniature fast-response wire heater implanted in the polymer sample is believed to be more reasonable. The authors' experiments show the possibility of measurements on small samples with a volume of several cubic millimeters. In turn, the procedure of
preparation of a set of polymeric samples with close molecular-weight distribution appears to be rather easy. Some uncertainty occurs with respect to the wire probes dimensions that are hard to determine directly. Preliminary experiments with nearly adiabatic pulse heating of wire probes in the air allow determining the basic values of length and diameter of wires for the subsequent numerical calculations.

![Figure 9](image-url)

**Figure 9.** A set of temperature dependences of thermal conductivity (left plot) and volumetric heat capacity (right) for PMMA obtained in this work. Thick lines correspond to the recommended computational results.

### 6. Conclusion

A device for programmable heating of a thin wire probe in various media has been developed. Depending on the measurement task, different heating modes can be selected. The examination of substances in the superheated state often requires rapid initial heating, followed by keeping the probe temperature close to the preset value. In this case, the double-pulse heating mode is used to record temperature changes in the second pulse section. Anomalous temperature deviations indicate changes in the substance and can be used, for example, to detect volatile impurities in transformer oil.

High accuracy and repeatability of digital device signals (both in single- and double-pulse modes) allows selecting the most suitable (with respect to the task details) heating conditions. The condition of the heating power constancy, which is necessary to determine the thermal conductivity of the substance, can be fulfilled automatically as a result of the software iterative selection of the optimal heating current values. This mode is used in the case of long-term measurements, when the parameter is the composition or temperature of the sample (Section 4). Another mode, the polynomial model coefficients selection mode (Section 5), is useful in case of possible irreversible changes in the samples, e.g. due to thermal decomposition of polymers. Step-by-step increase of heating power allows to determine the area of short-term thermal stability of the polymer on the basis of violations of thermogram monotony and select, on the basis of a single heating pulse, the most informative set of coefficients to evaluate the properties of a thermally unstable sample.

Finally, the device allows performing short-time measurements over a wide temperature range, including superheated states of a substance.
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References
[1] Starostin A A and Skripov P V 2019 Methodology for Measurements of the Properties of Not Fully Stable Fluids Proc.12th Int. Conf. on Measurement (Bratislava: Institute of Measurement Science, Slovak Academy of Sciences) p 75–8
[2] Echmaev S B and Zhukov S A 2013 Study of resistance of metastable states under repeated thermal impulse loads High Temperature 51 876–7
[3] Kozulin I and Kuznetsov V 2018 The dynamic of the water explosive vaporization on the flat microheater Journal of Physics: Conference Series 1105 (1) 012061
[4] Lukynov K V, Starostin A A and Skripov P V 2017 Heat transfer under high-power heating of liquids. 4. The effect of water admixtures on the heat transfer in superheated hydrocarbons Int. J. Heat Mass Transfer 106 657–65
[5] Starostin A A, Shangin V V, Bukhman V G, Volosnikov D V and Skripov P V 2016 Development of a Self-Contained Device for Rapid Detection of Volatile Impurities in the Oil System of a Turbine Therm. Eng. 63 544–50
[6] Assael M J, Antoniadis K D and Wakeham W A 2010 Historical Evolution of the Transient Hot-Wire Technique Int. J. Thermophys. 31 1051–72
[7] Rutin S B, Gal’kin D A and Skripov P V 2017 Returning to classic conditions of THW-technique: A device for the constant heating power mode Int. J. Heat Mass Transfer 115 769–73
[8] Starostin A A, Puchinskis S E, Efremov V P and Skripov P V 2015 Investigation of not fully stable fluids by the method of controlled pulse heating. 2. Short-term thermal stability of polymethyl metacrylate Thermochim. Acta 609 31–5
[9] S.B. Rutin, A.A. Smotritskiy, A.A. Starostin, Yu.S. Okulovsky, P.V. Skripov 2013 Heat transfer under high-power heating of liquids. 1. Experiment and inverse algorithm, Int. J. Heat Mass Transfer 62 135–41
[10] Pavlov P A and Skripov P V 1999 Bubble nucleation in polymeric liquids under shock processes Int. J. Thermophys. 20 1779–90