

Conference Paper

Optoelectronic Methods of IR-Photometry in Solving Thermal and Physical Problems

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Abstract

Results of the application of optoelectronic IR-photometry methods for solving the actual thermophysical problems using high-speed photodiode temperature (pyrometric) sensors are presented. The latest achievements of the Ioffe Institute in the field of middle IR spectral range (3-6 μm) photodiode production technology are used in the sensor development. The above-mentioned middle-IR photodiode sensors are based on heterostructures from A_3B_5 solid solutions and they do not require forced cooling. New data on thermophysical processes taking place under the complex experimental conditions, including dynamically changing properties of the object, have been obtained. The new experimental results are based on high-speed non-contact measurements of the absolute temperature of objects directly in the exposure region of laser radiation and / or powerful electromagnetic fields.

In the context of creating new experimental techniques for determining the thermophysical parameters of new promising materials the efficiency of middle-IR photodiode temperature sensors has been shown. In particular, a simple and effective measuring method for determining the heat transfer coefficient of solid objects and the pyroelectric coefficient of ferro- and pyroelectric materials is offered. The measuring method is based on laser thermowave techniques and direct non-contact measurements of the sample surface temperature under the pulsed / periodic laser action. The proposed experimental techniques make it possible to significantly improve the accuracy of measurements of thermophysical parameters of materials, and in particular, to eliminate a considerable spread of data on the parameters of the pyroelectric effect in ferroelectric materials.

Keywords: Pyrometric sensors, mid-IR photodiodes, laser thermowave techniques, heat transfer coefficient, pyroelectric coefficient

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1. Introduction

The development of various fields of science and technology is closely related with the study of thermal properties of new promising materials and technologies and at present it requires the creation of contactless temperature control systems (pyrometers) with new measurement capabilities. The new measurement capabilities of pyrometry are associated with the expansion of the measurements range in the region of low temperatures (from the pyrometry point of view, it corresponds to temperatures below 300 °C) while maintaining a high energy and time resolution. The significant moment is the reduction of the radiation measurement procedure error associated with the influence of the object itself and the intermediate medium properties on the true values of temperature results. The analysis of the fundamental Planck radiation law makes it possible to determine the optimal spectral region with maximum sensitivity temperature measurements for a given temperature range. For temperatures of 100-300 °C this corresponds to the mid-IR spectral range: 2.7-4.05 μm . The sensitivity lines of A_3B_5 photodiodes (PD) suggested by us earlier for low-temperature pyrometric sensors [1] precisely corresponds to this optimal spectral range. The high-speed response (~ 20 ns), detection ability ($D^* \sim 2 \cdot 10^{10} - 10^{11} \text{cm} \sqrt{\text{Hz}} / \text{W}$), Si- immersion lens configuration and the lack of cooling are far from being the only features of the mid-IR PDs attractive for modern temperature sensors.

The relative spectral narrowband provided by the configuration of the PDs under consideration makes it possible to implement simple methods for pyrometric sensors calibrating procedure directly at the workplace and also to create multispectral "sandwich" structure PDs [2]. The allocation of several operating spectral ranges of the pyrometric sensor directly in the PD heterostructure makes it possible not only to substantially simplify the optical and mechanical scheme of the measuring device, but also to significantly reduce its dimensions, mass and power consumption. But the main thing is that the accuracy and reliability of pyrometric measurements are significantly improved due to the information redundancy of the sensor. The original technical, circuitry and design solutions of the developed mid-IR dual-spectral (two-color) PD sensors made it possible to create a number of unique experimental setups for solving complex thermophysical problems:

- for the first time, the dual-spectral mid-IR PD pyrometric sensor allowed us to carry out temperature monitoring of the microwave sintering process of silver nanoparticles (AgNPs) onto polyethylene terephthalate (PET) film substrates [3].

Such control is necessary to solve the task of developing the most effective compositions and inkjet-printing modes for electronic and photonic functional films. The two-spectral PD pyrometric sensor provides accurate (not worse than 1%) measurements of the absolute temperature and its fast (tens of μs) fluctuations in the region of 100-300 C for a PET-based substrate with solvent-absorbing layers with optical properties changing under various microwave power levels and heating periods. The design of the sensor ensured the possibility of using various optical schemes for delivery of the object (film) heat radiation: lens with adjustable focal length or fiber optic radiation input (sapphire fiber). The fiber-optic input allows local spot (up to 400 μm) measurements of the dynamics of temperature changes within large film samples.

- The high-speed PD dual-spectral pyrometric sensor with the time resolution of up to 1 μs and measurement range from ≈ 200 to 5000 $^{\circ}\text{C}$ was used in an experimental setup for investigating the cyclic action of heat fluxes generated by a plasma gun on various materials used in Fusion Engineering Research Reactors. The interaction of plasma with the surface is an important problem when creating protective materials for thermonuclear reactors. Experiments on the interaction of tungsten elements with hydrogen, deuterium, and helium plasmas have shown that it can lead to a change in structure, the development of porosity, and the embrittlement of reactor structural materials [4]. The duration of the pulses created by the plasma gun did not exceed tens of microseconds, and the high temporal resolution necessary for surface temperature monitoring during such a short time interval can not be realized by any known measuring tools other than by using high-speed mid-IR PD pyrometric sensor.

A wide range of modern physical problems is connected with measurements of thermal [5], optical [6] and pyroelectric [7] parameters of materials and with the investigation of the influence of structural and contact inhomogeneities on heat transfer processes [8-11]. In particular, serious attention has been paid in recent years to study the pyroelectric properties of new promising materials for IR radiation sensors and energy storage devices (energy harvesters) [12, 13]. The most important characteristic of these materials is the pyroelectric coefficient that determines the magnitude of the pyroelectric and electrocaloric effects of the a test material. Currently, the transient heating methods using the laser radiation of samples are widely used in pyroelectric experiments [14-17]. In this case the thermal pulse (TP) method [17] and laser intensity modulation method (LIMM) [14-16] are used. Literature review and published data analysis show that in the vast majority of experiments the evaluation of the material

pyroelectric coefficient is based on indirect calculations of the sample temperature changing under conditions of thermal laser action. However, the correctness of the experimental data interpretation requires the direct non-contact sample temperature change control directly in the region of the thermal laser radiation action. In addition, most pyroelectric experiments are performed under conditions for which the face surfaces of the samples are in contact with the surrounding gaseous medium (usually air). In this case, an important thermophysical parameter is the heat transfer coefficient, " H " [11], indicative of the efficiency of heat exchange of a sample with the environment in specific experimental conditions. It can also be an important source of complementary error in thermophysical experiments. The heat transfer coefficient is an experimentally difficult to control parameter, it depends on specific experiment conditions and requires special studies [18]. Therefore, as follows from the publishing data, when carrying out TP and LMM pyroelectric experiments with ferroelectric materials, the heat transfer of the sample is usually considered to be negligibly small and in the calculations it is assumed that $H = 0$ [5]. At the same time, even in vacuum conditions, the heat transfer coefficient has a nonzero value as a result of thermal radiation [19]. Thus, when carrying out pyroelectric measurements, it is important to have techniques that provide not only direct contactless measurements of the sample temperature, but also independent measurements of the heat exchange coefficient and pyroelectric signal of the sample under the conditions of the same experiment.

In the present paper, the problem of increasing the reliability and accuracy of determining the pyroelectric coefficient of ferroelectric and pyroelectric materials is solved. The efficiency of the use of mid-IR PD pyrometric sensors to create new laser thermal waves experimental methods for determining the material pyroelectric coefficient is shown.

2. Materials and methods

To determine the material pyro-coefficient, taking into account the sample heat transfer effect, we proposed and used a technique based on the dynamic thermal laser action on the sample in the form of a "step" (or a single rectangular pulse), which simultaneously recording the dynamics of the temperature change of its surface and the pyroelectric response. An experimental verification of the method was carried out in [20] on a bulk sample of a well studied ferroelectric material-lithium tantalate monocrystal (LiTaO_3 , Z-cutoff, sample thickness 0.615 mm).

For the first time, in this paper the experimental results of measuring the pyroelectric coefficient of an AlN crystal bulk sample (thickness $L = 0.315$ mm) and AlN 10-12 μm thick films grown on substrates of various types (Si and SiC with a thickness of 400 μm) are presented. The samples of AlN films were prepared at the Ioffe Institute. The test samples were elements of an arbitrary shape with linear dimensions of at least 10 mm, considerably exceeding their thickness. To register the pyroelectric signal, metal (Ag) electrodes were placed on the samples, which completely occupied its surfaces. They were covered with a thin layer of absorbing material to increase the absorption coefficient of laser radiation and the surface emissivity for pyrometric temperature measurements.

2.1. Experimental setup.

The uniform illumination of one of the surfaces of the sample by laser radiation causing its heating was performed in the experimental setup. A block diagram of the experimental setup is shown in Fig. 1. A semiconductor laser with a wavelength $\lambda = 638$ nm and an adjustable radiation power of 0.05-0.3 W permitting the modulation of the radiation power from an external signal generator of an arbitrary shape was used as the source of thermal waves in the test samples. The laser beam uniformly illuminated the surface of the sample freely lying on the annular holder inside the cell with sapphire windows, transparent for exciting laser radiation and recorded thermal radiation. Such a design enabled the non-contact pyrometric control of the temperature of both surfaces of the sample. Optoelectronic sensors based on the immersion lens PD type PD42SrNB [21] sensitive in the range $\Delta\lambda = 3.4$ -4.4 μm (0.5 level) were used to monitor the sample surface temperature. In this case, the important advantage of immersion lens PDs was their complete insensitivity to radiation with $\lambda \leq 1$ μm , which makes it possible to use a variety of lasers to excite thermal waves. The PDs electronic circuitry provided the detection of sample surface thermal radiation in the 2 kHz band and generated the output volt signal proportional to the test sample surface temperature. The current-voltage conversion pyroelectric measurement circuit was used to monitor the sample pyroelectric response. The total conversion factor of the pyroelectric current to the measured voltaic signal (Pyroelectric voltaic signal) was $4 * 10^8$ V/ A.

A part of the radiation from a semiconductor laser was transferred to a Si-photodetector using a semi-transparent mirror to control the amplitude and shape of

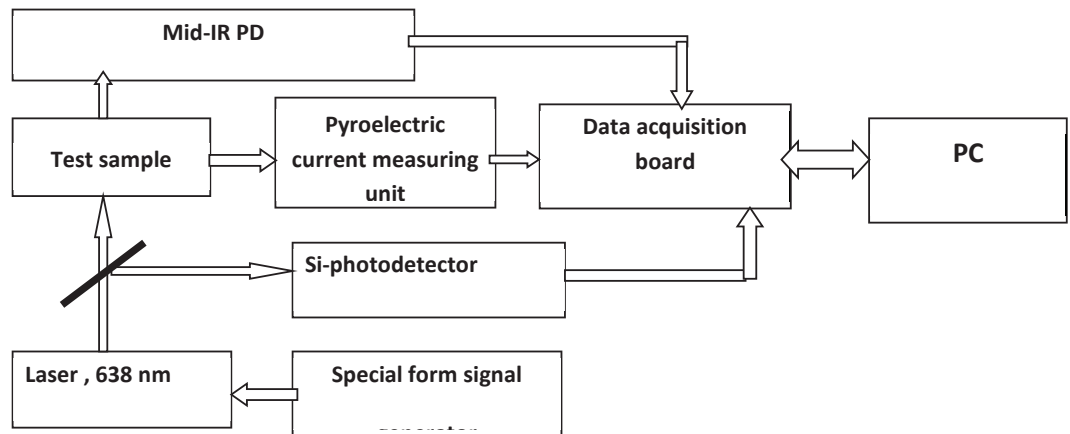


Figure 1: Block diagram of the experimental setup for studying the pyroelectric properties.

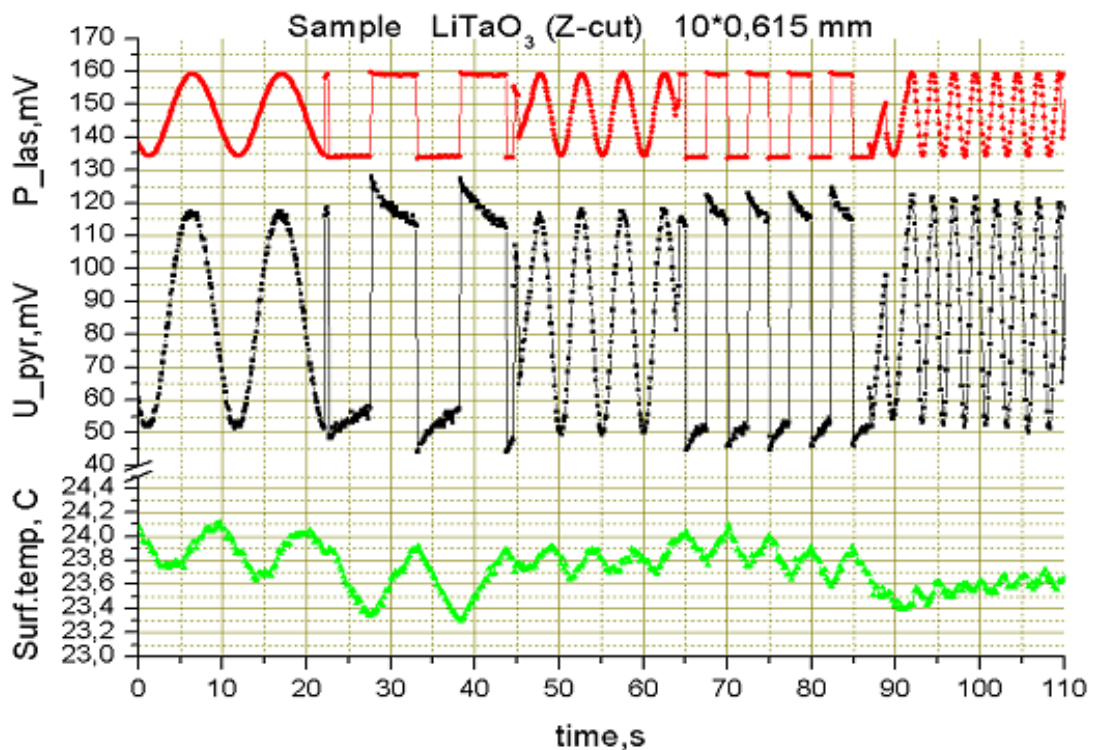


Figure 2: Pyroelectric experiments using the laser intensity modulation (LIMM) method and direct non-contact surface temperature measurements of the LiTaO₃ volume sample.

the laser radiation exciting the thermal wave in the test sample. The signal from the Si-photodetector (named "P_{las}") entered the data acquisition/ transmission circuit.

The data acquisition/ transmission board is based on a ADuC 7026 microcontroller for the precision processing of analog signals. It contains a 16-channel 12-bit ADC and provides simultaneous digitization and transmission of all measuring signals to the PC. The experimental setup software provided on-line calculation and display of the

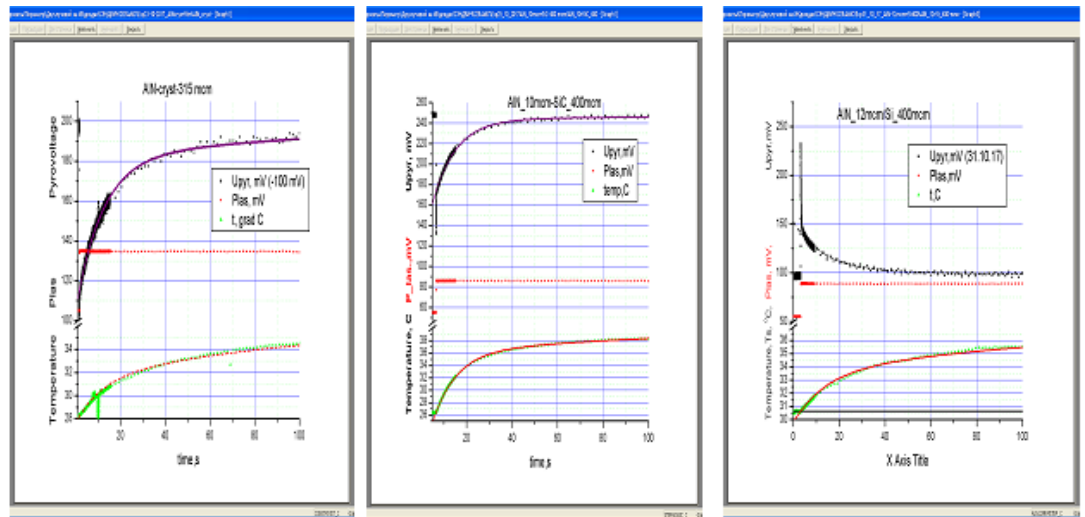


Figure 3: Experimental results of measuring the dynamics of the AIN samples surface temperature and pyroelectric response under the stepwise laser action with radiation power 0.1 W for the AIN single crystal, L= 315 μ m (a) AIN film L=10 μ m on a SiC substrate (b) and AIN film L=12 μ m on a Si substrate (c).

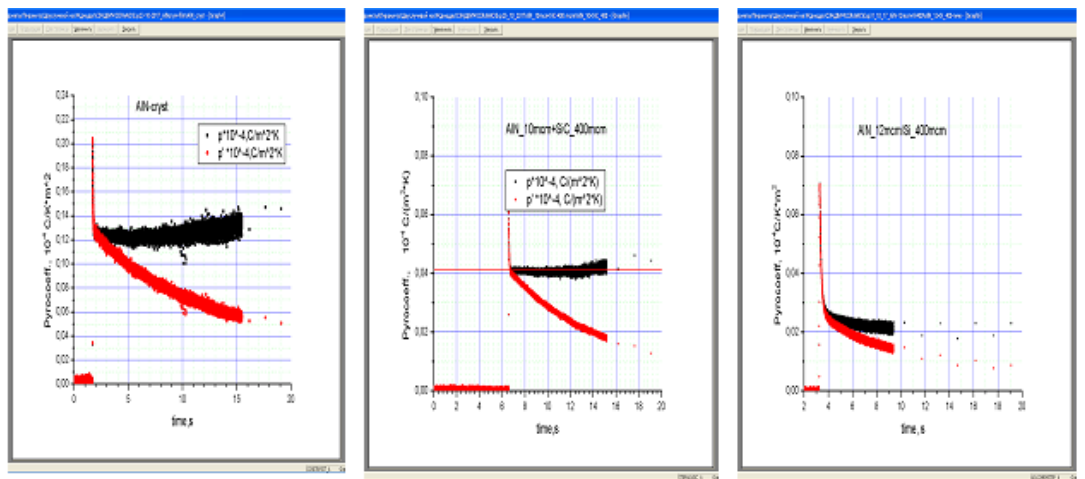


Figure 4: The results of the calculation of the pyroelectric coefficient with taking into account the experimentally determined value of the heat transfer coefficient H (\bullet) and under the assumption of $H=0$ (\circ): the AIN single crystal, L= 315 μ m (a) the AIN film L=10 μ m on a SiC substrate (b) and AIN film L=12 μ m on a Si substrate (c).

test sample surface temperature values in the range from 20 to 200 $^{\circ}$ C with accuracy not worse than 0.1 $^{\circ}$ C simultaneously with the recorded voltaic pyroelectric signal and “P_las” signal with a time resolution 1.47 ms.

As an example, Fig. 2 shows the experimental results of the output pyro-electrical response and the sample surface temperature modulation when a 1-Hz sinusoidal laser radiation is applied to a LiTaO₃ test sample.

2.2. Theoretical basis

For samples with linear dimensions significantly exceeding their thickness, the heat removal from their ends does not play an important role, since their total area is much smaller than the area of the facial surfaces. Under these conditions, the temperature distribution within the sample can be found using the one-dimensional heat equation

$$\rho C \frac{\partial T}{\partial t} = K \frac{\partial^2 T}{\partial x^2} + Q(x, t), \quad (1)$$

where “ ρ ” is the density of the sample material, “ C ” is its specific heat, “ K ” is the thermal conductivity, $Q(x, t) = \alpha(1 - R) I_0 e^{-\alpha x} f(t)$ is the power density of the thermal source generated by the laser radiation, “ α ” is the radiation absorption coefficient of the sample surface, “ I_0 ” is the intensity of the laser radiation, “ R ” is the sample surface light reflection coefficient, and $f(t)$ is the function describing the time dependence of the thermal effect.

It is necessary to set the boundary conditions to solve equation (1). For samples whose faces are located at $x = 0$ and $x = L$, taking into account the heat sink to the environment, these conditions have the form

$$K \left. \frac{\partial T}{\partial x} \right|_{x=0,L} = H(T - T_0) \Big|_{x=0,L}, \quad (2)$$

where H is the heat transfer coefficient, T_0 is the ambient temperature.

It is easy to find the temperature dependence of the sample surface T_s as a function of time by integrating equation (1) in terms of its volume. Using the Gauss theorem and taking boundary conditions (2) into account, we obtain this dependence in the form

$$T_s(t) = \frac{I_0}{\rho C L} \int_{t_1}^t dt' e^{-\frac{2H}{\rho C L}(t-t')} f(t') + T_0. \quad (3)$$

With the laser thermal action $f(t)$ in the form of a single rectangular pulse the solution of equation (3) will have the form:

$$T_s(t) = \frac{I_0}{2H} \cdot \left(1 - e^{-\frac{2H}{\rho C L} \cdot (t-t_1)} \right) + T_0, \quad \text{if } t_1 < t < t_2, \quad (4)$$

$$T_s(t) = T_s(t_2) \cdot e^{-\frac{2H}{\rho C L} \cdot (t-t_2)}, \quad \text{if } t > t_2,$$

where t_1 is the time of beginning of laser action, t_2 is the end time.

As can be seen from the expressions (4), measurements of the dynamics of the temperature change of a surface with known values of the parameters of the samples ρ , C and L under the action of a stepped laser action make it possible to determine the value of the heat transfer coefficient (H). In this case, the value of H determines the

transient thermal process relaxation time, $\tau_{th} = \rho CL / 2H$, which arises in the sample under the influence of laser radiation.

It is generally accepted that the heat transfer coefficient does not depend on the sample material, but it depends on the method and quality of its surface treatment and experimental conditions. The minimum value of the heat transfer coefficient is achieved for smooth surfaces and under conditions of free air convection it is $5.6 \text{ W / m}^2 \cdot \text{K}$ [22]. The value of H obtained by us [23] in the same experimental setup, for pure metal samples of *Ni* and *Li* with the similar linear dimensions was equal to $20 \text{ W/m}^2\text{K}$, which is in good agreement with the data ($5.6 \leq H \leq 25 \text{ W/m}^2\text{K}$) reducible for conditions of free air convection given by [22].

Taking into account (4) and under the condition of homogeneity of the pyroelectric effect along the thickness of the sample, the value of the pyroelectric current caused by the stepwise laser action can be written in the form:

$$i_p(t) = p \cdot A \cdot \frac{dT_s}{dt} = p \cdot A \cdot \frac{I_0}{\rho CL} \cdot e^{-\frac{2H}{\rho CL} \cdot t}, \quad (5)$$

where A is the area of sample electrodes, p is the integral (on the thickness of the sample) material pyroelectric coefficient.

Thus, expressions (4) and (5) show that independent measurements of test sample surface temperature and pyroelectric signal for known parameters of the sample material ρ , C and L , make it possible to determine its pyroelectric coefficient of the material under study taking into account the actually present convective heat exchange with the surrounding medium. The expressions obtained also allowed us to estimate the error in measuring the pyroelectric coefficient of the sample, neglecting its heat exchange with the surrounding medium.

3. Results and Discussion

Figure 3 shows the experimental results of measuring the dynamics of the AlN samples surface temperature and pyroelectric response under the stepwise laser action with the radiation power 0.1 W for the AlN single crystal test sample (Fig. 3a), AlN films test samples of thickness $L = 10 \text{ }\mu\text{m}$ on a SiC substrate (Fig. 3b) and $L = 12 \text{ }\mu\text{m}$ on a Si substrate (Fig. 3c). The different polarity of the pyroelectric response of the samples in Figs. 3a-c is due to the accidental connection of the sample electrodes to the pyroelectric measurement circuit. As follows from the obtained experimental data, the transition characteristics for the samples surface temperature and pyroelectric response on sufficiently long time intervals are well approximated by exponential

functions (4) (solid lines) and they allow us to calculate the relaxation times τ_{th} of different test samples and the corresponding heat transfer coefficient. As a result of the calculations, the values of the relaxation time constants for the single crystal and AlN films were found to be $\tau_{th} = 100-125$. The corresponding values of H lie in the range 20-30 W/m²K. The spread of the values of H can be determined by the different geometric dimensions of the sample surface.

It is clearly seen from the experimental data presented in Fig. 3 that in the initial sections of the pyroelectric response transition processes that have much shorter relaxation times τ_0 than the observed thermal processes are present ($\tau_0 \approx 20-70$ ms $\ll \tau_{th}$). The nature of these processes requires additional analysis and experimental studies, but it is obvious that their presence can significantly distort the estimates of the pyroelectric coefficient of materials carried out by various methods, including high-frequency ones, in various experimental setups.

The results of the calculation of the pyroelectric coefficients of the samples under consideration, taking into account the data on the heat dissipation (black dot) and assuming $H = 0$ (red dots) are shown in Figures 4a-c. As can be seen from the characteristic curves of Fig. 4, the frequently used practice of measuring the pyroelectric coefficient immediately after the laser radiation applied to the sample (at times $t < \tau_0$) gives an overestimate value of the coefficient for all the samples under study. For times $t \gg \tau_0$, the pyroelectric coefficient measurement mode can be considered quasi-stationary. In this case, according to (3), in order to calculate the pyroelectric coefficient from the measurements of the pyroelectric current, it is necessary to multiply its value by an exponent with a time constant depending on H . When processing the experimental data for $t > \tau_0$, we used the values of H obtained for each of the samples from an analysis of the dynamics of the surface temperature. At times $t \ll \tau_{th}$, the approximation $H = 0$ does not introduce a significant error in such a procedure, but it limits the data averaging time of the pyroelectric coefficient calculation procedure. In addition, the presence of various transient processes in this area does not make them completely reliable.

Measurements of the values of the pyroelectric response together with the sample surface temperature and the calculation of the material pyroelectric coefficient from these data under quasi-stationary conditions at $t \rightarrow \tau_{th}$ are not only more reliable, but also provide higher accuracy. The maximum averaging time for these calculations is limited only by the possibility of detecting a pyroelectric current with a sufficiently high signal-to-noise ratio.

The obtained results show that thermowave technique with a stepwise laser action on the sample together with the direct noncontact measurement of its surface temperature can be recommended for determining the pyroelectric coefficient of bulk and film materials without the use of special calorimetric equipment. This technique takes into account the presence of transient processes immediately after the onset of laser thermal action and estimates pyroelectric coefficients taking into account the heat transfer to the environment.

The developed method of the contactless determination of the surface temperature of a sample with an accuracy of not worse than 0.1° opens up wide opportunities for investigating pyroelectric and electrocaloric effects, both in bulk materials and in thin films, including a wide range of sample temperature variation and applied electric fields. This opens up wide experimental possibilities for studying new ferroelectric materials (in particular, relaxors [24]) and direct methods for measuring the electrocaloric effect [25]. The polarization effects that are inseparably associated with pyro- and electrocaloric effects, which are the fundamental characteristic of pyro- and ferroelectrics, can also be investigated by this method. The proposed experimental method for studying pyroelectric and electrocaloric effects in bulk and film materials has no analogues in its measuring capabilities.

4. Conclusions

Thus, the possibilities of the mid-IR PD pyrometric sensors and the experimental techniques developed on their basis for solving a wide range of physical-technical problems are demonstrated:

1. in the field of high speed (up to units μs) object true temperature measurements and the materials phase transformations controlling in the thermonuclear reactors and in technological processes with over a wide temperature range from tens to thousands of degrees Celsius
2. when determining of thermophysical parameters of promising materials by means of the laser thermowave techniques in a wide range of temperatures, with a high accuracy (no less than 0.1°C) up to the lower threshold of measurement to tens of degrees Celsius
3. to improve the accuracy of the determination of the pyroelectric coefficient of bulk and film materials when complex using of direct contactless temperature measurements and laser thermowave techniques

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