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Application of the Thermal Diffusivity Standard for the Heat Transfer Parameter Control in Absorbing Materials

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Abstract

Metrological support creation and use of heat transfer etalons are important stages in the development of modern materials science. This is especially concerned to the emergence of new materials in the world with previously unattainable thermophysical parameters. The purpose of this work was to develop and experimentally verify the idea of joint application of the transient gratings method which is well-known in nonlinear optics and the single thermal diffusivity etalon of conventional type for the heat transfer metrological control in materials of a wide values range. The method proposed is based on thermal diffusivity etalon application as a source of calibrated optical signals that are excited in it by short laser pulses. Their lifetime is formed by the etalon thermal diffusivity and on the transient grating spatial period. The etalon linear graph of gratings lifetimes as a function of the gratings periods squared and grating lifetime of the material under study are used for the thermal diffusivity calculation. Thermal diffusivity of thin sub-surface layers of the samples under study – duraluminium, monocrystalline silicon and thermoelectric lead telluride film was measured. The results obtained are in close agreement with the reference values.

Keywords: transient gratings, etalon of thermal diffusivity, metrological control, sub-surface layers

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Применение эталона температуропроводности для контроля параметра теплопереноса в поглощающих материалах

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Создание, контроль и постоянное использование эталонов теплопереноса являются важнейшими факторами в развитии современного материаловедения. Это в особенности актуально в связи с появлением новых материалов с недостижимыми ранее теплофизическими параметрами. Целью работы являлась разработка и экспериментальное апробирование идеи совместного применения известного в нелинейной оптике метода динамических решёток и одного эталона температуропроводности стандартного типа для метрологического контроля параметров теплопереноса в материалах с широким диапазоном значений. Предложенный метод основан на использовании стандартного эталона температуропроводности как источника калиброванных оптических сигналов, возбуждаемых в нём короткими лазерными импульсами. Их длительность определяется коэффициентом температуропроводности эталона и периодом динамической решётки. Последний легко контролируется средствами современной оптики. Построенный с помощью эталона линейный график зависимости времени жизни множества динамических решёток от квадрата их периодов в сочетании с измеренным временем жизни решётки в исследуемом материале позволяют вычислить коэффициент температуропроводности. В приповерхностных слоях микронной толщины в трёх образцах – дюралюминий, монокристаллический кремний и термоэлектрик теллурид свинца – проведены измерения искомых параметров. Результаты тестирования близко соответствуют справочным значениям.

Ключевые слова: динамические решётки, эталон температуропроводности, метрологический контроль, поверхностный слой

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Introduction

The heat transfer standards reproduction and storage are currently based on the comprehensively studied materials application [1]. Metrological value is supported by using a set of several measures and its implementation at a given temperature can be carried out only at a separate points of the mastered metrological range. The need to expand the range of measures, insufficiency of the existing set of measures and nonuniform of their distribution over the range of thermal diffusivity are problems that are relevant and have been discussed for a long period of time [2–4]. However, their solution by traditional means is a very long and expensive procedure. The substance used as a reference measure must have chemical inertness, physical homogeneity, non-hygroscopicity, absence of phase transitions, stability of properties over time, low cost, etc.

In [5] an attempt is made to create a new class of metrological control devices – a multivalued etalon of thermal conductivity. The essence of the proposed approach is to create a standard with controlled internal heat sources, which, depending on their power and distribution in space, form a specific thermophysical parameter, and it is then used for metrological control of materials. However, for a number of objective reasons of a fundamental nature, this idea have received a negative assessment by the experienced specialists (see, for example, [6]).

The aim of this work was to develop a method for multi-valued metrological thermal diffusivity control of a material in an extended range of values by using a standard unambiguous standard in combination with the transient grating method application. The method was tested by samples application with well-known thermal parameters.

Transient grating application for the metrological problem of heat transfer solving

Patent [7] proposes the idea of solving one of the metrological problems on the basis of the transient gratings method application, which is now widely used in the scientific world [8–10]. According to the method, two interfering beams from a pulsed laser light up the sample at an angle Θ to each other. In this case, an interference pattern is formed in the form of light and dark rectilinear equidistant bands following the period Λ, depending on the angle between the beams $\Lambda = \lambda/2\sin(\theta/2)$. Due to the absorption of light, spatially periodical heating of the sample occurs on the surface or in volume, which leads to a phase diffraction grating formation with the same period Λ. The third light beam from the continuous wave laser is directed to the sample and the light beam undergoes diffraction with the formation of diffraction orders of plus and minus the first orders. A diagram of a laser device for determining the thermal diffusivity of materials is shown in Figure 1. In different embodiments, it is given in numerous works concerning the transient gratings recording or application (see, for example, [11]).

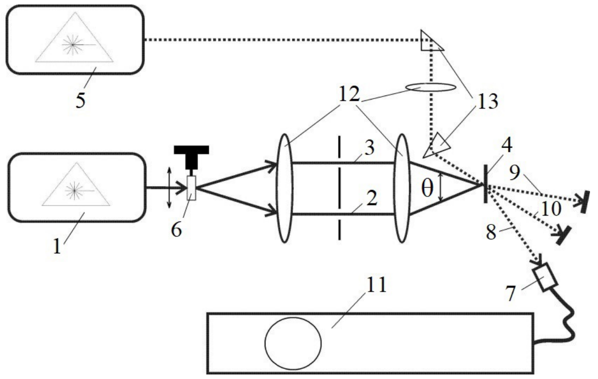

Figure 1 – Diagram of a laser-based device for the thermal diffusivity of materials measurement: 1 – source of pulsed laser radiation; 2, 3 – interfering light beams; 4 – sample under test; 5 – continuous wave laser; 6 – special diffraction beam splitter; 7 – detector for the diffracted signal kinetics recording; 8, 9, 10 – diffracted beams of zero and of the ±1-orders; 11 – digital system for the diffraction signal recording and processing; 12, 13 – optical elements for light beams bringing to the sample under study

According to the early developed theory [12] for three-dimensional transient gratings, in the approximation, which is obviously performed when making measurements, the intensity of the signal of the first order of diffraction varies according to the exponential law:

$$I(t) = I(0)\{\exp(-t/\tau)\}^2, \quad (1)$$

where the diffraction signal decay τ is inversely proportional to the thermal diffusivity χ of the sample under study in the direction of the grating vector:

$$\tau = \Lambda^2/8\pi^2\chi. \quad (2)$$

If the heating of the sample is essentially of surface nature and therefore the thermal gradient is formed simultaneously in two directions – along the sample surface (due to spatially periodic heating) and along the normal to it, the diffracted signal decay is recorded through an complementary error function [12]:

$I(t) = I(0)\ \{\mathrm{erfc}(t/\tau)^{0.5}\}^2$; (3)

$\tau = \Lambda^2/4\pi^2\chi$. (4)

From the above relations it follows that in order to measure χ by the transient grating method, it is necessary to experimentally determine two values: grating period and its lifetime. The grating period can be measured for example, by using a standard microscope equipped with an eyepiece-micrometer. The time constant τ is determined by standard comparison of the experimental kinetics of diffraction signals recorded with the theory in accordance with the relations (1) or (3) depending on the type of grating (volume or surface).

Metrological support of the thermal diffusivity measurements begins with the calibration of the measuring setup, shown in Figure 1. For this purpose a stainless steel etalon of thermal diffusivity No. MTO 01.01.005-30/062, manufactured and officially certified at the Research Institute of Metrology named after D.I. Mendeleev, St. Petersburg, is used. According to the passport attached to the etalon, its thermal diffusivity is $\chi_{\mathrm{etalon}} = 0.04$ cm^2/sec. Further, using the etalon as the sample under study, and the transient grating recording of different grating periods, a linear calibration graph $\tau_{\mathrm{etalon}}(\Lambda_{\mathrm{etalon}})^2$ should be constructed, as shown in Figure 2. The scale along the x axis from $x = 0$ to the maximum value $x = (\Lambda_{\mathrm{etalon}})^2$ is selected depending on the range of measured parameters expected.

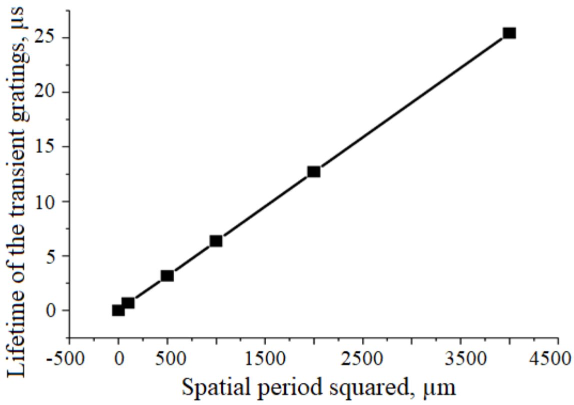

Figure 2 – Calibration graph $\tau_{\mathrm{etalon}}(\Lambda_{\mathrm{etalon}})^2$

Then, transient grating with an arbitrarily selected period Λ_x is excited in the sample under study and lifetime of the transient grating τ_x is determined by standard interpolation.

With the help of the graph in Figure 2, it is found to which period of the thermal grating $\Lambda_{\mathrm{etalon}}$ the equality $\tau_{\mathrm{etalon}} = \tau_x$ is valid. The desired value of thermal diffusivity χ_x is calculated using the formula below:

$$\chi_x = \chi_{\mathrm{etalon}} \left(\frac{\Lambda_X}{\Lambda_{\mathrm{etalon}}} \right)^2 . \quad (5)$$

It is convenient to determine the value of the $\Lambda_{\mathrm{etalon}}$ using the $\tau_{\mathrm{etalon}}(\Lambda_{\mathrm{etalon}})^2$, stored directly in the rather popular program OriginPro8. In this case, it is also advisable to use the Screen Reader option within this program. Using the manual movement of the crosshair, we find the point on the line graph that gives $Y = \tau_x$. At the same time, we find the value of $\Lambda^2_{\mathrm{etalon}}$ and calculate the thermal diffusivity of the sample under study using the ratio (5).

Results of control measurements

It is worth to note that since all the necessary information when performing measurements is taken from optical radiation, the surface and volume of the sample under study should be of high optical quality with minimal losses caused by light scattering.

Control measurements of thermal diffusivity were performed for duraluminium and silicon samples. The specimens are made in the form of plates 30×30 mm and 2 mm thick, one surface of which is polished to optical quality. The heat transfer in the semiconductor film of the currently popular narrow-bandgap thermoelectric PbTe with a thickness of 2.3 μm on a glass substrate was also studied. In all samples excited by laser beam at wavelength 0.532 μm, the surface heating is realized.

Duraluminium grade D16T

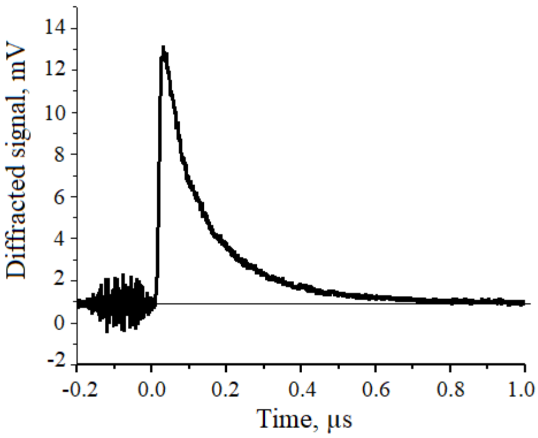

Figure 3 – Kinetics of the diffracted signal decay at surface excitation of duraluminium. Grating period is $\Lambda_x = 25$ μm. Its lifetime according to comparison with theory (3), is 0.31 μs

At Λ_x = 25 μm, by using the plot τ_{etalon} $(\Lambda_{etalon})^2$ and the OriginPro8 program, the value of Λ_{etalon}. is determined. By using the ratio (5), thermal diffusivity of sample was found to be χ_x = 0.496 cm^2/s. From the reference data [13] it follows that the thermal diffusivity of duralumin grade D16T, as the result of dividing its thermal conductivity by the bulk heat capacity, lies in the range of 0.48–0.51 cm^2/s.

Monocrystalline silicon

With this semiconductor, all the same actions are performed as with duralumin. At the grating period Λ_x = 25 μm, diffraction kinetics with a decay time of 0.21 μs was registered. It was found that in this case Λ^2_{etalon} = 30.7 μm^2. As a result, we determine the value of thermal diffusivity:

$$\chi_x = 0.04\left(\frac{625}{30.7}\right) = 0.83\ \text{cm}^2/\text{s}.$$

The reference value of monocrystalline silicon thermal diffusivity is 0.9 cm^2/s [14].

Lead telluride thermoelectric film

When studying thin film on a substrate, it is important, first of all, to use radiation at a wavelength that provides high surface absorption to excite the thermal grating, so that the initial depth of heating of the film is significantly less than its thickness. and secondly, to choose the grating period so small that during the relaxation of the DR the heat does not reach the surface of the substrate. The latter requirement is met when satisfying the inequality $\Lambda < \pi d$, where d is the film thickness [12]. Otherwise, the measurement result will refer to the effective thermal diffusivity of the film-plus-substrate system.

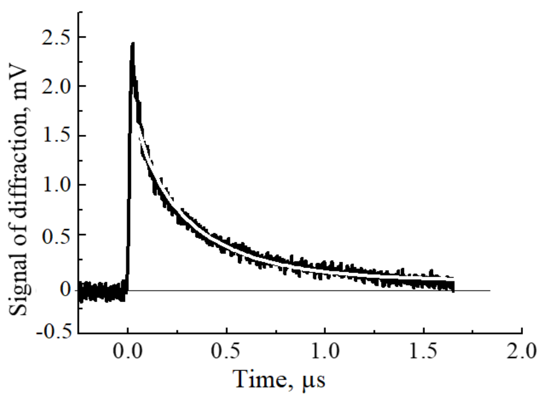

Figure 4 – Diffraction signal during excitation of a thermal transient grating in a 1.3 μm thick PbTe film on glass. The period λ_x and the lifetime τ_x of the lattice are 5 μm and 0.37 μs, respectively. The white line is the theoretical curve (ratio (3))

By using the graph in Figure 2, the OriginPro8 program, as well as ratio (5), the required value of χ_x is determined to be 0.018 cm^2/s. This value of thermal diffusivity well corresponds to the result of measurement obtained in [15].

Conclusion

A method is proposed that allows metrological support measurements of the thermal diffusivity χ_x of solid-state materials in a wide range of χ_x values by using a single etalon with a certified thermal diffusivity value χ_{etalon}. The basis of the measurements is a line graph $\tau_{etalon}(\Lambda_{etalon})^2$ constructed, which, due to the method application, can be considered as a multi-valued graphic material for metrological support of thermal measurements.

It has been experimentally shown that a thermal diffusivity etalon with a passport value χ_{etalon} = 0.04 cm^2/sec can be used for metrological support of measurements of samples with thermal diffusivity in different ranges compared to χ_{etalon}: χ_x = 0.496; 0.830 and 0.018 cm^2/s. This possibility is realized as well through the use of an easily controlled parameter – the transient grating period Λ.

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