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NON-CONVENTIONAL CONTACT-LESS METHOD FOR MEASUREMENT OF MATERIALS THERMAL DIFFUSIVITY COEFFICIENT

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ABSTRACT: Many application fields need information about the thermal properties of the used materials. Heat management is becoming very import for example in the building industries due to exploding energy costs, automobile industry or in the photonics and semiconducting industries as well. A lot of research and development has been done regarding the development of thermal diffusivity measurement methods. Recently, dynamic measurement methods, which are much faster than static ones are in progress. The paper relates to the optical, non-invasive method for measuring material thermal diffusion coefficient. Experimental results regarding the measurement of thermal diffusion coefficient of vitreous As-S materials are presented. The way to improve the sensibility is proposed and discussed.
KEY WORDS: thermal conductivity, methods for thermal conductivity, optical measurements, deflection of light

1. INTRODUCTION

Thermal conductivity (denote usually as κ) is the property of a materials to conduct heat.

Thermal conductivity plays an important role in various technological processes. For example, metals are characterized by high thermal conductivity, while dielectrics have low thermal conductivity.

The coefficient of thermal conductivity depends on the internal structure of the materials. In metals, thermal conductivity is mainly caused by the movement of free electrons. In dielectrics free electrons are missing and thermal conductivity is determined by the collective oscillations of atoms called phonons.

The mathematical model that reflects the thermal conductivity phenomenon is described by the Fourier's Law :

$$q = -k\nabla T \quad (1)$$

Which means that the heat flux is proportional to temperature gradient:

The minus sign indicates that heat is propagating from the body at higher temperature.

There are two categories of measurement techniques: steady-state and non-steady state or transient. Steady-state techniques infer the thermal conductivity from measurements on the state of a material once a temperature profile has been stabilized in time and space, whereas transient techniques are based on the state of a system during the approach to steady state. Steady-state techniques do not require complicated numerical simulations and measurement equipment. The disadvantage is that some uncontrolled heat leakage take place and the time required to reach steady state may be too long, both conditions may lead to systematic errors to the measurement process.

Steady-state methods. In steady-state method [1] the bulk material is prepared by a certain shape, either cylinder, disc have parallelepiped shape. Thermal conductivity is calculated using the Fourier equation of flow measurement and temperature difference at a certain distance along the body. The measurement lasts more than 30 minutes, the time it takes to reach the stationary state.

The heat flow is considered unidimensional and without heat loss. To minimize losses, in some cases the thermal conductivity is measured in vacuum [2] and with a screen that eliminates radiation losses. Other variants of the stationary flux method are known, such as the radial flow method [3, 4] or the parallel flow thermal conductivity method [5].

Transient methods. Among the non-steady-state methods, the most common method is the needle-type linear source of heat. These methods have been developed to overcome the shortcomings of stationary methods. As mentioned, there are parasitic heat losses and long waiting times until equilibrium is established.

The heat source in this method is provided either periodically or in the form of a pulse that manifests as corresponding phase and amplitude changes over time. The advantage lies in the fact that the measurements are carried out much faster, up to 30 s. The signal values are calculated more complicated in this case and are based on the heat equation:

$$\frac{\partial T}{\partial t} = \alpha \nabla^2 T \quad (2)$$

The coefficient $\alpha = k/\rho c$ is called thermal diffusion coefficient and is proportional to the thermal conductivity coefficient; ρ- is the density of the

material and c- is the specific thermal capacity. These two parameters of the material can be measured by other methods so that if we know the coefficient of thermal diffusion we can consider that we know the coefficient of thermal conductivity and vice versa.

Several variants of measurement are known by the non-stationary method. One of the other is the hot-wire method [6-8]. As a source of heat, a platinum wire embedded in the material, most often in the form of powder (soil, cereals, chemicals), is used.

At some point in time, heating is started by connecting to an electrical outlet. At a certain radial distance, temperature variation is measured over time. The method is mainly used for low thermal conductivity material, such as refractory materials, soil, etc.

Other variants of the hot-wire method, e.g. needle-sample method [9], are similar to the previous one, but the temperature variation is measured at zero. Just the thermocouple is electrically isolated from the heater wire.

Another transient method is the laser flash method, which measure thermal diffusivity of a disk-shaped sample [10, 11]. As previously seen, the thermal diffusion coefficient may be related to thermal conductivity. The method is unattached as it uses an optical light source (laser radiation) that is absorbed to heat the front of the disc. The temperature at the rear of the disc is determined from a distance from an infrared radiometer.

For film samples, specific methods of characterization have also been developed. The disadvantage for all these methods lies in the fact that the material subjected to typing (either bulk or film) requires preparation of the sample with well-defined dimensions. This does not allow the characterization of original objects such as rock, sculpture, thermal engine, small size material, etc.

Since 1980 some photo-thermal method began to be developed. The photo-thermal deflection was proposed as an optical non-contact and non-destructive method to measure coefficient of thermal diffusion. The method was based on measurement of the value of deflection angle of the laser beam and the determination of the value when it is zero.

This one corresponds to half period of thermal wave, determined by thermal diffusivity of material.

We develop another version of the thermo-optical measurement method relied on the measurement of the phase shift of the probe beam as function of distance from heat source. The basic theory is presented in section 2, experimental setup is presented in section 3 and in section 4 are presented the results for thermal diffusion of vitreous amorphous arsenic sulfide.

2. BASICS OF THERMO-PHOTO-DEFLECTION MEASUREMENT METHOD

Morphy and Aamodt [12, 13] and Jackson et al. [14] the first initiated theoretical base of method.

Let us consider the case presented schematically in Fig. 1a and 1b. The concept of this method is based on the thermal dispersion of the refractive index existing on all materials, including air.

A temperature gradient is formed on a heated surface. In the air, for example, the dispersion of the dn/dT refractive index is negative, therefore the beam of light deviates from the heated surface (Fig.1, left).

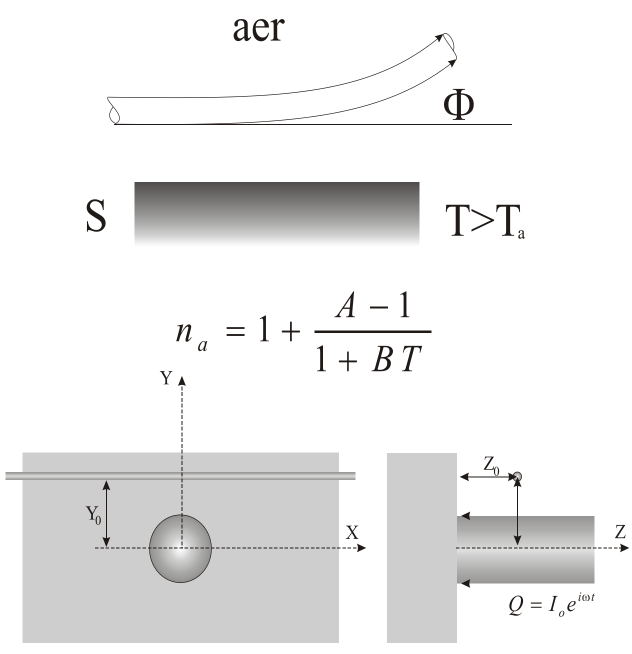

Figure 1. up: Deflection of light into a medium, air for instance, with thermal gradient ("Mirage" effect). The air refractive index $n = n(T)$ depends on temperature. down: Laser beam propagation geometry. The Probe beam slides along the surface and the pump beam falls perpendicularly to the surface (Are showed both top and front views).

A controlled deviation of the Φ angle is achieved using a beam of sample laser that propagates parallel to the surface of the body. The surface of the body is irradiated with a beam of laser light that falls perpendicular to its surface and is absorbed by the sample (Figure 1. down). If the laser light is periodically modulated over time, heat sources occur in the material and the environment. Laser radiation with P is absorbed in the sample and produces a heat source Q (r, t) that has the distribution [14]:

$$Q(r,t) = \frac{1}{2}\frac{4P\alpha}{\pi^2 a^2}\exp(-\alpha z)\exp\left(-\frac{2r^2}{a^2}\right)\exp(i\omega t) \quad (3)$$

The temperature distribution T (x, y, z) can be found by solving the heat conduction equations for each of three environments with the corresponding border conditions:

$$\nabla^2 T_0 - \frac{1}{k_0}\frac{\partial T_0}{\partial t} = 0 \qquad \text{region 0,} \qquad (4)$$

$$\nabla^2 T_1 - \frac{1}{k_1}\frac{\partial T_1}{\partial t} = \frac{-Q(r,t)}{k_1} \qquad \text{region 1,} \qquad (5)$$

$$\nabla^2 T_2 - \frac{1}{k_2}\frac{\partial T_2}{\partial t} = 0 \qquad \text{region 2,} \qquad (6)$$

Here ki are the coefficients of thermal diffusion for each of three adjacent environments. Typically, the specimen is in the air and the thermal air diffusion $k_0 = k_2$ is known. ki is the thermal conductivity of the sample to be characterized. The solutions are presented in the paper [14] in the general form.

The following particularities may be mentioned. In the case of applying a localized and harmonic source of heat to the material body, thermal waves are formed and propagated, which always have the form:

$$T(x,t) = T_0 R_{(x)} e^{-kx}\cos(\omega t - kx) \qquad (7)$$

The coefficient R(x) which determines the temperature distribution depends on the applied geometric configuration, the heating conditions, the absorption of the material, is influenced by the convection s.a. The wave vector $k = 1/\lambda = (\pi f/\chi)^{1/2}$, where λ is the thermal wave length. The thermal wavelength is determined by the expression $\lambda = (\chi/\pi f)^{1/2}$, where $f = \omega/2\pi$ is the frequency of laser beam modulation. They will note the following thermal wave characteristics from the expression for temperature T (x, t): The magnitude of the temperature oscillations decreases rapidly with the distance and the faster the higher the frequency ($e^{-kx} = e^{-2\pi x/\lambda}$). At the distance x = λ, the variable temperature value is already 500 times smaller than the temperature value at the location of the heat source. We can conclude that: a) a body with the wavelength (or higher) dimension can be considered semi-finite; and b) Actual temperature measurements can be made up to the departure of the λ order, otherwise the signal becomes insignificant to be detected. As an example, we can mention the thermal waves that occur during the periodical warming of the sun: Daily variation penetrates the soil to a depth of 15-20 cm, and the summer-winter season to the depth of 100-120 cm. At higher depths the temperature remains constant.

Conventional methods of temperature change with electric conductor or liquid flow and temperature measurement with thermocouples are limited to low operating frequencies, damaging the samples to be tested. Also, the measurement time and sample sizes are high.

In the optical method, temperature variation over time corresponds to the frequency of laser beam modulation and can be done very quickly. Transmission of heat is made without thermal contact through optical radiation. As it results from the theoretical results, the deflection angle Φ is proportional to the dispersion of the refraction index in the air and is related to the temperature profile by the following relation:

$$\emptyset = \frac{1}{n}\frac{dn}{dT}\int_{path} \nabla_{\perp} T(r,t)\,ds \qquad (8)$$

Obtaining absolute values for the deflection angle from this equation is rather complicated and uncertain if we refer to absolute values.

For these reasons, it is reasonable to measure the deflection angle as the distance offset of the sample beam y (offset) from the center of the parallel heating surface laser. The relationship is linear as demonstrated in the paper [15] if the phase of the sample signal is measured:

$$\varphi = y/l_T \qquad (9)$$

where the length of the thermal diffusion is determined by the chopper frequency ω and the thermal diffusion coefficient χ of the material by the relation:

$$l_T = (2\chi/\omega)^{1/2} \qquad (10)$$

3. EXPERIMENAL SETUP AND THERMAL DIFFUSIVITY OF AS_2S_3

The experimental installation is made using an optical system consisting of a beam extension beam and a cylindrical lens that focuses laser radiation on the surface of the object in the form of a narrow and long light beam (asymptomatic in the form of a line). The intensity of the continuous laser is periodically modulated in time by a chopper or an electro-optical or acoustic-optic modulator. The laser energy absorption on the surface of the object creates a linear heat source as shape and time modulated. This heat source produces heat waves that propagate in the sample to be characterized and in the air over the surface. If the object is transparent to the wavelength of the laser, cover the surface with a thin layer of graphite. Unlike the authors mentioned above, isotherms and isophases in this geometry represent cylinders with parallel axes at the source of the heat source.

The sample laser aligns in such a way that its radiation propagates parallel to the axis of the heat source (light beam), parallel to the surface and as close as possible to it. Once it propagates parallel to the axis, beam deflection occurs from the phase with the constant amplitude of the lane of the optical path. This leads to a cumulative summary and, consequently, an increase in the divergence angle. The deviation is measured with a sectorial radiation photodetector in the form of four segments connected differentially to a selectively nanovoltmeter synchronized with the lock-in signal. The distribution of the pumping laser energy over a long strip covers a larger irradiated surface compared to the surface of the spot used in other known schemes so that the local temperature increase is less at the same laser power.

The method can be done using the scheme and apparatus shown in Figure 2. As the source of the pumping beam, an argon laser will be used on the wavelength of 488 nm and the emission power of about 100 mW. As part of the characterization, pieces of calcogenic glass were used, characterized by an absorption coefficient of 103-104 cm-1 for the argon laser light used. The surface of the sample on which the light is focused is blotted. The laser beam extends 5 times with an optical lens and then focuses with a cylindrical lens with a focal length of 120 mm. In the focus, the beam has the shape of a 5x0.05 mm strip. Phase measurements require a shutter beam with laser diode and photoreceptor, from which the synchronization signal is transmitted to the phase detector. To obtain the sine wave modulation, the diameter of the laser beam must be approximately the same size as the holes on the modulator disk. As a source of the test beam, a less powerful (de ≈ 1 mW) laser such as the LG-208 He-Ne gas is used. It generates optical radiation with a wavelength of 630 nm, which corresponds to red light. Until the sample heating is initiated, the system is adjusted so that the beam of the sample laser falls on the position sensor matrix so that all photodiodes generate the same current.

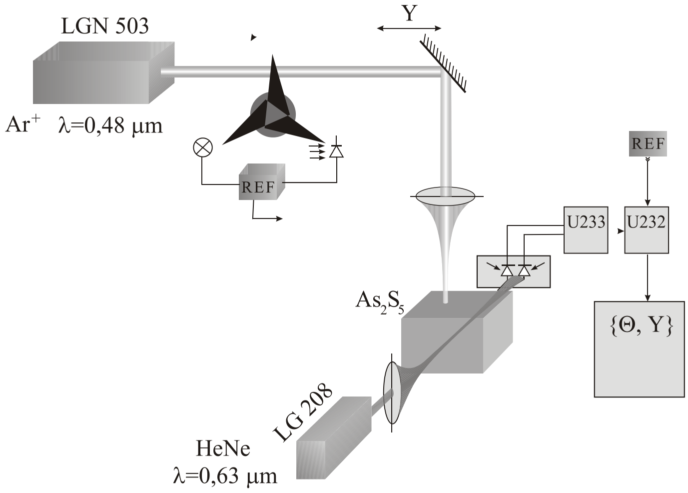

Figure 2. Example of the photographic thermo-deflection method for thermal diffusion measurement.

The reverse photodioded current is transmitted to a differential amplifier so that the current is zero. The reference signal is taken from a fairly simple device consisting of an ordinary bulb and a photodiode (REF) connected in series with a resistor to a DC source.

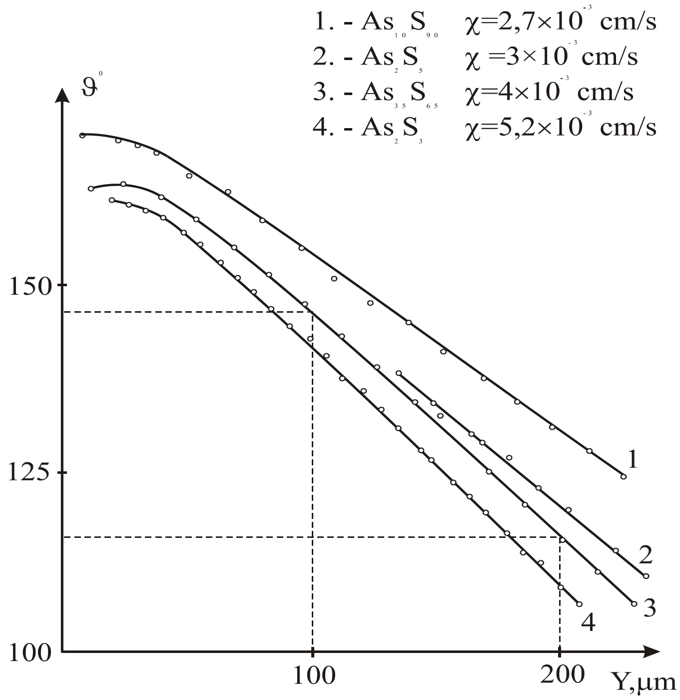

Figure 3. Dependency of the propagation phase for different compounds in the As-S system. The modulation frequency is 2 Hz. 1 – $As_{10}S_{90}$ (χ=2,7), 2 – $As_{29}S_{71}$ (χ=3,3), 3 – $As_{35}S_{65}$ (χ=4,0), 4 – $As_{40}S_{60}$ (χ=5,2).

The reference signal obtained is about 1 V, which is sufficient for the voltmeter to be synchronized.
The current from the position sensors is amplified and given to a "Unipan-232B" selective nanovoltmeter, which can measure voltages of 0,01 μV with a fairly small spectral band. The phase is fixed to the

reference signal by maximizing the phase detector signal.

In Figure 3. the phase dependence θ(y) is presented depending on the heat source removal from the heat source measured for different As-S system components.

4. CONCLUSIONS

Thermal deflection phenomenon using absorbtion surface of periodic modulated laser as point source of heat. The thermal waves were detected by a quadrant receiver. The wave phase was measured as dependance on distance between pump laser beam and the probe laser beam.

The thermal diffusion coefficient χ is determined by the angle of inclination of the phase $\Phi(y)$ on the linear portion.

We have measured the thermal diffusivity of AsxS1-x chalcogenide semiconductors. The pump laser radiation was focused in line shape. The phase interrogation demonstrates high level signal and reproducible values using photo-thermal deflection method.

It has been shown that the method can be used to measure material with a low value of thermal diffusion coefficient.

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