

obtaining not only the volume content of the filler but also of the binder and the pores by separating the porosity into open and closed and retaining the completeness of the specimen. Analysis of the curves in Fig. 4 shows that the porosity is reduced in layers with elevated binder content. The arrangement of the interlayers over the material thickness is seen from the curves, the tendency to increase the volume content of binder with the approach to the specimen outer surface is noticeable because of its extrusion during winding due to the contract pressure of the layers. Closed pores predominate as in the first experiment.

Therefore, the proposed tomographic method of determining the composition of composites permits computation of the volume content of the original components, the porosity, the volume content of the open and closed pores in each elementary layer of material, and clarification of the regularities of its structure.

NOTATION

ρ , ρ_T , ρ_{fi} , ρ_b , ρ_p , ρ_w , ρ_{im} , material densities, theoretical, fiber, binder, pore, water, impregnating fluid; μ_{fi} , μ_b , μ_p , fiber, binder, pore volume contents; f_{fi} , f_b , f_p , f_{di} , f_{di}^* , f_{cij} , f_{im} , linear coefficients of attenuation of the fiber, binder, air of the i -th layer of the dry specimen; i -th water-saturated layer of the specimen; j -th cell of the i -th layer of the specimen under investigation, impregnator; n , quantity of cells in the i -th layer, m_{fi} , m_1 , m_2 , weight of the dry and impregnated specimens in air and in water; C_{c1} , closed porosity; S_f , rms deviation of the linear attenuation coefficients; and α , volume closed pore content.

LITERATURE CITED

1. P. G. Cheremskoi, Methods of Investigating the Porosity of Solids [in Russian], Moscow (1985).
2. D. M. Karpinos (ed.), Composite Materials (Handbook) [in Russian], Kiev (1985).

MEASUREMENT OF THERMAL DIFFUSIVITY OF DIAMOND BY THE LIGHT-INDUCED THERMAL LATTICE METHOD

V. V. Gorbachev, V. M. Durasov, R. B. Zezin,
E. V. Ivakin, and A. S. Rubanov

UDC 636.2.023

The thermal diffusivity coefficient of natural diamonds is measured by optical induction of thermal diffraction lattices.

Diamond is the unique natural material which combines thermal and electrical conductivity properties most suitable for use in electronic technology. The wide use of diamonds as heat-sinks in microelectronics requires development of a rapid and reliable method for testing the thermal conductivity χ of crystals. The traditional methods of thermophysical experiment (see, for example, [1]) are cumbersome, require much time, and are unsuitable for studying crystals of small size. Spectral measurements [2] provide indirect information on χ and do not consider the presence of various defects, for example, dislocations, which affect the thermophysical properties of the diamond. There are available a few studies [3] using Rayleigh scattering of light within a diamond, on the basis of which it is in principle possible to determine the thermal diffusivity coefficient κ . However the extremely low level of the desired signal and noise produced by elastic scattering on crystal inhomogeneities require use of expensive optical and electronic instrumentation and lengthy processing of the measurement results.

Physics Institute, Academy of Sciences of the Belorussian SSR, Minsk. Translated from *Inzhenerno-Fizicheskii Zhurnal*, Vol. 56, No. 5, pp. 745-748, May, 1989. Original article submitted November 24, 1987.

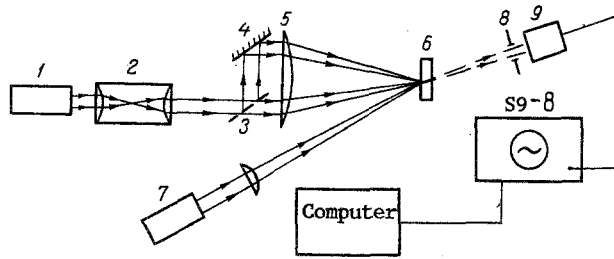


Fig. 1

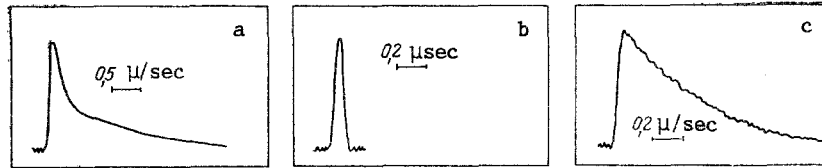


Fig. 2

Fig. 1. Diagram of experimental apparatus.

Fig. 2. Oscillograms of radiation intensity at input (a) and output (b) of plasma gate and light diffraction on thermal lattice in diamond (c), $\Lambda = 120 \mu\text{m}$.

The present study will demonstrate the possibility of measuring κ of natural diamond by forming a thermal diffraction lattice within the specimen [4]. This method has the advantages of lack of physical contact with the specimen, high spatial resolution, rapidity, and relative simplicity. It provides a significantly higher signal level as compared to Rayleigh scattering. Measurement of the thermal diffusivity coefficient κ by the thermal diffraction lattice method consists of forming a heat source within the specimen volume, the power of which can be described by a one-dimensional spatially periodic function. To do this the specimen is irradiated briefly by two coherent light beams propagating at an angle to each other which form an interference field. Due to absorption and heat liberation in the specimen the electromagnetic radiation power distribution is converted into an index of refraction distribution. The thermal diffraction lattice thus formed is then probed by a continuously acting laser. For adiabatic heating the diffraction efficiency of the lattice $\eta(t)$ after switchoff of the pulses forming the lattice is defined by the expression [5] (at $\eta \leq 0.1$)

$$\eta(t) = \frac{I_{\text{dif}}}{I_{\text{probe}}} = T \left[\frac{\pi}{\lambda} \left(\frac{\partial n}{\partial T} \right)_p \frac{1}{c\rho} \right]^2 Q_1 Q_2 \exp\left(-\frac{2\tau}{t}\right).$$

Here T is the specimen transmission at wavelength λ , $(\partial n/\partial T)_p$ is the thermal coefficient for change in the index of refraction of the medium, measured at constant pressure Q_1 and Q_2 are the surface energy absorption densities in the specimen at the end of the pulse, produced by action of the first and second light beams, $\tau = \Lambda^2/4\pi^2\kappa$ is the lattice constant of the thermal lattice with period Λ . Determining the value of κ reduces to measurement of the attenuation constant of the signal diffracted on the thermal diffraction lattice.

Unique features of κ measurement in diamond using the thermal lattice, as compared to similar measurements performed in liquids [6] and some crystals [7] are the thermal lattice relaxation times, which are orders of magnitude smaller. There are also definite difficulties in choosing the thermal lattice formation source, since the diamond absorption lies in the range 4–6 μm , where pulsed lasers are lacking, with the exception of laboratory CO-lasers. Therefore a TEA CO₂-laser operating at 9.2 was used, this wavelength corresponding to the edge of the diamond impurity absorption band related to appearance of A-, B₁-, and B₂-centers.

A diagram of the experimental apparatus is shown in Fig. 1. The pulsed radiation source was a single-mode TEA CO₂-laser (1) using a mixture CO₂:N₂:He = 1:1:3, which generated single pulses 1.5 sec long at half height with an energy of 0.8 J ($\lambda = 9.2 \mu\text{m}$) (Typical pulse form is shown in Fig. 2a).

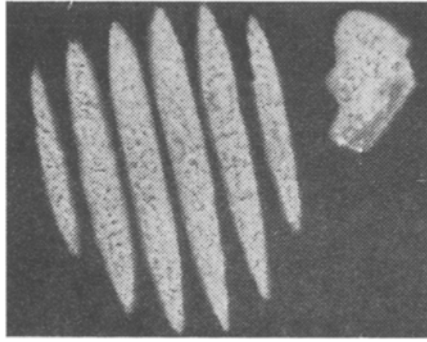


Fig. 3. Radiation intensity distribution in the specimen.

TABLE 1. Measured and Calculated Thermophysical Properties of Diamond

Characteristic	Specimen No.							
	1	4	7	8	9	10	11	12
χ_1 , w/cm-deg	10,09	9,03	8,48	12,09	19,3	10,3	10,04	6,28
κ_1 , cm ² /sec	4,63	4,14	3,90	5,55	8,85	4,72	4,60	2,88
κ_2 , cm ² /sec	3,6±0,2	3,0±0,5	2,9±0,5	4,2±0,4	—	3,4±0,5	3,4±0,5	2,2±0,4
κ_1/κ_2	1,28	1,38	1,34	1,32	—	1,39	1,35	1,31
Thickness, mm	0,22	0,29	0,65	0,5	1,0	0,53	1,0	1,3
Absorption at $\lambda = 9,2 \mu\text{m}$, %	14	21	36	24	7	29	54	84

To measure the brief relaxation times, which are related to the high value of κ , it is necessary to shorten the duration of the excitation pulse. Plasma gate 2 [8] is used for this purpose. It consists of a container filled with an inert gas and germanium lens input windows forming a confocal system. As is evident from Fig. 2b use of the plasma gate insures the required pulse duration, not greater than 100 nsec at half height. In its optimum mode the plasma gate transmits 15-20% of the applied energy.

The interferometer used to create thermal lattices in the specimen consists of semitransparent light splitter 3 based on a germanium plate polished on one side, mirror 4, and lens 5, which produces two focused beams in the specimen plane 6. The period of the interference pattern (Fig. 3) was 120 μm , the laser spot diameter in the specimen was 0.7 mm, and the pulse energy was 30-40 mJ. The thermal lattice was probed by a type LG-38 helium-neon laser 7 with 20 mW of power. The diffracted signal is extracted by diaphragm 8 and recorded by photomultiplier 9. Measurements were performed with an S9-8 oscilloscope and an "Elektronika DZ-28" microcomputer, with time resolution of 50 nsec.

The specimens used were natural diamond crystals type IIa (specimen No. 9), an intermediate type (specimen No. 8), and type Ia (specimens Nos. 1, 4, 7, 10, 11, 12) with various contents of basic structural-impurity defects. Diamonds with and without optical surfaces and volume inhomogeneities were studied.

Measurement results are presented in Table 1. The first line gives values of the thermal conductivity coefficient χ at 320 K, as calculated from optical absorption in the IR and uv regions [2]. The second line is thermal diffusivity coefficients found from the relationship $\kappa_1 = \chi_1/c\rho$. Unfortunately the literature lacks data on $c\rho$ measurements of natural diamond at temperatures near room temperature, so that $c\rho$ values from [9], extrapolated to a temperature of 320 K were used. In the κ_1 calculations the value $c\rho = 2.18 \text{ J}/(\text{cm}^3 \cdot \text{K})$ was used.

Values of the thermal diffusivity coefficient κ_2 were measured with the light-induced thermal lattices and averaged over 5-10 measurements. Table 1 also indicates the maximum deviations from the mean. The mean heating of the specimen due to thermal lattice formation comprised $T = 25\text{-}35 \text{ K}$ depending on the crystal's absorption. Therefore it can be assumed that the κ_2 data correspond to the same temperature range as the κ_1 data.

As is evident from Table 1, the measured κ_2 values are 25-30% lower than κ_1 . One reason for this divergence may be related to the fact that κ_1 was determined by an indirect method which did not consider the effect on thermal conductivity of crystalline imperfections other than type A and B₁ defects. There is a correlation between κ_1 and κ_2 values upon transition from specimen to specimen.

The relatively high deviations from the mean value of the measurements are caused by optical and electrical noise in the measurement system. As a result it was impossible to obtain reliable results for specimen No. 9, which had low absorption at 9.2.

NOTATION

χ , thermal conductivity coefficient; κ , thermal diffusivity coefficient; η , diffraction efficiency; I_{dif} , diffracted radiation intensity; I_{probe} , probe radiation intensity; λ , probe radiation wavelength; c_p , specific heat; Q , surface energy density; τ , thermal lattice relaxation time; Λ , thermal lattice period.

LITERATURE CITED

1. T. D. Ositinskaya, V. A. Tsentrovskii, and A. S. Vishnevskii, *Inzh.-Fiz. Zh.*, 32, No. 4, 620-624 (1977).
2. G. B. Bokii, G. N. Bezrukov, Yu. A. Klyuev, A. M. Naletov, and V. I. Nepsha, *Natural and Synthetic Diamonds* [in Russian], Moscow (1986).
3. H. E. Jackson, R. T. Harley, and M. H. Lindsey, *Phys. Rev. Lett.*, 54, No. 5, 459-461 (1985).
4. Method for Determining Thermophysical Characteristics of Materials, USSR Patent No. 568,303, MKI G 01 N 25/18.
5. A. S. Rubanov and E. V. Ivakin, in: *Quantum Electronics and Laser Spectroscopy* [in Russian], A. M. Samson (ed.), Minsk (1974), pp. 407-425.
6. H. J. Eichler, G. Salje, and H. Stahl, *J. Appl. Phys.*, 44, No. 12, 5383-5388 (1973).
7. H. J. Eichler and S. Knof, *Appl. Phys.*, 13, No. 2, 209-210 (1977).
8. E. Yablonovitch, *Appl. Phys. Lett.*, 23, No. 3, 121-122 (1973).
9. D. V. Fedoseev, N. V. Novikov, A. S. Vishnevskii, and I. G. Teremetskaya, *Diamond* [in Russian], Kiev (1981).