Calorimetric measurement of absorption loss in orientation-patterned GaP and GaAs

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ABSTRACT

Materials grown by vapor phase techniques such as chemical vapor deposition or hydride vapor phase epitaxy (HVPE) often exhibit very low losses which are difficult to quantify by simple transmission measurements. The measurement of extremely low absorption coefficients can be carried out by laser calorimetric or thermal rise techniques, which determine the absorption coefficients by measuring the temperature increase caused by the absorbed laser radiation. We report here on results of measuring absorption coefficients of bulk HVPE-grown orientation-patterned GaAs (OP-GaAs) and GaP (OP-GaP) crystals using one of the methods of laser calorimetry, called transient calorimetry. In our setup, the sample under test is attached to a high-conductivity copper holder and placed in a vacuum chamber. A 2-micron CW laser beam is transmitted through the sample and the temperature rise in the sample is measured and, through the calorimeter calibration process, related to the power absorbed in the sample. The absorbed power, P_a, is a function of the total attenuation coefficient α_{tot}, the length of the sample, and the laser power P_o, defined as $P_a = P_o \, exp \, (-\alpha_{tot} \, l)$, where total attenuation α_{tot} is the sum of absorption and scattering: $\alpha_{tot} = \alpha_{abs} + \alpha_{scat}$. Since scattered light does not cause heating, the calorimetric technique is only applicable to determining α_{abs}. By this technique we have measured 2-micron absorption coefficients in OP-GaAs and OP-GaP as low as 0.007 cm^{-1}.

Keywords: OP-GaAs, OP-GaP, Tm:fiber laser, quasi-phase-matching, QPM, nonlinear optical material, laser calorimetry, absorption

1. LASER CALORIMETRY APPROACH

The measurement of extremely low absorption coefficients can be carried out by laser calorimetric or thermal rise techniques, which determine the absorption coefficients by measuring the temperature increase caused by the absorbed laser radiation. There are two different techniques of handling samples and measuring the temperature. In one method, a sample is thermally isolated from its surroundings and the amount of heat is directly measured from the resulting temperature rise in a straightforward fashion.[1] In the other method, called transient calorimetry, the sample under the test is attached to high-conductivity material; the temperature rise of this material is measured and, through the calorimeter calibration process, related to the heat in the sample.[2] As the high-conductivity material, one can use heat–absorbing fluid or copper, which, in turn, is thermally isolated from its surrounding. Although in both techniques, for thermal isolation, the samples have to be placed in vacuum, we chose the latter technique as it allowed us to design a vacuum chamber and sample holder easily modified to fit various sample shapes and sizes.

In the transient calorimetry, a CW laser beam is transmitted through the sample, and the increase in temperature is monitored as a function of time by a linear thermistor, attached to the copper mount or, as we will refer to it below, the copper base. Assuming instant conductivity of copper, the temperature increases initially at linear rate with a slope dT/dt related to the absorbed power P_a by the expression

$$mc_p(dT/dt)|t_0 = P_a, \qquad\qquad (1)$$

where m is the mass, c_p is the specific heat, and T is the temperature of the copper base. The value of dT/dt could be calculated or determined experimentally by heating the copper base with a heater. The absorbed power, P_a, is a function of the total attenuation coefficient α_{tot}, the length of the sample, and the laser power P_o, defined as

$$P_a = P_o \, exp \, (-\alpha_{tot} \, l), \tag{2}$$

where total attenuation α_{tot} is the sum of absorption and scattering: $\alpha_{tot} = \alpha_{abs} + \alpha_{scat}$. Since scattered light does not cause heating, the calorimetric technique is only applicable to determining α_{abs}. It is necessary to note that P_o is the laser power corrected for scattering and reflection from the sample input and output surfaces.

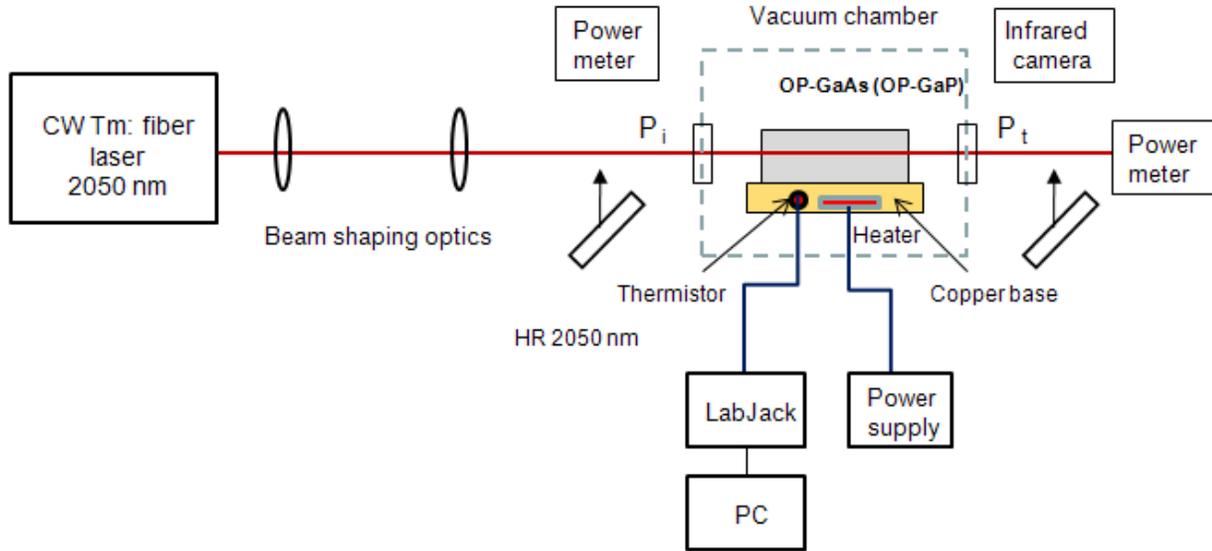The layout of the experimental setup for absorption measurements is shown in Fig. 1.

Figure 1. Block Diagram of Laser Calorimeter.

As a probe laser beam we use output from a 10-W CW Tm:fiber laser built by Q-Peak. The beam is collimated by a 50-mm-focal-length (f.l.) air-spaced triplet and then focused by a 250-mm-f.l. (in air) ZnSe lens to form a 140-micron waist in the focal plane with Rayleigh range of ~ 30 mm. A sample under test is placed in the vacuum chamber at distance from ZnSe lens greater than f.l. by t_s where $t_s = l \, (n-1)/2n$, where l is length of the sample and n its refraction coefficient. The data on temperature rise of the sample are taken when the pressure in the vacuum chamber achieves $3 \div 6 \times 10^{-6}$ Torr. We also measure the beam power, incident on the vacuum chamber, P_i, as well as the power unabsorbed by the crystal, P_t as depicted in Fig. 1. The laser beam profile and profile of the beam transmitted through the sample are recorded with the use of an Infrared Electro-Physics camera.

Evaluation of the sample absorption consists of the following major steps: (1) calorimeter calibration, i.e. defining the value of $(dT/dt)/P$ for the copper base, (2) measurements of sample temperature as a function of time and the incident beam power, and (3) calculation of the sample absorption coefficient. These steps as well as the results of absorption measurements for a few OP-GaAs and OP-GaP samples will be described in the sections below.

2. CALORIMETER CALIBRATION

As mentioned above, for thermal isolation of samples from surroundings except the copper base, we designed a vacuum chamber. The chamber consists of the following components (see Fig. 2): a main chamber, a 3.375" stainless steel cube with 6 CF view ports, vacuum windows, vacuum flanges, two CaF_2 windows, a 9 mm x 19 mm x 30 mm copper base for mounting crystal up to 30-mm long, and PEEK insulator.

The sample is attached to an oxygen-free (OFC) copper base using high thermal conductivity silver paint; the mount is screwed to a PEEK insulator to prevent heat dissipation through the stainless steel cube. The insulator is screwed to a stainless steel disk, and with an OFC gasket is bolted to the cube. Attached to the base are a linear thermistor and a

heater. The thermistor is an Omega Engineering linearized two-element device configured to give a voltage directly proportional to the temperature with a relative accuracy of ±0.05 ºC. The thin film resistive heater is used to apply known heat loads to the copper clamp, in situ, in order to calibrate its response to absolute heat load values.

(a)   (b)
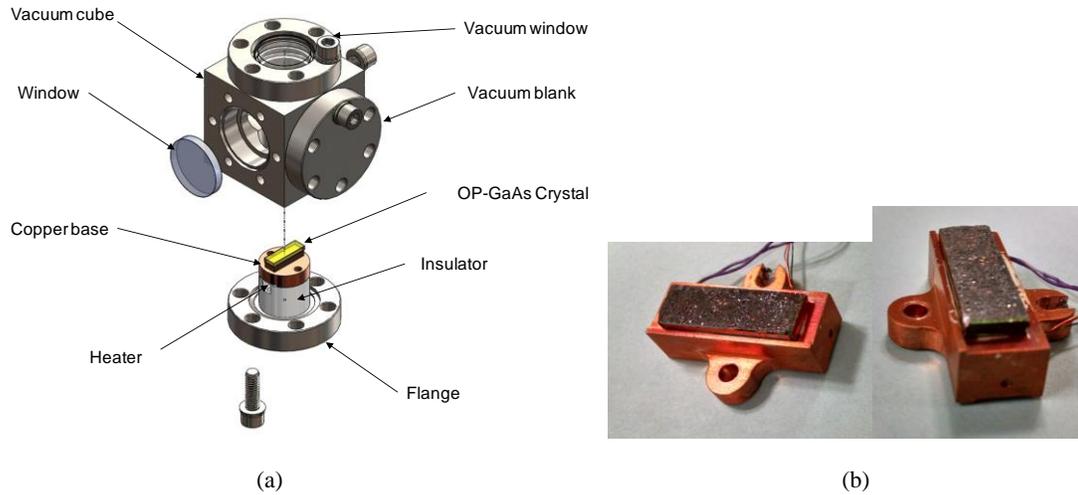
Figure 2: 3D Solid Works View of Vacuum Chamber (a) and Picture of OP-GaAs Crystal on Copper Base (b).

On top of the cube, a vacuum window is bolted and sealed with an OFC gasket. The uncoated CaF_2 windows are glued using an EP51HT epoxy. The wires of the thermistor are passed thru the last port of the cube and ran through a "T" CF flange that is bolted into place with the OFC gasket. The wires are soldered to the 8-pin CF electrical feed through vacuum flange and bolted into place with the OFC gasket. The last port on the "T" is connected to the Turbo Pump system using an OFC gasket as well.
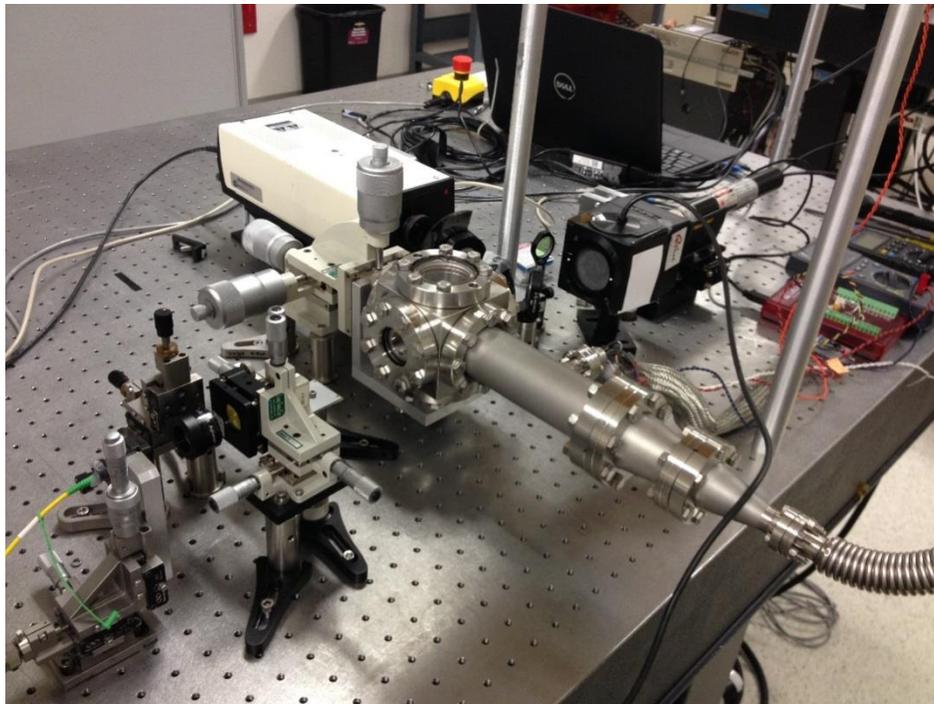

Figure 3. Picture of Laser Calorimeter Setup with Vacuum Chamber.

The assumption of instant transient response of heat from the sample to the sensor was confirmed by the results of the calculations performed in Autodesk Simulation Mechanical 2014 [3]. Gallium Arsenide was modeled using the following data: density, 2316 kg/m^3; specific heat 330 J/kg*K; thermal conductivity, 58 W/m*K. The material properties of UNS10100 oxygen free copper applied to the copper crystal mount were: the specific heat 0.385 J/g*K, thermal conductivity 385 W/m*K. The OP-GaAs sample in the model was 2.1 mm thick, 8 mm wide and 30 mm long with QPM layer thickness of 1.5 mm. The model shows that the sensor temperature becomes practically equal (the difference is 0.014^{o}C) to the sample temperature within ~ 500 msec.

In order to calibrate the calorimeter, i.e. to determine the value $(dT/dt)/P$ (we will refer to it as S_t), we calculated it as a product of base weight, m= 24.5 g and copper specific heat that gives S_t equal 0.106 Ks^{-1}W^{-1}. To confirm that our system design provides high accuracy of absorption measurements, we determined S_t experimentally by utilizing results of measuring the temperature rise at different heat power applied to the heater; the results presented in Fig. 4.
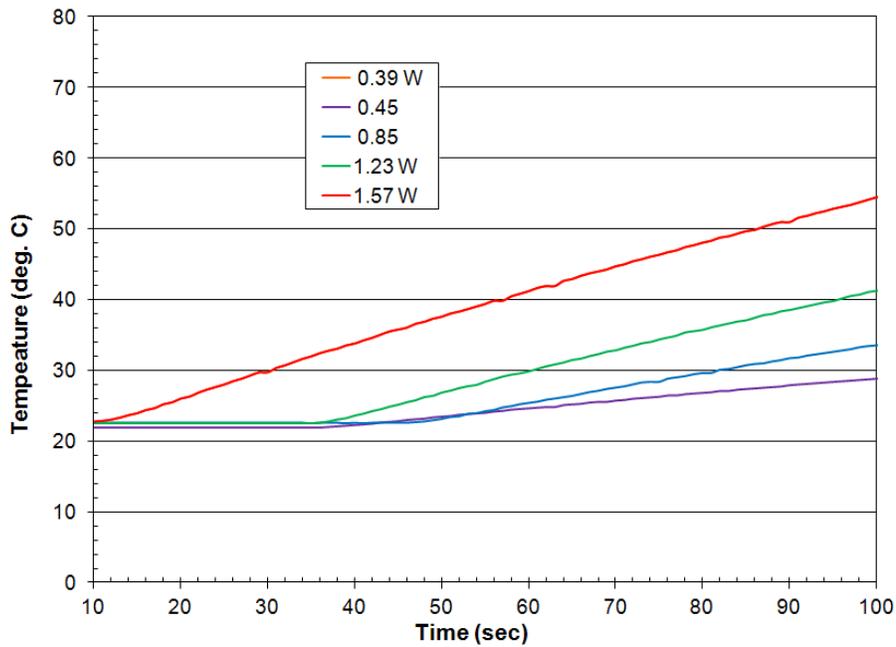

Figure 4. Copper Base Temperature Rise as a Function of Time at Different Heat Loads.

The data in Fig. 4 clearly shows the initial rate of temperature rise over 100 second to be linear. The slopes of temperature rise, or temperature derivatives dT$/dt$, plotted versus heater power loads from 0.39 W to 1.57 W are plotted in Fig. 5 and demonstrate a linear function. The slope of this line, in turn, represents the experimental value of $S_t =$ 0.102 Ks^{-1}W^{-1}, which is in good agreement with that calculated above.
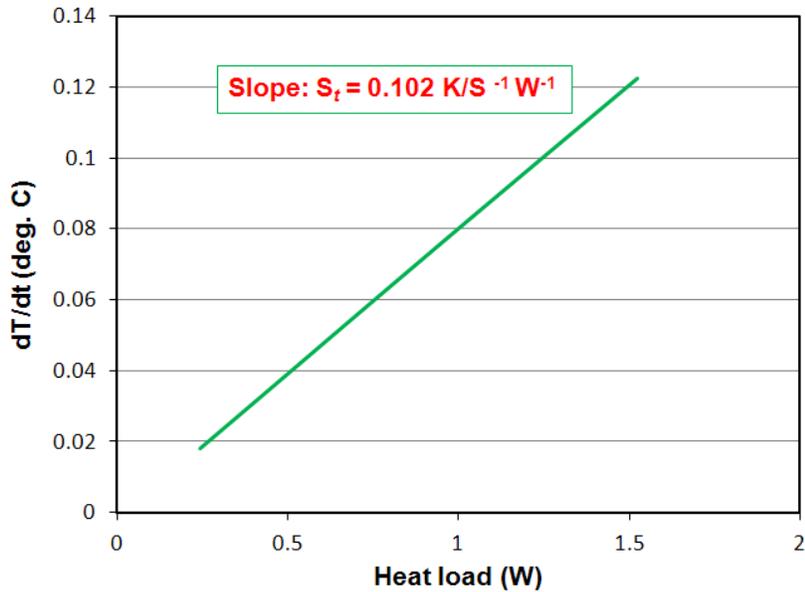
Figure 5. Temperature Derivative dT/dt as a Function of Heat Load for Copper Base.

3. ABSORPTION MEASUREMENTS

The essential part of absorption measurements consists of determining the temperature rise of the copper base caused by absorption of the laser radiation by a sample under test. Evaluating the slope of the temperature rise and relating it to S_t, allows one to calculate the power, P_a absorbed in the sample and, subsequently, determine the absorption coefficient.

The measurement of the temperature rise of the sample is straightforward: the laser beam is directed toward the sample and the temperature of the copper base is recorded as a function of time. It is sufficient to record the temperature rise at just a single laser power value; however, we repeated the experiment at various power points. We also measured the incident, P_i and transmitted, P_t powers.

Figure 6 depicts the CW Tm:fiber laser employed in our experiments.

Figure 6. CW Tm:fiber laser provides up to 10 W of single-mode output at 2050 nm.

As described above, we measured the temperature of the sample through the use of a linearized thermistor mounted in the copper base, which is part of a battery-operated circuit optimized to provide a linear relation between temperature and voltage. We employ LabJack (Lakewood, CO) U-6, USB-connected, 16-bit analog-digital converter to measure both the battery voltage and the thermistor voltage, with a provision for also measuring a power-meter-derived voltage

representing the laser power incident on the sample. We adapted a LabJack-supplied Labview-based software program to convert the thermistor voltage to temperature, sample the voltage at a fixed rate, and provide both a graphical display and a file output to show, as a function of time, the sample temperature and the time-derivative of the temperature.
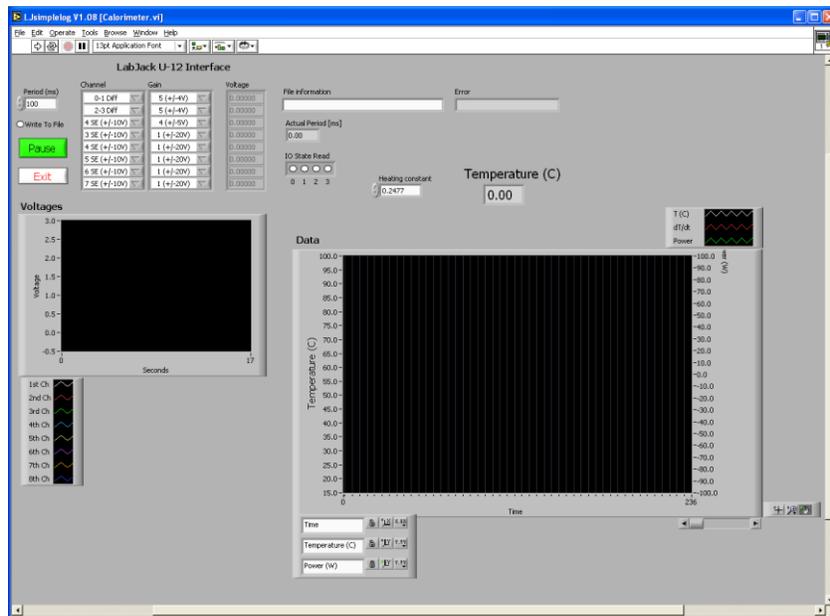

Figure 7. Control panel for Labview-based calorimeter-data acquisition.

In order to make sure that the laser beam propagates through the patterned area of the OP-GaAs sample without clipping, we recorded transmitted beam profiles while moving the vacuum chamber in the vertical direction across the laser beam. Figure 8 displays the output laser beam profile (a), the profile of the beam diffracted at the upper edge of the sample (b), and the beam transmitted through the sample. It can be seen from Fig. 8 (c) that the transmitted beam is slightly asymmetric but not substantially distorted.
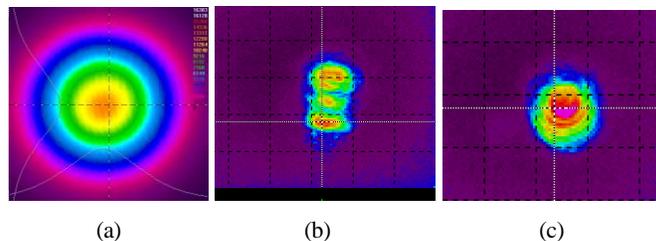

(a)   (b)   (c)

Figure 8. Probe Beam Profile (a), Beam Diffraction at Upper Edge of Patterned Layer (b), Beam Transmitted Through the Layer (c).

Below, we graphed the test results for the OP-GaAs sample #26P which had dimensions 1.2(t) x 8.5 (w) x 30 mm (L) with QPM layer thickness of 0.6 mm and grating period of 60.5 μm. The crystal faces were AR-coated for 1990 – 2090 nm and 3400 – 4900 nm. The reflection coefficients at 2050 nm were 0.008 and 0.012869.

The slopes of the lines in Fig. 9 represent temperature derivatives over time, which we will notate as D_t, at different incident laser powers,
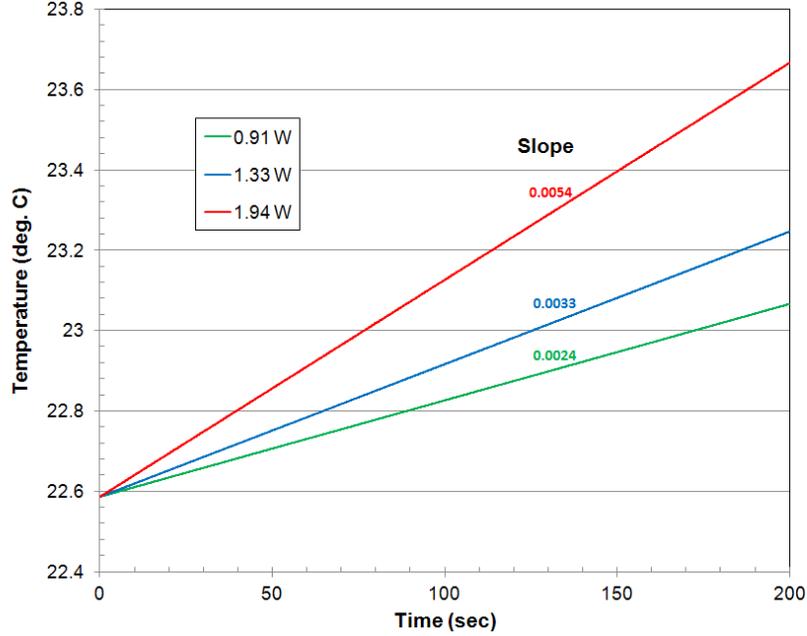
Figure 9. Temperature Rise as a Function of Time for OP-GaAs Sample #26P at different laser power.

4. CALCULATION OF ABSORPTION COEFFICIENT

In order to evaluate the absorption coefficient we need to determine the absorbed, P_a and incident power P_o, i.e. the laser power corrected for scattering and reflection from the sample input and output surfaces. P_a can be determined from an expression $P_a = D_t/S_t$. For instance, at the laser power 1.94 W, $D_t = 0.0054$ Ks^{-1}W^{-1} and $P_a = 0.0054/0.0102 = 0.529$ W.

For determining P_o we use the following rational: the beam, P_i, incident on the input surface is partially reflected; the coefficient of reflection is r; the transmitted portion of the beam is then reflected from the output surface back into the sample. Thus, the power inside the sample, P_o is equal

$$P_o = (P_i - r\,P_i) + (P_i - r\,P_i)\,r = P_i\,(1 - r^2), \qquad (3)$$

where r is the reflection coefficient of sample faces at 2050 nm and Pi is the laser power at the input face of the sample, i.e. behind the input window of the chamber . The absorbed power can be expressed as

$$P_a = P_o\,[1 - \exp(-\alpha_{abs}\,l)] \qquad (4)$$

where l is length of the sample. The coefficient of absorption then can be derived from expression (3) as:

$$\alpha_{abs} = (-1/l) \times Ln\,(1 - P_a/P_o) \qquad (5)$$

The results of evaluation of α_{abs} for two OP-GaAs samples both AR-coated for 1990 – 2090 nm and 3400 – 4900 nm are tabulated below in Tables 1 and 2.

Table 1. OP-GaAs sample #27I, l = 24.8 mm, t = 2.1mm, w = 8 mm wide, QPM layer thickness of 1.5 mm and grating period of 56 μm.

P_i, W	D_t, deg.C/sec	P_a, W	P_0, W	α_{abs}, cm-1
1.615	0.003	0.0283	1.6148	0.007
2.270	0.0048	0.0453	2.2696	0.008
2.891	0.0052	0.0491	2.8903	0.007

Table 2. OP-GaAs sample #26P, l = 30 mm, t =1.5-mm thick, w = 8.5 mm wide, QPM layer thickness of 0.6 mm and grating period of 60.5 μm.

P_i, W	D_t, deg.C/sec	P_a, W	P_0, W	α_{abs}, cm-1
0.865	0.0024	0.0226	0.8644	0.009
1.264	0.0033	0.0311	1.2634	0.008
1.843	0.0054	0.0509	1.8428	0.009

Results for more samples are shown in Table 3.

Table 3. Measured absorption coefficients for several OP-GaAs and OP-GaP samples

Sample	Length, mm	Grating period, μm	α, cm^{-1}
28P, OP-GaAs	25.7	63.3	0.017
33G1, OP-GaAs	16.5	55.6	0.015
31G, OP-GaAs	19.6	Unpatterned	0.014
42A, OP-GaAs	19.8	Fan 65-70	0.007
30A1, OP-GaAs	12.2	Unpatterned	0.012
21C1, OP-GaP	16.3	Unpatterned	0.018
21C1, OP-GaP	16.3	Substrate	0.049

5. SUMMARY

We have built and calibrated a Tm:fiber-laser-based calorimeter for measurement of absorption loss in materials in the 2000-nm wavelength region. Watt-level powers provided high SNR data for 1 %/cm absorption levels and the system could likely extend to < 0.1 %/cm absorption. We measured seven OP-GaAs samples with loss range 0.7-1.7 %/cm and average 1.2 %/cm and one OP-GaP sample, 1.8 %/cm.

ACKNOWLEDGEMENTS

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