Near-IR absorption in high-purity photothermorefractive glass and holographic optical elements: measurement and application for high-energy lasers

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Volume Bragg gratings (VBGs) in photothermorefractive (PTR) glass are widely used for laser beam control including high-power laser systems. Among them, spectral beam combining based on VBGs is one of the most promising. Achieving $100 +$ kW of combined laser beams requires the development of PTR glass and VBGs with an extremely low absorption coefficient and therefore methods of its measurement. This paper describes the calorimetric method that was developed for measuring a low absorption coefficient in PTR glass and VBGs. It is based on transmission monitoring of the intrinsic Fabry–Perot interferometer produced by the plane-parallel surfaces of the measured optical elements when heated by high-power laser radiation. An absorption coefficient at 1085 nm as low as 5×10^{-5} cm^{-1} is demonstrated in pristine PTR glass while an absorption coefficient as low as 1×10^{-4} cm^{-1} is measured in high-efficiency reflecting Bragg gratings with highest purity. The actual level of absorption in PTR glass allows laser beam control at the 10 kW level, while the 100 kW level would require active cooling and/or decreasing the absorption in PTR Bragg gratings to a value similar to that in virgin PTR glass.   © 2011 Optical Society of America

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1. Introduction

Photothermorefractive (PTR) glass is a multicomponent silicate glass that undergoes a refractive index change after UV exposure followed by thermal treatment [1]. Based on this photosensitivity, high-efficiency volume Bragg gratings have been recorded in this glass [2] and successfully used for laser beam control [3]. One of the very promising laser applications is spectral beam combining by means of volume Bragg gratings (VBGs) [4]. The spectral combining of five 150 W fiber laser beams with an efficiency of $90 + \%$ has been recently demonstrated [5]. An analysis of the scalability of this spectral beam combining to the multikilowatt level is presented in [6]. The main effect that lowers the quality of a combined beam is thermal lensing resulting from nonuniform

heating of the VBGs by laser radiation. Therefore, one of the key parameters for the success of this application is the level of absorption in PTR glass. However, creation of low absorbing PTR glass and VBGs depends on development of both high-purity technology and reliable measurements. They are challenging because the actual level of absorption in this material corresponds to an attenuation of the laser beam by a tiny fraction of a percent per centimeter and is extremely difficult to detect by conventional photometry. Another challenge comes from the presence of scattering losses with amplitude equal or higher than the absorption losses.

Numerous methods for measuring small absorption coefficient in glass and crystals have already been published. Among them, laser calorimetry [7] and photothermal deflections [8] are the most sensitive methods. Interferometric methods have also been developed [9]. However, each method requires preparation of special samples and some knowledge

about the thermal properties of the sample to be measured. The goal of this research is to make measurements in complex optical elements with a hologram recorded in the volume of the sample. Moreover, it would be of great interest to be able to correlate this absorption coefficient with the parameters of the VBGs operating in high-power laser beams. In this paper we introduce a setup that allows us to measure the change of the optical thickness of a PTR glass flat under exposure to high-power laser radiation. From this, the absorption coefficient can be calculated allowing an evaluation of the change of the properties of a VBG in the actual laser system. Based on this procedure, we studied the actual level of absorption in pristine glass produced at glass melting facilities available at CREOL/University of Central Florida and OptiGrate Corp. as well as in the VBGs recorded in this glass. Finally, we model the change of VBG temperature and resonant wavelength per incident beam power for different levels of PTR glass absorption. Based on an extrapolation of these results, we determine the level of absorption that is required to achieve multikilowatt laser beam control by means of passively cooled VBGs in PTR glass.

2. Method of Small Absorption Measurement in Optical Flats

A. Principle of the Method

The principle of the method that we developed is an evolution of interferometric methods that were previously developed and used for substrates [9,10], thin films [11], or even plasma characterization [12]. Let us consider an optical flat. When the angle of incidence or the wavelength of a monochromatic plane wave transmitted through this flat is scanned, the beam appears with a modulation of its intensity due to the presence of a Fabry–Perot interferometer resulting from the multiple reflections on each face of the flat (Fig. 1). This intrinsic interferometer can be used to measure the local optical thickness (a product of the refractive index and the geometrical thickness) of the flat if the coherence length of a light source exceeds the difference in optical paths of the beams after multiple reflections [13,14]. If the optical thick-
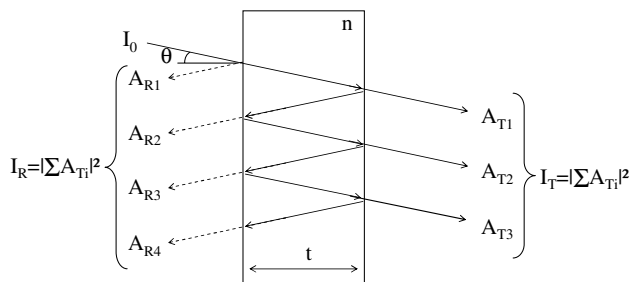

Fig. 1. Ray tracing of the beam transmitted through a Fabry–Perot-like structure. Transmitted (I_T) and reflected (I_R) intensities are obtained as a coherent sum of all transmitted beams (A_{Ti} and A_{Ri}).

ness of the flat is varied as a result of some external forcing function (e.g., heating), the modulation of the intensity of the overall transmitted beam (I_T) launched at the normal incidence angle is given by the Airy formula:

$$\begin{cases} I_T(nt, \lambda) = \dfrac{I_0}{1 + F \sin^2\left(\frac{\Phi}{2}\right)}, \\ \Phi = \dfrac{4\pi nt}{\lambda} \cos(\theta) \end{cases} \quad (1)$$

where I_0 is intensity of the incident beam, F is a parameter defining the finesse of the Fabry–Perot, Φ is the phase of the beam after propagating through the optical flat, n is its refractive index, t is its geometrical thickness, λ is the wavelength of the beam propagating through the window, and θ is the angle of incidence on the window. Figure 2 shows the dependence of the transmission on phase shift for a 5 mm thick PTR glass flat (refractive index 1.497) illuminated with an He–Ne laser at 633 nm. This phase could be changed by either a change of the incident wavelength (λ) or by a change of the optical thickness (nt) of the window that occurs when exposed to mechanical stresses, thermal effects, etc. The parameters I_0, n, t, and λ are known. Parameter F can be determined experimentally:

$$\begin{cases} I_0 = I_{\max} \\ F = \dfrac{I_{\max}}{I_{\min}} - 1 \end{cases}, \quad (2)$$

where $I_{\min}$ and $I_{\max}$ are the minimum and the maximum values of the transmission. Hence, Φ is the only unknown parameter in Eq. (1). If the wavelength and the angle of incidence are fixed, variations of the output intensity would only be due to a change of the optical thickness of the PTR glass window. The corresponding optical thickness can be extracted using the following equation:
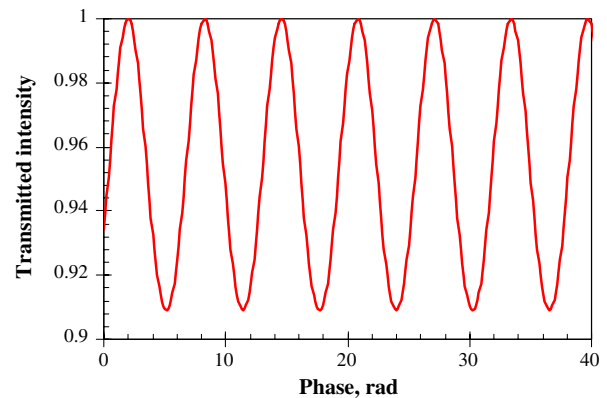

Fig. 2. (Color online) Dependence of the intensity of a beam transmitted through an optical flat on the total phase incursion in the plate.

$$\begin{cases} nt = \frac{\lambda_0}{2\pi} \arcsin \sqrt{\frac{1}{F} \left(\frac{I_0}{I_T} - 1 \right)} \\ nt \in \left[0, \frac{\lambda}{2} \right] \end{cases}. \quad (3)$$

In Fig. 2, the intensity of the transmitted beam (Airy formula) is shown as a function of the total phase incursion in the flat. However, because of the periodicity of the intensity of the transmitted beam, the only values of optical thickness change that can be unambiguously extracted are between 0 and $\lambda/2$. For the measurement of optical thickness variations greater than $\lambda/2$, this periodicity requires the continuous monitoring of the transmission evolution in order to take into account each 2π phase change.

B. Setup and Experimental Procedure

The method that we developed for the measurement of the absorption coefficient in PTR glass is based on the measurement of the phase incursions and therefore the optical thickness change during the laser-induced heating of the sample. The setup used for this measurement is presented in Fig. 3. It uses two lasers: an He–Ne laser operating at 633 nm used as a probe laser and a high-power laser (an IPG Photonics Yb-doped fiber laser with central wavelength of 1085 nm and output power up to 100 W) used as the pump or heating laser. A small fraction of the pump beam is reflected by a fused silica wedge and redirected to an InGaAs photodiode that measures the fluctuations in the pump power. The main part of the pump beam is transmitted through the fused silica wedge and overlaps with the He–Ne laser beam in the plane of the sample. The angle between the two beams is about 15°. The beam diameter of the probe beam is equal to 1 mm ($\mathrm{FWe^{-2}M}$), while the pump beam has a diameter of 7 mm ($\mathrm{FWe^{-2}M}$). Thus, the probed area of the sample can be considered to be homogeneously heated by the high-power radiation. After passing through the sample, the pump beam is sent to a beam dump while the probe laser beam is sent to a silicon detector to measure the variations of the transmitted power (Airy function). As the silicon detector has some sensitivity at 1085 nm, it can de-tect scattered radiation of the pump beam. To eliminate the effect of the scattered pumping radiation on the probe signal and improve the signal-to-noise ratio of our measurement, a chopper modulated at a frequency of 300 Hz is placed in the probe beam. The silicon detector is then connected to a data acquisition card associated with a computer using Labview programs that perform the signal acquisition, the lock-in detection, and all signal processing.

The measurement procedure is the following. The sample is placed in the setup at normal incidence to the probe beam. This condition allows us to obtain the maximum contrast of the modulation of the transmission. Then the sample is rotated from the normal incidence angle in order to determine the value of the maximum and minimum intensities that are required for calculating the I_0 and F parameters. When this is done, the sample is then realigned at normal incidence to the probe beam. Because the inclination angle needed to determine the values of the minimum and maximum intensities is small (only a few degrees), they can be considered as constant [15]. When the sample is thermally stable and the signal is therefore constant, the pump power is turned on and the time dependence of the transmitted probe beam power is measured; a typical signal is shown in Fig. 4(a). Intensity evolution after the pump beam is turned on follows the Airy formula. Depending on the initial position on this curve (rising or falling edge), the intensity can increase toward a maximum or decrease to a minimum [as seen in Fig. 4(a)]. Using Eqs. (2) and (3), it is then possible to extract the evolution of the optical thickness change of the glass plate during heating [Fig. 4(b)].

C. Calibration

Calibration of a calorimetric absorption measurement generally requires precise knowledge of the heat capacity of the sample as well as the thermal diffusivity or the thermal conductivity [16], the geometry of measurements (beam diameters, sample sizes, beam overlapping area), the conditions of thermal conductivity in the sample, and the parameters of heat exchange with the environment. While thermophysical parameters were precisely characterized
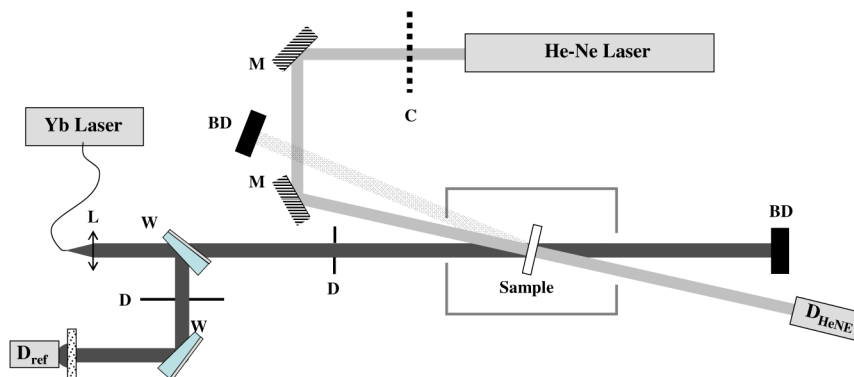

Fig. 3. (Color online) Optical setup designed for the measurement of the phase incursion induced by laser heating in an optical flat. W, wedge; L, lens; D, diaphragm; D_i, detector; M, mirror; C, chopper; and BD, beam dumps.
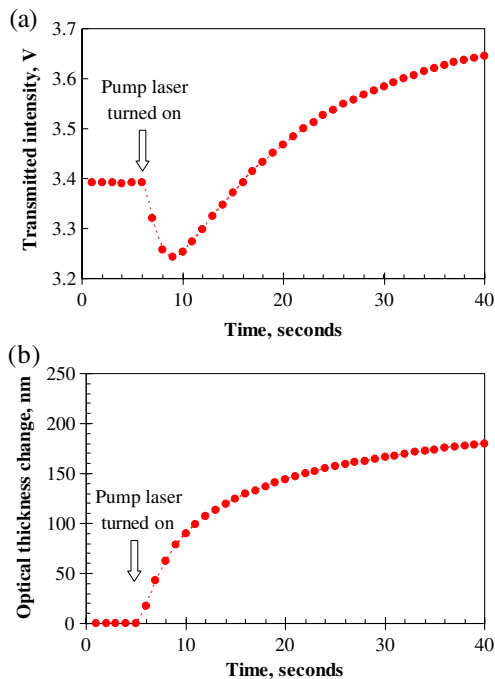
Fig. 4. (Color online) (a) Evolution of intensity of a probe beam transmitted through the sample exposed to a high-power pump beam and (b) corresponding evolution of the optical thickness. The exposure started at the time marked by the arrow.

for some of the classical glasses such as fused silica, BK7, etc., these data are not as precise when it comes to new materials such as PTR glass. In order to overcome this problem, we fabricated a calibration sample that was a specially prepared PTR glass doped with a very small amount of reduced iron Fe^{2+}. Fe^{2+} has a very well-known absorption band centered at $1.1 \mu m$ [17], and it is therefore possible to produce a glass with a predetermined absorption coefficient at 1085 nm. The calibration glass was doped with ~40 ppm of iron and resulted in an absorption coefficient ($A = \frac{\log_{10} T_i^{-1}}{t}$, where T_i is internal transmission and t is thickness) at 1085 nm of 3.3×10^{-2} cm^{-1}. The data for the calibration sample were determined with a precision of 10^{-3} cm^{-1} using a precise photometric measurement of the transmitted and reflected power. The basic composition of the calibration glass sample was identical to that of the glasses that were studied (except for the minor amount of Fe). Therefore, its thermal properties were identical to those of standard PTR glass. When this glass is inserted in the absorption measurement setup, a reference signal could be measured. The absorption coefficient of a PTR glass sample with unknown absorption could be calculated by comparing the signals detected for the test and the reference samples. This calculation requires knowing such measurable numbers as the intensities of the pump and probe beams, the thicknesses of the test, and the reference samples, but it does not require knowing thermophysical parameters of the studied material and the exact geometry of measurements. The single requirement is to have

identical thermophysical parameters and geometry for two sequential measurements. Moreover, it is important to note that we kept power low enough to avoid any nonlinear effects. We tested this assumption by showing that a 3× increase of the regular pump power as used for the calibration sample would result in a 3× increase of the measured optical thickness change.

Theoretically, the proportionality between optical thickness change and absorption coefficient is only correct for uniform heating of the sample and in the absence of energy exchange between the sample and environment. Actually, when the sample is heated by the laser, the geometry of the sample, the convection flows surrounding the glass, and the temperature of the surrounding air, etc., will influence the final measured phase shift and can affect the proportionality between the absorption coefficient and the temperature of the sample. In order to mitigate these effects, several precautions were taken. The first one was to construct a box surrounding the sample in order to minimize convection flows that can affect the temperature of the sample before and during the measurement. The second was to limit the measurements to 30 s in order to have a measurement long enough to obtain a good signal-to-noise ratio but also short enough to keep constant temperature of the surrounding medium and to minimize heat exchange between the sample and surrounding medium. The third and the most significant precaution was to vary the power of the heating beam such that the change in the optical thickness and therefore of the sample's temperature would be similar (within 20%) in all measured samples independent of their absorption coefficient. Using this additional precaution, it was therefore possible to prove that all thermal exchanges between the sample and the surrounding air were identical for both the unknown and the calibrated PTR glass samples and therefore to consider the measured changes of the optical thicknesses to be proportional to the glass absorption coefficient.

D. Precision and Repeatability of the Measurement

The repeatability of the measurement technique was determined by multiple measurements of the same sample that had an absorption coefficient of ~3 × 10^{-4} cm^{-1} Typical fluctuations of the absorption coefficient that were measured in this sample are summarized in Table 1. It is seen that the repeatability of the measurement is better than ±5% of the measured absorption coefficient. Moreover, this precision is the same for the samples with different absorption coefficient as laser heating of a sample is kept constant to allow accurate calibration (Subsection 2.C).

E. Effect of Sample Dimensions

The dimensions of the samples are another variable to be taken into account. Therefore, we experimentally investigated the influence of the sample's lateral size and thickness. A PTR glass sample with a thickness of 5 mm and lateral dimensions

Table 1. Repeatability of the Absorption Coefficient Measurements

Measurement No.	1	2	3	4	5	6
Absorption coefficient, cm^{-1}	1.06E − 04	1.08E − 04	1.16E − 04	1.20E − 04	1.10E − 04	1.12E − 04
					Average	1.12E − 04
					Standard deviation	4.5%

Table 2. Absorption Coefficient Measured in the Samples with Different Lateral Sizes

Sample size	35 mm × 35 mm	30 mm × 30 mm	25 mm × 25 mm	20 mm × 20 mm	15 mm × 15 mm
Absorption coefficient, cm^{-1}	1.06E − 04	1.09E − 04	1.09E − 04	1.06E − 04	1.12E − 04

Table 3. Absorption Coefficient Measured in the Samples with Different Thicknesses

Thickness	1.45 mm	2.82 mm	3.80 mm	5.15 mm
Absorption coefficient, cm^{-1}	1.31E − 04	1.37E − 04	1.28E − 04	1.29E − 04

of 35 mm × 35 mm was measured. Then lateral dimensions of this sample were sequentially decreased by 5 mm in each direction down to 15 mm × 15 mm, and the sample's absorption coefficient was remeasured after each cut. The results are summarized in Table 2. It is seen that, if the sample's lateral dimensions are kept at least two times larger than the diameter of the pump beam, no detectable changes within the accuracy of the measured absorption coefficient are seen. This effect is first associated with the fact that, with such a ratio, there is no interaction of the pump beam with the sample edges. Moreover, due to the low thermal conductivity of glasses associated with the short duration of the measurement (30 s), edge effects can be thus neglected. The same experiment was done with a series of 25 mm × 25 mm samples having different thicknesses (Table 3). Once again, no detectable changes of the measured absorption coefficient within the accuracy of measurements could be observed in this range of thicknesses.

F. Sensitivity of the Method

Regarding the sensitivity of the proposed method, based on the signal-to-noise ratio, the smallest optical thickness change that can be detected after 30 s of heating is in the range of ~2 nm. Based on the calibration curve, such a change can occur in a 5 mm thick sample with an absorption coefficient of $4 \times 10^{-6} cm^{-1}$. This value therefore constitutes the limit of absorption that can be detected by this method in this particular embodiment, which is based on a 100 W pump beam, a 1 mW probe He–Ne beam, associated with standard Thorlabs amplified silicon detectors and a National Instrument data acquisition card. Based on the thermal properties of PTR glass, it is possible to convert this optical thickness change into a change of temperature after 30 s of heating [18]. At the detection limit, the minimum detected temperature change is equal to 0.05 K. Hence, the sensitivity of the method in its current implementation is comparable to the sensitivity of other calorimetric techniques [19]. Further improvement of the sensitivity of the methods would require overcoming the temperature fluctuations of the surrounding medium as well as decreasing the level of noise of the laser. The first problem could be solved by placing the sample in vacuum in a temperature regulated chamber, while the second problem could be solved by adding a second detector and performing an autobalanced detection.

3. Absorption and Laser-Induced Heating in PTR Glass

A. Absorption in Virgin PTR Glass

A series of PTR glasses was prepared by melting high-purity raw materials in a Pt crucible with stirring. The absorption coefficient was measured in PTR glass samples selected from different melts, and the data are summarized in Table 4. One can see that absorption coefficients range from 5×10^{-5} to $2 \times 10^{-4} cm^{-1}$ from melt to melt. This absorption is extremely low compared to the one of glasses fabricated using conventional melting in an electric furnace and is 1 order of magnitude (or more) lower than the one of commercial BK7 glass [19]. Multicomponent silicate glasses similar to BK7 or PTR glass do not have intrinsic absorption in the near-IR spectral region. The residual near-IR absorption in such glasses is caused by different types of impurities. The most probable reason for absorption in the vicinity of 1 μm is due to residual contamination by iron. Such a low level of absorption in PTR glass is achieved through the use of high-purity chemicals and clean melting conditions that keep iron contaminations below 1 part per million (ppm). Variations in the absorption coefficient at 1 μm could be caused both by differences in the total concentration of iron and redistribution between different valence states of Fe^{2+} and Fe^{3+}.

B. Absorption in VBGs

The fabrication of VBGs in PTR glass requires complex processing [2] (UV exposure followed by thermal treatment) that might induce additional losses. We therefore measured the absorption

Table 4. Absorption Coefficient of Virgin PTR Glass Samples from Different Melts

Melt ID No.	PTR1	PTR2	PTR3	PTR4	PTR5	PTR6	PTR7
Absorption coefficient, cm^{-1}	1.02E − 04	8.61E − 05	8.96E − 05	9.30E − 05	1.15E − 04	1.56E − 04	6.55E − 05

coefficient in reflecting Bragg gratings having a central wavelength of 1064 nm and 95 + % diffraction efficiency; these gratings are similar to those that were used for spectral beam combining of high-power fiber lasers [5]. An absorption coefficient as low as 1×10^{-4} cm^{-1} was achieved in such VBGs by purification and improving the processing of the VBGs after fabrication. However, it is somewhat higher than the absorption coefficient that was measured in the virgin PTR glass samples. This increase can be explained by the change in oxidation state of several ions in the glass (cerium, silver, iron, etc.) that lead to additional absorption bands in both the visible and near-IR ranges [17,20]. The ability to measure a low absorption coefficient in both virgin PTR glass and in holographic optical elements produced from this glass is important for modeling of the performance of PTR VBGs in high-power laser beams.

C. Laser-Induced Heating in Near-IR Region by High-Power CW Radiation

To provide accurate measurements of the absorption coefficient, periods of laser heating and data acquisition were limited to 30 s. It allowed us to minimize temperature changes of the sample and the surrounding environment. However, the time duration of this measurement can be extended until an asymptote or saturation level is reached (i.e., it no longer heats) that is determined by the thermal conductivity in the test sample and the heat exchange with the environment. Using the data obtained after prolonged heating, it is possible to calculate for this experimental configuration the maximum optical thickness change that will occur for this level of heating. Both the absorption coefficient and the change in optical thickness per incident power at 1085 nm at saturation were measured and plotted against each other for PTR glass samples with different absorption coefficients (Fig. 5). The correlation appears linear and the slope of the linear relation has the value of ~5000 nm/W cm^{-1} for ~5 mm thick samples. This means that only linear absorption is responsible for heating of the grating (i.e., there are no nonlinear processes involved in the heating).

Let us consider the effect of high-power laser radiation on optical components (VBGs) fabricated from PTR glass. The first effect to consider is the possible change of the resonant wavelength. The resonant wavelength of a VBG (let us consider λ_{1064} for specificity) is given by the Bragg formula:

$$\lambda_{1064} = 2n_0\Lambda, \qquad (4)$$

where n_0 is the mean refractive index and Λ the grating period. For simplicity, let us consider uniform heating of the grating by laser radiation. This heat-

ing will result in a thermal shift of the central wavelength of the VBG:

$$\frac{1}{\lambda_{1064}}\frac{\partial\lambda_{1064}}{\partial T} = \frac{1}{n_0\Lambda}\frac{\partial n_0\Lambda}{\partial T} = \frac{1}{\Lambda}\frac{\partial\Lambda}{\partial T} + \frac{1}{n_0}\frac{\partial n_0}{\partial T}. \qquad (5)$$

The laser-induced change of the grating period is determined by the laser-induced expansion of the grating material. The geometrical thickness t of such a periodic structure as a VBG can be defined in the following way:

$$t = (N + \varepsilon)\Lambda, \qquad (6)$$

where N is an integer number of periods and ε the fractional number. These two quantities are constant and independent of the temperature of the grating; only Λ changes with heat or stress. Hence, we can write that

$$\frac{1}{n_0\Lambda}\frac{\partial n_0\Lambda}{\partial T} = \frac{1}{n_0 t}\frac{\partial n_0 t}{\partial T} = \frac{1}{\lambda_{1064}}\frac{\partial\lambda_{1064}}{\partial T}. \qquad (7)$$

In other words, the relative change of the central wavelength of the grating is equal to the relative change of the grating period or to the relative change of the optical thickness of the PTR glass window. Hence, the characterization of the optical thickness change that was described above can be directly used to assess the change of the Bragg wavelength of the VBG in PTR glass as a function of the PTR glass absorption coefficient and incident power. Figure 6 shows this thermal shift of wavelength for natural convective cooling of a glass plate. One can see that, if the PTR glass absorption coefficient is equal to 10^{-4} cm^{-1}, 1 kW is expected to shift the central wavelength by ~70 pm while 100 kW will shift it by ~7 nm. The thermal coefficient of the Bragg wavelength in a PTR VBG at 1 μm is equal to ~9 pm/K. It means that, for 1 kW and 100 kW of transmitted power with no active cooling, the temperature would
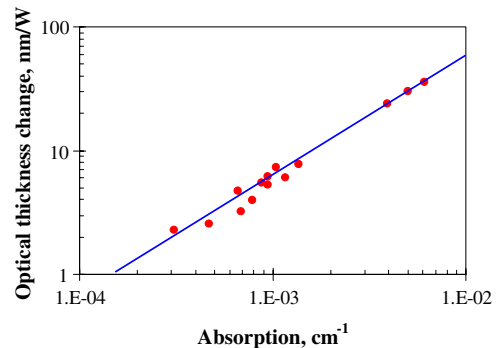

Fig. 5. (Color online) Correlation between specific optical thickness change ($\Delta nt/P$) and PTR glass absorption coefficient.
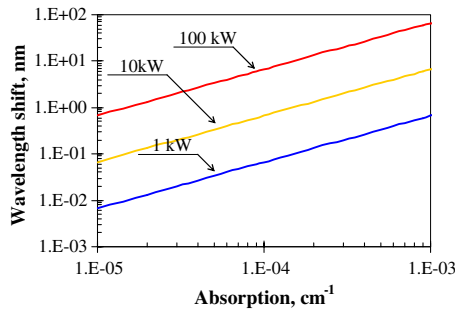
Fig. 6. (Color online) Bragg wavelength shift in a conventionally cooled in air PTR glass plate versus the absorption coefficient for different incident power of laser beams.

reach $8\,°C$ and $800\,°C$, respectively. Thus, the level of absorption in commercially produced PTR glass and VBGs allows it to be used for spectral beam combining and other types of laser beam control at several kilowatts without active cooling, while scaling to tens and hundreds of kilowatts requires decreasing the residual absorption down to $10^{-5}\,cm^{-1}$ or/and active cooling of elements.

4. Conclusions

An interferometric method for measuring absorption coefficient in optical flats and holographic optical elements recorded in these flats is presented. It is based on sample heating by a pump beam while using a probe beam to measure the increase in optical thickness. Measurement of the evolution of the intensity of a probe beam transmitted as a function of laser heating time enables calculation of the absorption coefficient of the sample. The calibration of the measurement was made with the use of a sample of the same glass with a known absorption coefficient. The sensitivity of the method is approximately $10^{-6}\,cm^{-1}$. An absorption coefficient as low as $5 \times 10^{-5}\,cm^{-1}$ was measured in commercially produced PTR glass, while an absorption coefficient as low as $1 \times 10^{-4}\,cm^{-1}$ was measured in high-efficiency VBGs. The achieved level of absorption in a PTR VBG allows its use at the $10\,kW$ level without active cooling, while operation at the $100\,kW$ level would require active cooling and/or decreasing the absorption coefficient in the PTR Bragg gratings by an order of magnitude.

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