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Optics and photonics — Laser and laser-related equipment — Photothermal technique for absorption measurement and mapping of optical laser components

Optique et photonique — Lasers et équipements associés aux lasers — Technique photothermique pour la mesure et la cartographie de l'absorption des composants laser optiques

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 172, *Optics and photonics*, Subcommittee SC 9, *Laser and electro-optical systems*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

With the rapid development of high-power/high-energy laser technology, laser-induced damage to optical laser components and laser-induced thermal distortion in laser components become the most important limiting factors for the operation and applications of high-power/high-energy laser systems. Normally, the laser-induced damages to optical laser components are caused by absorbing defects on the surface or within the laser components which result in thermal stress or melting of the laser components and lead to damage. The thermal distortions, which induce wavefront distortions and therefore beam quality deteriorations to the laser beam, are caused by non-uniform thermal expansion or refractive index change due to absorption irregularities (such as absorbing defects) inside the laser components. To improve the laser-induced damage threshold (LIDT) and reduce the laser-induced thermal distortion of laser components used in high-power/high-energy laser systems, there are needs not only to measure precisely the absorptance of the laser components, but also to detect various absorbing defects on/within the laser components, therefore to improve the performance of these laser components via optimizing fabrication/coating processes.

Currently, the ISO 11551 standardized testing method - laser calorimetry for absorptance measurements of optical laser components can only measure test samples with small sizes (normally less than 50 mm in diameter and 10 mm in thickness) and has almost no capability to measure the absorptance of large-sized laser components (100 mm in diameter and over) widely used in high-power/high-energy laser systems. In addition, laser calorimetry has only limited capability to map the absorptance distribution of an optical laser component.

The measurement procedures in this document have been optimized to allow the mapping of absorbing defects of optical laser components and measurement of absolute absorptance of large-sized laser optics actually used in high-power/high-energy laser systems using photothermal techniques which provide absorption measurement/mapping with high sensitivity, high spatial resolution, and high reliability.

In addition to absorption measurement/mapping of optical laser components with photothermal amplitude, the photothermal phase measurement/mapping can also find applications in thermo-physical characterization of laser optics, which will be helpful for a better understanding of defect properties of laser optics and laser-defect interaction that would lead to a better understanding of laser-induced damage mechanism of laser optics.

Optics and photonics — Laser and laser-related equipment — Photothermal technique for absorption measurement and mapping of optical laser components

1 Scope

This document specifies procedures for the absorption measurement and high spatial-resolution two-dimensional or three-dimensional absorption mapping of optical laser components, and upon calibration, the measurement of absolute absorptance of laser optics.

The methods given in this document are intended to be used for the two-dimensional or three-dimensional absorption mapping of optical laser components, that is, measurement of absorption as a function of position, as well as absorption/absorptance measurement and mapping of laser optics used in high-power/high-energy laser systems.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 11145, *Optics and photonics — Lasers and laser-related equipment — Vocabulary and symbols*

ISO 14644-1, *Cleanrooms and associated controlled environments — Part 1: Classification of air cleanliness by particle concentration*

ISO 80000-7, *Quantities and units — Part 7: Light and radiation*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11145 and ISO 80000-7 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
absorption
radiant flux absorbed by the optical laser component

3.2
absorptance
ratio of the radiant flux absorbed to the radiant flux of the incident radiation

3.3
absorption map
absorptance map
measured *absorption* (3.1)/*absorptance* (3.2) as a function of sample position

Note 1 to entry: The definition of absorptance used for this document is limited to absorptance processes which convert the absorbed energy into heat. For certain types of optics and radiation, additional non-thermal processes may result in absorption losses which will not be detected by the test procedure described here.

4 Symbols used and units of measure

Table 1 — Symbols used and units of measure

Symbol	Term	Unit
A	Absorptance of test sample	
A_0	Absorptance of calibration sample	
S	Photothermal amplitude of test sample	
S_0	Photothermal amplitude of calibration sample	
P, P_0	Pump laser power	W
$\Delta I, I_0$	Probe beam intensity change and dc probe intensity detected in photothermal lensing	
I_1, I_2	Probe beam intensity detected by the two photo-detectors of a bi-cell photo-detector in photothermal deflection	
D	Thermal diffusion coefficient of test sample	m^2s^{-1}
$\Delta\varphi(x, y)$	Photothermally induced optical phase shift to probe beam	
α_{th}	Linear thermal expansion coefficient of test sample	K^{-1}
dn/dT	Temperature coefficient of refractive index of test sample	K^{-1}
ν	Poisson ratio of test sample	
f	Modulation frequency of pump laser power	Hz
μ_{th}	Thermal diffusion length of test sample	m
a	Pump beam radius in test sample	m
λ	Probe laser wavelength	m
z	Detection distance	m
$T(x, y, z)$	Photothermally induced temperature rise distribution inside test or calibration sample	K
B, C	Proportional coefficients	
β	Slope of linear fit of the measured absorptance dependence of the power-normalized photothermal amplitude of calibration samples	

5 Test method

5.1 Test principle

5.1.1 General

Based on photothermal effects, photothermal techniques are highly sensitive for the measurement of weak absorptance of optical laser components. In a typical photothermal experiment, a continuous-wave or highly repetitive pulsed excitation laser (pump laser) beam is used to irradiate the sample under test, heat is created within the sample due to optical absorption, and a temperature rise distribution within the sample is formed. For optical laser components, a displacement is induced on the sample surface due to thermal expansion, and a refractive index gradient is formed within the sample due to the temperature dependence of refractive index. By employing photothermal techniques to detect the surface displacement or refractive index gradient with a second probe laser beam, the

absorptance (absorption) of the sample can be determined. The absolute absorptance can be obtained by calibrating the photothermal amplitude. By measuring the absorption/absorptance as a function of position, the absorption/absorptance map of a laser component is obtained.

Photothermal lensing (or thermal lens - TL) and photothermal deflection (PTD) are appropriate detection techniques for absorption measurement and mapping of optical laser components. Two detection schemes, both reflected and transmitted probe beam detections, can be used to measure the photothermal signal amplitude, which is linearly proportional to the absorption/absorptance of optical laser components under test when the photothermally induced optical phase shift to the probe beam is relatively small as compared to the probe beam wavelength (small signal approximation).

5.1.2 Photothermal lensing (TL)

In a typical TL scheme, an unfocused probe beam irradiates the pump laser-induced surface displacement zone or the refractive index gradient zone which acts as a negative (or positive) lens. The photothermal signal is represented by the intensity change at the centre of either the reflected probe beam (in surface TL – STL scheme) or the transmitted probe beam (in transmitted TL – TTL scheme). This probe beam intensity change can be detected by a pinhole photodetector (a photodetector with a pinhole in front of it).

5.1.3 Photothermal deflection (PTD)

In a typical PTD scheme, a tightly focused probe beam irradiates the pump laser-induced surface displacement zone or the refractive index gradient zone, the reflected probe beam is deflected due to the slope of the surface displacement in a reflected PTD configuration, or the transmitted probe beam is deflected due to the refractive index gradient in a transmitted PTD configuration. The photothermal signal is represented by the probe beam deflection, which can be easily detected by a position-sensitive photodetector (for example, a bi-cell photodetector).

5.1.4 Rules for selecting reflected and transmitted photothermal detection schemes

Selecting between the reflected or transmitted photothermal detection schemes (both TL and PTD) should be considered as follows. As a general rule, the detection scheme with a higher detection sensitivity should be selected for more sensitive and precise absorption measurement. For an optical laser component with a (much) larger linear thermal expansion coefficient so that the photothermally induced optical phase shift to the probe beam by the thermal expansion caused surface displacement is large, a reflected photothermal detection scheme is preferable. On the other hand, a transmitted photothermal detection scheme should be selected if the temperature coefficient of refractive index of the component is much larger so that the optical phase shift to the probe beam caused by the temperature rise induced refractive index change is much larger than that caused by the surface displacement. However, if the sample under test is opaque to the probe beam, the reflected photothermal detection scheme should be selected.

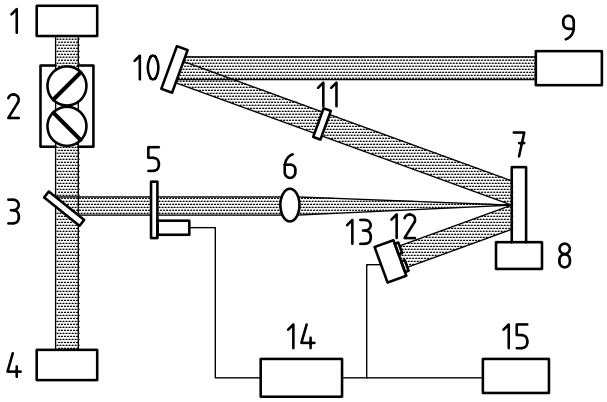
For photothermal absorption measurement of bulk samples and separation of surface absorption and bulk absorption, the transmitted photothermal detection scheme is preferable. The reflected photothermal detection scheme may be used for measurement of bulk samples only when the bulk sample is homogeneous and has negligible surface absorption.

5.2 Measurement arrangement and test equipment

5.2.1 Photothermal detection arrangement

There are four photothermal detection arrangements that may be used to measure and map the absorption of optical laser components. That is, surface thermal lens (STL), transmitted thermal lens (TTL), reflected photothermal deflection (or photothermal displacement) (reflected PTD), and transmitted photothermal deflection (transmitted PTD).

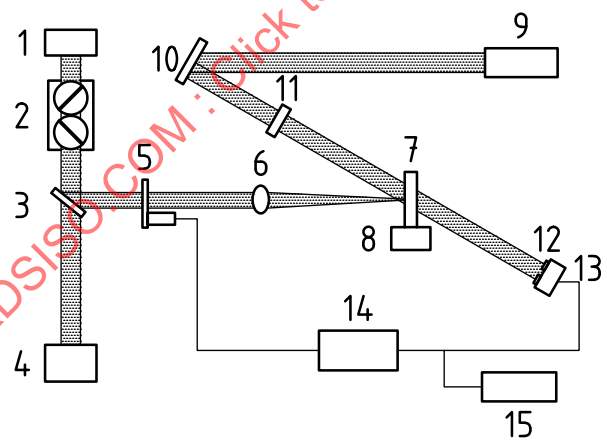
Figures 1 to 4 show typical experimental arrangements for the STL, TTL, reflected PTD, and transmitted PTD detection schemes, respectively.



Key

1	pump laser	9	probe laser
2	variable attenuator for pump beam	10	mirror
3	beam-splitter	11	attenuator for probe beam
4	power meter	12	pinhole
5	mechanical chopper	13	photo-detector
6	lens	14	lock-in amplifier
7	sample under test	15	oscilloscope
8	translation stage		

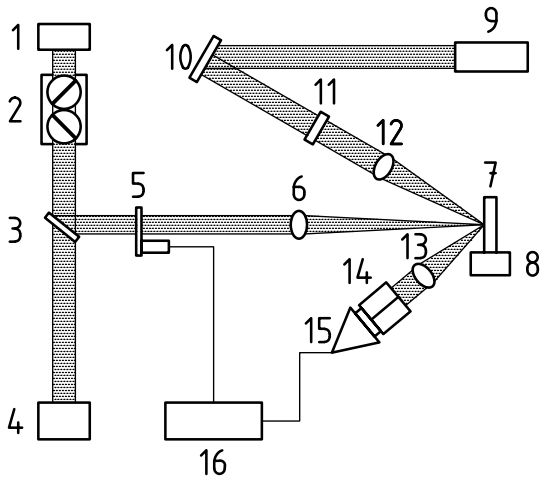
Figure 1 — Typical experimental arrangement for the STL detection scheme



Key

1	pump laser	9	probe laser
2	variable attenuator for pump beam	10	mirror
3	beam-splitter	11	attenuator for probe beam
4	power meter	12	pinhole
5	mechanical chopper	13	photo-detector
6	lens	14	lock-in amplifier
7	sample under test	15	oscilloscope
8	translation stage		

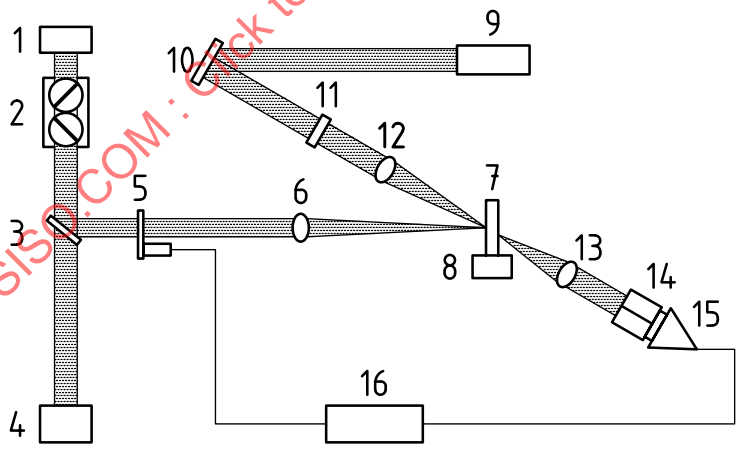
Figure 2 — Typical experimental arrangement for the transmitted TL detection scheme



Key

1	pump laser	9	probe laser
2	variable attenuator for pump beam	10	mirror
3	beam-splitter	11	attenuator for probe beam
4	power meter	12, 13	lens
5	mechanical chopper	14	bi-cell photo-detector
6	lens	15	differential amplifier
7	sample under test	16	lock-in amplifier
8	translation stage		

Figure 3 — Typical experimental arrangement for the reflected PTD detection scheme



Key

1	pump laser	9	probe laser
2	variable attenuator for pump beam	10	mirror
3	beam-splitter	11	attenuator for probe beam
4	power meter	12, 13	lens
5	mechanical chopper	14	bi-cell photo-detector
6	lens	15	differential amplifier
7	sample under test	16	lock-in amplifier
8	translation stage		

Figure 4 — Typical experimental arrangement for the transmitted PTD detection scheme

5.2.2 Pump laser

The pump laser is a continuous-wave or highly repetitive pulsed laser used to heat the test sample. Wavelength of the laser source, angle of incidence and state of polarization shall correspond to those specified by the manufacturer for the use of the test sample. The state of polarization (*p* or *s*) of the laser beam shall be selected by the polarizer. If the value ranges are acceptable for these three quantities, any combination of the wavelength, angle of incidence and state of polarization may be chosen within these ranges.

The (average) power of the pump laser should be sufficiently high so that the surface displacement or refractive index gradient created by the pump beam absorption of the test sample is highly detectable. The pump laser power should be adjustable via a variable attenuator which should not create any change to the beam profile of the pump beam during power adjustment. The pump beam profile shall be the same at all times, during the calibration and during the photothermal measurement at each adjusted power. The pump laser power is periodically modulated by a mechanical chopper or an acoustic-optic modulator. The modulation frequency is selected for optimal signal-to-noise ratio (SNR) of the photothermal signal.

For absorption mapping performed longer than several minutes, the power stability of the pump laser shall be monitored by a power meter or photo-detector as shown in Figures 1 to 4, and if needed its influence on the absorption mapping shall be eliminated by normalizing the photothermal amplitude with the monitored output power.

The pump beam is focused into the test sample to create enough temperature rise inside the test sample. The beam size of the pump beam on/inside the test sample is optimized taking into account the SNR of the photothermal signal, the area and spatial resolution of absorption mapping. Care shall be taken to avoid laser damage to the test sample due to too tight focusing.

5.2.3 Probe laser

The probe laser is a continuous-wave laser used to detect the photothermal signal. Normally a highly stable He-Ne or diode laser with a TEM_{00} mode output is used as the probe laser. The output power of the probe laser should be low so that the heat created by the probe beam absorption of the test sample is negligible.

For TL detection, the probe beam is normally not focused, or the probe beam size is at least five times larger than that of the focused pump beam in the interaction zone of the two beams. For PTD detection, the probe beam is focused. The size of the focused probe beam is at least smaller than that of the pump beam in the interaction zone. The separation between the pump and probe beams has to be adjusted to maximize the photothermal (lensing or deflection) amplitude for optimum SNR.

For absorptance measurements and two-dimensional absorption mapping, the angle of incidence of the probe beam with respect to the sample normal should be small, for example smaller than 10 degrees. For three-dimensional absorption mapping or separation of surface absorption and bulk absorption, the angle of incidence of the probe beam with respect to the pump beam should be adjusted taking into consideration depth resolution and SNR of photothermal signal. The angle of incidence of the probe beam should be documented.

A detailed description on the separation of surface and bulk absorption is given in Annex B.

5.2.4 Translation stage

A two-dimensional/three-dimensional translation stage is used to move the test sample in order to measure the absorption as a function of position, so that a two-dimensional or three-dimensional absorption map is obtained. The translation stage should be motorized with a position resolution better than 10 µm and controlled by a computer software.

5.2.5 Detection unit

For TL detection, the detection unit consists of a pinhole and a photo-detector, which is appropriate for the probe laser wavelength, and a lock-in amplifier. As a general rule, the size of the pinhole is comparable to that of the surface displacement or refractive index gradient zone.

For PTD detection, the detection unit consists of a position-sensitive photo-detector (e.g. a bi-cell photo-detector), a differential amplifier, and a lock-in amplifier. The position-sensitive photo-detector should be appropriate for the probe laser wavelength, and the total detection area should be larger than the probe beam size to make sure that all probe power is detected by the photo-detector. A positive lens may be used in between the sample and photo-detector to adjust the probe beam size on the detection area of the photo-detector. If a lens is used, it shall be ensured that no imaging of the probe beam spot at the sample onto the detector is established. A differential amplifier is used to delete the dc level of the probe beam deflection signal.

In both cases the reference frequency of the lock-in amplifier should be the same as the modulation frequency of the pump laser power, and the time constant of the lock-in amplifier should be set taking into consideration the temporal resolution and SNR of photothermal signal.

5.2.6 Data acquisition and processing

At each position of the test sample, the amplitude and phase of the modulated photothermal signal is recorded via the lock-in amplifier for a certain time and averaged over a certain number of repeat measurements. The photothermal measurement may also be repeated at several different sample positions. The reported absorption/absorptance value is the average of repeat measurements performed at a certain position or the average of measurements performed at the different positions. For absorption mapping, the test sample is moved by controlling the motorized translation stage and photothermal amplitude and phase are measured as a function of sample position. By choosing an appropriate scan step and a measurement area, a map of photothermal amplitude is obtained, which represents the absorption map. In addition, a map of photothermal phase is obtained, which gives useful information on the thermo-physical characteristic distribution of the test sample.

5.2.7 Environment

The environment of the testing place should consist of dust-free filtered air with 40 % to 60 % relative humidity. The residual dust shall be reduced in accordance with the clean-room Class 7 as specified in ISO 14644-1.

5.3 Preparation of test sample

In general, test samples should have flat surfaces, for example un-coated, highly reflective (HR) or anti-reflective (AR) coated plane substrates. The absorption/absorptance of samples with curved surfaces could also be measured. However, care shall be taken for absorption mapping of samples with curved surfaces as the surface curvature shall be considered to keep identical alignment at each position during position scanning.

Storage, cleaning and preparation of the test samples shall be carried out in accordance with the instructions of the manufacturer.

6 Test procedure

6.1 General

Either STL, TTL, reflected PTD, or transmitted PTD configuration shall be chosen to measure and map the absorption/absorptance of the optical laser component. The amplitude of the photothermal signal shall be measured to determine the absorption, and upon calibration, to determine the absorptance of the test sample. The sample position has to be scanned to map the absorption/absorptance of the test sample.

The incident angle of pump beam to the test sample shall be set according to the manufacturer's instruction. For a sample with normal incidence, the absorption measurement should be performed with the incident angle in between 1° to 10°, which shall be documented.

6.2 Measurements of photothermal amplitude and phase

The absorption of the test sample is determined photothermally by means of one of the photothermal measurement arrangements described in Figures 1 to 4 (STL, transmitted TL, reflected PTD, and transmitted PTD, respectively). Before the photothermal amplitude via lock-in amplifier readout is recorded, the experimental parameters of the photothermal measurement have to be optimized and the optical arrangement of the photothermal measurement has to be aligned carefully. The modulation frequency of the pump beam, the separation between the pump and probe beams, the detection distance (that is, the distance from the sample to the photo-detector), and the transverse position of the photo-detector are adjusted to maximize the amplitude and SNR of the photothermal signal. Also, the waiting time should be long enough, to allow for the photothermal signal to be stabilized after careful alignment. The photothermal amplitude is recorded at least 5 times and an average is obtained for one position.

For photothermal measurements, when setting the modulation frequency of the pump laser and time constant of the lock-in amplifier the thermal diffusion properties of the sample shall be taken into consideration. A lower modulation frequency and a longer time constant shall be set for a sample with a lower thermal diffusion coefficient. For laser optics with fused silica, N-BK7, or sapphire as the substrates, it is recommended to set the modulation frequency to between 50 Hz to 500 Hz, and set the time constant to between 0,1 s to 1 s. For TL measurement, the pump and probe beams should be overlapped with zero separation, the transverse position of the photo-detector should be nearly centred related to the probe beam profile to maximize the photothermal signal amplitude. The detection distance is set by the following Formula (1):

$$z = B \cdot \frac{\pi a^2}{\lambda} \tag{1}$$

where

- a is the radius of the pump beam at the sample surface or interaction zone;
- λ is the probe beam wavelength;
- B is a coefficient between 1 and 10, depending on the radius of the pump beam and the modulation frequency.

For PTD measurement, the probe beam should be separated from the pump beam by approximately one time the radius of the pump beam, and the transverse position of the position-sensitive photo-detector is adjusted to have nearly zero dc output of the differential amplifier.

NOTE The detection distance is at least 10 cm or larger, depending on the radius of the pump beam and the modulation frequency.

6.3 Maps of photothermal amplitude and phase

Control the position of the test sample via a motorized translation stage. Set up a measurement area of the test sample and a scan step for the mapping via a control software. Scan the test sample and record the photothermal amplitude and phase as a function of the scanning position. During the position scanning, at each position a waiting period of three to five times the time constant of the lock-in amplifier is required before signal recording to allow for the photothermal signal to be stabilized. The map of the photothermal amplitude represents the absorption map of the test sample, and the map of the photothermal phase provides information on the distribution of the thermo-physical properties of the test sample. As an example, Figure 5 shows a typical absorption map of a test sample measured by reflected STL scheme.

By tightly focusing the pump beam and scanning the sample position three-dimensionally, a three-dimensional absorption map of a bulk sample can be obtained. An example is presented in Figure 6, which is the three-dimensional absorption map of a fused silica substrate.

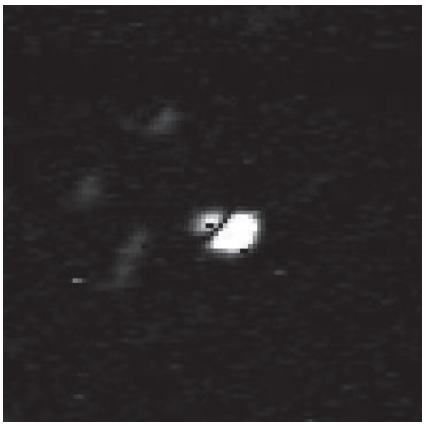


Figure 5 — Absorption map of a test sample measured by reflected STL scheme
(Mapping area: 5 mm × 5 mm)

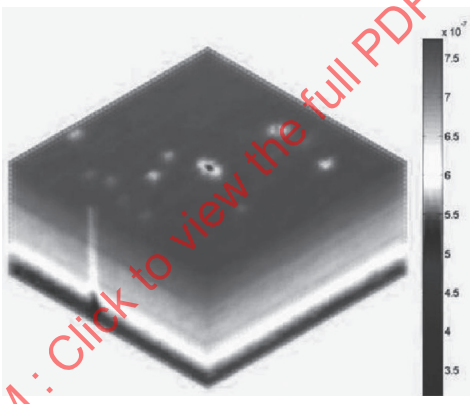


Figure 6 — Three-dimensional absorption map of an optical substrate measured by TL scheme

6.4 Calibration of photothermal amplitude

As the photothermal amplitude is linearly proportional to the absorptance of the test sample, by calibrating the photothermal amplitude with a calibration sample of a "known" absorptance and without absorption inhomogeneities, the measured photothermal amplitude can be used to determine the absolute absorptance of the test sample. It is recommended to calibrate the photothermal amplitude with "known" sample with the same or similar thermo-physical parameters as the test sample and under the same experimental conditions. The absorptance of the calibration sample can be determined by spectrophotometry, laser calorimetry, etc.

It is recommended to use calibration samples with the same functions as the samples under test. That is, an uncoated calibration sample (or sample set) for uncoated test samples, a HR-coated calibration sample for HR-coated test samples, and an AR-coated calibration sample for AR-coated test samples. For the AR-coated calibration sample, both the absorption coefficient of the substrate and the absorptance of the AR coating have to be known, and the substrate material shall be the same as that of the sample under test.

Prepare a calibration sample with a relatively high absorptance (up to approximately 0,1) or a relatively low absorptance (between 10^{-5} and 0,001). For the high-absorptance calibration sample, the absorptance may be determined by a commercial spectrophotometer or photometric method; for the low-absorptance calibration sample, it is recommended to determine the absorptance with laser

calorimetry, ISO 11551 for testing the absorptance of optical laser component. When calibrating the photothermal amplitude with the high-absorptance calibration sample, the power of the pump laser shall be reduced to obtain the photothermal amplitude which is comparable to that measured with the test sample without reduction of the pump power. It is not recommended to adjust the pump power when the low-absorptance calibration sample is used for calibration. It is important to note that when the high-absorptance calibration sample is used for calibration, the resulting photothermal amplitude is within the "small-signal approximation", which can be checked by measuring the pump power dependence of the photothermal amplitude.

The determination of the absorption of the AR-coated calibration sample can be performed as follows: First determine the absorption coefficient of the substrate by measuring its absorptance with laser calorimetry; then one-side AR-coat the substrate and measure the total absorptance of the AR-coated substrate by laser calorimetry again. The absorptance of the AR-coating is then determined by assuming the absorptance of the substrate does not change during coating process and subtracting the substrate absorptance from the measured total absorptance. It is recommended that only low-absorptance calibration samples are prepared and used for the absorptance measurements of AR-coated samples.

The calibration can also be performed with a set of (at least three) calibration samples of "known" absorptance ranging from a few (or a few tens of) 10^{-6} to approximately 0,1. A linear dependence of photothermal amplitude on the absorptance is expected by normalizing the photothermal amplitude by the pump power, which is adjusted for appropriate photothermal amplitude levels with different absorptance of calibration samples. The slope of this linear dependence can be used to calibrate the photothermal amplitude.

The structural parameters of the calibration sample should be specified. For HR-coated calibration samples, a circular sample is preferable with diameter much larger than that of the pump laser induced displacement region or refractive index gradient region as well as the thermal diffusion length. The thickness of the calibration sample is much larger than the thermal diffusion length. The thermal diffusion length is defined as given by Formula (2):

$$\mu_{\mathrm{th}} = \sqrt{D / \pi f} \tag{2}$$

where

D is the thermal diffusion coefficient of the test sample;

f is the modulation frequency of the pump laser.

NOTE Formula (2) is for the thermal diffusion length defined for a one-dimensional heat conduction only.

In this case the influence of the structural parameters on the photothermal signal of the calibration sample is negligible. This calibration procedure can be used for the calibration of the photothermal amplitude for the determination of absorptance of large-sized reflective laser components.

For the determination of absorptance of small-sized laser components, the calibration sample should have the same or similar structural parameters as the test sample.

Theoretical and practical considerations on the calibration are given in Annex A.

The calibration procedure for photothermal measurement in the depth direction of a three-dimensional absorption mapping is described in Annex B.

6.5 Assessments of the measurement

For absorption measurement and mapping, the amplitude map represents the absorption distribution of the test sample. For determination of the absorptance, the measurement error is mainly caused by the calibration error and uncertainties of other experimental parameters. The typical uncertainty for the absorptance measurement is in the order of 10 % to 20 %.

7 Evaluation

7.1 Determination of absorption via photothermal measurement

In general, for photothermal measurement, the photothermal signal is a complex function of many parameters as listed in Table 2. A rigorous expression for the photothermal signal of an optical laser component is complicated. However, for absorption measurements of optical laser components with the photothermal technique, the physical basis is that under small-signal approximation, the photothermal amplitude is linearly proportional to the absorptance of the test sample. With this linear proportionality, a photothermal amplitude can represent the absorption, a photothermal amplitude map linearly represents the absorption map, and upon calibration, the absorptance can be determined from the photothermal amplitude.

Table 2 — Parameters that affect the photothermal signals in photothermal absorption measurements

Test sample's parameters	Instrumental/experimental parameters
Optical parameters:	Pump beam's parameters
Absorption coefficient	Shape
Absorptance	Size
Thermo-physical parameters	Power
Thermal conductivity	Intensity distribution
Thermal diffusion coefficient	Modulation frequency
Linear thermal expansion coefficient	Probe beam's parameters
Temperature coefficient of refractive index	Shape
Mechanical parameter	Size
Poisson ratio	Power
Structural parameters	Intensity distribution
Shape	Wavelength
Size	Angle of incidence
	Other experimental parameters
	Separation of pump and probe beams
	Detection distance

For either TL or PTD detection scheme, the photothermal signal is linearly proportional to the photothermally induced optical phase shift, which, for the reflected photothermal configuration, is approximately expressed as given by Formula (3):

$$\Delta\varphi(x,y)=\alpha_{\mathrm{th}}\cdot\frac{4\pi}{\lambda}\cdot\frac{1+\nu}{1-\nu}\int T(x,y,z)\,\mathrm{d}z \tag{3}$$

where

a_{th} is the linear thermal expansion coefficient of the test sample,

ν is the Poisson ratio of the test sample,

λ is the probe beam wavelength, and

$T(x, y, z)$ is the photothermally induced temperature rise inside the test sample.

The integration is performed along the depth direction of the test sample For the transmitted photothermal configuration see Formula (4):

$$\Delta\varphi(x,y)=\frac{2\pi}{\lambda}\cdot\frac{\mathrm{d}n}{\mathrm{d}T}\int T(x,y,z)\mathrm{d}z \tag{4}$$

where dn/dT is the temperature coefficient of refractive index of the test sample.

The integration is performed along the propagation path of the probe beam in the sample. In both cases the temperature rise $T(x, y, z)$ is linearly proportional to the absorptance A of the test sample and the power of the pump laser P. For TL detection, the TL amplitude can be expressed as given by Formula (5):

$$S=\Delta I / I_0 \propto A\cdot P \tag{5}$$

where

ΔI is the intensity change of the probe beam detected with the pinhole photo-detector, and

I_0 is the dc intensity of the probe beam detected with the pinhole photo-detector.

For PTD detection, the PTD amplitude can be expressed as given by Formula (6):

$$S=(I_1-I_2)/(I_1+I_2)\propto A\cdot P \tag{6}$$

where I_1 and I_2 are the intensity values of the probe beam detected by the two photo-detectors of the bi-cell detector (position-sensitive photo-detector).

NOTE For more information on the mathematical description of TL and PTD amplitudes, see Reference [6].

7.2 Determination of absorptance via photothermal calibration

Absorptance of test sample can be determined by calibrating the photothermal amplitude with a calibration sample of "known" absorptance. For the calibration sample with absorptance A_0 and measured with pump laser power P_0, the photothermal amplitude is S_0 as given by Formula (7):

$$S_0=C\cdot A_0\cdot P_0 \tag{7}$$

where C is a proportional factor which is determined by the thermo-physical parameters of the calibration sample and experimental parameters of the photothermal detection configuration.

For the test sample with absorptance A (and with the same thermo-physical parameters of the calibration sample) and measured with pump laser power P under the same experimental photothermal configuration, the photothermal amplitude is S, as given by Formula (8):

$$S=C\cdot A\cdot P \tag{8}$$

The absorptance, A, of the test sample is therefore given by Formula (9):

$$A=(S/S_0)\cdot(P_0/P)\cdot A_0 \tag{9}$$

To minimize the calibration error, it is recommended that the pump power is adjusted to measure the photothermal amplitude of the calibration sample so that S_0 and S are comparable. However, the calibration sample can only present linear absorption, without any nonlinear absorption. This can be checked by measuring the pump power dependence of the photothermal amplitude of the calibration sample.

The calibration can also be performed with a set of "known absorptance" calibration samples (a minimum of three samples with a wide range of absorptance) with the same thermo-physical

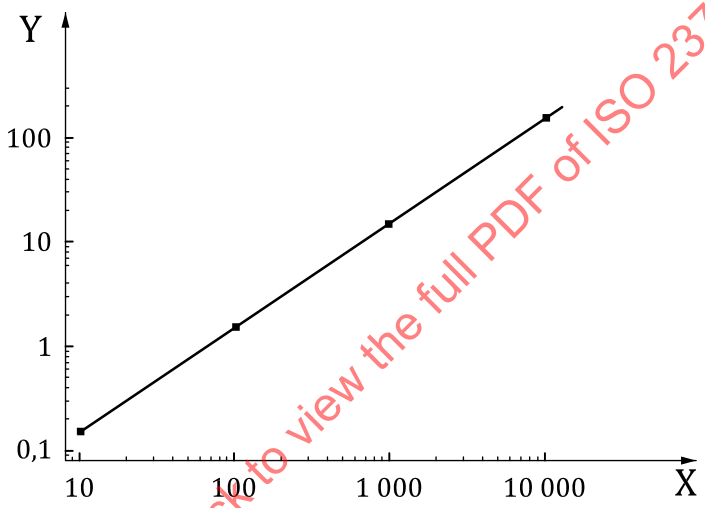
properties. The photothermal amplitude normalized by pump power (S_0/P_0) is measured as a function of the absorptance A_0, as presented in Figure 7. A linear fit is performed to the measured absorptance dependence of the power-normalized photothermal amplitude. From the linear fit, a slope β is obtained, that is, $(S_0/P_0) = \beta \cdot A_0$. Then the absorptance of the test sample can be obtained via Formula (10):

$$A = (S / P) / \beta \tag{10}$$

The calibration with a set of calibration samples is preferable whenever a set of "known absorptance" samples with a wide range of absorptance is available.

For the absorptance determination of the AR coatings of AR-coated samples, a separation of surface and bulk absorption has to be performed by line-scanning the sample along the depth direction. The absorptance of the AR coating is determined by calibrating the separated surface absorption.

Refer to Annex B for the separation of surface and bulk separation .



Key

X	A_0 in ppm
Y	S_0/P_0 (1/W)
▪	measurements
———	linear fit

NOTE The symbols represent measurements with calibration samples of different absorptance values.

Figure 7 — Dependence of photothermal amplitude normalized by pump power on the absorptance of the calibration sample

7.3 Two-dimensional/three-dimensional maps of absorption

A two-dimensional/three-dimensional absorption/absorptance map can be obtained by scanning the test sample two-dimensionally/three-dimensionally and measuring the photothermal amplitude as a function of sample position.

The three-dimensional map can only be obtained for bulk samples. In this case, a large interaction angle between the pump and probe beams is needed for a reasonable spatial resolution in the depth direction of the test sample. It is recommended that the interaction angle between the two beams is at least 30°.

7.4 Mapping area and spatial resolution

For photothermal absorption/absorptance mapping of an optical laser component, the mapping area has to be determined first. The scan step is determined by taking into consideration the acceptable

test time and spatial resolution requirements. The spatial resolution of the absorption mapping is determined by the pump beam size on the test sample, the scan step and the thermal diffusion length. The mapping area is under-sampled if the scan step is larger than the pump beam diameter and is over-sampled if the scan step is smaller than the pump beam diameter. When it is under-sampled, the spatial resolution is determined by the scan step. When it is over-sampled, the spatial resolution is determined by the mutual influence of pump beam diameter, the scan step and the thermal diffusion length. A smaller scan step results in a better spatial resolution of photothermal absorption mapping.

8 Test report

The following information shall be documented in the test report:

a) **Information on the test organization:**

   1) testing organization;

   2) date of test, time;

   3) examiner;

b) **Information concerning the test sample:**

   1) manufacturer of test sample;

   2) type of test sample;

   3) part ID, date of production;

   4) specifications of manufacturer for storage and cleaning;

   5) specifications of manufacturer for normal use (wavelength, polarization, angle of incidence, purpose of use, etc.);

c) **Information concerning the calibration sample(s):**

   1) manufacturer of calibration sample(s);

   2) type of calibration sample(s);

   3) structural parameters (shape, size, etc.) of calibration sample(s);

   4) part ID, date of production;

   5) specifications of manufacturer for storage and cleaning;

   6) absorptance at test pump wavelength and absorptance measurement method.

d) **Information concerning the test facility:**

   1) pump laser source (operation mode, wavelength, output power, power stability, state of polarization, modulation frequency);

   2) probe beam source (wavelength, beam quality, state of polarization)

   3) photothermal measurement configuration (reflected TL, transmitted TL, reflected PTD, or transmitted PTD);

   4) description of other relevant test equipment;

5) environmental conditions (temperature, degree of cleanliness when clean room is used, relative humidity, vibration control, etc.);

e) **Information concerning testing and evaluation:**

1) photothermal amplitude of calibration sample and used pump power;

2) photothermal amplitude of test sample and used pump power;

3) radius or diameter of pump beam on the sample surface;

4) mapping area and scan step for photothermal absorption mapping;

5) acquisition time;

f) **Test results:**

1) absorptance of test sample;

2) absorptance uncertainty of test sample;

3) graphs of photothermal absorption maps (two-dimensional or three-dimensional)

g) **A reference to this document, i.e. ISO 23701:** —:

An exemplary test report format is given in Annex C.

Annex A
(informative)

Theoretical and practical considerations on calibration

A.1 General

This annex describes the theoretical and practical considerations on the calibration issue.

A.2 Theoretical consideration on calibration

Theoretically, for reflected photothermal detection schemes, either STL or reflected PTD, the photothermal amplitude is related to the surface displacement caused by absorption induced thermal expansion, which is proportional to the integral of the temperature gradient along the depth direction of the sample. When the pump beam irradiates the sample at normal incidence, this surface displacement is approximately proportional to the total absorptance of the sample (that is, sum of coating absorptance and substrate absorptance). In this case, a "universal" calibration sample of known absorptance can be used to calibrate the photothermal amplitude of uncoated, HR-coated, and AR-coated samples if the substrates of these samples are the same. On other hand, in the case that the pump beam does not irradiate the sample at normal incidence, the photothermal amplitude becomes sensitive to the temperature profile along the depth direction, which is closely related to the angle of incidence of the pump beam and the absorption profile along the depth direction. The contributions of the coating absorption and substrate absorption to the photothermal amplitude are different. In this case separate calibration samples of known absorptance should be used to calibrate the photothermal amplitudes of uncoated, HR-coated, and AR-coated samples, respectively.

Similarly, for transmitted photothermal detection schemes, either TTL or transmitted PTD, the photothermal amplitude is related to the optical phase shift caused by the absorption induced refractive index change, which is proportional to the integral of the refractive index gradient along the propagation path of the probe beam inside the sample. When both pump beam and probe beam irradiate the sample at normal incidences, this optical phase shift is approximately proportional to the total absorptance of the sample (that is, sum of coating absorptance and substrate absorptance). In this case, again a "universal" calibration sample of known absorptance can be used to calibrate the photothermal amplitude of uncoated, HR-coated, and AR-coated samples if the substrates of these samples are the same. On the other hand, in the case that either the pump beam, or the probe beam, or both beams do not irradiate the sample at normal incidences, the contributions of the coating absorption and substrate absorption to the photothermal amplitude are different. In this case separate calibration samples of known absorptance should be used to calibrate the photothermal amplitudes of uncoated, HR-coated, and AR-coated samples, respectively.

The above discussions indicate that only when the pump beam irradiates the sample at normal incidence in reflected detection schemes, and when both pump beam and probe beam irradiate the sample at normal incidence in transmitted detection schemes, a "universal" calibration sample, no matter uncoated or coated, exists for calibrating the photothermal amplitudes of uncoated, HR-coated, and AR-coated samples with same substrates to determine the absorptance of the test samples. Practically, however, in real TL and PTD detection schemes, both pump beam and probe beam irradiate the samples at certain angles of incidence. The calibration samples of the same functions of the test samples have to be used for calibrating the photothermal amplitudes of uncoated, HR-coated, and AR-coated samples.

A.3 Practical consideration on calibration samples

Practically, the following are examples of calibration samples:

a) ND filters with absorptances in the order of 0,01 to 0,1, measured by a spectrophotometer;

b) Uncoated substrates with absorptances in the order of 10^{-5} to 10^{-4}, measured by a laser calorimeter;

c) Protected metal mirrors with absorptances in the order of 0,01, measured by a spectrophotometer;

d) Dielectric HR mirrors with absorptances in the order of several to several tens 10^{-6}, measured by a laser calorimeter;

e) One-side dielectric AR-coated substrates with total absorptances in the order of 10^{-5} to 0,001, and with absorptance of substrate measured in advance. The absorptances are measured by a laser calorimeter.

It is recommended that whenever possible, the preparation of the calibration samples with the substrate materials be the same as that of the test samples. It is also important that the calibration samples and test samples have the same functions. That is, HR calibration samples with same substrate materials for HR test samples, AR calibration samples with same substrate materials for AR test samples, and uncoated substrate calibration samples for substrates with same material but different absorptance. Do not use calibration samples with different substrate materials and different functions to calibrate the photothermal amplitude for absorptance determination. In general, it is not necessary that the coating materials are the same for calibration and test samples.

NOTE The calibration is highly dependent on the properties and design of the sample.

Annex B
(informative)

Separation of surface absorption and bulk absorption

B.1 General

This annex describes the photothermal configuration to separate the surface (coating) absorption and bulk (substrate) absorption of AR-coated optical laser component and considerations regarding the measurement results.

B.2 Photothermal detection configurations for separation of surface absorption and bulk absorption

The transmitted photothermal detection schemes, either transmitted TL or transmitted PTD, should be used to separate the surface absorption and bulk absorption. As an example, an optical configuration of transmitted TL detection for the separation is re-shown in Figure B.1. In this transmitted TL configuration, the pump beam irradiates the sample at nearly normal incidence, and the probe beam irradiates the sample at a relatively large angle of incidence. It is preferable to have a large interaction angle between the pump and the probe beams for a higher spatial resolution in the depth direction, which is needed when the substrate is thin. For separation of the surface absorption and bulk absorption, a line scan along the depth direction is performed by moving the interaction zone of the pump and probe beams across the sample and recording the TL amplitude as a function of the depth. Figure B.2 shows a typical line scan result of a one-side AR-coated fused silica sample. The TL amplitude is a vector sum of the TL signals from the coating and substrate, respectively. When the interaction zone of pump and probe beams is within the substrate but far from the coated surface, the TL signal is attributed to the substrate absorption only. In this case the TL amplitude, when calibrated, can be used to determine the absorption coefficient of the substrate (the bulk absorption). The maximum TL amplitude peak, which appears when the interaction zone of the pump and probe beams is close to the coated surface of the substrate, can be used to determine the absorptance of the coating. However, care has to be taken when determining the coating absorptance as the substrate absorption also contributes to the maximum TL amplitude peak, and its contribution shall be eliminated.