

T.R
GEBZE TECHNICAL UNIVERSITY
GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

**ABSORPTION COEFFICIENT DETERMINATION OF HIGH-
QUALITY OPTICS USING LASER CALORIMETRY**

CAN GÖZÖNÜNDE
A THESIS SUBMITTED FOR THE DEGREE OF
MASTER OF SCIENCE
DEPARTMENT OF PHYSICS

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**T.C.
GEBZE TEKNİK ÜNİVERSİTESİ
FEN BİLİMLERİ ENSTİTÜSÜ**

**LAZER KALORİMETRİ KULLANARAK
YÜKSEK KALİTELİ OPTİKLERİN SOĞRULMA
KATSAYISININ BELİRLENMESİ**

**CAN GÖZÖNÜNDE
YÜKSEK LİSANS TEZİ
FİZİK ANABİLİM DALI**

**DANIŞMANI
PROF. DR. FEVZİ NECATİ ECEVİT**

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SUMMARY

Precise determination of the absorption coefficient of optical elements plays a vital role in the design of integrated systems which involves a huge number of high-quality optical components. For this reason, accurate measurements of the optical absorption coefficient down to sub-ppm level by employing a low power laser have a great importance in the production of the high-quality optical components. Currently these measurements are performed according to laser calorimetric methods, regulated by ISO 11551 series standards. However, measurements of ultra-low optical absorption coefficients with interferometric methods especially photo-thermal common-path interferometry have become prominent in the last years. Nevertheless, due to the indirect nature of these methods, reference materials are still required for the absolute calibration.

In this study, a new laser calorimeter which can perform the measurements in both vacuum and atmospheric environments was designed, constructed, and employed. The absorption coefficient of two reference materials, namely high-grade Fused Silica and BK-7[®] glasses were assessed at different laser output powers. The experimental setup includes 532 nm, 640 nm, 1070 nm single mode lasers, thermally isolated chamber, vacuum chamber, power meter, and NTC sensors, where the sample geometry and thermometer positions can be varied by demand.

The measurement results were analyzed by applying different non-linear fitting methods and compared. Additionally, a finite-element method-based simulation was applied for the calculations of the thermal performance of fused silica and BK-7[®] bulk materials, using the same experimental parameters. The obtained results are presented and discussed in detail.

Key Words: Absorption coefficient, Laser calorimetry, Optical absorption, ISO 11551.

ÖZET

Optik elemanların soğurma katsayısının hassas bir şekilde belirlenmesi, çok sayıda yüksek kaliteli optik bileşen içeren entegre sistemlerin tasarımında hayati bir rol oynamaktadır. Bu nedenle, düşük güçlü bir lazer kullanılarak optik soğurma katsayısının ppm altı seviyeye kadar doğru ölçümlerinin sağlanması, yüksek kaliteli optik bileşenlerin üretiminde büyük öneme sahiptir. Şu anda bu ölçümler ISO 11551 serisi standartlar tarafından düzenlenen lazer kalorimetrik yöntemlere göre yapılmaktadır. Ancak son yıllarda ultra düşük optik soğurma katsayılarının diğer yöntemlerle, özellikle de fototermal ortak yol interferometrisi tabanlı yöntemlerle ölçümü öne çıkmaktadır. Bununla birlikte, bu yöntemlerin dolaylı doğası nedeniyle, mutlak kalibrasyon için hala referans malzemeler gerekmektedir.

Bu çalışmada, ölçümleri hem vakum hem de atmosfer ortamında gerçekleştirebilen yeni bir lazer kalorimetre tasarlanmış, kurulmuş ve kullanılmıştır. Yüksek dereceli kaynaştırılmış silika ve BK-7[®] camları olmak üzere iki referans malzemenin soğurma katsayısı farklı lazer çıkış güçlerinde değerlendirilmiştir. Deney düzeneği 532 nm, 640 nm, 1070 nm tek modlu lazerler, termal olarak izole edilmiş kabin, vakum kabini, güç ölçer ve örnek geometrisinin ve termometre konumlarının isteğe göre değiştirilebildiği NTC sensörlerini içermektedir.

Ölçüm sonuçları, farklı doğrusal olmayan fit yöntemleri uygulanarak analiz edilmiş ve ardından karşılaştırılmıştır. Ayrıca, aynı deneysel parametreler kullanılarak kaynaştırılmış silika ve BK-7[®] gövde malzemelerinin termal performansının hesaplanması için sonlu elemanlar yöntemine dayalı bir simülasyon uygulanmıştır. Elde edilen sonuçlar ayrıntılı olarak aktarılmış ve tartışılmıştır.

Anahtar Kelimeler: Soğurma katsayısı, Lazer kalorimetri, Optik soğurma, ISO 11551.

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LIST OF ABBREVIATIONS AND ACRONYMS

Abbreviations : **Explanations**

and Acronyms

S	: Scattering
T	: Transmittance
A	: Absorbance
R	: Reflectance
I_0	: Initial intensity of light
I	: Final intensity of light
μ	: Optical density
d	: Thickness of the material
R	: Radius
T_e	: Environment temperature
T_s	: Sample temperature
T_c	: Chamber temperature
t_0	: Irradiation start
t_1	: Irradiation End
H	: Density
c_p	: Specific heat under constant pressure
K	: Thermal Conductivity
H	: Surface heat loss coefficient
K	: Thermal diffusivity
Γ	: Temperature loss coefficient
Q	: Heat source
$\theta(t)$	: Time dependent Heaviside function
M	: Mass
T_e	: Environment Temperature
t_p	: Half irradiation time
P	: Laser power
C_{eff}	: Effective heat capacity
f_c	: Correction factor

ΔT	: Temperature rise
ΔT_{el}	: Temperature rise caused by electrical heating
V_h	: Heating voltage
I_h	: Heating current
CRDS	: Cavity Ring-down Spectroscopy
DAQ	: Data Acquisition
GTÜ	: Gebze Teknik Üniversitesi
ISO	: International Organization for Standardization
LC	: Laser Calorimetry
LC1	: Laser Calorimetry setup depicted at Figure 3.1
LC2	: Laser Calorimetry setup depicted at Figure 3.4
LED	: Light-emitting Diode
NIR	: Near-infrared
NTC	: Negative Temperature Coefficient
OH	: Hydroxide
PAS	: Photoacoustic Spectroscopy
PC	: Personal Computer
PCI	: Photo-thermal Common-path Interferometry
PID	: Proportional Integral Derivative
PZT	: Piezoelectric Transducer
STL	: Surface Thermal Lensing
TÜBİTAK	: Türkiye Bilimsel ve Teknolojik Araştırma Kurumu
UME	: Ulusal Metroloji Enstitüsü
UV	: Ultraviolet

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1. INTRODUCTION

The foundation of the laser was laid in 1916 by Albert Einstein, who argued that an excited atom could be re-excited by a photon to produce an identical photon with the same phase, direction, polarization and wavelength [1].

Despite lasers' experimental history is little over half a century, utilization of laser is increasing day by day in every field from toy industry to quantum mechanical experiments. In recent years, interest in high-power lasers has increased rapidly due to their wide use in various industries and defense. This growing interest in high power lasers has led to the need for the production of high-quality optical components that can be utilized with such lasers. The main reason for this is that the lack of power compatibility of these components can lead to fatal consequences in the system.

In order to obtain a stable high power laser system, the characterization of the optical components in this system is indispensable. Method for the measurement of scattering, reflection, transmission, and degree of absorption values of these high-quality optical components must be measured very precisely to overcome this issue. Some of the methods which are employed to measure these characteristics and corresponding ISO standards are listed in Table 1.1 [2]-[7].

Table 1.1: List of the ISO standards about reflectance, transmittance, absorptance and scattering

Characteristic	Method	ISO Standard
Reflectance and Transmittance	Spectrophotometry	ISO 15368
	Precise Ratiometric Method	ISO 13697
	Cavity Ring Down	ISO 13142
Absorption	Laser Calorimetry	ISO 11551
Scattering	Total Scattered Radiation	ISO 13696
	Integrated Scattering	ISO 19962

When electromagnetic radiation interacts with a material it transfers its energy in lots of ways which causes changes in the properties of the material such as temperature, density, pressure, refractive index, transparency, and thermal expansion. These changes allow us to indirectly measure material characteristics which are scattering, absorption, reflection, and transmission of the material. Of these four fundamental properties, the degree of absorption of the optical component is often

considered the most essential element when characterizing a laser system. To determine the degree of absorption of light per unit length, absorption coefficient parameter is used.

1.1. Purpose, Contribution and Content of the Thesis

Absorption coefficient is widely used parameter in high-power laser optics, photonics, optoelectronics, and solar energy conversion. Accurate and reliable measurement of the absorption coefficient plays a vital role for the design, characterization and optimization of the devices which uses the absorption coefficient or endeavors to avoid absorption losses.

Traditionally, optical absorption is calculated by using methods like transmission, reflection and scattering spectroscopy. The absorption can then be derived from the requirement of energy conservation using equation 1.1.

$$1 = S + T + A + R \quad (1.1)$$

Here S, T, A, and R represent scattering, transmittance, absorbance, and reflectance phenomena respectively. However, the precision and accuracy of these methods do not meet the requirements of modern technology standards. The continuous development in optical components has created a demand for optical characterization methods such as absorption coefficient measurement. For many years, various methods have been developed to ensure the measurement of even lowest absorptions. The measurement of absorption coefficients using laser calorimetry has become a well-established technology that has been developed and redefined over time in the 1970s [8]. Other modern methods for measuring absorption coefficients including photoacoustic spectroscopy, cavity ring down spectroscopy, photo-thermal interferometric methods are more sensitive than laser calorimetry. Nevertheless, the reference method used by the ISO 11551 series standards, laser calorimetry, offers a potent, cost-effective, and versatile approach for precise and accurate measurement of optical absorption down to ppm/cm levels in optical components [8].

The objective of this thesis is to design, build, and use a novel laser calorimeter that can perform measurements in both vacuum and atmospheric environments. At

various laser output levels, the absorption coefficients of reference materials, high-grade Fused Silica and BK-7[®] glasses, were measured in both atmospheric and vacuum conditions. The compliance of the experimental results obtained under high vacuum ($\sim 1 \times 10^{-6}$ mbar) settings with the ISO 11551 series standards were studied. Furthermore, the experimental absorption coefficient data were compared with the results produced by using the finite element approach using the COMSOL Multiphysics[®] tool and numerical simulation with MATLAB[®]. The proposed technology will be used for both absolute calibration of interferometry-based systems and measurement of the relevant parameters of space-qualified optics. The experimental setup contains three separate single mode lasers with wavelengths of 532 nm, 640 nm, and 1070 nm which can operate sequentially with mirror adjustment, a thermally isolated chamber, vacuum chamber, power meter, and NTC sensors with variable sample shape and thermometer sites. Different mathematical fit tools were used in line with the measurement results throughout the data processing stage, and the results were compared with reference materials with known absorption coefficients.

The underlying physics of the absorption mechanism, brief summary of the contemporary approaches for measuring the absorption coefficient, laser calorimetry, experimental phases, and findings will all be covered in this thesis.

2. ABSORPTION

Absorption is one of the fundamental processes in light-matter interaction. It has been studied since the early 18th century [9]. In optics, absorption is defined as the transfer of the energy from electromagnetic field of the light to the medium resulting in the attenuation of the light. A significant phenomenon called absorption can take place in a material by a variety of processes, including electronic, vibrational, or rotational transitions [10]. This phenomenon has a substantial role in a wide range of technological fields such as spectroscopy, material science and high-quality optics. The amount of light absorbed by the material is determined by a number of parameters. These parameters include wavelength of the light, thickness, optical qualities, and composition of the material.

For the characterization of the absorption of a material, absorption coefficient is an essential parameter. Absorption coefficient quantifies how much of the incident radiation is absorbed while passing through the material. The absorption coefficient is described by fractional attenuation of the incident light per unit distance traveled through the material. It is typically expressed in unit of cm^{-1} .

Johann Heinrich Lambert described the absorption coefficient in the mid-18th century mathematically (theoretically). Lambert's study provides the link between incoming, transmitted light intensity, and absorbing medium thickness [11]. Thereafter, in the mid-nineteenth century, August Beer improved Lambert's study by connecting the absorption coefficient to the concentration of absorbing species in liquids [12]. This development resulted in the establishment of the Beer-Lambert Law, which is frequently used to determine light absorption in spectroscopy and related fields. According to Beer-Lambert law absorption coefficient is related to material's optical density, which is a measure of how much of the light is absorbed by the material at a particular wavelength. Beer-Lambert law can be expressed by the equation 2.1.

$$I = I_0 e^{-\mu d} \quad (2.1)$$

According to this equation, the amount of light that passes through a substance depends on its thickness, absorption coefficient, and incident light intensity. However, certain presumptions must be made for this method to produce an accurate absorption

coefficient. Examples include assuming a linear relationship between path length and absorption as well as the absence of scatter or other complicated mechanisms. To overcome these limitations, more accurate absorption coefficient measurement systems are developed. For example, more precise and accurate assessments can be made by using photoacoustic spectroscopy, surface thermal lensing, PCI, and laser calorimetry.

Depending on the incident wavelength of the light absorption coefficient can change significantly. Some material may have strong absorption lines in the UV regions while other materials may have strong absorption lines in the visible regions. These absorption lines may have correlation with the temperature humidity or environmental factors. Numerous scientific advances have been made thanks to our growing understanding of material absorption mechanisms. The exploration of material absorption mechanisms has led to significant advancements across various scientific fields. Numerous breakthroughs have been achieved, resulting in notable applications, such as lithography, energy conversion in photovoltaic devices, heat conversion in thermoelectric devices, and preservation of human health, among others.

2.1. Absorption Mechanisms

Absorption phenomena can be classified into two main categories: linear absorption and non-linear absorption. Linear absorption is characterized by its independence from the light intensity. In contrast, non-linear absorption exhibits a relationship with the light intensity that can be either linear or expressed through higher-order functions (e.g. two-photon, multiphoton absorption).

Absorption mechanism in materials can vary with the incident wavelength of the light and purity of the material. Absorption mechanisms can be classified into two categories: intrinsic and extrinsic absorption. Intrinsic absorption arises from fundamental properties inherent to the material, such as electronic transitions and phonon coupling. On the other hand, extrinsic absorption is attributed to the presence of impurities, encompassing factors like OH groups, lattice defects, metallic nanoparticles, and rare-earth elements, among others [13].

For example, most of the SiO₂-based glasses has strong absorption in the UV region this is because energy required for the electronic transition of SiO₂-based

glasses are around ~ 9 eV (138 nm) [14]. When an electron is excited from the valence band to the conduction band, resulting in the formation of an electron-hole pair. Subsequently, the electron and hole can bind together, forming a stable bound state known as an exciton. In addition to the exciton absorption, fused silica contains a small number of impurities which causes additional absorption bands according to the impurity type. OH groups, intrinsic defects, transition metal complexes, alkali ions are some of the most common impurity types which are mainly located around UV and visible region. Although most of the impurities located in the UV and visible spectrum, infrared absorption which is mostly caused by hydroxyl groups, in the near and mid infrared region is still contributing to the absorption coefficient (e.g. multi-phonon absorption around $2\mu\text{m}$ [15]).

Infrared absorption can be divided in two categories namely, vibrational, and rotational absorption. Solid materials contain molecular vibrational modes, where the atoms of the material vibrate at allowed frequencies. These vibrational modes are categorized as the stretching, bending, or twisting of chemical bonds in the material. When light interacts with the material, it can be absorbed by exciting vibrational modes. Absorption occurs when the energy of the incident light has enough energy to excite the atoms to higher vibrational energy levels.

Vibrational absorptions tend to exhibit increased intensity within the far infrared region. Nevertheless, NIR absorption holds significance in solid materials due to its non-destructive nature. Conversely, rotational absorption in solid materials remains negligible, owing to the narrower band structure characterizing solids [16], [17]. However, in certain cases, impurities or dopants in the solid materials may cause rotational absorption.

It's important to note that the extent and specific frequencies of vibrational, and rotational absorption in glasses depend on factors such as the purity of the material, dopants or impurities present, and the specific energy levels involved. The absorption characteristics can be influenced by factors such as temperature, pressure, and the presence of other molecules or impurities.

Overall, the absorption of light in glasses involves a combination of electronic transitional, vibrational, and rotational processes, with vibrational absorption being the most significant in the mid-infrared and near-infrared regions.

2.2. Thermodynamic Property-based Techniques for Absorption Detection

The determination of the absorption coefficient is critical for a wide range of applications in materials science and optics. Several methods for measuring the absorption coefficient have been devised, each having merits and limitations. Among different analytical techniques, photo-thermal spectroscopic approaches have grown in favor. Their popularity stems from their exceedingly sensitive and non-destructive character. Photo-thermal spectroscopy causes changes in numerous critical thermodynamic parameters of the material, including temperature, pressure, and refractive index, by utilizing photon absorption [18]. These changes provide important information on the absorption behavior and properties of the sample under investigation. Photo-thermal spectroscopy is a useful tool for studying the absorption properties of various materials due to its ability to properly monitor these thermodynamic changes. Some of the absorption coefficient determination methods are listed in Table 2.1.

Table 2.1: Absorption coefficient determination methods

Thermodynamic Property	Method
Temperature	Laser Calorimetry
Pressure	Photoacoustic Spectroscopy
Refractive Index	Laser Induced Deflection Photo-thermal common-path Interferometry Surface Thermal Lensing Cavity Ring-down Spectroscopy

The following presents a brief review of some of the above techniques based on refractive index and pressure changes. Figure 2.1 shows the schematic of surface thermal lensing method. Surface thermal lensing represents an innovative approach that combines the sensitivity inherent in photo-thermal deformation techniques with the simplicity of the thermal lensing method. This technique serves as a potent tool for the photo-thermal characterization of optical thin films with weak absorption properties. Owing to the characteristic wherein the probe beam has a larger beam size than the pump beam, this method proves to be optimal for industrial applications. This advantage stems from its ease of alignment and reduced susceptibility to environmental influences [19]. Within the interaction volume, a Gaussian distribution

of temperature is created by the pump beam's absorption. The interaction of thermal diffusion within the medium and heat injection by absorption results in the formation of a thermal lens. The probe beam's trajectory as it travels through the material is then affected by this lens [20]. The transmitted beam's spatial profile variations as a result are then seen in the far field. Notably, the chopped beam is phase-modulated in synchrony with the periodic emergence of the surface lens, which imparts a characteristic diffraction pattern onto the reflected probe beam.

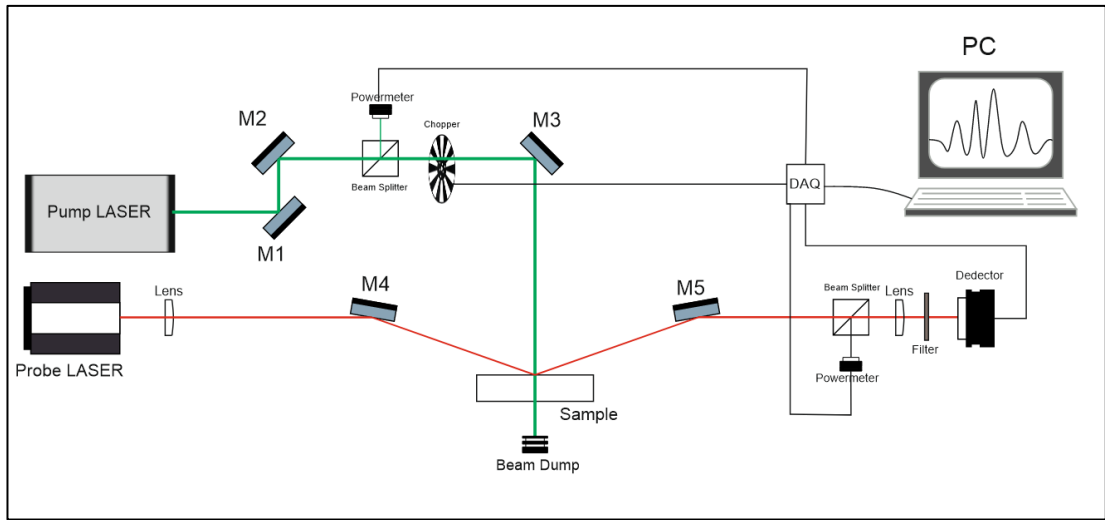


Figure 2.1: The schematic of Surface thermal lensing setup [21]

Figure 2.2 shows the schematic of photo-thermal common-path interferometry. PCI is a technique used for the determination of the absorption coefficient of materials. It combines the principles of photo-thermal spectroscopy and common-path interferometry to achieve highly sensitive and accurate measurements [22].

In PCI, a laser beam called pump laser is directed onto the sample, and the absorbed light energy generates localized heating [23]. A probe beam which has a size that covers the pump beam (concentric), passes through the sample and directed to a photodetector. The locally absorbed pump laser induces changes in the refractive index and optical path length of the material. Due to the locally changed refractive index the center of the probe beam gains a different phase from the rest of the beam. As a result of the phase difference there occurs a distortion at the center of the probe beam. Distortion originates from the interference phenomenon between distorted and undistorted parts of the probe beam. The interference phenomenon contains

information about the absorption characteristics of the sample [24]. By analyzing the interference pattern, the absorption coefficient of the sample can be determined.

The common-path interferometry configuration is advantageous because it eliminates the need for separate reference arms and compensates for environmental disturbances and path-length fluctuations. It provides robust and stable interference signals, enhancing measurement accuracy.

The PCI technique offers several benefits for absorption coefficient determination. Firstly, it is highly sensitive, capable of detecting even small changes in the refractive index induced by absorbed light energy. This enables the measurement of low absorption coefficients. Additionally, it is a non-destructive method that allows for repeated measurements on the same sample, making it suitable for studying dynamic processes or monitoring changes over time.

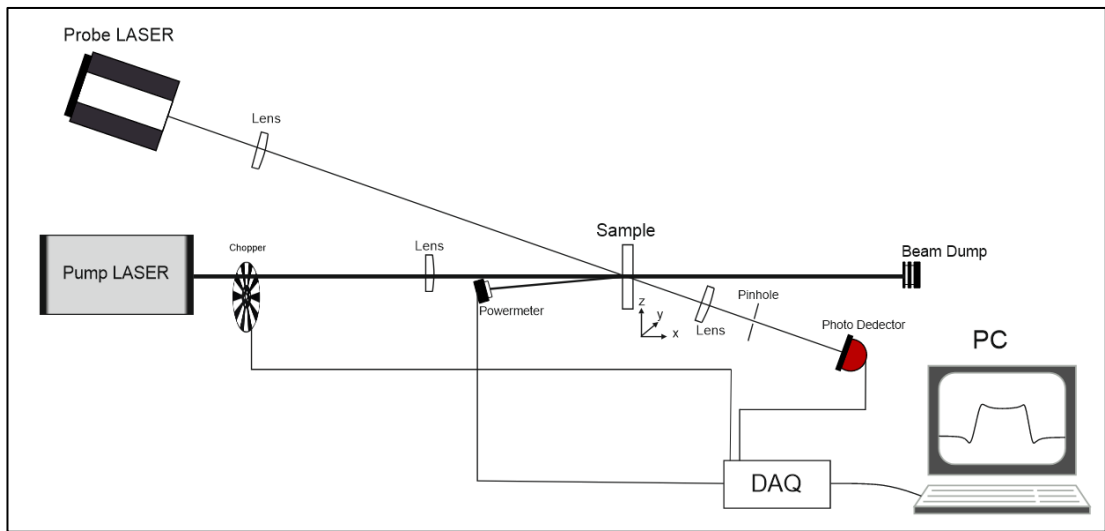


Figure 2.2: The schematic of photo-thermal common-path interferometry system [23]

The PCI system is designed as a linear setup, which means when the power falling on the tested optical component increases, there is a corresponding increase in the photo-thermal signal obtained from that component. Thanks to this feature, the calibration of the system can be performed relatively. In relative calibration, reference materials with known absorption coefficient needed in order to calibrate the system. For measurements involving coated optical components, surface reference materials are employed, whereas uncoated bulk optical components are measured using bulk reference materials.

Figure 2.3 shows the schematic of Photoacoustic spectroscopy. PAS is a substantial method for figuring out a material's optical absorption characteristics. It combines optical and acoustic principles to offer insightful information about the absorption behavior of a variety of materials, such as solids, liquids, and gases.

An acoustic wave is produced when a sample absorbs light energy and then goes through a prompt thermal expansion. By detecting and analyzing this wave one can extract the optical absorption characteristics of the sample.

Typical photoacoustic spectroscopy experimental setup comprises irradiating the sample with modulated light of a specific wavelength or a range of wavelengths. Pulsed or modulated lasers can be utilized as light sources, depending on the desired spectral range and resolution. However pulsed laser PAS is known for efficiency and resistance to environmental change [26].

The absorbed modulated light energy causes localized heating, leading to periodic thermal expansion and contraction. These acoustic waves are generated by thermal cycling that propagate through the surrounding medium, typically air or an inert gas. These acoustic waves can be detected using specialized microphones, such as sensitive piezoelectric transducers [27].

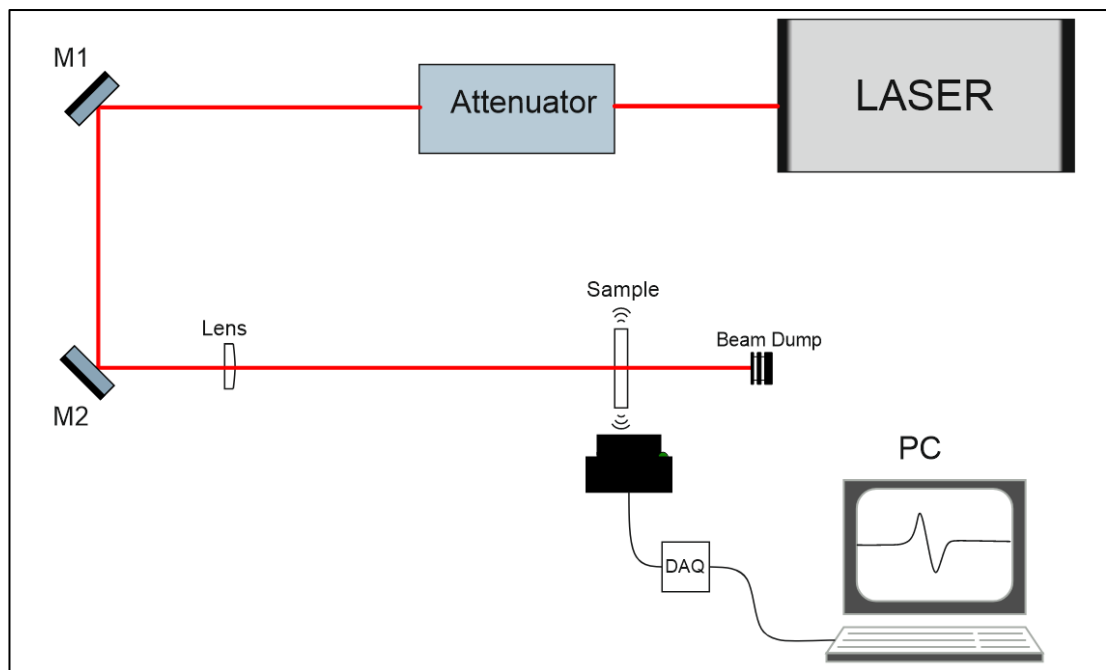


Figure 2.3: The schematic of Photoacoustic Spectroscopy [27]

In order to extract the absorption characteristic of the sample the detected acoustic signal is then demodulated. One can determine the sample's absorption spectrum by adjusting the modulation frequency and examining the photoacoustic signals.

Figure 2.3 shows the schematic of Cavity Ring-down Spectroscopy. CRDS is a sensitive and highly precise technique utilized for the determination of optical absorption and scattering characteristics of samples. It is an effective method for investigating impurity amounts of substances in gas, liquid and recently solid phases [28]-[30].

The fundamental principle of CRDS is based on the measurement of the decay of light intensity within a high-finesse optical cavity. The resonant optical cavity consists of two highly reflective mirrors facing each other. One of the mirrors is partially transmissive, allowing a small portion of the light to escape from the cavity. A pulsed laser is typically used as the light source in CRDS setups, providing light at a single wavelength or a range of wavelengths. The optical cavity receives laser light and is adjusted to resonate at the laser wavelength. A standing wave pattern is produced inside the cavity as a result of the light's many reflections between the mirrors as it enters. When a sample interacts with the light inside the cavity photons experience absorption or scattering phenomena depending on the sample characteristics. This interaction causes an attenuation in the light intensity within the cavity. The decay of the light intensity is measured by monitoring the time it takes for the light to "ring down" or diminish to a certain fraction of its initial intensity [31].

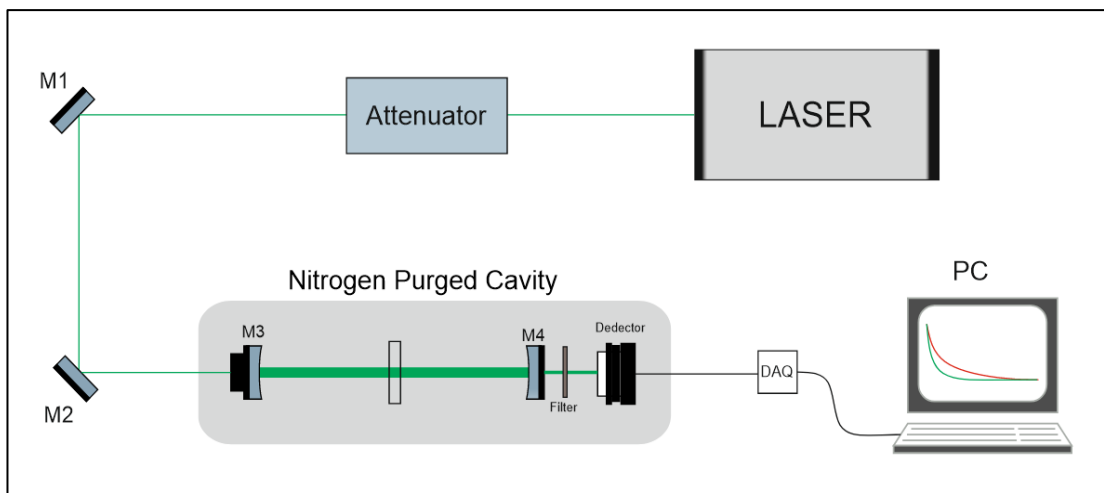


Figure 2.4: The schematic of Cavity Ringdown Spectroscopy [32]

The ringdown time measurement yields valuable data regarding the sample's absorption or scattering characteristics. The optical characteristics of the sample, such as its absorption cross-section or scattering coefficient, can be identified by comparing the ringdown times with and without the sample within the cavity. CRDS is an ideal method for trace analysis applications since it can detect compounds at incredibly low concentrations [29].

2.3. Laser Calorimetry

In the 1990s, a standard testing method for assessing the absorption of optical components was required due to the expanding market demand for lasers and laser systems. To address the requirements of standardization, the aforementioned intricate methods proved to be overly laborious for practical use. As opposed to laser calorimetry, deflection techniques and photoacoustic methods are frequently complimented for their increased sensitivity and superior adaptability for carrying out characterizations with spatial resolution.

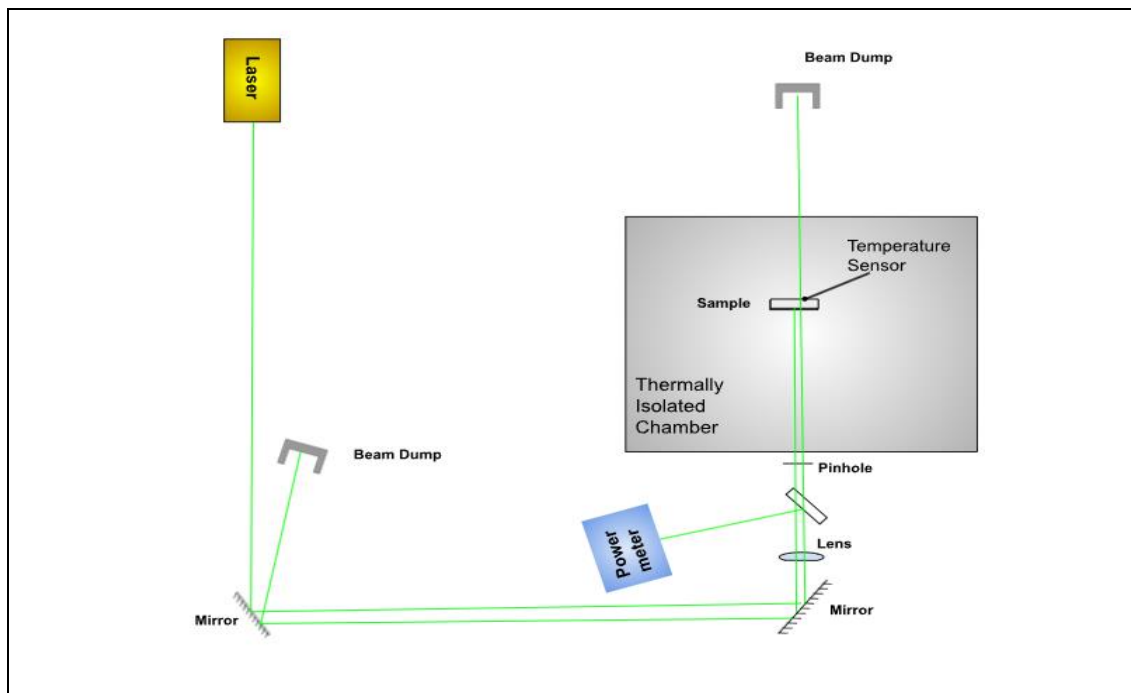


Figure 2.5: The schematic of Laser Calorimetry Measurement System

However, an essential aspect of this standard procedure is the ability to achieve absolute calibration. It is possible to assess the absorbed power accurately and sensitively without the need for elaborate apparatus by monitoring the temperature rise in the heated sample directly [8]. In order to characterize absorption losses in laser optics, the International Organization for Standardization introduced ISO 11551 in 1997, enabling laser optics research, development, and production to be conducted in accordance with this established standard ever since.

When measuring absorption coefficients at the ppm level with laser calorimetry, a thorough illustration of the relatively minimal equipment required as shown in Figure 2.5. This graphic demonstrates how the laser calorimetry approach is streamlined and effective, requiring only a small number of tools and equipment to produce reliable findings. The characterization and analysis of optical components can be done with astounding efficiency and effectiveness by utilizing this technique, which enables researchers and practitioners to perform absorption coefficient measurements with the maximum ease and practicability. Figure 2.5's representation of laser calorimetry is a testament to its economic viability, making it an important tool for laser optics research, development, and production.

The theoretical aspect of this method will be explored through the lens of two distinct categories: the homogeneous method and the inhomogeneous method. This classification offers a comprehensive framework for understanding the underlying principles and complexities of the method.

2.3.1. Inhomogeneous Model

Although the inhomogeneous model offers a more detailed representation of the system, it requires a higher level of computational and mathematical rigor, making it a less straightforward choice compared to the homogeneous method. The following model will be presented to study the temperature dynamics of a continuous-wave Gaussian laser-absorbing sample: The sample under consideration is a cylinder of radius R and thickness d . The sample is radially symmetrically exposed to a laser beam for the time span between t_0 and t_1 . $Q(\rho, z)$ stands for the heat injection per volume element. Additionally, the heat losses per unit area element on the sample's surface,

which include convection and radiation, are proportional to the difference in temperature between the surface and the surrounding air.

For an approximation of the influence of thermal conductivity, the simple case of a cylindrical substrate is considered. Introducing the temperature rise $\vartheta(\rho, z, t) = T_s(\rho, z, t) - T_c(\rho, z, t)$, we can write the heat equation as follows [8]:

$$\nabla(k\nabla\vartheta) + Q(\rho, z) = \eta c_p \dot{\vartheta} \quad (2.2)$$

By using the Newton-Rikhman boundary conditions that describe the interaction with the environment are [33]:

$$k\partial_\rho\vartheta(\rho, z, t) = \begin{cases} -h\vartheta & \rho = R \\ 0 & \rho = 0 \end{cases}, \quad k\partial_z\vartheta(\rho, z, t) = \begin{cases} -h\vartheta & z = d \\ h\vartheta & z = 0 \end{cases} \quad (2.3)$$

Since the specimen's temperature is low, the radiative heat losses are relatively smaller than the convective losses, making them negligible [34]. Without any heat source $Q(\rho, z)=0$ equation can be solved by:

$$\left(\partial_\rho^2 + \frac{1}{\rho} \partial_\rho + \partial_z^2 - \frac{\partial_t}{\kappa} \right) \vartheta = 0 \quad (2.4)$$

In order to solve the equation separation of variables is used

$$\vartheta(\rho, z, t) = f(\rho) * g(z) * h(t) \quad (2.5)$$

The solution is then:

$$0 = \frac{f''(\rho) + f'(\rho)/\rho}{f(\rho)} + \frac{g''(z)}{g(z)} - \frac{h'(t)}{\kappa h(t)} \quad (2.6)$$

$$\begin{aligned} -\varepsilon^2 &= \frac{f''(\rho) + f'(\rho)/\rho}{f(\rho)} \\ -\lambda^2 &= \frac{g''(z)}{g(z)} \end{aligned} \quad (2.7)$$

$$\varepsilon^2 + \lambda^2 = - \frac{h'(t)}{\kappa h(t)}$$

Radial temperature dependence can be solved with linear combination of Bessel J_0 and the Weber function Y_0 [35].

$$T(r) = A * J_0(c * r) + B * Y_0(c * r) \quad (2.8)$$

These mathematical functions, known as zeroth-order Bessel and Weber functions, have applications in scenarios requiring circular symmetry, wave propagation, heat conduction, and a variety of other areas of mathematics and physics. Where A and B are usually constant and can be determined from initial conditions. The equation used to describe the temperature distribution in problems with cylindrical symmetry is given below.

$$f(\rho) = FJ_0(\varepsilon\rho) + F'Y_0(\varepsilon\rho) \quad (2.9)$$

Since the F' term is zero at $\rho=0$, the term $J_0(\varepsilon\rho)$ alone is sufficient in some cases. Boundary condition for dependency in z direction can be written as [35]:

$$g(z) = G \sin(\lambda z) + G' \cos(\lambda z) \quad (2.10)$$

at $z=0$ boundary condition: $G' = G \frac{k\lambda}{h}$

For time dependency $h(t)$ can be re written from equation 2.7.

$$h(t) \propto e^{-\kappa(\varepsilon^2 + \lambda^2)t} \quad (2.11)$$

In the absence of heating $\left(\lim_{t \rightarrow \infty} h(t)\right) = 0$ the boundary condition that the difference between sample and ambient temperature should decrease. This leads to the limitation of the constants $\varepsilon^2 + \lambda^2 > 0$ $\varepsilon, \lambda \in R$.

This boundary condition is a commonly used boundary condition in thermal conduction problems. This condition refers to a scenario where the temperature difference decreases with time in the absence of heating. This boundary condition can be expressed as:

$$T_s(t) - T_e(t) > 0 \quad (2.12)$$

This boundary condition describes situations where the sample interacts thermally with its environment and reaches thermal equilibrium over time [36]. That is, when there is a temperature difference between the sample and the medium initially, this difference decreases over time. This is the case where energy is dispersed into the environment by reducing the temperature difference in the sample through heat transfer.

According to the formula 2.9, 2.10, 2.11, the temperature rise equation 2.5 can be rewritten as:

$$\vartheta(\rho, z, t) = \sum_{i,j} F_i J_0(\varepsilon_i \rho) G_j \left(\sin(\lambda_j z) \frac{k\lambda_j}{h} + \cos(\lambda_j z) \right) e^{-\kappa(\varepsilon_i^2 + \lambda_j^2)t} \quad (2.13)$$

The discrete coefficients ε_i and λ_j result from the boundary conditions for $z = d$ and $\rho = R$. Which can be found by solving $f(\rho)$ and $g(z)$.

$$\begin{aligned} \varepsilon_i &= x_i/R \quad , \quad 0 = \frac{hR}{k} J_0(x_i) - x_i J_1(x_i) \\ \lambda_j &= x_j/d \quad , \quad 0 = 2 \cos x_j + \left(\frac{hd}{kx_j} - \frac{kx_j}{hd} \right) \sin x_j \end{aligned} \quad (2.14)$$

Figure 2.6 shows first two roots of the discrete coefficients ε_i and λ_j .

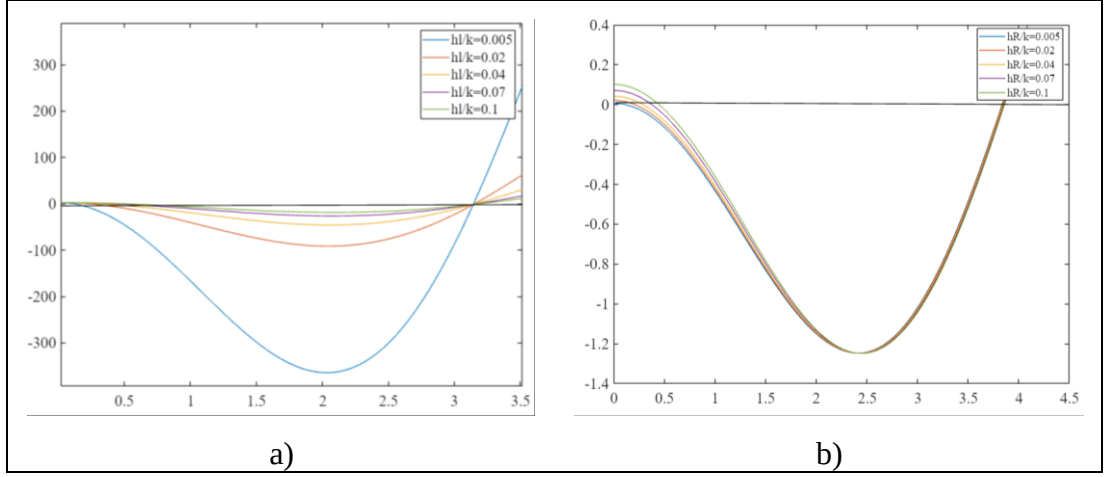


Figure 2.6: The discrete coefficients a) ε_i and b) λ_j

To streamline subsequent calculations, a practical estimation for the cooling coefficients γ can be obtained based on the aforementioned considerations.

$$\gamma_{i,j} = \kappa(\varepsilon_i^2 + \lambda_j^2)$$

$$\gamma_{0,0} \approx \frac{2h}{\eta c_p} \left(\frac{1}{R} + \frac{1}{d} \right), \quad \gamma_{i>0,j>0} \approx \frac{k\pi^2}{\eta c_p} \left(\frac{(i + 1/4)^2}{R^2} + \frac{j^2}{d^2} \right) \quad (2.15)$$

As the Bessel functions $J_0(\varepsilon_i \rho)$ build a complete set of orthogonal functions in $[0, R]$ or $[0, d]$, the heat-source term can be developed [36]:

$$q_{i,j} = \frac{1}{N_{i,j}} \int_0^R \int_0^d dz d\rho \rho Q(\rho, z) J_0(\varepsilon_i \rho) \left(\sin(\lambda_j z) + \frac{k\lambda_j}{h} \cos(\lambda_j z) \right)$$

Where

$$N_{i,j} = \underbrace{\int_0^R d\rho \rho J_0^2(\varepsilon_i \rho)}_A \underbrace{\int_0^d dz \left(\sin(\lambda_j z) + \frac{k\lambda_j}{h} \cos(\lambda_j z) \right)^2}_B \quad (2.16)$$

with $Q(\rho, z, t) = Q(\rho, z)[\Theta(t - t_0) - \Theta(t - t_1)]$, and $\Theta(t)$: Heaviside function. A solution for the inhomogeneous differential equation can be determined with generalized coefficients $c_{i,j}(t)$:

$$\begin{aligned}
\vartheta(\rho, z, t) &= \sum_{i,j} c_{i,j}(t) J_0(\varepsilon_i \rho) \left(\sin(\lambda_j z) + \frac{k \lambda_j}{h} \cos(\lambda_j z) \right) e^{-\gamma_{i,j} t}, \\
0 &= \left(\partial_\rho^2 + \frac{1}{\rho} \partial_\rho + \partial_z^2 - \frac{1}{K} \partial_t \right) \vartheta + \frac{Q}{k} \\
&= \sum_{i,j} \left(0 - \frac{1}{k} c_{i,j}(t) e^{-\gamma_{i,j} t} + \frac{q_{i,j}}{k} \right) J_0(\varepsilon_i \rho) (\sin(\lambda_j z) \\
&\quad + \frac{k \lambda_j}{h} \cos(\lambda_j z)) \Rightarrow c_{i,j}(t) = \frac{q_{i,j}}{\gamma_{i,j} \eta c_p} e^{\gamma_{i,j} t} + \text{const}(t).
\end{aligned} \tag{2.17}$$

This can be expressed in terms of both heating and cooling time as follows:

$$\begin{aligned}
T(\rho, t) - T_0 &= \\
\begin{cases} 0, & t \leq t_0 \\ \sum_{i,j} \frac{q_{i,j}}{\gamma_{i,j} \eta c_p} J_0(\varepsilon_i \rho) \left(\sin(\lambda_j z) + \frac{k \lambda_j}{h} \cos(\lambda_j z) \right) (1 - e^{-\gamma_{i,j}(t-t_0)}), & t_0 < t \leq t_1 \\ \sum_{i,j} \frac{q_{i,j}}{\gamma_{i,j} \eta c_p} J_0(\varepsilon_i \rho) \left(\sin(\lambda_j z) + \frac{k \lambda_j}{h} \cos(\lambda_j z) \right) (1 - e^{-\gamma_{i,j}(t_1-t_0)}) e^{-\gamma_{i,j}(t-t_1)}, & t_1 < t \end{cases}
\end{aligned} \tag{2.18}$$

By using equation 2.18 any radially symmetric heat source, location-dependent temperature development in and on the sample may be estimated analytically. Simulation result for BK-7[®] glass with 25mm diameter and 6 mm thickness, by taking Gaussian laser beam as heat source $Q(\rho, z) = \exp\left(-\frac{200\rho^2}{R^2} - \frac{15z}{d}\right)$ Figure 2.7 can be obtained [36].

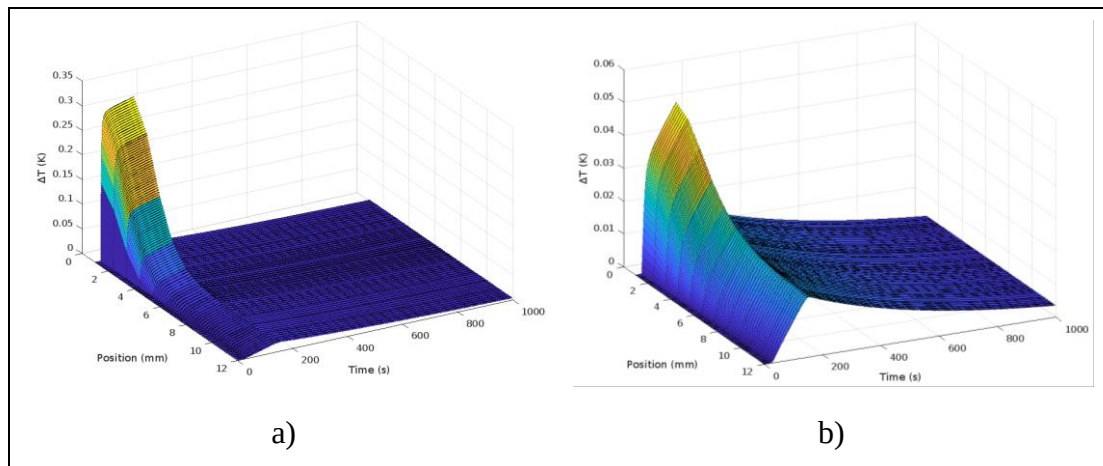


Figure 2.7: 3D modelling of the temperature rise simulation according to equation 20 a) for $z=0$ b) $z=d$

2.3.2. Homogenous Model

The homogeneous method, widely recognized and frequently employed, takes precedence as the primary approach in this context. The homogeneous method, in terms of mathematical workload, offers a simpler approach. However, it should be noted that this method poses several optimization challenges that need to be addressed. In order to utilize homogeneous model in the system there are some rules that needs to be followed.

The temperature elevation caused by the laser within the sample must remain significantly lower than the ambient temperature. Moreover, both the sample and its environment should attain thermal equilibrium prior to laser exposure. Furthermore, given the assumption of infinite thermal conductivity of the sample, a uniformly distributed temperature increase is anticipated within the sample. Consequently, the measured temperature elevation remains consistent across all detection positions (r and z) [37]. These presumptions lead to the following descriptions of the evolution of the laser-induced temperature rise in the sample:

$$\dot{\vartheta} = \frac{\alpha P}{mc_p} - \gamma \vartheta \Rightarrow \vartheta = (T_s - T_e) \quad (2.19)$$

γ can be known from previous measurements or can be calculated from:

$$\gamma = \frac{h}{mc_p} * 2\pi r(r + d) \quad (2.20)$$

The solution then can be found from equation 2.19 as follows:

$$\vartheta(t) = \begin{cases} 0, & t < t_0 \\ \frac{\alpha d P}{\gamma m c_p} (1 - e^{-\gamma(t-t_0)}), & t_0 < t < t_1 \\ \frac{\alpha d P}{\gamma m c_p} (e^{-\gamma(t-t_1)} - e^{-\gamma(t-t_0)}), & t > t_1 \end{cases} \quad (2.21)$$

2.3.3. Fitting Methods

According to the ISO 11551 fitting methods that are acceptable: The exponential method utilizes equation 2.21 to analyze the heating curve ($A_h - B_h e^{-C_h t}$) or the cooling curve ($A_c - B_c e^{-C_c t}$). These coefficients can then be used to derive the bulk absorption coefficient. In the context of laser calorimetry, it is stipulated that the irradiation duration for the exponential method should exceed a minimum threshold of 60 seconds, as indicated by reference [5]. The sample's absorption characteristics can be quantitatively evaluated using the exponential method, making it easier to fully comprehend the sample's optical features. An example of this method is shown in Figure 2.8.

$$\alpha = \frac{A_h C_h \sum_i m_i c_{p,i}}{dP} = \frac{A_c C_c \sum_i m_i c_{p,i}}{dP} \Rightarrow \gamma \sim C_c \cong C_h \quad (2.22)$$

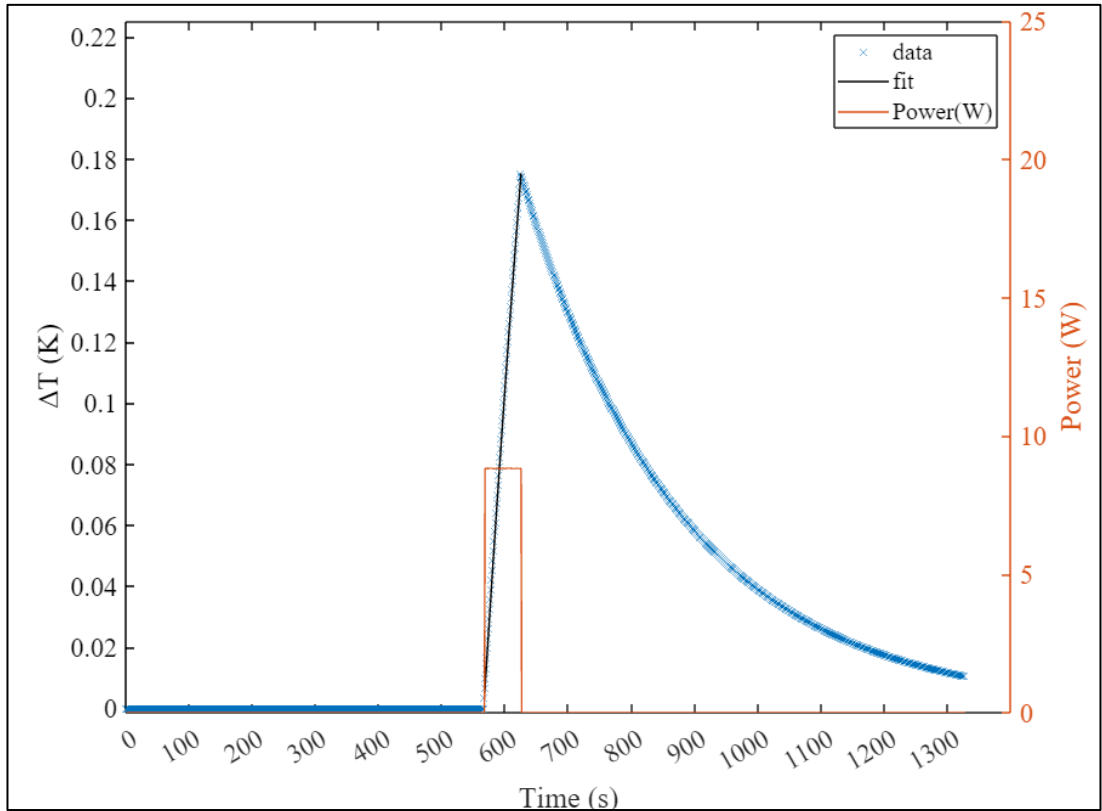


Figure 2.8: Exponential fitting method according to the ISO 11551

The so-called pulse method is an additional evaluation option that is especially helpful if the heating interval is not viable for fitting because of a brief irradiation time.

To employ the Pulse method effectively, the irradiation time must not exceed a predefined minimum threshold of 120 seconds, as indicated by reference [5]. In the pulse approach, the cooling curve is fitted exponentially, and the fitted function is extrapolated to the half-irradiation time (t_p). This method makes it possible to calculate the bulk absorption coefficient via the equation 2.23. Through mathematical analysis and extrapolation techniques, the pulse method offers a dependable method for learning more about the sample's absorption behavior.

$$\alpha = \frac{\vartheta_{pulse} C_c \sum_i m_i c_{p,i}}{dP} (1 - e^{-C_c t_p}) e^{\frac{C_c t_p}{2}} \quad (2.23)$$

In order to obtain more precise and dependable findings in actual experiments, it is critical to take into account the mass and specific heat coefficients of additional parts, such as holders or supporting structures. Despite their apparent insignificance, these extra parts can affect the system under study's total heat transmission and temperature dynamics. An example of this method is shown in Figure 2.9.

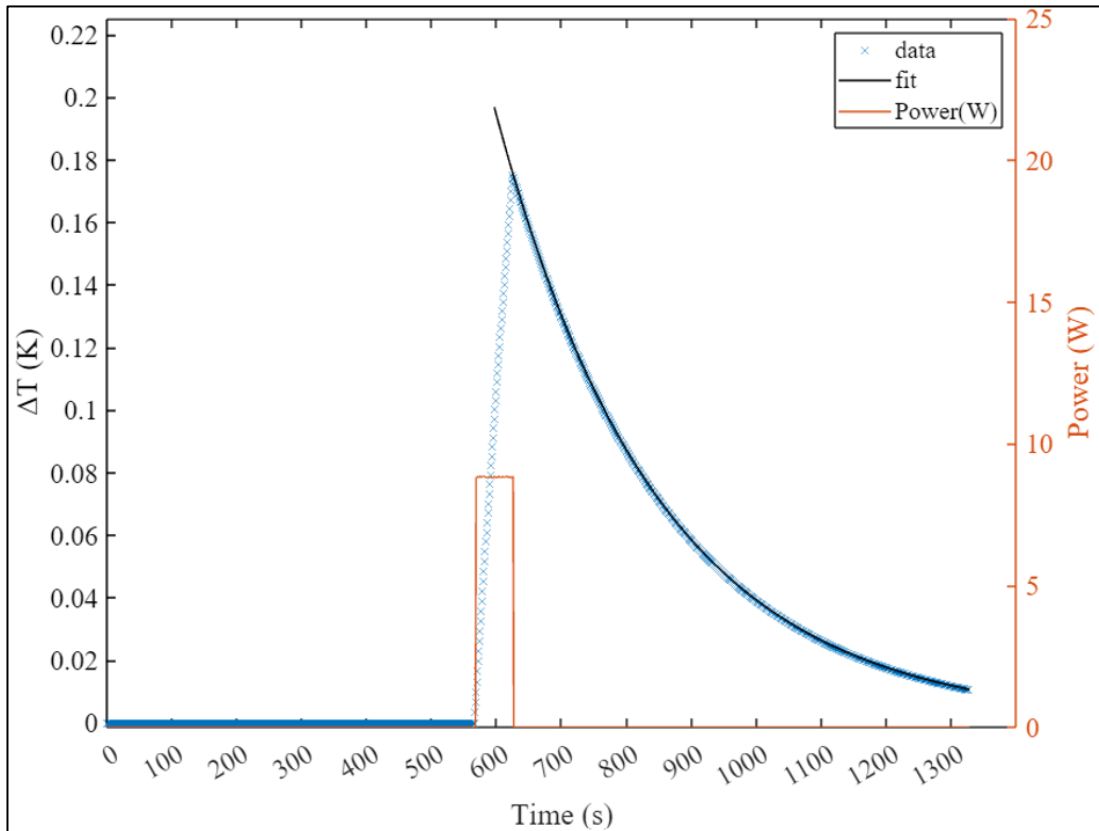


Figure 2.9: Pulse fitting method according to the ISO 11551

3. EXPERIMENTAL PROCESS

3.1. Atmospheric Procedure

Laser Calorimetry systems measure the extremely low absorption values of optical components down to parts per million (ppm) [38]. As a result, temperature readings from temperature sensors (NTC) are also at the level of a few mK during the measurement. In order to observe such small temperature changes, it is essential to minimize temperature fluctuations arising from the external environment. Therefore, the measurement system should be separated from the external environment as much as possible. For this purpose, the laser calorimeter system has two nested thermal isolation enclosures, as seen in Figure 3.1. A PID-controlled resistance heating system has also been included to eliminate any drift in the sample temperature during the test.

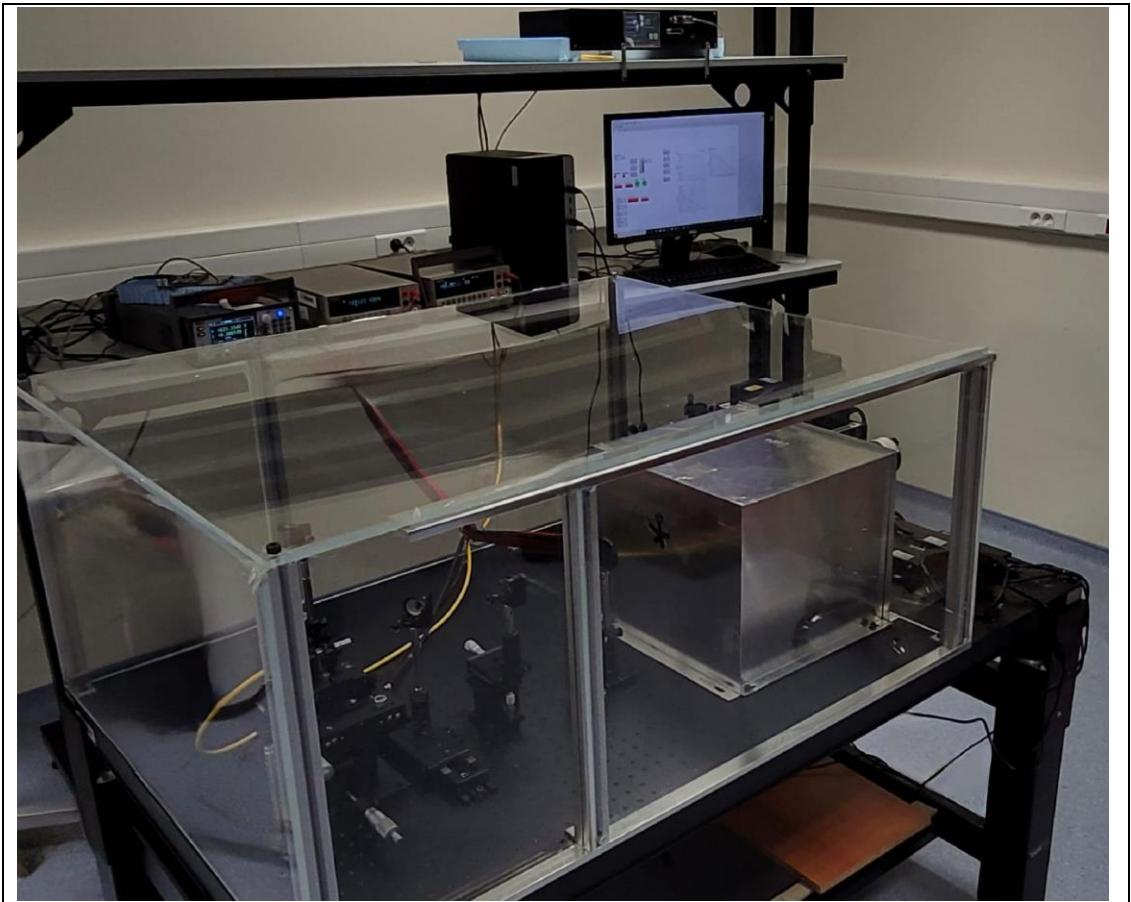


Figure 3.1: Image of laser calorimetry setup experimental in atmospheric environment

As a result, a temperature change of 10 mK per hour was achieved. According to the literature this value is considered acceptable [39]. An example of thermal stabilization of the system is shown in Figure 3.2.

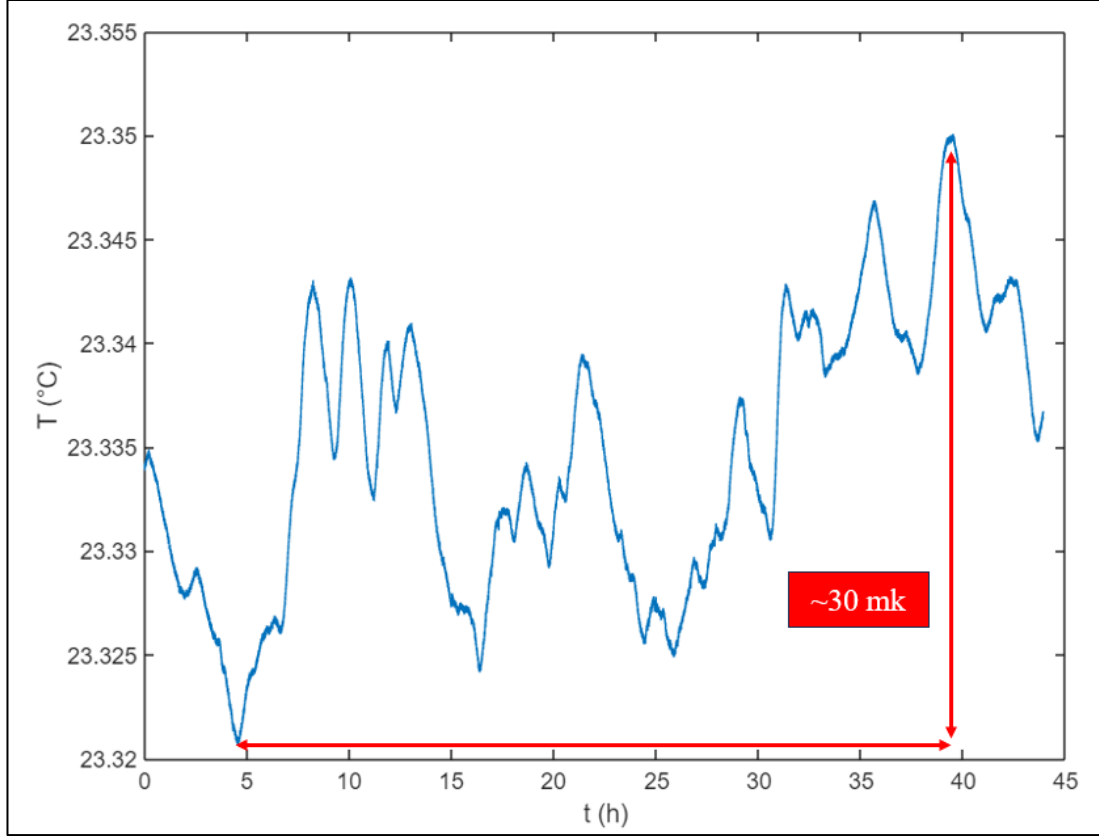


Figure 3.2: Thermal stabilization of the laser calorimetry in atmospheric condition for 45 hours

The laser calorimetric system in the atmospheric setup has various essential components. The system incorporates laser sources that operate at specific wavelengths within the visible to near-infrared (VIS-NIR) spectrum. These lasers are precisely tuned to emit light at 532 nm, 640 nm, and 1070 nm, with maximum power outputs of 1.5 W, 0.5 W, and 10 W respectively. This configuration enables the system to effectively perform measurements within the desired wavelength range for the intended applications in the VIS-NIR spectrum. The system is equipped with a thermally isolated chamber, to ensure an adiabatic environment for the accurate calorimetric experiments. A power meter is integrated into the setup to precisely monitor and quantify the laser power during the measurements. The setup includes an NTC thermistor to guarantee precise temperature measurements. A motorized stage

can be used to precisely position this thermistor, enabling small adjustments and improving the contact between the thermistor and the sample being evaluated. The exact positioning of the thermistor permits accurate temperature readings, which are necessary for a precise calorimetric study. The data acquisition systems that are included with the laser calorimetric system also play a critical role in gathering and preserving the experimental data. These systems make sure that the measured data is efficiently collected, stored, and analyzed, making it easier to thoroughly review and interpret the calorimetric results. Within the scope of this investigation, a software application has been developed using LabVIEW[®], equipped with the capabilities for data acquisition, analysis, and reporting. This software, showcased in Appendix A.1, offers a comprehensive toolset for efficiently managing and processing collected data throughout the course of the study. Figures 3.1 and 3.3 provides visual representations of the measuring system. Figure 3.1 shows a thorough image that depicts the actual configuration and componentry. A schematic illustration of the important parts and how they link is shown in Figure 3.3.

Before the measurement, the laser beam is aligned by using 2 mirrors, polarizer and focusing lens. If the sample has any dust contamination, cleaning gas will then be injected onto the surface and held there until all contaminants are eliminated. It is essential to start collecting temperature data at least two minutes before pointing the laser beam at the sample under examination. Using the temperature data obtained prior to the irradiation, extrapolation is done during the analysis phase of the measurement findings to account for the error produced by linear thermal drift. This extrapolated value is then subtracted from all measurement data, including both the heating and cooling phases, in order to eliminate the error. Subsequently, the sample is subjected to irradiation using a single mode laser for a duration of 30 s to 300 s. The laser beams are focused onto the surface of the sample with a spot of about 500 μm diameter at $1/e^2$.

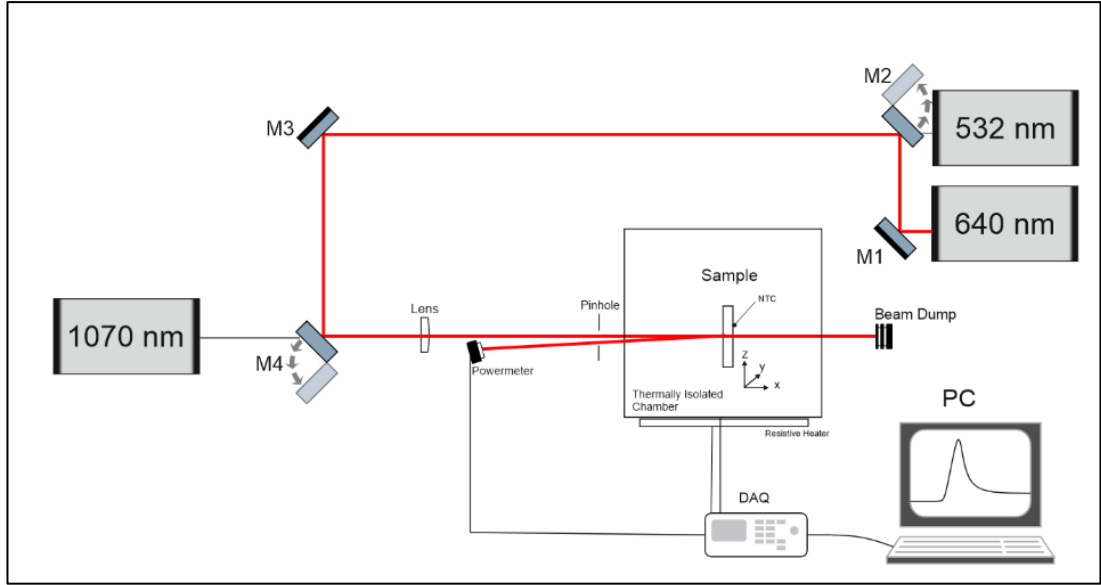


Figure 3.3: Schematic of laser calorimetry experimental setup in atmospheric environment

After the irradiation process is complete, a waiting period of at least 200 seconds is required before proceeding with the application of the abovementioned fitting procedure. This waiting period gives the sample enough time to settle and attain a condition that is suitable for precise fitting. The accuracy and precision of the fitting operation are improved by providing an adequate waiting period, producing findings that are more accurate and significant in compliance with ISO 11551 requirements.

3.2. Vacuum Procedure

The procedural steps for vacuum conditions closely resemble those under atmospheric conditions. However, a distinct divergence lies in the requirement for a thorough cleaning of the vacuum chamber prior to commencing measurements. It is crucial to obtain $\sim 10^{-6}$ mbar vacuum level where heat loss is predominantly influenced solely by contact and radiation. Figure 3.4 depicts the photographs of the developed vacuum laser calorimetry system. In addition to the atmospheric procedure, 1 mm apertures are added inside the vacuum chamber for minimizing thermal disturbances from reflections of vacuum windows as shown in Figure 3.5.

Both benchmarks consist of a single mode laser, a power meter to measure the power of the laser, a focusing optical arrangement to focus the laser beam onto a

specimen, a couple of contact thermometers, a system for the holding of a sample, PID controlled thermally isolated cabin and data acquisition system.

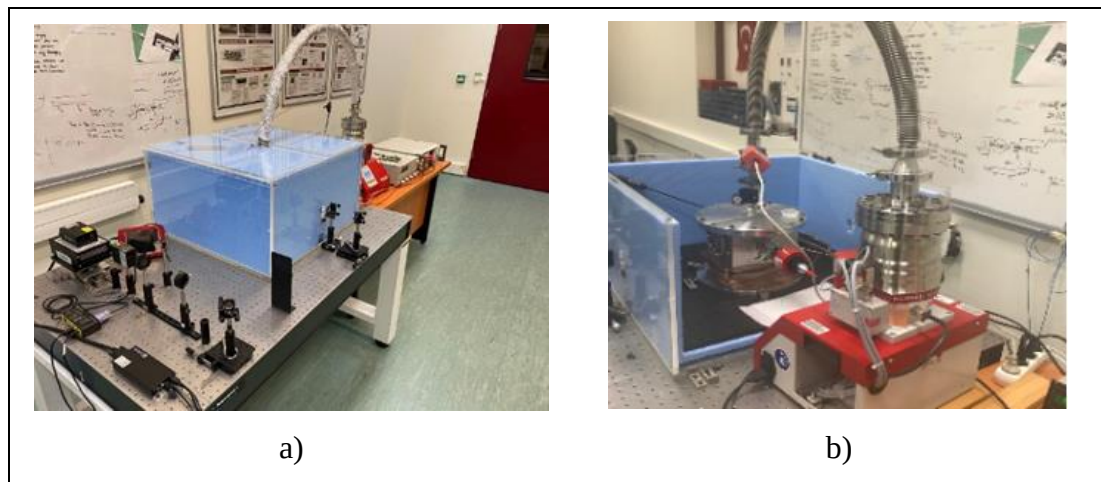


Figure 3.4: Images of laser calorimetry experimental setup in vacuum environment, a) optical path and b) vacuum chamber

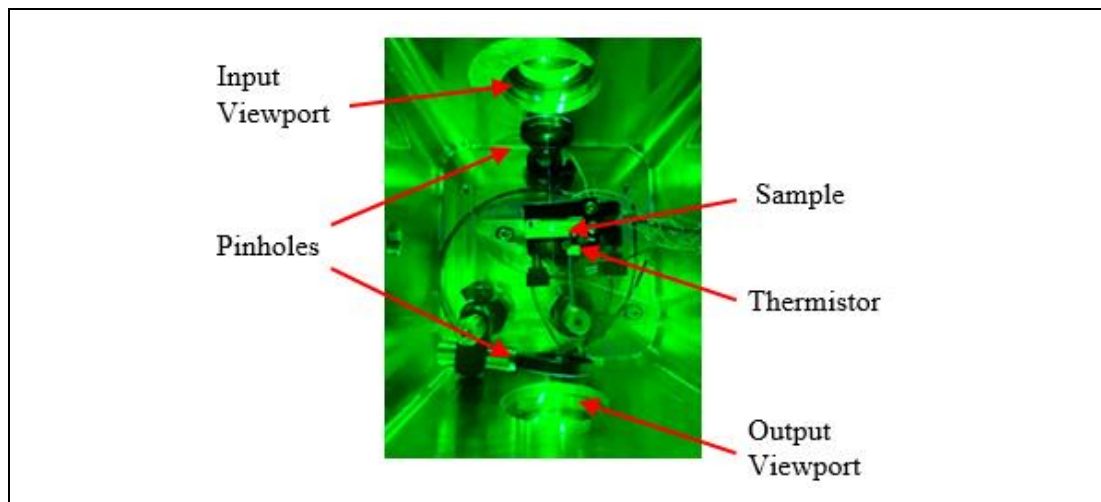


Figure 3.5: The view of the vacuum chamber inside.

Two high-precision and small-size temperature sensors (thermistors) are used in the vacuum chamber to monitor the sample surface temperature by direct contact with the surface. An example of thermal stabilization of the system is shown in Figure 3.6. Figure 3.7 showcases a schematic illustration of the laser calorimetry system operating within a vacuum environment.

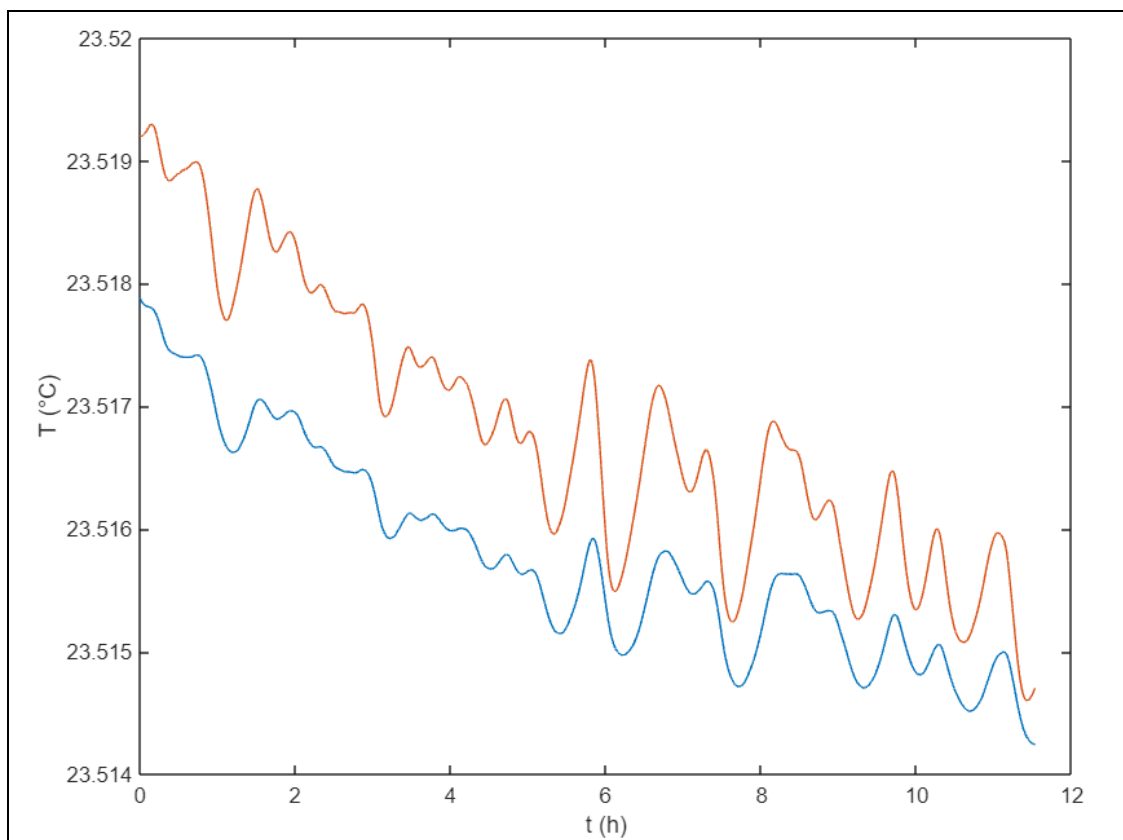


Figure 3.6: Thermal stabilization of the laser calorimetry in vacuum condition for 12 hours

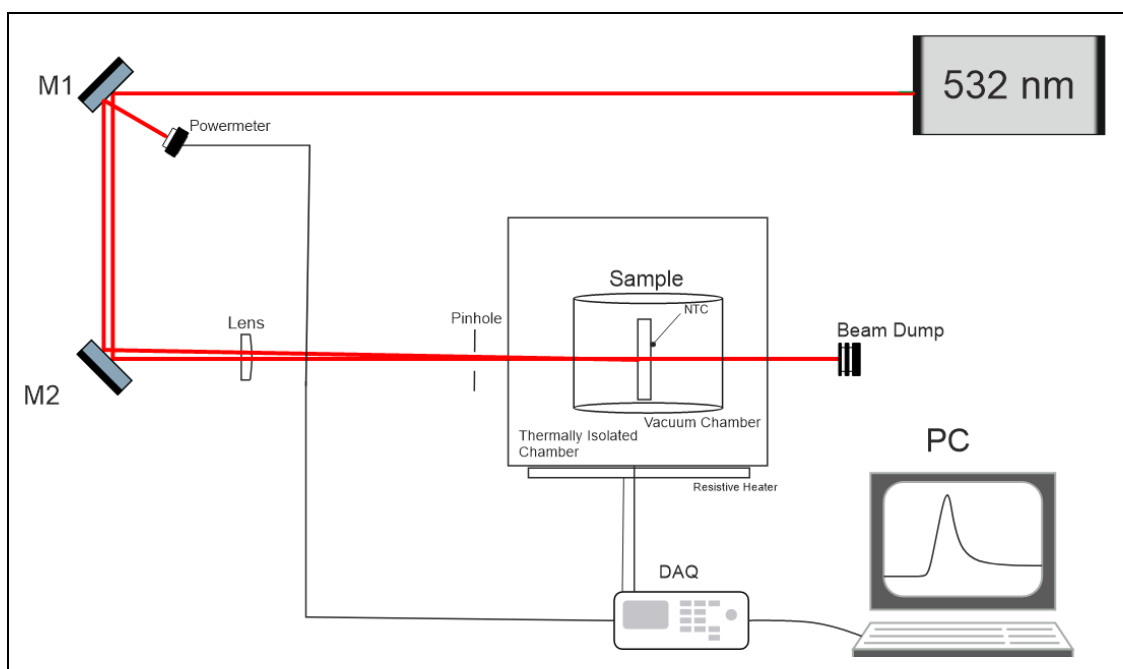


Figure 3.7: Schematic of laser calorimetry experimental setup in vacuum environment

3.3. Optimization

Within the framework of both experimental processes, several optimization problems emerged, necessitating careful attention and resolution. These three primary optimization challenges encompassed diverse aspects of the experimental setup.

The first major concern was the removal of scatter radiation which is obvious in Figure 3.5. A laser system may produce scatter radiation from a number of different sources, including optical scattering at reflective or transmissive beam guiding elements and diffraction at beam-limiting apertures. In order to overcome this issue laser beams carefully tuned, optical components regularly cleaned and spatial filters were used. Standard suggests that, in order to neglect to scatter radiation system must be calibrated each time when the sensor position changed, or the sample changed. According to this calibration, the temperature sensor's sensitivity must be one order lower than the sample's temperature rise.

Finding the exact spot where the detector would be on the sample was the next optimization issue. For accurate measurements and representative data to be recorded, the detector's placement had to be done precisely. An initial solution to address this problem involved the utilization of a self-centering holder and a specially designed 3D printed part.



Figure 3.8: Schematic of laser calorimetry experimental setup in vacuum environment

This configuration was designed to securely position the temperature sensor at the specified location on the homogeneous model. A visual representation of this arrangement can be observed in Figure 3.8.

Nevertheless, this particular configuration did not yield satisfactory results in terms of achieving optimal thermal contact and mitigating scatter radiation. Finally, it was essential to make sure the sample and thermistor had adequate thermal contact. It is essential to create trustworthy thermal connections between the sample and the thermistor sensor for accurate temperature measurements. A new and compact sample holder was designed and manufactured as shown in Figure 3.9.a) and 3.9.b), in order to address the aforementioned issues and enhance the experimental setup. To improve the overall performance and dependability of the system, this design includes a number of crucial elements.

A special holder is designed and manufactured to hold the sample under test (as shown in Figure 3.9). Two pieces of thermistors are contacted to the rear side of the sample surface. An x-y translational piezo step motor was installed in the sample holder to provide accurate and controlled movement in two dimensions. This made it possible to precisely position the temperature sensor within the sample at various points. The incorporation of the piezo step motor increased measurement precision by ensuring repeatability and reproducibility in sample positioning in addition to facilitating simple adjustment. In order to adjust the sample surface with respect to the impinging laser beam, three axis controlling stage is placed on the bottom side. Furthermore, the compact design of the sample holder offered several benefits. The utilization of a blackened aluminum outer part helped to mitigate scatter radiation. The sample was also supported by glass rods in order to produce superior thermal performance and equal temperature dispersion. This design decision reduced direct temperature gradients and improved thermal stability by minimizing contact between the sample and the holder. The glass rods offered a solid support framework and enabled effective heat conduction, leading to more accurate temperature readings and improved experimental results.

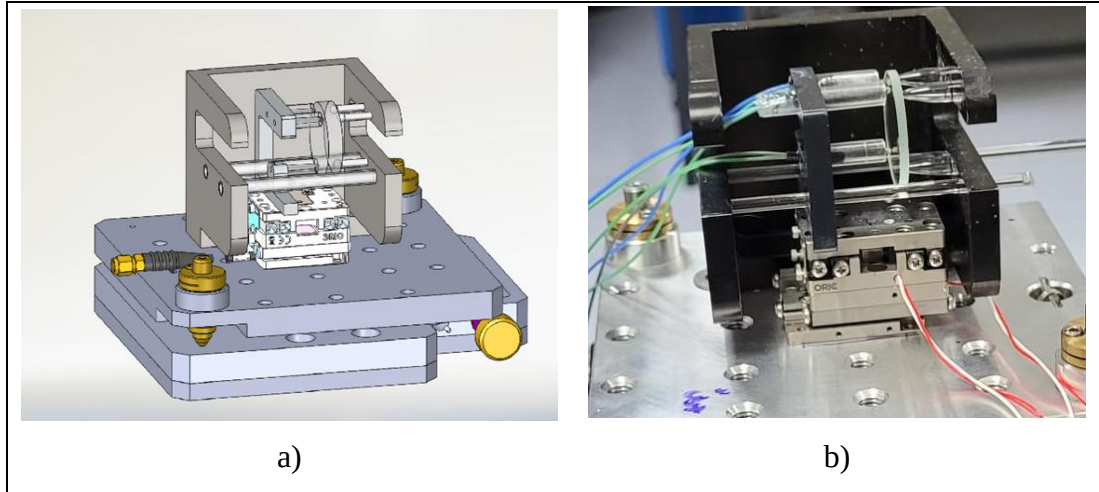


Figure 3.9: a) SolidWorks drawing of the new sample holder and b) an image of the new sample holder.

3.4. Calibration

The main advantage of laser calorimetry is that it does not need any reference material in order to obtain absolute values. The absorption coefficient value can be deduced through equations 2.22 and 2.23, provided that the mass and specific heat coefficient of the heated sample are known. Furthermore, it's crucial to perform calibration for both the laser power and the temperature sensor before the measurements.

Prior to conducting measurements, it is essential to carry out preliminary calibration procedures to ensure the accuracy of the subsequent results. A crucial aspect of this calibration involves assessing the characteristics of the beam profile and beam radius. In the context of this study, it has been observed that the beam profile aligns with a Gaussian distribution, and the beam radius measures $500\text{ }\mu\text{m}$ for both systems. In situations involving multiple laser systems, ensuring the lasers converge precisely onto a singular point on the sample is crucial. This alignment is accomplished by adjusting the centroid of the beams using a beam profiler. This initial calibration process plays a pivotal role in establishing a robust foundation for the subsequent measurement activities, facilitating accurate and reliable outcome evaluation. Subsequently, as pointed out earlier, power meter calibration plays a crucial part in laser calorimetry. Calibration procedure which includes measurement of the absolute power at the sample's position followed by the measurement of the secondary power

meter using the reflected beam from the beam splitter is illustrated in Figure 3.10.a). As depicted in Figure 3.10.b), by employing a linear fitting approach between the two power meters, it becomes feasible to acquire the actual power value for real-time measurements.

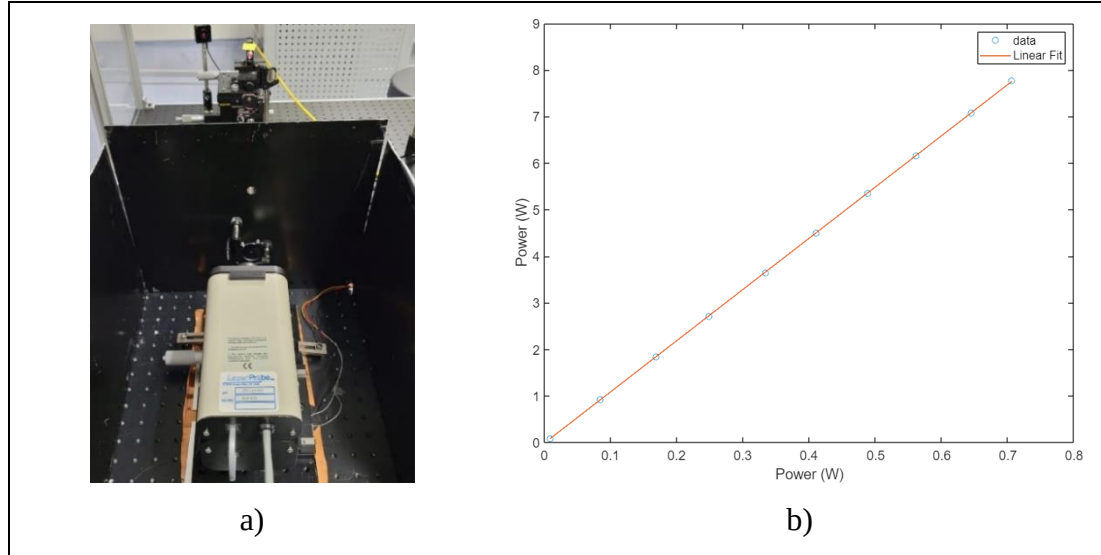


Figure 3.10: Comparative power meter calibration a) image of the setup and b) fit graph

It's well-established that glasses exhibit low thermal diffusivity at room temperature [41]. Illustrated in Figure 3.11.a), when thermal diffusivity decreases, the sensor's location gains increased significance. Nevertheless, there comes a juncture where the detector records a comparable value regardless of diffusivity. This specific outcome is depicted in Figure 3.11.b) [42]. By implementing this numerical calibration, the homogeneous model can be employed in accordance with the guidelines in the ISO 11551 standard. Nevertheless, as depicted in Figure 3.11.a), the ideal sensor positions began to diverge below $0.002 \text{ cm}^2/\text{s}$. This divergence is shown in Figure 3.1.b). This observation indicates that the efficacy of this approach diminishes when the thermal diffusivity reaches a sufficiently low threshold.

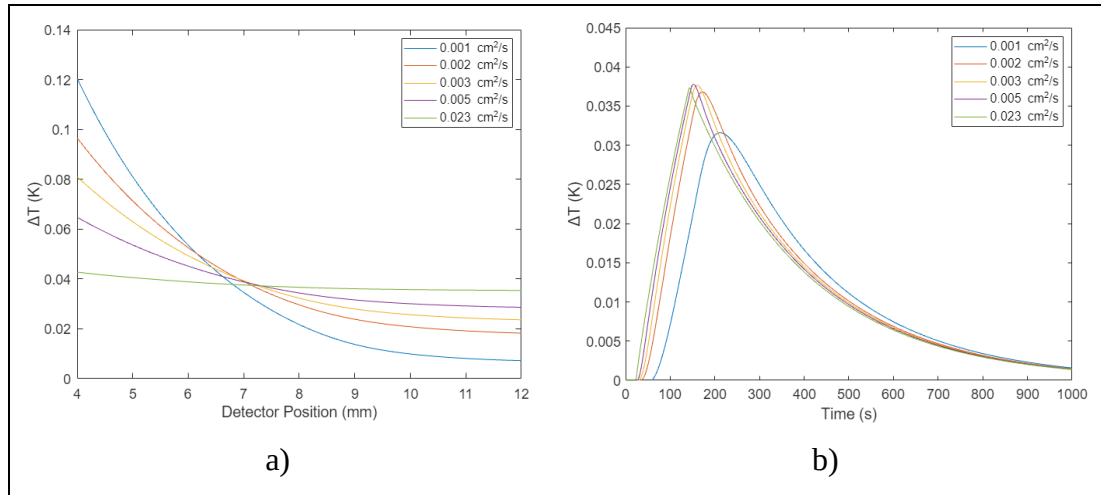


Figure 3.11: Numerical simulation according to inhomogeneous model for 25mm diameter and 6 mm thickness samples a) varying sensor position with varying thermal diffusivity b) temperature rise at 7.2 mm

In order to obtain the optimal sensor position, which is less effected from scattering, the detector is subjected to a scanning process with increments of 0.5 mm, in the absence of the sample. This step enables the determination of the additional temperature influenced by the sample holder which is around few mK with the current sample holder at 8.7 W. The precise position of the sensor on the sample is determined using millimetric paper as shown in Figure 3.12.a), a technique that allows for accurate alignment and determination of the coordinates of sensor in reference to the surface of the sample. As illustrated in Figure 3.12.b), the obtained results are in line with the findings presented in Figure 3.11.a), wherein proximity of the detector to the incident beam results in a notable increase in temperature rise.

In case of obtaining influence of the heat capacity, a couple of samples with known heat capacity must be coated with known absorptivity material such as graphite. During the evaluation of a calibration measurement, the effective heat capacity (C_{eff}) is calculated as the value that needs to be used in place of the summation ($\sum_i m_i c_{p,i}$) in order to accurately reflect the degree of absorption [43]. This effective heat capacity can then be used for samples with the same geometry and substrate properties. Furthermore, this approach can serve as a means to validate the optimal sensor position, along with confirming the application of ISO 11551's recommended correction factor (f_c). This correction factor acts as a compensation for samples with low thermal conductivity.

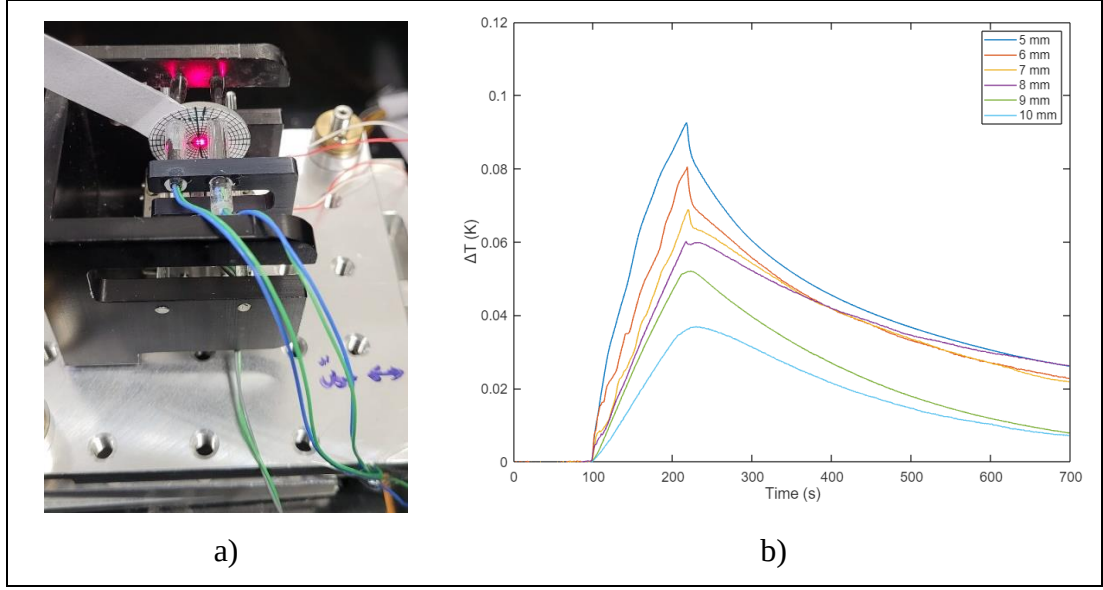


Figure 3.12: Sensor position calibration a) image with millimetric paper and b) scanning result.

In order to achieve higher levels of certainty, it is recommended to utilize the electrical calibration technique, as introduced by Decker, Temple et al. [44], [45]. This technique involves the use of an adiabatic chamber, as depicted in Figure 3.13, where the sample is held within a measuring ring. Inside the chamber, a temperature sensor and a heating resistor are attached to the measuring ring. This setup, known as adiabatic laser calorimetry, enables the measurement of the temperature increase of both the sample and the measuring ring when irradiated with a laser. When the sample is exposed to laser irradiation, it undergoes heating along with the measuring ring, and the resulting temperature increase is measured using a thermistor. In order to calibrate the system, a voltage is applied to the heating resistor connected to the measuring ring, inducing a comparable temperature rise through a heating current. Subsequently, the optically absorbed power can be calculated based on the input electrical power, which can be determined with relatively high precision with equation 3.1.

$$\alpha P = \frac{V_h I_h \Delta T}{\Delta T_{el}} \quad (3.1)$$

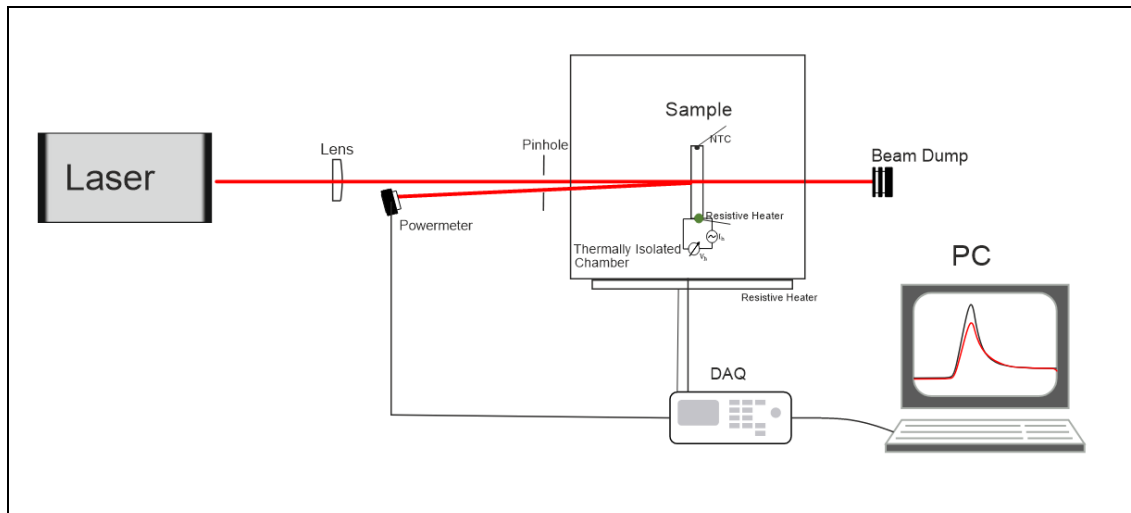


Figure 3.13: Electrical calibration system according to [44]

In this measuring concept, there is no requirement for knowledge regarding the heat capacities and masses of the sample and retaining ring, or the absolute calibration of the temperature sensor. However, it should be noted that when dealing with samples of low thermal conductivity, a time delay may occur during the start and stop of the laser due to the placement of the temperature sensor and resistive heater on the metal heater ring. This delay is absent when heating with a resistive heater due to the efficient thermal conductivity of the metal ring. To address this issue, a resistive heater with the same radius as the laser is positioned at the laser's location, as depicted in Figure 3.14.a). Example results obtained under varying electrical power conditions are visually depicted in Figure 3.14.b). These results hold significant relevance, as they form a crucial component of the calibration process.

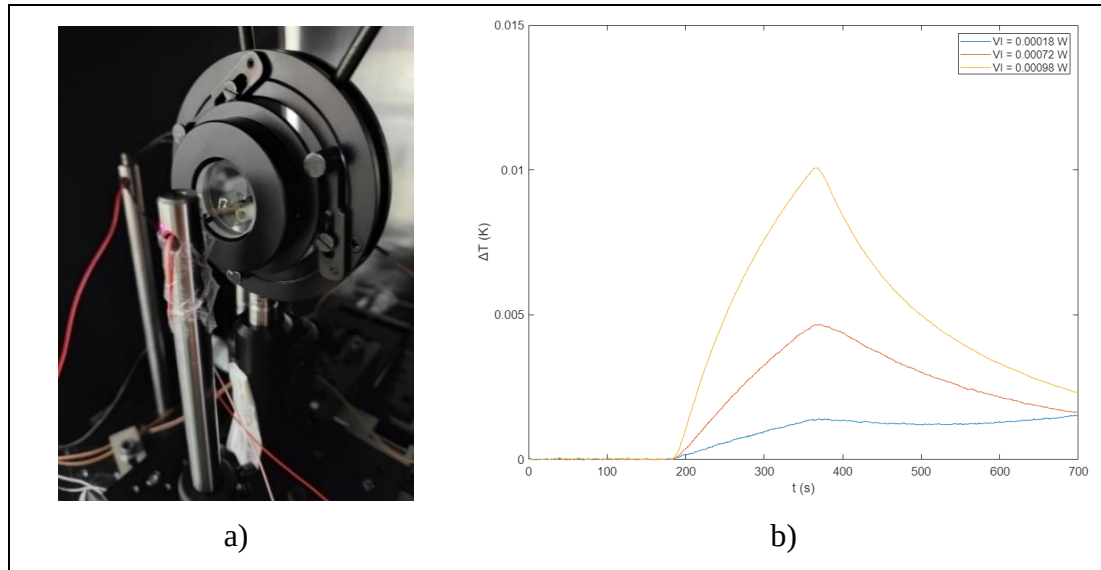


Figure 3.14: Electrical calibration a) image of resistive heater and b) temperature rise with different electrical power.

However, it is important to acknowledge a significant challenge associated with this approach. Coupling the electrical heating power into the sample in a controlled and defined manner becomes complex. Since the heating point is located on the optical functional surface, great care must be taken to ensure proper coupling of the heating resistor to the sample while avoiding any potential degradation. Traditional methods such as adhesive bonding or applying excessive pressure are not viable options due to their potential adverse effects. Consequently, alternative techniques and careful considerations are required to establish a reliable and stable connection between the heating resistor and the sample, ensuring accurate and consistent results throughout the experiments.

4. RESULTS

4.1. Atmospheric Results

Atmospheric Measurements are done in both vacuum chamber and thermally isolated chamber separately in order to compare the results from the vacuum measurements. In both measurements setup (LC1 and LC2), measurement results for BK-7[®] and Fused Silica were obtained and compared with each other. In addition, an analysis was conducted on a Zerodur[®] sample employing LC1. This process was undertaken to enhance the Laser Calorimetry system's performance and precision when dealing with materials exhibiting elevated levels of absorption. Both absorption coefficient and heat loss coefficient of the BK-7[®] sample is shown in Figure 4.1.a) and Figure 4.1.b).

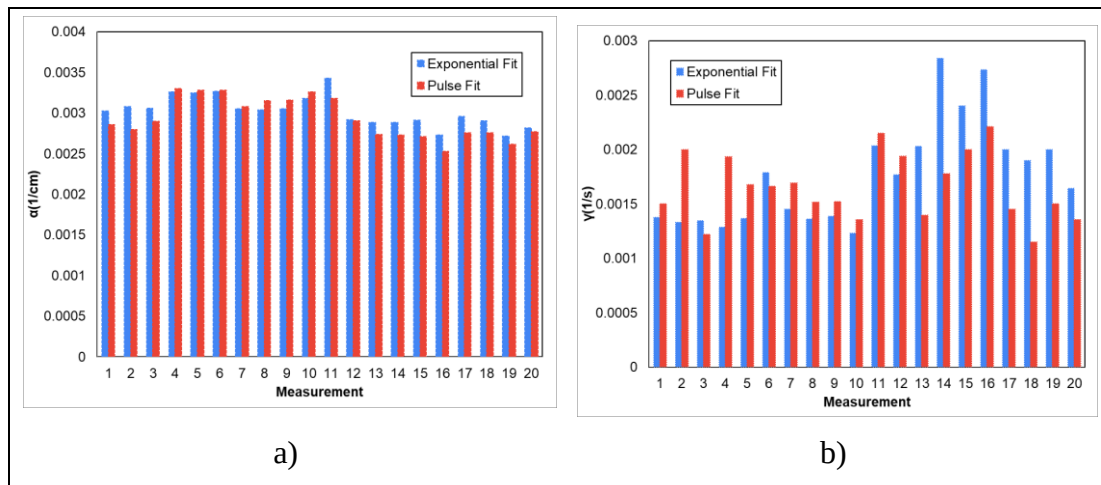


Figure 4.1: Examination of BK-7[®] properties under atmospheric conditions, showcasing results acquired through LC2 at 532 nm with a power output of 1W.

a) Depiction of the absorption coefficient and b) heat loss coefficient.

As illustrated in Figure 4.1.a), the accuracy of the measurements approximately 10%, with its primary source attributed to the inherent variability arising from sensor positioning. Furthermore, as illustrated in Figure 4.1.b), the heat loss coefficient, as derived through curve fitting, demonstrates a more variation, particularly in the context of exponential fit as opposed to pulse fit. This variation is believed to be influenced by the specific placement of sensors, given that the exponential fit is applied to the irradiation region. Nevertheless, there exist additional parameters that could

potentially contribute to this observed variation. These parameters encompass factors such as irradiation duration, potential scattering originating from the chamber environment, and the potential influence of temperature drift.

As referenced in Section 3.1, the management of temperature drift is executed through a PID controller. This ensures that measurements are conducted within a linear temperature drift regime, wherein the drift remains under 10 mK/h. Furthermore, concerning the issue of scattering, preliminary measurements are conducted using an empty holder prior to the actual measurements. During these preliminary measurements, the temperature escalation is controlled to be at least an order of magnitude lower than that during the subsequent measurements involving the sample which suggested by ISO 11551 standard. Nonetheless, as illustrated in Figure 4.2.a), it is evident that the heat loss coefficient is significantly contingent upon the duration of laser irradiation in the case of the exponential method. Conversely, the pulse method, as depicted in Figure 4.2.b), remains unaffected by the duration of laser irradiation. Moreover, as indicated in Figure 4.3, the duration of laser irradiation also impacts the absorption coefficient, albeit with a comparatively lesser degree of influence. Collectively, this information contributes to a more comprehensive understanding of the dynamics underlying the laser calorimetry analysis process.

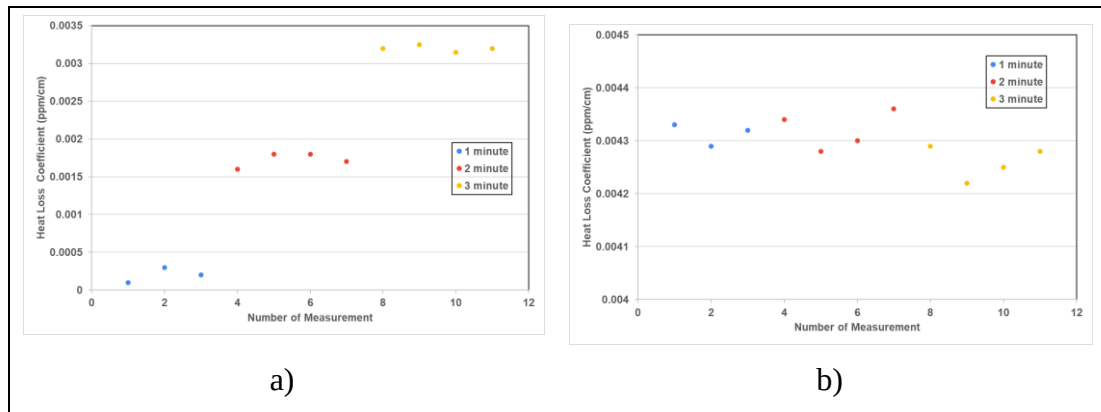


Figure 4.2: Examination of Zerodur® properties under atmospheric conditions, showcasing results acquired through LC1 at 1070 nm with a power output of 10W.
a) Depiction of the absorption coefficient and b) heat loss coefficient.

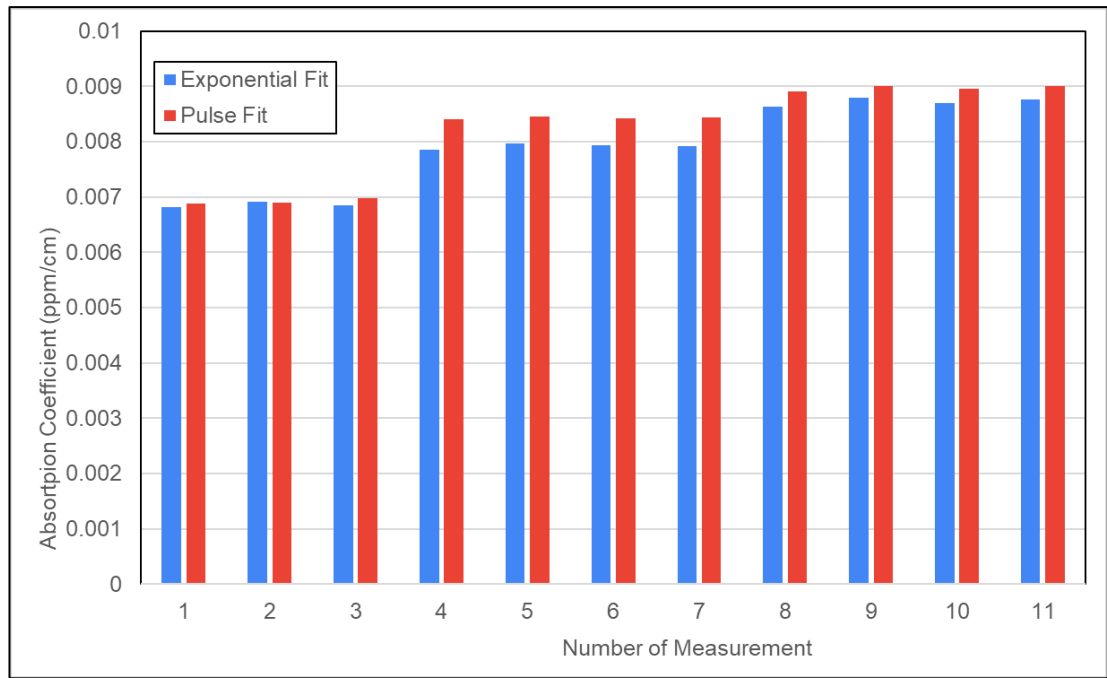


Figure 4.3: Zerodur® absorption coefficient under atmospheric conditions, showcasing results acquired through LC1 at 1070 nm with a power output of 10W.

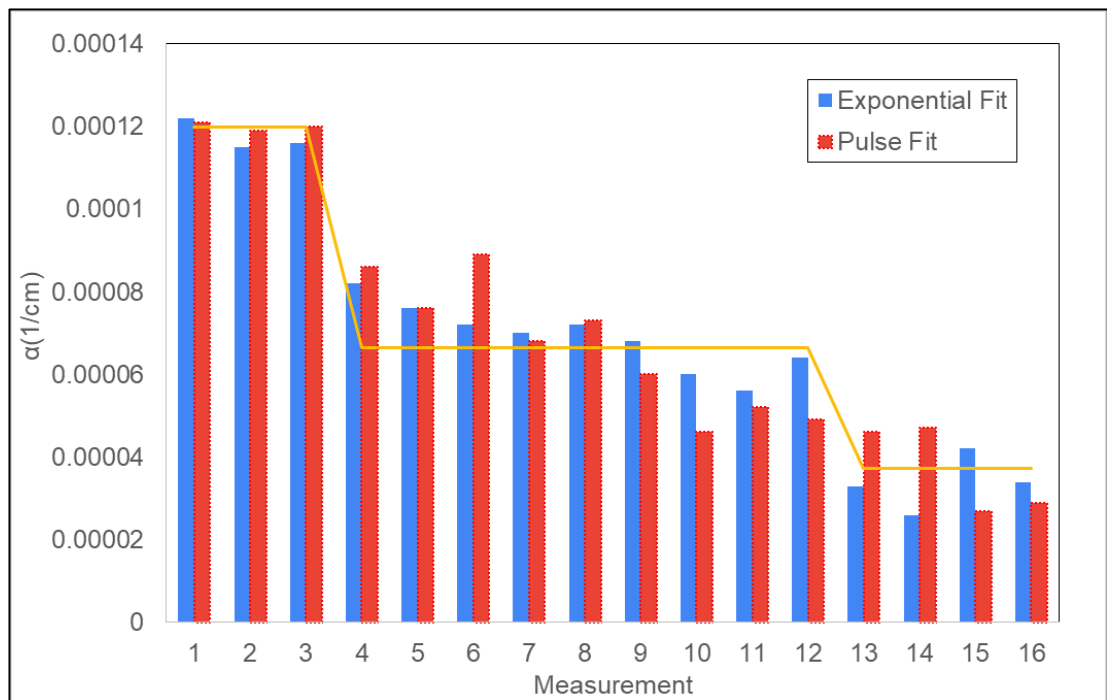


Figure 4.4: Variation in the absorption coefficient of the Fused Silica sample throughout iterative optimization procedures in LC1 at 1070 nm and 10W power.

Illustrated in Figure 4.4, the absorption coefficient of the Fused Silica sample displays a consistent trend of reduction across successive optimization processes.

These iterative processes encompassed modifications such as altering the sample holder configuration, mitigating scattering effects through the incorporation of a pinhole, and refining the positioning of the sensor.

As demonstrated in Figure 4.5, the optimization processes have yielded minimal improvement in the absorption coefficient of Fused Silica.

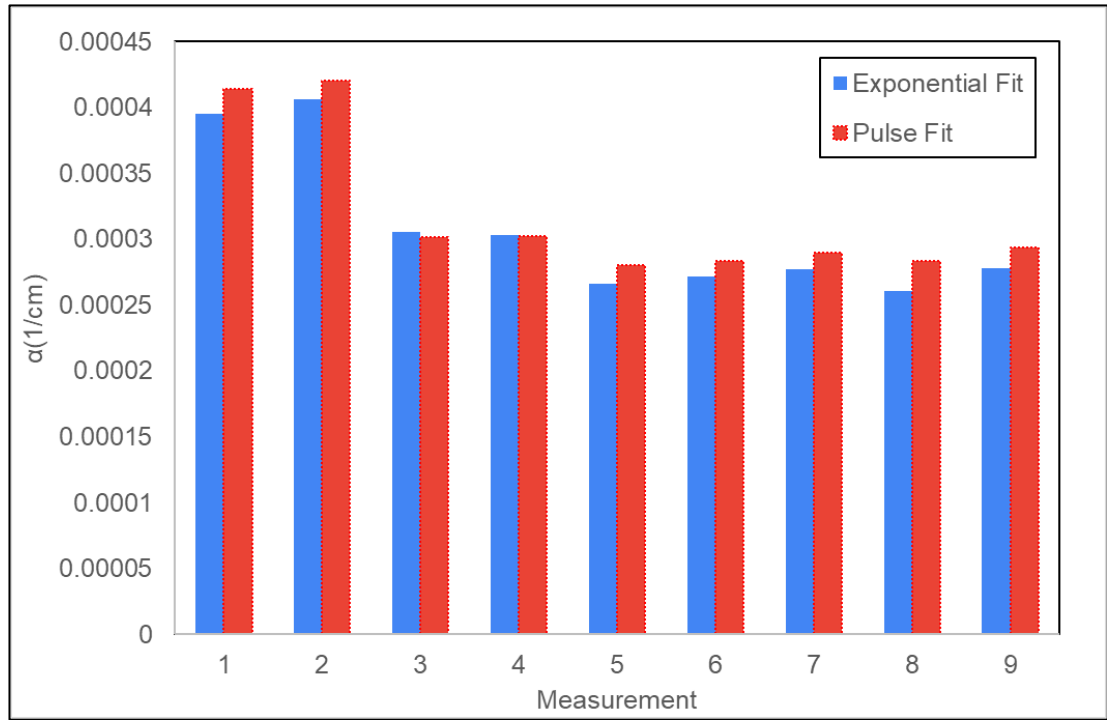


Figure 4.5: Variation in the absorption coefficient of the Fused Silica sample in LC2 at 532 nm and 1 W power.

To facilitate measurements at three distinct wavelengths, a configuration involving three lasers was implemented. These lasers were precisely overlapped on the sample's surface, ensuring alignment at the same point and with an identical beam radius as described at Section 3.4.

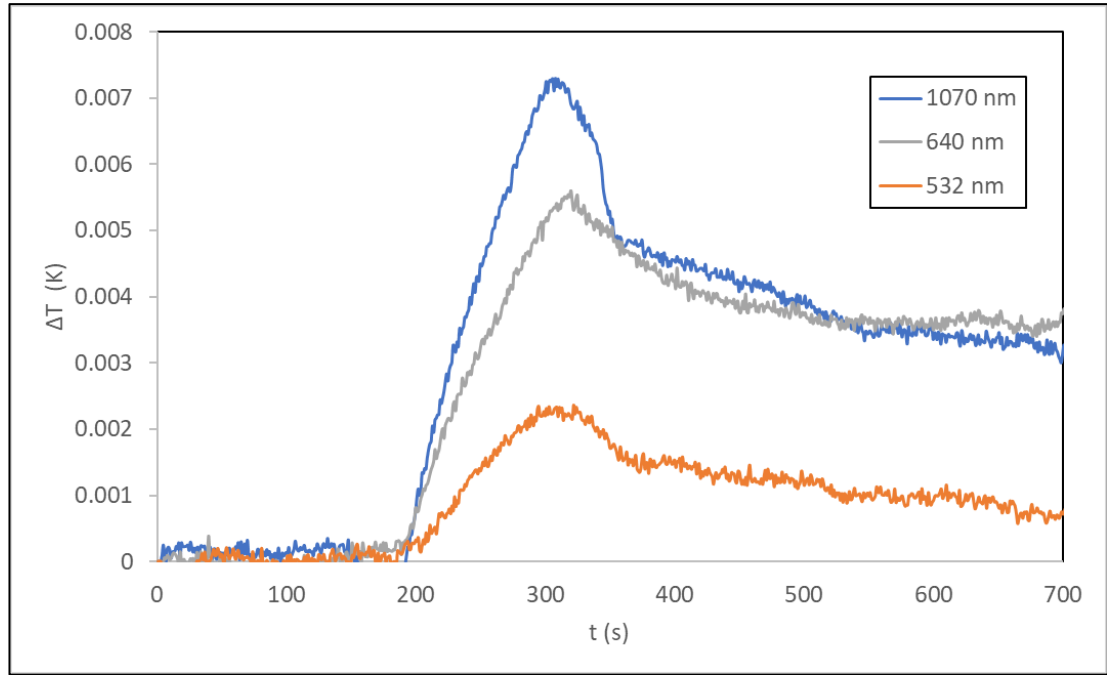


Figure 4.6: Temperature rise of the BK-7® sample in LC1 at 3 different wavelength and 0.5 W power.

Subsequently, the lasers were irradiated sequentially, resulting in the acquisition of three temperature rise (as shown in Figure 4.6) that align with the values reported in the existing literature [46].

4.2. Vacuum Results

The decision of performing laser calorimetry measurements in vacuum is a strategic one. Working in a vacuum has various advantages, one of which is the lack of potential interference from ambient gases, which might impair the accuracy of optical absorption coefficient measurements. The lack of these gases in a vacuum creates a more controlled and stable environment for precise optical characterization. Furthermore, vacuum circumstances can be beneficial when studying the optical properties of materials in space or other low-pressure situations where atmospheric conditions do not apply, making it a versatile approach for a variety of scientific and technical applications.

Vacuum measurements were conducted within a controlled vacuum chamber environment. In the measurement setup, as illustrated in Figure 3.4, the results displayed a notable consistency among themselves. However, as the pressure was

systematically reduced, a consequential increase in the absorption coefficient was observed, as depicted in Figure 4.7. Furthermore, as evident in Figure 4.8.a) and Figure 4.8.b), both the absorption coefficient and heat loss coefficient of the BK-7[®] sample were determined across varying pressure conditions.

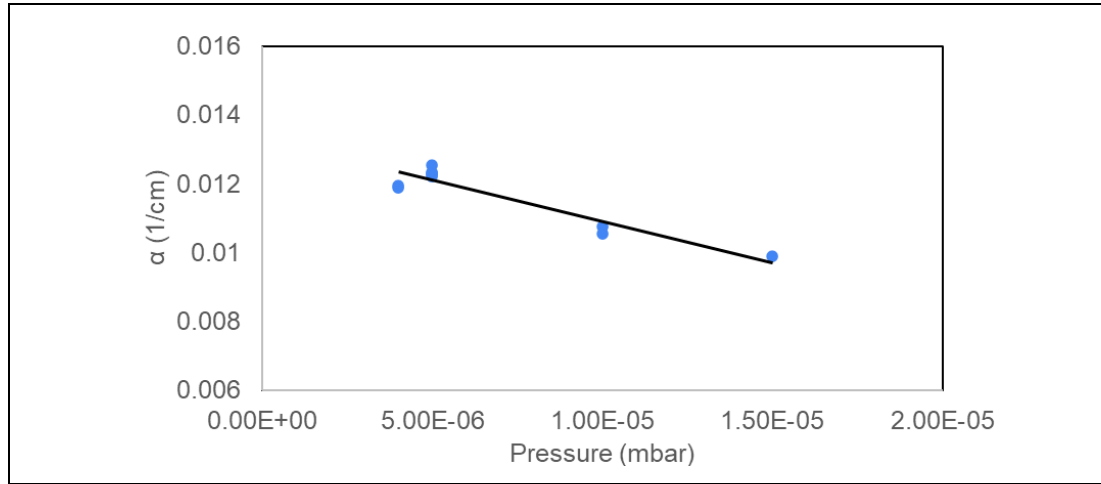


Figure 4.7: Absorption coefficient of the square BK-7[®] sample under different vacuum levels

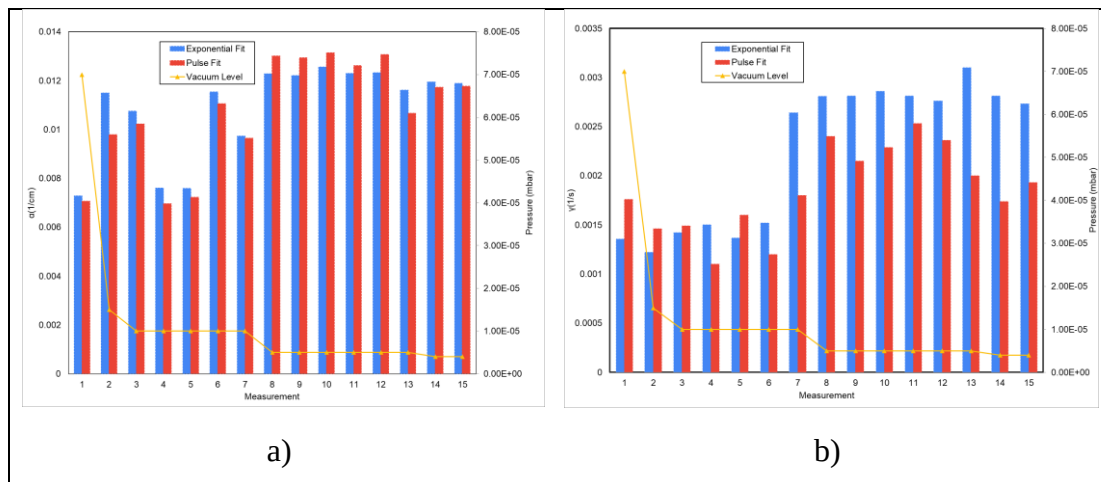


Figure 4.8: Examination of BK-7[®] properties under varying vacuum conditions, showcasing results acquired through LC2 at 532 nm with a power output of 1W.
a) Depiction of the absorption coefficient and b) heat loss coefficient.

As depicted in Figure 4.9.a) and Figure 4.9.b), both the absorption coefficient and heat loss coefficient of the Fused Silica sample were determined under vacuum conditions, employing both the pulse and exponential methods. Subsequently, a comparative analysis was conducted to assess the variations between the two methods.

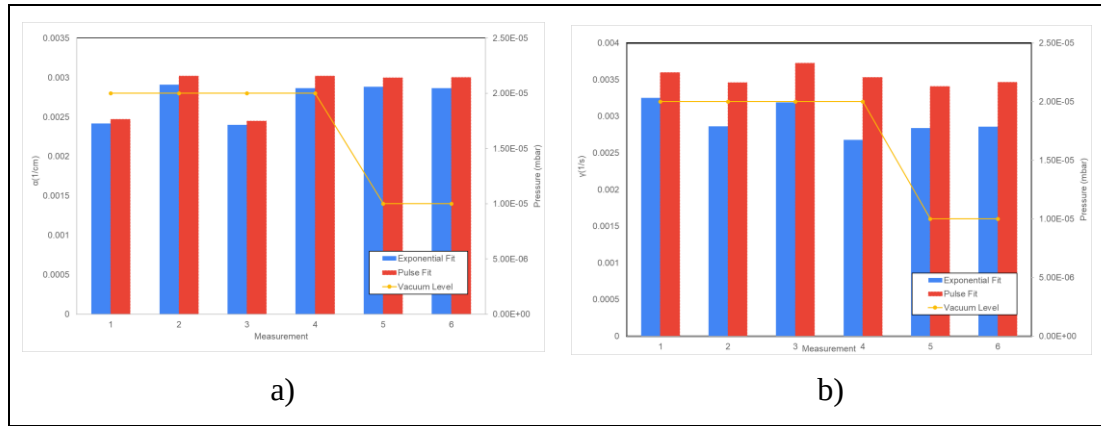


Figure 4.9: Examination of Fused Silica properties under varying vacuum conditions, showcasing results acquired through LC2 at 532 nm with a power output of 1W. a) Depiction of the absorption coefficient and b) heat loss coefficient.

4.3. Simulation Results

In this thesis, during the data processing stage, various mathematical fitting techniques were employed in line with the measurement results, and the obtained outcomes were compared to reference materials. The conformity of the measurements conducted under high vacuum conditions ($\sim 1 \times 10^{-6}$ mbar) with ISO 11551 standards was examined. Furthermore, a comparative analysis was performed by comparing this data with results obtained through the implementation of the finite element method using the COMSOL Multiphysics® program and numerical simulations conducted with MATLAB®.

4.3.1 Finite Element Method Simulation

To utilize the finite element method, the first step involves establishing the fundamental physics principles underlying the system. This entails creating a foundation based on the principles of heat transfer. For instance, a straightforward ideal case of a heating process can be simulated on a BK-7® sample within the COMSOL Multiphysics® program as shown in Figure 4.10.a) and result is shown in Figure 4.10.b). This initial simulation serves as a starting point for understanding the thermal behavior and response of the sample under controlled conditions.

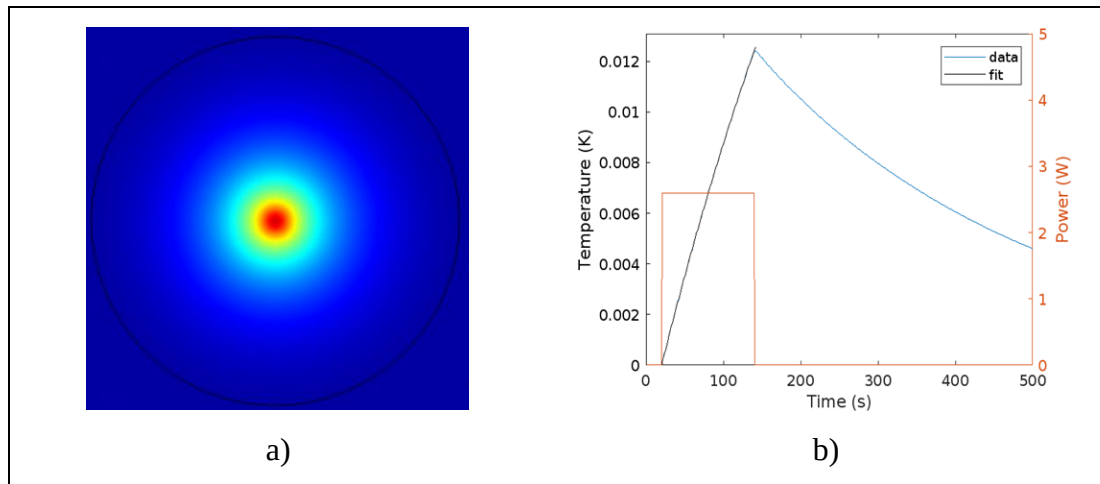


Figure 4.10: Finite Element Methods Simulation of BK-7[®] (25mm diameter and 4 mm thickness) a) simulation and b) temperature rise graph at 7.2 mm graph in COMSOL Multiphysics[®]

A new sample holder is subsequently created using SolidWorks in order to provide a more accurate representation, as seen in Figure 3.9. This newly created sample holder takes into account practical concerns and more closely resembles the actual experimental setup. The COMSOL Multiphysics[®] tool is then used to build the improved model that reflects the conditions in the real world. As depicted in Figure 4.11.a), the final model, which more accurately depicts the actual setup, enables a thorough examination of the temperature distribution and behavior (as seen in 4.11.b)). It is discovered that the outcomes continue to support the homogeneous model that was previously created. This demonstrates that the simulation is accurate and reliable because the average temperature increase matches the values predicted by the homogenous model. These results give us more assurance that the finite element method is accurate and appropriate for simulating and assessing the heating process inside the experimental setup.

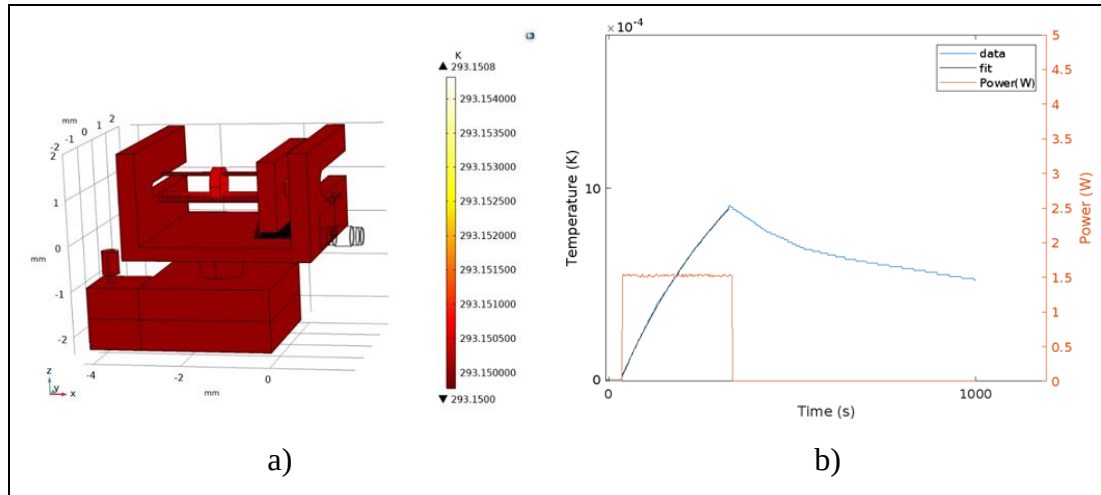


Figure 4.11: Finite Element Methods Simulation of Fused Silica sample (25mm diameter and 6 mm thickness) with the a) new designed holder simulation and b) temperature rise graph at 7 mm graph in COMSOL Multiphysics®

4.3.2. Numerical Simulation

This numerical simulation was primarily conducted as part of section 2.3.1, utilizing an inhomogeneous model of the heat transfer equation. The aim was to investigate the intricate details of the heat transfer process within the BK-7[®] sample. By employing advanced numerical techniques, such as the Finite Element Method, the simulation allowed for a thorough examination of the temperature distribution and behavior.

Figure 4.12.a) and Figure 4.12.b) presents a direct visual comparison of sensor placement and its impact on temperature changes. This graphical representation serves as a fundamental tool for evaluating how well the inhomogeneous model aligns with Finite Element Method Simulations. By carefully examining the similarities and differences in the results, it can be assessed effectively the strengths and limitations of each method. This analytical approach significantly contributes to the validation and refinement of the models used in numerical simulations, enhancing overall understanding of heat transfer dynamics.

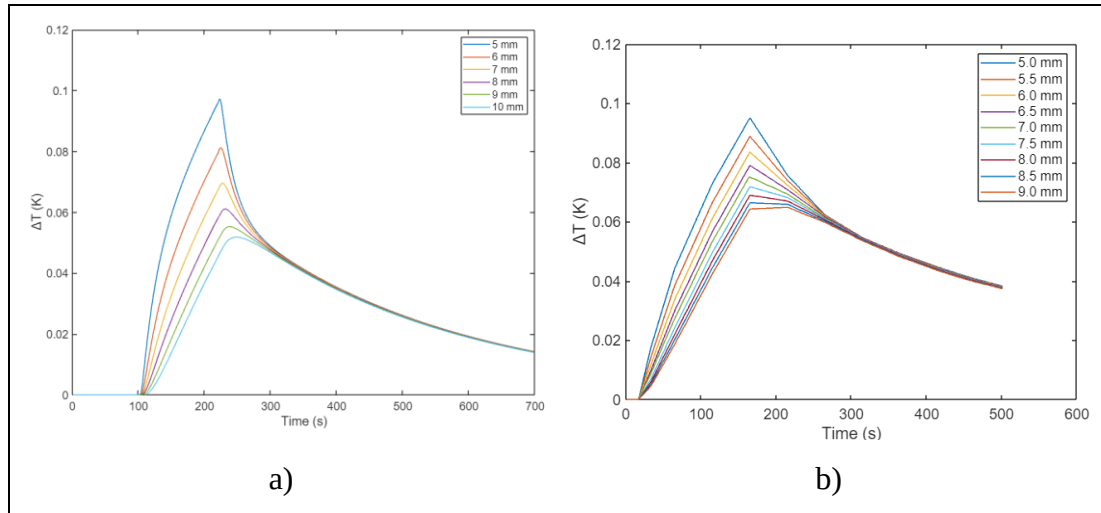


Figure 4.12: A visual comparison of sensor placement and temperature changes of BK-7[®] sample, aiding the evaluation of modeling approaches a) inhomogeneous model and b) COMSOL Multiphysics[®] model.

Simulation parameters for both sensor positions were meticulously calibrated prior to the actual measurements, as depicted in Figure 3.12.b). The outcomes of these simulations and measurements exhibited a high degree of concordance, indicating their robust compatibility.

5. CONCLUSION

This thesis presents a thorough investigation into the measuring of absorption coefficients for high-quality optical components such as BK-7[®], Zerodur[®] and Fused Silica under atmospheric and vacuum conditions. This study intended to improve our understanding of the optical characteristics of these materials at three different laser wavelengths. Several important aspects of this research have provided vital insights and contributions to the field of optical characterization.

Firstly, thorough adjustment of the sample holder design (Figure 3.9) was critical in improving the precision and reproducibility of absorption coefficient measurements. By carefully adjusting the holder arrangement to allow temperature sensors to be scanned across the back end of the sample surface. This dynamic feature added the dimension of temperature gradients to the experiments, allows to characterize the heat transfer process within these optical components more thoroughly. Additionally, this holder configuration addresses scattering effects through the incorporation of a pinhole and blackened aluminum outer shell.

Furthermore, the use of different laser wavelengths (i.e., 532 nm, 640 nm and 1070 nm) provided a stable platform for in-depth study. It is possible to distinguish wavelength-dependent differences in absorption coefficients using our multi-wavelength technique. Table 5.1 presents the evaluation of absorption coefficients for high-quality optical components under atmospheric conditions. Notably, the determination of the optimal absorption coefficient at 640 nm was impeded due to the constrained power output at this wavelength.

Table 5.1: Evaluation of high-quality optical component absorption coefficients in atmospheric conditions.

Optical Component	Wavelength (nm)	Power (W)	Time (min)	Absorption Coefficient (ppm/cm)
BK-7 [®]	532 / 640 / 1070	1 / 0.5 / 10	5 / 2 / 3	2950 / 1630 / 3150
Zerodur [®]	1070	10	3	9000
Fused Silica	532 / 1070	1 / 10	5 / 3	121 / 38

As illustrated in Table 5.2, the absorption coefficients of high-quality optical components under vacuum conditions are notably higher compared to those observed under atmospheric conditions. This discrepancy can be attributed to several primary factors.

Table 5.2: Evaluation of high-quality optical component absorption coefficients in vacuum conditions.

Optical Component	Wavelength (nm)	Pressure (mbar)	Power (W)	Time (min)	Absorption Coefficient (ppm/cm)
BK-7 [®]	532	4e-6	1	8	10800
Fused Silica	532	1e-5	1	8	620

Primarily, in vacuum conditions materials undergo desorption, where gases or molecules previously absorbed or adhered to the optics are released. Desorption depending on the rate could cause changes in absorption coefficient.

Furthermore, in the presence of gases, vacuum conditions reduce convective and conductive heat transfer processes. Because of the restricted heat transmission, sample temperatures can rise during irradiation, affecting the absorption coefficients. Heat dissipation owing to convective cooling can impact temperature profiles under atmospheric conditions, resulting in reduced absorption coefficients.

Moreover, the introduction of scattering from vacuum windows significantly disrupts the precision of measurements. Additionally, achieving adequate temperature sensor contact with the sample becomes more challenging in vacuum conditions due to the reduced thermal conductivity of the surrounding environment.

To summarize, our research resulted in the successful design and fabrication of two laser calorimetry devices capable of operating in both vacuum and atmospheric environments. These systems, which are equipped with three different lasers, have been carefully created in the laboratory, achieving an excellent precision level with an error margin of less than 5%.

Furthermore, this effort included detailed thermal modeling of these systems, which included seamlessly integrating Finite Element Method (FEM) and numerical simulations to assure accuracy and dependability. Furthermore, as shown in Appendix B, the development of sophisticated software for data gathering, system control, analysis, and reporting has been an important achievement within the scope of this thesis.

REFERENCES

- [1] Einstein A., (1972), “On the quantum theory of radiation,” in *Laser Theory*, F. A. Barnes, 5–21, IEEE Press, New York.
- [2] ISO 15368 (2021), “Optics and photonics: Measurement of reflectance of plane surfaces and transmittance of plane parallel elements”, International Organization for Standardization, Switzerland.
- [3] ISO 15368 (2001) (E), “Optics and optical instruments-measurement of reflectance of plane surfaces and transmittance of plane parallel elements,” International Organization for Standardization, Switzerland.
- [4] ISO 13142 (2015), “Electro-optical systems -- Cavity ring-down technique for high-reflectance measurement”, International Organization for Standardization, Switzerland.
- [5] ISO 11551 (2019), “Optics and optical instruments–Lasers and laser-related equipment – Test method for absorptance of optical laser components, International Organization for Standardization, Switzerland.
- [6] ISO 13696 (2022), “Optics and photonics — Test method for total scattering by optical components”, International Organization for Standardization, Switzerland.
- [7] ISO 19962 (2019), “Optics and optical instruments,” International Organization for Standardization, Switzerland.
- [8] Willamowski U., Rista, D., Welsch E., (1998), “Measuring the absolute absorptance of optical laser components.” *Applied optics*, 37(36), 8362-8370.
- [9] Newton I., (1998), “Opticks: or, a treatise of the reflexions, refractions, inflexions and colours of light”. Royal Society of London, 53.
- [10] Frenkel J., (1931), “On the transformation of light into heat in solids”. I. *Physical Review*, 37(1), 17.
- [11] Lambert J.H., (1760), “Photometry, or, on the measure and gradations of light intensity, colors, and shade.” Augsburg, (Germany).
- [12] Beer (1852), "Bestimmung der Absorption des rothen Lichts in farbigen Flüssigkeiten" [Determination of the absorption of red light in colored liquids]. *Annalen der Physik und Chemie* (in German). 162 (5): 78–88.
- [13] France P. W., (2012), “Fluoride glass optical fibres.” Springer Science & Business Media.

- [14] Tan G. L., Lemon M. F., Jones D. J., French R. H. (2005), "Optical properties and London dispersion interaction of amorphous and crystalline SiO₂ determined by vacuum ultraviolet spectroscopy and spectroscopic ellipsometry." *Physical Review B*, 72(20), 205117.
- [15] Griscom D. L., (1991), "Optical properties and structure of defects in silica glass." *Journal of the Ceramic Society of Japan*, 99(1154), 923-942.
- [16] Davis S. P., Abrams M. C., Brault J. W., (2001), "Fourier Transform Spectrometry", in *Fourier Transform Spectrometry* Academic Press, San Diego.
- [17] Griffiths P. R. , de Haseth J. A. (2006), "Fourier Transform Infrared Spectrometry", in *Fourier Transform Infrared Spectrometry* John Wiley & Sons, Inc., 1.
- [18] Prasad N., (2008), "Laser surface thermal lensing (STL): An optical method for surface analysis of glass transition effects in transparent polymers."
- [19] Hussain B., (2005), "Investigation of the optical, thermal, and mechanical properties of a bulk polymer using the surface thermal lensing technique."
- [20] Ke K., Chen J., Gao F., Zhang X., Yuan X., (2020), "Study of UV repetition laser-induced absorption on fused silica surface using a surface thermal lensing technique." *Optics Letters*, 45(8), 2379-2382.
- [21] Chow R., Taylor J. R., Wu Z., Krupka R., Yang T. L., (1997), "High reflector absorptance measurements by the surface thermal lensing technique." In *Laser-Induced Damage in Optical Materials: 1996* (2966, 354-361). SPIE.
- [22] Skvortsov L. A., (2013), "Laser photothermal spectroscopy of light-induced absorption." *Quantum Electronics*, 43(1), 1.
- [23] Alexandrovski A., Fejer M., Markosian A., Route R. (2009), "Photothermal common-path interferometry (PCI): new developments." In *Solid State Lasers XVIII: Technology and Devices* (7193, 79-91). SPIE.
- [24] Bialkowski, S. E. (1996), "Photothermal spectroscopy methods for chemical analysis" (177). John Wiley & Sons.
- [25] Steinlechner J., Martin I. W., Bell A., Cole G., Hough J., Penn S., Steinlechner S. (2015), "Mapping the optical absorption of a substrate-transferred crystalline AlGaAs coating at 1.5 μm ." *Classical and Quantum Gravity*, 32(10), 105008.
- [26] Tam, A. C., (1986), "Applications of photoacoustic sensing Techniques." *Reviews of Modern Physics*, 58(2), 381.
- [27] Waasem N., Fieberg S., Hauser J., Gomes G., Haertle D., Kühnemann F., Buse, K. (2013), "Photoacoustic absorption spectrometer for highly transparent dielectrics with parts-per-million sensitivity." *Review of Scientific Instruments*, 84(2).

- [28] Cotirlan C., Logofatu C., Rizea A., Lazarescu M. F., (2011), "Angle-resolved evanescent-wave cavity ring-down spectroscopy for thin film-solid interface characterization." *Optoelectronics and Advanced Materials-Rapid Communications*, 5(7), 709-714.
- [29] Kessels W. M. M., van de Sanden M. C. M., (2003), "Thin Film Cavity Ringdown Spectroscopy (tf-CRDS)."
- [30] Engeln R., von Helden G., van Roij A. J., Meijer G., (1999), "Cavity ring down spectroscopy on solid C 60." *The Journal of chemical physics*, 110(5), 2732-2733.
- [31] O'Keefe A., Deacon D. A. (1988), "Cavity ring-down optical spectrometer for absorption measurements using pulsed laser sources." *Review of scientific instruments*, 59(12), 2544-2551.
- [32] Aarts I. M. P., Hoex B., Smets A. H. M., Engeln R., Kessels W. M. M., Van de Sanden, M. C. M., (2004), "Direct and highly sensitive measurement of defect-related absorption in amorphous silicon thin films by cavity ringdown spectroscopy." *Applied physics letters*, 84(16), 3079-3081.
- [33] Grishchenko I. V., Vorob'ev A. K., Ostapiv A. Y., Stirmanov Y. S., Konyashkin A. V., Ryabushkin O. A., (2022), "Measurements of small optical absorption and scattering coefficients of high-power polarised laser radiation in lithium triborate crystals." *Quantum Electronics*, 52(3), 301.
- [34] Gospavic R., Sreckovic M., Popov V., Todorovic G., (2006), "3D modeling of material heating with the laser beam for cylindrical geometry." *Mathematical and computer modelling*, 43(5-6), 620-631.
- [35] Hahn D. W., Özisik M. N. (2012), "Heat conduction". John Wiley & Sons, 3.
- [36] Willamowski U., (1999), "Standardisierbare Verfahren zur präzisen Charakterisierung des Absorptions-, Reflexions-und Transmissionsgrads optischer Komponenten."
- [37] Wang Y., Li B., (2011), "Accurate temperature model for absorptance determination of optical components with laser calorimetry." *Applied Optics*, 50(9), C264-C273.
- [38] Willamowski U., Gross T., Ristau D., Welling H., (1996), "Calorimetric measurement of optical absorption and transmissivity with sub-ppm sensitivity." In *Specification, Production, and Testing of Optical Components and Systems* (2775, 148-158). SPIE.
- [39] Becker H. W., Scheuer V., Tschudi T. T., (1994), "Low-power laser calorimetry with high resolution. In *Optical Interference Coatings*" (2253, 1152-1161). SPIE.

- [40] Salazar A., (2003), "On thermal diffusivity". European journal of physics, 24(4), 351.
- [41] Willer Y., Hao L., Balasa I., & Ristau D., (2017), "Calibration accuracy of laser calorimetry for common crystal geometries." In Laser-Induced Damage in Optical Materials 2017 (10447, 158-166). SPIE.
- [42] Willamowski U., Gross T., Ristau D., Welling H., (1996), "Calorimetric measurement of optical absorption at 532 nm and 1064 nm according to ISO/FDIS 11551." In Third International Workshop on Laser Beam and Optics Characterization (2870, 483-494). SPIE.
- [43] Decker D. L., Temple P. A., (1977), "The design and operation of a precise, high sensitivity adiabatic laser calorimeter for window and mirror material evaluation." Glass, AJ; Guenther, AH, ed. In Proceedings of the 9th annual symposium on optical materials for high power lasers (4-6).
- [44] Temple P. A., (1984), "Experimental and theoretical considerations in thin-film laser calorimetry." Optical Engineering, 23(3), 326-330.
- [45] Abdulagatov I. M., Emirov, S. N. Tsomaeva, T. A., Gairbekov K. A., Askerov S. Y., Magomedova N. A., (2000), "Thermal conductivity of fused quartz and quartz ceramic at high temperatures and high pressures." Journal of Physics and Chemistry of Solids, 61(5), 779-787.
- [46] Pérez M. V., (2014), "New concepts and techniques for the development of high-efficiency concentrating photovoltaic modules" Doctoral dissertation, Universidad Politécnica de Madrid.

BIOGRAPHY

Can GÖZÖNÜNDE successfully completed the Physics Engineering Department at Istanbul Technical University, Faculty of Science and Letters, which he began in 2016, in 2021. In the same year of his graduation, he started his master's studies in the Gebze Technical University, Institute of Science and Technology, Department of Physics. Since 2023, he has been working as a researcher at TÜBİTAK UME's Thermodynamics Metrology Laboratory.

APPENDICES

Appendix A: Publications Made in the Scope of Thesis Study

GÖZÖNÜNDE C., ŞENDOĞDU D., NASİBLİ H., ECEVİT F. N., (2022),
“Ultra-Low level Optical Absorption Coefficient Measurements with Laser
Calorimetry” 6. GTÜ Lisansüstü Araştırmalar Sempozyumu

GÖZÖNÜNDE C., ŞENDOĞDU D., NASİBLİ H., ECEVİT F. N., (2022),
“Lazer Kalorimetri ile Yüksek Kaliteli Optik Camların Soğurma Katsayısının
Ölçümü” 23. Fotonik Çalıştayı.

Appendix B: Software Written for This Thesis

The real-time data acquisition and system control software interface, as shown in Figure B1.1, provides a wide range of functionalities to facilitate experimental procedures. In addition to its capacity to accurately control the shutter mechanism, this comprehensive program serves as a versatile tool for experimental management.

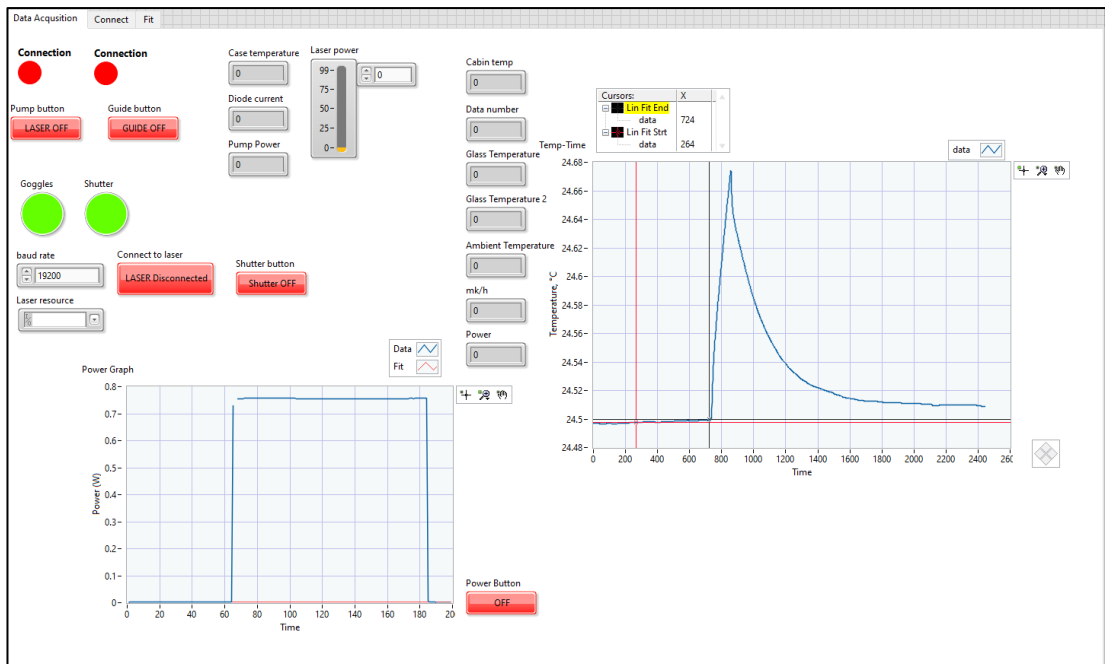


Figure B1.1: The real-time data acquisition and system control software interface

Alongside its various functions, the software offers the ability to record power levels, allowing meticulous monitoring of laser output. It also provides control over the laser source and PID control of the temperature-controlled chamber. In addition, the application simplifies the recording and storage of essential experimental data in real time.

This software exhibits a sophisticated ability for using advanced fitting methods in addition to its essential duties in data collecting and system control as shown in Figure B1.2. Furthermore, the software goes beyond data manipulation by creating complete reports that meet the demanding ISO 11551 criteria as shown in Figure N1.3. These reports provide structured and compliant documentation of the experimental processes, results, and analyses, ensuring transparency and traceability in compliance with industry and regulatory requirements.

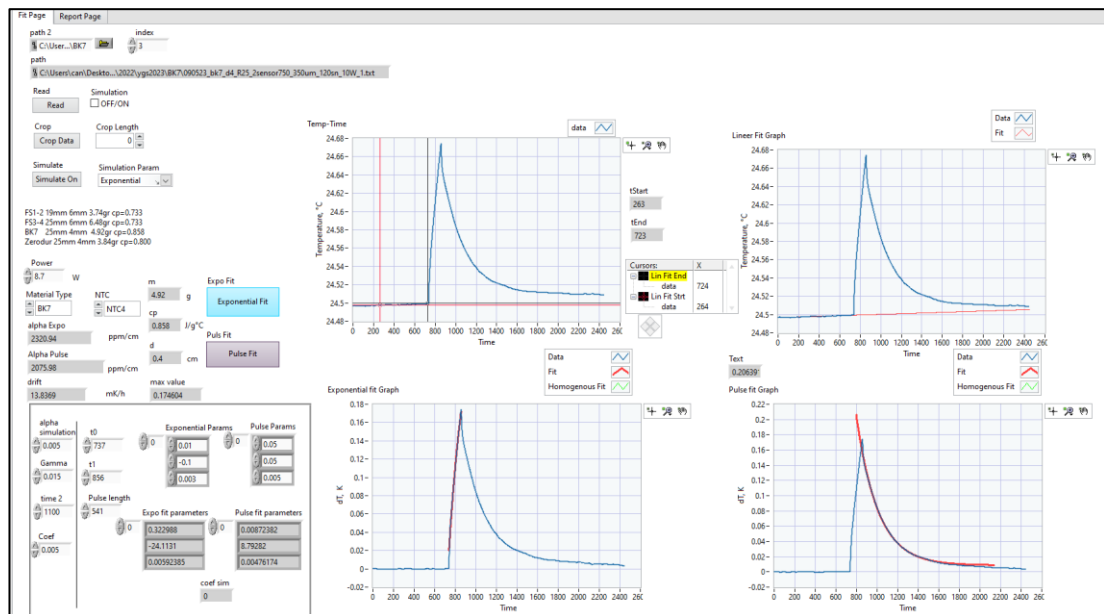


Figure B1.2: Data analysis and report interface

