

Article

Fluorescence Detection-Based Rapid Non-Destructive Testing of Large-Aperture Fused Silica Glass Components

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Featured Application

This work proposes an efficient, non-destructive detection protocol for absorptive defects in the surface layer of large-aperture fused silica optical components, offering practical support for optimizing processing techniques and mitigating laser-induced damage susceptibility in high-power laser systems. The integration of absorptive defect-specific fluorescence response screening with PTWA verification establishes a robust defect detection framework, contributing to enhanced reliability and performance of optical materials in high-power laser systems.

Abstract

As precision polishing and post-processing advance, surface-layer absorptive defects in fused silica optics increasingly show random distribution, low quantity, and ultra-low concentration—making efficient, non-destructive inspection of large-aperture components challenging. In this study, fused silica samples made by conventional ring polishing and acid cleaning were analyzed using photothermal weak absorption (PTWA), micro-X-ray fluorescence (μ -XRF), micro-X-ray diffraction (μ -XRD), and ultraviolet fluorescence microscopy spectroscopy. Results show that process-related contaminants emit strong spontaneous fluorescence between 500 and 620 nm under 375 nm ultraviolet (UV) excitation. Using this optical signature, a high-throughput detection system was developed that combines rapid fluorescence imaging for screening with PTWA for verification. Full-area scanning of a 100 mm $\times$ 100 mm sample successfully identified absorptive defects with significantly improved efficiency over conventional methods. This work provides a practical quality control solution for large-aperture fused silica optics and supports process optimization to reduce laser damage risks in high-performance systems.



Academic Editor: John Xiupu Zhang

Received: 3 February 2026

Revised: 2 March 2026

Accepted: 5 March 2026

Published: 10 March 2026

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Keywords: fused silica optical components; fluorescence spectroscopy; photothermal weak absorption detection; non-destructive testing

1. Introduction

Fused silica materials, owing to their outstanding physicochemical properties—particularly their superior optical performance—are widely utilized in the fabrication of optical components for high-power laser systems. However, the manufacturing process of fused silica optics is complex, and surface contamination, such as residual polishing slurries and

metallic impurities, is commonly observed [1–4]. These absorptive defects can absorb laser energy at operational wavelengths, leading to a significant reduction in the laser-induced damage threshold (LIDT) and premature component failure before reaching the theoretical intrinsic limit. This phenomenon critically undermines the performance stability and operational reliability of optical systems.

With advancements in conventional precision polishing and wet chemical etching post-processing techniques, the quantity and concentration of surface defects on fused silica components have been significantly reduced, thereby forming a distribution pattern characterized by extremely low occurrence frequency and spatial sparsity. Despite this progress, effective detection techniques for identifying such low-level absorptive defects remain under development. Conventional methods such as chemical etching [5,6] and cross-sectional microscopy [7–9] are straightforward and cost-effective but inherently destructive, causing irreversible sample damage and requiring prolonged processing times. Contact-based surface profilometry [10,11], although offering broad measurement range, high resolution, and good repeatability, risks mechanically damaging the surface during scanning, thereby distorting true topography and introducing artifacts into measurements. Currently, non-destructive testing (NDT) approaches are still in the exploratory stage. Total internal reflection microscopy (TIRM) [12–14], which integrates total internal reflection with scattering imaging and digital image processing, enables visualization and sizing of subsurface defects. However, due to the limited depth of focus inherent to optical microscopy, its application to large-aperture components necessitates extensive image tiling, rendering the inspection process laborious and time-intensive. Alternatively, the PTWA technique [15–18] allows non-contact, non-destructive evaluation of surface and near-surface defects; However, its point-by-point measurement mode covers only a microscopic area (on the order of tens of micrometers), and this results in prohibitively low detection efficiency, rendering it impractical for implementation, when applied to large-scale components (e.g., $>100\text{ mm} \times 100\text{ mm}$). Consequently, there remains an urgent need for a detection methodology that combines high efficiency with non-destructiveness for practical engineering applications.

In this paper, based on spectral characterization of fused silica glass samples fabricated using conventional ring polishing and acid-washing processes, we propose an efficient and non-destructive detection approach. The method exploits the characteristic spontaneous fluorescence emission of absorptive defects in the ultraviolet spectral region, combined synergistically with the principle of PTWA. This integrated strategy offers a promising solution for rapid inspection of large-aperture fused silica optical components and provides valuable feedback for optimizing fabrication processes.

2. Materials and Methods

All fused silica samples employed in this study were supplied by Shanghai Hengyi Optical Precision Machinery Co. Ltd., Shanghai China, and subjected to multiple cycles of synergistic annular polishing and acid cleaning processes. Their nominal dimensions are $20\text{ mm} \times 20\text{ mm} \times 5\text{ mm}$ and $100\text{ mm} \times 100\text{ mm} \times 30\text{ mm}$, respectively. The root-mean-square surface roughness (R_q) of all specimens was measured to be below 0.7 nm . The spatial coordinates of absorptive defects on the sample surfaces were determined via point-by-point scanning using the PTWA technique. Measurements were conducted using a small-aperture photothermal weak absorption instrument equipped with a 355 nm pump laser, operating in reflection mode, with a focused spot diameter of $20\text{ }\mu\text{m}$ and a system sensitivity exceeding 50 ppm .

To investigate the elemental composition and crystalline phase of absorptive defects, $\mu\text{-XRF}$ spectroscopy and $\mu\text{-XRD}$ were employed for localized characterization of defect

regions. Microscopic UV fluorescence spectroscopy was employed to acquire the spontaneous fluorescence spectral characteristics of absorptive defects upon UV excitation. LIDT testing was carried out under 355 nm irradiation with a pulse duration of 7.6 ns, using an irradiated spot area of 0.33 mm², to assess the laser damage resistance performance of the samples.

3. Results and Discussion

3.1. Absorptive Defects and Their Fluorescence Characteristic Responses

3.1.1. Spatial Localization of Absorptive Defects

Laser-induced damage in fused silica optical components is primarily attributed to surface defects that exhibit anomalous optical absorption at the operational wavelength [1]. In the case of continuous-wave or long-pulse laser irradiation, absorptive defects on fused silica surfaces absorb significantly more energy than the surrounding material under high-flux conditions. Prolonged exposure leads to localized heat accumulation, resulting in temperature elevation and thermal deformation. When the induced thermal stress exceeds the tensile strength of fused silica, microcracking or fracture initiates, giving rise to initial damage that may propagate under continued irradiation. Furthermore, if the absorbed energy is sufficient to raise the local temperature beyond the melting point of fused silica, localized melting or ablation can occur, thereby inducing irreversible damage. Thus, scanning the sample surface at a wavelength of 355 nm to characterize weak photothermal absorption properties enables effective identification of the locations of absorptive defects and their corresponding absorption intensities.

The PTWA detection system employed in this study used a 355 nm laser as the pump source and a 632 nm laser as the probe beam, performing point-by-point scanning on fused silica samples. The results are shown in Figure 1. In the two-dimensional photothermal weak absorption distribution map, the artificial indentation is designated as O(0, 0; N), where N represents its absorption coefficient (unit: ppm). High-absorption points or regions are labeled as P(x, y; M), with x and y denoting spatial coordinates relative to the origin O (unit: mm), and M indicating the corresponding absorption coefficient (unit: ppm). The PTWA signal in defect-free regions reflects the baseline response of the sample to the pump laser. In contrast, local regions containing absorptive defects exhibit enhanced absorption of the 355 nm pump light, inducing thermal expansion and refractive index variations. These effects lead to significant deviations in the detection signal, characterized by a marked increase in measured values relative to the surrounding defect-free regions.

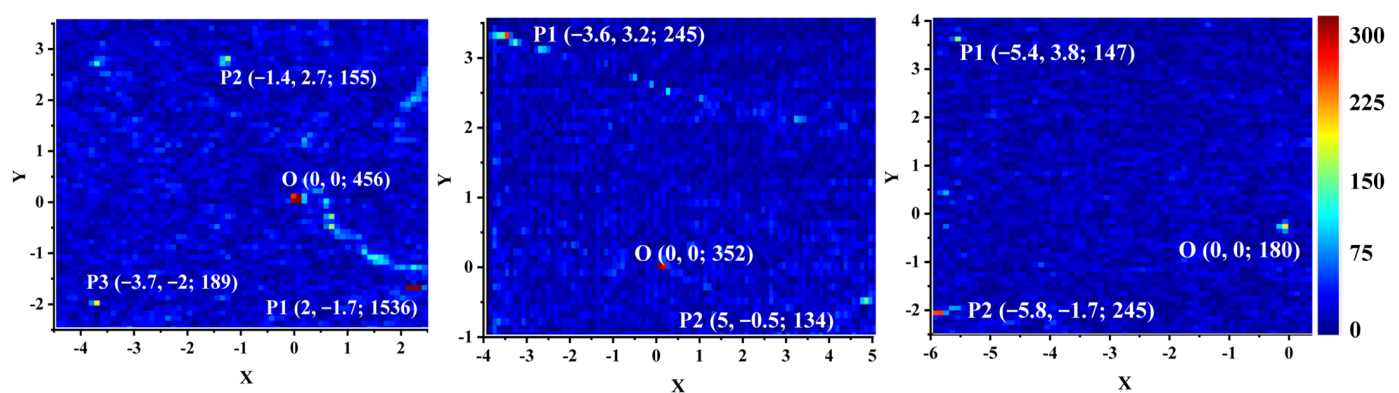


Figure 1. Two-dimensional distribution of weak photothermal absorption on the surface of fused silica glass samples after traditional ring polishing and acid cleaning processes; the characterization was performed using a small-aperture photothermal weak absorption instrument equipped with a 355 nm pump laser, with a focused spot diameter of 20 μ m.

3.1.2. Characteristic Spectra of Absorptive Defects

The composition of absorptive defect regions on the sample surface was analyzed using μ -XRD (Figure 2) and μ -XRF (Figure 3). μ -XRD analysis revealed the absence of distinguishable crystalline diffraction peaks in both the absorptive defect regions and adjacent defect-free regions. μ -XRF results demonstrated that only the matrix elements Si (100.00 wt%) were detected in defect-free regions; conversely, in all absorptive defect sites, foreign elements including Ce, Fe, and Br were identified alongside Si. Notably, Si exhibits the highest mass fraction of 99.5433 wt%, whereas the mass fractions of Ce, Fe, and Br are determined to be 0.2370 wt%, 0.1823 wt%, and 0.0375 wt%, respectively. These impurities, primarily derived from process residues introduced during the fabrication of fused silica optical components (e.g., polishing fluid additives, acid cleaning reagents, and environmental particle deposition), represent one of the key triggers for the enhancement of localized optical absorption [2].

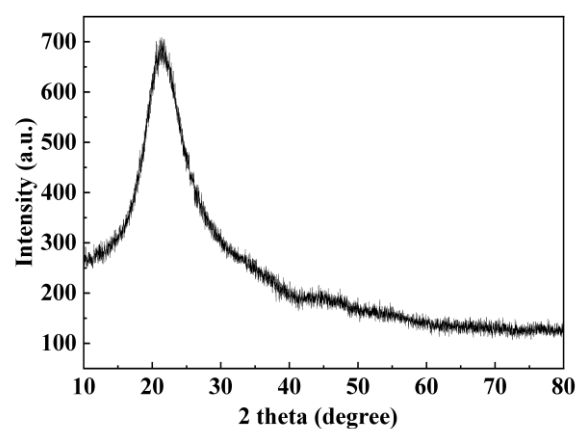


Figure 2. Micro-XRD analysis of surface absorptive defect regions in fused silica samples.

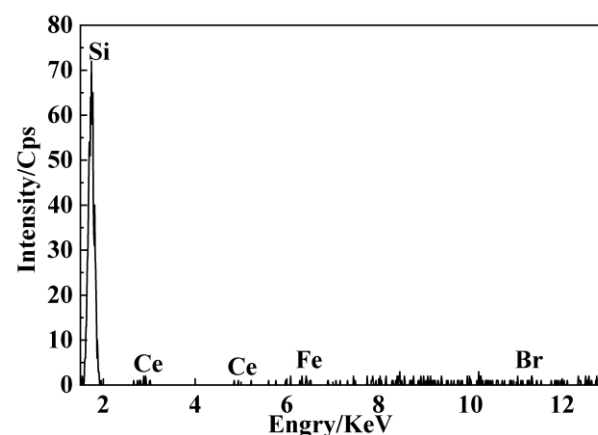


Figure 3. Micro-XRF analysis of surface absorptive defect regions in fused silica samples.

As illustrated in Figure 4, this study performed UV fluorescence spectral characterization on the polishing slurry used for fused silica samples and the corresponding polished sample surfaces. The red line denotes the fluorescence emission spectrum of the polishing slurry, while the black line corresponds to the fluorescence response signal of absorptive defects on the sample surface. Under 325 nm laser excitation, three distinct fluorescence peaks were observed on the fused silica sample surface, centered at 410 nm, 515 nm, and 575 nm, respectively. The peak around 410 nm is attributed to oxygen-containing intrinsic defects in fused silica, such as non-bridging oxygen hole centers (NBOHCs) [19,20]. The 515 nm peak is strongly associated with hydrogen-related defects: owing to the small atomic radius

and high diffusivity of hydrogen, it readily bonds with broken bonds or vacancies in the silicate network, forming structures like Si–H and Si–OH, which give rise to this characteristic green emission band. These defects are also classified as intrinsic defects of fused silica [21]. Notably, the broad emission band near 575 nm is prevalent across all samples with absorptive defects, and its peak shape, intensity, and spectral position are highly consistent with the fluorescence spectrum of the polishing agent—this clearly indicates residual contaminants introduced by the conventional annular polishing-acid cleaning process. Through comparative analysis of the fluorescence spectra of surface absorptive defects and the fluorescence features of the polishing agent, this work established the 500–620 nm band as the characteristic response window for identifying processing-induced surface absorptive defects.

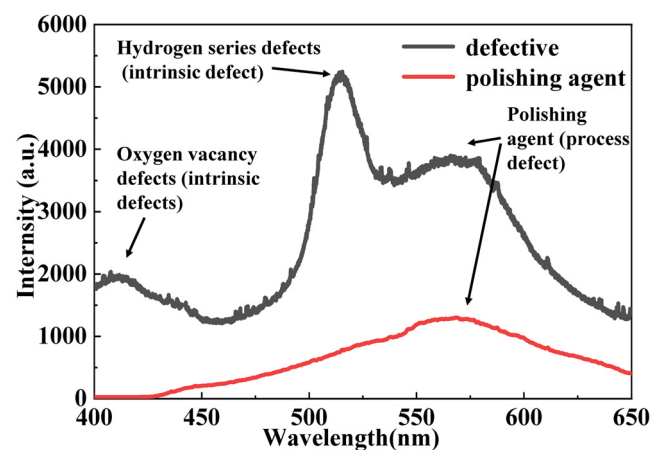


Figure 4. Comparison of UV fluorescence emission spectra between absorptive surface defects of fused silica optical components and the employed polishing slurry. The red line denotes the characteristic fluorescence emission spectrum of the polishing slurry, while the black line corresponds to the characteristic fluorescence response signal generated by surface absorptive defects under 325 nm UV excitation.

To further validate the applicability of this characteristic spectral range, micro-UV fluorescence spectroscopy was conducted on multiple known absorptive defect regions and adjacent defect-free areas under 375 nm excitation. As shown in Figure 5, all defect sites exhibited multiple fluorescence peaks within the 500–620 nm range, with significantly higher intensities compared to non-defective regions. Notably, the fluorescence intensity of defect-free regions exhibits a global minimum at ~560 nm. Concurrently, at the two characteristic wavelengths of 520 nm and 600 nm, the fluorescence response intensity of absorptive defect regions is significantly higher than that of their corresponding adjacent defect-free counterparts. These findings collectively demonstrate the excellent contrast and diagnostic specificity of the 500–620 nm band for surface defect identification in fused silica optical components. Compared to the spectrum obtained under 325 nm excitation, the broad peak originally centered at 575 nm exhibits a redshift to the 585–600 nm range. This phenomenon is ascribed to the Stokes shift effect induced by the redshift of the excitation wavelength.

Fluorescence microscopy imaging experiments were performed on the samples. The excitation source employed was a 375 nm central-wavelength UV LED, and the imaging band of the camera was set to the 500–620 nm range. As demonstrated in Figure 6, UV fluorescence imaging clearly resolves absorptive defects on the fused silica surface, outperforming conventional bright-field microscopy. Moreover, absorptive defects beneath the sample surface were successfully detected using this method (see the red-circled regions in Figure 6). Notably, although surface dust particles emitted detectable fluorescence,

they did not exhibit concurrent photothermal weak absorption anomalies. Therefore, to ensure reliable identification of true absorptive defects, candidate regions identified via fluorescence characteristics must be corroborated through subsequent photothermal weak absorption measurements.

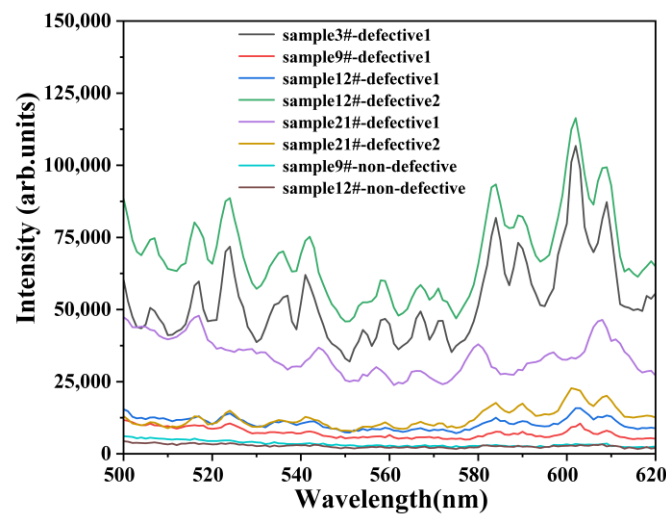


Figure 5. Microscopic UV fluorescence emission spectra of absorptive surface defect regions and adjacent defect-free regions on fused silica optical components (excitation wavelength: 375 nm; detection band: 500–620 nm). All absorptive defect-containing regions exhibit significantly enhanced fluorescence responses, with intensities substantially higher than those of their corresponding adjacent defect-free regions.

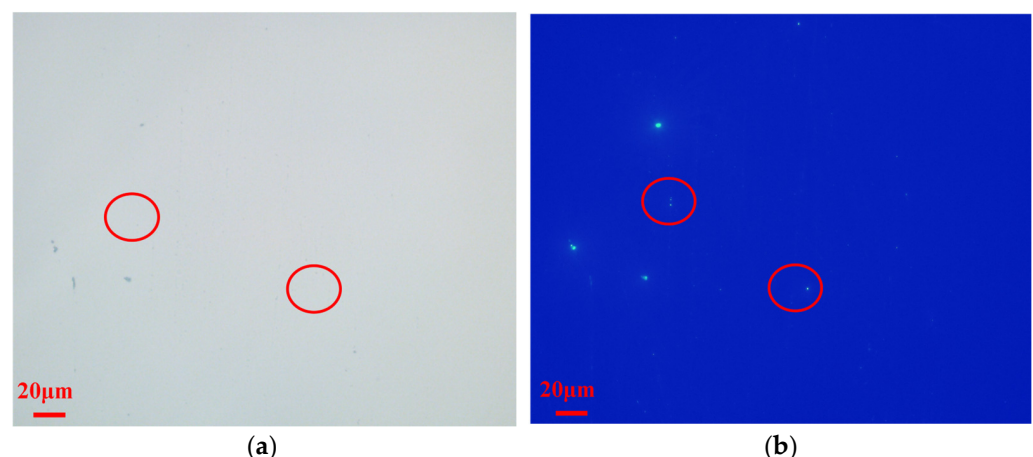


Figure 6. Micro-UV fluorescence imaging results of fused silica samples: (a) conventional white-light microscopy image; (b) micro-UV fluorescence image.

3.2. Rapid Non-Destructive Testing System for Large-Aperture Fused Silica Optical Components

By exploiting the characteristic fluorescence response of absorptive defects in fused silica within the UV spectral range, a scanning microscopic fluorescence imaging system was developed to achieve high-efficiency localization of fluorescence-emitting sites on the sample surface. Subsequent point-by-point validation of the spatial coordinates associated with these fluorescence signals is performed using a photothermal weak absorption detection system, enabling accurate identification of surface-layer absorptive defects. Thus, a rapid non-destructive testing system for large-aperture fused silica optical components was developed, integrating a fluorescence characteristic imaging module and a photothermal weak absorption detection module. To ensure the high precision and stability of system operation, the displacement platform employs a closed-loop feedback control system based

on a grating ruler: the positioning resolution of the X, Y, and Z axes is 10 nm; the repeat positioning accuracy in the XY plane is better than $\pm 0.5 \mu\text{m}$, and that of the Z axis is better than $\pm 1 \mu\text{m}$. Meanwhile, the system integrates a high-precision auto-focus module, which has a positioning resolution of 0.1 μm and a steady-state repeat accuracy of $\pm 0.5 \mu\text{m}$. The above design synergistically enables sub-micrometer-level spatial relative position control between the sample and the detection probe, ensuring that the overall system spatial alignment accuracy is better than 100 nm (steady-state error).

3.2.1. Fluorescence Feature Spectral Imaging Module

A UV fluorescence scanning imaging module was established based on experimental results, with its optical configuration illustrated in Figure 7. The system employs an upright microscope architecture with coaxial excitation geometry. Excitation light passes through an excitation filter and is reflected by a dichroic mirror before being focused onto the sample surface via an objective lens. Fluorescence emitted from excited absorptive defects retraces the incident path, transmits through the dichroic mirror, and is directed to an imaging camera after passing through an emission filter, thereby generating a fluorescence image. Concurrently, a fraction of the emitted fluorescence is collected by a focusing lens and coupled into an optical fiber for spectral analysis using a fiber-optic spectrometer (Avaspec-ULS2048L-USB2, Avantes, Apeldoorn, Netherlands), allowing acquisition of UV fluorescence spectra from defect regions. The system achieves a field of view of $0.9 \text{ mm} \times 0.9 \text{ mm}$ per measurement point, with an integration time of 100 ms, demonstrating high detection efficiency.

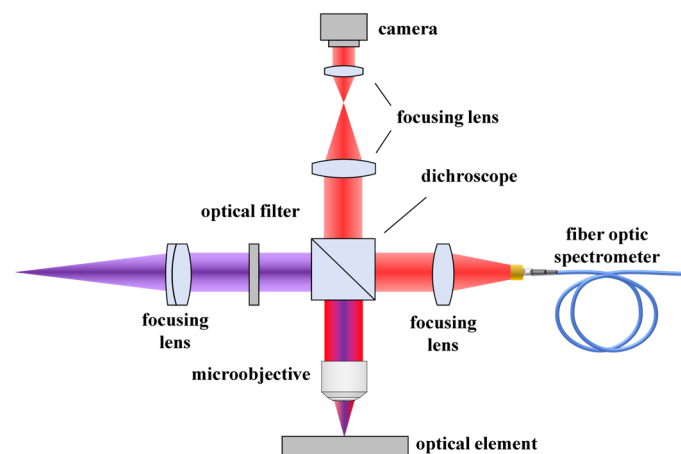


Figure 7. Schematic illustration of the micro-UV fluorescence imaging module in the rapid non-destructive testing system for large-aperture fused silica optical components.

3.2.2. Photothermal Weak Absorption Confirmation Module

As shown in Figure 8, the PTWA detection module consists of two distinct optical paths: the pump light path and the probe light path. A continuous-wave ultraviolet laser (355 nm) serves as the pump source, which is intensity-modulated by a mechanical chopper and subsequently expanded and focused onto the sample surface using a beam expander (Thorlabs, BE05-355, Thorlabs Inc., Newton, MA, USA) and a focusing lens. Upon absorption of photon energy by electronic states in the fused silica substrate, non-radiative relaxation processes transfer energy to the lattice, resulting in localized heating and the formation of a spatially periodic temperature distribution at the surface. This thermal gradient induces periodic nano-scale surface displacements due to thermoelastic deformation. A separate probe beam, generated by a helium-neon (He-Ne) laser (632 nm), is collimated, expanded, and focused onto the thermally modulated region of the sample surface via a reflective mirror, forming a confocal detection spot. During reflection, the

probe beam acquires phase modulation induced by the oscillating surface topography, leading to a periodically varying diffraction pattern in the far field. A pinhole spatial filter selects the diffracted component exhibiting maximum intensity modulation depth, and a photodetector converts this modulated optical signal into a time-varying electrical signal. Finally, the lock-in amplifier extracts the amplitude of the alternating current (AC) component synchronized with the pump modulation frequency, yielding the photothermal signal. The magnitude of this signal is proportional to the amplitude of thermoelastic surface displacement, thereby enabling the measurement of light absorption in defect regions within the surface layer.

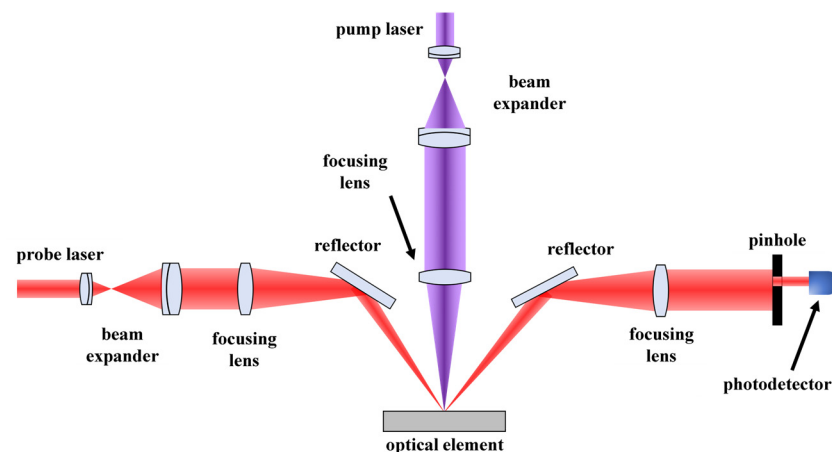


Figure 8. Schematic illustration of the photothermal weak absorption detection module in the rapid non-destructive testing system for large-aperture fused silica optical components.

3.2.3. System Detection Results and Discussion

The aforementioned rapid non-destructive testing system for fused silica glass optical components was employed to characterize absorptive defects in a 100 mm × 100 mm sample. The results are presented in Figure 9a,b, with a total testing duration of 1.5 h. Given the inherent limitation of the photothermal weak absorption measurement method, which features a small single-point detection aperture, theoretical estimates indicate that a full-coverage inspection of a 100 mm × 100 mm fused silica optical component using this method alone would require thousands of hours. Therefore, this study introduces a pre-screening strategy based on the specific fluorescence spectra of absorptive defects, which can significantly reduce the time cost of subsequent high-precision detection. During the measurement, one absorptive defect was identified at coordinates ($X = 25.93$ mm, $Y = 86.50$ mm). Subsequently, the PTWA detection system was employed to validate the suspected absorptive defect region.

Figure 9a presents the characteristic fluorescence image of the defect location, while Figure 9b provides a magnified view of the region. Figure 9c illustrates the spatial distribution map of the PTWA coefficient over the corresponding area. The photothermal measurement was centered on the defect, covering a 0.3 mm × 0.3 mm region with a 31 × 31 sampling grid, achieving sub-micrometer spatial resolution. As shown in Figure 9c, the defect exhibits a significantly elevated absorption signal compared to the background level, confirming its anomalous absorptive behavior. Meanwhile, microscopic UV fluorescence spectroscopy of the region adjacent to the defect was acquired using a fiber optic spectrometer, with the results presented in Figure 10. In comparison to the defect-free region, this region exhibits distinct fluorescence intensity in the 500–620 nm wavelength band, further confirming that it corresponds to an absorptive defect induced by process residues.

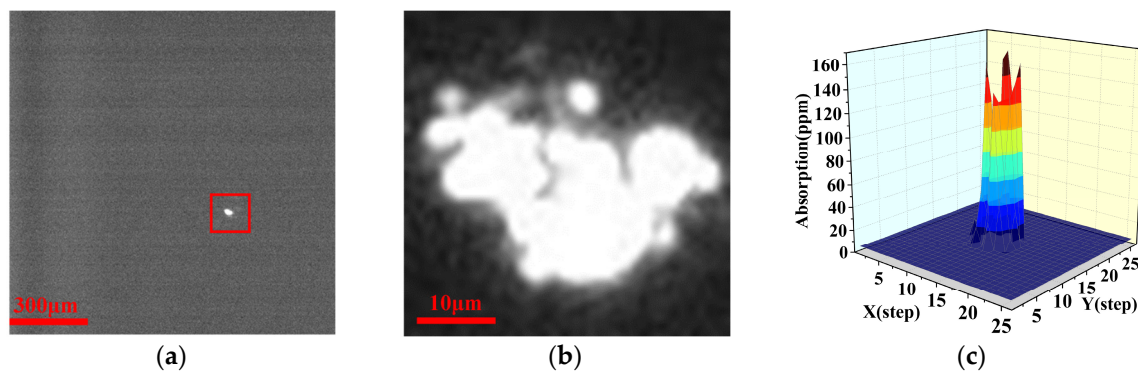


Figure 9. Magnified view and PTWA coefficient distribution map of a suspected defect detected by the rapid non-destructive testing system for large-aperture fused silica optical components ($X = 25.93$ mm, $Y = 86.50$ mm). (a) Micro-UV fluorescence image of the suspected defect; (b) Local magnified view of the defect region; (c) PTWA coefficient distribution map of the defect region.

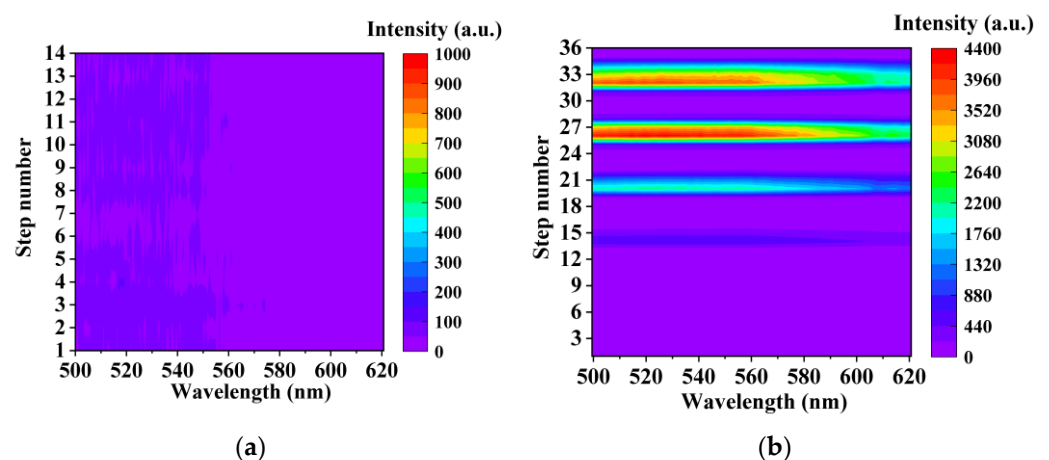


Figure 10. Scanning micro-UV fluorescence spectral heatmap of the absorptive defect region ($X = 25.93$ mm, $Y = 86.50$ mm), with a single-point scanning spot aperture of 0.1 mm. (a) Scanning micro-UV fluorescence spectral heatmap of the normal region; (b) Scanning micro-UV fluorescence spectral heatmap of the region adjacent to the absorptive defect.

To further validate the impact of this absorptive defect on the LIDT of fused silica optical components, LIDT measurements were performed on the defect region. Under laser irradiation conditions of 355 nm wavelength and 7.6 ns pulse width, distinct damage initiation was observed at the defect site when the cumulative fluence exceeded 16.3 J/cm^2 ; in contrast, the LIDT of the absorptive-defect-free region within the same sample reached 50.8 J/cm^2 . The significant discrepancy between these two values demonstrates that the absorptive defect substantially degrades the local laser damage resistance of the component.

This system leverages the specific fluorescence response of absorptive defects under ultraviolet excitation, complemented by quantitative validation of the photothermal weak absorption coefficient, enabling efficient identification of residual absorptive defects on the surface of fused silica optical components fabricated via traditional ring polishing processes. A typical defect has been successfully spatially localized and subjected to multi-modal physical confirmation. However, constrained by the inherent random spatial distribution, extremely low areal density (with typical dimensions ranging from sub-nanometer to single-atom scale) of such defects, as well as the current system's motion control and optical positioning precision (≈ 100 nm), limitations persist in detection sensitivity and spatial resolution for isolated, dispersed nanoscale and even atomic-level absorption centers. This technical bottleneck hinders the comprehensive characterization of trace process-residue-

induced defects, representing a core challenge that demands urgent breakthroughs in subsequent research.

4. Conclusions

This study addresses the critical challenge of efficient detection of absorptive defects in large-aperture fused silica optical components by systematically integrating defect origin analysis, characteristic signature identification, and the development of an integrated detection methodology. A multimodal analytical approach—combining PTWA detection, μ -XRF, and μ -XRD—confirmed the presence of impurity elements (Ce, Fe, Br) within absorptive defects, which are attributable to residual contamination introduced during fabrication processes. Complementary UV micro-fluorescence spectroscopy under 325 nm excitation revealed three distinct emission peaks at 410 nm, 515 nm, and 575 nm, thereby establishing a characteristic fluorescence response in the 500–620 nm wavelength range. Further validation through UV fluorescence imaging under 375 nm excitation demonstrated that this spectral fingerprint enables reliable visualization of surface-layer absorptive defects in fused silica components.

Leveraging this characteristic fluorescence signature, a high-throughput NDT system was developed, integrating a rapid fluorescence screening module with a photothermal revalidation unit. Full-area scanning of a 100 mm $\times$ 100 mm sample successfully identified and confirmed the presence of absorptive defects, demonstrating the system's feasibility. This work presents an efficient and non-destructive detection protocol for large-aperture optical components, offering valuable insights for refining fabrication processes and reducing laser-induced damage susceptibility in high-power laser systems.

Author Contributions: Conceptualization, Z.S. and R.H.; methodology, Z.S.; validation, J.C., L.L. and K.N.; formal analysis, J.C., K.N., Z.S. and R.H.; investigation, J.C., L.L. and K.N.; data curation, J.C., L.L. and K.N.; writing—original draft preparation, J.C. and K.N.; writing—review and editing, J.C. and K.N.; supervision, Z.S. and R.H. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data available on request from the authors. The data that support the findings of this study are available from the corresponding author (K.N.), upon reasonable request.

Conflicts of Interest: The authors declare no conflicts of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

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