

Transmission laser micro-welding of preheated glass substrates

by

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M.Sc., University of Basra, 2009

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AN ABSTRACT OF A DISSERTATION

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DOCTOR OF PHILOSOPHY

Department of Industrial and Manufacturing Systems Engineering
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Abstract

Glass is a widely used material for multiple applications such as building windows, medical implants, solar cell modules, and sensor packaging. In these applications, glass joining is a key process for producing the final functional design, which is traditionally performed by applying or inserting a second material like adhesives or polymer films. Cured adhesives and polymer films can join glass to glass and attain acceptable strength and transparency, however durability of the structure is limited by the material-environment interaction. Cured polymers absorb moisture and change color during service leading to a degradation in the sealed joints that affects the functionality of the respective application.

Laser glass welding is a technique for joining surfaces without using an intermediate layer. Generally, the technology can be used to weld a transparent material to a transparent or non-transparent material such as glass-to-glass, glass-to-ceramic and glass-to-metal. The technique requires focusing and scanning a laser beam at the interface between pre-contacted surfaces to produce a seam weld. Hermetic sealing with a strength comparable to the glass shear strength can be achieved by optimizing laser and scanning process parameters. Laser process parameters are pulse energy, pulse duration, repetition rate, hatch distance and scanning speed. Generally, process parameter combinations fall into two categories: filamentation and accumulation. However, concurrent filamentation and accumulation can also be utilized for laser glass welding but effects of the filamentation may affect the quality of the welded parts. The current research investigates mitigation of the filamentation-related defects utilizing preheating that can also enhance modification topology and welding strength.

This research provides fundamental knowledge and insights into laser glass-to-glass welding using preheated glass substrates by investigating pre-heating welding requirements compared to room temperature conditions.

Since determination of the focal position before laser scanning is a critical requirement, especially when preheating is adopted, a fundamental analytical model is derived from basics of thermo-optical phenomenon for focal position prediction. The model is validated using focal position measurements of accumulation-induced tear-drop experiments under a range of pulse energy, scanning speed, distance between glass and lens, and temperature using femtosecond and picosecond laser pulses. The applicability of the model is investigated using welding experiments under picosecond conditions by introducing a compensation parameter to the analytical model. Temperature effects on the tear-drop modification are investigated under filamentation or self-focusing and accumulation conditions to elucidate temperature enhancement mechanisms by defining teardrop's height (width) gain ratio. At last, effects of preheating on absorptivity rate, shear strength and defects of welded glass slides are explored to optimize the process parameter window and make the technique available for industrial applications.

Predictions of the derived analytical model match the measurements of focal position in the distance of 9.56–9.96 mm at 200 °C where self-focusing is not significant while underestimating the measurements for the distance of 10.76 mm and 10.36 mm by 30–50 μm . Tear-drop modifications at 200 °C show a relaxed discoloration compared to 150 °C and room temperature. It is also found that in the focal range of 1.6 to 3.2 mm and using a 14 μJ pulse energy, the width and height gain are more than 18% and 13%, respectively. At 150 °C, tear-drop's aspect ratio is enhanced by around 25% at a focal position of 1 mm for the pulse energy of 4.5, 6, 10 μJ and speed of 5 mm/s. Welding using optimum conditions of 5 scans and 200 μm hatch produces a

strength of around 50 MPa at RT and 40 MPa at 150 °C for 184 fs compared to 35 MPa at RT and 32 MPa at 150 °C for 10 ps. While preheating mitigates self-focusing discoloration, a lower fracture toughness is expected to be the major reason behind higher strengths for RT welding that needs independent future determination.

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Chapter 1 . Introduction

1.1 Transmission Laser Welding Process

Laser glass welding involves joining two surfaces of glass workpieces with/without an intermediate layer of absorbing material. The laser is shined through the first workpiece, transmitted to the interface between the two workpieces, and absorbed by the intermediate layer if used causing melting and joining of the glass material at the interface [1]. The intermediate layer's material should have a matched coefficient of thermal expansion (CTE) to the glass being welded to avoid cracking by thermal stresses [2]. Selection of the intermediate material is challenging, especially when a wide range of transparent materials are considered for welding. Alternatively, Tamaki et al. developed a glass welding technique without inserting an intermediate layer for the first time in 2005 [3], where two fused silica glasses were welded using a femtosecond laser having 880 nm, 130 fs and 1 kHz of wavelength, pulse duration, and repetition rate, respectively.

Generally, the technology can be used to weld a transparent material to a transparent or non-transparent material [4]. The process is also flexible to weld similar and dissimilar glass substrates like fused silica to fused silica and fused silica to BK7 [5], borosilicate to fused silica, borosilicate to titania-silica binary glass (ULE), and ULE to zerodur [6]. Parameters that determine the processing window and mechanism of glass welding are pulse energy, pulse duration, repetition rate, scanning speed, and wavelength [7]. Laser glass welding without intermediate layer has multiple applications such as MEMS hermetic sealing, sensor packaging for RF medical implants, and welding glass end-caps to optical fibers [8]. Such applications utilize laser welding because it is localized, contactless and does not require additional adhesive material [9]. More details about current practical applications of laser glass welding can be found in Section 2.4. Figure 1.1 shows a schematic of the transmission laser glass welding at the interface between two

contacted glass workpieces. A tear-drop shaped modification is generated during the laser scan inside glass because of the intensity-dependent nonlinear absorption of optical energy [8].

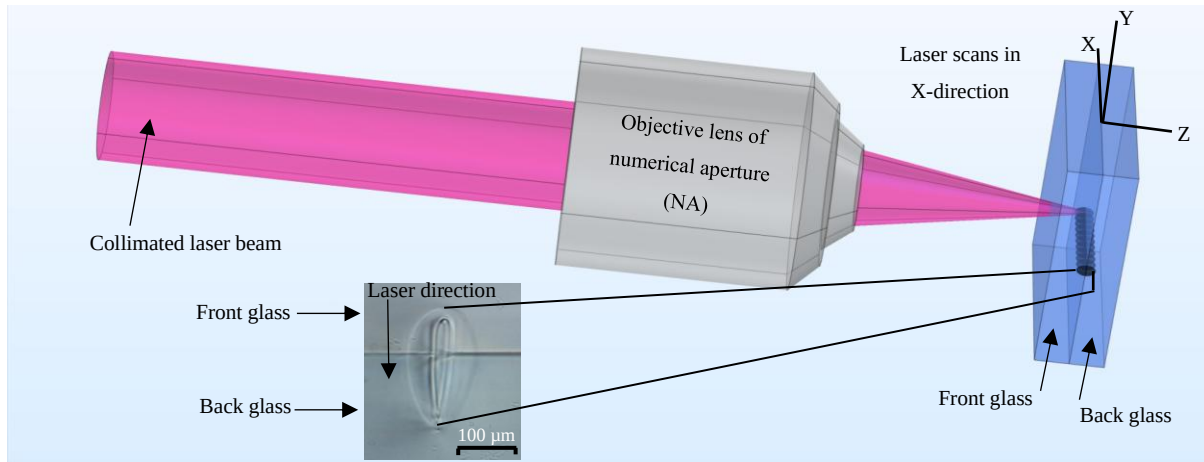


Figure 1.1 Transmission laser glass welding by overlapped tear-drop modifications

1.2 Requirements of Laser Glass Welding

The study of Tamaki revealed that the smaller the pre-welding gap between contacted glass workpieces the better for a successful joining. When a gap of $\frac{1}{4} \lambda$ or 100 nm exists, where λ is the light wavelength prior to welding, an optical contact can be achieved. Non-optical contact laser glass welding is also possible, but welding defects and strength could be affected. Table 1.1 lists reported welding cases of optical and non-optical contact using different glass materials and sample thicknesses. Table 1.1 lists the highest gap that has been bridged by a tear-drop of around 10 μm [10] while the thickest welded glass substrates of 6.4 mm [6].

Additionally, focal position is another critical parameter for successful glass welding, even if full optical contact is not achieved. Successful welding can be achieved when the focal position lies in a welding range, generally when the position is located under the contact surfaces as observed from the front glass sample. Establishing full optical contact is essential, but it is

technically costly even for highly polished surfaces, especially when a large welding area of several square centimeters is required [11].

Table 1.1 Laser glass welding requirements for initial gap, thickness and material

Contact type and pre-weld gap	Material and thickness	Ref.
Optical	Fused Silica-0.8 mm	Tamaki et al. [3]
Optical	Fused Silica-2.3 mm, BK7-1.0 mm	Hélie et al. [5]
Optical	Fused silica, Borosilicate, BK7, ULE, and Zerodur-6.4 mm	Richter et al. [6]
Optical	BK7-1 mm	Kongsuwan e al. [9]
Non-optical (9.82 μm)	Borosilicate-2.0 mm	Chen et al. [10]
Optical and Non-optical (3 μm)	Fused Silica-1.0 mm	Chen et al. [11]
Optical and Non-optical (1 μm)	Soda Lime, NBK7, BF33, D263, and Fused Silica-not specified	Cvecek et al. [12]
Non-optical (5 μm)	Soda lime-2 mm	Jia et al. [13]
Optical	Fused Silica-2.0 mm	Zhao et al. [14]

1.3 Laser Glass Welding Approaches

Without using an intermediate layer, there are two approaches for laser transmission glass joining: filamentation and thermal accumulation. Filamentation-induced welding utilizes self-focusing, which is significant when incident laser peak power exceeds the material's critical peak power of self-focusing. The peak power is calculated by $P_{\text{peak}} = E_p / t_p$, where E_p is the pulse energy and t_p is the pulse duration. Lower repetition rates of a couple of kHz and slow scanning speeds are used for this welding approach to join glass surfaces even when a mismatch in CTE exists because filamentation makes it more tolerant to thermal stresses compared to the accumulation-dependent approach [15]. Thermal accumulation is significant when repetition rate is higher than 200 kHz using a laser intensity exceeding the material's intensity threshold for nonlinear absorption. The intensity is calculated using $I = F / t_p$ where the fluence $F = E_p / (\pi w_0^2)$. The spot size $2w_0$ is calculated using $2w_0 = 2(M^2 \cdot \lambda) / (\pi \cdot \text{NA})$ where $M^2 \geq 1$ is the beam quality factor, λ is the laser wavelength, and NA is the lens numerical aperture. This approach can utilize higher scanning

speeds where a tear-drop modification is generated at the interface between the contacted glass surfaces [12]. While the tear-drop is a localized modification where the focal position controls the modification location (see Figure 1.1), filamentation welding is accompanied by a discoloration that extends into the glass pieces being welded. Figure 1.2 shows cross-sections of two fused silica glasses welded by a filamentation focused at different focal positions where the filamentation discoloration is evident throughout the glass volume. The sample thickness is 2.3 mm and scans A, B, and C are shined using different focal positions. The corresponding interfacial weld modifications have widths of 5.34, 4.27 and 2.14 μm for scans A, B, and C, respectively.

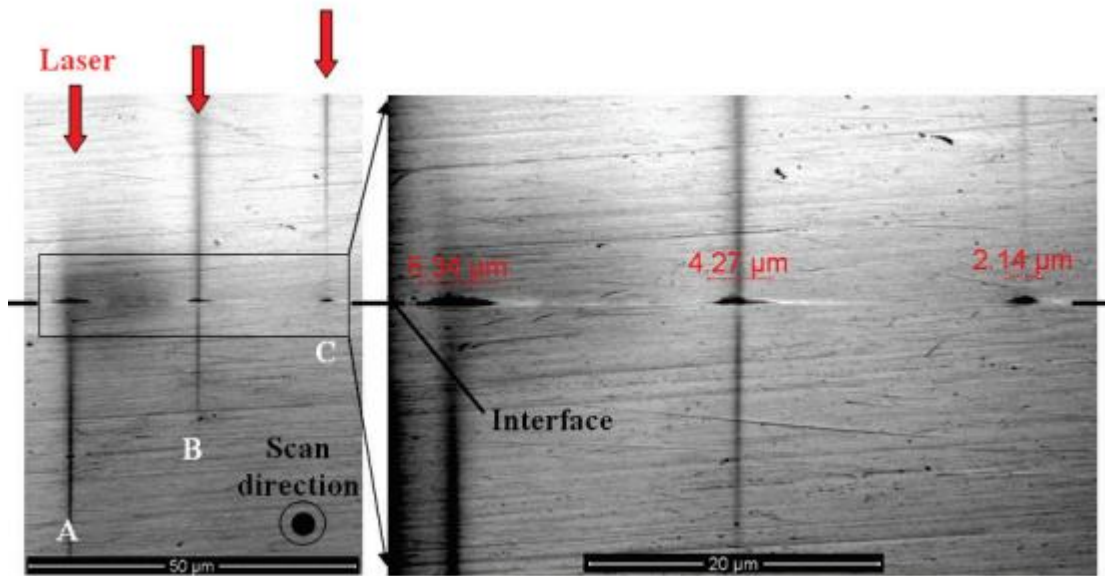


Figure 1.2 Laser fused silica welding by filamentation at different focal positions [5]

When a laser beam is focused at the interface of contacted glasses and both of the incident peak power and intensity exceed the threshold, both approaches take place. However, accumulation effects dominate over filamentation leading to the formation of a tear-drop modification preceded by a discoloration [16].

Laser glass welding requirements are practically challenging to achieve, especially the optical contact. The stochastic nature of the problem rising from uncertainties in sample thickness,

flatness and roughness inhomogeneity have rendered the technique uncommercialized so far. Extending the laser parameter processing window for wider ranges including the self-focusing range provides not only a flexibility in the parameter selection but also can improve welding strength and reduce modification defects. In the current research, introducing a novel preheated glass substrate welding conditions may alleviate the process requirements and extend the parameter window, which requires fundamental analysis of the focal position determination prior to laser scanning.

1.5 Objectives of the Current Work

The goal of this project is to investigate the transmission welding of preheated glass substrates using femtosecond and picosecond pulses utilizing tear-drop modifications' focal position prediction. Specific research objectives include:

- a) To develop an analytical model of focal position prediction under room temperature and high temperature conditions.** Focal position prediction is a critical parameter for controlling the tear-drop modification initiation position to meet the welding requirement of gap bridging at the interface between contacted glass workpieces. Ray optics method of beam propagation is utilized to derive the focal position shift model upon preheating compared to room temperature. An analytical model of spherical aberration effects due to thermal dilatation and refractive index evolution is derived from thermo-optical analysis of a preheated glass slab compared to room temperature. The model can predict the focal position at any temperature up to the limit of the current work of 200 °C for the cases where self-focusing and accumulation are significant.
- b) To investigate tear-drop internal modification morphology and focal position of preheated glass slabs using femtosecond and picosecond laser pulses.** Internal

modifications are generated by scanning a focused laser beam at different focal positions by changing the distance between the lens and the glass surface. Tear-drops are generated using a range of pulse energy and scanning speed for each distance level. An experimental procedure is developed to create visible tear-drops having minimum interaction between adjacent modifications for femtosecond and picosecond cases. In the femtosecond laser case, self-focusing and accumulation conditions occur concurrently, while in the picosecond case, only the accumulation condition occurs.

- c) **To establish welding procedures and fundamental understanding of preheating effects on enhancing tear-drop morphology and welding strength.** Focal position prediction compensated for welding of glass slides at higher temperature is developed. Tear-drop modifications under compensated and non-compensated focal position conditions are created using optimum processing window of pulse energy and scanning speed. The introduction of compensation is to account for the cases where the tear-drop height evolves due to preheating and to keep a corresponding focal position for a successful welding. The fundamental effect of temperature is investigated by monitoring the beam transmission at the wake of preheated glass slides and measuring the transmitted pulse energy at different propagation locations and temperatures under self-focusing conditions. Pulse energy is varied in a range up to glass damage that is determined by far-field power measurements after the glass slides. Lap shear welding strength tests are performed using glass slides welded by different hatching distances. The optimum processing window and hatching distance based on lap shear strength are identified and investigated.

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Chapter 2 . Literature Review of Laser Internal Modification and Glass Welding

Abstract

Exploration of laser internal modification of glass includes investigation of focal position and tear-drop modification evolution for proposed laser process parameters. During this exploration, process parameters are optimized before the actual welding experiments. Focal position determination is a critical step for a successful welding with minimum defects and optimum strength. This paradigm has been implemented by most of the research in the field. Recent studies have focused on accumulation-based picosecond laser welding to avoid the nonlinear effects of filamentation that is likely to happen for femtosecond laser pulses. Additionally, repetition rates in the MHz and GHz ranges have also been utilized, which is far exceeding the accumulation onset of around 100 KHz. In spite of the recent advances in the industrial applications of laser transmission welding, the technique is still not commercialized due to the critical optical contact and process parameter requirements.

2.1 Introduction

Internal modification of glass by a focused pulsed laser beam triggers the multi-photon absorption when the localized incident intensity exceeds the intensity threshold of the glass material. Tear-drop modification evolves locally at the focal position, which is attributed to multiphoton absorption [1] by free electrons generation. These free electrons can move valence band electrons to the conduction band by linear absorption induced avalanche ionization. Laser energy can also be absorbed by the free electrons producing plasma absorption [2] and extending the tear-drop core to form a melted plasma-modified region. When heat diffuses from the plasma-

modified melt to the surrounding glass, a dual-structure tear-drop shaped modification is created by the heat affected zone [3]. Tear-drop height, width and aspect ratio, which is the height to width ratio, depend on the incident laser pulse energy, repetition rate, pulse duration [4], and scanning speed [5].

When the tear-drop modification is located at the interface between contacted glass substrates and due to the localized melting of the plasma-modified region, the glass substrates can be welded by gap bridging [6]. The plasma-modified region plays a significant role in glass welding compared to the heat affected zone of the tear-drop, especially for bigger pre-welding gaps [7]. Welding defects like longitudinal, transverse and tunnel-like cracks, gas bubbles, and stress-induced birefringence can appear when laser processing window parameters are not optimized [8]. However, preheating effects on tear-drop modification height, width and aspect ratio, and welding defects and strength have not been considered in the literature, which is the focus of the current work.

2.2 Tear-drop Modification using Femtosecond and Picosecond Pulses

The tear-drop modification has a distinctive dual-structure of a central core, which is called the plasma-modified region due to electronic damage. Figure 2.1 depicts the tear-drop structure and formation mechanism. The plasma-modified region is initially created by a localized nonlinear absorption at the focal position (highlighted by a yellow-colored object in Figure 2.1). Then, when heat accumulation takes place by the incidence of multiple pulses, linear avalanche ionization extends the plasma column towards the laser source during which laser intensity decreases progressively terminating the formation of the plasma column of length l [9]. The surrounding blue-colored boundary in Figure 2.1 or the HAZ is generated due to thermal diffusion forming a

dual-structure tear-drop. Simulations show that the plasma-modified region has a temperature exceeding 2000 °C, which results in a localized melting of the glass inside the region [10].

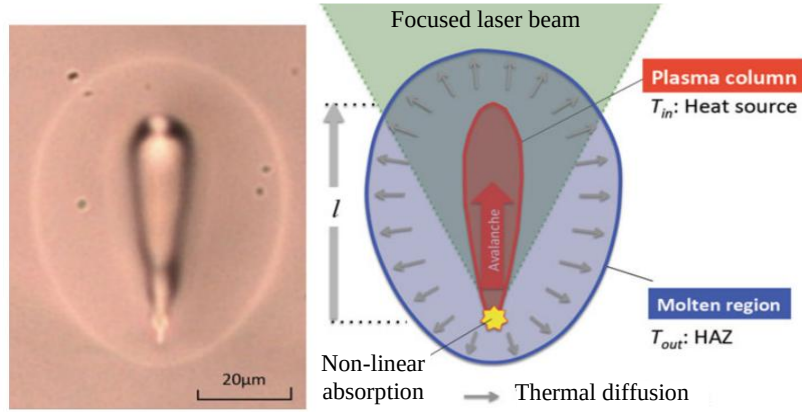


Figure 2.1 A dual-structure tear-drop formation by focused short pulses

Miyamoto et al. [11] investigated the effects of pulse energy, repetition rate and scanning speed on the tear-drop modification generated by scanning 10 ps and 1064 nm laser pulses inside a borosilicate glass. Microscopy images of the tear-drop morphology showed increased tear-drop height and width when the repetition rate is increased in the range of 50 to 1000 kHz. By increasing pulse energy in the range of 1 to 11 μJ and repetition rate, nonlinear absorptivity also enhanced by 40% to 95%. Ouyang et al. [12] used ray tracking to determine the effects of NA = 0.45, 0.65, and 0.85 on the spot size in air and glass. Focal position dynamics and absorption rate are characterized by a high-speed camera and transmitted laser power measurements. Spot size analysis showed more than 5 μm increase when the beam propagates in glass compared to air. By increasing pulse energy, absorption rate increased for all NAs, which was around 80% for NA=0.65 at laser and scanning parameters of 2.5 μJ, 1 MHz, 12.5 ps and 100 mm/s, respectively.

Table 2.1 lists reported tear-drop modification size for different laser processing parameters and scanning speeds.

Table 2.1 Tear-drop height and width using femtosecond and picosecond pulses

Pulse energy	Pulse duration	Rep. Rate	Scan speed	Tear-drop height	Tear-drop width	Ref.
9.25–75.25 μJ	8 ps	400 kHz	10 mm/s	437–591 μm	48–162 μm	[5]
1–4 μJ	10 ps	500 kHz	20 mm/s	40–120 μm	-	[9]
0.41–2.46 μJ	10 ps	500 kHz	20 mm/s	13–112 μm	5.7–76.6 μm	[10]
0.5–4 μJ	12.5 ps	1.0 MHz	100 mm/s	20–120 μm	18–58 μm	[11]
2.45–4.43 μJ	5.9 ps	400 kHz	2 mm/s	40–125 μm	3–13 μm	[12]
1–10 μJ	0.5 ps	200 kHz	1 mm/s	75–450 μm	18–160 μm	[13]

The maximum listed tear-drop height and width were around 600 and 160 μm , respectively [5]. This case corresponded to the highest pulse energy of around 75 μJ . Also, the highest scanning speed was 100 mm/s, which is required for high productivity in welding applications. This case corresponded to a lower range of height and width of 20–120 and 18–58 μm , respectively. The vast majority of cases utilized the picosecond pulse duration to minimize the filamentation effects and induce accumulation-based multiphoton absorption required for the tear-drop formation and growth.

However, the effects of preheating temperature on tear-drop modification focal position and size have not been considered in the literature. The current work focuses on investigating thermal effects on focal position by analytical analysis of spherical aberration and tear-drop morphology by experimental investigation using femtosecond and picosecond laser pulses.

2.3 Laser Glass Micro-welding

As mentioned in Chapter 1, a pre-weld contact is required between the glass workpieces to accomplish the transmission welding process. Optical contact can be achieved by mechanical clamping [1,3,5] of commercial glass slides or using highly polished samples [14,15] having a flatness better than $\frac{1}{4} \lambda$. The former can produce a partial optical contact while the latter may induce a wider contact area. In either case, an initial optical contact region is required where Van-der-Waals forces temporarily join the contacted surfaces to accomplish a crack-free welding

process [6]. Figure 2.2 shows examples of mechanical clamping setups and an optical contact region for laser glass welding experiments [1,5, 6].

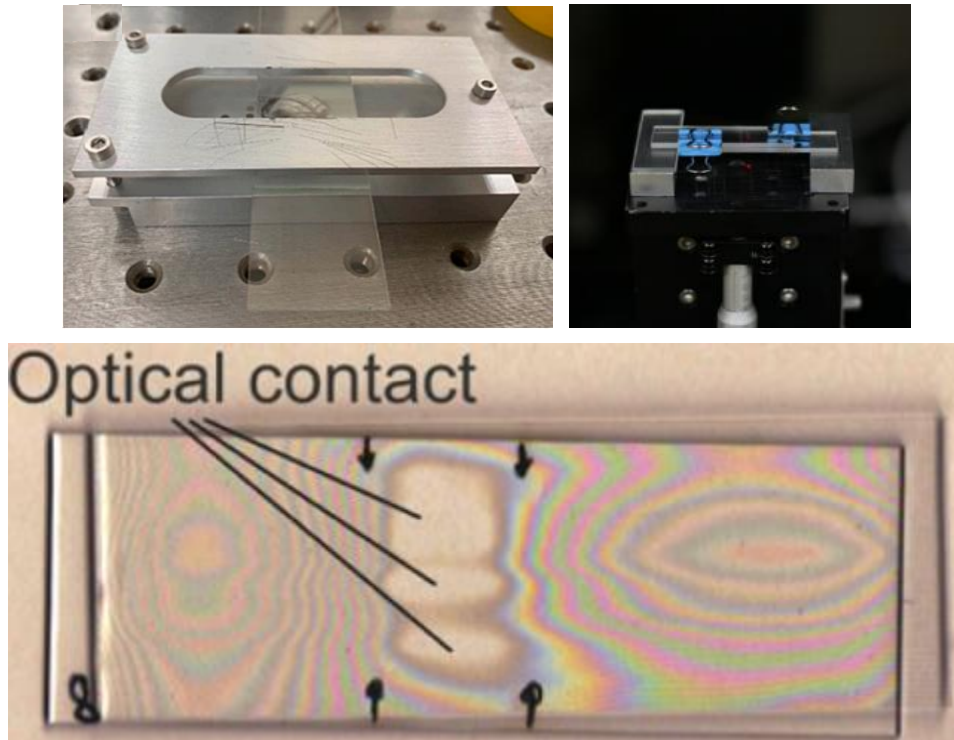


Figure 2.2 Examples of mechanical clamping setups and optical contact for laser welding

In addition to the transmission laser micro-welding, surfaces can be joined by scanning a focused laser beam through a narrow gap between adjacent surfaces [16]. In this technique, laser transmits through the contacted surfaces directly without propagating through the bulk of the transparent materials.

Post-welding, the partial optical contact can be extended by laser scans to a larger area indicating a successful welding of the surfaces [6]. A number of parallel scans are recommended to weld glass-to-glass to produce consistent strength measurements [17] and minimize the uncertainty effects stemming from variations in sample surface quality and thickness. Different number of parallel scans have been used in the literature resulting in a discrepancy of the welding

strength measurements. Jia et al. [1] reported a tensile shear strength of 3.3 and 6.3 MPa for 10 and 15 welding scans for a soda lime glass, while Chen et al. [7] used 40 scans and reported a 1325 N compression shear force for borosilicate glass. However, the number of scans has not been reported by Richter et al. [13] and Chen et al. [17], which corresponded to 72.9 and 39 MPa for laser welded fused silica and borosilicate glass, respectively. Table 2.2 lists recent contributions in the literature of transmission laser glass welding.

Table 2.2 Selected literature in transmission laser glass welding

Author and Year	Material	Parameters	Contribution
Chen et al., 2015 [12]	Silica	5.9 ps, 400 kHz	Avoiding the optical contact
Zhao et al., 2019 [5]	Silica	8 ps, 400 kHz	Welding using Low NA= 0.052
Jia et al., 2023 [1]	Soda lime	13 ps, 50 kHz	Multi-scan laser processing
Chen et al., 2023 [17]	Borosilicate	10 ps , 50 MHz	Beam shaping to crack suppression
Chen et al., 2024 [7]	Borosilicate	10 ps , 50 MHz	Multifocal beam shaping
Zhang et al., 2024 [18]	Aluminosilicate	1.2 ps, 1 MHz	Spatial shaping using a Bessel beam
Chen et al., 2024 [19]	Silica	300 fs, 1087 kHz	Pre-weld water layer between samples
Lafargue et al. 2025 [20]	Silica and Soda lime	500 fs, 40 MHz and 1.28 GHz	Burst-mode: MHz and GHz region

Commercialization of the transmission laser glass welding faces multiple challenges due to optical contact and process parameter requirements. The reported literature explored four major directions to solve these issues: welding strategy [1], beam shaping [5,7,17, 18], ultra-high repetition rate [20], and gap reduction techniques [12,19]. The multi-scan laser welding procedure of soda lime glass produced a successful bridging of 5 μm gap, but welding strength did not exceed 6.5 MPa and came at the expense of welding seam transparency, procedure complexity, and low

productivity. Beam shaping created welding seams with defects due to low NA and increased system complexity and power loss associated with spatial light modulators to generate a multi-focal beam. Other effects of beam shaping were a reduced laser processing window due to burst-mode and increased control requirements over the center of the non-diffractive zone of the Bessel beam affecting the welding transparency. Gap reduction techniques by applying a water layer have limited applications, especially for moisture-sensitive electronics or sensors. Comparatively, mechanical clamping can still produce equivalent gaps. Despite the aforementioned investigations, transmission laser welding of preheated glass substrates has not been explored in the literature.

2.4 Industrial Applications of Transmission Laser Welding

Traditional industrial solutions for glass joining utilize glues to bond glass-to-glass. During the solidification of the glue, gas volatiles and contamination by small parts of the gluing material are inevitable. Glue contamination issue has a significant reduction on yield of critical optics applications' production. Additionally, solidified glues suffer aging effects of hardening and moisture absorption, which shortens the service life of critical laser optics applications. Alternatively, laser glass welding does not require a second glue material, significantly localized, and durable [21].

Hélie et al. [22] reported the welding of a 100 μm thick fused silica glass slide to a highly-polished fiber optic for the first time in 2013 using a two-step procedure for the fiber end-cap encapsulation applications. First the glass-fiber interface was transmission-welded using a focused laser beam of 70 fs, 250 kHz, 787 nm, 0.2 mm/s, and 600 nJ. Second, glass slide edges were milled starting from the rear surface of the glass using a liquid-assisted rear surface drilling. Figure 2.3 shows the two-step fiber end-cap encapsulation schematic and results.

Sensor packaging is another application that utilizes transmission laser glass welding. Kim et al. [23] introduced an implantable blood pressure sensor packaged by a laser beam of 370 fs, 2 MHz, 1030 nm, 20 mm/s, and 4 μ J. Three scans having a rectangular profile were used to weld the interface between optically contacted flat and trench quartz glasses to produce a hermetic and bio-compatible blood pressure sensor. Penilla et al. [16] utilized the through-gap laser scanning to weld ceramic surfaces for hermetic encapsulation of sensors for severe enviromntal applications like nuclear reactor monitoring. The application was filed as a patent in the filed of sensor packaging using transparent ceramics. In their study, a gap of ~ 12.5 μ m was bridged between a cap and cylinder made of yttria-stabliazed zirconia using laser scan conditions of 230 fs, 1 MHz, 1028 nm, 13 mm/s, and 8.8 μ J, as shown in Figure 2.4.

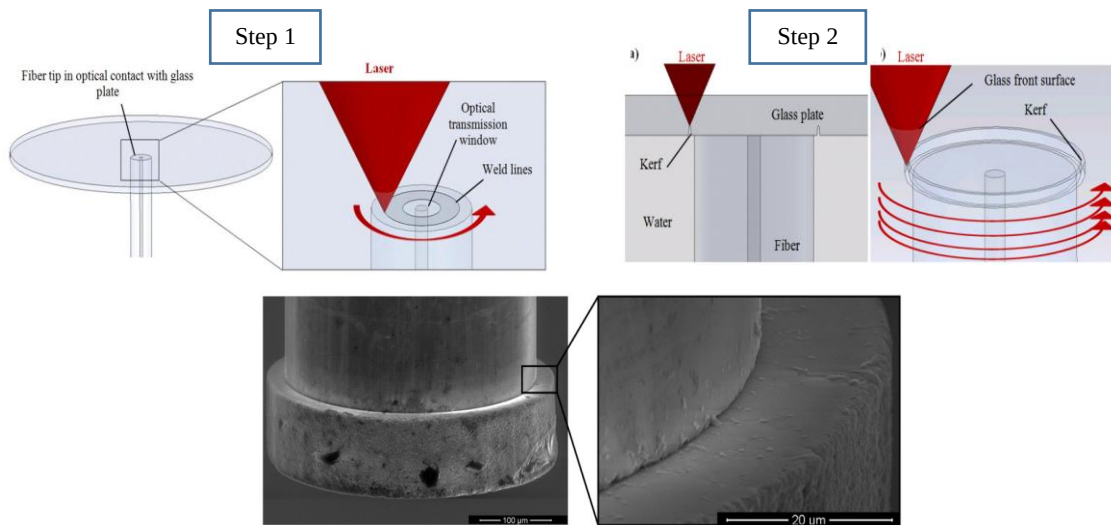


Figure 2.3 Fiber end-cap encapsulation: step 1- laser welding and step 2-laser milling

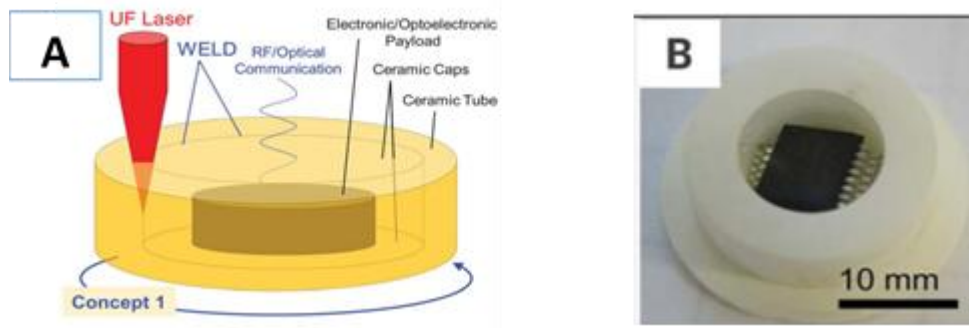


Figure 2.4 Ceramics sensor packaging: (A) concept and (B) integrated circuit inside tube

Recently, researchers from the National Renewable Energy Lab (NREL) [24] reported the application of transmission laser welding of low-iron glass replacing the intermediate ethylene vinyl acetate polymer film that is traditionally used to bond glasses for solar modules. The application involved welding optically contacted flat-to-trench solar glasses utilizing a new design. The study involved mechanical testing, multiphysics simulation, economic analysis and the realization of a fs laser system for a compact industrial plant. Details about the laser parameters were not reported.

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Chapter 3 . On the Effects of Substrate Temperature on Glass

Internal Modification

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On the Effects of Substrate Temperature on Glass Internal Modification using Femtosecond
Laser Pulses

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Abstract

The effects of substrate temperature on laser focal position and tear-drop morphology in laser internal modification of glass are investigated. A model is derived to predict the shift of the tear-drop at high substrate temperature. Femtosecond laser pulses are scanned inside borosilicate glass at room temperature, 150, and 200 °C using a pulse energy of 4.5 – 18 μJ , a scanning speed of 5 – 20 mm/s, and a distance between lens and glass of 9.56 – 10.76 mm. Temperature effects are characterized by defining a height (width) gain ratio as the ratio between the tear-drop height (width) measured at high temperature to that measured at room temperature. Thermal expansion is simulated using a profile temperature acquired by a thermal camera and image processing. Results show that substrate temperature has a significant effect on self-focusing, and modifications at 200 °C show a relaxed discoloration compared to 150 °C and room temperature. Analytical predictions match the measurements of focal position in the distance of 9.56 – 9.96 mm at 200 °C where self-focusing is not significant while underestimates the measurements for the distance of 10.76 and 10.36 mm by 30 – 50 μm . At 200 °C, tear-drop's gain ratio is increased when the pulse energy is increased in the range of 4.5 – 18 μJ . Within this pulse energy range and at 200 °C, the maximum width gain is 10 to 100% higher compared to the maximum height gain.

3.1 Introduction

Glass internal modification by laser radiation incorporates focusing a laser beam inside a glass substrate with an intensity exceeding the glass fluence threshold. The focused beam leads to a localized energy deposition and melting followed by a solidification as the laser beam scans through the modified region. Laser energy absorption takes place by multiphoton ionization, tunneling [1, 2], avalanche ionization, and plasma absorption [3]. Laser energy is absorbed by valence band electrons moving them to the conduction band, and free electrons producing an

absorption volume, which grows away from the focal position in the direction of the incident beam forming a tear-drop shaped modification when thermal diffusion follows the plasma evolution [4].

A practical application of glass internal modification is glass-to-glass laser welding. Generally, the technology can be used to weld a transparent material to a transparent or non-transparent material utilizing the nonlinear absorption [5]. Additionally, the process is flexible to weld similar and dissimilar glass substrates like fused silica to fused silica and fused silica to BK7 [6], borosilicate to fused silica, borosilicate to ULE, and ULE to zerodur [7]. Parameters that determine the processing window and mechanism of glass welding are pulse energy, pulse duration, repetition rate, scanning speed, and wave length [8].

Process parameters for laser glass welding are distinguished for two steps: a conditioning step and laser welding step. In the conditioning step, two samples of glass are brought together under full optical contact conditions, which require highly smooth, flat, and clean surfaces leaving a gap of around $\frac{1}{4} \lambda$ or 100 nm [1, 5] between the contact surfaces. Still part of the conditioning step is to determine the focal position precisely.

Achieving optimum focal position is critical for a successful glass welding, even if full optical contact is not achieved. A successful welding can be achieved when the focal position lies in the welding range, generally when the position is located under the contact surfaces as observed from the front glass sample. Establishing full optical contact is essential in the preparation step, however it is technically costly even for highly polished surfaces, especially when large welding area of several square centimeters is required [9].

Aspect ratio of the tear-drop modification, which represents the tear-drop's height to width ratio is another critical parameter that controls post welding defects like longitudinal crack formation. When the aspect ratio approaches unity for a semi-spherical tear-drop, the longitudinal

crack formation is minimized due to less concentrated stresses are generated after the weld seam is cooled down. A semi-spherical tear-drop can be realized using objective lens having a high numerical aperture of $NA \approx 0.8$; however, spherical aberration can affect the expected aspect ratio especially when the focal plane is located relatively deep into the glass material [10]. The focal position and tear-drop's morphology are of great importance for a successful glass welding. The absorption volume of plasma and the size of the heat-affected zone (HAZ) determine the welding quality when the focal position is optimized.

Despite the extensive research that has been reported so far, the effect of substrate temperature on laser glass welding has not been considered in the literature. Substrate temperature may be introduced as a parameter of the conditioning step of the laser glass welding. It is believed that using a preheated glass substrate enhances the tear-drop's size and aspect ratio, which in turn improves the femtosecond laser weldability by bridging larger gaps especially when thicker substrates are considered. As a first step towards laser glass welding under preheated substrate, the effects of substrate temperature on tear-drop modification inside a thick borosilicate glass samples of ~10 mm is investigated in this work. The thermal effects are investigated at two temperature levels of ~150 °C (MT) and ~200 °C (HT), and compared to room temperature (RT). The analyses of the current work aim to establish the fundamental theoretical and experimental background of temperature effects on the tear-drop modification for future applications based on laser internal modification.

3.2 Thermo-optical Analysis

Relatively high substrate temperature affects laser focal position due to thermal expansion compared to room temperature (RT). Additionally, temperature is expected to affect the modification mechanism and tear-drop's aspect ratio, which are analyzed in the current

investigation. For a laser beam passing through an objective lens of numerical aperture $NA = \sin \theta_1$ and traveling from a low refractive index material of n_1 to a high refractive index material of n_2 , the beam refracts at the interface between the two materials. The incidence and refraction angles, θ_1 and θ_2 , are determined by Snell's law [11]:

$$\frac{\sin \theta_1}{\sin \theta_2} = \frac{n_2}{n_1} \quad \text{Equation (1)}$$

where $n_1 = 1$ and $n_2 = 1.5$ are the refractive indices of air and glass, respectively. Figure 3.1 shows a schematic representation of a laser beam refracting at the interface between air and glass.

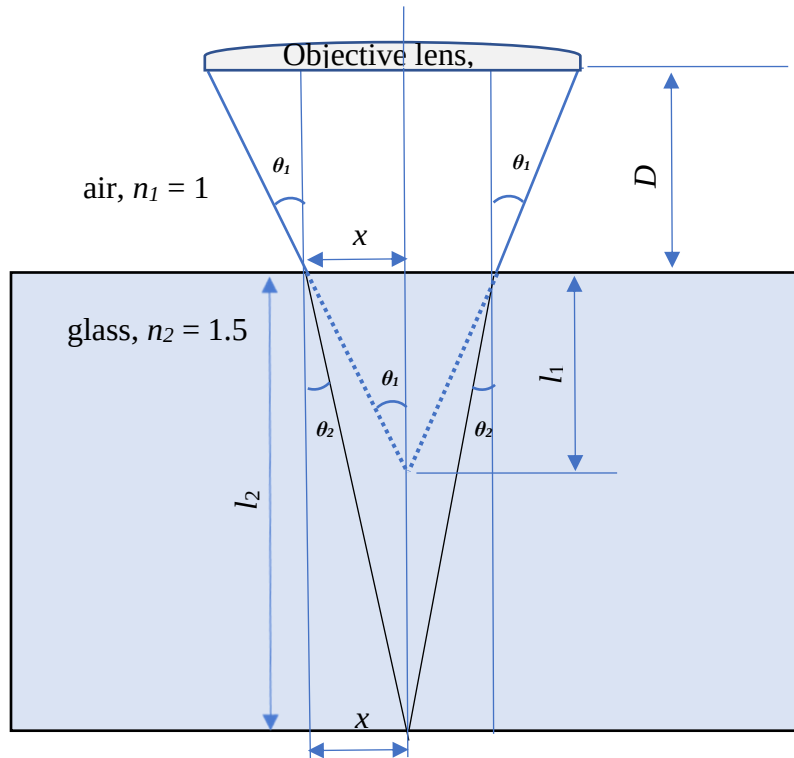


Figure 3.1 Laser beam refraction at the air-glass interface

For objective lens, NA is determined by half of the cone angle at the point of focus and the refractive index [12]:

$$NA = n_1 \sin \theta_1 \quad \text{Equation (2)}$$

From the schematic of Figure 3.1, the depth of focal position in air, l_1 , and inside the glass, l_2 , are determined using Equations 3 and 4:

$$x = l_1 \tan \theta_1 = l_2 \tan \theta_2 \quad \text{Equation (3)}$$

$$\frac{l_2}{l_1} = \frac{\tan \theta_1}{\tan \theta_2} \quad \text{Equation (4)}$$

From Equations 1 and 2 when $n_1=1$, $\theta_1 = \sin^{-1} NA$ and $\theta_2 = \sin^{-1} \left(\frac{NA}{n_2} \right)$. Equations 1 to 4 yield $l_2/l_1 = b$, where b is an aberration parameter that depends on the NA and the refractive index n_2 . For a NA= 0.3 and $n_2= 1.5$, $b \approx 1.54$.

Additionally, Figure 3.1 shows the focal length of the objective lens, FL , determined by Equations 5 and 6:

$$FL = D + l_1 \quad \text{Equation (5)}$$

$$FL = D + \frac{1}{b} l_2 \quad \text{Equation (6)}$$

It should be noted that the current analysis using ray tracing is equivalent to the analysis of spherical aberration effects in [13, 14]. However, the current model estimates the deepest focal position and its shift due to temperature effects.

The effects of thermal expansion at high temperature (HT) on focal position shift compared to RT are schematically illustrated in Figure 3.2. Two glass samples are modified by a laser beam incident from an objective lens fixed at location L for both cases, one at RT and the other HT. The refractive index of glass is assumed to be temperature-independent for the RT and HT cases. In the HT case, it is assumed that glass is fixed at the bottom boundary at $y=0$; expansion happens in +y direction only. Thermal expansion takes place in +y direction for the upper boundary of HT sample by Δl_1 where the focal position at HT is located at point b' that is shifted by Δl_2 compared

to the focal position of point b at RT. Since Equation 4 and its consequence of $l2/l1 = b$ represents a general relation of focal position at any temperature, for the HT case in Figure 3.2 it is:

$$\frac{l2 + \Delta l2 + \Delta l1}{l1 + \Delta l1} = b \quad \text{Equation (7)}$$

The result of Equation 7 yields $\Delta l2 = (b - 1)\Delta l1$, which means the shift in laser focal position after thermal expansion equals $(b - 1) \times 100\%$ of the thermal dilatation in +y direction. Since the angles of $\theta1$ and $\theta2$ are not changing with temperature, Equation 7 can be verified using CAD models after applying different dilatations in +y direction while keeping the lens location unchanged. By accounting for the thermal expansion effects, Equation 6 can be modified by adding the shift in focal plane position due to thermal expansion Δl_2^{HT} and thermal dilatation of the top surface Δl_1^{th} :

$$\begin{aligned} l2^{HT} &= (FL - D) * b + \Delta l_2^{HT} = (FL - D) * b + (b - 1) \Delta l_1^{th} \\ &= \left(FL + \frac{(b - 1)}{b} \Delta l_1^{th} - D \right) * b \end{aligned} \quad \text{Equation (8)}$$

where $l2^{HT}$ is the focal position at HT. It should be noted that even if thermal expansion happens in the x-direction, it should not affect the validity of the current analysis for three-dimensional cases. One more observation is that Equation 8 determines the focal position $l2^{HT}$, which is the distance between the top surface of glass at HT and the point b' . After laser processing when the glass sample of HT cools down to RT, there will be a differential contraction between the y-coordinate of top surface and the y-coordinate of point b' . This differential contraction takes place when there is a temperature variation between the top surface and the point b' position, which may render the actual focal position $l2^{HT}$ to be smaller than the calculated one from Equation 8 by the amount of differential contraction. This effect will be investigated by FEA simulations in Section 3.3.

Laser beam propagation is affected by self-focusing or Kerr effect, which is significant when the applied peak power exceeds a critical level. For borosilicate glass, the critical peak power is 3.2 MW for a laser of 1046 nm wavelength and a refractive index of 1.52 [15]. The laser peak power used in the current work lies in the range of 17.5-70 MW when 30% of lens power loss is considered, which is higher than the critical peak power for self-focusing. The shift due to self-focusing is out of the scope of the current work, which is not significant when self-focusing is minimized at HT as will be verified by the current experimental results.

Mechanical/thermal stresses can induce a refractive index variation due to a stress-induced birefringence. However, borosilicate glasses are tolerant to stress-induced birefringence where the variation in refractive indices is less than 30×10^{-6} when the absolute stress level is as low as 10 MPa [16]. This effect supports the assumption of a constant refractive index when glass temperature is raised for the current thermo-optical analysis, and the thermal stress levels are analyzed in the FEA results section for further verification.

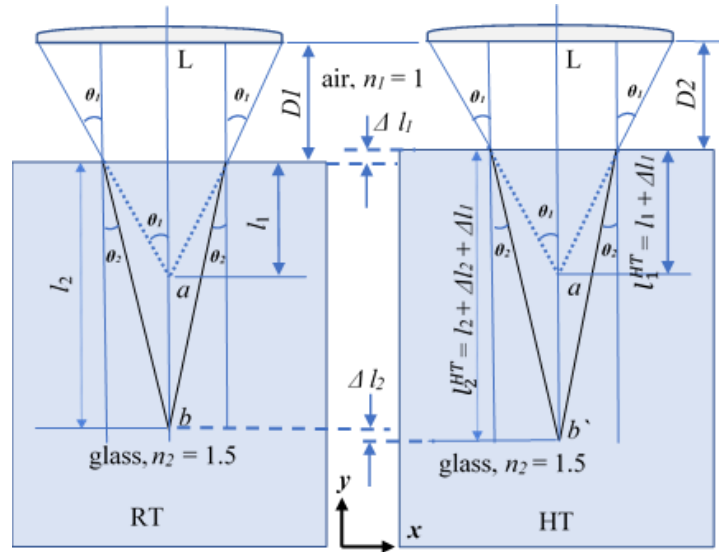


Figure 3.2 Effect of thermal expansion on the focal position shift

3.3 Thermal Dilatation Simulation for HT Experiments

The thermal dilatation Δl_1^{th} of the glass surface facing the laser beam during experiments is determined by the 3D domain and boundary conditions of the glass sample shown in Figure 3.7. Due to computational complexity of determining convective and radiative heat transfer, the following procedure for finite element analysis (FEA) is adopted. The simulation domain has the dimensions of 50.8 mm x 50.8 mm x 10 mm that represents the glass sample. The heating and thermal expansion process is simulated using a coupled thermo-mechanical module in COMSOL, a commercial software package. Steady-state coupled simulation is conducted using fixed boundary displacements in the x-, y-, and z-direction for the glass surface that is attached to the optical plate. A temperature boundary condition is mapped from a thermal camera image using IRimage software [17] as a plugin for imageJ. The IRimage is used to extract the raw temperature 2D profile for the captured image shown in Figure 3.3. Due to the presence of the right-angle bases that secures the glass to the optical plate, only the upper third of the glass surface temperature profile is used. The 2D temperature profile is interpolated using the extracted raw data by linear interpolation, as shown in Figure 3.4.

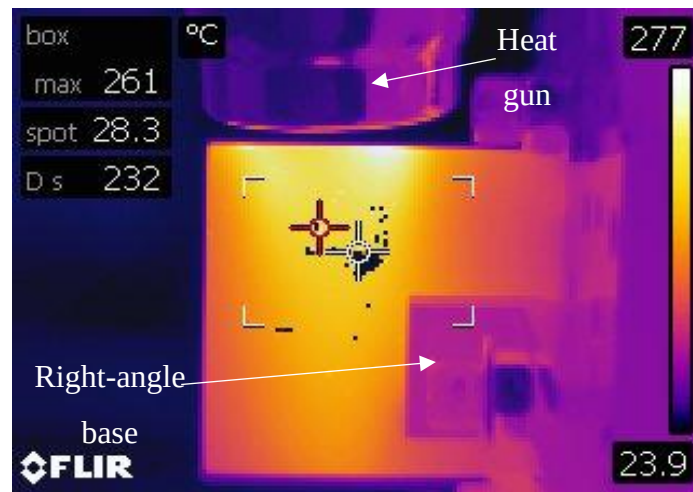


Figure 3.3 Thermal image captured by a camera for the HT experiment

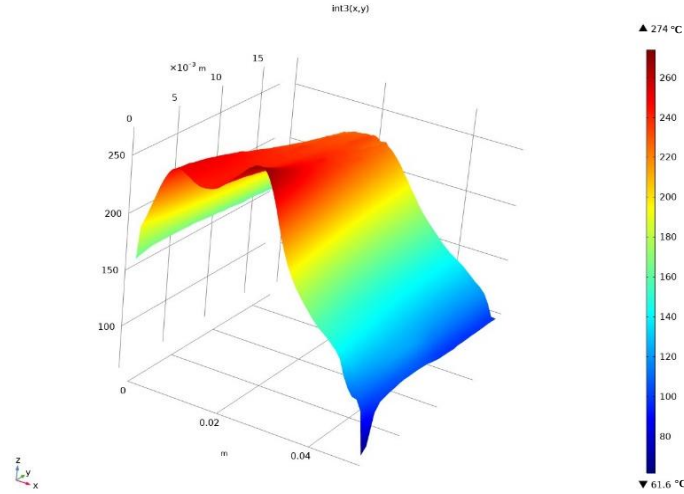


Figure 3.4 The interpolated temperature profile for the top third of the glass surface

The mechanical and thermophysical properties of borosilicate glass are taken from the literature [18, 19] and listed in Table 3.1 below.

Table 3.1 Mechanical and thermophysical properties of borosilicate glass

Property	Value	Property	Value
Young's modulus	64 GPa	Thermal conductivity	1.25 W/m.K
Poisson's ratio	0.22	Specific heat capacity	746 J/kg.K
Density	2.23 g/cm ³	Coefficient of thermal expansion	3.3 x 10 ⁻⁶ 1/K

Figure 3.5 shows the simulated temperature distribution of the glass slab compared to the thermal camera image. A minor difference of 3 °C in the peak temperature of the simulation (left of Figure 3.5) and the captured image (right of Figure 3.5) is observed.

Figure 3.6 shows the temperature, von Misses stress and thermal dilatation at the surface facing the laser beam and at 3 mm deep into the glass to account for the deepest tear-drop modification location. The modification sets of HT are located inside the marked region in Figure 3.5 within 10 mm (0.01 m) from the top of the sample, which corresponds to 10 mm on the x-axis

(arc length) of the plots in Figure 3.6. The temperature range is within 190 to 210 °C where the modifications are produced during the experiments for which the HT case has the average temperature of ~ 200 °C.

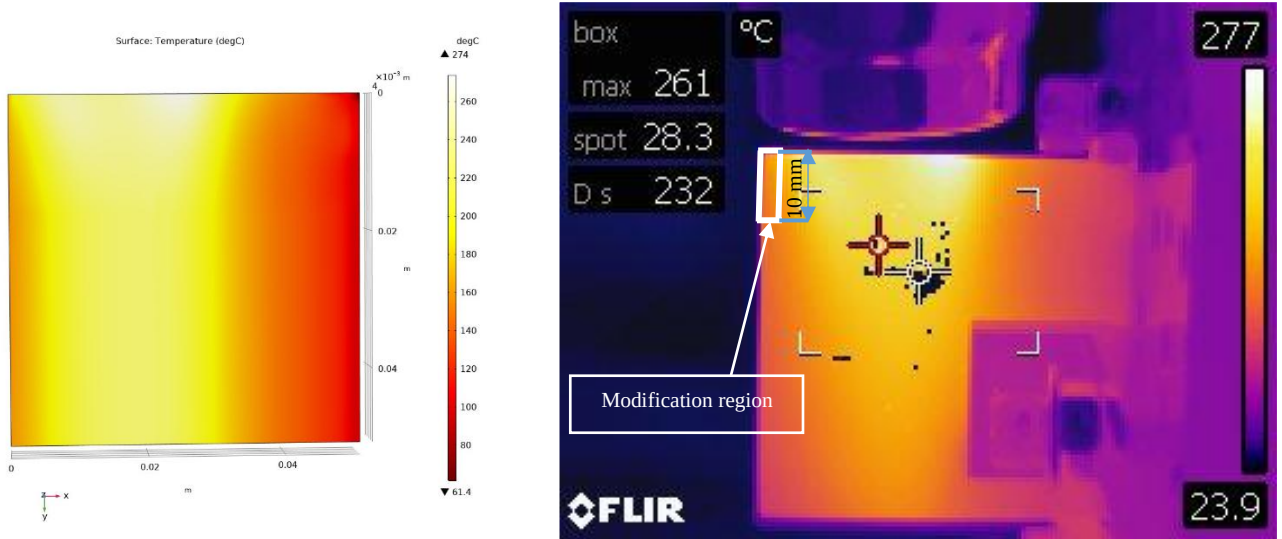


Figure 3.5 Temperature distribution of the captured image compared to the FEA results.

The von Misses stress is less than 3 MPa for all cases that should not produce a stress-induced birefringence as explained in Section 3.2 of the paper. The thermal dilatation is marked by -u as the displacement takes place in the negative x-direction. The thermal dilatation is between 29 to 30.6 μm for the focal position shift of Equation 8. Thermal dilatation difference between the surface and the 3 mm deeper curves is almost 1.5 μm , which reflects the differential contraction between these locations when the glass sample cools down to RT after laser processing. This differential contraction is negligible compared to the measured focal positions of 1.35 to 3.15 mm for the experiments of the current work in Section 3.4.2. Similar FEA simulations are conducted for the MT case where the average temperature within the modified region is ~ 150 °C.

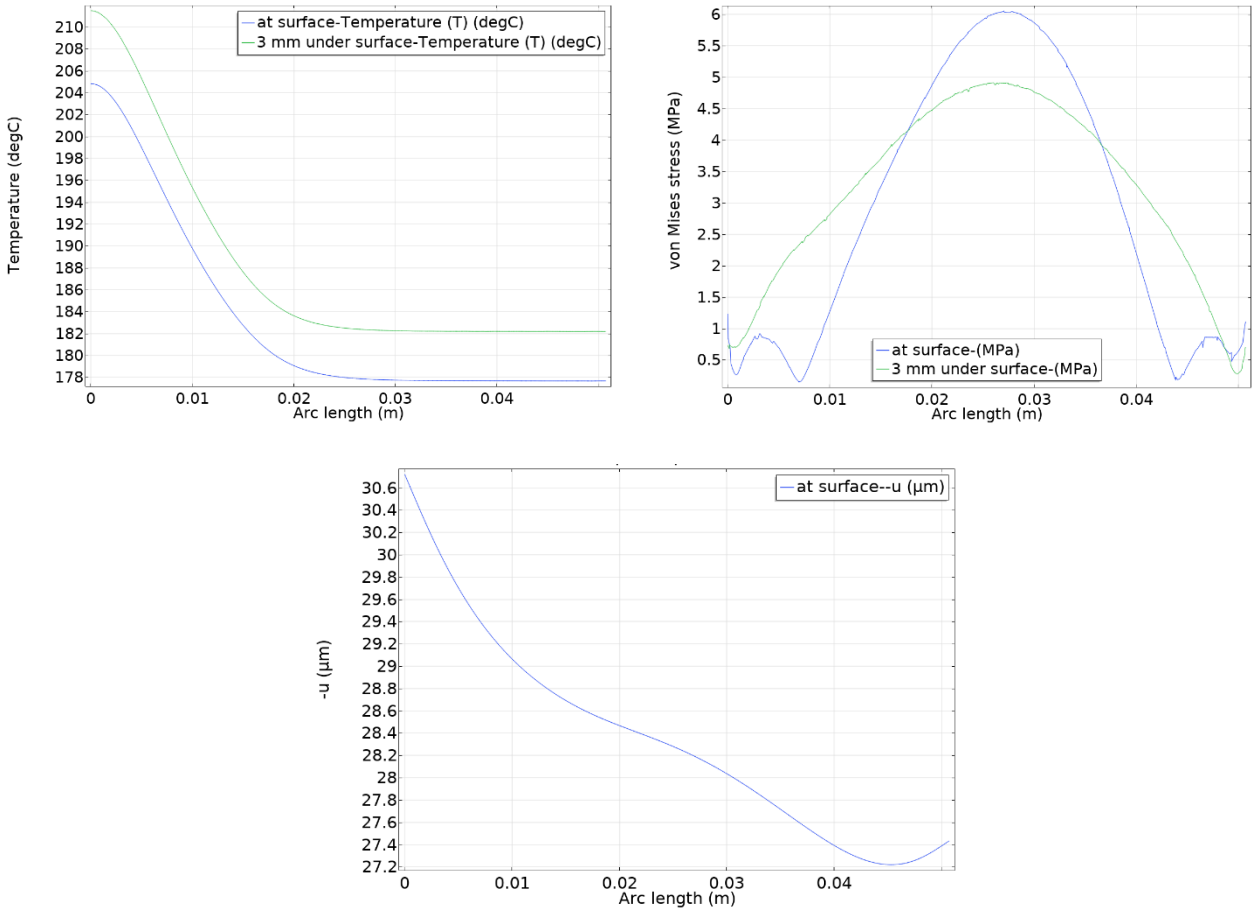


Figure 3.6 Temperature, von Misses stress, and thermal dilatation based on FEA results

3.4 Experimental Procedure of HT Modification

An experimental setup is utilized to heat up one thick borosilicate glass sample to the required temperature, using a hot air flow from a heat gun attached to a stage, and keep it relatively constant during laser scanning. The setup is shown in Figure 3.7 below. The thermal expansion effects are investigated at ~ 150 °C (MT) and ~ 200 °C (HT), and compared to room temperature (RT). Femtosecond laser pulses of 1028 nm, 184 fs, and 200 kHz for wave length, pulse duration, and repetition rate are utilized for the current experiments. Pulse energy, scanning speed and focal plane position are varied during the experiments where an objective lens of $NA = 0.3$ is used to focus the laser beam. Three glass replicates are considered where tear-drop size and focal positions

are measured using optical microscopy. The glass substrate temperature is measured by a thermal camera where images are captured and processed for the finite element analysis in Section 3.3. The analytical model of Section 3.2 is used to predict the focal plane position with thermal expansion taking place and to compare with the experimental measurements.

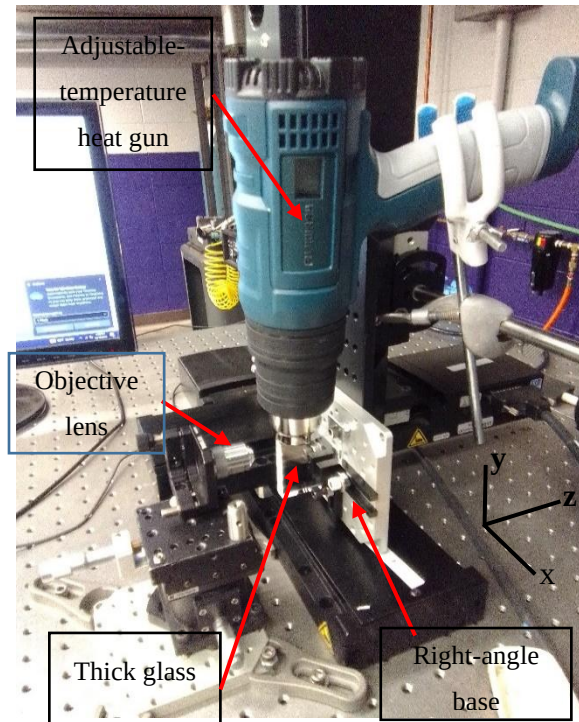


Figure 3.7 Experimental setup of preheated glass modification.

The experimental procedure for each replicate is repeated using similar conditions to minimize sample-to-sample variations. All glass samples have chamfered corners, the overall dimensions are 50.8 mm x 50.8 mm x 10 mm, for the side lengths and thickness, respectively. The thick glass sample is attached to an optical plate and secured using a right-angle base from all sides to avoid detachment between the glass sample and the optical plate when hot air is applied using a temperature-adjusted heat gun mounted on the stage. The hot air temperature is adjusted where the glass temperature is monitored using a thermal camera until the maximum temperature reaches

277 °C at the glass surface facing the camera. Initially, laser pulse energy is set to 4.5 μJ while keeping other laser parameters fixed. When the required temperature is stabilized after around 5 minutes, laser is scanned by moving the stage with 5 mm/s in the x-direction shown in Figure 3.7 for each distance of 9.56 – 10.76 mm with a step of 0.4 mm. This procedure is repeated for the speeds of 10 mm and 20 mm/s while keeping the hot air flow to insure consistent temperature for all scans. When the first set of experiments is accomplished for the 4.5 μJ and scanning speeds of 5, 10, and 20 mm/s, the procedure is repeated for the pulse energy of 6, 10, 14, and 18 μJ using similar levels of scanning speeds. The interaction between successive modifications is minimized by performing deeper scans first for each set of similar pulse energy and speed, and after each scan of a specified distance, the stage is moved in the y-direction by 0.1 mm.

Similarly, the temperature of the heat gun is adjusted until the maximum temperature reaches 185 °C at the glass surface facing the thermal camera where the distance, speed, and pulse energy are varied for this level of temperature (MT). This step-by-step procedure is repeated for three replicates where the tear-drop modifications are analyzed using optical microscopy.

4.5.1 Self-focusing Effects at High-temperature

The source pulse energy range of the current experiments is between 4.5 and 18 μJ as measured before the objective lens. When considering the lens loss of 30%, the fluence range is between 56.6 and 226.5 J/cm^2 , which corresponds to an intensity range of 0.31×10^{15} to 1.2×10^{15} W/cm^2 , respectively. The spot size is calculated using $2w_0 = 2(M^2 \lambda)/(\pi \cdot \text{NA})$ where $M^2 = 1.22$, $\lambda = 1028$ nm, $\text{NA} = 0.3$, and $2w_0 = 2.66$ μm . The intensity is calculated using $I = F/t_p$ where F is the fluence and $t_p = 184$ fs, which is the pulse duration. This intensity range is higher than the intensity threshold of borosilicate glass of 2.15×10^{13} W/cm^2 , as reported in [4]. The peak power for the 184 fs scans using 18 μJ and 30% loss is 70 MW, which is higher than the critical peak power of

3.2 MW [15] for borosilicate glass. Figure 3.8 shows two groups of RT modifications, the nine tear-drops in the upper part are scanned using a source pulse energy of 14 μJ and the other nine in the lower part are scanned using a source pulse energy of 18 μJ . Tear-drops' numbers of 1, 2, 3 and 10, 11, 12 are scanned using a speed of 5 mm/s, while those designated as 4, 5, 6 and 13, 14, 15 are scanned using a speed of 10 mm/s. The rest are scanned using a speed of 20 mm/s. The numbering scheme follows the scanning order of the tear-drops. The modifications that are created at RT are preceded by an evident conical discoloration/darkening as shown in Figure 3.8, which is on-axis with the beam direction for all pulse energies and speeds. This discoloration is the result of color center formation that is associated with borosilicate glass under femtosecond pulses [20]. To determine the formation mechanism of color centers in the current femtosecond cases, a set of RT picosecond experiments is conducted using similar pulse energy, repetition rate, scanning speed, and depth of focus to the femtosecond cases, but with a 10 ps pulse duration. For the picosecond scans, the 18 μJ source pulse energy has a peak power of 1.3 MW when lens loss is considered, which is below the critical peak power for self-focusing. When self-focusing is hindered using the 10 ps laser scans, the discoloration is not significant as shown in Figure 3.9. This gives strong evidence that color centers are generated due to self-focusing for the 184 fs scans, while tear-drop modifications due to a multi-photon absorption happen in both cases due to the applied intensities exceeding the threshold. Hence, the presence of discoloration is used as an indication of significant self-focusing effects.

For the high temperature cases where substrate temperatures of $\sim 150^\circ\text{C}$ and $\sim 200^\circ\text{C}$ are used at the modification region, Figures 3.10 and 3.11 show the tear-drop modifications generated using 184 fs pulses. The discoloration is moderate in the MT case and insignificant in the HT case. It has been reported that color center formation is a reversible phenomenon and can be relaxed

when borosilicate glass is annealed at high temperatures after exposure to femtosecond pulses [20]. The explanation of the results in Figures 3.10 and 3.11 is twofold: either a concurrent discoloration nucleation and relaxation happen or a discoloration does not take place at high temperatures. In either scenario, the result is that self-focusing-assisted discoloration is moderate for the MT case and insignificant for the HT case, which has an effect on the focal position compared to RT case.

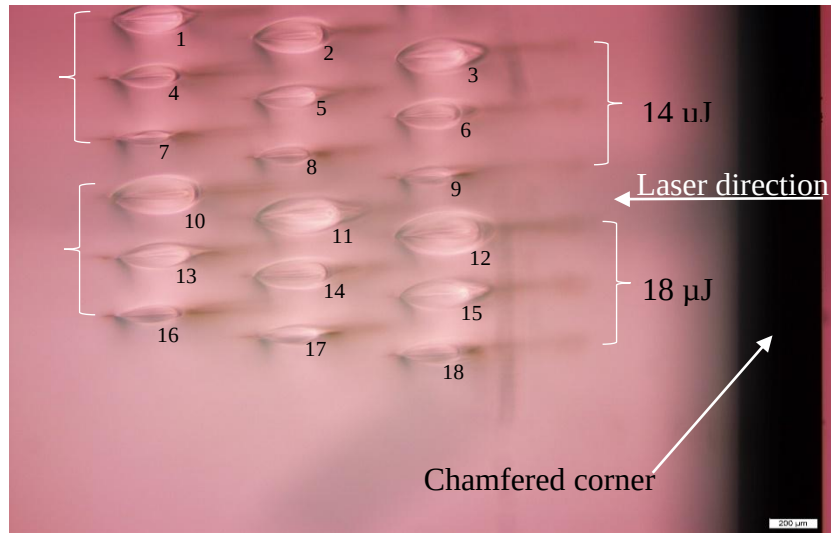


Figure 3.8 Tear-drop modifications for the 184 fs laser scans at RT

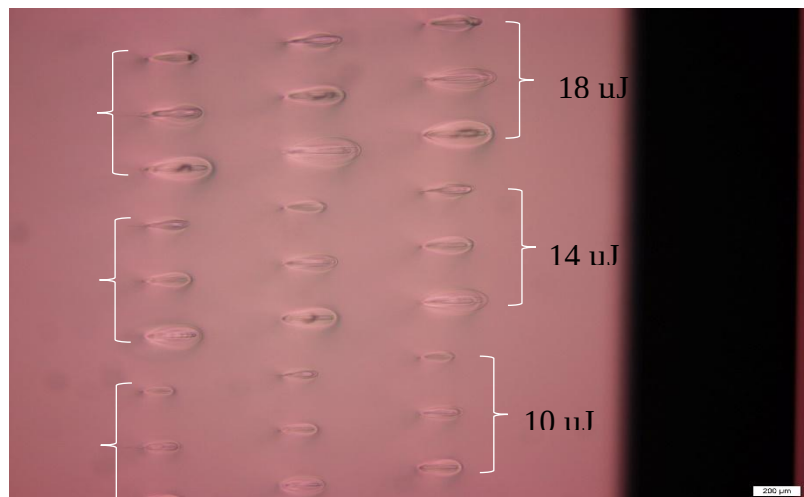


Figure 3.9 Tear-drop modifications for the 10 ps laser scans at RT

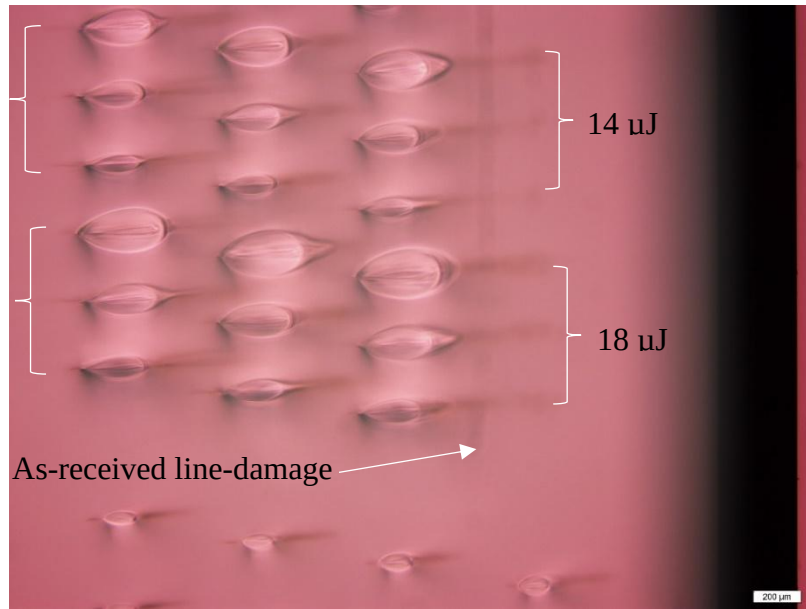


Figure 3.10 Tear-drop modifications for the 184 fs laser scans at MT case

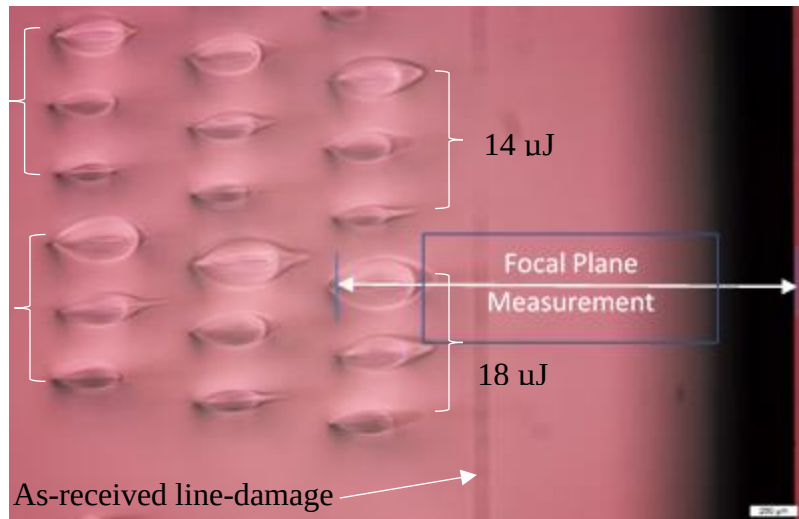


Figure 3.11 Tear-drop modifications for the 184 fs laser scans at HT case

4.5.2 Focal Position

Focal position depth is measured using Image J for the lower tip of the tear-drop core where the plasma-modified region is started as shown in Figure 3.11. To allocate the focal position with minimum uncertainty, shadowing effects are added to the images during the measurements.

However, there has been uncertainty in the measurements due to small-sized tear-drops, modification distortions, and longitudinal microcracks in the beam direction where low pulse energy or higher speeds are used, especially for deeper scans.

Figure 3.12 shows the measured depth of focus for RT, MT, and HT scans using 5 mm/s scanning speed and a distance range of 9.56 – 10.76 mm with a step of 0.4 mm. All three replicates' measurements are included in Figure 3.12 for the source pulse energy range of 4.5 to 18 μJ . For some cases, especially the HT cases with high pulse energy and low scanning speed, the distance level of 10.76 mm is not considered due to high probability of glass damage by crack/cleavage propagating from the modified locations to the exposed surface. For all temperatures, the depth of focus follows a linear dependence on the distance, as predicted by Equation 8. However, there has been a constant shift for the RT and MT cases rendering the focal position shallower compared to the HT case, which is consistent for all pulse energy levels. This is the effect of self-focusing, which is significant for the RT and moderate for the MT case, respectively. This result is in agreement with the color center relaxation effect of temperature that is explained in Section 3.4.1. As the self-focusing-assisted color center formation is relaxed/inhibited, the experimental results are closer to those from Equation 8, which takes place for all pulse energy levels from 4.5 to 18 μJ .

Comparing the interpolation lines of experimental results for all pulse energy levels at HT with the line of Equation 8, there is a slight discrepancy between the interpolation lines for the distances of 10.35 and 10.75 mm. This discrepancy has the experimentally measured focal position deeper compared to the calculations of Equation 8. Further investigation is required to analyze this discrepancy.

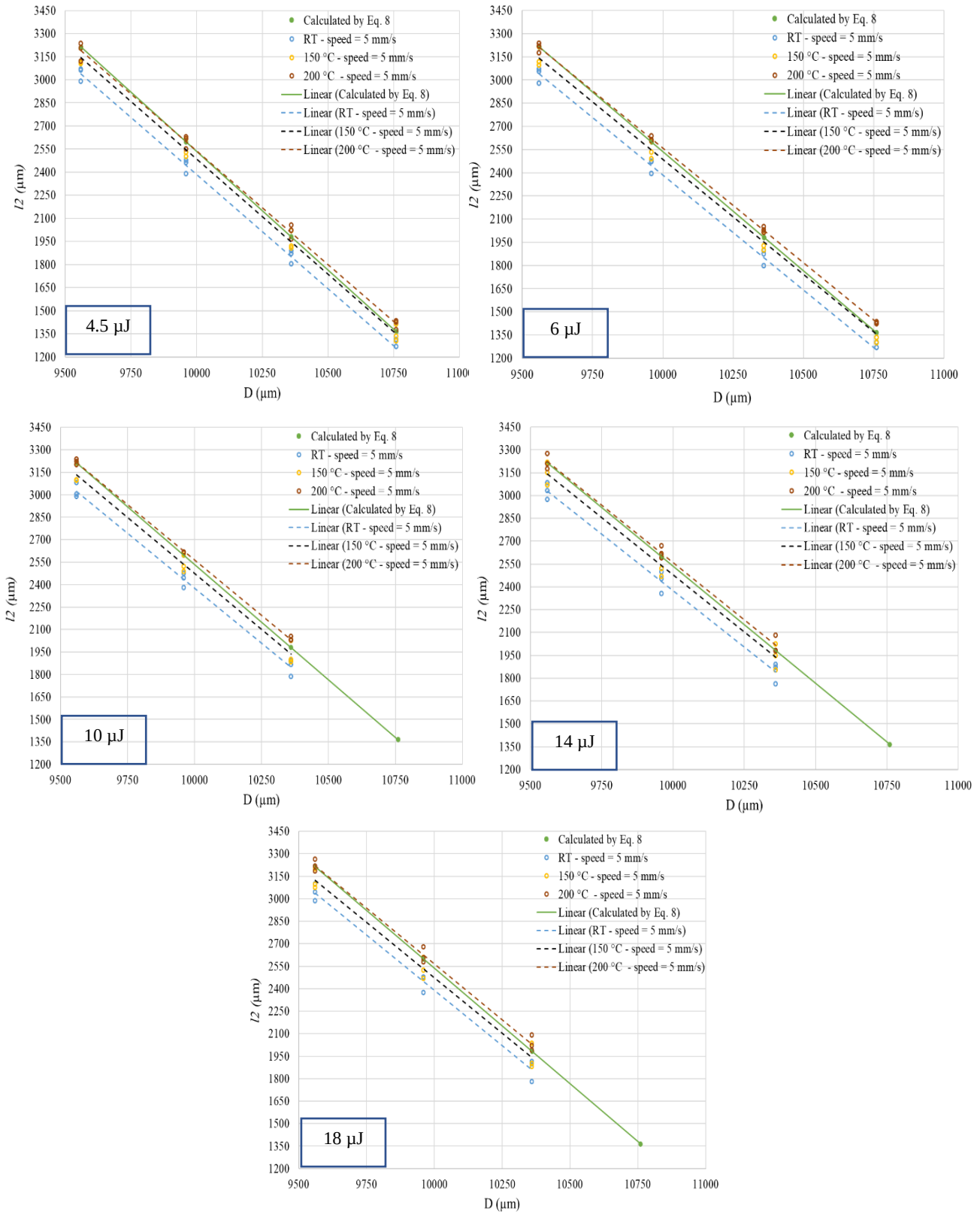


Figure 3.12 Focal position predictions and measurements at RT, MT, and HT cases

4.5.3 Tear-drop Morphology

The effect of substrate temperature on tear-drop height and width is introduced by height and width gain ratio, which represent the ratio of the height or width of the tear-drop at the MT or HT condition to the corresponding height and width of the tear-drop at RT condition. The gain ratio is defined for tear-drops having similar scanning conditions of pulse energy, speed, and depth of focus. The scattering nature of the measurements shown in Figures 3.13-3.15 stems from the modification defects like longitudinal and transverse microcracks [10], nonlinear propagation effects of self-focusing, spherical aberration [13, 14], and glass refractive index inhomogeneity [16], in addition to the measurement uncertainty due to uncertainty in determining the tear-drop boundaries. The raw measurements of three replicates are combined into six groups for each pulse energy. Each group has the gains of similar speed and temperature. All six groups are filtered by 1st and 3rd quartile outlier analysis to exclude measurements abnormally larger or smaller compared to the data bounds.

Filtered gain ratio results are shown in Figures 3.13-3.15 for pulse energy of 4.5 to 18 μJ , and the measurements are grouped by pulse energy and temperature for scanning speeds of 5, 10, and 20 mm/s. For all pulse energy levels, the gain ratio has a general trend that for the HT case, the height and width ratio increase when the focal position l_2 is increased. For the MT case, the gain ratio is almost unchanged when the l_2 is increased for all pulse energy levels. Within the HT conditions, the increase of the gains for deeper scans is a result of having higher temperature for deep scans of 3 mm by 7 °C compared to surface temperature, as shown in Figure 3.6. This difference, though relatively small, has an effect on incremental relaxation in self-focusing for deeper scans compared to near surface scans, which in turn enhances the modification topology. However, the dependence of self-focusing relaxation on temperature for the HT conditions within

the 3 mm focal position range is still qualitative depending on visual inspections by microscopy. A quantitative measure is required for establishing precise temperature-discoloration correlation to support the previous observation. Qualitatively, the self-focusing is inhibited when substrate temperature is increased for the HT case compared to the MT case, which is the apparent reason for the difference in gain ratio trend of these cases. When the self-focusing dominates at RT and partially at MT, the gain ratio is almost unchanged. Either the tear-drop's height and width at MT and RT are constant for increased l_2 or they are changing in a similar trend resulting in the relatively flat gain ratio for increased l_2 . To conclude, assuming that spherical aberration is not affected by the substrate temperature where refractive index and NA are unchanged, inhibition of self-focusing is the unique reason for the beneficial gain of HT over MT case, as confirmed by the results of Section 3.4.1.

A quantitative assessment of the general trends in Figures 3.13-3.15 for the % of measurements that show an increase in width and/or height is introduced in Table 3.2. For each pulse energy entry, the measurements tab is for all scanning speeds that show an increase in a topology metric. For all cases, the % of measurements with increase in height and width are higher for the HT case compared to the MT case, having less uncertainty for HT measurements as well. With the exception of the 4.5 μJ pulse energy, the pulse energy of 6, 10, 14 and 18 μJ have higher % of increase in topology range for the HT compared to the MT condition. In addition, the % increase in width is higher compared to the % of increase in height for all pulse energy levels, which decreases the aspect ratio of the tear-drops and is beneficial for glass-to-glass welding as an example of laser application for internal modification.

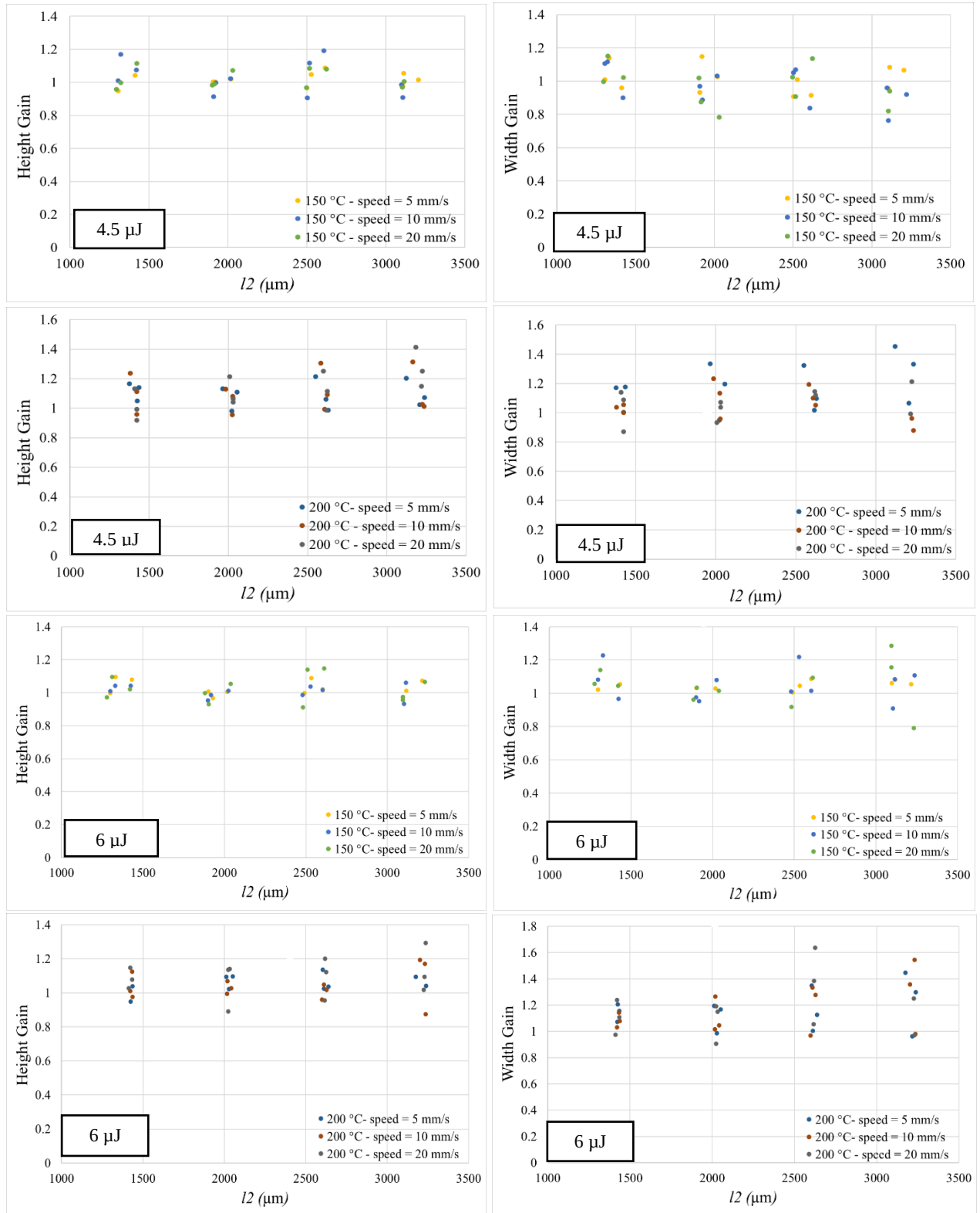


Figure 3.13 Tear-drops' height and width gain ratio for 4.5 and 6 μJ at MT and HT

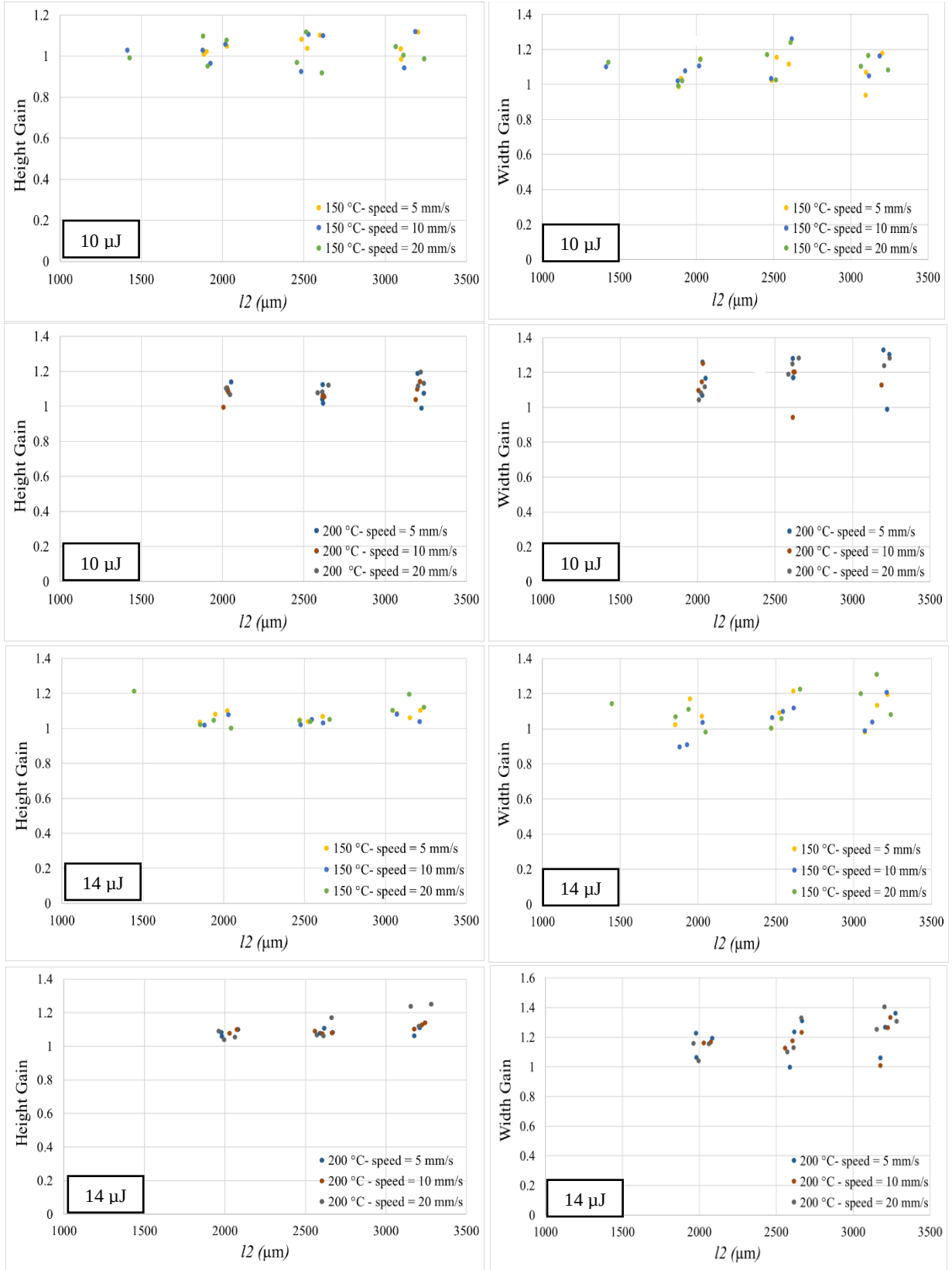


Figure 3.14 Tear-drops' height and width gain ratio for 10 and 14 μJ at MT and HT

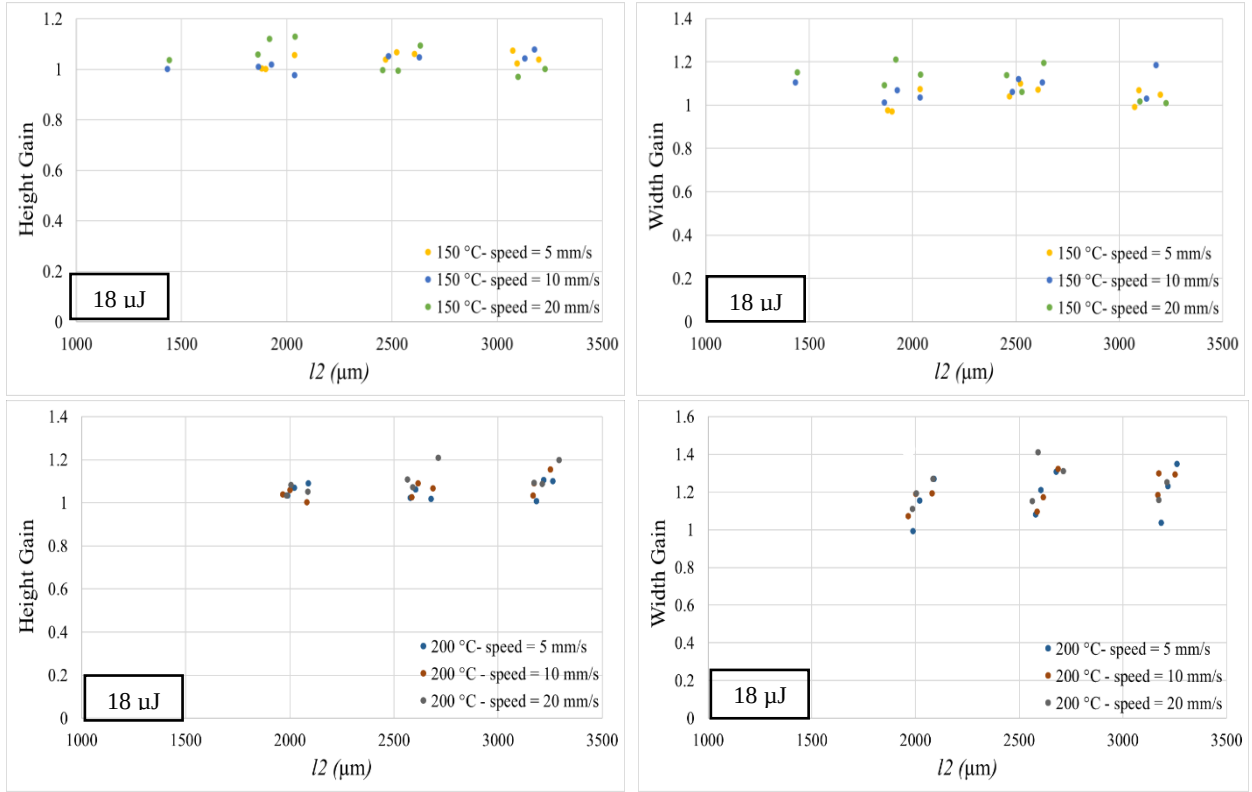


Figure 3.15 Tear-drops' height and width gain ratio for 18 μJ at MT and HT

Table 3.2 Percent of measurements having an increase in width and/or height for each category

Pulse Energy (μJ)	Case	Measurements	Morphology	Range of % Increase
4.5	MT	30 %	Height	5.7 % - 32.9 %
4.5	MT	30 %	Width	3.0 % - 15.1 %
4.5	HT	60 %	Height	6.4 % - 41.1 %
4.5	HT	50 %	Width	8.8 % - 45.3 %
6	MT	30 %	Height	4.3 % - 14.7 %
6	MT	22 %	Width	8.8 % - 28.8 %
6	HT	40 %	Height	8.9 % - 29.2 %
6	HT	55 %	Width	13.1 % - 63.6 %
10	MT	60 %	Height	2.0 % - 11.9 %
10	MT	60 %	Width	7.5 % - 26.1 %
10	HT	60 %	Height	8.0 % - 19.7 %
10	HT	80 %	Width	9.2 % - 32.9 %
14	MT	40 %	Height	7.0 % - 21.4 %
14	MT	40 %	Width	10.1 % - 31.0 %
14	HT	70 %	Height	7.6 % - 25.2 %
14	HT	77 %	Width	12.1 % - 40.5 %
18	MT	30 %	Height	6.0 % - 13.0 %
18	MT	30 %	Width	9.2 % - 33.0 %
18	HT	65 %	Height	5.4 % - 21 %
18	HT	82 %	Width	10.3% - 41.2 %

3.5 Conclusions

Internal glass modification using femtosecond pulses where self-focusing does not dominate under high temperature conditions for 10 mm thick borosilicate substrates is investigated in the current work. Pulse energy, scanning speed and distance have the ranges of 4.5 – 18 μJ , 5 – 20 mm/s and 9.56 – 10.76 mm, respectively. The thick borosilicate glass slabs are preheated to two levels: MT and HT of $\sim 150^\circ\text{C}$ and $\sim 200^\circ\text{C}$, respectively. A significant self-focusing-assisted color center formation is observed for the femtosecond cases at RT and moderately at MT condition. However, when a temperature level of HT is used, the color center formation is inhibited/relaxed, suggesting that the $\sim 200^\circ\text{C}$ temperature has the effect of making self-focusing in borosilicate glass insignificant. The mechanism of color center formation under RT and MT cases is self-focusing where the discoloration is not observable when picosecond pulses are scanned with 10 ps, 18 μJ and 1.3 MW of pulse duration, pulse energy, and peak power, respectively.

In the femtosecond and picosecond cases, a multi-photon absorption mechanism is significant where tear-drops and color centers happen when the peak power is higher than the critical peak power or when the substrate temperature is within the RT to MT range. The derived analytical model determines the focal position l_2 for the deepest point in the focal range, which matches the experimental measurements of l_2 in the distance of 9.56 – 9.96 mm for the HT case where self-focusing is not significant. However, the predicted l_2 underestimates the measurements for the distance of 10.76 and 10.36 mm by 30 – 50 μm . Further investigation is required to determine the mechanism and manipulation for this deviation.

Measurements at HT show higher consistency and range of increase for width gain compared to height gain ratio. For the HT cases, when pulse energy is increased in the range of

4.5 – 18 μJ , the maximum width increase is 10, 100, 67, 60 and 96% higher compared to the maximum height increase. The overall qualitative result is that tear-drop's aspect ratio can be decreased when pulse energy is increased for the HT case when self-focusing is inhibited, alluding to the potential benefits in applications for glass-to-glass welding.

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Chapter 4 . Transmission Laser Welding of Preheated Glass

Substrates

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Abstract

Tear-drop morphology and welding quality using a picosecond laser beam focused at the interface between preheated glass substrates are investigated. Focal position inside preheated borosilicate substrates is predicted utilizing a thermo-optical model considering temperature-dependence of refractive index and thermal dilatation. The model is validated using focal position measurements of tear-drops generated by a 10 ps laser beam scanned thorough-thickness of 10 mm thick borosilicate substrates at room temperature and 150 °C. The validation tear-drops are created using a pulse energy of 4.5, 6, 10, 14, and 18 μJ , and scanning speed of 5, 10, and 20 mm/s. Four focal positions are considered for each pulse energy, speed and temperature utilizing a distance between the lens and the glass surface in the range of 9.56 to 10.76 mm with a step of 0.4 mm. Thermal dilatation during experiments is simulated by finite element analyses using temperature profiles extracted from thermal camera images. The effects of temperature on the tear-drop morphology are assessed by defining a height (width) gain ratio. Welding experiments of 1 mm thick glass slides are performed at room temperature, and 150 °C using similar conditions to the tear-drop experiments. It is found that in the focal range of 1.6 to 3.2 mm and using a 14 μJ pulse energy, the width and height gain are more than 18% and 13%, respectively. At 150 °C, tear-drop's aspect ratio is enhanced by around 25% at a focal position of 1 mm for the pulse energy of 4.5, 6, 10 μJ and speed of 5 mm/s. Higher pulse energy shows less aspect ratio enhancement due to excessive growth in tear-drops' height in addition to width.

4.1 Introduction

When a laser beam is focused inside a transparent material with an intensity exceeding the threshold, non-linear absorption takes place leading to a localized melting initiated at the focal position. Multiple phenomena contribute to the laser energy absorption: multiphoton, tunneling,

avalanche and plasma [1]. Due to linear and non-linear absorption, plasma is created, which can also absorb laser energy leading to plasma growth in the laser incidence direction. When accumulated heat inside the laser modified region diffuses, the heat affected region is created forming a tear-drop shaped modification [2]. Laser glass welding utilizing the nonlinear absorption has multiple applications such as MEMS hermetic sealing, sensor packaging for RF medical implants, and welding glass end-caps to optical fibers [3]. Such applications utilize laser welding because it is localized, contactless and does not require additional adhesive material [4]. The first attempt to weld silica glass was by Tamaki et al. [5]. Welding parameters were 880 nm, 130 fs and 1 kHz of wave length, pulse duration, and repetition rate, respectively. The study of Tamaki revealed that initial gap prior to welding is the critical parameter for a successful joining. Soda lime glass samples were welded by a 13 ps pulse duration and 50 kHz repetition rate laser utilizing a multi-scan laser processing by Jia et al. [6]. The irreversible expansion of the modified glass was considered the major contributor for the welding. The process involved adjusting the focal position as the multi-scan process was repeated for 10 to 25 scans. An investigation to weld fused silica glass without optical contact at the interface was conducted by Chen et al. [7] using a 5.9 ps, 1030 nm, 400 kHz, and 2.45 to 4.43 μ J of pulse duration, wave length, repetition rate and pulse energy range, respectively. Tear-drop modifications were captured and analyzed using counts of 280–2400 pulses. A correlation was established between tear-drop volume, geometrical focus and number of pulses. Zhao et al. [8] demonstrated the welding of fused silica glass with ~ 10 μ m gap by a low numerical aperture $NA = 0.052$ lens using a laser of 8 ps and 400 kHz. Due to the high sensitivity to laser parameters, a numerical model was built to predict the shift of the bottom tip location of the tear-drop. Chen et al. [9] investigated the welding of borosilicate glass using an ultrahigh repetition rate of 50 MHz and pulse duration of 10 ps. Nonlinear absorption was

enhanced by the repetition rate resulting in increased tear-drop size. The formation of radial cracks was suppressed by the beam reshaping when the major axis of the focused beam was parallel to the scan direction. Laser scanning parameters were optimized and shown to produce a maximum bulge of $\sim 21\text{ }\mu\text{m}$. Most recently, Chen et al. [10] introduced an intermediate layer of water between non-optical contact silica glasses prior to welding using a laser of repetition rate of 1087 kHz and pulse duration of 300 fs. Two glass samples were clamped by applying a pressure using a fixture for the welding case of intermediate water layer and compared to the case where no water layer was applied. The shear strength of the welded workpieces was 1.4 times greater when intermediate water layer was used due to gap reduction up to $2.6\text{ }\mu\text{m}$ owing to water surface tension and differential pressure. In a recent work of Zhang et al. [11], a Bessel beam was used for welding aluminosilicate glass. The focused Bessel beam relaxed the focal position uncertainty assuming a negligible spherical aberration. Spindle-shaped modifications were created using these parameters: 1 MHz, 1.2 ps, $2.75\text{ }\mu\text{J}$ and 1-4 mm/s. The focal tolerance using the Bessel beam at high repetition rate was claimed to be 3 times higher compared to a Gaussian beam. Chen et al. [12] investigated the utilization of multifocal beam shaping for borosilicate glass welding. This welding was analogous to focus multiple beams spaced by a horizontal distance. The number of foci and the separation distance were optimized experimentally to weld naturally stacked glass using a laser of 46 nJ, 50 MHz, and 1 mm/s. The shear force of the welded glass using 40 scans of 5 focal points spaced by $16\text{ }\mu\text{m}$ was around 1.3 kN compared to 0.66 kN when 40 scans of a single focus point were used.

Despite the extensive research that has been reported so far, the effect of preheating on laser welding has not been considered in the literature. In this work, welding and tear-drop experiments are conducted using 10 ps laser pulses having a pulse energy of 4.5, 6, 10, 14, 18 and

20 μJ , and scanning speed of 5, 10, and 20 mm/s. Welding experiments are conducted to join preheated glass slides of 1 mm thickness to enhance the tear-drop aspect ratio. Welding strength is also expected to improve due to tear-drop's morphology enhancement. Since laser welding requires precise beam focusing, the authors' previously developed analytical model [13] of focal position's prediction is used to determine the focal position inside thick borosilicate glass samples of ~ 10 mm at a temperature of ~ 150 °C (MT) and room temperature (RT). In this work, the distance D refers to RT conditions, unless it is stated.

4.2 Focal Position Model: Effects of Temperature

Laser focal position depends on the distance between the objective lens and the glass surface D^{RT} , the refractive index of glass $n_2(RT)$ and the numerical aperture $\text{NA} = \sin \theta_l$ of the lens, as shown in Figure 4.1 (left). However, when temperature is increased from RT to MT, thermal dilatation makes the glass thicker by Δl_1^{th} and induces a change in refractive index from $n_2(RT)$ to $n_2(MT)$, as shown in Figure 4.1 (right).

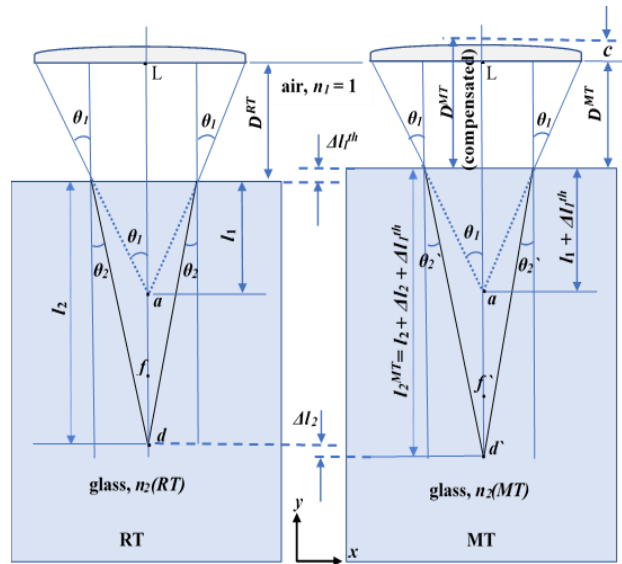


Figure 4.1 Effect of preheating on the focal position

The focal length FL is the distance between a and L . Figure 4.1 shows a scenario when a laser beam is focused at point d at RT where the depth of focus inside glass l_2 determines the focal position. When a laser beam is focused inside a similar glass workpiece but at MT, the focal position d' is shifted by Δl_2 compared to the RT case and θ_2 is evolved into θ_2' .

In Figure 4.1 (left), FL of the lens, the distance D^{RT} , and the focal position l_2 are related by [13]:

$$FL = D^{RT} + l_1 = D^{RT} + l_2/b(RT) \quad \text{Eq. (1)}$$

where $b(RT)$ is a temperature-dependent aberration parameter that can be determined using the equation:

$$b(RT) = \frac{l_2}{l_1} = \frac{\tan \theta_1}{\tan \theta_2} = \tan (\sin^{-1} NA) / \tan (\sin^{-1} \frac{NA}{n_2(RT)}) \quad \text{Eq. (2)}$$

where l_1 determines the geometrical focus in air. Details of deriving Equation 2 can be found in [13]. For the MT case in Figure 4.1, $b(MT)$ can be written as:

$$b(MT) = \frac{l_2 + \Delta l_2 + \Delta l_1^{th}}{l_1 + \Delta l_1^{th}} \quad \text{Eq. (3)}$$

Substituting for l_2 and l_1 from Equation 1 into Equation 3, Δl_2 is determined, which represents the shift in focal position when the glass at MT is cooled down to RT:

$$\Delta l_2 = (FL - D^{RT}) * (b(MT) - b(RT)) + \Delta l_1^{th} * (b(MT) - 1) \quad \text{Eq. (4)}$$

It should be noted that up on cooling down to room temperature, differential contraction takes place. The top surface of glass shrinks back to the original position in the y-direction. This also happens at the y-position of point d' ; but this difference is around 5% of Δl_1^{th} , which is relatively small and can be neglected as determined by finite element simulation (FEA) in the authors' previous work [13].

Equation 4 lists two sources of focal position shift due to thermal effect: refractive index variation and thermal dilatation. The refractive index variation generally has a larger effect than the dilatation. When paraxial rays are considered in the analysis of focal position prediction, the focal position lies in an aberration range, and the closest position to the glass surface coincides with the paraxial focus. For the current work, assuming that the focal positions at RT and MT coincide with the paraxial foci and following the derivation of the foci range in [14], the predicted focal position l_2 is adjusted by a factor of $n_2(T)/b(T)$. The paraxial foci for RT and MT are f and f' as shown in Figure 4.1.

The apparent correlation between Δl_2 and refractive index change is linear if $b(MT)$ changes linearly with temperature when Δl_1^{th} is relatively small. For specific types of glass such as N-PK51 [15] and borosilicate 7059 [16], refractive index decreases with temperature increase, which can decrease the focal position depth.

4.3 Tear-drop and Welding Experiments

Two sets of experiments are performed: tear-drop modifications and welding. For both sets, picosecond laser pulses of 1028 nm wave length, 10 ps pulse duration, and 200 kHz repetition rate, are utilized. Pulse energy and scanning speed of 4.5, 6, 10, 14, 18 and 20 μ J, and 5, 10, and 20 mm/s are used. Focal position is controlled by adjusting the distance D between the lens and the glass surface using an objective lens of NA = 0.3.

For the tear-drop experiments, a thick borosilicate glass of $50.8 \times 50.8 \times 10$ mm is heated up using a heat gun, as shown in Figure 4.2. Laser is scanned through-thickness of the thick glass in x-direction. The thermal expansion effects are investigated at ~ 150 °C (MT) and compared to room temperature (RT). Four focal positions are considered for each pulse energy, speed and temperature by adjusting D in the range of 9.56 to 10.76 mm using a step of 0.4 mm. The glass

substrate temperature is measured by a thermal camera where images are captured and processed for the FEA. Three replicates are used for the tear-drop experiments. Welding experiments are performed utilizing the setup shown in Figure 4.3.

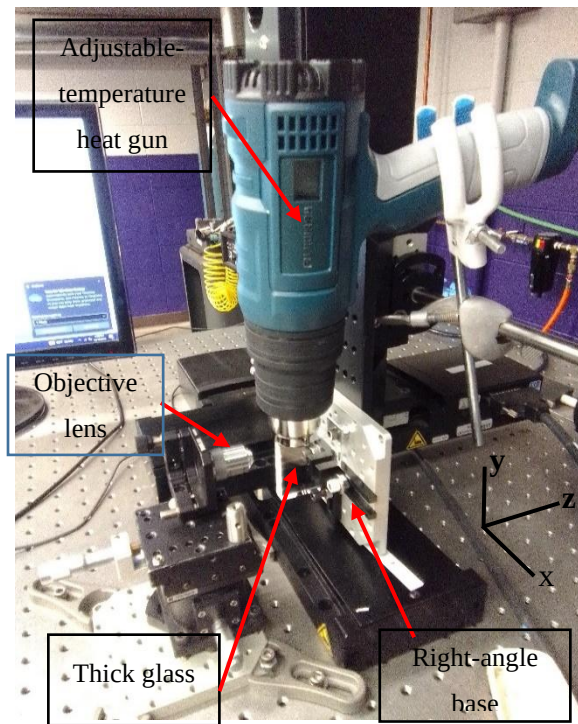


Figure 4.2 Setup of tear-drop modification

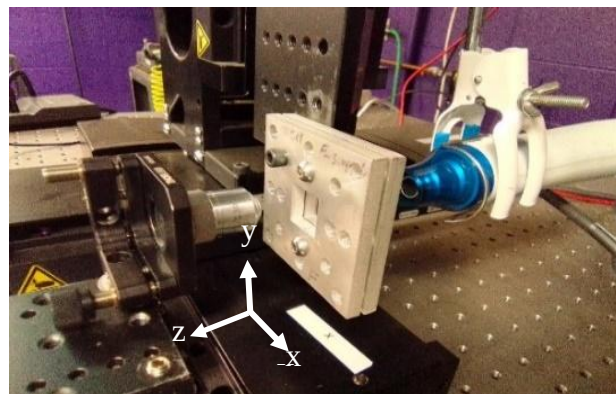


Figure 4.3 Experimental setup of preheated glass welding

A two-plate aluminum fixture with dimensions of 3" (76.2 mm) $\times$ 3" (76.2 mm) $\times$ ¼" (6.35 mm), has holes to apply a holding force through bolts. The fixture has a central window of 1" (25.4 mm) $\times$ 1" (25.4 mm) to pass the laser beam for welding two pieces of glass slides of 1 mm thickness sandwiched between the plates. The clamping force in the current experiments does not produce full optical contact between the glass workpieces, with only limited optical contact is seen at the center of the workpieces for some cases. The glass slides are cut to 1.5" (38.1 mm) $\times$ 1" (25.4 mm) $\times$ 0.039" (1 mm) samples, and the edges are ground and cleaned using acetone and a lint-free tissue prior to joining. Welding procedure starts when two samples are stacked together, placed between the aluminum plates that are secured by two hand-tightened bolts to avoid excessive pressure while welding at MT. The setup is attached to the stage shown in Figure 4.3. The distance between the lens and the outer glass sample is optimized to focus the beam at the interface between the glass samples. A heat gun is used to heat up the backside glass sample for 3–4 minutes. Laser scanning is initiated by moving the stage in the x-direction when the thermal camera measures a maximum temperature of around 140 °C.

Three replicates are welded per combination of pulse energy, scanning speed and temperature, as listed in Tables 4.1 and 4.2. Welding is considered successful when at least three replicates are consistently joined after the samples are taken out of the fixture. Tables 4.1 and 4.2 list the process parameters window of welding at RT and MT, respectively. All samples are welded using 7 scans spaced by 0.5 mm in the y-direction and having an increment in distance D of 5 μ m between adjacent scans. First, the RT welding is performed, and results show that the 4.5 and 6 μ J pulse energy are not repeatable to produce consistent welds. For the MT conditions, the 20 μ J pulse energy produces damage and cracking during welding. Similar cracking during welding is noticed for the 14 and 18 μ J using 5 mm/s. These combinations are excluded from the competition

for the processing window determination, and the acceptable processing parameters for comparison between RT and MT are highlighted in Tables 4.1 and 4.2.

Table 4.1 Process parameters of welding at RT

Speed (mm/s)	Pulse Energy (μJ)				
	4.5 & 6	10	14	18	20
5	X (not repeatable)	✓	✓	✓	X (excluded by MT)
10	X (not repeatable)	✓	✓	✓	X (excluded by MT)
20	X (not repeatable)	✓	✓	✓	X (excluded by MT)

Table 4.2 Process parameters of welding at MT

Speed (mm/s)	Pulse Energy (μJ)				
	4.5 & 6	10	14	18	20
5	X (excluded by RT)	✓	X (glass breaks during laser scan)	X (glass breaks during laser scan)	X (glass breaks during laser scan)
10	X (excluded by RT)	✓	✓	✓	X (glass breaks during laser scan)
20	X (excluded by RT)	✓	✓	✓	X (glass breaks during laser scan)

4.4 Thermal Dilatation Simulation

For the tear-drop experiments, thermal dilatation of the 10 mm thick samples is simulated by FEA to determine the z-displacement of the glass surface facing the laser. The results were reported in the authors' previous work [13]; Δl_1^{th} was around 16.5 μm and that the differential contraction was around a couple of microns that can be neglected.

4.4.1 Thermal Dilatation Simulation for Welding Setup

Thermomechanical FEA of the fixture shown in Figure 4.3 using mapped temperature and displacement boundary conditions (BCs) is implemented. The distribution of temperature profile is interpolated by COMSOL using pixel to pixel temperature measurements of the thermal camera images shown in Figure 4.4.

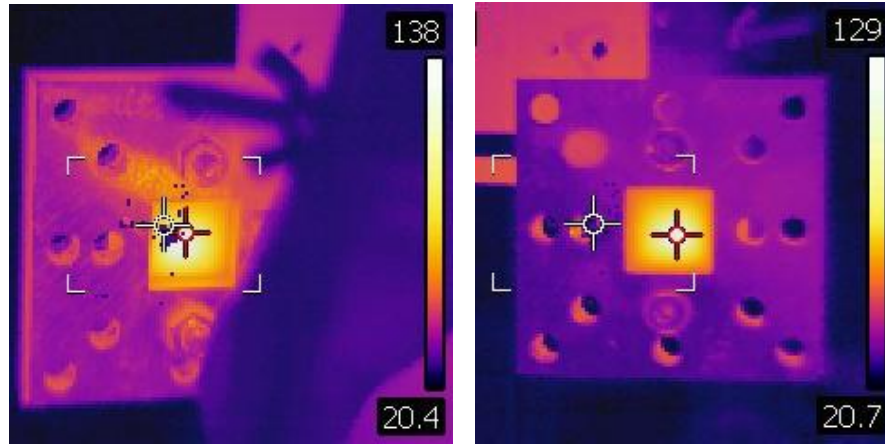


Figure 4.4 Thermal images: backside, facing the heat gun (left) and front, facing the laser beam (right)

The IRimage software [16] is plugged into imageJ to process the thermal images and extract the pixel-wise temperature measurements for the backside and front aluminum plate and glass workpieces. The raw temperature measurements of the front plate show a relatively constant temperature in the level of RT, thus a constant temperature BC for the front plate is assigned. A steady-state thermomechanical simulation is conducted utilizing the extracted temperature fields and by assigning fixed displacement BCs for the bolts' locations. Figure 4.5 shows the surface temperature BCs of the glass samples interpolated by COMSOL.

A minor difference of around 2 °C in the maximum temperature can be seen between the BCs in Figure 4.5 and the thermal images of Figure 4.4 due to the linear interpolation functions

used to fit the data. The mechanical and thermophysical properties of borosilicate glass and aluminum are taken from the literature [17, 18].

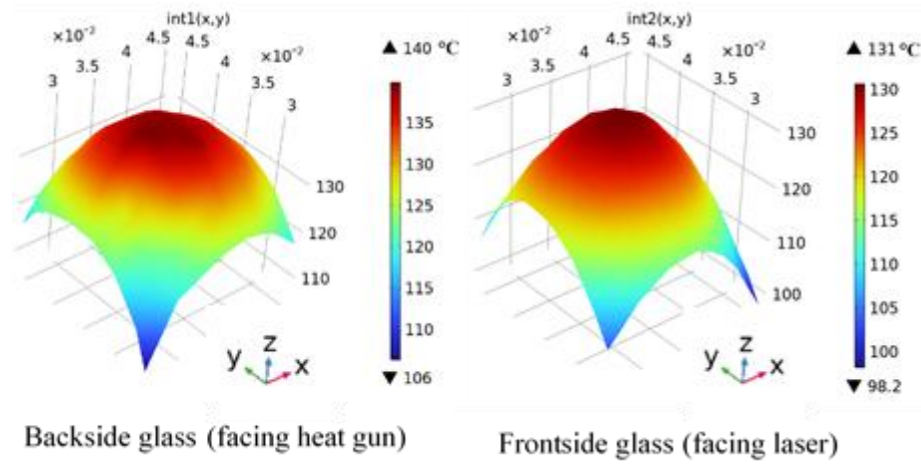


Figure 4.5 The interpolated temperature profiles

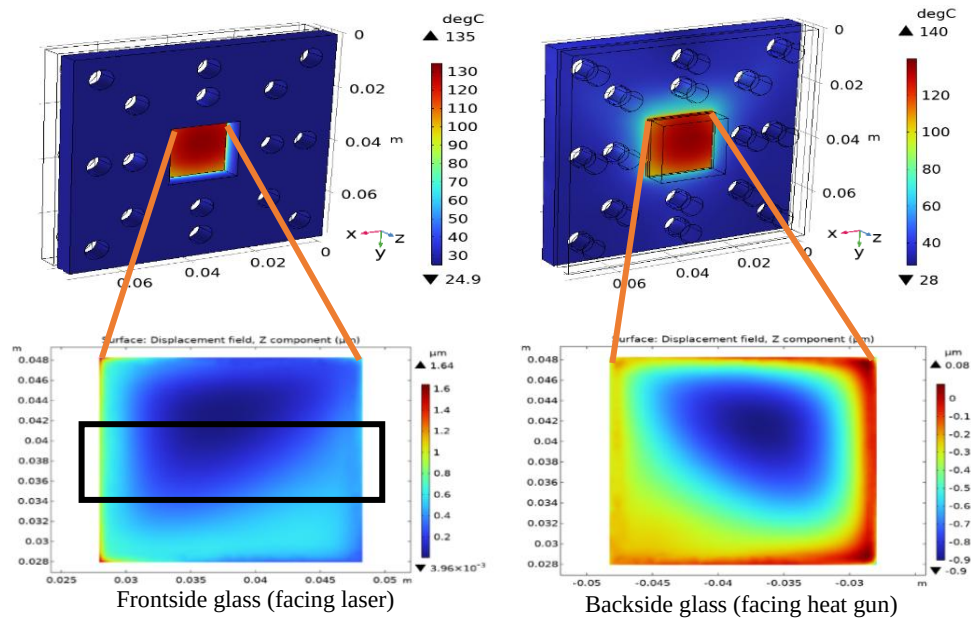


Figure 4.6 Temperature and z-dilatation distribution of the glass

Figure 4.6 shows the steady-state temperature and z-dilatation distribution of the front and backside plates and glass. The front glass that faces the laser has a negligible dilatation of less than 1 μm, especially in the area enclosed by the dark rectangle where laser scanning takes place.

Accordingly, the dilatation can be neglected in Equation 4 for the welding experiments. However, the tear-drop experiments show a higher dilatation of 16.5 μm , which is considered for the validation of experimental results of the focal position in Section 4.5.2.

4.5 Tear-drop Modification Experiments

4.5.1 Refractive Index at RT and MT

For quantitative comparisons, refractive indices of the thick borosilicate glass are estimated at RT and MT. Previous work's results [13] showed that the focal position shifts deeper when the glass slabs were preheated to 150 °C and 200 °C compared to RT. Qualitatively and according to Equation 4, the focal position shifts deeper when the refractive index increases upon heating during laser scanning. Uncertainty analysis is performed by assuming initial values of the refractive indices and changing them to minimize the uncertainty between the predicted and measured focal position at RT and MT. The refractive index estimations at RT and MT are $n_2(\text{RT}) = 1.418$ and $n_2(\text{MT}) = 1.468$. These estimates show an increase in the refractive index of around 4% at MT. Reported measurements in [19] showed that refractive index increases by around 0.03 % and 4 % for BK7 and fused silica, respectively, when temperature is raised to 150 °C. The current work's estimates of refractive indices are within a reasonable range of increase due to preheating to 150 °C.

4.5.2 Focal Position

Figure 4.7 shows the tear-drops for MT using a pulse energy of 4.5, 6, 10, 14, and 18 μJ . It shows that changing pulse energy and scanning speed levels does not shift the focal position remarkably. Focal position l_2 is measured from the tip of the plasma modified region to the outer glass surface as shown. For borosilicate glass, the critical peak power is 3.2 MW for a laser of 1046 nm wavelength [20]. The maximum peak power in the current work is around 1.3 MW when

30% of objective lens power loss is considered, which is lower than the critical peak power for self-focusing. Measurements of focal position for the three replicates are performed using imageJ as shown in Figure 4.8.

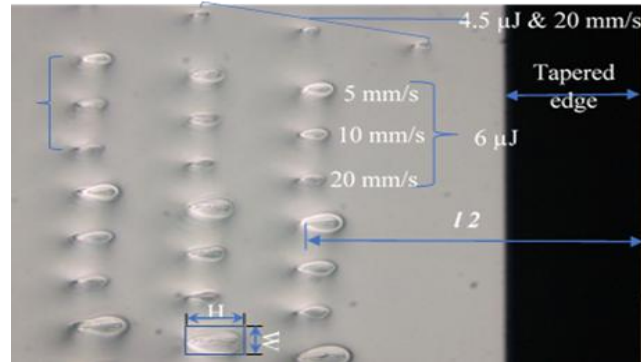


Figure 4.7 Tear-drops: focal position and aspect ratio at MT

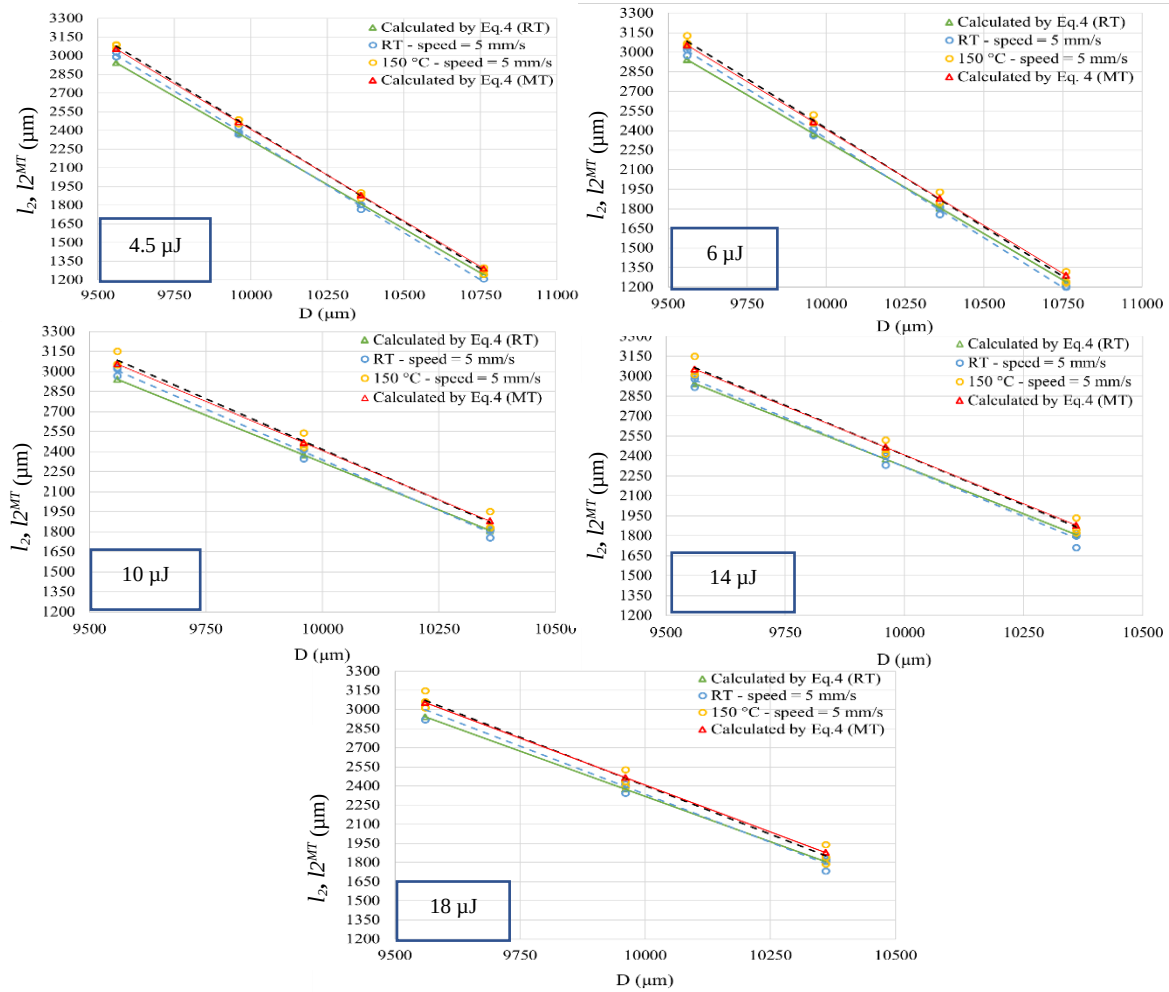


Figure 4.8 Focal position of tear-drops: predictions and measurements at RT and MT

The focal position is calculated using Equation 4 for the RT and MT conditions by considering the estimated refractive indices of Section 4.5.1 and the thermal z-dilatation of 16.5 μm determined in Section 4.4.1. The linear trends of l_2 measurements match the predictions of Equation 4 for all pulse energy levels as the distance D is increased. The root-mean-square error (RMS) between the measured and predicted l_2 is listed in Table 4.3 for the RT and MT conditions. Since three replicates are considered, the RMS error is calculated between the mean of the measured l_2 at any distance D and the corresponding calculated l_2 using Equation 4.

The results of Table 4.3 show that the RMS error is around two times for RT compared to MT for all pulse energy levels. The major source of this discrepancy is attributed to the estimation uncertainty in refractive indices. However, the highest RMS error in predictions of Equation 4 is around 3% and 1% of the measured l_2 for the RT and MT conditions, which is considered insignificant for a linear model of laser beam propagation.

Table 4.3 RMS error of the calculated and measured l_2

Pulse Energy	RMS error at RT	RMS error at MT
4.5	46.5	16.1
6	46.9	20.6
10	39.3	19.09
14	23.2	9.2
18	34.9	18.9

4.5.3 Tear-drop's Morphology

Effects of preheating prior to welding on the tear-drop height (width) and aspect ratio are analyzed using optical microscopy. To account for the temperature effect, a gain ratio is defined as the ratio between the tear-drop height (width) measured at MT to the respective height (width) measured at RT. The aspect ratio is defined as the ratio of a tear-drop's height to width. Figure 4.9 shows the height and width gain of 4.5 and 14 μJ for all speeds in the range of focal position of

$1.2 \text{ mm} < l_2 < 3.2 \text{ mm}$. The gain measurements are filtered by 1st and 3rd quartile outlier analysis to exclude measurements abnormally larger or smaller compared to the data bounds.

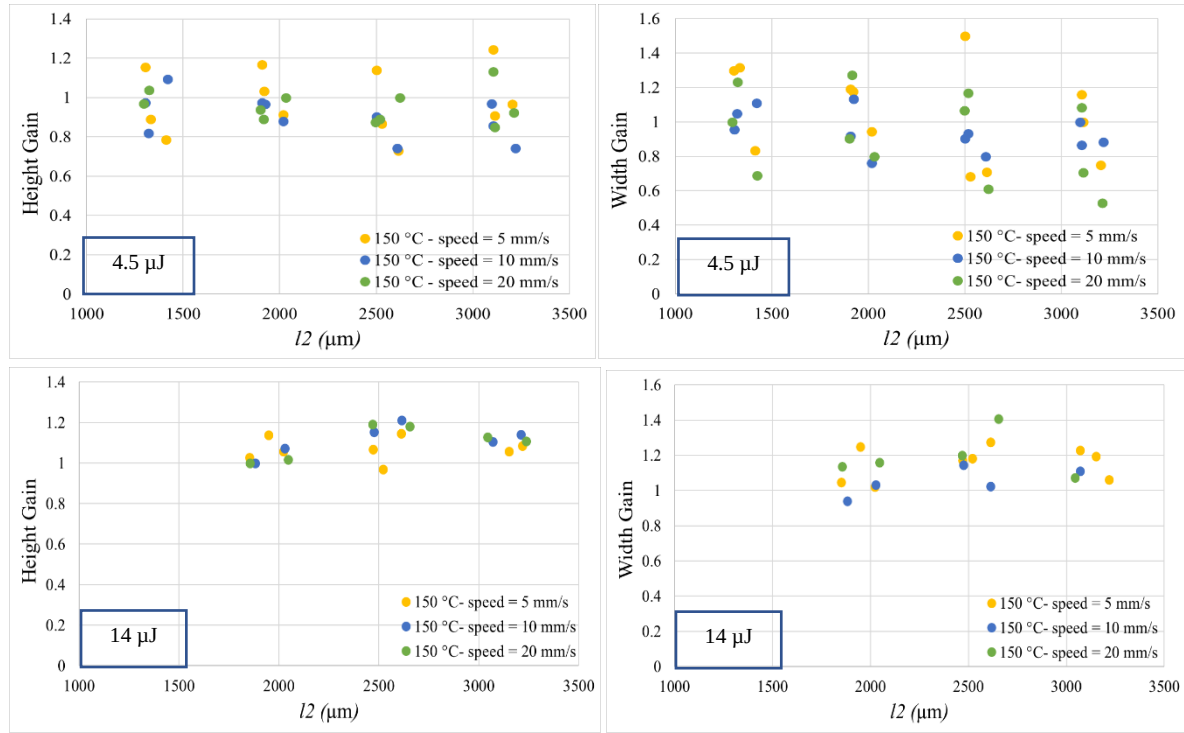


Figure 4.9 Tear-drops' height and width gain ratio for 4.5 and 14 μJ

In Figure 4.9, scattered measurements result from modification defects like longitudinal and transverse microcracks [21] and glass refractive index inhomogeneity [22], and the uncertainty in measuring the tear-drops. To highlight the effect of temperature, Figure 4.10 shows a breakdown of the measurements by pulse energy into five groups. Each group has all speeds and distance D levels included.

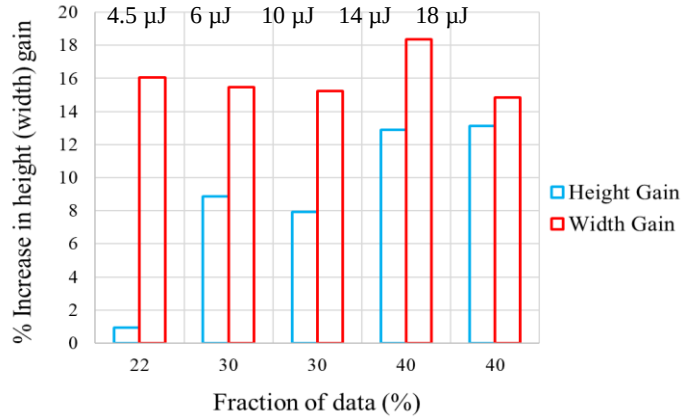


Figure 4.10 Height and width gain chart for pulse energy effect

For all pulse energy, width gain is higher compared to height gain when temperature is increased to MT, but the difference between height and width gain is decreasing for the 14 and 18 μJ . This means that higher pulse energy shows less aspect ratio enhancement due to increase of tear-drop height in addition to width.

To address the effects of the temperature on the tear-drop's aspect ratio, Figure 4.11 shows the effects of temperature on aspect ratio for 6 and 14 μJ at a scanning speed of 5 mm/s. Other cases of 4.5, 10 and 18 μJ have similar trends to the 6, and 14 μJ , respectively, which are not shown in Figure 4.11. For all pulse energy and temperature, the aspect ratio increases as depth of focus is increased, which does not support a good welding quality. For the pulse energy of 4.5, 6 and 10 μJ , the aspect ratio is enhanced (lowered) in the focal range of $1100 \mu\text{m} < l_2 < 2000 \mu\text{m}$ due to preheating. For the focal position $l_2 > 2000 \mu\text{m}$, the RT condition has a lower aspect ratio compared to the MT condition. For 14 and 18 μJ , a higher temperature shows an enhanced aspect ratio for all depths of focus. Since the welding experiments have glass slides of 1 mm thickness, the extension of the aspect ratio trends in Figure 4.11 to a focal depth of 1 mm helps in predicting the expected quality of welding at that level. The expected aspect ratio at MT compared to RT are

enhanced by 25% to 29% for pulse energy of 4.5 to 10 μJ and only 5% for 14 and 18 μJ . This result of aspect ratio is in agreement with the relative increase in gain ratio of Figure 4.10 for pulse energy of 4.5 to 10 μJ , which is higher compared to the 14 and 18 μJ . This can be explained as tear-drop's height and width are increasing simultaneously when higher pulse energy is used, which affects the aspect ratio adversely.

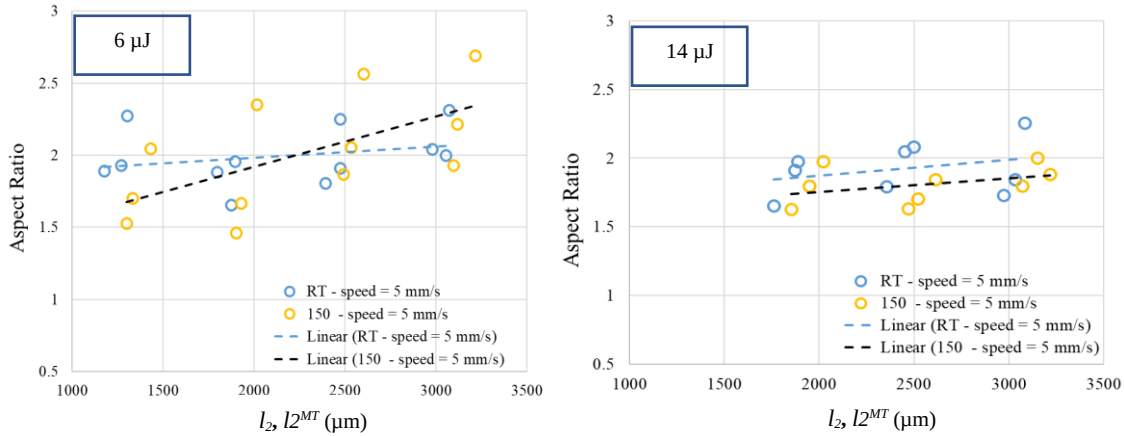


Figure 4.11 Aspect ratio for pulse energy of 6 and 14 μJ at RT and MT

4.6 Compensation for MT Welding

Figure 4.12 shows three cross-sectional micrographs of welding two glass slides of 1 mm thickness each using a pulse energy of 10, 14 and 18 μJ and scanning speeds of 10 and 20 mm/s for the selected process parameters of Table 4.2. The cross-sections are polished prior to microscopy. A compensation parameter, c , can be introduced into Equation 4 to account for the required shift in distance D in the y -direction at MT to bring the focal position l_2 at the interface, which can be seen in Figure 4.1. The compensation can be realized by moving the lens in Figure 4.1 at MT by c in the y -direction and adjusting Equation 4. For the compensated case, the distance D^{MT} can be calculated by $D^{\text{MT}} = D^{\text{RT}} + c - \Delta l_1^{\text{th}}$, where D^{RT} is the focal distance of the RT case, as shown in Figure 4.1. Equation 1 can be rewritten for predicting the focal position at MT as

$l_2^{MT} = (FL - D^{MT})b(MT)$. Additionally, from Equation 3, $l_2^{MT} = l_2 + \Delta l_2 + \Delta l_1^{th}$. Utilizing the last three relations and l_2 from Equation 1, the shift in focal position up on compensation is:

$$\Delta l_2 = (FL - D1) * (b(MT) - b(RT)) + \Delta l_1^{th} * (b(MT) - 1) - c * b(MT) \quad \text{Eq. (5)}$$

Equation 5 is applied to the tear-drop modifications measurements of Figure 4.12 by subtracting Equation 4, which yields $c * b(MT)$. In other words, the difference in focal position between two adjacent tear-drops having similar pulse energy, scanning speed and temperature is $c * b(MT)$. Since a compensation of $c = 60 \mu\text{m}$ is used in the current welding experiments, Figure 4.13 (left) shows a comparison between the measurements and calculations. The refractive indices of the glass slides are assumed as $n_2(RT) = 1.526$ and $n_2(MT) = 1.557$ for the calculations of Figure 4.13.

Figure 4.13 represents the ratio of the measured focal position l_2 at MT to RT for tear-drops having similar pulse energy and scanning speed but a different distance ratio of D^{MT}/D^{RT} . The model's predictions are also included for the basis of comparison. In all cases, the model predicts the corresponding measurement with minimal uncertainty.

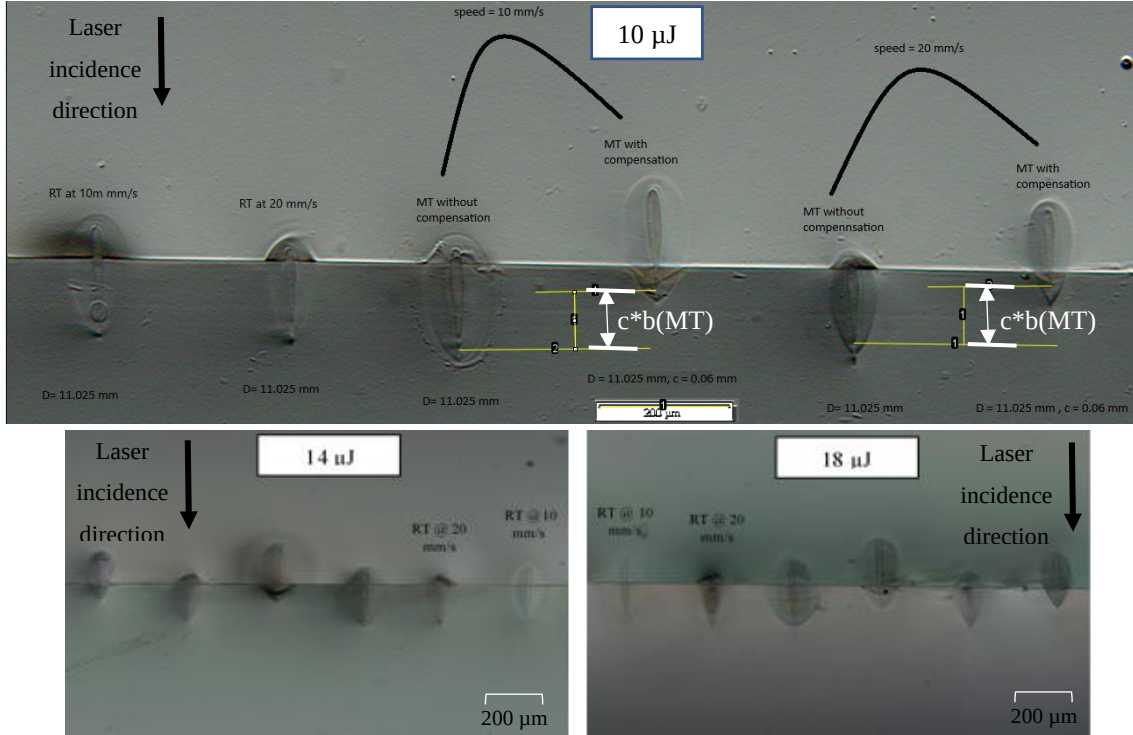


Figure 4.12 A cross-section of weld seams with and without compensation

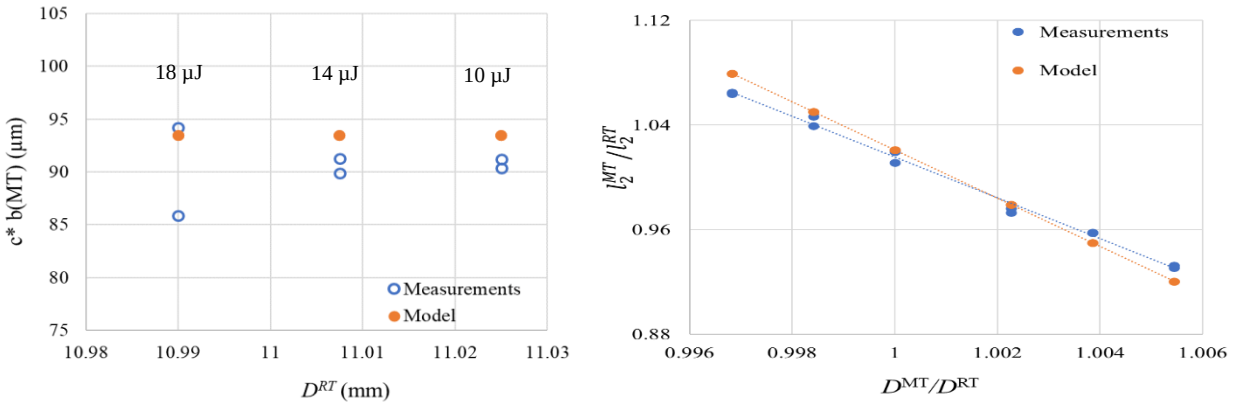


Figure 4.13 Focal position compensation and ratio l_2^{MT}/l_2^{RT} due to temperature effects

4.7 Conclusions

Precise prediction of focal position is a critical step towards high quality welding of preheated glass substrates. The current work introduced an analytical model for focal position prediction at RT and MT conditions taking into account the refractive index variation and thermal

dilatation. The model is validated using focal position measurements of tear-drop experiments where thermal dilatation is around $16.5\text{ }\mu\text{m}$ for 10 mm thick borosilicate glass slabs. Refractive index variation plays a dramatic role in determining the eventual focal position at higher temperatures. The current model is calibrated using a glass material having a higher refractive index at MT compared to RT. It is also expected that the current model can capture temperature effects on focal position for transparent materials that shrink and or have a decreased refractive index at higher temperatures.

Focal position measurements of welding experiments utilizing the compensation feature confirm the viability of the current model where interfaces are present without sacrificing the model generality. Uncertainty in material homogeneity, sample dimensions, welding defects and temperature distribution produce scattering in the refractive index, focal position and tear-drop morphology. The current work introduces a statistical analysis of the measured height (width) gain when substrates are preheated. A general trend shows that using a pulse energy of 4.5, 6, and 10 μJ for a 1 mm focal depth enhances the aspect ratio by around 25% when scanning speed is 5 mm/s. This effect is in agreement with the overall trend of the height (width) gain ratio enhancement over increased pulse energy up to 10 μJ as shown by the respective histograms. For higher pulse energy levels of 14 and 18 μJ and because of the simultaneous growth in tear-drop height in addition to width, the aspect ratio enhancement is marginal. Welding strength is expected to enhance when tear-drop width is increased using preheated borosilicate glass substrates; but this requires further verification through welding tests and shear strength measurements considering the stochastic nature of this application.

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Chapter 5 . Effects of Preheating on Glass Modification and Laser Welding Strength

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Abstract

Glass preheating prior to laser scanning is expected to enhance internal modification morphology; however, its effect on weld seam topology and welding strength have not been investigated. In the current work, the effects of preheating on ultrafast laser (184 fs and 10 ps) internal modification and welding strength of borosilicate glass slides are investigated. For the internal modification experiments, pulse energy of 30-100 μJ and repetition rate of 10 kHz are used by focusing a laser beam at the interface of optically contacted slides at room temperature ($\text{RT} \approx 23^\circ\text{C}$), 150 and 200 $^\circ\text{C}$. Welding is conducted by a pulse energy of 4.5-18 μJ and repetition rate of 200 kHz using pre-clamped glass slides with a scanning speed of 10 mm/s at RT and 150 $^\circ\text{C}$. Also, for welding, optimum number of scans and hatching spacing are identified. Filamentation experiments show that discoloration is not significant when preheat temperature reaches 200 $^\circ\text{C}$. Compared to 10 ps, pulse duration of 184 fs can produce 19 % narrower plasma-modified region at both RT and 150 $^\circ\text{C}$ and 13 % wider heat-affected zone at 150 $^\circ\text{C}$. Welding using optimum conditions of 5 scans and 200 μm hatch, and “crack-free” laser parameters produces an average strength of: 50 ± 3.2 MPa at RT and 40 ± 2 MPa at 150 $^\circ\text{C}$ for 184 fs compared to 35 MPa at RT and 32 MPa at 150 $^\circ\text{C}$ for 10 ps, using 10 replicates each. However, the welding strength upon preheating to 150 $^\circ\text{C}$ using 184 fs is still 25 % higher compared to average reported laser welding bonding strength, while the 10 ps strength is within the reported average. The enhanced welding strength for 184 fs can be attributed to reduced microcracking, especially when “crack free” combinations are utilized.

5.1 Introduction

Transmission laser glass welding utilizes localized melting and gap bridging taking place at interface of contacted transparent surfaces by generating a tear-drop shaped modification [1].

The tear-drop bridges the interfacial gap, producing a permanent weld, mainly due to a plasma-modified core of the tear-drop [2]. Nonlinear absorption triggers the initiation of the tear-drop at the focal position. The tear-drop grows towards the incident laser by avalanche ionization and plasma absorption. The incident intensity at the focal position should exceed the intensity threshold for nonlinear absorption initiation [3].

Optimization of the incident beam parameters is a critical step for producing defect-free (crack-free) weld seams with enhanced tear-drop morphology and strength [4]. Investigations of tear-drop morphology show that tear-drop's height and width are increased when pulse repetition rate is increased in the range of 50 to 1000 kHz. By increasing pulse energy in the range of 1 to 11 μJ and repetition rate, nonlinear absorptivity is also enhanced from 40 to 95% [5]. Decreasing scanning speed can also increase the tear-drop width and height, which is significant when the speed is decreased by orders of magnitude for higher pulse energy levels [6]. Absorption volume simulations of a 20 μJ laser beam using a numerical aperture NA of 0.4 to 1.2 revealed that a constant-width tear-drop can be achieved with nonlinearly decreasing height and aspect ratio of height-to-width, where aspect ratio can reach 1 for NA = 1.2 [7]. The effect of NA on the tear-drop morphology can be even more complicated if the spherical aberration and self-focusing are considered, especially when the focal position is located deeper inside thick glasses [8].

In the authors' previous papers [9, 10], it has been determined experimentally that preheating glass to 150 and 200 °C prior to laser scanning can increase tear-drop height and width. In the current work, the authors first introduce an experimental investigation of the preheating temperature effects on laser absorptivity under self-focusing modification. The width of tear-drop's heat-affected and plasma-modified zone are then investigated at RT and 150 °C for pulse energy of 4.5 – 18 μJ and duration of 184 fs and 10 ps.

Welding defects (cracks) formation can also depend on the initial gap before laser scanning; optical contact with a gap < 200 nm can produce crack-free weld seams as tensile stresses generated by the glass melt are balanced up on solidification [1]. Pre-welding gap between glass substrates can be controlled using mechanical clamping to get partial optical contact where Van-der-Waals forces temporarily join the contacted surfaces [11]. Full optical contact is also achievable by high-quality polishing, but it is technically expensive [12].

A number of parallel scans are recommended to weld glass-to-glass to produce consistent strength measurements [13] and minimize the uncertainty effects stemming from variations in sample surface quality and thickness. Jia et al. [14] investigated welding of soda lime glass and reported a tensile shear strength of 3.3 and 6.3 MPa using 10 and 15 welding scans, respectively, while Chen et al. [2] used 40 scans and reported a 1325 N compression shear force for borosilicate glass. Richter et al. [15] did not report the number of welding scans in their experiments, which corresponded to 72.9 and 39 MPa for welding of fused silica and borosilicate glass, respectively. Zhang et al. [16] utilized a line-to-line (hatch) spacing of 250 μm , producing a shear strength of 10.9 MPa; however, when the spacing was reduced to 120 μm , the strength increased to 17.4 MPa. Tian et al. [17] and Chen et al. [18] used a scan spacing of 50 and 200 μm producing a shear force and strength of 76 N and 18 MPa, respectively. Most recently, Jiang et al. [19] conducted a quasi-synchronous welding using 5 scans of 10 μm hatch, producing a weld compression lap-shear strength of 21.42 and 16.33 MPa for a pre-welding gap of 1 and 5 μm , respectively.

Effects of preheating temperature on the welding quality, and strength have not been investigated in the literature yet. Additionally, hatch spacing effects and its correlation to laser-induced cracking defects have not been reported so far.

In the current work, preheating temperature effects on filamentation modification pattern, absorption rate, and welding defects and strength are investigated. Room temperature, 150 and 200°C are denoted as RT, MT, and HT, respectively. Absorptivity rate is calculated using incident and transmitted pulse energy measurements at three distance levels in the z-direction after the beam exits contacted glass slides at distance: $dl = 103.5$, $dp = 292$, and $dh = 502$ mm. Welding experiments are performed at RT and 150 °C using an optimum hatch distance h and number of scans with 10 replicates for each parameter combination where welding defects are analyzed and the corresponding strengths are determined using a single-lap compression shear test.

5.2 Absorption Rate Determination and Modification Pattern

In the current work, absorption rate measurement is performed by focusing a laser beam at the interface of optically contacted glass workpieces and by measuring the transmitted laser power for a known incident laser power, as shown in Figure 5.1. Absorption rate measurement techniques assume negligible reflection and scattering due to plasma generation [20]. Absorption rate A can be calculated using Equation 1 [21]:

$$A = \frac{E_a}{E_i} = \frac{E_i(1 - R)^2 - E_t}{E_i(1 - R)} \quad \text{Eq. (1)}$$

where E_a , E_i , E_t and R are the absorbed, incident, transmitted pulse energy, and reflectivity of the glass surface, respectively. In the literature, determination of the absorption rate using this approach has been implemented for laser intensities exceeding the threshold [5, 20] where tear-drops are generated, which are technically permanent damages. Under this damage condition, the measured E_t when the tear-drop modification happens could affect the calculated absorption. Heat accumulation can also affect the measurements when pulse repetition rate exceeds 100 kHz. On the other hand, absorptivity can also be measured under conditions of self-focusing as nonlinear propagation inside glass is still happening, which is a general procedure for Z-scan tests [22].

However, for Z-scan self-focusing conditions, the sample's thickness should be smaller compared to the lens' Rayleigh range [23] and the repetition rate should be in the neighborhood of 10 kHz to minimize accumulation heating [24]. Experiments of this section are conducted under self-focusing condition where laser peak power exceeds the critical peak power of the glass but not the tear-drop formation, as discussed below and elaborated in section 5.4.1.

For the calculations of A and glass modification patterns, a laser system of 1028 nm wavelength producing a beam spot size $D = 5$ mm is utilized. The collimated laser beam is focused using a plano-convex lens of focal length $f = 750$ mm at the interface of two optically contacted glass slides of 1 mm thickness each, as shown in Figure 5.1. First, the beam spot size w_0 at the focal plane is measured in air using a BC207VIS camera profiler. The measured spot size is $w_0 = 112.71$ μm , which is only 2.71 μm higher compared to the calculated spot size of $w_0 = 2M^2\lambda f/\pi D$ for a Gaussian beam, where $M^2 = 1.1$. An incident laser beam of 0.3–1 W (30–100 μJ), 184 fs, and 10 kHz for power (pulse energy), pulse duration, and repetition rate, respectively, is used to run the absorption rate experiments at RT and HT. Two optically contacted glass slides are edge-sealed using a commercial glue, then a heat gun is utilized to preheat the slides to 200 °C, which is measured using a thermal camera. Prior to laser scanning, preheating by a heat gun can trigger a background effect on the power meter measurements. These effects are accounted for by subtracting the background measurements from the real measurements after laser exposure. The transmitted power is measured after the beam exits the glass at three levels of z-distance: 103.5, 292, and 502 mm; these are measured from the outer surface of the rear glass slide to the meter, (Figure 5.1). Three replicates for RT and other three for HT are utilized. For each replicate, when a distance level or a pulse energy is reset, a new intact spot of the glass replicate is utilized to create the required modification and acquire the measurements. Commercial borosilicate glass slides of

dimensions $76 \times 25 \times 1 \text{ mm}^3$ under optical contact are utilized for experimental investigations of modification patterns and laser welding. For welding experiments, the glass slides are cut to $38 \times 25 \times 1 \text{ mm}^3$. Material properties are listed in Table 1 below.

Table 5.1 Physical properties of glass slides [27]

Physical Property	Value
Density (g/cm ³)	2.51
Refractive index	1.5
Heat capacity (J/kg. K)	2.2
Coefficient of thermal expansion (1/°C)	7.2×10^{-6}
Melting point (°C)	827

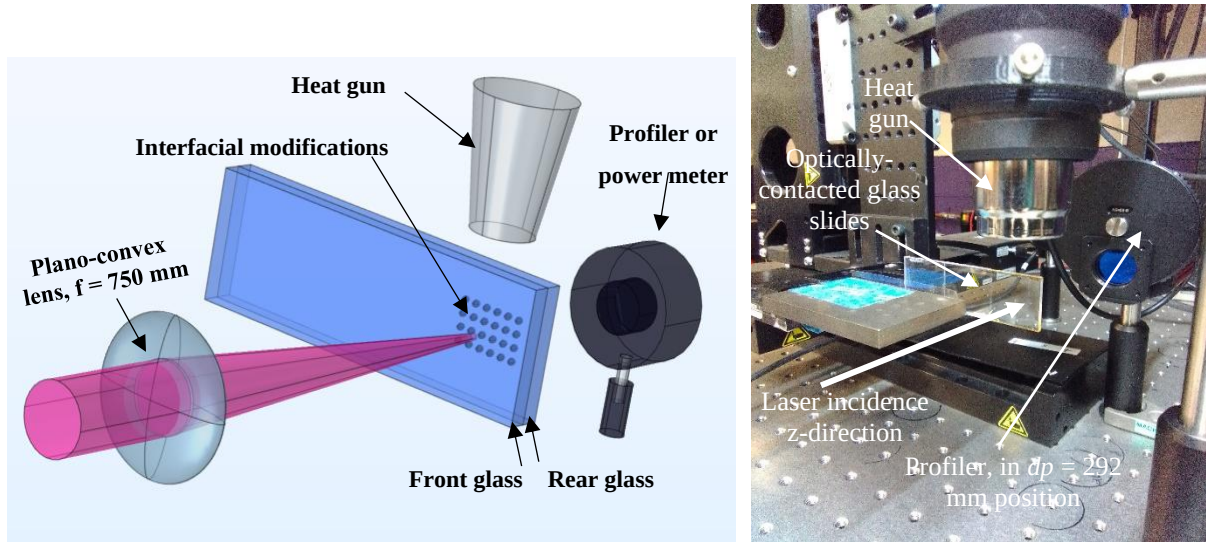


Figure 5.1 Experimental setup for transmitted pulse energy E_t measurements at $dl = 103.5$, $dp = 292$, and $dh = 502$ mm; the setup shows measurement of E_t at $dp = 292$ mm

5.3 Glass Welding and Single-lap Compression Shear Test

Welding experiments are performed to weld two glass slides placed between two aluminum plates as shown in Figure 5.2. Two bolts are used to secure the glass slides at a processing window of $23 \times 23 \text{ mm}^2$ where the bolts are hand-tightened prior to laser scanning. Pre-weld optical contact regions are observed but are limited and varied in location inside the window due to sample-to-

sample variations. First, the distance between the lens and glass slides is optimized by initial welding experiments to locate the focal position at the welding condition. Then, welding experiments are conducted at RT and MT using the combinations of pulse energy, pulse duration, scanning speed, and hatch distance listed in Table 5.2.

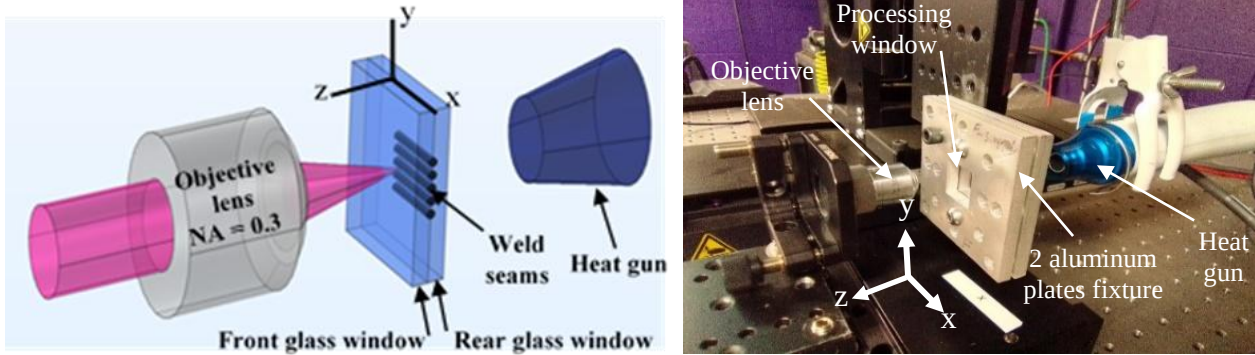


Figure 5.2 Experimental setup of glass welding at RT and MT; for MT the gun heats up the glass slides from rear side which is attached to the stage that scans in x-direction

Table 5.2 Laser transmission welding parameters of preheated glass slides

	Pulse Energy (μJ)				
	4.5	6	10	14	18
Pulse Duration (fs)	184	184	184	X	X
Hatch, h (μm)	200	200	200		
Pulse Duration (ps)	X	10	10	10	10
Hatch, h (μm)		200	200	300	400

The glass temperature upon preheating is monitored by a thermal camera to measure rear and front aluminum and glass surfaces temperature and ensure that it does not build up excessively. First laser scan starts when the maximum temperature of the rear glass reaches around 140 °C, while the maximum temperature may rise to 155 °C upon finishing the last welding scan. More details are included, in addition to FEA simulations in a previous work [10]. Similar procedure is

followed for the internal modification preheating. There have been technical limitations over the pulse energy and preheat temperature levels for a successful welding. For the MT conditions, the 14 and 18 μJ pulse energy produce significant cracking during welding. Similar cracking during welding is also noticed for almost all pulse energy levels for HT conditions. Accordingly, these conditions are excluded.

The selection of h for the 184 fs and 10 ps is optimized to minimize the possibility of crack extension perpendicular to parallel scans for the welded samples to make a fair comparison between fs and ps conditions. Initial welding experiments show that h should be set proportional to pulse energy if the cracks perpendicular to the parallel laser scans should not connect to each other. If these cracks connect, then strength is reduced drastically, which is confirmed by initial strength measurements.

Figure 5.3 shows microscopy images of interface of samples welded by 10 μJ , 10 ps, 200 kHz, 10 mm/s and 15 scans with h of 50, 100, and 200 μm as marked by a, b, and c, respectively. An interesting observation is that cracks produced by the initial scans (1st scan and so on) have extended perpendicular to the weld seams when $h < 200 \mu\text{m}$, which is later selected as the optimum hatch distance. Figure 5.3 (c) shows the effect of using $h = 200 \mu\text{m}$ where cracks do not extend across weld seams in this case, which is also confirmed by other 4 replicates.

A Shimadzu universal testing machine of 5 kN maximum load is used to conduct single-lap compression tests to determine the lap shear strength of the welded slides using an in-house made fixture, as shown in Figure 5.4. A platen speed of 0.02 mm/s is used and the test is stopped after the weld seam failure when a catastrophic drop in shear force happens. Initial samples are welded by 5 and 15 scans; however, testing of the 15-scan welded samples shows failures occur at the bulk glass not the welded interface. Apparently, the weld seams are strong enough causing

bulk glass slides welded with 15 scans to break prior to shearing of the weld seams. Accordingly, the 15-scan welding scheme is not considered for further investigation. Figure 5.3 (c) also shows the determination of width of plasma-modified W_p and heat-affected W_h zone, which will be discussed in Section 5.4.3.

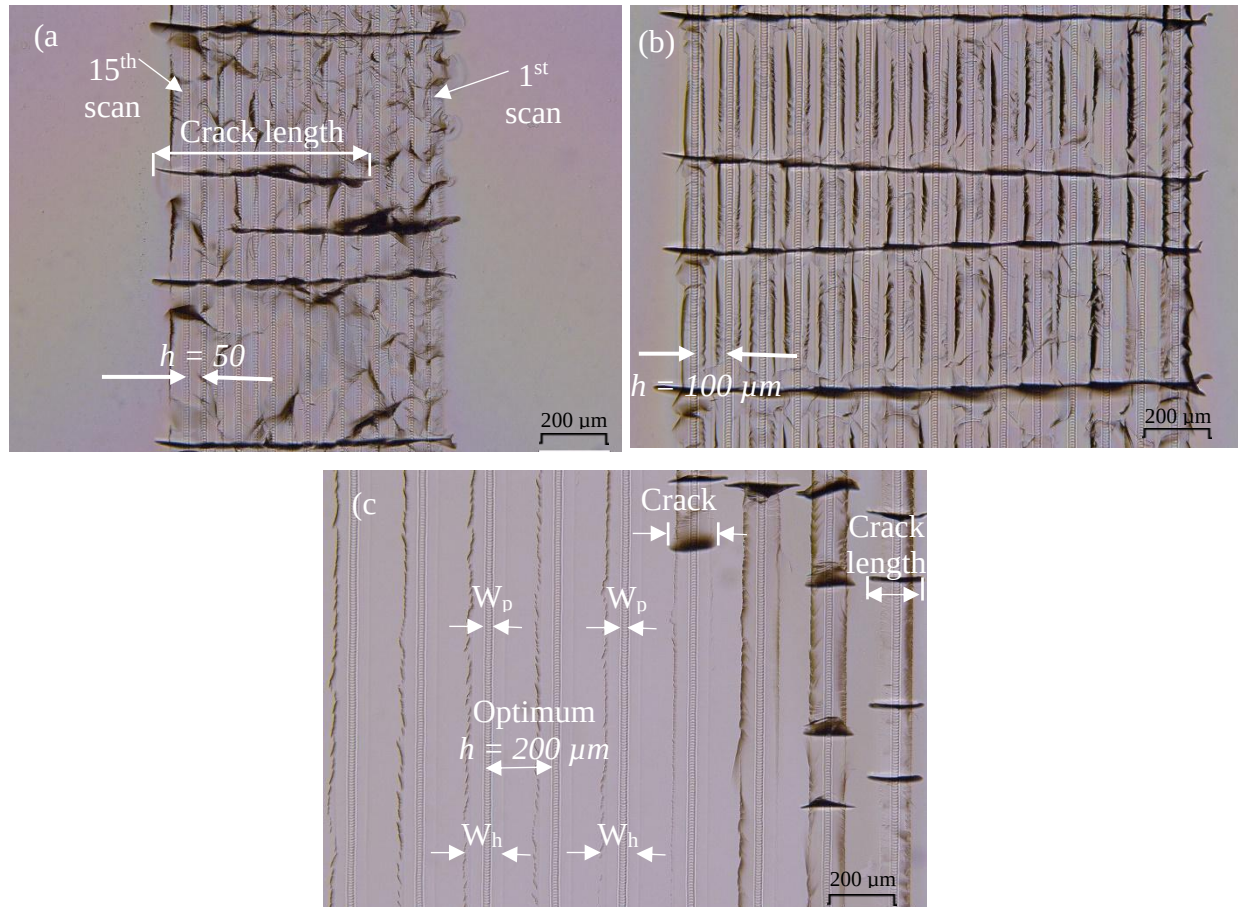


Figure 5.3 Effects of hatch h on crack connection of three 15-scan welded samples at RT: (a) $h=50 \mu\text{m}$, (b) $h=100 \mu\text{m}$, and (c) $h=200 \mu\text{m}$. Measurements of W_p , W_h , and microcracks length are also shown

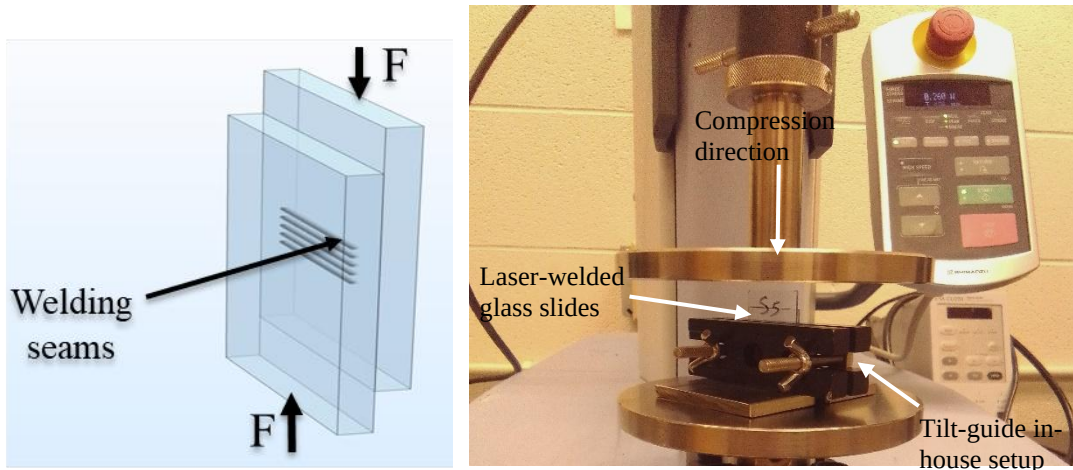


Figure 5.4 Experimental setup of lap shear strength testing; a tilt guide is utilized to avoid sample tilt during compression loading

5.4 Results and Discussion

5.4.1 Glass Modification Pattern

Figures 5.5 and 5.6 show modifications at the interface of a front glass sample of the contacted pair created at RT, MT, and HT. For the RT case, apparent circular discoloration happens under the effects of laser-glass interaction, the intensity of which is proportional to the pulse energy. This discoloration is attributed to the color-center formation where a change in refractive index of borosilicate glass happens due to self-focusing [25]. Additionally, a clustered-particulate modification centered at the discoloration circles is also observable, especially for the 80 – 100 μJ pulse energy. For the MT and HT, the discoloration is not significant for the former and not discernable for the latter. However, the clustered-particulate modification is still remarkable.

The analysis below explains the effects of pulse energy on discoloration intensity. The incident laser beam of $E_i = 30 \mu\text{J}$ has a peak power of around 163 MW, which is around 50 times the critical peak power for self-focusing of 3.2 MW for borosilicate glass, as reported in [3]. The peak power can be calculated by $P_{\text{peak}} = E_i/t_p$; however, glass can only absorb a portion of the

incident pulse energy, which is estimated by the absorption rate analysis of Section 5.4.2. From Figure 5.7 (Section 5.4.2), $A \approx 1\%$ for $E_i = 30 \mu\text{J}$; $P_{\text{peak}} \approx 1.6 \text{ MW}$, which is lower than the critical peak power for self-focusing. This explains the absence of discoloration for the $30 \mu\text{J}$ at RT in Figure 5.5. For $E_i > 30 \mu\text{J}$, Figure 5.7 shows that it is possible to exceed the critical peak power based on the absorption rate level; for example, when $E_i = 40 \mu\text{J}$, $A \approx 3\%$ and $P_{\text{peak}} \approx 4.8 \text{ MW}$, which exceeds the critical peak power. In all cases of Figure 5.5 and 5.6, the incident beam intensity I do not exceed the intensity threshold of $2.15 \times 10^{13} \text{ W/cm}^2$ [7] for borosilicate glass. Intensity I is calculated by $I = F/t_p$ where $F = E_i/A_s$ is the incident laser beam fluence and A_s is the spot area.

Apparently for RT, MT, and HT, the discoloration has a higher intensity at the center of the modification circles, which abates as the radial distance from the circles' center increases. Assuming that the filamentation modification of glass still has a Gaussian or a semi-Gaussian intensity, the radial dependence of discoloration intensity can be correlated to the intensity's radial distribution.

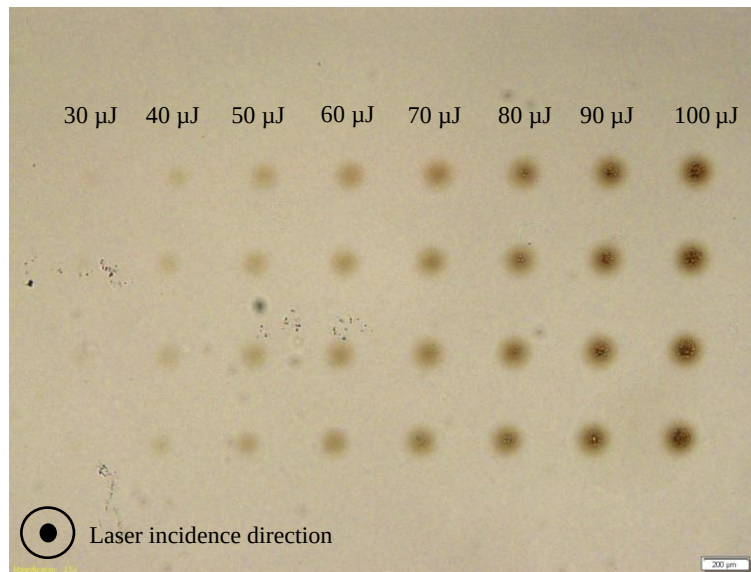


Figure 5.5 An interface of a glass slide under optical microscope after laser transmission at RT showing four modification sets per pulse energy for consistency

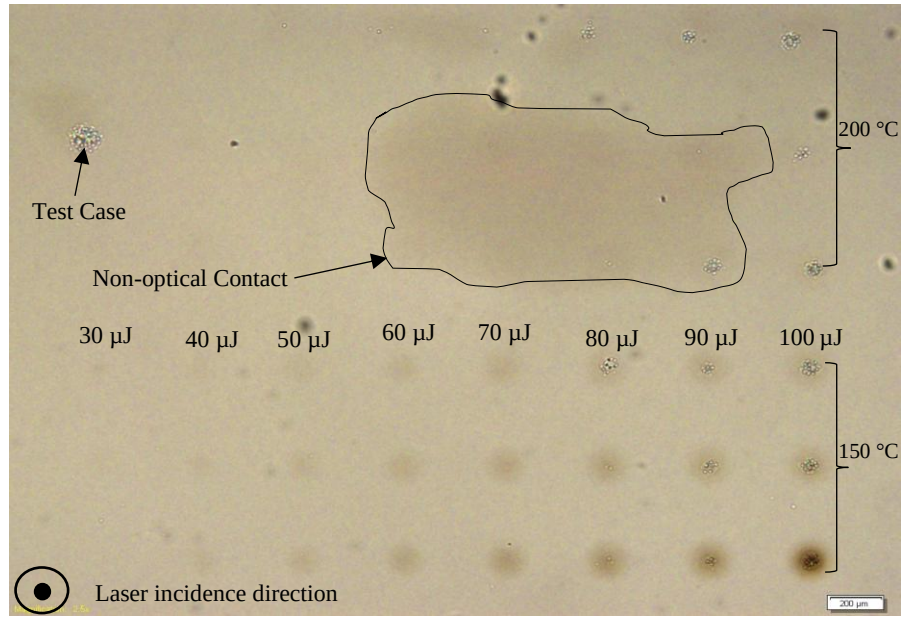


Figure 5.6 An interface of a glass slide after laser transmission at MT and HT showing three modification sets per pulse energy per temperature for consistency

The discoloration generation is attributed to the formation of color centers due to trapped electrons/holes under the exposure of femtosecond pulses [25,26]. These electrons/holes are unstable which can recombine upon annealing heating.

Free electrons in dielectric materials like glass are generated as a result of laser material interaction. When an incident laser beam of wavelength λ is transmitted through a glass sample, laser energy is absorbed by valence electrons of the dielectric material. If the photonic energy is high enough to move electrons from the valence band to the conduction band, a multiphoton (N-photon) ionization phenomenon takes place and generates free electrons. The photon energy E_{photon} is inversely proportional to λ , and the number of photons, N required to trigger one electron can be determined by $N = E_{\text{gap}}/E_{\text{photon}}$, where E_{gap} is the band gap of the dielectric material. The freed electrons by multiphoton absorption can also collide to other valence electrons moving them from the valence band to the conduction band and generating more free electrons by avalanche ionization [19].

The observations in Figures 5.5 and 5.6 suggest two possible scenarios: either concurrent discoloration formation and recombination happens or color center formation does not take place upon preheating. In the current work, preheating above RT where the temperature difference of 125 for MT and 175 °C for HT can still relax the self-focusing-assisted discoloration but not to the level that stops self-focusing, which is still significant in terms of clustered-particulate interfacial modifications. Figure 5.6 has a feature marked as a “Test case”, which is used as a reference for the HT modification’s location when inspected under microscopy. Also in Figure 5.6, the “non-optical contact” contour marks a region at the interface where optical contact is re-laxed/lost under the effect of preheating during laser scan, which did not affect the out-comes of the modification pattern creation and analysis.

5.4.2 Absorption Rate Measurements

Figure 5.7 shows the measured transmitted pulse energy E_t and the calculated absorption rate $A\%$ for the incident pulse energy E_i in the range of 30–100 μJ at three far-field positions in z -direction after the beam exits the glass slides: $dl = 103.5$, $dp = 292$, and $dh = 502$ mm. In Figure 5.7 (right), $A\%$ is calculated for each E_i using Equation 1 by taking E_t as the average of nine measurements, three replicates at all three far-field positions at RT. Similarly, there are other nine measurements for HT at all three far-field positions. From Figure 5.7(left), the E_t can be linearly correlated to E_i at RT and HT up to 50 μJ while the absorption rate is not significantly affected by glass temperature within this pulse energy range as shown in Figure 5.7 (right); A is in the range of 1 – 3 %. At HT and for $E_i > 50$ μJ , the E_t is around 5 % higher for all positions compared to the RT up to the highest E_i of 100 μJ . Assuming that the level of clustered-particulate damage is not affected by the temperature as shown in Figures 5.5 and 5.6, the lower level of E_t in the RT curve for $E_i > 50$ μJ (Figure 5.7, left) can be attributed to the discoloration. The discoloration at RT

contributes to higher absorbed energy E_a for $E_i > 50 \mu\text{J}$ compared to the HT, which is confirmed by the marginal increase in $A \%$ as shown in Figure 5.7 (right).

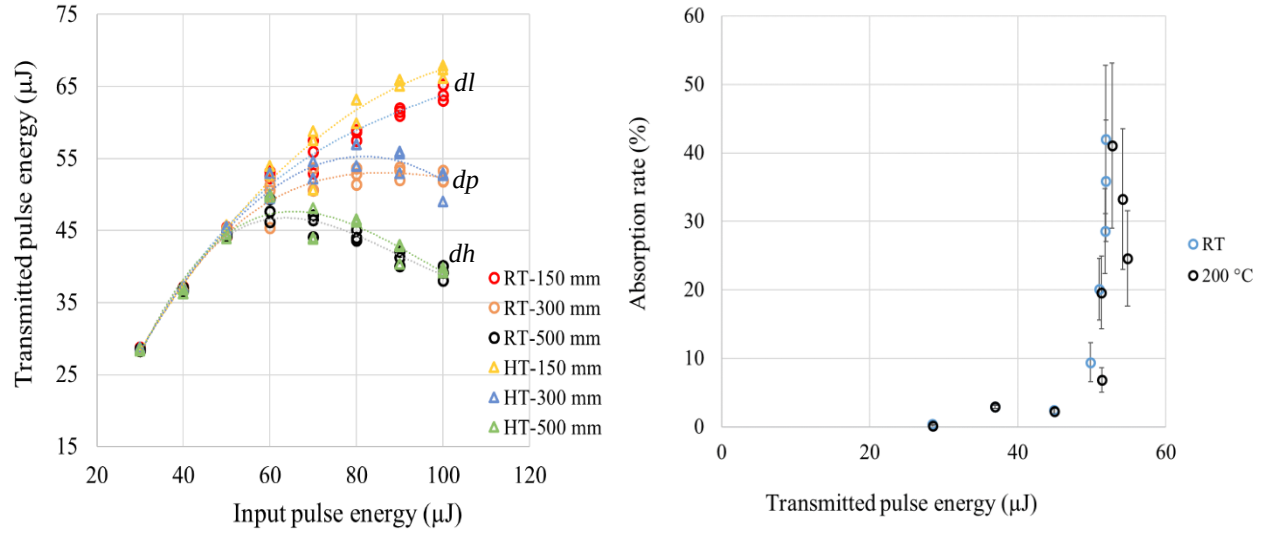


Figure 5.7 Transmitted pulse energy measurements of three replicates using three positions of power meter at both RT and HT, and the calculated absorption rate %

For RT and HT, as the distance is increased from dl to dh , the measured E_t decreases significantly for pulse energy of $E_i > 50 \mu\text{J}$ (Figure 5.7, left). Additionally, the reduction in E_t when the distance is increased is proportional to pulse energy. This distance-dependent reduction in the measured E_t (Figure 5.7, left) is attributed to the scattering effect of the clustered-particulate modification formation at RT and HT, which is proportional to pulse energy and does not change with temperature, as shown in Figures 5.5 and 5.6.

5.4.3 Weld Seam Topology: Heat-affected and Plasma-modified Region

Welding of glass slides is performed utilizing the parameter combinations of Table 5.2. Ten replicates of $38 \times 25 \times 1 \text{ mm}^3$ are welded for each treatment combination using five parallel scans, repetition rate of 200 kHz, and scanning speed of 10 mm/s at RT and MT utilizing the setup of Figure 5.2. Then, the replicates are inspected by optical microscopy to capture images of the

interfacial weld seam. Figure 5.8 shows six welded glass slides having optical contact created at the weld seams surrounded by interference fringes.

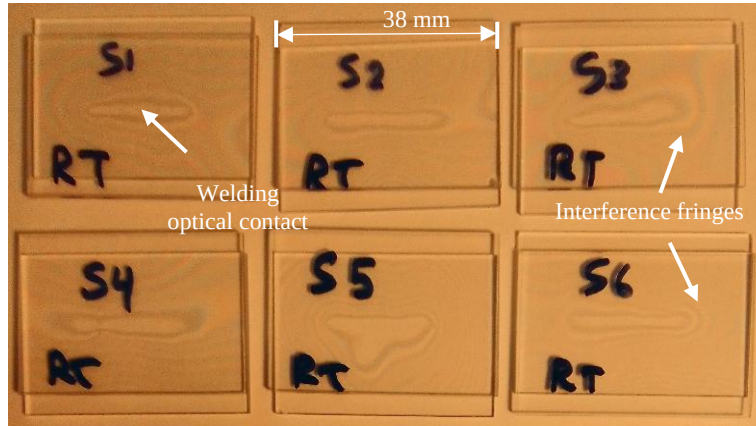


Figure 5.8 Six welded pairs of glass slides using 10 μJ and 10 ps at RT showing the welding optical contact of 5 scans per sample and the surrounding interference fringes

After welding, the width of the heat-affected zone, W_h , the plasma-modified region, W_p , and the length of cracks are measured using imageJ. Figures 5.3 (a) and (c) show sample measurements of W_h , W_p , and crack length. Cracks are projected where only straight measurements are taken, even for tilted cracks, to avoid the complexity of measuring branched, oblique cracks, and cracks with different opening directions. Only cracks that are perpendicular to the weld seam direction are considered for the analysis in Section 5.4.5 as these cracks represent the most significant encountered defect's type for the inspected samples that affects the strength.

Figure 5.9 shows the measurements of four groups of 10 replicates. The W_h and W_p measurements at RT of Figures 5.9 (a) and (b) are for the same group of ten replicates. Another group of ten replicates is utilized to measure W_h and W_p at MT of Figures 5.9 (a) and (b). Similar measurements are made for the pulse duration of 184 fs and shown in Figures 5.9 (c) and (d). A minimum of 3 measurements are acquired for each scan of the 5 weld seams per sample; meaning that at least 15 measurements are taken per sample. Qualitatively in Figures 5.9 (a) and (c), the tear-drop width, which is equal to W_h is dependent on the preheating temperature; the MT

measurements are shifted within the top 50 % of the range compared to the RT condition. In Figures 5.9 (a) and (c) and at MT, $W_h > 78.69 \mu\text{m}$ for the 10 ps while $W_h > 97.81 \mu\text{m}$ for the 184 fs. Comparatively, there is an insignificant dependence of W_p on preheating temperature for both 10 ps and 184 fs welding conditions, as shown in Figures 5.9 (b) and (d).

To better understand the dependence of W_h and W_p on preheating temperature and pulse duration or incident intensity, average measurements of W_h and W_p for the pulse energy of 4.5, 6, 10 μJ and 6, 10, 14, 18 μJ for the femtosecond and picosecond pulse durations are plotted in Figure 5.10.

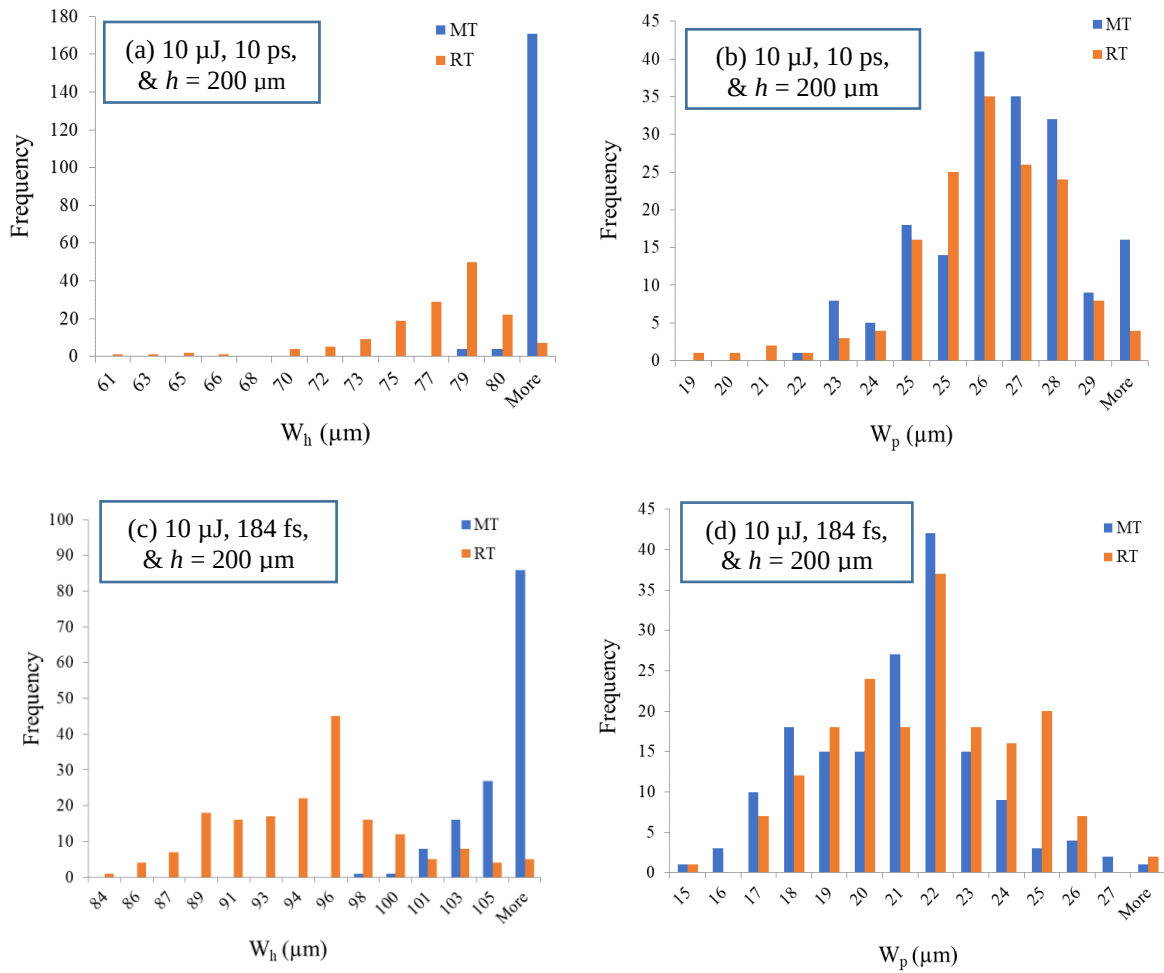


Figure 5.9 Histograms of measured W_p and W_h for 184 fs and 10 ps at RT and MT, measurements of 10 replicates per histogram per temperature

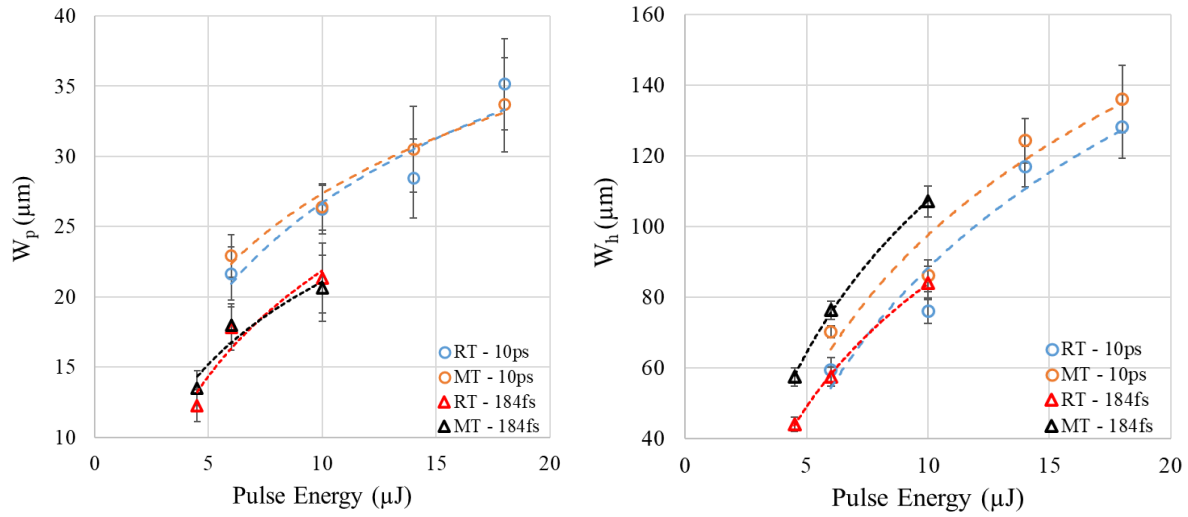


Figure 5.10 Average measurements and deviation of W_p and W_h for Figure 5.9 histograms

Interestingly, there are two important outcomes from Figure 5.10: first, the 10 ps plasma-modified seams where glass welding is expected to occur have shown a wider W_p compared to the 184 fs; W_p is around 19% higher for the 10 ps compared to 184 fs within the range of 6 – 10 μJ at both temperatures. Second, W_h is not dependent on pulse duration at RT while it depends on temperature at MT; at MT, W_h is around 13% higher for the 184 fs compared to the 10 ps within the range of 6 – 10 μJ . In short, W_p depends on pulse duration and not temperature, while W_h depends on temperature and pulse duration for any preheating temperature higher than RT.

Another finding for the comparison of weld seams generated by 184 fs and 10 ps pulse durations can be seen in Figure 5.11. The plasma-modified region where welding is expected to occur for the 10 ps has a clear pulse-to-pulse staircase topology compared to the 184 fs where the staircase topology is not discernable, as shown in the magnified inserts. This effect can be seen for all replicates of RT and MT cases; apparently it does not depend on temperature. The staircase is not visible for the 184 fs due to the fact that the top portion of the plasma-modified region has a pin-shaped topology compared to a curve-shaped top portion for the 10 ps weld seams, as shown

in Figure 5.12. Additionally, the red arrows point to the discoloration due to self-focusing at RT and 184 fs, which does not happen at MT and 184 fs, and both temperatures at 10 ps.

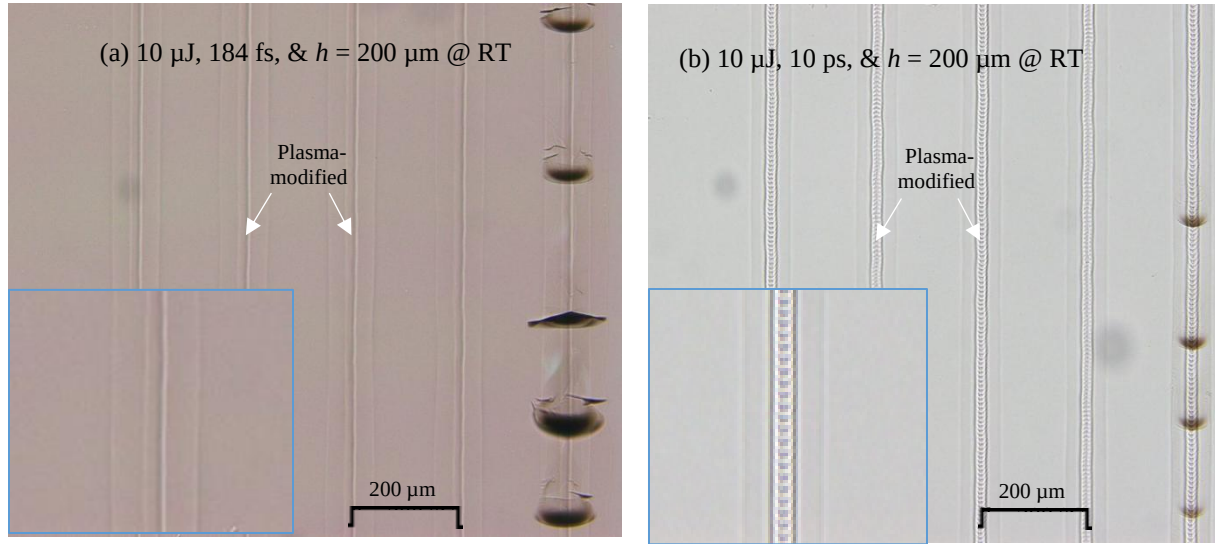


Figure 5.11 A comparison of plasma-modified regions of 184 fs and 10 ps weld seams showing a stair-case topology for 10 ps, which happens for all pulse energy at RT and MT

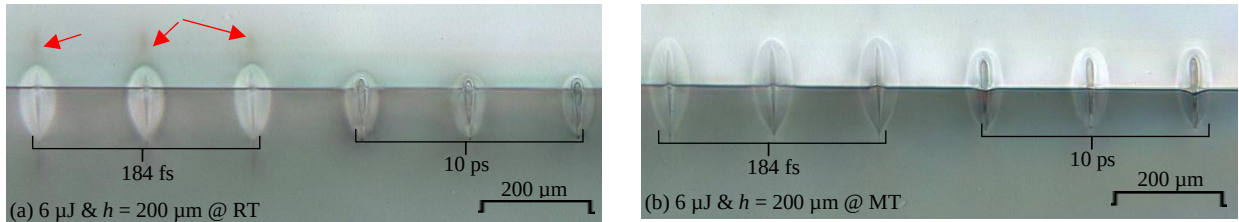


Figure 5.12 Effects of temperature on discoloration and plasma-modified morphology explaining a possible reason for the stair-case topology of 10 ps weld seams at RT and MT

5.4.4 Single-lap Compression Shear Strength

Figure 5.13 shows load-displacement curves of the lap shear strength testing for samples welded with 10 μJ pulse energy, 5 scans, and $h = 200 \mu\text{m}$ at RT and MT. Figures 5.13 (a), (b) and (c), (d) are for the 184 fs and 10 ps pulse duration, respectively. Variations between samples produced scattering in the load-displacement behavior, shear strength load and shear displacement

at failure. Effects of pre-weld and post-weld partial optical contact take place for the elastic portion prior to maximum load in terms of a limited zigzag elastic behavior. Post maximum load, there have been progressive failures for some samples due to breaking of weld seams introduces glass flakes at the interface between samples and could increase local friction forces while under the compaction of the tilt guide. However, since the major outcome of the single-lap shear test is the maximum shear load, the progressive failure should not have a significant effect.

Shear strength is calculated by dividing the maximum load by the shear area. The shear area is determined by the product of W_p and the seam length of five scans, which is around 23 mm per scan. W_p is determined by the measurements of Section 5.4.3; W_p is taken as the average of 15 measurements per sample for strength calculations. To exclude strengths that are abnormally larger or smaller compared to the measurements' bounds, the 1st and 3rd quartile outlier analysis is performed for the calculated shear strengths. Filtered strengths data are depicted in Figure 5.14 for the 184 fs and 10 ps at RT and MT.

Surprisingly, the shear strength of samples welded at RT is higher than the shear strength for samples welded at MT regardless of the incident beam pulse duration. However, when a linear trend is fitted to the strength vs. pulse energy, an enclosed region is formed between the RT and MT lines, which is wider for the 184 fs. The fitted lines show that the strength at RT is between 11– 66 % higher than that at MT for 6 – 18 μJ using 10 ps pulse duration. Similarly, the strength at RT is between 19– 83 % higher than that at MT for 4.5 – 10 μJ using 184 fs pulse duration. Apparently, the effect of preheating temperature on welding strength reduction is more significant at 184 fs compared to 10 ps. To explain this finding, cracks generated due to laser glass welding are analyzed for all replicates of 184 fs and 10 ps pulses.

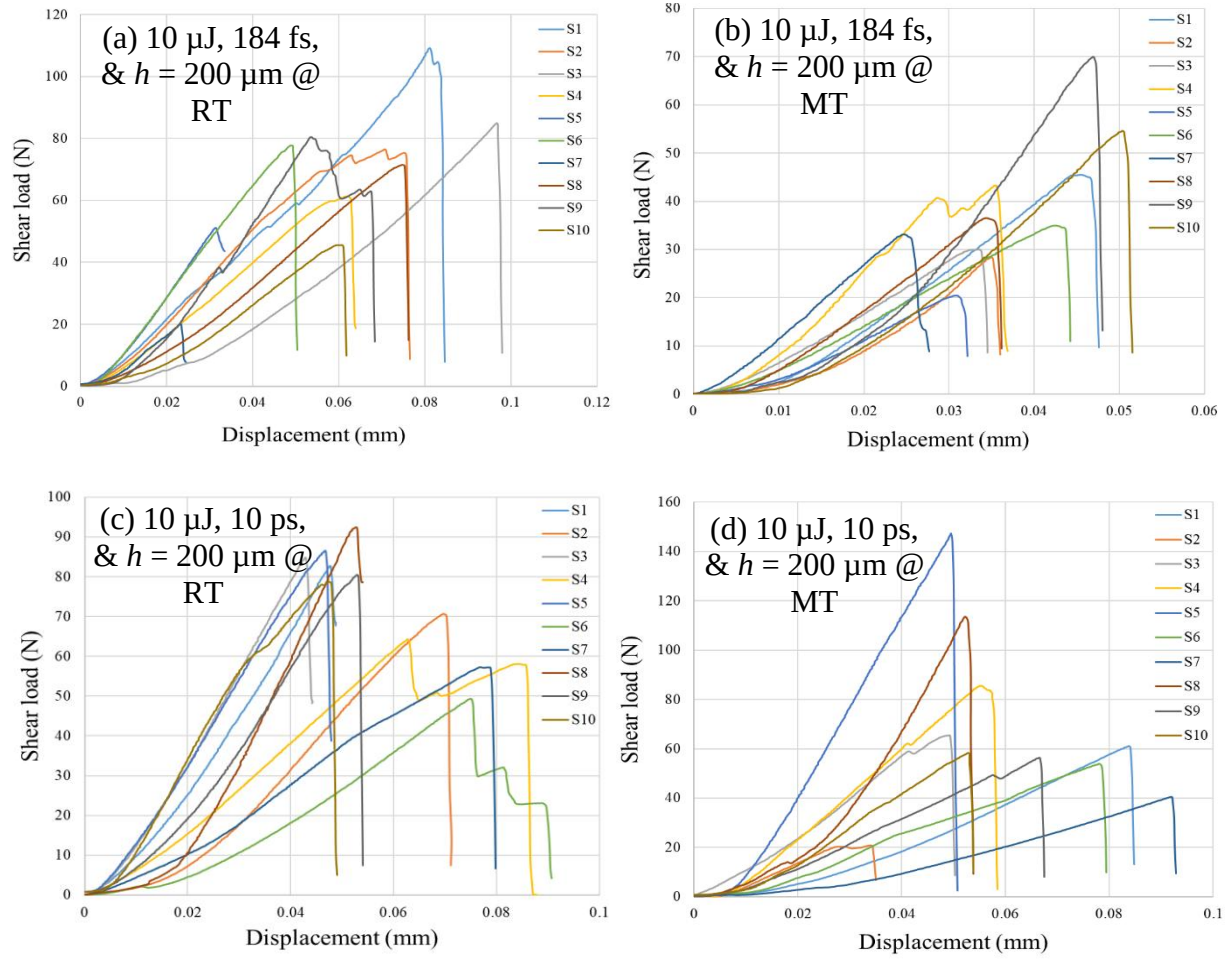


Figure 5.13 Sample load-displacement curves of single-lap shear tests showing fitted elastic and post-failure portions of the curve

The difference in strength of the “crack-free” combinations (see Section 5.4.5) of Figure 5.14 shows that the 184 fs pulse duration exhibits a maximum strength of around 50 MPa compared to 35 MPa of 10 ps. This difference can be caused by laser-induced defects at smaller scales where the connectivity of the ring structures of glass material decreased at specific locations of nonbridging oxygen hall centers and compacted at other locations, which can impede the propagation of cracks generated after mechanical loading application [7]. This scenario can also explain a possible reduction in fracture toughness that can happen for laser glass welding implemented at MT using “crack-free” combinations where preheating contributes to reduce the

beneficial effects of impeding defects leading to micro-crack initiations and lower fracture toughness. The possible reduction in fracture toughness is higher for the 10 ps compared to the 184 fs; however, this hypothesis for “crack-free” and “crack-rich” conditions is yet to be proved in future work.

Welding of preheated substrates using “crack-free” process parameters producing promising strengths for the 184 fs and 10 ps, which are 25 % and 10 % higher compared to average laser welding bonding strength of 32 MPa reported in [18]; the 32 MPa strength is the average of nine different investigations.

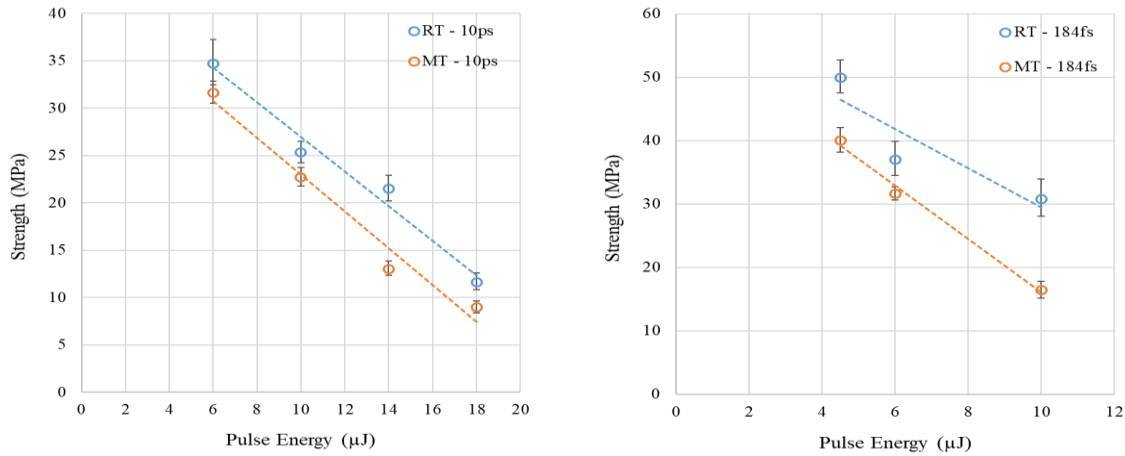


Figure 5.14 Average shear strength and deviation of weld seams vs. pulse energy for 184 fs and 10 ps at RT and MT; strength decreases with pulse energy and preheating

5.4.5 Post-Welding Cracking

Figure 5.15 shows the projected measurements of perpendicular cracks for 6 and 10 μJ of 184 fs and 10, 14, and 18 μJ of 10 ps at RT and MT, respectively. Pulse energy of 4.5 μJ at 184 fs and 6 μJ at 10 ps are denoted as “crack-free” combinations that have not shown significant cracking; number of cracks is around 10 for no more than two replicates. This is why these cracks are not reported in Figure 5.15; however, Figure 5.14 still shows a higher strength for RT compared to MT for 4.5 μJ at 184 fs and 6 μJ at 10 ps.

In Figure 5.15, crack length measurements are plotted against crack length-to-hatch ratio. The number of cracks, their lengths relative to hatch, and the slope, which represents the hatch can be investigated. At both RT and MT, when a group of combinations of 6 μJ for 184 fs and 10 μJ for 10 ps are used to weld glass slides, significant cracks are generated; however, their lengths are still not exceeding the respective hatch; crack length-to-hatch ratio does not exceed 1, as shown in the dark colored circles of Figures 5.15 (a – d). Among this group's four combinations, the 6 μJ for 184 fs at RT shows an average strength of around 42 ± 1 MPa (based on trend line of Figure 5.14) compared to 27 ± 1.1 MPa for 10 μJ for 10 ps (based on trend line of Figure 5.14). Comparing these two cases, the 184 fs one has a 55 % higher strength compared to the 10 ps, which is the result of a combined effect of fracture toughness and average crack morphology, as stated by Equation 2. In Figure 5.15, comparing the MT cases to their counterparts of RT cases, MT welding produced more and longer cracks compared to RT conditions, which could be the reason behind lower strengths for welding at MT shown in Figure 5.14, assuming that fracture toughness does not change significantly by temperature.

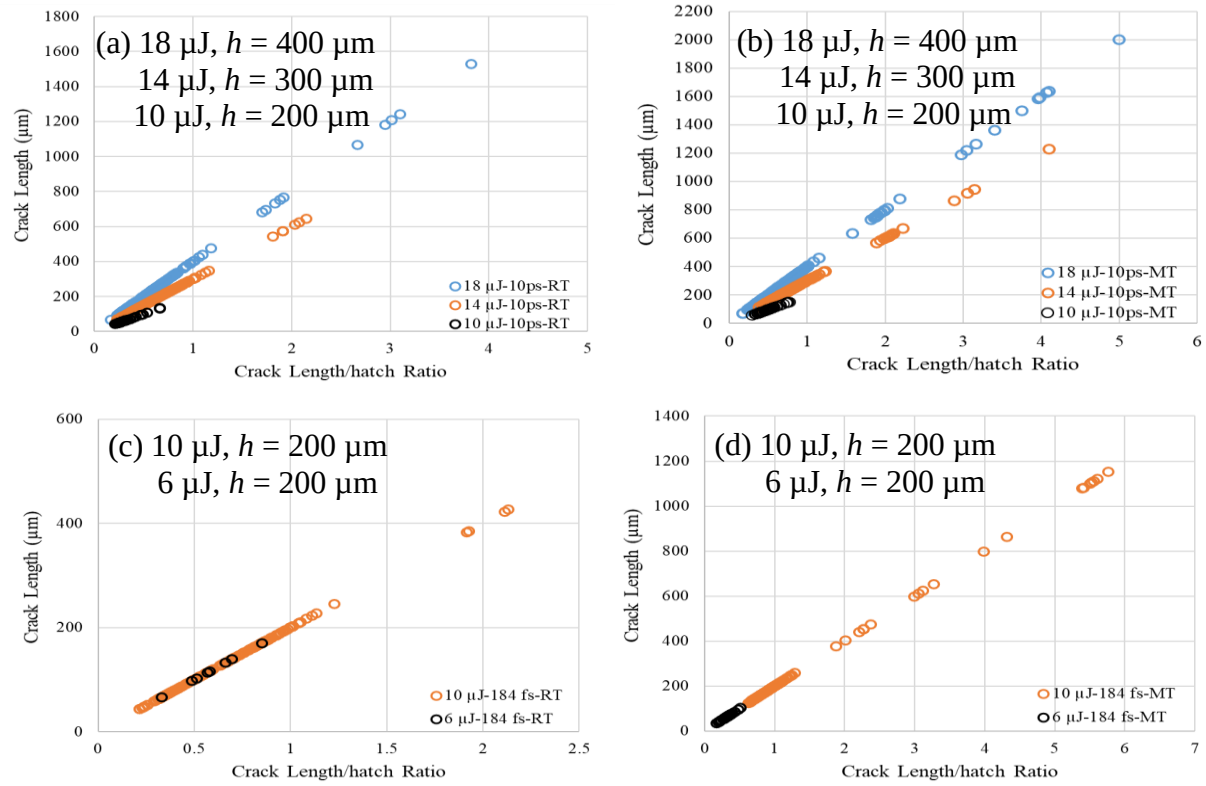


Figure 5.15 Post-welding crack measurements for the combinations of Table 5.2, 4.5 μJ at 184 fs and 10 μJ at 10 ps (not plotted) have shown less than 10 small microcracks for only 3 replicates out of 10 and hence are considered as “crack-free” combinations

5.5 Conclusions

The role of preheating in laser glass welding under ultrafast laser pulses of 10 ps and 184 fs is investigated in the current work. While the pulse energy range for welding of 4.5 – 18 μJ at 184 fs produces self-focusing discoloration, similar pulse energy range at 10 ps does not, as the incident peak power does not exceed 3.2 MW for 10 ps. Investigating the effects of preheating on absorption rate when only self-focusing is significant can still explain the effect of preheating on nonlinear absorption, without the generation of tear-drops. Based on this hypothesis, outcomes of modification pattern experiments and absorption rate determination using 184 fs are applicable for preheated glass welding under 10 ps and 184 fs pulse durations. Internal modification analyses show that pre-heating has a significant effect on inhibiting discoloration due to color center

formation associated with self-focusing filamentation; however, preheating within the current range does not seem to enhance absorption rate. The particulate-clustered damages that are significant at pulse energies higher than 50 μJ may not happen during welding, as the highest welding pulse energy is 18 μJ . The outcome of unchanged absorption rate due to pre-heating to 200 °C under 10 ps and 184 fs is in agreement with the results of measured width of plasma-modified region W_p , which shows independency on preheating to 150 °C. Contrary to W_p , the width of heat affected zone W_h depends on preheating; both 10 ps and 184 fs have enhanced W_h ; however, the 184 fs shows 13 % higher W_h compared to the 10 ps. Accordingly, the effects of preheating on glass welding under 10 ps and 184 fs conditions are mainly mitigation of self-focusing discoloration, which only happens for the 184 fs pulse.

However, the wider W_h does not contribute to enhancing welding strength in the current work, as welding mainly happens within the W_p . While preheating to MT mitigates self-focusing discoloration and enhances W_h , heat induced defects/microcracks could be the main reason behind lower shear strengths for glasses welded at MT compared to RT at 184 fs and 10 ps. Welding strength at RT using 184 fs in the “crack-free” region is higher by 42 % compared to the 10 ps which could be due to the reduction in fracture toughness under the 10 ps pulses. Future work is required to investigate the preheating effects on glass material evolution under femtosecond and picosecond laser pulses in terms of fracture initiation and propagation.

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Chapter 6 . Summary and Future Work

This thesis reports on innovative laser glass welding of preheated glass substrates prior to laser scanning, utilizing the localized non-linear absorption of the laser beam at the focal position without using solders or a second absorbing layer. With preheating, process parameters' window of pulse energy and duration are not limited by the significant self-focusing filamentation that leads to glass discoloration. Glass discoloration not only alters refractive index and transparency but also contributes to a shift in focal position, which may disrupt the relative location of tear-drop to the interface for a successful laser glass welding.

The applicability of preheating for laser glass welding is investigated by developing a linear analytical model to estimate the focal position at any temperature. The model predicts the focal position beforehand that can be used for transparent materials having different thickness, temperature, refractive index and thermal dilatation properties. The developed analytical model is simple, physics-based, and computationally efficient, which can be used by scientific and education community for other transparent materials. An internal modification process using femtosecond pulses is also developed by minimizing the interaction between adjacent tear-drops and providing a reliable morphological and focal position measurement. The analytical model is robust for predicting focal positions of tear-drop internal modifications generated at RT, 150 and 200 °C as validated by experimental measurements of multiple replications to account for uncertainty due to glass and geometry variations.

The concept of distance compensation between the lens and glass surface for focal position determination using picosecond pulses is investigated and an analytical model is also developed. The compensation distance is required to account for refractive index variation and its effect on focal position shift upon preheating to different temperature levels. It also accounts for inevitable

uncontrolled variations stemming from glass material inhomogeneity and gap and thickness uncertainty, which can lead to inward/outward variations in local focal position. The compensation parameter is controllable and can be predetermined to locate the focal and teardrop-to-interface relative position, which is also validated by varying temperature, scanning speed, and lens-to-glass distance at different pulse energy levels.

A comprehensive investigation of welding defects and strength utilizing femtosecond and picosecond pulses shows that cracking perpendicular to weld seams can be minimized using optimal hatch distance at first hand. While femtosecond laser pulses generally produce discoloration due to high peak power compared to less possibility of discoloration using picosecond pulses, preheating is shown to significantly control self-focusing discoloration. Teardrop, plasma-modified region, and crack length measurements have significant dependence on pulse energy. However, plasma-modified width depends on pulse duration but not temperature, while width of heat-affected zone depends on temperature and pulse duration for any preheating temperature higher than the RT.

Moreover, compression-shear strength of welded glass shows a strong dependence on pulse energy, which is attributed to length of welding-induced cracking perpendicular to the seam weld direction. Upon preheating, a marginal drop in strength of “crack-free” welding conditions is observed, which is magnified when pulse energy is increased. The marginal drop in strength is also investigated and attributed to variation in laser-glass interaction leading to plasma-modified glass seams having an intensity-dependent fracture toughness, as supported by the mode I crack-opening analytical model.

In conclusion, application of the innovative preheating concept establishes a new processing parameter map where self-focusing effects are alleviated, making it feasible to use

femtosecond conditions of high peak power without significant self-focusing effects. Glass preheating strategy is a major contribution for situations where transparency and refractive index are critical requirements of post-welding for optics applications. The analytical, numerical and experimental procedures developed in this work are of great importance for the current industrial efforts to process commercialization and future exploration of novel transparent materials welding and joining processes.

Future work recommendations:

- A comprehensive investigation of preheating effects on absorptivity parameters should be conducted for linear, non-linear, and plasma absorption using “preheated” z-scan experimental analysis. Analytical modeling can be utilized to extract absorption parameters using experimental intensity measurements for a target transparent material.
- Scaling of the proposed preheating strategy should be examined for a part-level practical application like sensor packaging using contour welding of thick glass samples to investigate the limitations of spherical-aberration on welding thick glass samples under preheating conditions.
- The validity of the developed analytical model for applications in transparent materials whose refractive index decreases upon heating should be investigated. This includes model validation and experimental measurements of temperature-dependent refractive index.
- The applicability and effects of preheating on transparent-to-non-transparent laser welding like glass-to-ceramic and glass-to-metal should be studied. This investigation includes internal modification and weld seam topology analysis.
- The effects of post-welding heating on self-focusing discoloration, welding defects, and weld strength should be studied in terms of post-welding annealing time and heating and cooling

rate. An independent testing of fracture toughness is required to validate the effects of laser processing parameters on the evolution of glass material and mechanical properties.