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EFFECT OF PULSE DURATION ON THE PROPERTIES OF LASER-WELDED GLASS JOINTS

VPLIV TRAJANJA IMPULZOV NA LASTNOSTI LASERSKO VARJENIH SPOJEV STEKLA

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To explore the effect of pulse duration on the micro-morphology and mechanical properties of laser-welded glass joints, a glass welding experiment was carried out. The microstructure of the molten layer with different pulse durations was shown by field-emission scanning electron microscopy. The pores and distribution characteristics were analyzed, and the porosity variation rule of the welding layer for different pulse durations was obtained. The line-scan analysis of the joint interface was carried out with a D8-Advance type crystal X-ray diffractometer, and the relationship between the thickness of the reactive wetting layer and the pulse duration was obtained. The hardness and welding strength of the laser-welded glass joints were obtained with a Vickers hardness tester and an electronic universal testing machine. This study can provide a theoretical and experimental basis for the laser welding of glass.

Keywords: laser welding, glass, porosity, mechanical properties

Avtorji so izvajali preizkuse varjenja stekla zato, da bi raziskali vpliv trajanja impulzov na mikro morfologijo in mehanske lastnosti lasersko varjenih spojev stekla. Avtorji v pričujočem članku opisujejo mikrostrukturo staljene plasti, vidne pod vrstičnim elektronskim mikroskopom na emisijo polja (FE-SEM), nastale pri različnih časih trajanja impulzov. Analizirali so nastalo poroznost in njene spremembe, nastale zaradi različnega časa trajanja impulzov. Z modernim D8-Advance rentgenskim difraktometrom so izvedli linijsko mikrokemijsko analizo na nastalih spojih in ugotovili povezavo med debelino reaktivnega sloja omakanja in časom trajanja impulzov. Trdoto in trdnost izdelanih lasersko varjenih steklenih spojev so določili z merilnikom trdote po Vickersu in na univerzalnim trgalnem stroju. Avtorju zaključujejo, da je ta študija lahko temelj za teoretične in eksperimentalne osnove laserskega varjenja stekla.

Ključne besede: lasersko varjenje, steklo, poroznost, mehanske lastnosti

1 INTRODUCTION

Welded glass is applied in many fields, such as optics, insulation for windows, microelectromechanical systems (MEMS), opto-electronic and medical devices.[1–3] The laser welding of glasses has developed in the recent years.[4,5] Many methods have been developed to laser weld glass.[6] Wang et al. welded Ti-based bulk metallic glass (BMG) plates together, and the mechanism of successful welding of the BMG was discussed in terms of the thermal history of the weld-fusion zone (WFZ) and the heat-affected zone (HAZ).[7] Zhao et al. used picosecond laser irradiation with a low numerical aperture in the transmission welding of two fused-silica glass plates. A model, which considered the propagation property of the laser beam and the incubation effect of modification threshold, was provided to predict the position of the bottom tip of the modification (BTM).[8] Soren et al. provided a new approach for the high-stability bonding of different glasses: ultrashort pulse-laser welding. It was found that ultrashort laser pulses at high repetition rates offer the possibility to realize strong bonds between different glasses. And this technique could be used in integrated microoptical devices.[9] Zhang et al. studied the influence of air gap and focus position on the quality of ps-laser glass-to-glass welding, and a method of detecting the air gap between the glass surfaces based on optical thin-film interference was provided. The processing parameters of the picosecond laser welding were determined and the welding strength and the morphology of the bonded zone were detected and observed. This study is useful for the packaging of electronic components.[10] Kind et al. found that the laser glass frit sealing process was suitable for materials with crucial thermal properties, like soda-lime glass.[11] In conclusion, the laser-welding glass technique is practical. To develop this technique, the effect of pulse duration on the property of laser-welded glass joints is investigated by experiment and theoretical analysis.

During laser welding, different pulse durations correspond to different laser energy, and the laser energy will change the microstructure of the molten layer, the pores and the distribution characteristics, then affect the hardness and strength of the welded joint. This study carried

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out the experiment of laser welded glass to research the effect of pulse duration on the micro-morphology and mechanical properties of pulsed-laser welded glass joints, aiming to provide a theoretical and an experimental basis for the laser welding of glass.

2 MATERIALS AND METHODS

2.1 Experimental methods

Since powdered solder is difficult to coat on a glass surface, a solvent was used to adjust the solder to a liquid slurry before use. In this study, the ethyl cellulose and the terpineol mixed solution were used as a blending agent. The preparation process was as follows: place ethyl cellulose and terpineol in a mass ratio of 1:9 in a beaker, mix under heating at 80 °C in a water bath, and dissolve the ethyl cellulos. Then, mix solder with a mass ratio of 10:1 with a solvent. It was stirred for 30 min and then rested for 30 min, then defoaming was performed to complete the preparation of the coated solder.

The size of the soda-lime glass selected for the welding experiment was 50 mm × 50 mm × 4 mm, and the chemical compositions were shown in **Table 1**. If the glass surface was smooth, this was not conducive to fusion with the solder. Therefore, the glass surface to be welded was polished with a BD46N abrasive belt-sanding machine, so that the glass had a certain roughness. Due to dust, oil and other dirt stained on the glass surface, it should be ultrasonically cleaned in absolute ethanol for 10 min, then dried in a DUG-9053A electric thermostatic blast drying oven to coat the prepared solder. The periphery of the plate glass was then placed on a D20 digital display heating plate for heating at a temperature of 150 °C for 30 min to completely remove the adhesive, and avoided defects such as pores and bubbles in the sealed portion. Then it was combined with another piece of glass and warmed to 250 °C for preheating, and the preheating time was 10 min. Finally, the experimental samples were placed on the HGL-LCY300 Nd:YAG laser platform, and the glass samples were obtained for various process parameters. The prepared sample was cut into 20 mm × 20 mm × 8.3 mm using a SYJ-160 wire saw and then polished the cut surface, finally placed in a drying dish after ultrasonic cleaning.[12,13]

Table 1: Chemical composition of the common soda-lime glass (*w*/%)

SiO_2	CaO	MgO	Na_2O	Al_2O_3
67–74	5–11	0–4	10–17	0–3

The welding strength of the glass directly affected the reliability and durability of the glass products. The welding strength was insufficient, causing easily the glass cracked and the welding failed. Therefore, it was necessary to test the welding strength. The sample was made by coating glass solder on the center of a glass strip with a size of 120 mm × 20 mm × 4 mm and an area of 20 mm × 20 mm. Another glass strip of the same size was placed on the solder and the two glass strips were perpendicular to each other. The prepared samples were placed on the laser platform, and the two glass strips were bonded together by solder after laser-welding cooling.

2.2 Performance tests

A BX41M-LED metallographic microscope was used to observe the bonding between the solder and glass at the joint interface under different process parameters, and Image-J software was used to calculate the porosity of the sealing layer after metallographic photograph processing. The porosity is the percentage of the pore area to the surface area of the weld.

A Zeiss Supra 55 field emission scanning electron microscope was used to observe the micro-structure of the molten layer under different process parameters, and then the self-contained energy spectrometer was used to perform line scan analysis on the reactive wetting layer. Finally, the sample analysis was carried out by the D8-Advance type crystal X-ray diffractometer.

The hardness test on samples was tested for different process parameters using a MHV-1000 Vickers hardness tester. The test conditions are as follows: the load is 200 g, and the holding time is 15 s, then the average value is obtained after multi-point measurement. The hardness test position can refer to the reference [14].

Welding strength includes tensile strength and shear strength at the welding interface. The detailed test can refer to the reference 14.

2.3 Process parameter determination

In the LCY300 type Nd:YAG laser welding system, the influence of pulse current on the microstructure of the sealing layer was studied by a single factor experimental control method. Therefore, the main factors affecting the laser-welding experiment include pulse duration, spot size and clamping force. Since the fixture used in this experiment uses mechanical transmission control, the value cannot be measured. To ensure that other process parameters were tested under the same pre-tightening force conditions, the pre-tightening force was used as the quantitative experiment, and ignored the influence of the fixture. By adjusting the position of the lens, the diameter of the spot irradiated on the solder surface was 10 mm, which was equal to the welding duration of the glass.

During the welding process, the average laser power should be controlled at 30–55 W, and the welding speed should be maintained at 90 mm/min. The relationship between the laser output energy and the pulse current and pulse duration is shown in Equation (1).

$$W = UI\tau \times 3\% \tag{1}$$

In Equation (1) W is the single pulse energy of the laser. U is the external voltage of the laser, which is 380 V.

I is the pulse current. τ is the pulse duration. The photoelectric conversion rate of the laser is 3 %.

The relationship between the laser average power P and the laser single pulse energy W and the pulse frequency f is shown in Equation (2).

$$P = W \times f \tag{2}$$

The pulse duration is adjusted to obtain the laser power required for the test. Through a preliminary experiment exploration, the range of parameters is determined in this experiment, as shown in **Table 2**.

Table 2: Pulse duration variation range in this experiment

pulse duration (ms)	pulse current (A)	pulse frequency (Hz)	welding speed (mm/min)
1; 2; 3; 3.5	160	18	90

3 RESULTS

3.1 Effect of pulse duration on the microstructure

When the pulse current I was 160 A, the pulse frequency f was 18 Hz, the welding speed v was 90 mm/min, and the pulse duration was changed. **Table 3** shows the welding process parameters.

Table 3: Welding process parameters when changing the pulse duration

pulse duration (ms)	pulse current (A)	pulse frequency (Hz)	welding speed (mm/min)	single pulse energy (J)	average power (W)
1	160	18	90	1.82	27.4
2	160	18	90	3.65	54.8
3	160	18	90	5.47	82.1
3.5	160	18	90	6.38	95.8

Figure 1 shows the microstructure of the molten layer under different pulse durations.[15,16] When the pulse duration was 1 ms, the solder was not completely melted, and the molten layer mostly existed in a massive agglomeration structure. The bonding effect between the solder particles was poor, forming a large number of pores, and the density was low. When the pulse duration was 2 ms, the solder melted more completely, and the solder spread better. Moreover, the surface of the molten layer was flat, and the density was high. When the pulse duration was increased to 3 ms, the crystal grains of the molten layer were coarsened, and a large number of spherical solid particles existed. When the pulse duration was increased to 3.5 ms, the spherical particles became large, and the distribution area increased. This phenomenon was a "spheroidization" phenomenon formed during laser sintering of the powder. The larger the pulse duration was, the longer the heat source acted, making the solder absorb more energy, forming more liquid phase and reducing the melt viscosity significantly. However, too low liquid viscosity could lead to a "spheroidization" effect, which could reduce the density. When the pulse duration is too large, the molten pool will be overheated severely, and generate large thermal stress, resulting in deformation and cracking, as shown in **Figure 2**.

Figure 2 shows the distribution of pores and the porosity variation curve of the sealing layer under different pulse durations.[17] Results show that the porosity of the sealing layer decreased first and then increased. This was because when the pulse duration was 1 ms and 2 ms, as the pulse duration increased, the heat source acted longer, prolonging the relative thermal action time of the liquid solder convection, so the bubbles had enough time

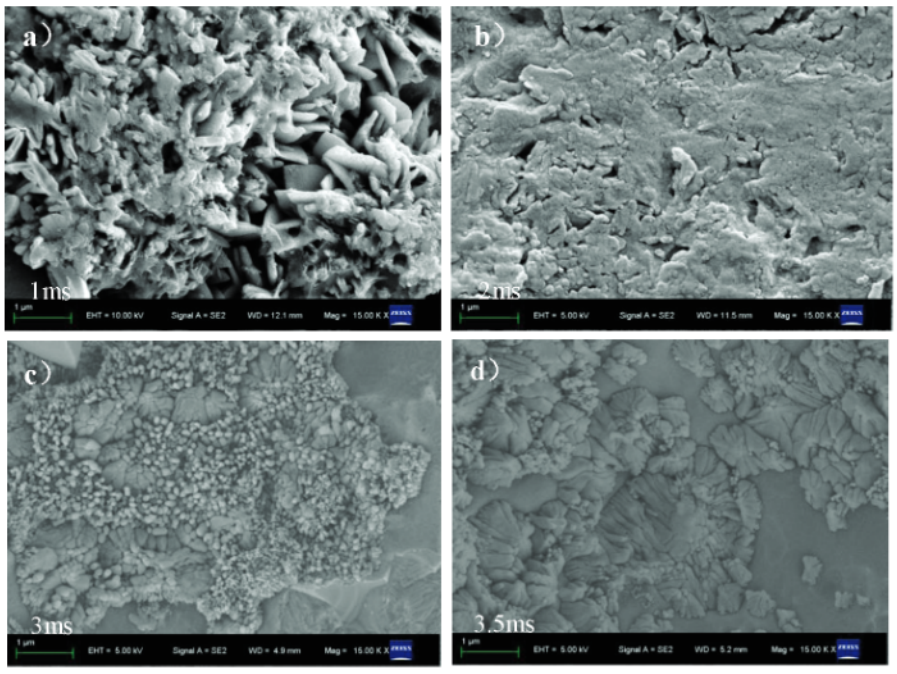

Figure 1: Microstructure of the molten layer for different pulse durations

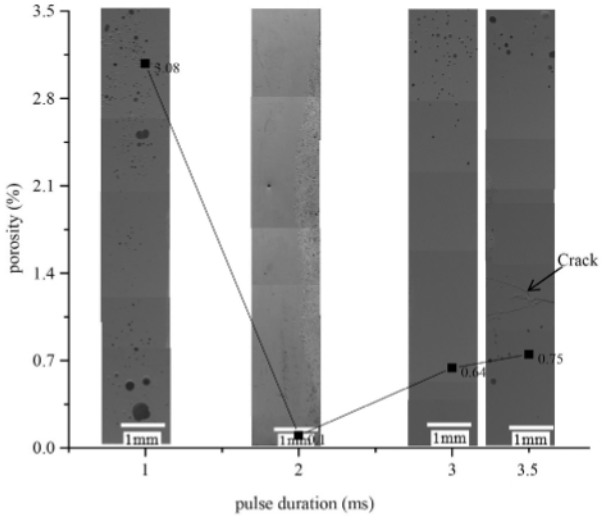

Figure 2: Distribution of pores and the porosity-variation curve of the sealing layer for different pulse durations

to escape from the sealing layer. However, an excessively large pulse duration will cause the solder to have a higher temperature than other conditions, resulting in a small amount of solder vapor inside the sealing layer, which may be the cause of an increase in the porosity of the pulse duration at 3 ms and 3.5 ms. At the same time, when the pulse duration was 3 ms and 3.5 ms, due to the occurrence of a "spheroidization" effect, grain coarsening during the cooling process led to an increase in melt viscosity and was not conducive to gas evolution. When the pulse duration was 1 ms, the number of pores in the sealing layer was large and widely distributed, and the porosity was 3.08 %. When the pulse duration was 2 ms, the pores were the least, and the porosity was only 0.1 %. When the pulse duration was 3 ms and 3.5 ms, the pores were fewer and small, and the porosity was only 0.64 % and 0.75 %, respectively.

Figure 3 shows the results of a line scan of the joint interface for different pulse durations. The thickness of the reactive wetting layer increased first and then decreased with the increase of pulse duration. The main reason was that when the pulse duration was 1 ms and 2 ms, the pulse duration increased, the liquid phase increased, enhancing the wettability of the solder and the glass. At the same time, the element diffusion at the interface increases with the increase of temperature, so the thickness of the wetting layer increased. When the pulse duration was 3 ms and 3.5 ms, the larger the pulse duration was, the more serious the grain coarsening was, which was not conducive to the wetting of solder and glass, so the thickness of the wetting layer become smaller and smaller.

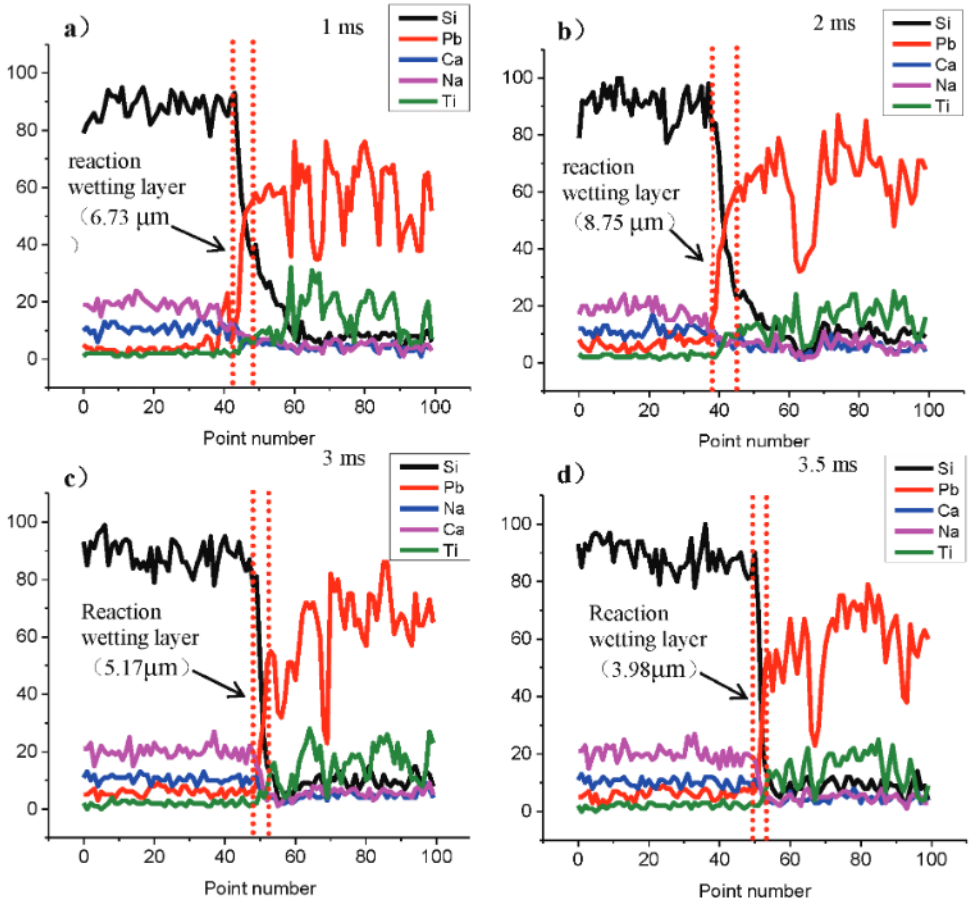

Figure 3: Results of line scan of the joint interface for different pulse durations

3.2 Effect of pulse duration on the mechanical properties of the welding layer

Table 4 shows the hardness, tensile strength and shear strength values of the welding layer for different pulse durations. Results show that the hardness of the welding layer increased first and then decreased and the peak appeared at the pulse duration of 2 ms. This was because when the pulse duration was 1 ms and 2 ms, the pulse duration increased, the liquid phase structure increased, enhancing the wetting and bonding effect between the solder particles. At the same time, the hardness was improved, and the tensile strength and shear strength were also increased. Increasing the pulse duration will decrease the mechanical properties, which is related to the grain coarsening of the molten layer.

By analyzing the above test data, it was found that when the pulse duration τ was 2 ms, the morphology of the molten layer was better, and the porosity of the sealing layer was lower. Moreover, the thickness of the interface reaction wetting layer was large, and it had good mechanical properties. In summary, when the pulse current I was 160 A, the pulse frequency f was 18 Hz, the welding speed v was 90 mm/min, and the pulse duration τ was 2 ms, the best set of experimental data could be obtained.

Table 4: Hardness, tensile strength and shear strength values of the sealing layer under different pulse durations

pulse duration (ms)	hardness (HV)	tensile force (N)	tensile strength (MPa)	shear force (N)	shear strength (MPa)
1	425.23	192.1	0.47	982	2.46
2	432.47	252	0.63	1737	4.34
3	419.28	190.5	0.48	895	2.24
3.5	399.19	170.3	0.43	884	2.21

4 CONCLUSIONS

In this study the experimental materials and methods of laser-welded glass were introduced. Then the effect of pulse duration on the micro-morphology and mechanical properties of laser-welded glass joints was studied. The conclusions are as follows:

1) The larger the pulse duration, the longer the heat source acted, making the solder absorb more energy, forming more liquid phase and reducing the melt viscosity significantly.
2) The porosity of the sealing layer decreased first and then increased with the increase of pulse duration.
3) When the pulse duration was 1 ms and 2 ms, the bubbles had enough time to escape from the sealing layer. When the pulse duration was 3 ms and 3.5 ms, due to the "spheroidization" effect, grain coarsening during the cooling process led to an increase in the melt viscosity and was not conducive to gas evolution.
4) When the pulse duration was 1 ms, the number of pores in the sealing layer was large and widely distributed, and the porosity was 3.08 %. When the pulse duration was 2 ms, the pores were the least, and the porosity was only 0.1 %. When the pulse duration was 3 ms and 3.5 ms, the pores fewer less and small, and the porosity was only 0.64 % and 0.75 %, respectively.
5) Increasing the pulse duration will decrease mechanical properties, which is related to the grain coarsening of the molten layer.

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