

Wettability of Laser Surface Modified Soda Lime Silica Glass

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1. Introduction

Soda lime silica glass is widely used in almost all engineering activities due to its unique characteristics that most materials are unable to replicate. Having good mechanical, thermal, electrical and optical properties has made this material use up to 80% of ceramic usage around the globe [1,2]. Recently, modification on glass surfaces has attracted the intention of researchers as the capability to extend the glass properties to exhibit anti-slip, anti-fog and self-cleaning features. Demand from the industries also creates the desire to improve the material's characteristics while maintaining its properties. Surface modification of glass using a non-contact technique is quite limited in research activities as this process creates a lot of defects on the glass surface, such as cracks and decreasing light transmissibility properties.

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Current mechanical and chemical processes such as polishing, etching, grinding, and blasting have significant drawbacks, especially in the processing time, while certain processes generate fumes and silica dust that are very hazardous for our environment and mankind [3–5]. The involvement of hazardous chemicals in the surface modification process contradicts the Sustainability Development Goals (SDG) objectives and can be dangerous if the usage of these chemicals is uncontrolled properly. Toward 2023, an increasing trend in surface wettability studies has been reported by Jothi and Prasanth [6] and supported by Liu *et al.,* [7] where laser processing on surface modification is the future technology in surface modification.

Experimental works and research focusing on modified glass surfaces using laser processing started to evolve over the past few years as laser processing has shown great advantages and are effective in transforming the glass surface wettability [3,8–14]. Even though these researchers are focusing on glass surface modification, fewer authors are applying the optimization process on the laser parameters to achieve better outcomes based on desired aspects. Besides that, the effect of laser parameters using Design of Experiment (DOE) methods also is not in favour of most researchers. This study intended to fill the research gap while extending knowledge on laser surface modification on glass.

Crack formation on the glass during laser processing has been a major setback in processing glass using thermal application. Uncontrolled heat induced by glass created inconsistent thermal expansion and contraction, thus resulting in micro-cracks [11,15]. A detailed study made by Nategh *et al.,* [10] reported that no intensive study had been made on laser parameters optimization of soda lime glass. The authors also concluded that increasing the cutting speed is more preferred than decreasing the pulse energy in minimizing crack formation.

Research on soda lime silicate glass using 355 nm UV laser by Dinh *et al.,* [3] has found that improvement in the wettability occurred after laser processing and drastic transformation to superhydrophobic properties occurred after the specimens were heated. The effect of frequency has been studied by Yang *et al.*, [16] while working with silica glass coated with TiO₂, where frequency is one of the parameters influencing surface topography that is reflected in contact angle. Besides, by varying the laser power, the surface roughness can be altered, and a higher contact angle can be achieved [17].

A comparison between line hatch and cross hatch on silica-based glass has found that cross hatch produces a higher contact angle compared to line [11]. A similar result was found in 2017 [13], where comparisons between line spacing were evaluated on water contact angle. A recent study using a 1064 nm wavelength laser on ultra-white glass has found that the hatch doesn't greatly affect the hydrophobicity, but the microgroove size has a bigger effect on the water contact angle [14]. Studies on groove formation through variation in laser power, scanning number and hatch spacing were conducted by Ouchene *et al.,* and Liao *et al.;* where both papers reported that glass surface wettability is highly correlated with the micro-nano structure, also known as groove formation, depending on the laser parameters used [18–20].

Based on the literature review and previous studies, most authors are using low surface energy agents to increase water contact angle after laser processing in various methods. The salinization and immersion method is the most common method used in reducing the surface energy of silica-based working specimens [16,17,21–24]. Besides that, some others are using the vapour deposition method as a secondary process to achieve superhydrophobicity in glass [25,26].

From the introduction, a lot of methods and factors affect the modification of surface energy of laser-modified glass. This research intended to study the effect of processing parameters on 1 mm thickness Soda Lime Silica (SLS) glass using a 1064 nm wavelength laser with evaluation on the water contact angle according to ASTM D7334-08 standard. A D-optimal RSM with a variation in hatch style, hatch spacing, frequency and scanning speed is studied to understand the effect of each parameter chosen while proposing the optimal combination of parameters to achieve the desired outcome. Besides that, the threshold value for crack formation is reported in the final finding for other researcher reference. The final mathematical model was also generated to provide early prediction to other researchers on improving the water contact angle based on selected parameters.

2. Methodology

2.1 Materials

A 1 mm thickness glass with the size of 76x25x1 mm³ microscope slide (CAT 7101 FisherBrandTM) with the composition i[n Table 1](#page-2-0) was chosen to be the working material for this project. The selection of this material is based on commercial usage of this type of glass and the capability of the result to reflect the most common glass used in the world [1,27]. The sample underwent pre-treatment and standard cleaning procedure using ethanol and deionised water for 5 minutes each before submersion in an ultrasonic bath[13,14,26]. The final cleaning procedure was nitrogen gas with a purity of 99.9% cleaning to ensure the sample surface was free from any contamination before laser processing. Figure 1 illustrates the sample preparation process before undergoing laser surface processing.

Fig. 1. Material Cleaning Process

2.2 Methods

Table 1

Laser processing was performed using a 50-watt CK-FB3D50 laser machine operated at 1065 nm wavelength with scanning galvo (X-Y axis) application to control the beam movement. The execution process is illustrated in Figure 2, and the constant parameters are listed in [Table 2.](#page-3-0) By applying the direct method texturing, a 0.05 mm beam diameter was focused on the top glass surface with the detailed beam movement as in Figure 3. Laser beam path, also known as groove, is mentioned as a hatch in this paper, was varied in term of spacing and style. [Table 3](#page-3-1) tabulated the manipulated parameters for determining the wettability transformation of laser-modified SLS glass.

Table 2

Fig. 2. Laser processing on glass with different hatch style

 Table 3

During the experimental process, the laser power was kept at maximum value to maximize the machining capability. The process is in ambient pressure and room temperature; the surface modification process was conducted for a total of 28 runs based on the D-optimal method RSM using Design Expert[™] software. Sample processing was designed using four parameters of hatch spacing, hatch style, scanning speed and frequency, as shown in [Table 3.](#page-3-1) Hatch style was selected as the categorical factor compared to others that were set as numerical factors. Three levels were selected for numerical factors, while two levels were set for the categorical factor. To ensure the consistency of the experiment, seven lack-of-fits and seven replicate points were selected, with some axial check points used as a run experiment to generate the result for contact angle. The usage of D-optimal RSM was to ensure the distribution on check points is equivalent for both categorical factors thus resulting a better accuracy on the design of experiment result.

Fig. 3. Illustration on the experimental operation

2.3 Characterisation

Visual inspection of the glass surface using a Scanning Electron Microscope (SEM, JEOL JSM-IT200) was conducted to investigate any defect or crack at the glass surface. Crack formation in glass surface modification was reported by various authors, and in most cases, a large thermal effect induced on glass has generally formed cracks [11,15]. Understanding the threshold value of thermal exposure on soda lime glass could be beneficial to other researchers in reducing the major defect that occurred during laser processing.

Evaluation on contact angle and surface wettability were done according to ASTM D7334-08, where the test liquid is Type II distilled water in accordance with specification ASTM D1193-06. A Hamilton™ syringe with an exact droplet of 10 μL distilled water was used to measure the water static contact angle on the glass-modified surface. An average of five sets of measurements were taken at different positions to ensure the consistency of the glass-modified surface. Measurement on each contact angle was used using ImageJ TM software using the drop-analysis LB ADSA plugin. The initial Contact Angle (CA) was 56.7° (Figure 6(b)), indicating hydrophilicity on the pristine glass. Before each CA measurement, modified glass was cleaned using 99.9% ethanol and deionized water to remove unwanted particles and contamination.

2.4 Statistical analysis

Analysis of variance (ANOVA) was used to determine the contribution of each factor while identifying the significant factors or their interaction. Model fit test, analysis of variance with 95% confidence level and fit statistic were performed to analyze the response analysis to each dedicated factor. A mathematical model equation was developed for contact angle response based on Eq. (1) before the optimization process to validate the model equation. With the understanding of each factor's contribution to the response, the optimization process on the static contact angle has been done with three validation tests to measure the accuracy of the statistical analysis.

$$
y = b_0 + \sum b_{ii} X_{ii} + \sum b_{ii} X_{ii}^2 + \sum b_{ij} X_i X_j + \varepsilon
$$
 (1)

3. Results

Table 4

3.1 Surface Inspection

Visual inspection using a scanning electron microscope on glass showed that certain parameters happened to form microcracks on the glass surface. This clear microcrack at the surface was found on some samples that operated using a cross-hatch pattern at 60 kHz, which is equivalent to 0.0204 $J/mm²$. The crack becomes worse as the spacing is at 0.06 mm due to unresolved heat and thermal differences. Runs 7, 14, 15, 17 and 23 generate clear crack propagation starting at the laser beam path toward the non-beam movement area. A similar result was found by Kang and Shin[28] while working on 1.1 mm thickness soda lime glass using a $CO₂$ laser. The author reported that the sample was broken and generated a critical crack at 0.0203 J/mm² while operating above the laser energy level, which could result in complete cutting. [Table 4](#page-5-0) tabulated the results of this experimental work.

The threshold value for scribing a laser layer on 4mm thickness silica glass was reported at 0.0179 J/mm2, and damages during laser processing were due to the thermal expansion of the glass [10]. A similar result was found by Gao *et al.,* [29] while working on a modified quartz glass surface where a broken sample occurred at an energy density of more than 0.07 J/mm2. Crack-free grooves on glass can be produced by a UV laser with an accumulated laser energy of 0.0171 J/mm² [30]. The uncontrolled local heating from the laser beam due to the high sensitivity of glass to thermal shock may generate cracks and chipping; a higher speed should be considered to reduce the potential of surface fracture and cracking. In addition, microcrack was developed due to thermal expansion and contraction strain induced by laser plasma [2,10,14].

A frequent issue in laser processing on glass is cracks, and overcoming this defect has been a major setback in laser processing on silica-based materials. The reason for these damages is due to the high coefficient of thermal expansion of soda lime silica glass at 9×10^{-6} /K, which is significantly higher than metal and other materials. This issue can be resolved by reducing the energy density, but minimum energy is always required to generate grooves and texturing of the glass. Differences in threshold energy density reported by previous research are seen in Figure 4 and [Table 5.](#page-6-0)

Fig. 4. Comparison table for threshold value based on literature

Figure 5 shows the micrograph of cracks that occurred on the modified glass surface. From the observation made, most micro-cracks tend to happen at beam-overlapping paths, especially on crosshatch patterns. The overlapping path is where high thermal energy accumulates, increases the thermal effect, and creating thermal shock and fracture, thus, resulting in microcracking [10,15]. On the other hand, crack-free surfaces are also found in most runs where thermal energy is able to dissipate through the glass, thus forming a nice textured surface.

Fig. 5. Microcrack happened on the modified glass surface at 0.0204 J/mm2

3.2 Contact angle

The contact angles recorded were scattered between 49° to 92.3° (as i[n Table 4\)](#page-5-0) for a total of 28 runs compared to pristine glass, which is 56.7°, as shown in Figure 6(b). The static contact angle of pristine soda lime glass matches with previous works [3,14,31]. By using Type II distilled water in accordance with specification ASTM D1193-06, the transition of wettability for modified glass occurred significantly. For most of the run, the wettability of modified soda lime silica glass reduced and transformed into a hydrophobic phase, while at certain parameters, the modified glass became more hydrophilic. Figure 6(a) illustrates the scatterplot of the water contact angle for all runs. The horizontal red line across the scatterplot indicates the transition of wettability of the laser-modified glass.

Fig. 6. Result for contact angle: (a) a total of 28 runs, (b) pristine glass, and (c) laser-modified glass

From the total runs, it was found that five samples improved their wettability characteristic on the laser-modified surface. Samples 7, 14, 16 and 25 were textured with a hatch spacing of 0.5mm. The spacing is an integral effect in determining the surface wettability of the glass. On the other hand, the transformation of hydrophobic occurred on samples of 0.06mm hatch spacing. Spacing between laser beam paths is crucial in determining the contact angle [3,11–14,17]. Factors such as surface roughness, hatch spacing and style affect the wettability of laser-modified glass [14,31]. Figure 7 illustrates the water contact angles at different hatch spacings.

Fig. 7. Water contact angle of laser-modified glass at different hatch spacings

3.3 Analysis of variance (ANOVA)

[Table 6](#page-9-0) summarises the result of ANOVA for each factor and their interaction. A significant model was obtained from the ANOVA and a fit data distribution. By using Design ExpertTM software, this statistical software is able to produce an accurate result on the relationship between factors and their interactions. Based on the ANOVA result, the model F-value of 8.52 indicates less than a 0.01% chance that the model depends on noise and variation. A model with a p-value less than 0.05% shows that the models are reasonably good and suitable for further analysis. In this design model, significant factors are A, D, AB, AD and CD. All significance was selected with the p-value less than 0.05 calculated in ANOVA.

The percentage contribution of each significant factor can be determined from the distribution of the F-value. Based on the calculation made, factor A (hatch spacing) and factor D (hatch style) contributed approximately 28% and 9%, respectively, to the contact angle of laser-modified glass. The mathematical model in coded factor for the resulted contact angle is listed below as in Eq. (2). From this mathematical model, prediction on the response can be made at any value within the operating range.

 $contact angle = +70.08 - 6.67A - 1.09B - 1.28C - 3.34D + 6.10AB -$ 0.8147AC + 5.64AD + 1.90BC - 3.10BD - 5.10CD

3.4 Effect of laser process parameters on the contact angle

A comparison of the effect of each parameter on the contact angle for all factors can be seen in Figure 8, where the biggest slope of the graph represents the most significant factor on the response. The result of the perturbation plots is aligned with the model developed in ANOVA [\(Table 6\)](#page-9-0), and its main factors effect on the response. Negative interaction between hatch spacing and static contact angle can be concluded based on the perturbation plot shown in Figure 8. Contributing up to 28% on contact angle, this factor is the most significant factor to be controlled in order to modify the wettability of soda lime silica glass. This interaction is due to the groove formation from the laser beam that generates a micro pit. The smaller hatch spacing created a narrower micro pit, thus storing more air and forming a layer of air cushion and cavities between the glass surface and water. Meanwhile, having a larger hatch spacing enables the water droplet to penetrate into the gap of the adjacent pillar, thus spreading rapidly, producing better wettability. The comparison of water contact angle on different hatch spacings can be seen in Figure 9. The same trend was reported by various authors on the hatch spacing as smaller hatch created more micro/nanostructure and generated an air gap between micropillar that transformed surface wettability into hydrophobic state [3,11– 14,17].

A recent study on 2 mm thick soda lime glass using femtosecond laser found that the optimal setting for producing a superhydrophobic glass is with 0.06 mm spacing with a carbon polymer plasma deposition process [32]. Meanwhile, a previous study on a 0.04 mm groove spacing resulted in a bigger water contact angle compared to a 0.08 mm groove spacing while working on flat soda lime glass [18]. Both results reported were in agreement with current findings that smaller hatches reduce the wettability of soda lime glass and transform the hydrophilic glass into a hydrophobic state.

 (a) (b) **Fig. 8.** Perturbation plots illustrating the effect of each factor on contact angle at 0.28 mm hatch spacing, 50 mm/s scanning speed and 90 kHz frequency; (a) cross-hatch and (b) line hatch

Fig. 9. Comparison water contact angle for different hatch spacing at 20 mm/s scanning speed, 120 kHz frequency and cross-hatch style; (a) 0.06 mm; (b) 0.5 mm

From the perturbation, it is clearly seen that the cross-hatch midpoint (Figure 8(a)) is higher compared to the line-hatch (in Figure 8(b)); indicating a highly significant effect compared to the line style of hatch. The hatch pattern effect is in agreement with previous works as the hatch pattern affects the surface area that is in contact with water droplet, thus resulting in changes in surface wettability [33–35]. Comparison between different styles and patterns of the hatch on water contact angle aspect can be seen in Figure 10. Based on this figure, the same laser processing parameters (hatch spacing = 0.28 mm; speed = 50 mm/s; frequency = 60 kHz) except different hatch styles were used on sample 11 and 12; resulting significant differences between them in term on static contact angle result.

The reason behind this interaction was subject to the surface area and micro pit formation from the laser beam movement. Forming a cross-hatch style reduces the surface contact area with water and produces many more micro pillars compared to the line hatch. A higher number of micropillars has the ability to support the water droplet sphere shape by storing more air gaps, thus increasing the water contact angle. Zhao *et al.,* [11], Nguyen *et al.,* [12]*,* and Bakhtiari *et al.,* [22] applied different patterns and hatching styles on silica-based material using laser processing method. They indicated creating more micro/nanostructures on the silica-based surface results in better air traps, thus creating better hydrophobicity and a higher possibility of detecting water droplets.

 (a) (b) **Fig. 10.** Contact angle result for different styles of hatch; (a) cross-hatch for sample 11; (b) line hatch for sample 12

The effect on speed and frequency is found to be less significant on the response. Positive interaction on the cross-hatch while negative interaction for line-hatch has shown that these parameters don't contribute much to surface wettability transformation. Results from statistical analysis and ANOVA, in [Table 6,](#page-9-0) indicate that these factors are not significant to the model term generated, as in agreement with the results reported by Jing *et al.,* [14] and Liao *et al.,* [26].

Contoured and 3D surface plots for cross and line hatch illustrated in [Table 7](#page-12-0) indicate the effect of laser processing parameters on the surface wettability transformation for soda lime silica glass. [Table 7](#page-12-0) illustrates the resulting water contact angle response on the laser processing parameter, namely, hatch spacing, scanning speed, frequency and hatch style. Comparing the cross and line hatch, the cross-hatch has more red area, indicating a higher contact angle; it shows a significant transformation to soda lime glass wettability. Meanwhile, for both graphs, having a smaller spacing resulted in a higher water contact angle. This statement was in agreement with the details mentioned in the previous paragraph.

Table 7

3.5 Optimisation

Based on the DOE, the optimization of laser processing parameters was predicted using statistical software before the result was validated through experimental works. A single criterion has been chosen, that is, to obtain the highest contact angle with the maximum speed that could be achieved using the selected parameters within the machine range. Speed was selected at maximum value to expedite the process, thus reducing the actual cost especially if the selected parameters were used in industry. [Table 8](#page-12-1) and [Table 9](#page-13-0) tabulate the criteria of optimization and experiment validation. Since the statistical software proposes multiple solutions with the highest desirability up to 95.1%, three random tests were checked to evaluate the margin of error compared to the predicted model by the statistical software. With an average margin of error of less than 10%, this confirms and concludes that the model developed through statistical analysis was within reasonable confidence and successful.

Table 8

Table 9

4. Conclusions

This experimental work and study investigated the effect of laser processing parameters on the surface wettability of soda lime silica glass. On the surface analysis, a crack-free result was obtained by texturing the soda lime glass less than the threshold value of 0.02 J/mm². Implementing multipath above this range induces a constant thermal effect on the working material that results in cracks.

From the statistical analysis made, hatch spacing and hatch style are the most significant factors in determining the water contact angle. It is calculated that these laser parameters recorded up to 28% and 9% contribution to the response, respectively. Having a cross-hatch with small spacing introduces hydrophobicity on pristine glass. This is due to the formation of microstructure after laser processing, where more micro/nanostructure results in transformation of soda lime glass wettability. Having a water contact angle of 92.3°, the transformation of a hydrophilic soda lime glass into a hydrophobic state is possible without using any chemical or low surface energy agent.

The optimization process that recorded less than 10% error supports the experimental work and thus validates the mathematical model developed. Therefore, selecting suitable and ideal laser processing parameters is crucial in determining whether the outcome improves the surface wettability or vice versa.

Since thermal induced using laser processing has various influencing factors, achieving dedicated wettability still requires further research study. The method of creating micropillar and pit could be optimized by incorporating other factors such as assist gas. Another important issue of laser surface modification is associated with improving processing time and efficiency. Future work in improving the processing time and cost is welcomed to improvise this method for better output and less chemical modification in the post-processing method.

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