

## Article

# Effect of Laser Conditioning on Surface Modification and Laser Damage Resistance of SiO<sub>2</sub> Antireflection Film

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**Abstract:** SiO<sub>2</sub> sol-gel antireflection film coated on fused silica can reduce the reflection loss and improve the transmittance of the optical component, although it is still prone to laser induced damage. Laser conditioning is an effective way to improve the laser induced damage threshold (LIDT) of SiO<sub>2</sub> sol-gel antireflection film. In this paper, single-layer SiO<sub>2</sub> sol-gel antireflection films pretreated by triple-frequency laser with different parameters are characterized by the macroscopical parameters, such as transmittance, refractive index, and thickness. The law of surface modification and the defect removal mechanism of the SiO<sub>2</sub> sol-gel antireflection film by laser conditioning are obtained. It is found that laser conditioning can reduce the thickness of the film and introduce densification. In addition, laser conditioning can eliminate micro-defects, such as vacancies and voids in the preparation of SiO<sub>2</sub> sol-gel antireflection films, which is the main reason to improve the laser damage resistance of films. Finally, the laser conditioning process with three step laser energy combinations of (0.2–0.6–1.0) F<sub>th0</sub> (zero damage threshold) is the best one to obtain high transmittance, and excellent effects on structure modification and defect removal of films. The research in this paper provides data support for the engineering application and mechanism research of laser conditioning.



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**Keywords:** fused silica; laser conditioning; modification

## 1. Introduction

Fused silica are widely used in laser optical systems and space explorers due to high transmittance, high laser damage resistance, and excellent refractive index homogeneity [1–4]. However, fused silica optics are still prone to laser induced damage under long-term and high nanosecond (ns) laser fluences. This limits the load capacity improvement of optical components. Therefore, the reduction in laser transmission efficiency and the lifetime of optical components under the high-power laser have received special attention [5–8]. The optical transmission efficiency of the optical component can be greatly improved by depositing different types of optical films on the surface of the optical component. The basic principle is that the optical film coated on the surface of the optical component reduces the reflection loss, and thus, improves the transmittance of the overall optical component. Nevertheless, optical films are still prone to laser induced damage in laser systems, which limits the further improvement of laser system energy [9–11]. It is well demonstrated that initiation damage with nanosecond laser pulses arises from the presence of defects that either absorb the laser energy or can cause field intensification.

In order to extend the lifetime of optical components, many approaches have been taken to improve the laser damage resistance of optical films. In these methods, laser conditioning has been widely concerned [9,10,12–23]. Laser conditioning is an effective technique that uses sub-threshold energy to irradiate the surface of optical components to improve the laser damage resistance [12]. Additionally, this technique has showed beneficial results

in the practice of improving the performance of various optical components, although the exact mechanisms are not well understood. It is generally agreed that pre-exposure laser fluences, starting at low and incrementally increasing to higher laser fluences, can remove nodules to form small scale damage pits. Additionally, these small damage pits will not really have an influence on the performance of the optics used in the laser system [24]. In addition, there is a strong implication that the interaction of intrinsic and/or extrinsic defects with sub-threshold laser fluence performs an important role in an increase in the laser induced damage threshold of optics [25].

At present, several theoretical models are proposed to explain the mechanism of laser conditioning, including laser cleaning model, electronic defect elimination model, laser heating annealing model, and surface modification model [16,17]. Shao and Zhao have done a lot of research on laser conditioning of optical components, such as optical films and crystals. Additionally, the results show that laser conditioning is effective for threshold enhancement of optical components [14,15,18–20]. Yang Lihong studied the influence of laser conditioning on the micro-properties of optical thin films and found that laser conditioning has the effect of polishing and oxidation film [21]. Li's team studied the effect of laser conditioning on irradiated fused silica and found that laser conditioning could restore the reduced damage threshold after irradiation [22]. Jiang Yilan carried out a study on the effect of laser conditioning, and the results show that laser conditioning can effectively inhibit the occurrence of initial damage [23]. Wu et al. presented a method monitoring the laser conditioning effects on optical film based on the thermal reflectance principle. Their results demonstrated that the laser fluence controlled by surface reflectance response method has successfully suppressed the initiation damage during laser conditioning process and increased the laser induced damage threshold of optical film [26]. Kafka et al. performed a laser conditioning process with pulses in a rapid termination of laser energy to increase the laser damage performance by 2.5 times [27]. These studies enable us to more clearly understand the significant improvement effects of laser conditioning on the laser damage threshold of optical films, and the influence law of laser conditioning on the micro-structure of optical films. However, there is a lack of systematic research on the influence of laser conditioning methods and parameters on the surface modification corresponding to transmittance and refractive index of optical films. This will lack guidance for engineering application of optical films laser conditioning.

In this paper, the single-layer SiO<sub>2</sub> sol-gel antireflection film was pretreated by triple-frequency laser conditioning with different parameters. The changes of laser induce damage threshold, transmittance, and refractive index of SiO<sub>2</sub> sol-gel antireflection film under different laser conditioning parameters were systematically studied. These three parameters are very important in the engineering application of SiO<sub>2</sub> sol-gel antireflection film. In this study, we put forward the concept of LIDT growth rate, which is used to compare the enhancement degree of laser damage threshold of SiO<sub>2</sub> sol-gel antireflection film under different process conditions. This allows us to judge the influence of laser conditioning on the laser damage resistance characteristics of the SiO<sub>2</sub> sol-gel antireflection film more accurately. By studying the surface modification law and mechanism of laser conditioning on SiO<sub>2</sub> sol-gel antireflection film, combined with the LIDT growth rate of samples under various process conditions, the effect of laser conditioning surface modification on the laser induced damage resistance of SiO<sub>2</sub> sol-gel antireflection film is discussed.

## 2. Materials and Methods

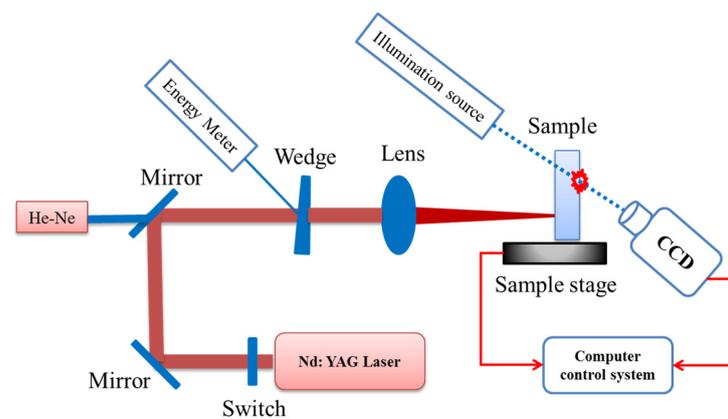
### 2.1. Sample Preparation

Fused silica samples (UV-grade fused silica) with a surface roughness of less than 1 nm RMS (root mean square) were used as substrates. The samples were etched by buffered hydrofluoric acid solution (the mixture of 2 wt% HF and 12 wt% NH<sub>4</sub>F), then treated by ultrasonic cleaning in order to remove the surface re-deposition layer. SiO<sub>2</sub> antireflection film of the fused silica samples were coated by the sol-gel process.

In order to avoid the error caused by the individual difference of samples, each sample is divided into several regions in the research. Each sample is divided into original coating area (0# area) and laser conditioning area. According to the designed laser parameters, laser conditioning area can be divided into several regions for verifying the effect of laser treatment on the laser induced damage threshold (LIDT) of samples. The range of laser irradiation and damage test for each region is not less than  $100 \text{ mm} \times 10 \text{ mm}$ . By using this method, the measurement error caused by individual difference in the process of laser induced damage threshold measurement can be avoided. That is to say, the more accurate test results can be obtained on the same sample under laser treatment. The samples of each batch in the research were divided by the same method, and the difference of the laser conditioning effect on different samples can be compared.

## 2.2. Laser Induced Damage Threshold Measurement System and Laser Conditioning Scheme

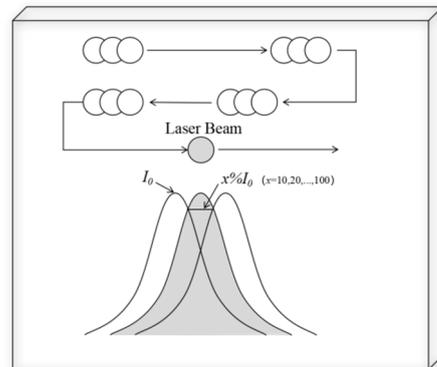
The laser induced damage threshold measurement system is shown in Figure 1. A 6.8 ns, 10 Hz, 355 nm Nd: YAG laser is employed in the experiment. The laser spot is near Gaussian distribution with a  $0.8 \text{ mm}^2$  laser spot area. The laser fluence was adjusted by an energy attenuator consisting of a half-wave plate and a polarizer. A He–Ne laser and a CCD camera are employed to detect the initial damage of laser action area in real time. An energy meter is used to measure the output laser energy separated from the wedged split plate with the maximum measurement energy range of 50 mJ. When the initial laser damage occurs, light scattering signals derive from the damaged sites in the surface of sample. These signals are caught by a scientific grade CCD camera coupled with a 3 times magnification objective lens. The illumination source is transmissive, which can make the CCD camera on the back surface of the sample observe the laser damage area more clearly.



**Figure 1.** The schematic diagram of laser damage test system.

To form a nearly uniform laser fluence on the  $\text{SiO}_2$  sol-gel antireflection film, laser conditioning was performed by raster scanning method. The laser conditioning process with raster scanning method also adopts the optical system, as shown in Figure 1. During raster scanning process, the spatial pulse-to-pulse overlap of x and y directions were set to 50% by adjusting the sample moving speed. The scanning frequency was 10 Hz. In order to determine a suitable combination of laser parameters, 1-on-1 method was used to test the damage probability of the film without laser conditioning. The 1-on-1 test measured the single-pulse LIDT by exposing each testing site to one laser pulse, and the zero-damage threshold were defined as the zero-probability damage threshold. The zero-damage threshold ( $F_{\text{th}0}$ ) was obtained by linear fitting from damage probability curves, and  $0.1 n F_{\text{th}0}$  ( $n = 1, 2, 3 \dots$ ) was used as the incremental energy ( $n$  selected according to the design). Finally, the sample surface was scanned by the raster scanning mode, as shown in Figure 2. After laser conditioning, the laser induced damage threshold measurement was performed immediately, and at least three different damage probabilities were tested during the experiment. The test points of each damage probability were no less than 20,

and the damage probability curves under different conditions were drawn. In order to make the experiment more comparative, the damage threshold was converted into laser energy at 3 ns.



**Figure 2.** The schematic diagram of laser conditioning scanning mode of film.

The laser induced damage threshold of the samples with different substrates is different. In order to accurately judge the laser damage resistance of SiO<sub>2</sub> sol-gel antireflection film by laser conditioning, we propose a concept of “LIDT growth rate”, as shown in Formula (1):

$$\text{LIDT growth rate} = \frac{\text{LIDT}_{\text{laser conditioning}} - \text{LIDT}_{\text{as-grown coating}}}{\text{LIDT}_{\text{as-grown coating}}} \quad (1)$$

where  $\text{LIDT}_{\text{laser conditioning}}$  is the LIDT value of laser conditioning sample, and  $\text{LIDT}_{\text{as-grown coating}}$  is the LIDT value of as-grown coating.

### 2.3. Structural Characterization

The structure changes of the samples after laser conditioning were characterized by measuring the macroscopically parameters, such as transmittance, refractive index, and thickness. The transmittance was characterized by ultraviolet-visible spectrophotometer. Air is used as background for zero correction, and the surface of the sample is perpendicular to the measurement light. In order to ensure the objective reality of the data, the measured data have not been made any amendments, such as data smoothing and baseline correction. The film thickness and refractive index were measured by an ellipsometer. The measurement accuracy of film thickness can reach 0.1 nm, and the measurement angle is 70°. The measurement wavelength is 400–800 nm that in the visible and near-infrared bands due to the strong absorption of organic groups in the UV membrane. Using the classic Cauchy model, the film thickness and refractive index are calculated on the fitting of measured data. The fitting software is provided by the equipment.

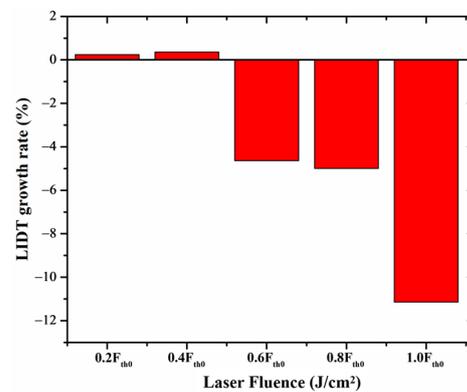
## 3. Results and Discussion

### 3.1. Single-Step Laser Conditioning Process

It is demonstrated that laser fluence is one of the key factors of all the parameters during the laser conditioning process [28]. Additionally, it is necessary to adjust the laser fluence accurately to achieve the effect of laser conditioning. It is difficult to remove the damage precursor and laser absorbing defect when the laser fluence is too small. On the contrary, laser induced damage will result if the laser fluence is too high during the laser conditioning process. Therefore, precise control of laser fluence parameters before laser damage-initiation can optimize laser conditioning process.

Based on the description of Section 2.2 in this paper, the zero-damage threshold  $F_{\text{th}0}$  of the SiO<sub>2</sub> sol-gel antireflection film without laser conditioning was obtained by 1-on-1 damage testing and linear fitting (the zero damage threshold of the film without laser

conditioning was  $8.04 \text{ J/cm}^2$  in the experiment). Then, different regions of the same sample were treated with  $0.2 F_{th0}$ ,  $0.4 F_{th0}$ ,  $0.6 F_{th0}$ ,  $0.8 F_{th0}$ , and  $1.0 F_{th0}$  laser fluence, respectively. After laser conditioning, the zero-damage threshold  $F_{th0}$  by 1-on-1 damage testing and linear fitting was measured, respectively, and compared with the  $\text{SiO}_2$  sol-gel antireflection film without laser conditioning. The result is shown in Figure 3. It can be seen from Figure 3 that in a single-step laser conditioning region of  $\text{SiO}_2$  sol-gel antireflection film, the zero-damage threshold drops rapidly when the laser fluence in the treated region reaches or exceeds  $0.6 F_{th0}$ . Only when the laser fluence is lower than  $0.6 F_{th0}$  is there a certain laser conditioning effect, but the laser induced damage threshold is not significantly improved. As shown in Figure 3, the LIDT growth rates of the  $\text{SiO}_2$  sol-gel antireflection film treated with  $0.2 F_{th0}$  and  $0.4 F_{th0}$  were 0.24% and 0.36%, respectively. Such small LIDT growth rates may be annihilated in the test error, and, obviously, they were impossible to have the engineering significance of improving the laser damage resistance of  $\text{SiO}_2$  sol-gel antireflection film. Therefore, the experiment shows that the single-step energy laser conditioning process does not have the effect of improving the laser damage resistance characteristics of  $\text{SiO}_2$  sol-gel antireflection film. If the laser fluence is too high, it is easy to greatly reduce the laser damage threshold of optical film, which has the opposite effect.



**Figure 3.** The LIDT growth rate of  $\text{SiO}_2$  sol-gel antireflection film after single-step laser conditioning.

### 3.2. Multi-Step Laser Conditioning Process

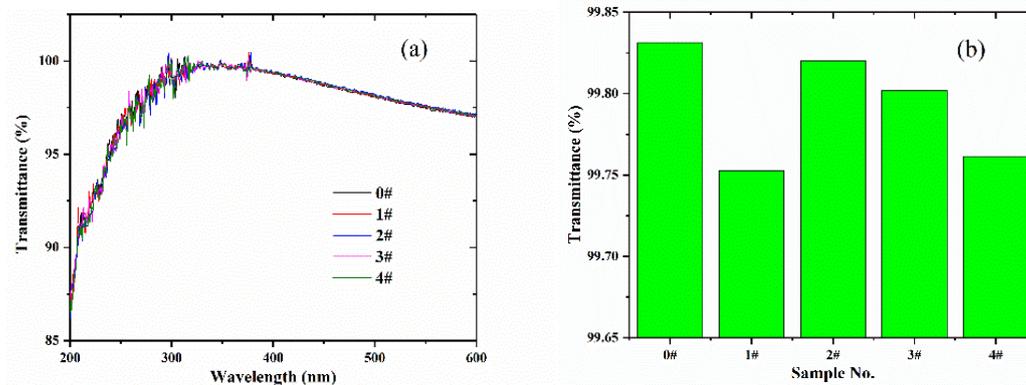
Table 1 shows the multi-step laser conditioning process parameters of the experimental samples.

**Table 1.** Laser conditioning parameters.

Sample Number	Laser Conditioning Scheme
0#	as-grown coating
1#	$(0.4-1.0) F_{th0}$
2#	$(0.2-0.6-1.0) F_{th0}$
3#	$(0.4-0.6-0.8) F_{th0}$
4#	$(0.2-0.4-0.6-0.8-1.0) F_{th0}$

We carried out the transmission characterization experiment on the ultraviolet—visible spectrophotometer. Figure 4a reflects the changes of transmittance of sample 0#–4# with wavelength. It is not clear how to reflect the influence of laser conditioning on the transmissivity of the film through Figure 4a, due to the small film transmittance change. Thus, the transmittance corresponding to the wavelength of 351 nm is extracted, and the histogram is used to reflect the change of transmittance. As shown in Figure 4b, the transmittance of sample 1# is 99.75%. Compared with the as-grown coating (sample 0#), the transmittance decrease rate of sample 1# reaches 0.8%, while the transmittance decrease rate of other

samples is below 0.7%. It is shown that the transmittance of the samples almost no obvious change after multi-step laser conditioning process.



**Figure 4.** Transmittance changes of film with different laser conditioning parameters: (a) the transmittance distribution as a function of wavelength; (b) the distribution of transmittance at 351 nm wavelength.

The results of ellipsometer test have some fluctuation at different points, so each sample is tested at five points. Figure 5 shows the ellipsometer test result, and the error bar is the error range of five tests. It can be seen that laser conditioning can significantly reduce the thickness of the SiO<sub>2</sub> sol-gel antireflection film, as compared with the as-grown coating (sample 0#). The direct-action processes of laser conditioning in SiO<sub>2</sub> sol-gel antireflection film are optical and mechanical processes, but the most basic one is thermal process. Through intrinsic absorption, impurity absorption, and nonlinear absorption, SiO<sub>2</sub> sol-gel antireflection film converts the laser energy into heat. Then, the structure of the film is changed by thermal melting or thermo-mechanical coupling. The compactness of SiO<sub>2</sub> sol-gel antireflection film increased obviously after laser conditioning. This indicates that the modification caused by laser conditioning can lead to the change of porosity of SiO<sub>2</sub> sol-gel antireflection film. In addition, it can be clearly seen that, the smaller laser energy interval between laser conditioning, the smaller change in film thickness. It is shown that the compactness of SiO<sub>2</sub> sol-gel antireflection film is increased significantly by the large energy interval between laser conditionings. That is, excessive laser energy interval has significant effect on film structure modification.

Figure 5b shows that the change of refractive index after laser conditioning is consistent with the change of film thickness. The refractive index of SiO<sub>2</sub> sol-gel antireflection film measured in our experiment exceeds 1.26. The main reason is that when we measure the refractive index of coating with an ellipsometer, the coating substrate is fused silica. However, the fused silica substrate itself is transparent, and the refractive index difference between the fused silica substrate and the coating is not very large. It will lead to a high refractive index measured by all samples. The results show that the compactness of the film increases, but the refractive index decreases. According to the refraction index Formula (2):

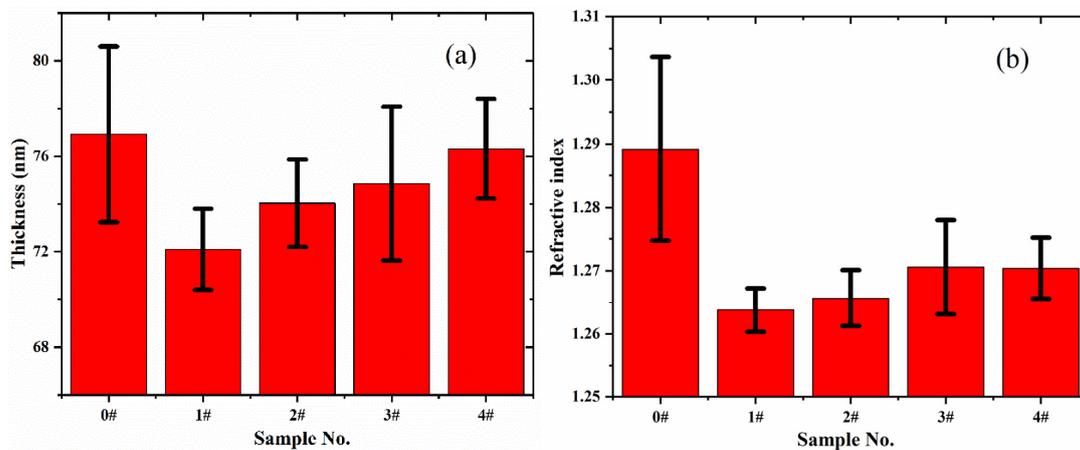
$$\frac{n^2 - 1}{n^2 + 2} \times \frac{M}{d} = R \quad (2)$$

where  $n$  is the refractive index,  $M$  is the molecular weight,  $d$  is the density, and  $R$  is the molecular refractive index.  $R$  can be obtained by the following Formula (3):

$$R = \sum R_i x_i \quad (3)$$

where  $R_i$  is the ionic refractive index (or atomic refractive index) of the ion (or atom) contained in the film, and  $x_i$  is the molar fraction of the ion (or atom). In the experiment, the thickness of the film decreases, indicating that the compactness of the film increases,

while the refractive index of the film decreases. This shows that the ion defects in the films decrease after laser conditioning, i.e., the  $R$  value decreases obviously. These results indicate that laser conditioning can eliminate the microelectronic defects, such as vacancies and voids in the preparation of  $\text{SiO}_2$  sol-gel antireflection film.

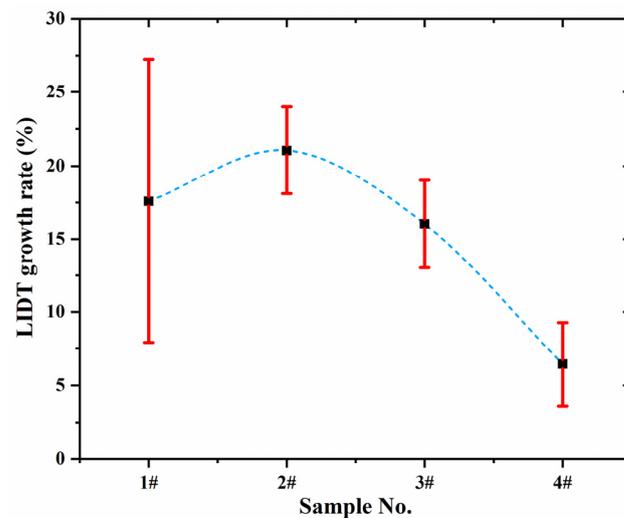


**Figure 5.** The changes of film layer (a) thickness and (b) refractive index with different laser conditioning parameters.

The thickness of the  $\text{SiO}_2$  sol-gel antireflection film changes to a certain extent after the multi-step laser conditioning process with a different parameter. It can be seen that the structure and densification of the  $\text{SiO}_2$  sol-gel antireflection film are changed. These results indicate that laser conditioning of  $\text{SiO}_2$  sol-gel antireflection film has obvious surface modification effect.

Based on the description of Section 2.2 in this paper, the zero-damage threshold  $F_{th0}$  of the  $\text{SiO}_2$  sol-gel antireflection film without laser conditioning was obtained by 1-on-1 damage testing and linear fitting. Then, laser conditioning was performed on different regions of the same sample through the laser conditioning process parameters shown in Table 1. In order to avoid individual fluctuation and test error, we carried out four experiments (corresponding to four samples). In each experiment, the same sample was used. Additionally, the laser conditioning process with different treatment parameters of 1#–4# was carried out for different areas on the surface of the same sample. Figure 6 shows the LIDT growth rate of sample 1#–4# (compared to sample 0#). The error bar reflects the fluctuation of the LIDT growth rate affected by the laser conditioning process.

As shown in Figure 6, the LIDT of laser conditioning process with the two step laser energy combinations (sample 1#,  $(0.4-1.0) F_{th0}$ ) is high, while the fluctuation of LIDT growth rate is very large. Based on the experimental results of the film thickness and refractive index of sample 1#, this combination of laser conditioning parameters has a great influence on the structure of the film. It is shown that the large laser conditioning energy gradient will lead to the obvious evolution of the film structure, which is also the main reason for the instability of the LIDT growth rate. The LIDT of laser conditioning process with the five step laser energy combinations (sample 4#,  $(0.2-0.4-0.6-0.8-1.0) F_{th0}$ ) also has an improvement of about 6%, and the fluctuation of LIDT growth rate is low. This is because the low-interval energy laser conditioning can steadily enhance LIDT. However, the LIDT growth rate decreases due to the long laser treatment time. The LIDT of laser conditioning process with the three step laser energy combinations (sample 2# and 3#) is also enhanced more than 15%, and the fluctuation of LIDT growth rate is low. It shows that the laser damage resistance of  $\text{SiO}_2$  sol-gel antireflection film after three step laser conditioning has a certain degree of stability.



**Figure 6.** Distribution of film LIDT growth rate with different laser conditioning parameters. Blue dash lines are used to aid the eyes.

The laser damage resistance of laser conditioning process with the two step laser energy combinations (sample 1#,  $(0.4-1.0) F_{th0}$ ) is great. However, the large laser energy gradient reduces the thickness of the film, then leads to the increased compactness and the decreased porosity. Thus, the micro-defects, such as vacancies and voids, are partly eliminated, which makes the laser damage resistance unstable. The laser damage resistance of laser conditioning process with the five step laser energy combinations (sample 4#,  $(0.2-0.4-0.6-0.8-1.0) F_{th0}$ ) is poor. We can see that the change of compactness is small through the change of film thickness. It shows that laser conditioning with the low-interval laser energy and multi-step laser energy combinations has a poor effect on the structure modification of  $SiO_2$  sol-gel antireflection film. Additionally, it is possible that new defects have been introduced after the laser conditioning process with the five step laser energy combinations, which results in ultraviolet absorption. Finally, negative effects are produced on the laser damage threshold of  $SiO_2$  sol-gel antireflection film.

Laser conditioning with three step laser energy combinations (sample 2# and 3#) can reduce the laser energy interval, and make the laser treatment time not too long. The transmittance of the sample is basically the same as the original sample. The improvement of laser damage resistance of samples 3# is worse than that of samples 1# and 2# because of the lower laser energy interval. Samples 2# ( $(0.2-0.6-1.0) F_{th0}$  three step laser energy combinations) exhibits high transmittance; additionally, it has favorable effects on film structure modification and defect removal. The results show that the laser conditioning process with  $(0.2-0.6-1.0) F_{th0}$  three step laser energy combinations is the best laser conditioning process for the  $SiO_2$  sol-gel antireflection film. The research in this paper provides experimental support for the laser conditioning mechanism of defect elimination and laser surface modification.

#### 4. Conclusions

The single-step laser conditioning process has no engineering significance to improve the laser damage resistance characteristics of  $SiO_2$  sol-gel antireflection film. The laser damage threshold of  $SiO_2$  sol-gel antireflection film can be significantly improved by multi-step laser conditioning process. This is due to the fact that laser conditioning can remove the microscopic defects, such as vacancies and voids, introduced in the preparation of  $SiO_2$  sol-gel antireflection film. In addition,  $SiO_2$  antireflection film after laser conditioning also has obvious surface modification effect, which significantly changes the structure of the film layer and the compactness of the network structure of the film. Finally, the laser conditioning process based on  $(0.2-0.6-1.0) F_{th0}$  three step laser energy combinations can

obtain high transmittance, favorable membrane structure modification and defect removal effect of SiO<sub>2</sub> sol-gel antireflection film, which is the best laser conditioning process scheme. The research in this paper provides experimental support for the engineering application of laser conditioning process and laser conditioning mechanism of defect elimination and laser surface modification.

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