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Original research article

SSD evolution model in HF etching of fused silica optics

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ABSTRACT

SSD (Subsurface damage) appears on optical elements after grinding, and do harm to the further quality improvement. HF etching can remove SSD and help following polishing processes, which improve polishing efficiency and quality. Based on the 2-D geometric model and the definition of SSD, a SSD evolution model in HF etching of fused silica optics is established. The SSD depth distribution of the samples after HF etching was measured by MRF (Magnetorheological Finishing) spotting test, and the results verified the validity of the model. The parameters of the model were also fitted according to the experimental results. This model can be used to predict the processing time of HF etching, ensure the complete removal of SSD, and improve the processing quality and efficiency.

1. Background

Fused silica components are generally processed by grinding, lapping, polishing and post-processing to achieve the final shape accuracy and surface quality. Grinding process leaves broken cracks and defects on the workpiece surface, which are hidden beneath the surface and cannot be seen directly, often referred to as SSD (sub-surface damage) [1]. In addition to reducing the service life, long-term stability and coating quality of optical components, SSD can directly affect the imaging quality and laser damage threshold of optical components [2–5]. In order to obtain optical components without SSD, it is necessary to further upgrade existing traditional processing methods to control the production of SSD; on the other hand, new processing methods such as MRF (Magnetorheological Finishing) [6], HF etching [7] can be introduced to remove SSD.

HF can react with SiO_2 and dissolve it. It has been widely used and studied in semiconductor industry [8]. Preston firstly studied the effect of HF on the glass sample after grinding, and proposed that HF could be helpful in detecting the ground glass [9]. Zhou employed HF to observe SSD of ground glass sample [10]. Wong studied the effect of HF on the ground fused silica sample, and indicated that HF can eliminate surface and sub-surface cracks [7]. The etching process of concentrated HF was studied systematically by Yong and the results showed that concentrated HF can be used as a connecting process between grinding and rough polishing to improve the machining efficiency and quality of optical components [11].

When describing the evolution of cracks in HF etching, Wong deduced the variation of crack depth with etching time based on a 2-D geometric model [7]. As the crack depth is difficult to be effectively measured, the initial value of the model is inconsistent with the experimental results. In this manuscript, the crack depth is characterized by SSD depth, and the evolution of surface and sub-surface cracks in HF etching process is described based on SSD depth.

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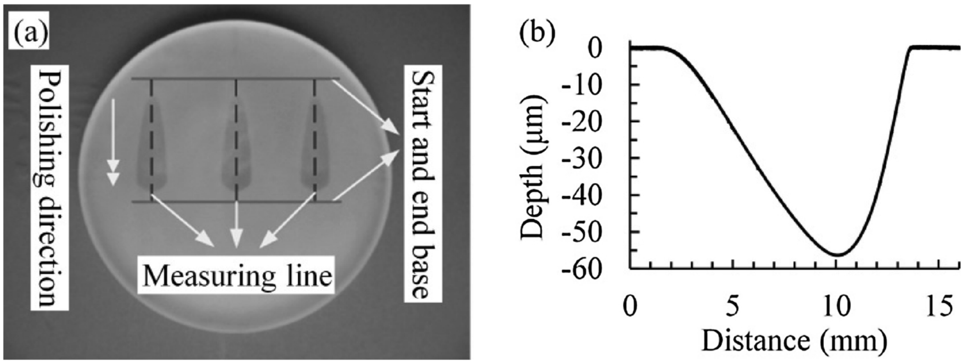

Fig. 1. SSD depth measuring method (a) MRF spots (b) The profile of a spot.

2. Theoretical backgrounds

2.1. SSD depth measurement

In order to study the characteristics of SSD and remove SSD, it is necessary to measure the depth of SSD. As the principle is to expose SSD for observation and measurement, it requires that no new SSD will be produced during the exposure process. Conventional exposure methods are polishing [12] and MRF [13]. The shearing removal mechanism of MRF allows the removal of materials without introducing new damage and is therefore ideal for SSD testing. Compared to the MRF wedge technique, MRF spotting method is relatively easier. The principle of MRF spotting is to generate spots on the surface of the sample by MRF to expose the sub-surface crack layer of the finished specimen, and to determine the surface crack layer depth according to the horizontal distance of the sub-surface crack and the MRF spot contour after acid etching. In this test method, the MRF spot acts as a sub-surface crack amplifier. The measurement of micro-scale crack depth is transformed into the measurement of millimeter-scale crack propagation distance in horizontal direction and the amplification ability of sub-surface crack can reach to hundreds of times.

The specific steps are as follows: Generate MRF spots (inclined pits) on the surface of the specimen, as shown in Fig. 1(a). The spot passes through the SSD layer to ensure that the depth of the spot exceeds the depth of the SSD to expose all SSDs. Using the profiler to measure the centerline contour of the spot from the starting base to the end base of the MRF spot. Fig. 1(b) is a typical center line profile. Then the cleaned specimen is placed on a linear mobile platform, and an optical microscope is used to observe the damage along the measuring line from the start base of spot. The moving distance of the platform was recorded when the damage disappeared, and the sub-surface crack depth could be obtained according to the profile of spot along the polishing direction.

2.2. 2-D geometric model [7]

When HF is used to etch the sample after grinding, the surface cracks will expand to intersect with each other and eventually disappear. An example of two scratches is analyzed to describe the change of scratches in the etching process.

Fig. 2 illustrates the propagation process of two scratches under the action of HF. The initial distance between these two scratches is s, and the depth of scratches is c. Assuming that the two scratches are the deepest scratches on the surface, the depth of the SSD is then determined by the depth of the scratch, i.e. the SSD depth equals c according to its definition.

At the initial moment when $t = 0$, scratches are covered. At this time, the depth of scratches can be measured by the MRF spotting method, which is the depth of the sample's SSD. After etching for t_1 time when $t = t_1$, scratches expand in all directions simultaneously. The scratches become wider and deeper during the etching process, and expand into two separate pits. As the surface of the sample is also corroded, the depth of the scratch remains unchanged and the value is still c.

As the etching progress makes the scratches grow wider and closer, the interval between them becomes thinner and thinner. When $t = t_2$, the two scratches overlap each other, and the interval between them disappears, resulting in a sharp reduction in the depth of the scratches. At this point the deep holes on the samples surface have all disappeared, only leaving some microstructures. Compared with the depth of the initial scratches, these microstructures are relatively small in height and can be regarded as the surface roughness of the sample. As the microstructures keep evolving during the etching process, its height keeps decreasing and the surface of the sample becomes smoother and smoother.

2.3. SSD evolution model

When describing the evolution of ground surface in HF etching process, Wong deduced the evolution model of scratch depth with etching time based on the 2-D geometric model [7]. This model describes the change of scratch depth during etching. At the initial stage of etching, the depth of the scratch remains unchanged. When $t = t_2$, as the scratches are expanded, the pits disappear and the depth of the scratches decreases sharply, which degenerates into the PV value of the surface roughness. As corrosion continues, the surface roughness decreases gradually. This model effectively describes the variation of scratch depth in the etching process, and the

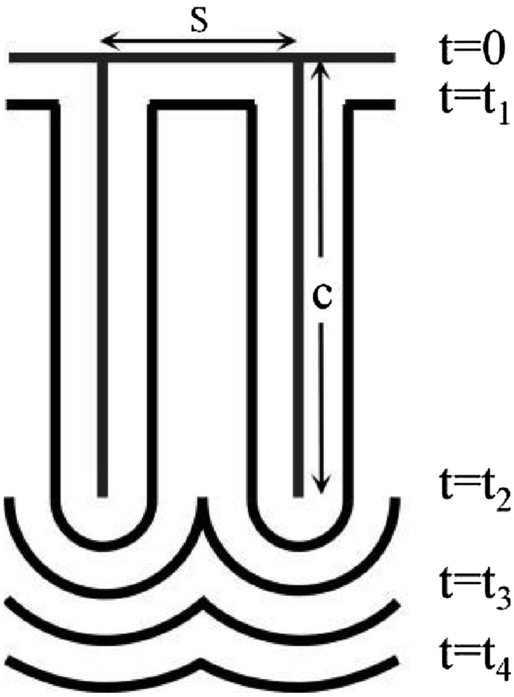

Fig. 2. 2-D geometric model of HF corrosion.

validity is also verified by experiments. However there is a defect in this model. As the scratches are deep and the width of the scratches is very narrow, the tip of the detector cannot penetrate into the scratch and draw the outline of the scratch. So it is difficult to measure the depth of the scratch directly and accurately. This is also why the initial stage of the model is not in consistence to the actual data.

Depending on the definition of SSD, the depth of the scratches can be considered to be the same as the SSD depth. At the initial stage of etching, the scratch depth remains basically unchanged, so the SSD remains unchanged. When the scratches intersect and disappear at $t = t_2$, the depth of scratches degenerates into surface roughness. As the scratches disappear, the depth of SSD can be considered as 0. Based on the above analysis, the SSD evolution model in HF etching process is proposed as follows:

$$d_{SSD} = \begin{cases} c, \; t < t_2 \\ 0, \; t \geq t_2 \end{cases} \tag{1}$$

In Eq. (1), d_{SSD} is the depth value of SSD and c is the initial subsurface crack depth. The critical time t_2 can be calculated by $t_2 = s/2r$, where r is the etching rate of HF and s is the distance between two adjacent cracks. It can be seen from Eq. (1) that in order to eliminate the subsurface cracks of the sample, it is necessary to etch enough time to make the adjacent cracks intersect with each other to reduce the depth of crack pits on the surface and remove the subsurface cracks.

3. Experiments and results

3.1. Experimental setup

13 pieces of fused silica sample with a diameter of 25 mm were selected for grinding with the same parameters. The specific grinding parameters were: a 270 # resin grinding wheel was employed, the wheel speed was 5000 r/min, the feed depth was 0.01 mm, line sweeping path was used to uniformly process the fused silica samples, X-axis feed speed was 1000 mm/min, and line spacing was 2 mm. After grinding, 1mm-thick material was removed from the surface to ensure the surface of all samples is in the same state. The sample was etched by a concentrated HF (49%HF) according to the parameters in Table 1. The SSD depth of each sample was measured by the MRF spotting method, and the profile of the MRF spot was measured by the Talysurf PGI 1240 Surface

Table 1
Experiment parameters.

Sample parameters	Φ25 mm fused silica sample												
Grinding parameters	270# resin wheel, speed 5000 r/min												
Sample number	1#	2#	3#	4#	5#	6#	7#	8#	9#	10#	11#	12#	13#
Etching time (min)	0	5	10	15	20	25	30	35	40	45	50	55	60

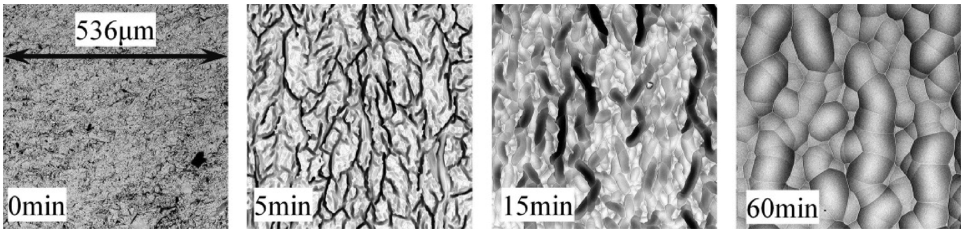

Fig. 3. The propagation process of cracks during etching.

Profiler.

3.2. Evolution of ground surface

A Phenom Desk top SEM model G2 Pro was used to observe the surface morphology of different fused silica samples after etching. Fig. 3 shows the results of the sample surface after HF etching for different times by the SEM. For the sample without corrosion, the brightness of the picture is not uniform and there were a lot of dark spots, which indicated that the surface of the sample is uneven. After 5 min etching, a large number of black stripes were observed on the surface of the sample. These black stripes represent cracks, and the darker the color, the deeper the cracks. The reason for these cracks are invisible at first is that the cracks are covered by residue after grinding and it is difficult to observe the shape of the cracks. After HF etching, the crack is slightly expanded and the residue on the surface is removed so that the crack can be clearly seen.

The crack width expanded with the increase of etching time. After 15 min of etching, the width of scratches increased obviously, and there were still dark spots on the surface, which indicated that the depth of these cracks was still deep. It was found that after a long period of 60 min etching, the adjacent scratches interlaced with each other and formed an uneven topography. There are almost no black areas on the surface, and the scratches have been completely removed. This will be confirmed by two examples of SSD depth measurements.

3.3. SSD measurement examples

The SSD depth of a sample after 5 min concentrated HF etching was measured by MRF spotting method, as shown in Fig. 4.

Fig. 4 shows the distribution of cracks at different depths of the sample. At 1.2 μm away from the surface, cracks are distributed extensively. With the increase of depth, the density of cracks decreases gradually, which indicates that the number of cracks with deep depth is decreasing. Sporadic cracks could also be observed at a distance of 38.2 μm from the surface. No cracks were found for the first time at a distance of 44.7 μm from the surface, and no further cracks were found, which suggested that the SSD depth of the grinded fused silica sample was 44.7 μm.

The SSD depth of a sample after 60 min concentrated HF etching was detected by the same method and the results are shown in

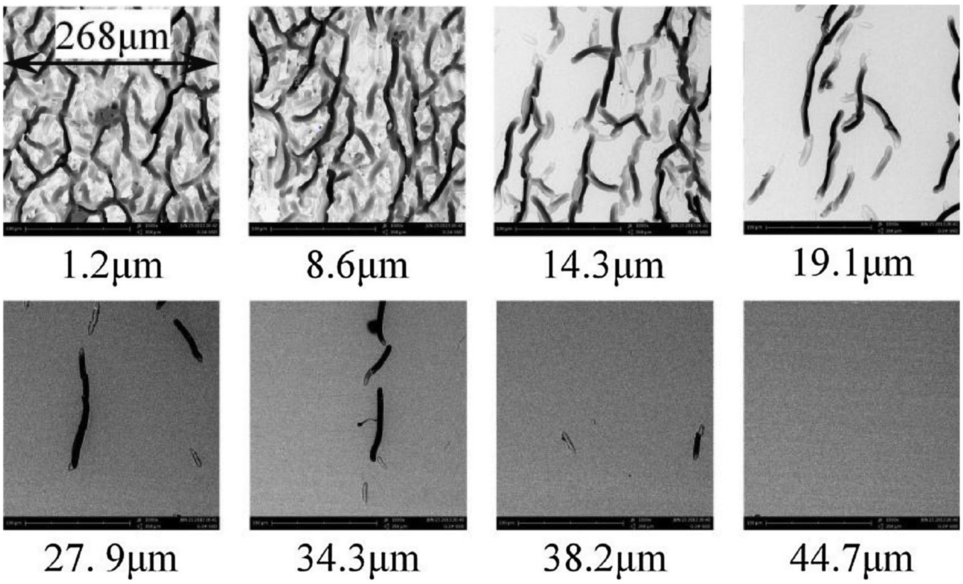

Fig. 4. SSD distribution of the 5 min etched samples.

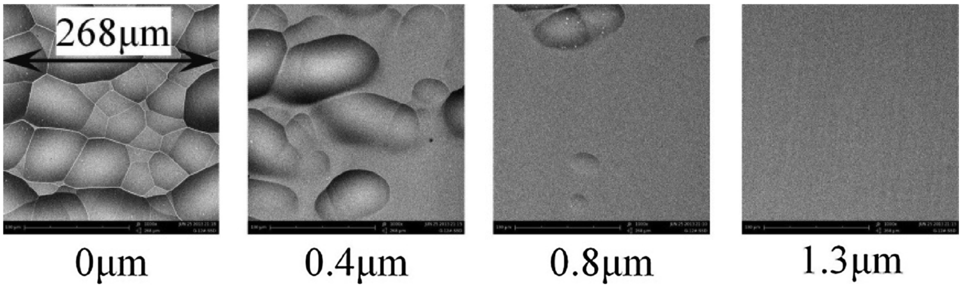

Fig. 5. SSD distribution of the 60 min etched samples.

Fig. 5.

Fig. 5 shows the distribution of cracks at different depths of the MRF spot. Some bumps and dents could be seen on the surface. No bulge or pit was observed for the first time at 1.3 μm from the surface, and no more bulge or pit was observed in deeper place, indicating that the SSD depth of the sample was 1.3 μm. Since the etched surface is not completely smooth but with a large number of convex and concave, the measured 1.3 μm SSD depth is actually the depth of residual concave and convex topography on surface, which means that the SSD of fused silica samples has been completely removed by HF etching.

4. Discussions

The SSD depth of 13 samples and the curve defined by Eq. (1) were drawn in a same picture (Fig. 6) to verify the effectiveness of the model.

The diamond points in Fig. 6 represent the SSD depth of the measured 13 fused silica samples. It can be seen from the point distribution that when the etching time is less than 35 min, the SSD depth remains basically unchanged, maintaining the SSD state after grinding; when the etching time exceeds 35 min, the SSD depth decreases sharply to about 0.

According to the evolution model in Eq. (1), we draw it in the graph with dashed line. The c in Eq. (1) will be the SSD depth of the ground sample, that is, 44.7 μm. The critical point t_2 for crack propagation is 35 min. Compared with the distribution of diamond points, it is found that the theoretical model is basically in agreement with the experimental SSD depth, which indicates that it is feasible to use the evolution model to describe the variation of SSD depth in the process of HF etching of ground samples.

An important purpose of this model is to find the critical point of etching. When we use HF etching to remove the cracks in ground samples, as long as the etching time reaches t_2, the cracks degenerate into the surface micro-morphology because of interlacing. It can be considered that the SSD depth of the element is zero, and the HF etching purpose has been realized. According to Eq. (1), it is necessary to know the HF corrosion rate and the distance between cracks to calculate t_2. The corrosion rate of HF can be measured by technological experiment, but the distance between cracks is difficult to describe by formula. It can be measured by the experimental method, then the critical time is calculated according to the $t_2 = s/2r$, and the time will be used as the processing time of HF etching. This can not only guarantee the totally removal of SSD, but also to ensure that no time will be wasted and improve the efficiency of processing.

5. Conclusions

Based on the 2-D geometric model and the definition of SSD depth, the SSD evolution model in HF etching process is presented. In order to verify the correctness of the model, a series of experiments were carried out. The experimental results show that the model can effectively describe the evolution of SSD in HF etching process. According to the experimental results, the parameters of the

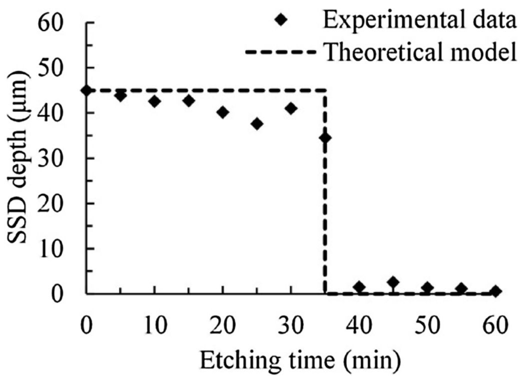

Fig. 6. Relationship between SSD depth and etching time.

model are fitted. The fitted model can be used to predict the least etching time to remove the SSD. HF etching for this time guarantees complete removal of the SSD and improve the machining efficiency.

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