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Effect of Chemically Etched Surface Microstructure on Tribological Behaviors

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Abstract: This study investigates the effect of the surface microstructure on the tribological characteristics of glass substrates. Chemical etching using hydrofluoric acid and ammonium hydrogen fluoride was employed to create controlled asperity structures on glass surfaces. By varying the etching time from 10 to 50 min, different surface morphologies were obtained and characterized using optical microscopy, surface roughness measurements, and water contact angle analysis. Friction tests were performed using a stainless steel ball as the counter surface to evaluate the tribological behavior of the etched specimens. The results showed that the specimen etched for 20 min exhibited the lowest and most stable friction coefficient, which was attributed to the formation of a uniform and dense asperity structure that effectively reduced the stress concentration and wear at the contact interface. In contrast, specimens etched for shorter (10 min) or longer (30-50 min) durations displayed higher friction coefficients and accelerated wear owing to nonuniform asperity structures that led to local stress concentration. Optical microscopy of the wear tracks further confirmed the superior wear resistance of the 20-minute etched specimen. These findings highlight the importance of optimizing the etching process parameters to achieve the desired surface morphology for enhanced tribological performance, suggesting the potential of chemical etching as a surface modification technique for various materials in tribological applications.

Keywords: Surface microstructure, Chemical etching, Wear resistance, Friction coefficient

1. Introduction

With the advancement of industrial technologies, there is a growing demand for high efficiency, precision, and longevity of mechanical systems. To achieve this, precise control of the tribological behavior at the contact interfaces between machine components, along with the improvement of individual component performance, is essential [1,2]. Wear occurring at the contact interfaces not only deteriorates the dimensional and geometrical accuracy of the components, but also leads to the degradation of material properties and the reduction of fatigue life, which are the main factors threatening the reliability and safety of mechanical systems [3,4]. Moreover, high friction coefficients at the contact interfaces decrease the energy efficiency and cause vibration and noise problems, negatively affecting the operational performance of mechanical systems [5,6]. Therefore, the development of precise surface modification technologies that can control the stress distribution and lubrication conditions at contact interfaces is urgently required to enhance the precision, reliability,

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and lifespan of machine components [7]. Generally, friction and wear are influenced by various factors, such as surface roughness, hardness, and lubrication conditions [8,9]. In particular, the surface roughness is closely related to the friction coefficient, which tends to decrease as the roughness decreases. Additionally, the friction and wear characteristics can be significantly improved by controlling the surface microstructure. For example, forming a regular pattern of micro-nano structures can reduce the actual contact area and friction force, and forming protrusions of appropriate size and shape can effectively discharge wear particles and suppress wear [10,11]. Therefore, the key to improving the durability and wear resistance of machine components is to optimize the surface structure to control friction and wear.

Previous studies have investigated the tribological properties of various materials and the effects of surface modification techniques on friction and wear behavior. For instance, Pratap and Patra studied the combined effects of tool surface texturing, cutting parameters, and minimum quantity lubrication (MQL) on the microgrinding of BK7 glass [12]. They found that tools with smaller and more numerous texture units reduced the cutting forces, surface roughness, and edge chipping compared with untextured tools. Jiang et al. prepared bulk metallic glass (BMG)/graphite composites with a 3D lubricating layer and evaluated their tribological properties under dry sliding and in seawater [13]. The synergistic effect of graphite lubrication and the microtexture significantly reduced friction and wear, although seawater affected the reliability of the composites owing to corrosion.

This study aimed to systematically analyze the effect of surface structure on wear resistance using a glass material, which is inexpensive, easy to process, and has a uniform surface. This is similar to the approach used in previous studies that utilized silicon wafers to investigate friction/wear mechanisms [14]. The lubrication characteristics of glass surfaces play a crucial role in various mechanical components, especially in precision machinery, optical devices, and Micro Electro Mechanical Systems (MEMS), where friction and wear have become critical issues. Therefore, understanding and optimizing the tribological characteristics of glass surfaces is essential for improving the reliability and durability of these systems. The goal of this study was to form a uniform micro-nano surface structure through the surface treatment of the glass substrate, thereby improving the wear resistance. To achieve this, the chemical/physical surface treatment process conditions were optimized to form regular asperity structures ranging from tens of nanometers to several micrometers in size on a glass substrate, and the effects of surface shape and roughness on friction and wear resistance were evaluated. Through this, we aim to derive a surface structure that can exhibit excellent improved wear resistance and secure basic data for future applications in machine components. The results obtained in this study are expected to provide surface structure design guidelines to improve the durability and reliability of machine components. In addition, the approach of this study is expected to be applicable to various mechanical elements in which friction and wear are problematic.

2. Materials and Experiments

In this study, a chemical etching method was used to control the surface structure of the glass substrate. Chemical etching has the advantage of being able to control the surface structure relatively easily by adjusting the process conditions, such as the time, temperature, and concentration of the etching solution. A mixture of ammonium hydrogen fluoride (NH_4HF_2) and hydrofluoric acid (HF), which is widely used for glass etching, was used, with distilled water as the etching solution.

Fig. 1 schematically shows chemical etching of the glass substrate. First, an etching solution was prepared by mixing hydrofluoric acid (50 wt%), ammonium hydrogen fluoride, and distilled water in a volume ratio of 2:2:1. The prepared etching solution was stirred at 300 rpm for 2 h to maintain uniform composition. Subsequently, the glass substrate specimens were immersed in the etching solution for chemical etching. All etchings were performed at room temperature, and the etching time was varied from 10 to 50 minutes at 10-minute intervals. The etched specimens were washed

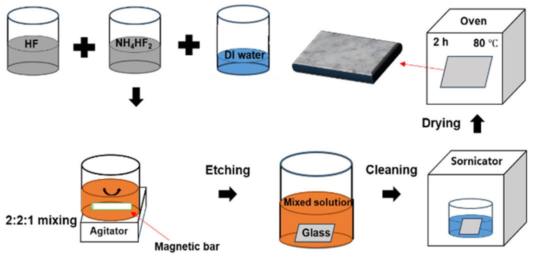

Fig. 1. Schematic diagram of the glass etching process using a mixed solution of hydrofluoric acid (HF), ammonium hydrogen fluoride (NH_4HF_2), and deionized water (DI water).

with ethanol and dried in an oven for 2 h. Each specimen was named 10, 20, 30, 40, and 50 min, according to the etching time. The glass specimens used in this study had dimensions of 76 × 76 × 1 mm.

During the chemical etching process, the composition of the etching solution and the etching time are the main factors that determine the size and shape of the microstructure formed on the surface of the glass substrate. The etching mechanism involves a reaction between the glass surface and the etching solution, resulting in the dissolution of the glass material. A higher concentration of hydrofluoric acid (HF) in the solution led to a faster etching rate and the formation of larger and deeper microstructures, whereas a higher concentration of ammonium hydrogen fluoride (NH_4HF_2) reduced the etching rate, resulting in shallower and more planar microstructures. Longer etching times allow more glass material to be dissolved, forming larger and deeper microstructures, but excessive etching can lead to over-etching and a reduction in the microstructure size. Therefore, by properly adjusting the composition of the etching solution and etching time, a uniform and dense surface structure can be obtained, which is expected to significantly contribute to improving the wear resistance of the diaphragm. This study aimed to systematically analyze the surface characteristics of glass substrate specimens manufactured through this chemical etching process and evaluate the effect of microstructural changes according to the etching conditions on wear resistance.

The surface of each specimen was analyzed before and after the friction test using an optical microscope, and by dropping 10 μL of DI water on the specimen using a pipette, the contact angle was measured using a microscope camera. Surface roughness was evaluated using a surface roughness tester with an evaluation length of 5 mm at a speed of 0.5 mm/s. For the friction test, a stainless steel ball with a diameter of 1 mm was selected as the counter tip, and the test was performed for 2,000 cycles with a stroke of 2 mm at a speed of 4 mm/s under a load of 100 mN.

3. Results and Discussion

Fig. 2 shows optical microscope images of the surface morphologies of the glass specimens obtained at different chemical etching times. The bare specimen exhibited a relatively smooth surface, while the 10 min specimen showed the formation of the most uniform and smallest protrusions on the order of tens of micrometers. This is because hydrofluoric acid selectively corrodes the glass surface in the initial etching stage, forming a fine asperity structure [15]. However, as the etching time increased, size differences occurred as the protrusions were further corroded, as observed in the

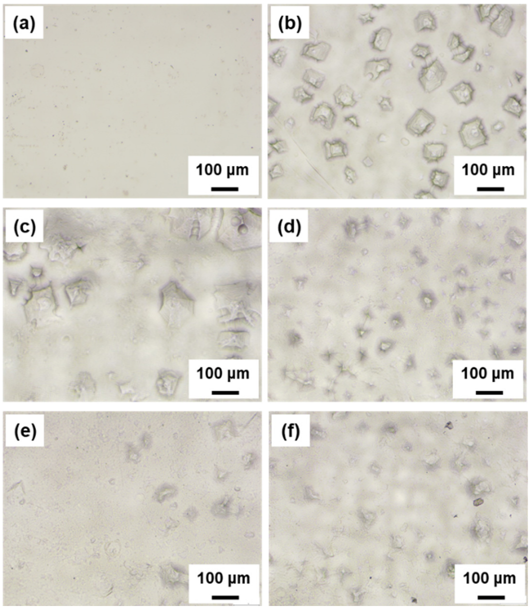

Fig. 2. Optical microscope images of the glass surfaces after chemical etching for different durations: (a) Bare, (b) 10 min, (c) 20 min, (d) 30 min, (e) 40 min, and (f) 50 min.

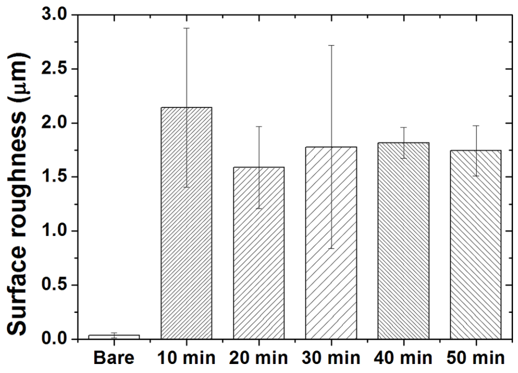

Fig. 3. Surface roughness of glass specimens as a function of chemical etching time.

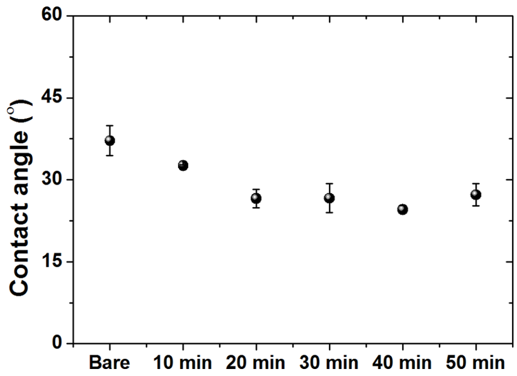

Fig. 4. Water contact angles of glass specimens with respect to chemical etching time.

20 min and 30 min specimens. This is because the top of the asperity structure was more exposed to hydrofluoric acid than the valley and quickly corroded. In contrast, in the 40 min and 50 min specimens, most of the protrusions were removed owing to long-term etching and were hardly observed. This can be seen as a result of the flattening of both the top and valley of the asperity structure owing to the excessive corrosion.

Fig. 3 shows the surface roughness of the specimens. The surface roughness of the bare specimen was 0.04 μm, whereas those of the 10-, 20-, 30-, 40-, and 50 min specimens were 2.14, 1.59, 1.78, 1.81, and 1.74 μm, respectively. The surface roughness reached its maximum value at 10 min, decreased at 20 min, increased again up to 40 min, and decreased at 50 min. This trend is due to the repetition of the process, in which the fine asperity structure formed at the beginning of etching was partially removed, and a new asperity structure was formed as the etching time increased. In particular, the specimens between 10 and 30 min exhibited relatively large standard deviations, which can be interpreted as the largest non-uniformity of the surface in this range.

Fig. 4 shows the change in the water contact angle with respect to the chemical etching time of the specimens. The contact angle of the bare specimen was 37° and that of the etched specimens was measured to be 24.58-32.59°. Overall, the difference in the contact angle with etching time was not significant. However, it appears that the hydrophilicity of the glass surface was somewhat improved owing to the fine asperity structure formed by chemical etching. This can be interpreted as the phenomenon of water droplet spreading being promoted owing to the increase in the actual contact area between the water droplet and glass surface caused by the asperity structure.

Fig. 5 shows the change in the friction coefficient of the glass specimens as a function of the number of cycles according to the chemical etching time. Figure 5(a) shows the friction coefficient evolution for the entire test duration, and (b) shows the initial 800 cycles to highlight the low friction coefficients at the beginning of the test. The bare specimen maintained a low friction coefficient of less than 0.2 during the initial several tens of cycles, and then rapidly increased to a high value of approximately 0.8. This can be interpreted as showing a low friction coefficient at the beginning of the test owing to the smooth characteristics of the glass surface; however, the surface was abraded and wear particles were generated owing to repeated sliding contact, rapidly increasing the friction coefficient [16]. In contrast, for specimens with a fine asperity structure formed on the surface through chemical etching, the behavior of the friction coefficient changed significantly. In particular, the specimen etched for 20 min exhibited the lowest and most stable friction coefficient, which was attributed to the uniform and dense fine asperity structure alleviating the stress concentration at the contact interface and exerting a lubricating effect. In contrast, the 10 min specimen showed a low friction coefficient initially but gradually increased. This seems to be because local stress

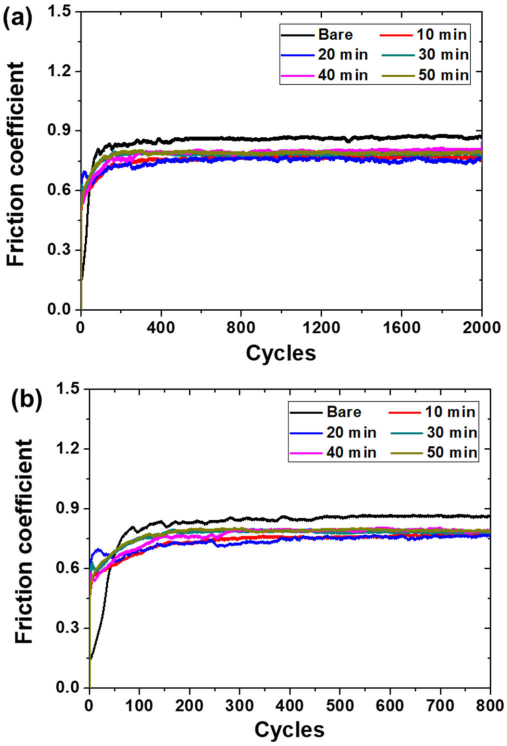

Fig. 5. Friction coefficient evolution of glass specimens with different chemical etching times: (a) entire test duration and (b) initial 800 cycles.

concentration occurs owing to the relatively non-uniform surface shape, and as a result, the fine asperity structure is gradually destroyed, thereby increasing the friction coefficient. Specimens etched for 30 min or longer generally exhibited a high friction coefficient. This can be interpreted as a decrease in surface hardness owing to long-term etching, and wear is accelerated owing to the uneven distribution of contact pressure caused by the nonuniform asperity structure. In particular, the 40 min specimen, it showed the highest friction coefficient, which is attributed to the intensified stress concentration owing to the relatively non-uniform size of the asperity structure.

Fig. 6 shows a graph comparing the average friction coefficients according to the chemical etching time of the specimens. The bare specimen exhibited the highest average friction coefficient of 0.84, while the specimen etched for 20 min exhibited the lowest value of 0.75. As mentioned earlier, this is because the uniform and dense fine asperity structure of the 20 min specimen was the most effective in reducing friction. On the other hand, in the case of the 10 min and 50 min specimens, the average friction coefficients were relatively low at 0.75 and 0.79, respectively, which can be interpreted as the formation of a fine asperity structure and surface flattening due to longterm etching having a positive effect on the frictional characteristics.

Fig. 7 presents optical microscope images of the wear tracks formed on the surface of each specimen after the friction test. In the case of the bare specimen, wear tracks were observed owing to the abrasive wear of the glass surface and adhesion of wear particles, but the wear width was relatively narrow. This phenomenon occurs when the surface is worn because of the contact between the smooth glass surface and counter material. For the specimen etched for 10 min, the wear track exhibited a distinct shape, where the surface structure was cut away because of contact with the rough surface caused by the microasperity structure, and a relatively wide wear width was observed. This is because the rough surface structure causes stress concentration at the contact interface and accelerates the wear of the surface protrusions. In contrast, the morphology of the wear track of the specimen etched for 20 min is similar to that of the bare specimen. This is interpreted as the result of the formation of a uniform and dense microasperity structure, which evenly distributes the stress at the contact interface and effectively reduces friction and

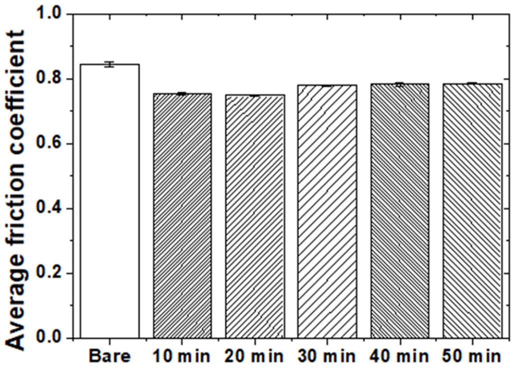

Fig. 6. Average friction coefficient of glass specimens as a function of chemical etching time.

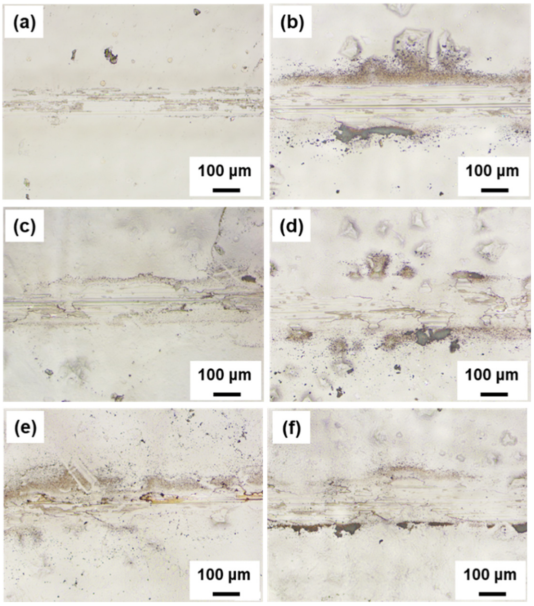

Fig. 7. Optical microscope images of wear tracks formed on glass surfaces after reciprocating sliding tests: (a) Bare, (b) 10 min, (c) 20 min, (d) 30 min, (e) 40 min, and (f) 50 min.

wear. For the specimens etched for 30 min or longer, the wear behavior appeared irregular owing to the nonuniform asperity structure. In particular, in the case of the 40-minute specimen, numerous wear particles were observed within the wear track owing to the destruction of the asperity structure, and the wear width also appeared relatively wide.

Summarizing the above results, it can be concluded that the chemical etching time of the glass surface has a significant influence on the shape and size of the fine asperity structure, which is closely related to the sliding friction and wear characteristics. In particular, among the etched specimens, the specimen etched for 20 min showed the best wear resistance because a uniform and dense fine asperity structure was formed, making the stress distribution at the contact interface uniform and effectively reducing the friction and wear. On the other hand, specimens etched for 10 min or less or 30 min or more tended to show accelerated wear owing to the local stress concentration caused by nonuniform asperity structures.

Table 1 lists the surface roughness values of the wear tracks formed on the glass specimens after the friction tests. The bare specimen exhibited the lowest wear track roughness of 0.01 μm, while the specimens etched for 30, 40, and 50 min showed the highest values, ranging from 0.41 to 0.42 μm. The 20 min specimen displayed a moderate wear track roughness of 0.19 μm, which is consistent with its enhanced wear resistance owing to the uniform and dense micro-asperity structure. These additional surface roughness measurements of the wear tracks further support the conclusions drawn from the optical microscopy observations, confirming the superior tribological performance of the 20 min etched specimen.

Table 1. Surface roughness values of the wear tracks formed on glass specimens with different chemical etching times.

Roughness (μm)	
Bare	0.01
10 min	0.12
20 min	0.19
30 min	0.42
40 min	0.41
50 min	0.42

Therefore, the results of this study suggest that forming a fine asperity structure with an appropriate size and shape is very important for improving the friction and wear resistance characteristics of glass surfaces. To this end, it is necessary to implement a uniform and dense fine asperity structure through optimization of the chemical etching process.

4. Conclusion

This study comprehensively investigated the relationship between surface roughness and tribological behavior of chemically etched glass surfaces under reciprocating sliding conditions. The etching time played a crucial role in determining the size, density, and shape distribution of the micro-asperity structures, which in turn significantly affected the friction and wear characteristics. An optimal etching time of 20 min produced a surface texture with uniform and densely distributed micro-asperities, resulting in the lowest friction coefficient and wear. This enhanced tribological performance was attributed to the effective distribution of contact stress and improved lubrication conditions at the interface.

Conversely, specimens etched for shorter or longer durations exhibited higher friction coefficients and accelerated wear owing to the stress concentration caused by non-uniform asperity structures. Microscopic observations of the wear tracks revealed that the dominant wear mechanisms, such as abrasive wear, adhesive wear, and surface fatigue, varied depending on the surface roughness. The findings of this study provide valuable insights into the optimization of the chemical etching process parameters for achieving the desired surface morphologies and enhanced tribological performance. Furthermore, the results suggest that chemical etching can be effectively employed as a surface-modification technique for various materials in tribological applications.

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