Improving the anti-seizure ability of SiC seal in water with RIE texturing

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The demand for higher pressure and higher speed of sealing systems is currently increasing due to stricter standards for permissible emissions. Silicon carbide (SiC) is thought to be a promising material for sealing systems operating in water, since the combination of SiC and water is both environmentally friendly and energy saving. The purpose of this study is to improve the anti-seizure ability of SiC seals working in water by means of surface texturing. The texture pattern of micro-pits evenly distributed in a square array is formed on one of the contact surfaces by reactive ion etching (RIE). Experiments which simulate the working condition of mechanical seals were carried out to evaluate the effect of micro-pits on the critical seizure load. It is found that micro-pit texturing is an effective way to stabilize friction, to reduce the friction coefficient, and to expand the low-friction range ($\mu < 0.05$) of SiC seals working in water.

KEY WORDS: texture, micro-pit, SiC, water, seizure

1. Introduction

Silicon carbide (SiC) is regarded as a promising material for mechanical seals operating in water due to its excellent tribological properties, such as anti-wear, anti-seizure, anti-corrosion, and in particular, very low friction while it slides against itself in water [1–4]. The main reason for the low friction is that a kind of tribochemical reaction occurs on the contact surfaces. Consequently, hydrodynamic lubrication is easily established with the smooth surfaces polished by tribochemical wear [5]; a film of silicic acid is proven to be formed on the contact surface by the dissolution of the reaction product SiO_2 in water, and it is supposedly effective in decreasing stress concentration and friction while contact of the materials occurs [6,7].

However, the problem of seizure still remains in some particular situations of severe friction contacts. Furthermore, the demand for higher pressure and higher speed of sealing systems, under stricter standards on permissible emissions, has recently sharply increased [8]. Therefore, improving anti-seizure ability is still important for the reliability of mechanical seals made of ceramics.

For the hydrodynamic pressure generated between parallel sliding surfaces, several contributing factors were summarized in refs. [9,10]. Surface roughness is recognized as an important role and this discovery has brought about the method of surface texturing for improving the anti-seizure ability of mechanical seals and sliding bearings. Along with the developments of new techniques, both the design and processing methods of surface texturing have recently been advanced. Two kinds of lubrication effect of surface texturing have been theoretically discussed in past research.

One is the hydrodynamic effect, as shown in figure 1 [11–13]. An asperity on the surface projects down into the fluid flow so as to create a step bearing. As the flow approaches the asperity, the pressure increases; meanwhile on the symmetrical side, the pressure decreases but not to the ideal antisymmetrical value of the opposite side, due to the occurrence of cavitation. As a result, additional load-carrying capacity is generated.

Another effect is known as "secondary lubrication effect" [14], which acts in the regime of mixed lubrication. As shown in figure 2, the liquid trapped in the low region of the texture can be considered as a secondary source of lubricant, which is drawn up by the relative movement to permeate the surface and to reduce the friction and retard seizing.

Various manufacturing techniques are utilized to produce textures on the contact surfaces. Photoetching is a traditional method, which is used on copper surfaces. Laser treatment is generally efficient and convenient for metals. Erosion is an effective technique for hard materials. Reactive ion etching (RIE) is a widely used method for the processing of MEMS and IC. With the aid of ion sputtering, the etching of SiC is possible and the pattern can be accurately controlled.

Research on the lubrication of textured surfaces has been carried out for a long time. So far, many types of surface texture, including micro-islands [11,12], spiral grooves [15] and micro-pits [16,18,19], have been studied. However, analytical solutions that yield true and exact results are still challenging when dealing with

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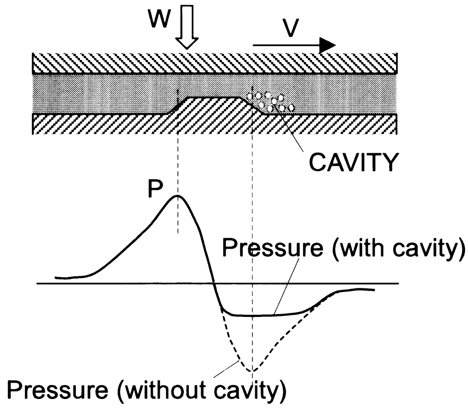


Figure 1. The hydrodynamic pressure generated by an idealized micro-asperity.

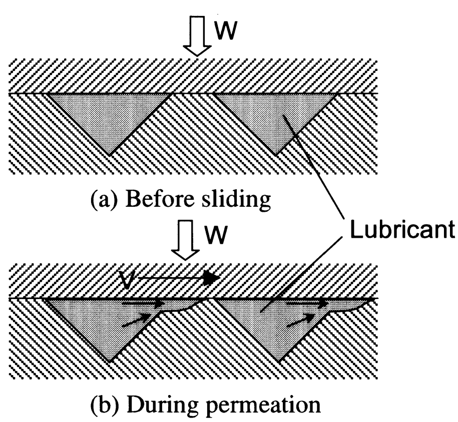


Figure 2. Secondary lubrication mechanism induced by micro-pits.

textured lubrication, although they are recognized as useful methods to explain some phenomena and to show some trends of the influence of surface texture. Especially in the case of Si-based ceramics sliding in water, so little is known about the properties of the tribochemical products that it is very difficult to simulate the lubrication conditions, even without surface texture. Comparatively, experimentation is a realizable method which can provide insights into the lubrication effect of a particular texture pattern.

Representative experiments on the effect of micro-pits (pores) include laser-textured pores on a metal seal in oil by Halperin [16] and micro-blast-formed micro-pits on SiC surface in water by Tejima *et al.* [18]. In ref. [19], the authors report the effect of RIE-textured micro-pits on the performance of a water-lubricated SiC thrust bearing, where two grooves are also formed on the contact surface to supply sufficient water to the friction surfaces.

The purpose of this research is to study the effect of micro-pits on the anti-seizure ability of a SiC seal, which does not contain grooves on its contact surfaces for supplying water.

2. Experiments

2.1. Specimens

Although SiC is often used against the face of a carbon-graphite composite in a sealing system, in order to investigate the texture effect in the case when tribochemical reaction occurs, sliding friction experiments were performed between the flat surfaces of a SiC cylinder [figure 3(a)] and a SiC disk [figure 3(b)]. Both of those were made of the SiC, sintered without pressurization. Their physical properties are shown in table 1. The contact surfaces of the cylinder and disk were ground to a roughness of around 0.2 μm RMS.

Then the contact surface of the disk was textured by RIE to form a pattern of micro-pits arranged in a square array, as shown in figure 4. Since the material is etched mainly in the direction of ion movement, the pit is formed in the shape of a well, where its cylindrical surface is near normal to the contact surface. The diameter and arrangement of the pits are determined by the metal mask formed on the surface. The depth of pits is controlled by the etching time.

All the cylinders were the same, with a hole in the center, which could be filled with purified water while testing. The purpose of this design is to simulate the contact condition of a mechanical seal.

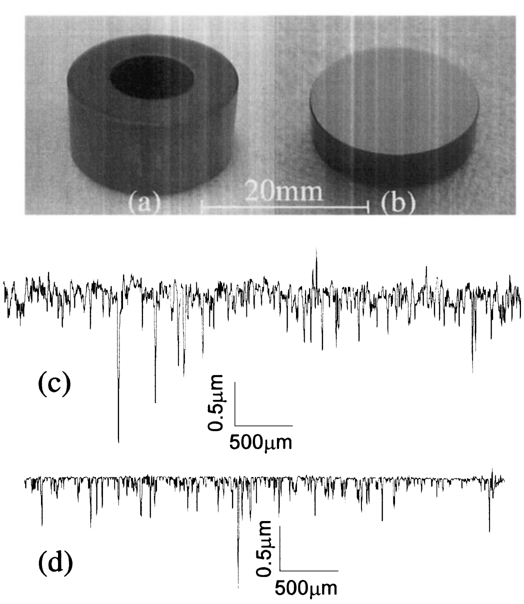


Figure 3. The cylinder (a) and disk (b) specimens and examples of surface roughness before (c) and after (d) running-in.

Table 1
Physical properties of the SiC used in this experiment.

Density	3100 kg/m^3
Bending strength	470 MPa
Vickers hardness	2800
Coefficient of thermal expansion	4.02×10^{-6} K
Thermal conductivity	125.6 $W/m \cdot K$

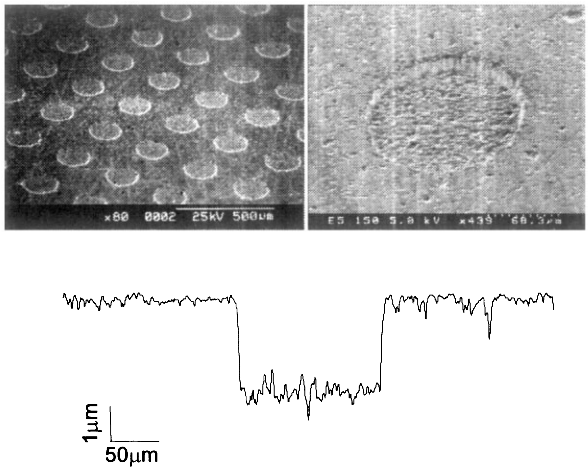


Figure 4. SEM photographs and cross-section profile of the micro-pits.

Table 2 shows the disks used in this study. They have pits with diameters ranging from 50 to 650 μm, depths from 2.0 to 13.3 μm, and pit-area ratios from 0 to 22.5%.

2.2. Apparatus

Figure 5 shows the apparatus used in this experiment. The cylinder is mated to the disk and driven by a motor with an adjustable rotational speed. The disk is supported by a half-spherical pivot, so that its friction surface is automatically aligned to match that of the cylinder. Load is applied by a hydraulic system from the bottom of the disk. Purified water is filled into the hole of the cylinder by a volume-controllable pump. The temperature of the water supplied to the friction surfaces was controlled at around 20 °C. Load and friction torque are detected by the load cells. An air bearing is used to support the disk so that very small friction torque (< 0.001 Nm) can be accurately detected.

An auto-stop system is used to stop the load-applying system and the driving motor to avoid damaging the apparatus and specimens when a rapid increase of friction torque occurs.

Table 2
The specimens used in this experiment.

	Pit diameter d (μm)	Pit depth h (μm)	Pit-area ratio r (%)
Cylinder	0	0	0
	0	0	0
	50	3.2	5
	150	2.0–13.3	5
	150	3–4	2.8–22.5
Disk	250	3–4	5
	250	3–4	5
	350	3–4	5
	500	3–4	5
	650	3–4	5

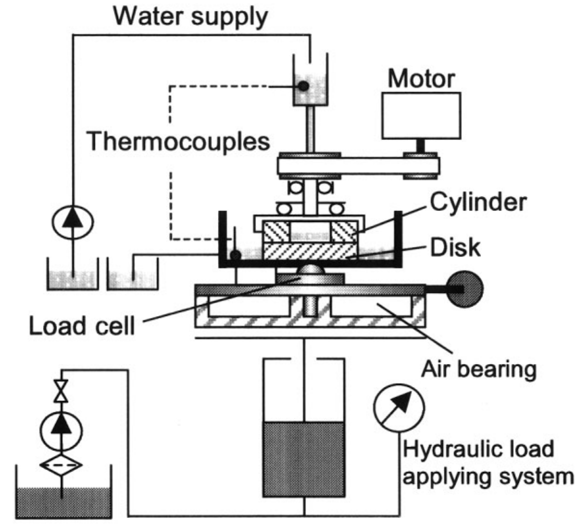


Figure 5. Schematic diagram of apparatus.

2.3. Test procedure

Previous work [4] has shown that surface roughness plays an important role on the friction of SiC since the thickness of the water film is estimated to be very thin. Thus, all the specimens were run-in with the same procedure before the test. The typical profiles of the surface before and after running-in are shown in figure 3.

Figure 6 shows the test method and the definition of the critical load, Wc. The load is increased with an increment of about 50 N at a time until the rapid rise of friction torque happens. Every load is maintained for 5 min since the experimental results show that the increase in friction sometimes follows an increase of load. This phenomenon can be found in this figure, where the friction torque rises 3 min after the load of 300 N is applied. It can be explained as the result of the existence of tribochemical reaction film, which delays the direct contact of materials by consuming itself.

The load at which friction torque suddenly increases can be considered as the beginning of seizure, because it increases so rapidly that the surface would be damaged if the auto-stop system were not available. Therefore, this load is defined as the critical load Wc of seizure for a SiC seal and is used to evaluate the effect of micro-pits on the anti-seizure ability of a SiC seal working in water.

3. Results and discussion

Figures 7 and 8 show the friction properties of the untextured and textured specimens obtained at a rotational speed of 400 and 1200 rpm, respectively. For the untextured specimens, both friction torque and load swung with a big amplitude at 400 and 1200 rpm. Comparatively, the friction torque and load of the textured specimens were relatively stable. This is possibly due to the friction properties of the tribochemical

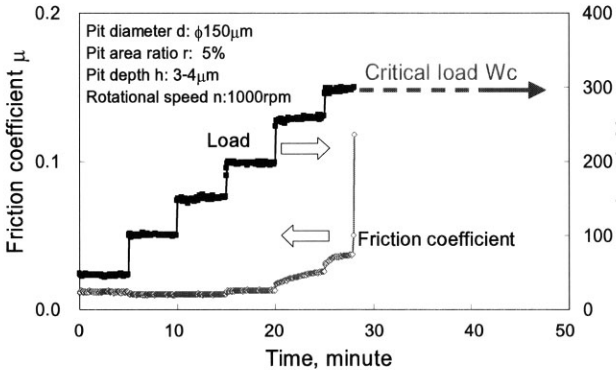
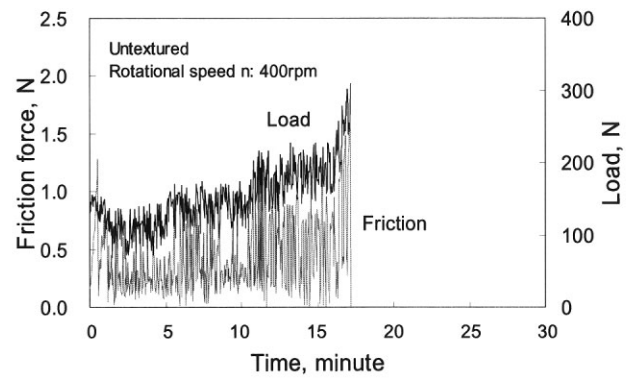
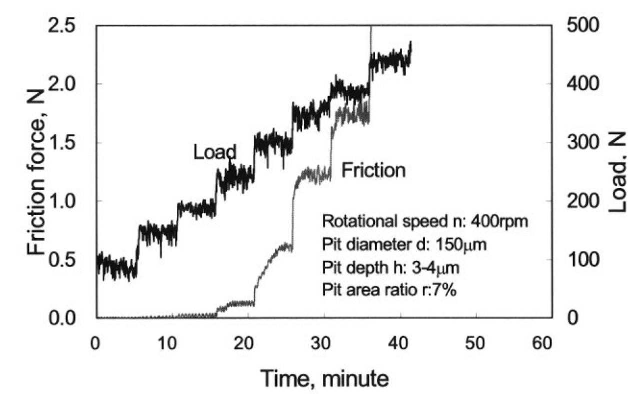


Figure 6. The definition of the critical load, Wc, for the transition from low friction to seizure.

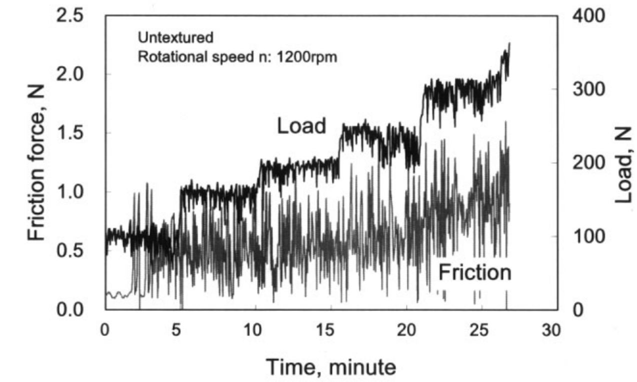


(a) Untextured

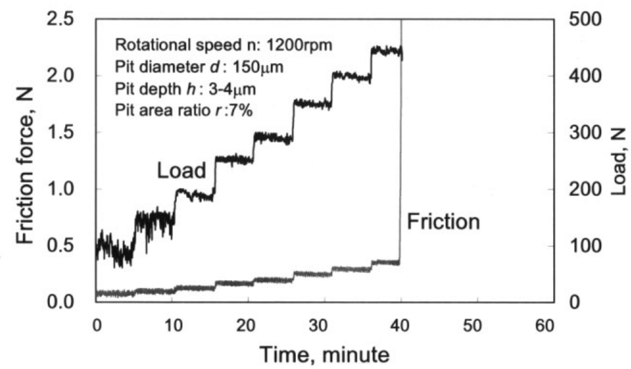


(b) Textured

Figure 7. The friction properties of untextured and textured specimens at a rotational speed of 400 rpm.



(a) Untextured



(b) Textured

Figure 8. The friction properties of untextured and textured specimens at a rotational speed of 120 rpm.

reaction film formed on the contact surfaces. Since it is known as a gel-like film by the dissolution of SiO_2 in water, the friction properties of this film would change if water were not stably supplied. Compared to untextured specimens, the surface of textured specimens should have more areas which could be well lubricated by the water stored in micro-pits. Thus, the friction of textured specimens is relatively stable. These figures also show that higher rotational speed results in a stable friction. It is probably due to the speed dependence of the friction of the gel-like film. The swings of load happen particularly in the case of untextured specimen, which has unstable friction. This could be a result of a mutual influence, where unstable friction makes the system vibrate, generating load fluctuation, and a fluctuation of load causes unstable friction.

Figures 9 and 10 show the Stribeck curves of untextured and textured specimens. The friction coefficient of the untextured specimen is $0.03 \sim 0.05$ on average, fluctuating with a large amplitude, and suddenly increasing along with the decrease of the duty parameter, G. The greatest friction coefficient, at which the experiment was stopped, is not displayed in this figure because it happened instantaneously and thus was difficult to record. Comparatively, the Stribeck curves of the textured specimens present both the lubrication regime and the transition clearly. The friction coefficient in the range before the rapid increase is less than 0.05 and exhibits a weak relationship with the duty parameter, G. This fact suggests that it is in the regime of hydrodynamic lubrication. Thus, the increase of friction coefficient from this regime means that solid contact occurs, and along with the continuous decrease of the duty parameter G, seizure would occur.

The G value of the untextured specimen at the transition of lubrication regime is higher than 10^{-8}, while

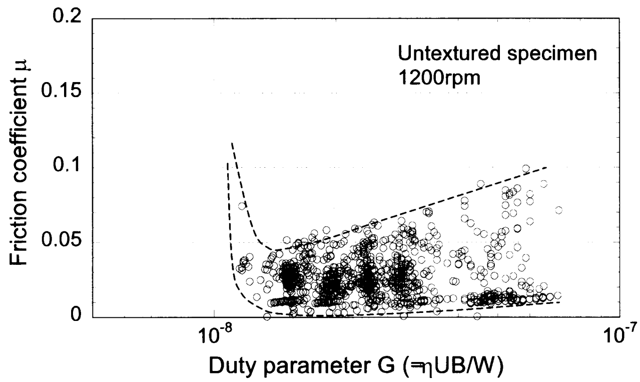


Figure 9. The Stribeck curve of an untextured specimen.

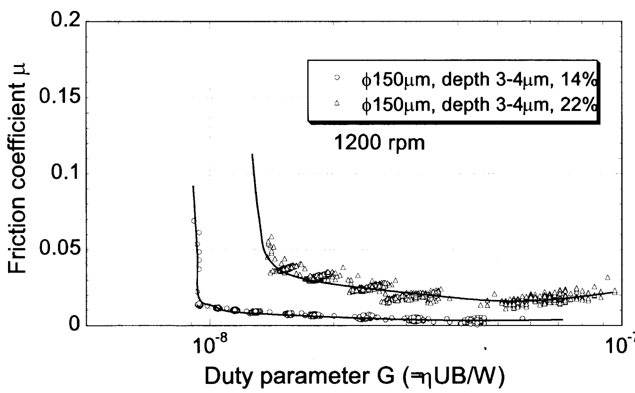


Figure 10. The Stribeck curves of the textured specimens.

that of textured specimen ($\phi 150\,\mu$m, depth 3–4 μm, 14%) is lower than 10^{-8}. This means that the low-friction range (hydrodynamic lubrication regime) could be expanded by surface texturing. Although another texture ($\phi 150\,\mu$m, depth 3–4 μm, 22%) does not show any improvement in the low-friction range, the friction coefficient of textured specimens in the hydrodynamic regime is low and stable, no matter what pattern it has.

Figure 11 shows the experimental result obtained from the textured specimens with a pit-area ratio of 5%, a depth of 3–4 μm and a pit diameter varying from 50 to 650 μm. Although the specimens with pit diameter of 50 and 650 μm show greater increase of the critical load than the others, in comparison with the fluctuations in the experimental data, it does not present a clear trend of the effect of the pit diameter. There is a need for more experimental results.

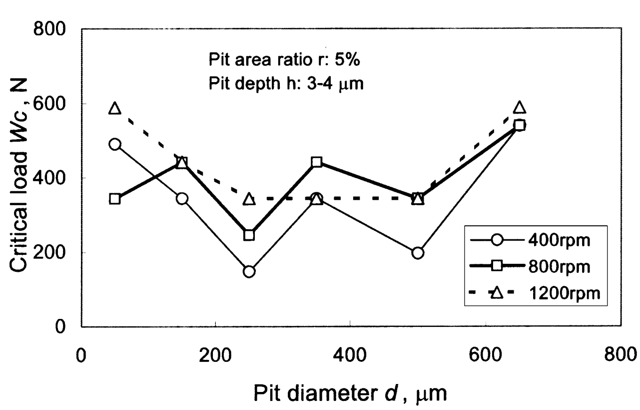


Figure 11. The effect of the pit diameter d on the critical load, Wc.

Figure 12 shows the effect of the pit-area ratio on the critical load, Wc, at the rotational speeds 400, 800 and 1200 rpm. It is clear that the critical load has an obvious increase in the pit-area-ratio range from 5 to 15%. The average increment in this range is over 2 times that of untextured specimen. It also shows that the increments are greatest at the rotational speed of 800 rpm in most cases.

Figure 13 shows the effect of the pit depth on the critical load, Wc, at the rotational speed of 1000 rpm. In the depth range from 2 to 13.3 μm, it shows that the deeper the depth is, the greater the increase in critical load.

The effect of pit depth obtained in this experiment is different from the results reported in refs. [16] and [19], which suggest that the preferable depth is 2–7 μm (diameter 90 μm) for a steel seal working in oil, and an optimum depth-diameter ratio is 0.015 for a SiC bearing lubricated by water. Both of those denote that deep pits are not beneficial for load-carrying capacity. The reason for this difference may be attributed to the fact that

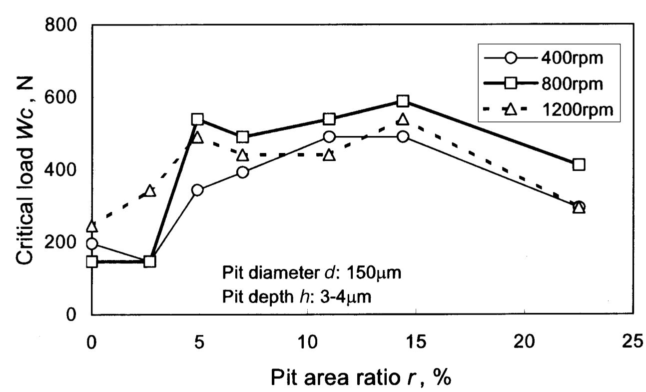


Figure 12. The effect of the pit-area ratio r on the critical load, Wc.

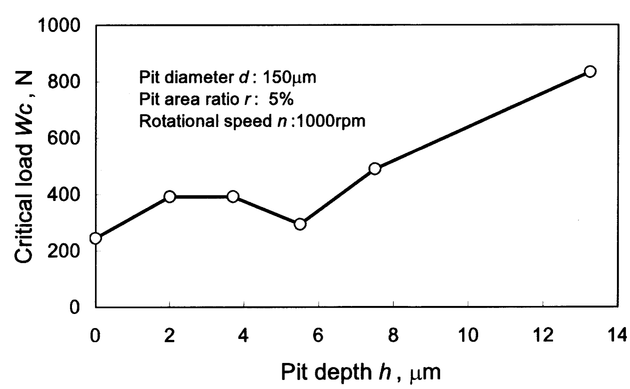


Figure 13. The effect of the pit depth h on the critical load, Wc.

water is not only a lubricant but also a necessary material for tribochemical reaction in this case. In addition, the effect of tribochemical reaction may be more important than the effect of hydrodynamic lubrication for the intimate contact of the SiC seal. Therefore, the deep pits can hold more water, which would be supplied to the contacting surfaces during sliding. This is helpful to maintain the existence of the tribochemical reaction film, which results in the improvement of the anti-seizure ability.

4. Conclusions

In order to increase the anti-seizure ability of a SiC seal working in water, micro-pits were textured on one of the contact surfaces by reactive ion etching (RIE). The critical load for seizure was used as the index to evaluate the anti-seizure ability of untextured and textured specimens. The effect of pits on the critical load was studied experimentally. The following conclusions have been drawn as the most significant:

1. Micro-pit texturing is an effective way to stabilize the friction, to reduce the friction coefficient, and to expand the low friction range ($\mu < 0.05$) of a SiC seal working in water.
2. For specimens textured with micro-pits of diameter 150 μm and depth of 3–4 μm, the critical load can be improved at least 2 times over that of untextured specimen in the range of the pit-area ratio from 5 to 15%.
3. The maximum increment of the critical load in this experiment was more than 3 times that of the untextured system and was obtained with a specimen with a pit diameter of 150 μm, a pit depth of 13.3 μm and a pit-area ratio of 5%.

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