《論文》 Journal of the KSLE Vol. 3, No. 2, 1987
Printed in the Republic of Korea

The Friction and Wear of Sic Ceramic at High Temperature with Solid Lubricant.

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(Received October 13. 1987)

고체윤활제를 사용한 경우 고온에서 SiC 세라믹의 마찰 마모에 관하여

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(1987. 10. 13 접수)

요 약

윤활제가 없는 경우와 고체 윤활제를 사용하였을 경우 세라믹Sic의 마찰 마모시험을 상온 및 고온에서 실시하였다. 이때 사용한 마모시험기는 Two-disk 형이고, 마찰속도는 132mm/sec 이며, 하중은 2.6N 에서 10.4N 의 범위이다.

실험결과에 의하면 고체윤활제(흑연. Mos_2)는 세라믹의 마찰마모를 300℃ 이하에서 모두 효과적으로 감소시킬 수 있었다. 또 300℃ 이하에서는 Mos_2가 더 효과적인 윤활제 역할을 하고, 300℃ 이상에서는 흑연이 더 효과적이었다. 그 원인은 윤활제 피막형성 및 파괴가 윤활 효과에 지배적인 영향을 미치고 온도상승에 따라 각 윤활제의 특성이 달라지기 때문이었음이 SEM 이나 EDS에 의해 밝혀졌다. 따라서 고온에서 윤활효과를 증대시키기 위해서는 윤활피막의 접착을 강하게 해 줄 수 있는 접착 첨가물이 필요하다. 고온에서 윤활이 안된 경우 마모는 결정경계를 통한 입자들의 파괴가 주로 원인되나 낮은 온도나 윤활상태에서는 결정경계에 무관한 연마 마모가 마모를 지배하고 있었다.

INTRODUCTION

For the tribological application, ceramic materals are a great deal of interest in research recently. In particular, the high wear resistance is a good property to use in situation of severe service [1]. Despite of the unique tribological properties, the friction and wear is still desirable to reduce enough to apply in the industry.

In literature, ceramic/ceramic or ceramic/metal couples were investigated to understand the wear of ceramics [2–7]. But there is no information how to reduce friction which is higher than that of metals [8]and wear. Thus to understand the wear and friction behavior of ceramics with lubricant, it is important to investigate the effect of lubricant at room and elevated temperature.

Also there are various papers on the use of solid lubricant to reduce the wear and friction of metals where liquid lubricant could not be used [9−11]. But the application of solid lubricant to ceramics has not reported yet. Because the chemical and mechanical properties of ceramic are different from metal, it is unclear to what extent the lubrication are effectively affected the friction and wear and which is best to use at various conditions.

This paper reports on the wear and friction of SiC/SiC and SiC/Steel pairs at various temperature with and without lubricants. At high temperature, graphite and molybdenum disulphide(MoS_2) were used as lubricant.

It was found that the wear mode and the solid film formation on the worn area affected the wear and friction of ceramic. Also the were debris formed on the contact area during sliding with water and without lubricant at high temperature accelerated the wear of ceramic. At the elevated temperature, graphite showed better performance than MoS_2 while MoS_2 was better at room temperature.

EXPERIMENTAL APPARATUS, MATERIAL AND TESTPROCEDURE

The wear and friction experiments were performed on a two disk sliding machine which one is stationary and the other is rotating shown in figure 1. These arrangement makes a good line contact between the two disks without edge effect. Also, it is possible to measure the wear scar length and coefficient of friction very accurately.

A hot pressed SiC(carborundum) was studied as well as steel(AISI 1045). The diameter of upper disk was 31.5 mm and of lower disk was 42.06 mm. Surface finish of ceramics were 0.114 μm (Ra) for the upper disk and 0.58 μm (Ra) for the lower disk. They were used as received from factory made. The steel disk which was used was machined to make the same geometry as the lower disk of ceramic of ceramic and the surface finish was 0.39 μm (Ra)

Sliding experiments were conducted in three temperature zone ; room temperature (25℃), 300℃, 500℃. The temperature control was done by adjusting the nozzle distance from the lower disk and controlling to O_2 and C_2H_2 valves. Until the temperature of the contact zone was stabilized as desired, the lower disk was rotated without sliding test and continuously measured with thermocouple. After the temperature was stabilized, the sliding test was conducted with removing the thermocouple to a little back of contact area.

The sliding speed of the whole test was 132 mm/sec (60 rpm) which did not increase the surface temperature by frictional heating.

During the test, the temperature was not varied larger than 10℃ at 300℃ and 15℃ at 500℃. In order to minimize the thermal effect of the strain gage, water cooling device was attached on the machine.

The specimen used were cleaned ultrasonically with acetone prior to use. The lubricants used were graphite (Josep Dixion Crucible Co.), MoS_2(Alpha Corp.), distilled water and mineral oil. Only graphite and MoS_2 were used at elevated temperature. The soild lubricants were dropped near the contact area of the lower disk continuously.

RESULTS AND DISCUSSION

SiC vs itself sliding at room temperature.

For the examination of the lubrication effect, unlubricated sliding test was conducted at room temperature. The load to be applied were 5.2 N and 10.4 N. Figure 2 is the SEM picture of original and worn surfaces. At low load (5.2 N), the surface worn away making smooth and about 0.025 μm (Ra). But at high load, the surfaces were became rough (0.05 μm). It was shown that the upper disk a lot of scratch lines along the sliding direction. There were no sign of intergranular were or fracture mode on the both surface.

With the commercal flake graphite, the ceramic pairs were lubrecated at 10.4N. The worn surface of the upper disk was not covered with graphite nor any solid film (Fig.3). But the lower disk was covered with graphit film. It was uneven and some was flaked off. Where the film was removed away, the worn surface showed that the wear mode was transgranular fracture type. It also showed that the surface was worn away during the removal of solid film which will be verifiad later. The graphite film was formed on the ceramic gradually and it takes abort 70 seconds with some frictional vibration after the graphite was introduced and then stabilized as shown in figure 4. It also show that the film was broken away by stylus tip (load : 0.5g, radius : 12.7 μm). With the upper disk has not graphite film, the friction coefficient and wear rate were between those of unlubricated sliding and lubricated with Mos_2.

When the contact zone was lubricated with MoS_2, the worn surfaces of the both disk were covered with solid film came from lubricant itself (Fig. 3). It showed that the film was pushed along the sliding direction and broken away. The area which film was removed showed the bare ceramic. With solid film on the both worn area, the friction coefficient and wear rate were less than graphite (Fig.5,6). The formation of MoS_2 during sliding did not take long time as graphite as shown in Fig.4. It was formed immediately after lubricant was introduced. Also there was no frictional vibration as graphite. Easy film formation of Mos_2 could be the reseaon of the low friction coefficient and wear than graphite at room temperature.

The mineral oil was used as lubricant in order to examine the difference from the solid lubricants. The both worn surfaces were very smooth. The wear rate and coefficient of friction were higher than that of MoS_2 lubrication, but less than graphite (Fig. 7). It could not protect the wear of ceramic which did not make solid film. When steel was used with mineral, it reduced the wear and friction at the same time (later described).

The distilled water also used as lubricant. After sliding, the upper disk was very smooth (Ra =0.01 μm). The upper surface also had some pits as showm in figure 7. The wear debris was very fine and might be abrasive material to make smooth the worn area. But the formation of pits was not known. The coefficient of friction with water was 0.3 and less than that of unlubricated sliding. But the wear rate (Fig.5) was the highest among the other conditions. It was 20 times more than that of MoS_2 lubrication. This results was also found at the other papers [3,12]. The severe wear might be caused by the abrasion of ceramic by the fine wear debris.

From this results, the wear and friction of ceramic were strongly affected by the wear debris, surface film formation.

SiC vs itself sliding at 300℃

In order to find the effect the lubrication at elevated temperature, the lower disk was heated to 300℃ with gas welding equipment. Because of the high thermal conductivity of ceramic and the hollow shape of the lower disk, it makes stabilize temperature at the lower disk. In these test, the sliding time was set to 15 minutes.

At first, the sliding test was conducted without lunbricant. The worn area was covered evenly with wear debris (Fig.8). The wear debris was adhered to the surface strongly. It was not removed from the worn area by ultrasonically cleaning with acetone. But some of them was removed away with razor blade showing some of worn surface. The debris was just same shape as water lubricated sliding except some of them was attached .o worn areas.

The fine wear debris was mere effectively worn the ceramic surface and the wear rate was 4－5 times larger than that of solid lubricated surface. Also it was larger than by 2 times which was slid at room temperature without lubrication. But the coefficient of friction was little higher than that of room temperature.

When the sliding zone was lubricated with graphite, the coefficient of friction was also increased than that of room temperature and still higher than MoS_2 lubrication. Both of the worn surface were partially covered with graphite and the film removed area showed the intergranular wear mode. But the wear rate was the least and 1/2 of the unlubricated sliding wear. So the effect of graphite lubrication was most prominence when the temperature is high. Also it is better than MoS_2 lubrication which will be discussed next.

Whit MoS_2, the wear was higher than of graphite even the friction coefficient was less (Fig.5, 6). Some of worn surface was covered with lubricant just as the worn area lubricated with graphite. Also the solid film which formed on the surface could not survice for a long time as shown in figure 9. It was formed on the ceramic instantly and worn away within 30 seconds. From this result, the wear was more affected by the solid film then its coefficient of friction. Easy removal of film increased the wear even the friction was low. It was the same result when the lower disk was changed to steel which will be described later. So, if MoS_2 is enough around the sliding area, friction is low. But if lubricant became scarce, the friction could be increased soon.

At 300℃, graphite gave better performance to reduce wear of ceramic even the coefficient of friction was a little higher than MoS_2.

SiC vs itself sliding at 500℃

At the temperature of 500℃, the color of lower disk became red and was broken by thermal shock during test at 10.4 N load. So the applied load was reduced to 2.6 N. Also the sliding time was set to 5 minutes.

The worn area of the unlubricated sliding showed the smeared and fractured film. The wear rate was higher than that of 300℃ as shown in Fig.5. Coefficient of friction was also higher and became to f=0.7(Fig.6). So, without lubricant both the wear and friction at high temperature were much worse than at low temperature.

When the graphite was used as lubricant, the worn surfaces have a lot of intergranular wear scar with some graphite film(Fig.10). The friction coefficient and wear were almost same order as those of unlubricated sliding. During the test, the frictional vibration is much higher than that of low temperature.

MoS_2 was also used as lubricant at the sliding zone. The wear of ceramics was higher than that of graphite. But still the coefficient of friction is lower than that of lubricated with graphite. The worn surface(Fig.10) was almost same as the surface lubricated with graphite. It became more clear that MoS_2 could not protect the wear of ceramics more effectively than graphite at elevated temperature.

At the hige temperature at 500℃, the solid lubrcant did not decrease the wear and friction from unlubricated sliding. In the literature [13], the solid lubricant could reduce the friction and wear up to 800℃ when metals were slid. But ceramic pairs was not shown same results partly because the hardness of ceramics was much higher than metal, so adhesion of solid lubricant film to substrate might be weak.

SiC vs steel sliding at room temperature.

The lower disk was changed to AISI 1045

steel in order to verify the former results and understand the effect of lubricants on the ceramic/steel sliding.

Without lubricant, the were rate and coefficient of friction were high as shown in figure 11 and 12. But all were much less than that of ceramic pairs. The worn surface of ceramic has not any transferred metal from lower steel disk.

The steel surface was covered with oxide is land but some of them was flaked off as shown in figure 13. From SEM and EDAX, it was found that the thick iron oxide layer had higher concetration of silicon than the oxide removed area as shown in figure 14. During the formation the oxide, creamic wear debris could be accumulated in the iron oxide.

Water was introduced to the sliding zone as a lubricant. The wear rate was still higher than unlubricated sliding. The picture of worn surface showed that steel surface has a lot of pits. These was not known as before but it could be made from ceramic wear debris. Graphite was used as lubricant during sliding. The solid film was formed on the both disk. Steel surface was more evenly formed with graphite. The time to formation of solid film was less than ceramic pairs (70 sec.) and it took about 45 sec. From the EDS, the solid film formed with graphite had more silicon conentration than the film removed area.

When mineral oil was used, the wear and friction were much smaller than those of other tests or ceramic pairs slid with oil (Fig. 11, 12). The both worn surfaces were covered with oil oxide film [14], while the ceramic disks have not any film when slid ceramic pairs slid itself.

The iron oil oxide on the steel surface did not contain as the other solid film. As a result, mineral oil was reacted only to the steel surface during sliding and some of them was transferred to ceramic. Also the formation and removal of the solid lubricant film always accompanied with the wear of ceramic while mineral oil oxide did not.

CONCLUSIONS

From the sliding and wear test with SiC ceramic, the following conclusions can be drawn :

1) During the unlubricated sliding, ceramic wear debris was attached on the lower disk surface which made to increase the friction coefficient.
2) The fine wear debris of ceramic which formed during water lubricated sliding or unlubricated sliding was so abrasive that it accelerated the wear of ceramics severely, which could be reduced effectively with solid lubricants.
3) Solid lubricants (MoS_2, graphite) were good enough to reduce the friction and wear of ceramics effecively at 300℃ as well as room temperature. But at the temperature at 500℃, the effectiveness of solid lubricants were not prominence as that of the temperature at 300℃ or less.
4) The time to formation of graphite film on the ceramic surface as well as steel is usually longer than that of MoS_2. Thes may be the reason that the graphite was adhered more firmly to base material than MoS_2, which makes better performance at high temperature.
5) The worn area of SiC showed that at high temperature or high load, intergranular wear mode was prevalent while at low temperature or light load, transgranular wear mode was controlled.

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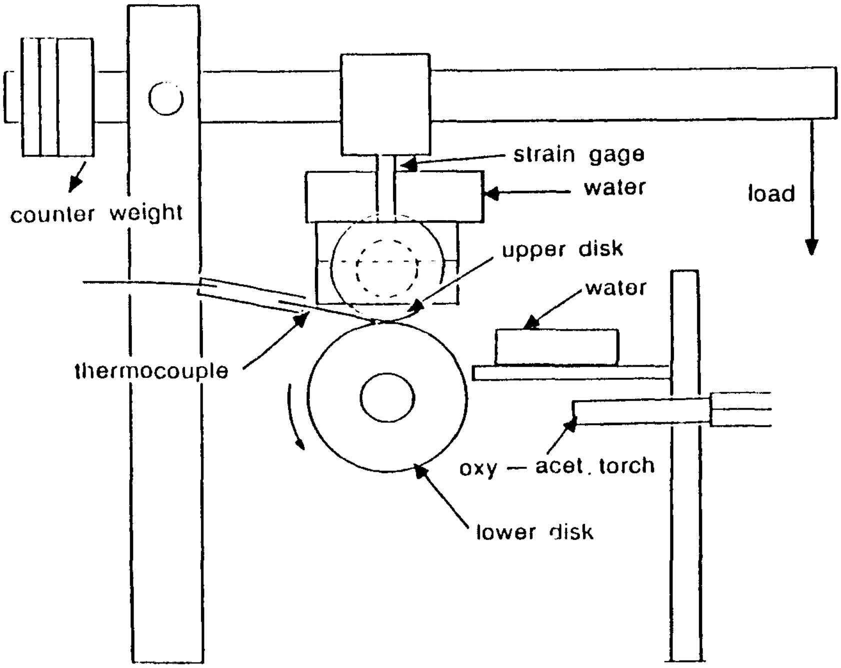


Fig 1. Schematic diagram of the two disk machine

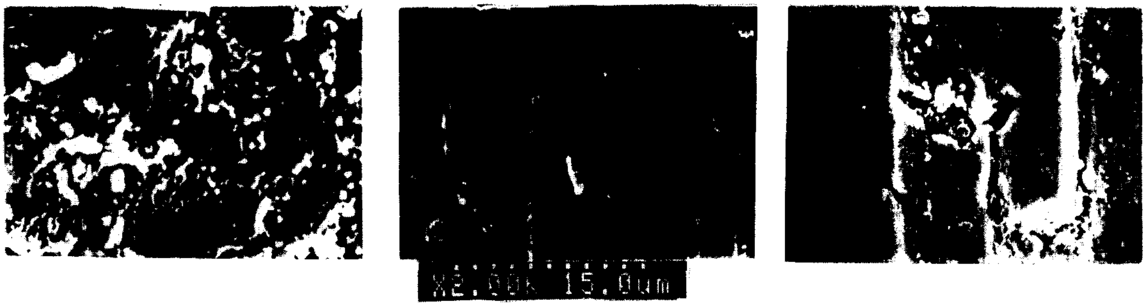
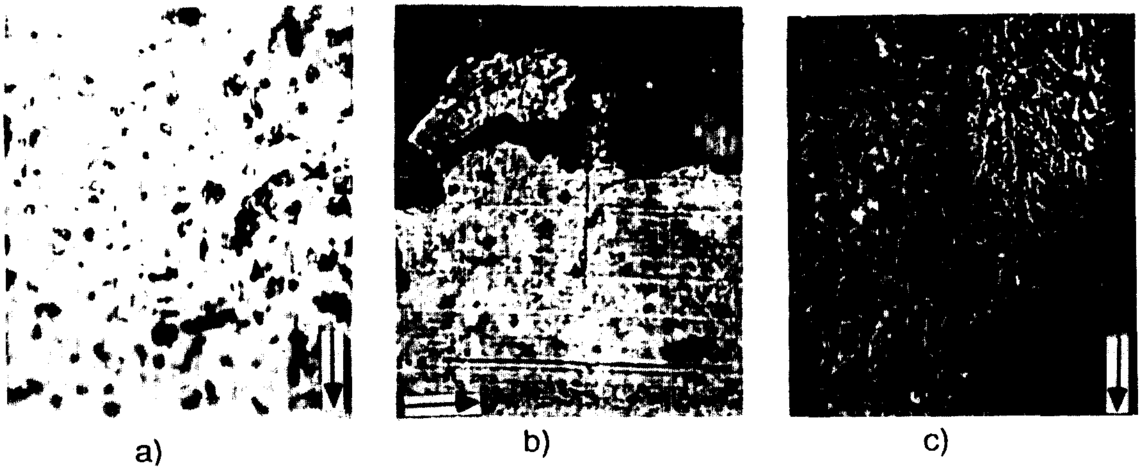


Fig2 . SDM micrograph of the original surface and unlubricated wear scar.
a) original surface, b) surface slid at 5.3N, c) surface slid at 10.6N.



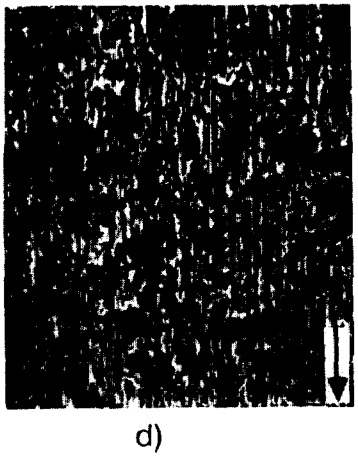


Fig 3. Optical micrograph of the wear scar.
a) upper disk slid with graphite,
b) lower disk slid with graphite,
c) upper disk slid with MoS₂,
d) lower disk slid with MoS₂.
arrow: 20 μm in sliding direction.

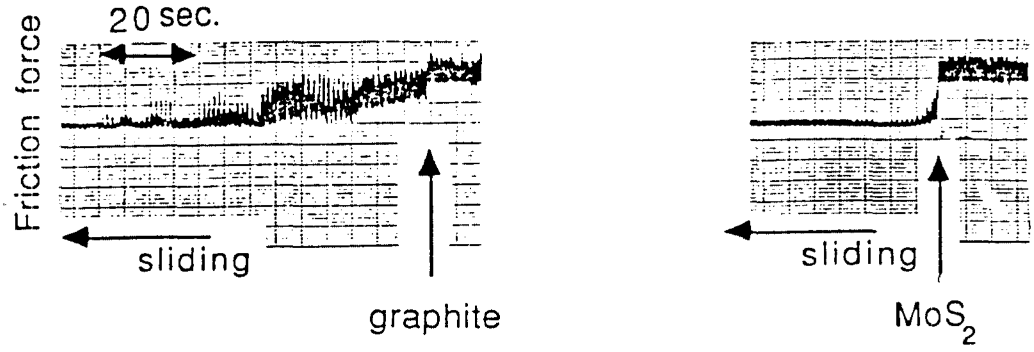


Fig 4. Friction force changes with lubricant after unlubricated sliding.

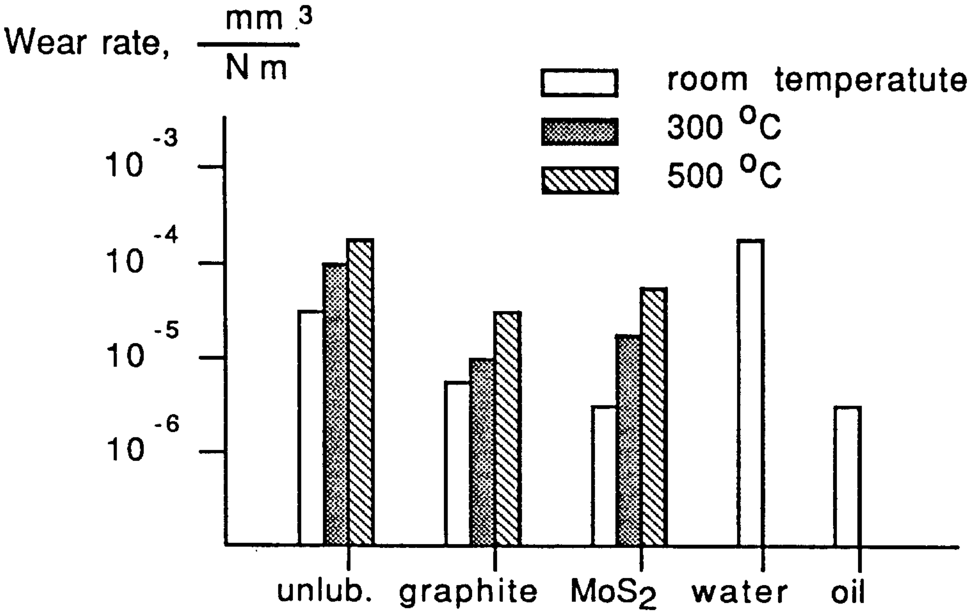


Fig 5. Wear rate of the upper disk at various temperature and lubricant.

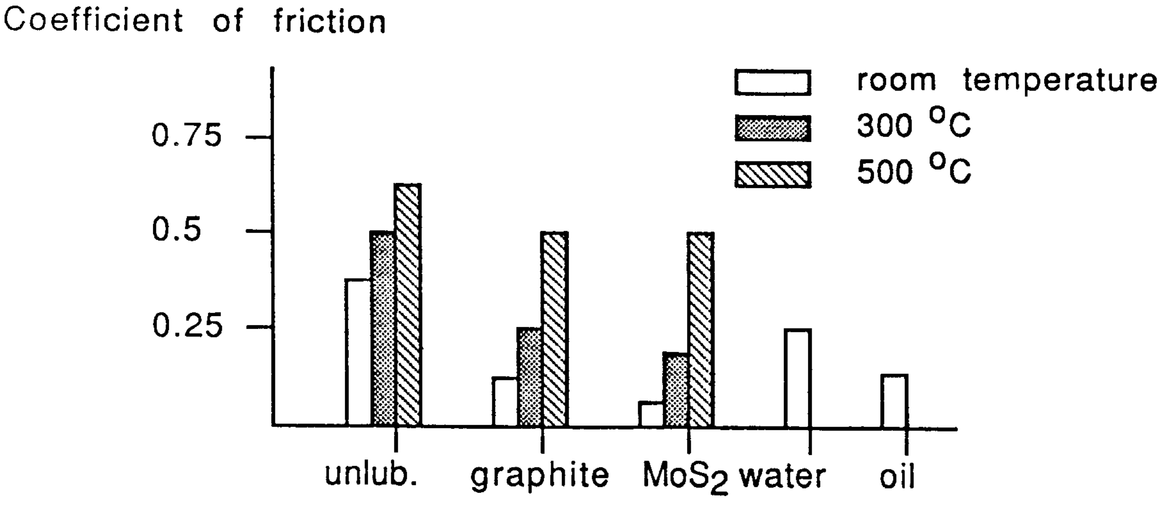
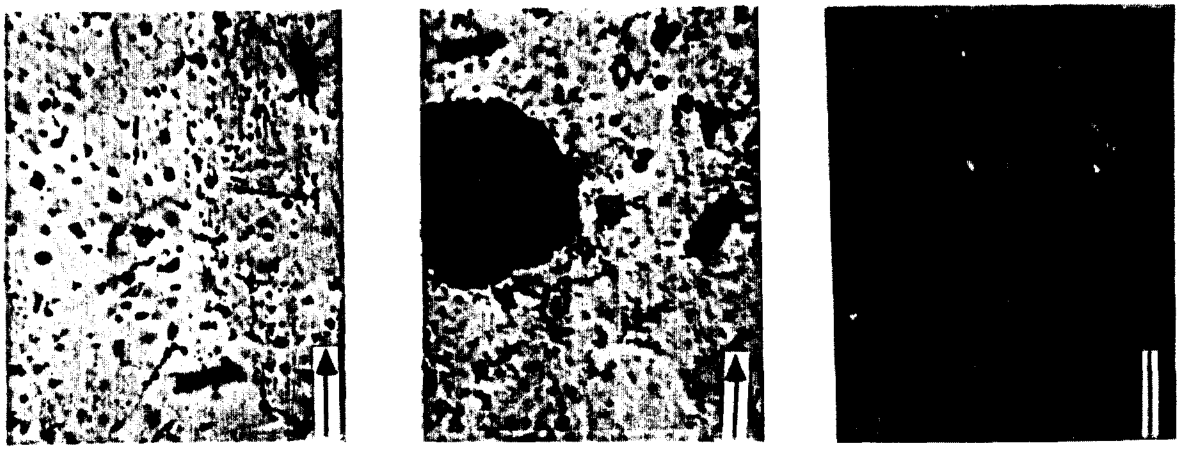


Fig 6. Coefficient of friction of ceramic pairs at verious temperature and lubricant.



a) b) c)

Fig 7. Optical micrograph of the wear scar.
a) upper disk slid with oil, b) upper disk with distilled water,
c) wear debris formed during sliding with water or oil.
arrow: 20μm in sliding direction.

Fig 8. Optical micrograph of the wear scar slid at 300℃.
a) unlubricated sliding surface, b) lubricated sliding with graphite,
c) lubricated sliding with graphite MoS_2.
arrow: 20μm in sliding direction.

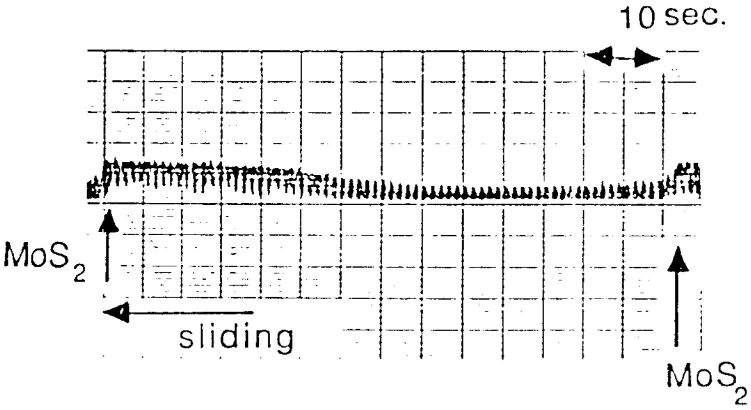


Fig 9. Friction force change after MoS_2 introduced on the contact zone at 500℃.

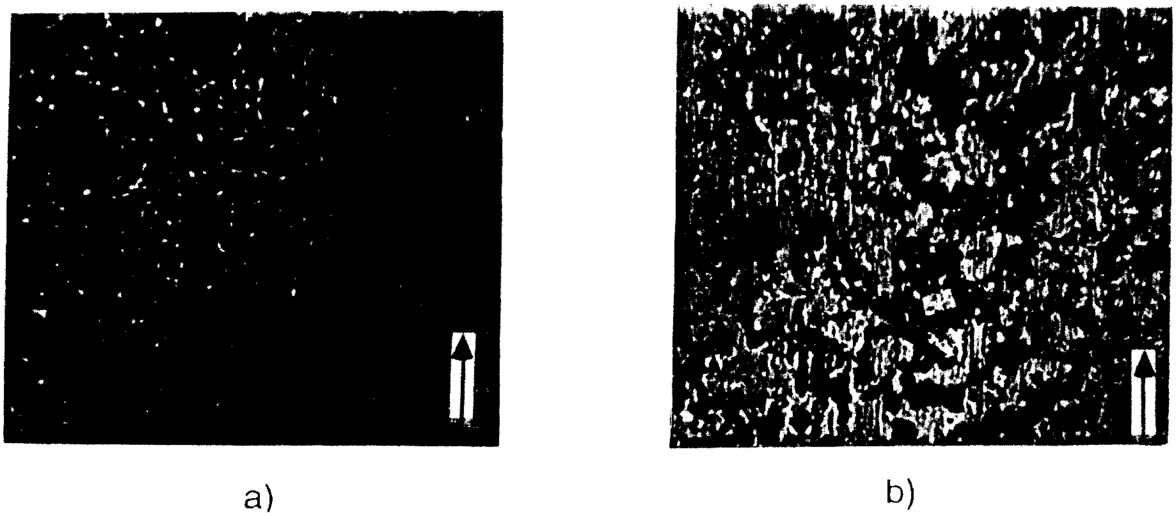


Fig10. Wear track of the upper disk slid at 500℃.

a) lubricated surface sliding with graphite. b) lubricated surface sliding with MoS_2.

arrow : 20μm in sliding direction.

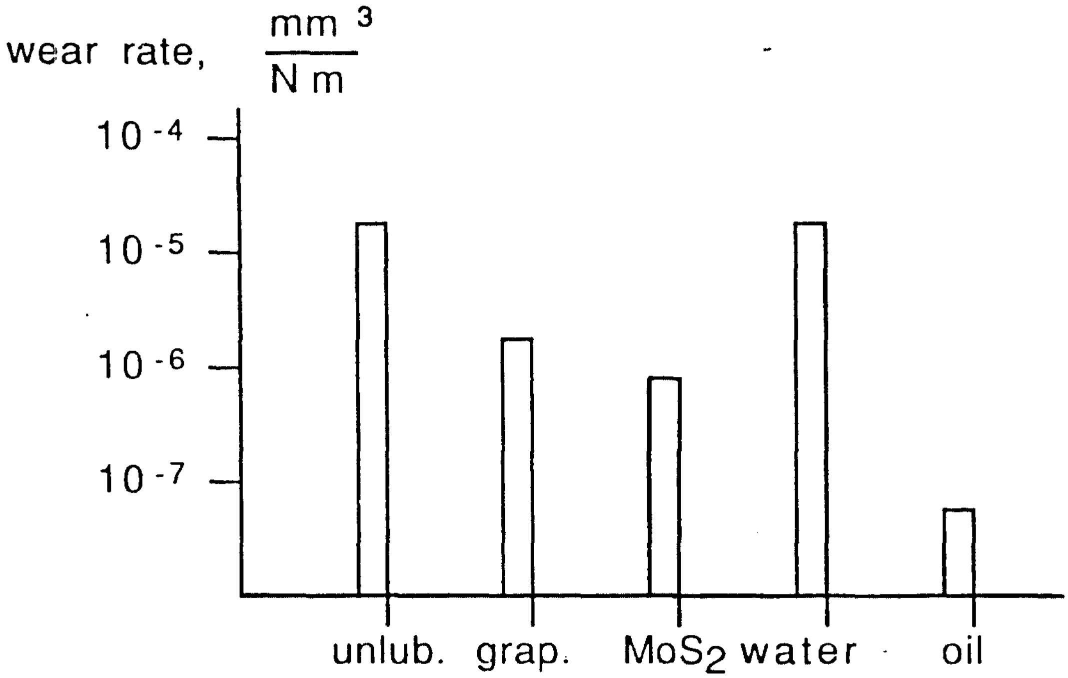


Fig11. Wear rate of SiC sliding against steel at verious lubricant.

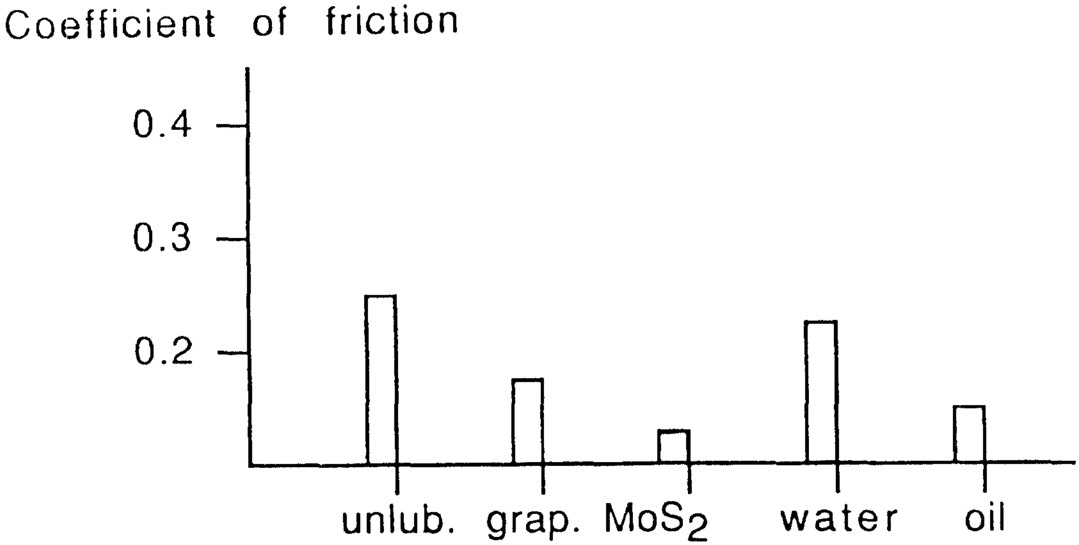


Fig12. Coefficient of friction of SiC sliding at room temperature temperature lubricant.

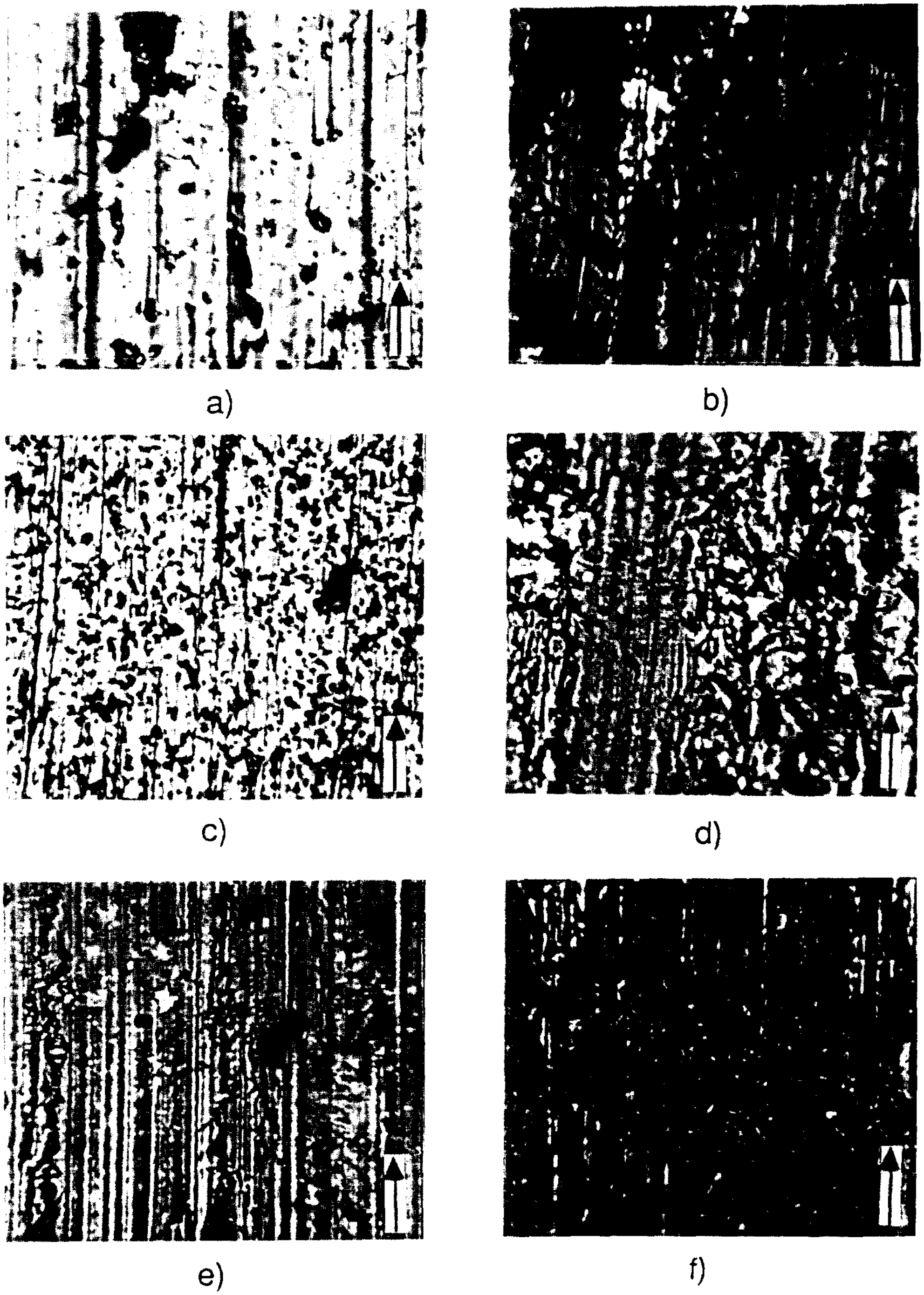


Fig13. Optical micrograph of wear track sliding SiC against steel.
a) upper disk slid without lubricant, b) lower disk slid without lubricant,
c) upper disk lubricated with water, d) upper disk lubricated with graphite,
e) upper disk lubricated with MoS_2, f) upper disk lubricated with mineral oil.
arrow:$20\mu m$ in sliding direction.

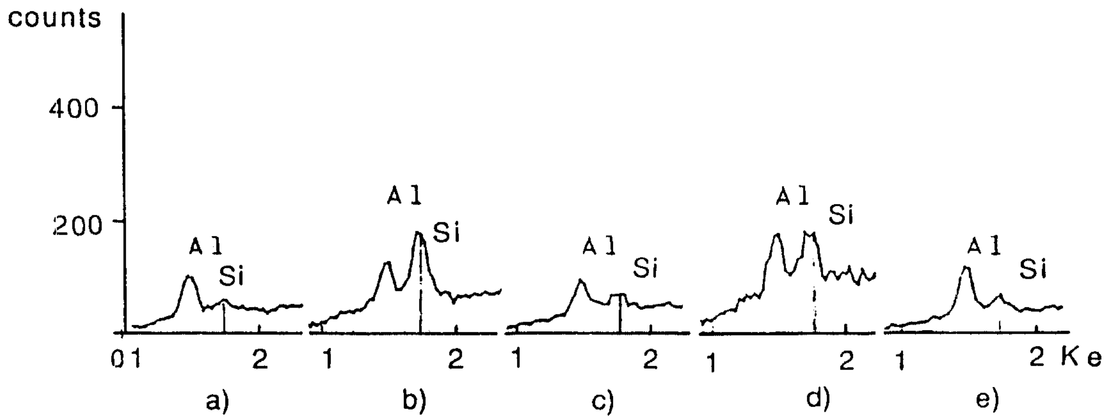


Fig14. EDS spectra of the steel disk sliding against SiC at verious condition.
a) original surface, b) unlubricated sliding surface (thick oxide layer)
c) unlubricated sliding surface (thick oxide removed area)
d) lubricated sliding surface with graphite,
e) lubricated sliding surface with mineral oil.