Tribological Properties of DLC for Die Applications

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Abstract : Friction and wear affect all processes involved in the extraction of materials and their conversion into finished products in the die applications such as drawing, extrusion etc. Originating phenomenon from the contact surface between the tool and workpiece, they are usually a hindrance to materials process operations which usually result in damaging the tools, increasing energy consumption, the contamination of processed material by wear particles and also some problems associated with technologies to control friction and wear. The most well established method to control friction and wear is by the application of lubricant such as fluorocarbon. Besides, a surface technique so-called surface modification can be applied to solve the tribology problems of the die applications for both the economical and ecological reasons.
In this article, we applied DLC(diamond-like carbon) thin film on alumina ceramic for HT test using the PIID(plasma ion immersion deposition), 4 groups of test specimens were tested up to 200ºC which is a little higher than the normal working temperature of die application. Pin-on-disc tribo-tester was used to test the friction and surfaces were characterized by SEM and EDS and else, the morphology changes of DLC coatings were studied. The present work indicated that the DLC had a great potential to reduce the friction and wear in the alumina die application without lubricants.

Key words : DLC, PIID, Tribology, Pin-on-Disc, Scratch Test, Die Application.

1. Introduction

With the rapid development of the surface modification technique, DLC which is served as a solid lubricant have been widely studied for the past two decades for its chemical inertness, high hardness and wear resistance and super low COF(coefficient of friction)[1,2,3,4] compared with other lubricants.

In the tribology applications of die, up to now, more and more dies are applied the ceramic tools such as alumina, silicon carbide, zirconia which all have good tribo-properties, they have been investigated widely in tribology, but a relative high COF over 0.4 is observed, while with DLC coated at the contact surface which eliminates the oil lubricant with much better performance is possible as our work indicated later.

For the sliding components, the high friction and wear are usually caused by adhesion wear due to the strong adhesive bonds that form across the sliding interface after the intervening films contacting the component removed, while for hydrogenated DLC which is an amorphous carbon has hydrogen to passivate the dangling bonds, once passivated, the DLC becomes chemical inertness and has little adhesion to most of the material.

In this article, using the PIID, we tested the tribology performance of DLC coating on alumina under 200 ºC. The friction, wear test, scratch test and adhesion problems were studied in this article.

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접수 : 2011. 10. 10. 채택 : 2012. 04. 13.

2. Experiment

2.1 Pretreatments and PIID

The alumina specimens were first polished to mirror-like with a roughness of 0.05μm Ra, then divided into five groups in which one was un-treated while the rest four were DLC-coated, each group consisted of 4-5 specimens.

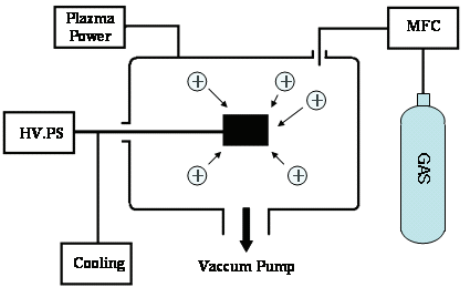

Figure 1. PIID process

We used precision ceramic balls (99.5% Al2O3) as the pin specimen. We selected the Plasma Immersion Ion Deposition (PIID) process [5, 6] as shown in Figure 1.

Table 1. Detail parameters for specimens

Sample #	Interlayer Deposition Method	DLC Coating Precursor	DLC Thickness (μm)
1	Si/PIID	C_2H_2	1.2
2	Si/PIID	CH_4+C_2H_2	1.3
3	Cr/IBAD+ Si_xC/PIID	CH_4	2.0
4	Cr/IBAD	C_2H_2	1.5

In our tests, the typical PIID process started with Ar sputtering to remove surface contaminants, then an interlayer was deposited. Compared with Si interlayer, Cr was deposited using Ion Beam Assisted Deposition (IBAD) technique [6, 7] to study the effect of interlayer type to the adhesion. For sample #3, before the DLC deposition, a second interlayer of SixCwasdepositedaslistedinTable1.

2.2 Pin-On-Disc Test

The tribological performance of the DLC films was tested using pin-on-disc tribo-tester under room temperature, 100 ºC, 200 ºC. The relative humidity was about 50%. At each test temperature, a number of specimens were used. The friction force was measured through the strain gauge on the load cell, and it was recorded continuously by a data acquisition system.

The uncoated 5mm-diameter alumina ceramic ball was used against the DLC coated alumina disc specimen. A normal load of 2.9N was applied, which corresponded to an initial contact stress of 1.3GPa, and the sliding speed was 0.1m/s in all tests, after a total sliding distance of 1000m for the tests conducted at room temperature and 500m for others, the wear track was examined using Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS).

Using Si wafer coupon samples that were coated along with the ceramics, hydrogen content was measured using Hydrogen Forward Scattering (HFS) analysis associated with Rutherford Back Scattering (RBS). A nano-indentation tester was employed to measure the nano-hardness of the DLC. The indentation measurements were repeated ten times at different spots on the same surface, and then the average value was calculated.

3. Result

3.1 Hydrogen Concentration and Nano-hardness

The hydrogen concentration in all DLC films is about 32% using HFS. The nano-hardness (an average of ten tests) is shown in Figure. 2, while #1,4 show hardness over 20Gpa, #2,3 show about 15Gpa mainly due to the precursor used with a lower C-H bonding while using acetylene as the precursor, the nano-hardness of #1,4 is relative higher.

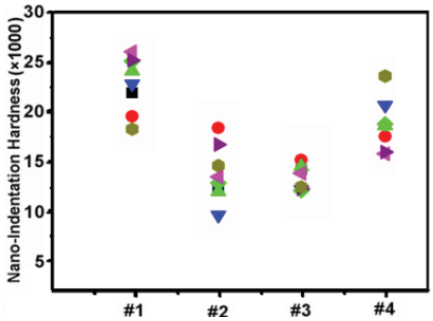

Figure 2. Nano-hardness of DLC coatings

3.2 Friction

Figure 3 shows the difference of COF of various lubricants on the alumina ceramics, from this figure, we can clearly understand that the DLC thin film can reduce the friction better than all lubricants.

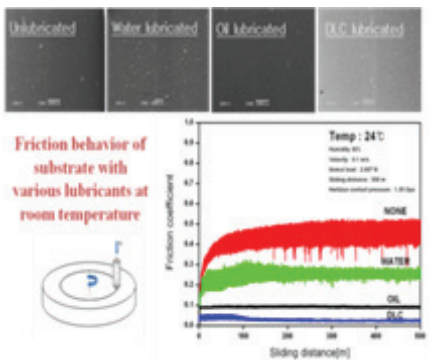

Figure 3. General comparison of ordinary lubricants with DLC on COF

At room temperature, the COF of untreated sample started at about 0.2, but it went up quickly and stabilized at an average of about 0.4, then increase further with rising temperature due to loss of the surface absorption and surface contaminants, which served as lubricants, a wide changeable area of COF can also be observed too.

DLC-coated samples #1 starts to fail at 200ºC due to the DLC delamination, a later SEM consideration showed that the failure was caused by the adhesion of the DLC to the substrates. #3 showed very high COF and oscillatory patterns due to the delamination of the coating observed from the SEM study, which will be discussed later. The COF of #2 and #4 remained super low which lasted for 1000 meters at the ambient and 500 meters at 200ºC without any noticeable degradation, and the COF of DLC increases substantially with the humidity [8, 9].

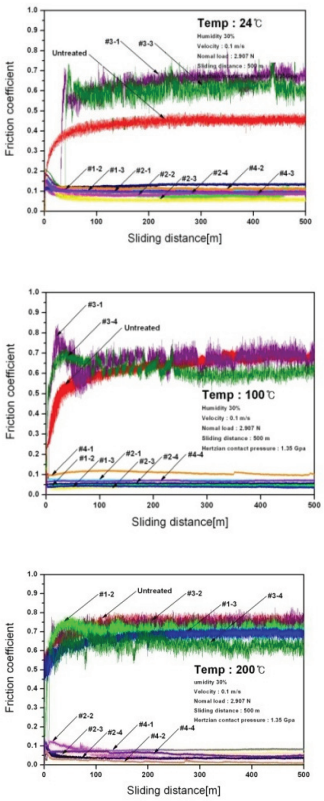

Figure 4. Coefficients of friction for all the samples tested at (a) room temperature, (b) 100ºC, (c) 200ºC

3.3 Wear

3.3.1 Wear morphology

After the pin-on-disc wear test, the samples were examined using an SEM. Shown in Figure. 5 are the wear tracks for all samples tested at the room temperature. As can be seen, the untreated sample showed a wide wear track. Samples #2 and #4 showed little damage while #1 showed some light damage. In contrast, the DLC coating was completely removed on

the #3 sample due to the serious delamination.

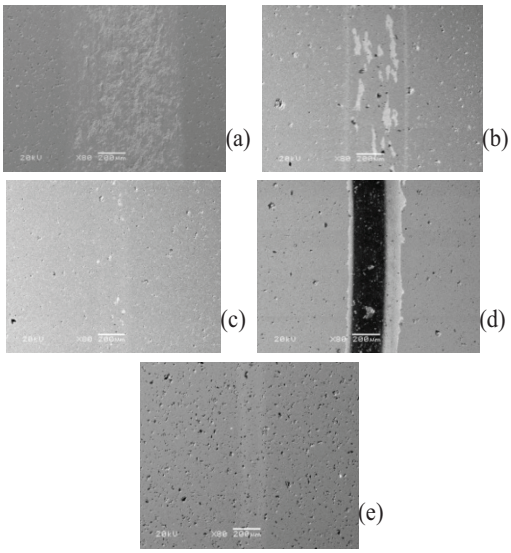

Figure 5. SEM photographs of the wear track tested at 24ºC for the samples:(a) uncoated (b) DLC-1 (c) DLC-2 (d) DLC-3 and (e) DLC-4.

3.3.2 Wear failure mode study

To study the failure mode, we examined the wear track border of sample #3 and the inside wear track for sample #2 as shown in Figure 6.

As can be seen, for sample #1 the coating seemed to be very brittle and delaminated to fail at 200ºC and the damage is of fractural nature.

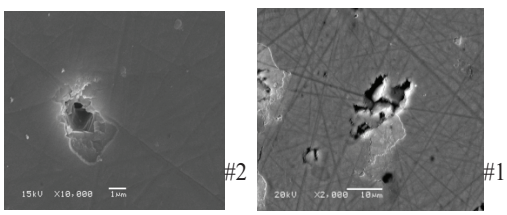

Figure 6. Failure mode of sample #2 and #1 at 200 ºC.

In contrast, sample #2 lost only a small area of the coating and it occurred only at the pores of the alumina substrate, the remaining coating around the damaged area still adhered well to the substrate. This was why the friction was still low even though part of the coating came off due to the impact of the ball.

3.4 Scratch Test

The scratch test is a widely method used to characterize the adherence of DLC coatings. In this test, the continuously increasing load on the diamond indenter will eventually cause damage to the DLC thin film, while the failure limit is reached, a succession of a single shock wave bursts and not a continuous signal spectrum is emitted, in this test the loading rate is 100N/min, and the speed of the specimen displacement is 10mm/min.

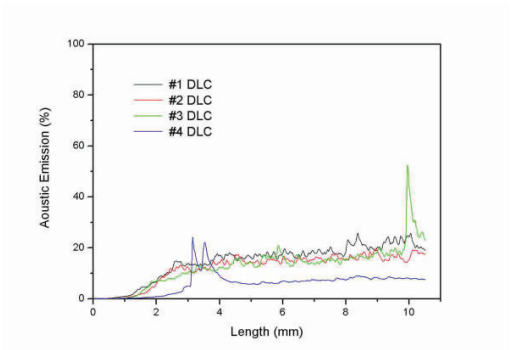

Figure 7. The scratch test of 4 DLC films

The low critical load #1,2,3 at which the load in the indenter just sufficient to generate a chipping failure on the film is almost similar around 11N, and a high critical load at which the load applied on the indenter just sufficient to generate a complete fake off the film is about 28N, for #4, the low critical load is around 20N, and the high critical load is around 33N, it does not match the exactly pin-on-disc test mainly due to the porous of the alumina oxide as shown in Figure 7, interlayer was used to accommodate for the internal stress and seemed to form compounds such as SiC and CrC to prevent the inter-diffusion between the DLC and the alumina substrate.

4. Conclusion

With the alumina alloy widely used in the industrial applications, DLC coated on the alumina become more

and more essential for further study for its high hardness, low friction and wear rate. DLC can be expected to achieve great performance in tribology of various die applications such as deep-drawing, minting, micro-die. As the experiment indicated, DLC #2, 4 showed a super low COF ranged 0.03~0.08 and good adhesion to the alumina alloy, so using DLC coated under PIID can be applied in the die applications. Compared with other DLC coating without interlayer in many die applications, in our article, interlayer Si, Cr is used to accommodate the internal stresses, the adhesion need to be further investigated using different interlayer and substrate.

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