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Short Communication

Evaluation of anti-adhesion characteristics of diamond-like carbon film using high-frequency, linear-oscillation tribometer

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ABSTRACT

The basic significance of a method of evaluating the anti-adhesion characteristics of diamond-like carbon (DLC) films by a reciprocating wear test using a high-frequency, linear-oscillation tribometer under a step loading condition was verified with a single test condition, and processes up to the occurrence of delamination were investigated in this study. The failure load, corresponding to the anti-adhesion capacity of the DLC film, varied greatly. When delamination of the DLC film occurred, the friction coefficient increased and the oscillation stroke decreased. However, these trends up to the occurrence of delamination were not uniform. Even when a delamination did not occur, the friction coefficient may have increased and the oscillation stroke may have decreased owing to the embedment of wear debris. It was revealed that the process from running-in to the occurrence of delamination exhibited two patterns depending on whether or not the embedment of wear debris occurred. A model representing the relationship between the phenomena occurring at sliding surfaces, the friction coefficient and the oscillation stroke was proposed.

1. Introduction

Diamond-like carbon (DLC) film is a next-generation tribological material with low friction and superior wear durability [1]. DLC films have been applied in various fields such as for industrial and medical use because of their excellent performance [2]. We have investigated the tribological characteristics of DLC films in terms of the effect of the surface roughness of the substrate material under a water lubrication condition and the difference between the characteristics under water lubrication and dry conditions [3,4].

The need for high-quality and reliable DLC films is increasing with their increasing use, as well as the establishment of standard technology to meet these requirements. The scratch test, Rockwell indentation test, bending test and pull test are typical examples of tests used to ensure satisfactory anti-adhesion characteristics between a DLC film and the substrate material. The scratch test is widely used to evaluate the anti-adhesion characteristics of thin solid films including DLC films, and it has already been standardized as an ISO standard [5]. A certified DLC film for use as a reference material is now available [6], and a round-robin test using this film has also been carried out [7]. The Rockwell indentation test is widely used in similar situations to the scratch test and has also been standardized as an ISO standard [8]. Although the ISO standard requires an evaluation involving four stages, a six-stage evaluation is often adopted (the Daimler-Benz Rockwell adhesion test) [9]. Regarding the bending test, the results of an evaluation involving 90° bending conforming to ISO 1519:2002 and U-shaped bending assuming metal forming have been reported [10,11]. The pull test is a popular and conventional evaluation method for verifying the improvement of anti-adhesion characteristics due to optimized film formation [12,13]. Recently, a new method involving the repeated pressing of a ball for the evaluation of anti-adhesion characteristics from the viewpoint of fatigue life has been proposed [14]. However, in the scratch test, the Rockwell indentation test and the bending test, the substrate material is markedly deformed because the contact stress is extremely high. In the pull test and the repeated pressing test, the effect of sliding is not considered. Therefore, neither test carried out under practical sliding conditions, and it is difficult to simulate actual delamination failures using these tests.

On the other hand, wear tests are an effective means of improving the reliability of DLC films, and are carried out under practical sliding conditions because the contact stress is much lower than that in the above-mentioned methods and because tests include frictional phenomena. The rotational wear test is a popular wear test, and a method of evaluating the friction and wear characteristics of DLC films using the rotational wear test has recently been standardized as an ISO standard [15]. Although the reciprocating test is also a popular wear test, regardless of the type of motion, delamination of the DLC film does not often occur under constant-load conditions [16]. A rotational wear

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test with a continuous increase in the load to induce delamination has been developed [17]. However, for the reciprocating wear test under a continuous increase in the load, there have been few reports on the occurrence of delamination [18]. Although the reciprocating wear test under a step loading condition has been standardized as an ASTM standard, this standard merely defines the test conditions and procedures, and concrete data are not included [19]. Furthermore, anti-adhesion and anti-wear performances measured in the scratch test, the Rockwell indentation test and wear test are often not in agreement [20]. We previously reported that there was no correlation between the critical load measured in the scratch test and the failure-resistant load measured in the wear test [21].

To evaluate the reliability of DLC films under sliding conditions similar to those in practical use, it is essential to establish a new method of investigating the performances of DLC films from an intermediate viewpoint between the scratch test and the wear test. As reported in this paper, the basic significance of a method of evaluating the anti-adhesion characteristics of DLC films by a reciprocating wear test using a high-frequency, linear-oscillation tribometer under a step loading condition was verified with a single test condition, and processes up to the occurrence of delamination were investigated.

2. Experimental procedure

2.1. Friction and wear tests

Fig. 1 shows an outline of the high-frequency, linear-oscillation tribometer (SRV model 3, Optimol Instruments Prüftechnik, Germany) that we used to evaluate the anti-adhesion characteristics of DLC films. In this test machine, a ball was pressed and rubbed against a plate, and then the friction coefficient was calculated on the basis of force signals obtained from load cells installed directly under the stage of the plate and attached to the loading system of the ball.

A DLC film (a-C:H) was deposited on a stainless-steel test plate (ISO X5CrNiCuNb16-4/UNS S17400/AISI 630/JIS SUS630) by an ionized deposition method. The film thickness was 4 μm including a gradient interlayer of Cr and Si. All tests were executed on the same test plate. Stainless-steel balls (ISO X110Cr17/UNS S44004/AISI 440C/JIS SUS440C) with a diameter of 9.525 mm used as rolling bearings were employed as test balls.

The tribometer was operated under a step loading condition with an

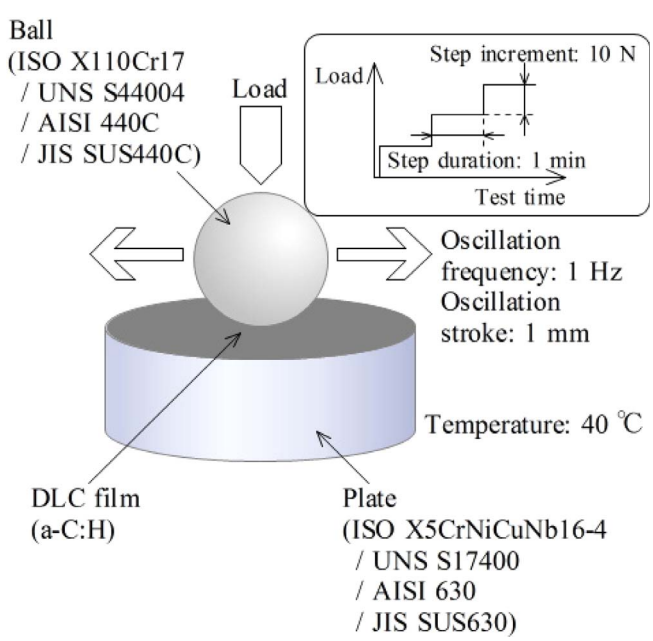

Fig. 1. Experimental setup.

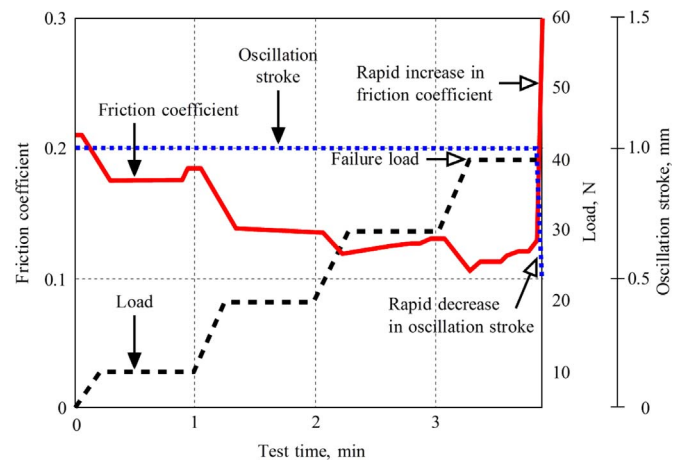

Fig. 2. Method of determining the failure load of a DLC film.

oscillation frequency of 1 Hz, an oscillation stroke (double amplitude) of 1 mm and a plate temperature of 40 °C in a dry environment. Because it was found in a previous study that the atmospheric conditions affect the friction and wear characteristics of DLC films [22], a dry environment was adopted to simplify the evaluation conditions where the relative humidity was 34–49%. The normal load was increased in increments of 10 N every 1 min.

Fig. 2 shows the method used to determine the failure load of a DLC film. The test was terminated when the friction coefficient increased by more than 0.15 from its value in the stable state and the oscillation stroke decreased by more than 0.25 mm from its value in the stable state. The load when the test was terminated was evaluated as the failure load, which was considered to be the anti-adhesion capacity of the DLC film. The test was executed six times (Nos. 1–6 as shown in Table 1) to confirm repeatability. The wear scars were observed by an optical microscope and their cross sections were measured by a surface profiler. Although the oscillation stroke of the reciprocating motion generated by a voice coil linear actuator in this test machine is controlled to maintain a constant value, if a large failure which inhibits smooth oscillation occurs at the sliding surface, fluctuations and the decrease of the oscillation stroke are induced. Therefore, the proposed method using the trend of the oscillation stroke is based on such feature of this test machine and the same concept is not worked for test machines that have a mechanically fixed oscillation stroke.

2.2. Investigation of cause of increased friction coefficient

From the results of the friction and wear test, it became clear that it was necessary to investigate the changes in the friction coefficient and the sliding surface. Thus, the following investigation was carried out. Some delamination tests were suspended when the friction coefficient reached about 0.2, 0.4 and 0.6 to investigate the surface states inside wear scars as the friction coefficient increased. The surface states were observed by an optical microscope and their cross sections were measured by a surface profiler. Furthermore, the surface states were observed by a scanning electron microscope (SEM), and also analyzed by

Table 1
Test results of the failure load of the DLC film (a-C:H).

Test no.	Failure load, N
1	220
2	170
3	160
4	310
5	220
6	160