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Excellent durability of DLC film on carburized steel (JIS-SCr420) under a stress of 3.0 GPa

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Abstract. To improve durability of transmission gears, Diamond Like Carbon (DLC) film coated on roller was estimated as well as TiN film. These films were coated on JIS-SCr420 steel, which was carburized, quenched, and tempered. DLC and TiN films were deposited by PCVD and PVD process, respectively. These surface modified rollers were estimated by usual metallurgical methods (observation of microstructure by optical microscope, SEM, and TEM, measurement of hardness by Vickers hardness tester and nano-indentator), measurement of friction coefficient by ball-on-disk in dry atmosphere, analysis of carbon by Raman spectroscopy and hydrogen by EDRA, and lifetime of pitting by the roller-pitting test. The hardness values were 21 GPa and 26 GPa, the elasticity coefficients were 192 GPa and 336 GPa, the friction coefficients were 0.1~0.15 and 0.5~0.6 for DLC and TiN films, respectively. The present DLC was a typical DLC called as hydrogenated amorphous carbon (a-C: H). The hydrogen content was about 20 %. The surface fatigue resistance of DLC-coated specimen had 100 times longer life than that the carburized and quenched one even under Hertzian contact stress of 3.0 GPa. TiN coated specimen was failed at 3.0 GPa by 5.17·105 cycles despite that the strength of the surface of the substrate was reduced due to the exposure at higher temperature in the coating process than the temperature for tempering.

1. Introduction

It has been required to reduce the size of automobiles for fuel economy and to lighten the adverse impact on the environment. To fulfil the requirement, it is needed to strengthen automotive components in a powertrain. Especially it is required of improvement in strength of gear, which is the core component of powertrain. Gears should be operated with durability at high-speed friction and/or high load, since gears are the major parts of the powertrain. Recently, the fatigue of tooth surface, especially pitting, has been a focus of attention as a serious problem [1]. Tooth surface is always operating under a rolling fatigue by a local high stress with slip [2][3]. To prolong the lifetime of gears, it is necessary condition to harden the tooth surface [4] and to provide a lower friction coefficient [5].
The present study aimed to clarify the differences in pitting resistance between the Diamond Like Carbon (DLC) and TiN-coated steels. The DLC and TiN coatings on the Carburized-Quenched-Tempered (CQT) JIS-SCr420 steel attract attention due to the superior machinable property \(^6\)[7][8], and the high effect on the decrease in friction.

2. Experimental procedures

2.1. Specimens

JIS-SCr420 steel, which is widely used in automotive parts such as gear, shaft, and so on, was carburized at 1,203 K for 420 min and then tempered in oil at 453 K for 120 min, in order to prepare specimens for roller pitting test and metallurgical estimation (specimen size of 20×30×7 mm\(^3\)). The R\(_a\) roughness of the small roller surface was finished in 0.4 \(\mu\)m after removal of the heterogeneous carburized structure by the machining. In addition, the bearing steels JIS-SUJ2 were used for the large roller of other partner specimen in the roller-pitting test. It was hardened higher than HRC62. The quenching and the tempering temperatures were controlled at 1103 and 453 K, respectively.

Some specimens were coated with DLC film by Plasma Chemical Vapour Deposition (PCVD) \(^9\) deposited with a mixture of acetylene at 503 K for 150 min, other specimens were covered with TiN film by ion plating method at 723 K for 70 min in order to estimate the effect of surface substance on durability of pitting resistance. The thicknesses of DLC and TiN films were adjusted to 1.0 and 3.0 \(\mu\)m, respectively.

The hydrogen content in DLC film was measured by Elastic Recoil Detection Analysis (ERDA), and its chemical combination state of the carbon were analyzed with Raman spectrometer.

The observation of the microstructure and measurement of hardness of the specimen before and after coating were carried out for estimation of the influence of coating. The microstructure in the cross section of specimens was observed by an optical microscope. In addition, the hardness of films were determined by a nanoindenter \(^10\) and the friction coefficient measured by the ball-on-disk in a dry condition.

2.2. Roller pitting test

A roller pitting test method, schematically shown as Fig.1, was used for the study on the characteristic of the pitting, under the following conditions; the load condition: 2.5~3.5 GPa (Hertzian contact stress at the sliding surface of small roller), lubricant: injecting amount of 2L/min Automatic Transmission Fluid (ATF) at 363 K, a rotation speed: 1,500 rpm, slip rate: 60 %. The test was ended when the test machine detected the vibration caused by the destruction of specimen or the number of rotation reached 2×10\(^7\) cycles.

The microstructure before and after examination was observed by Scanning Electron Microscope (SEM) and Transition Electron Microscope (TEM).
3. Results and discussion

3.1. Microstructure and hardness

Fig. 2 and Fig. 3 show the hardness profile and microstructure of the cross section before and after deposition, respectively. DLC, TiN and SCr420 (CQT) in Fig. 2 refer to the DLC-coated SCr420 (coated at 503 K and tempered at 473 K), TiN-coated SCr420 (coated at 723 K, tempered at 473 K) and SCr420 (CQT) without coating (tempered at 473 K), respectively. The microstructure of the DLC-coated specimens before and after deposition was almost the same as that of SCr420 (CQT), being consisted of martensite.

Meanwhile, the microstructure and hardness profile of the TiN-coated specimen were quite different from those of DLC-coated and SCr420 (CQT) specimens. The substrate of the TiN-coated specimen was evidently softened, since it is heated up to temperature of 723 K during the coating process. That is to say, high temperature tempering was caused by Ion Plating process.

The hardness-values of DLC and TiN films were 192 GPa and 336 GPa, the elasticity coefficients were 21 GPa and 26 GPa, and the friction coefficients were 0.1~0.15 and 0.5~0.6 in dry atmosphere, respectively.

The content of hydrogen in the present DLC film was measured to be about 20 %. From the Raman spectrum, the present DLC was identified as the typical DLC called as hydrogenated amorphous carbon (a-C:H)\[^{[11],[12]}\].

3.2. Roller pitting test

The result of roller-pitting test is shown in Fig. 4. In the SCr420 (CQT) specimen without coating, the crack initiated on the specimen surface while the maximum stress was generated at a depth of 0.3~0.4 mm from the specimen surface.

Meanwhile, the specimens coated with DLC or TiN films prolong the life equivalent or more than the uncoated specimen at all stress levels investigated. Accordingly, DLC film improved the pitting
resistance of 3.0 GPa and TiN 2.5 GPa. DLC prolonged the life by 100 times as that of uncoated specimen and ran without fracture on surface even under 3.0 GPa. DLC was, however, damaged on the surface at higher stress than 3.5 GPa, though DLC film remained.

Fig.5 shows the SEM image on the cross section of the DLC-coated specimen tested under 3.0 GPa, DLC-coated one tested under 3.5 GPa and TiN-coated one tested under 3.0 GPa. These found no damage and uniform structure in the DLC-coated specimen tested under 3.0 GPa. However when the stress level was high as 3.5 GPa, the cracking of the DLC film and plastic of the substrate near the surface were caused, while the film still remained. This implies that it is thought the DLC film could not follow the plastic flow of the substrate due to too high stress 3.5 GPa. In TiN-coated specimen, the plastic deformation was caused considerably so that the film was rolled.

![DLC-3.0 GPa](image1.png) ![DLC-3.5 GPa](image2.png)

Fig.6 shows the SEM image on the cross section of the DLC-coated specimen tested under 3.0 GPa, DLC-coated one tested under 3.5 GPa and TiN-coated one tested under 3.0 GPa. These found no damage and uniform structure in the DLC-coated specimen tested under 3.0 GPa. However when the stress level was high as 3.5 GPa, the cracking of the DLC film and plastic of the substrate near the surface were caused, while the film still remained. This implies that it is thought the DLC film could not follow the plastic flow of the substrate due to too high stress 3.5 GPa. In TiN-coated specimen, the plastic deformation was caused considerably so that the film was rolled.

**Fig.6 Microstructure on the cross section of TEM image**

Fig.6 shows the TEM image on the cross section the DLC-coated specimens tested under 3.0 and 3.5 GPa. It was comprehended that substrate of under 3.0 GPa became equality and minute structure to compare these images, though there was the imprint of plastic flow under 3.5 GPa. It is considered that the nanosize crystal was formed when high stress such as 3.0 and 3.5 GPa was added repeatedly during the roller-pitting test.

4. **Conclusion**

The main results are summarized as follows.

I. The durability performance of pitting resistance was drastically improved by the DLC coating. The 1μm thickness-DLC film prolongs the life by 100 times or more as it of pitting resistance under Hertzian contact stress of 3.0 GPa.

II. The TiN-coated specimen showed slightly higher than or comparable to the SCR420 (CQT) specimen without coating. The reason, why the improvement by the TiN coating was far smaller than that by DLC coating, could be attributed to the high temperature exposure in the TiN coating process (723 K for 70 min) in advance of CQT layer tempering (453 K for 120 min), which led to the reduction in surface strength of the substrate.

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