

# Effects of plasma carburizing and DLC coating on friction-wear characteristics, mechanical properties and fatigue strength of stainless steel

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## ABSTRACT

This study was conducted to investigate the effects of hybrid surface treatment on the friction-wear characteristics, mechanical properties and fatigue strength of austenitic stainless steel JIS SUS316. In hybrid surface treatment, after plasma carburizing as a pre-treatment had been performed at 773 K for 43.2 ks (12 h), DLC (diamond-like carbon) coating was performed. The formed duplex layer had the DLC layer (thickness 1.6–1.8 μm) outermost and the hardened layer (thickness 90 μm) below it. The DLC layer was markedly effective in decreasing the friction coefficient and improving wear resistance. At the same time, the durability of the DLC layer was improved by the existence of the hardened layer. Since no marked change in the microstructure of the substrate occurred through hybrid surface treatment, the mechanical properties were almost unchanged. Furthermore, fatigue strength was greatly improved by hybrid surface treatment (improvement rate 53%) because the generation of fatigue cracks from the surface was strongly suppressed by the duplex layer.

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## 1. Introduction

Austenitic stainless steel possesses excellent corrosion resistance in various environments and its crystal structure is stable in a wide range of temperature; however, stainless steel has inherent tribological problems, such as a high friction coefficient and low wear resistance. In some engineering applications, surface treatments are needed for stainless steel to overcome the above limitations. Plasma carburizing improves its wear resistance without marked deterioration of corrosion resistance when the treatment condition is selected appropriately [1]. Nevertheless, since this treatment cannot reduce the friction coefficient of stainless steel [2], further surface treatments are required.

In recent years, various DLC coatings, including multilayer DLC [3] and metal-doped DLC (Me-DLC) [4–7], have been investigated to improve the friction-wear characteristics of metals. For stainless steel, it has been reported that DLC coating was effective in reducing the friction coefficient even under water lubrication conditions [8–11]. If DLC coating is further performed after plasma carburizing, the friction coefficient of stainless steel can

be greatly improved as well as wear resistance. In this hybrid surface treatment, plasma carburizing as a pre-treatment plays an important role. Namely, the formed hardened layer effectively suppresses plastic deformation, which occurs below the DLC layer. As a result, the durability of the DLC layer will be improved. Actually, Ueda et al. reported that the friction coefficient of stainless steel, carburized initially and then DLC-coated, showed a low and stable value (0.20) over a long sliding distance [2].

On the other hand, the above hybrid surface treatment naturally involves the heating process of the stainless steel. Marked growth of crystal grains during surface treatments should be avoided to maintain the strength. Furthermore, high fatigue strength is desired in machine parts which are used under sliding conditions to prevent rolling contact fatigue. Some studies have reported the effect of DLC coating and its hybrid surface treatments [12–16] on the fatigue strength of metals. According to these studies, the fatigue strength of metals was improved by the formation of DLC layers. Nevertheless, more data concerning fatigue properties should be further accumulated to assure the safety of products as well as the friction-wear characteristics, which are directly related to functionality.

Based on the above background, this study was conducted to investigate systematically the effects of hybrid surface treatment on the friction-wear characteristics, mechanical properties and

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fatigue strength of austenitic stainless steel JIS SUS316. The hybrid surface treatment involved plasma carburizing and DLC coating, as mentioned above. In this study, four materials were prepared, that is, untreated material, plasma-carburized material (PC material), DLC-coated material (DLC material) and hybrid-surface-treated material (PC/DLC material). The first three materials were used for comparison with the PC/DLC material.

A variety of experiments were carried out to obtain comprehensive data. For all materials, the features of the formed layers and the microstructures of the substrates were observed on the cross sections and then the hardness distributions were obtained. The fundamental characteristics of the DLC layers of the DLC and PC/DLC materials, such as the thickness, hardness, Young's modulus and adhesion force, were examined, and then the indentation test was carried out. Moreover, the following tests were performed for all materials: the X-ray residual stress measurement, ball-on-disk type friction-wear test, EDS (energy dispersive X-ray spectroscopy) analysis of the wear tracks, tensile test, plane-bending fatigue test and observations of the fracture surfaces by SEM (scanning electron microscope).

## 2. Surface treatments and experimental procedures

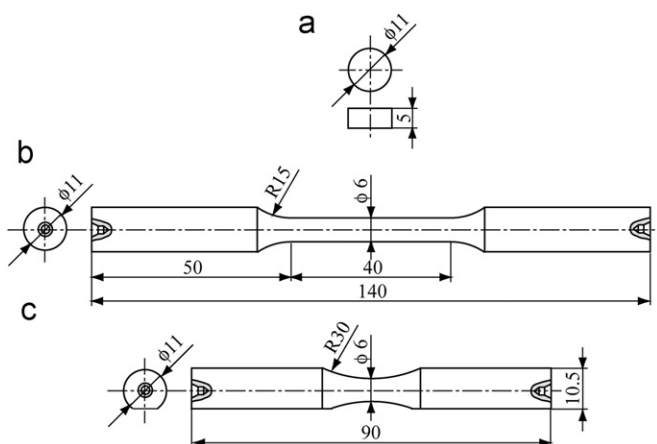
### 2.1. Surface treatments

Table 1 shows the chemical composition of the austenitic stainless steel JIS SUS316 used in this study. Stainless steel was supplied as round bars with a diameter of 12 mm. To homogenize the microstructure, the material was solution-treated at 1323 K for 3.6 ks (1 h) and quenched in water. The material was then machined to the configurations shown in Fig. 1.

Prior to surface treatments, the test sections of the button-type specimens were polished to a mirror surface with emery papers (#100–#2000) and alumina powders (#3000–#10000). The test sections of the tensile specimens and fatigue specimens were polished with emery papers in the same manner and finally electro-polished to a mirror surface. For electro-polishing, a mixture of acetic anhydride and perchloric acid was used.

**Table 1**  
Chemical composition of stainless steel JIS SUS316 (mass%).

C	Si	Mn	P	S	Ni	Cr	Mo	Fe
0.05	0.29	1.37	0.035	0.023	10.10	16.95	2.02	Bal.



**Fig. 1.** Specimen configurations (mm): (a) button-type specimen, (b) tensile specimen (JIS Z 2201, No.14), and (c) fatigue specimen (JIS Z 2274, No.2).

Plasma carburizing was conducted at 773 K for 43.2 ks (12 h). The test sections of the plasma-carburized specimens were polished again to a mirror surface with alumina powders. DLC coating was performed on untreated material and PC material using the UBMS (unbalanced magnetron sputtering) method. The details of DLC coating were as follows. Firstly, bombarding by Ar ions was carried out in a vacuum at  $2.6 \times 10^{-3}$  Pa to clean the test sections of the specimens. Then, after pre-heating at 773 K for 1.8 ks (0.5 h), an intermediate layer was formed to improve the adhesion force of the DLC layer. Finally, the DLC layer was generated at 473 K for 16.8 ks (4.67 h). In addition, plasma carburizing and DLC coating were respectively performed for the same batch to assure the equality of the specimens.

### 2.2. Experimental procedures

The button-type specimens were cut off and the cross sections were polished to a mirror surface with emery papers and alumina powders. The DLC layers of the DLC and PC/DLC materials were observed on the cross sections using SEM in detail. The hardness distributions of all materials were then obtained on the cross sections from the surface to 200  $\mu$ m depth. Micro-Vickers hardnesses were measured five times at each depth under a test force of 245 mN (25 gf) and their average was used as the hardness at each depth. After electrolytic etching was conducted using 10% oxalic solution, the microstructures were optically observed near the surfaces and the grain sizes of the substrates were measured.

For the DLC and PC/DLC materials, the thicknesses of the DLC layers were measured by the Calotest (spherical drilling method) [17–19]. The Calotest is a method to determine the precise thickness of coatings. In this test, a rotating sphere with known diameter is pressed on the coating surface and wears down the coating and substrate. Optical inspection of the depression reveals the projected surfaces of the abraded coating and substrate section. The thickness can be obtained by measuring the widths of the coating and substrate.

The hardness and Young's modulus of each DLC layer were obtained by the nano-indentation test under the maximum test force of 5 mN [20,21]. In the obtained ten data, the highest and lowest data were eliminated, and the average of the remaining eight data was used as the experimental result. Their adhesion forces were measured by the scratch test [22,23]. The indentation test was further carried out to investigate the adhesion condition of the DLC layers by the micro-Vickers hardness test under the test force of 9.8 N (1 kgf) and the indentations were observed by SEM.

The residual stresses of the untreated material and PC material were obtained on the surfaces of the button-type specimens using the X-ray residual stress measurement method; those of the DLC and PC/DLC materials were measured for the substrate through the DLC layers. The experimental conditions were as follows: Cr K $\alpha$  X-ray, diffraction plane (220), diffraction angle  $2\theta=128.7^\circ$ ,  $\sin^2\psi$  method ( $\psi=0^\circ, 5, 10, 15, 20, 25, 30, 35, 40, 45^\circ$ ), stress constant  $K=-628.5$  MPa/deg. Under the assumption of total diffracted intensity fraction  $G_x=0.95$ , the minimum penetrating depth of X-rays ( $\psi=45^\circ$ ) was calculated as 224  $\mu$ m for carbon (graphite), based on Ref. [24]. Accordingly, it can be said that X-rays passed through the DLC layers (thickness: 1.6–1.8  $\mu$ m), and the residual stress measurement was successful.

The relationship between the sliding distance and the friction coefficient was investigated using the ball-on-disk type friction-wear test. This test was conducted using alumina balls (diameter 5 mm) under a test force of 9.8 N (1 kgf) in dry condition at room temperature. The rotating diameter was 3 mm and the sliding speed was 40 m/s. The tests for the untreated material and PC material were terminated at a sliding distance of 1000 m because

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