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Evaluation of tribo-characteristics of diamond-like-carbon containing Si by metal forming simulators

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ABSTRACT

Environmental protection is an important issue among current manufacturing. Meanwhile, tribology in metal forming is making a great contribution to solve the problems related to both environmental and manufacturing aspects. High performance hard coatings and lubricants are in great demands for solving those problems. Diamond-like-carbon containing silicon, DLC-Si, which is an attractive material to reduce friction and prevent pick-up, has already been developed and used in the sliding parts of automobiles. This new coating has high adhesion strength, which has been proved in the application of metal forming. In this paper, its tribological characteristics are evaluated with different pressures and sliding velocities by four typical tribo-simulators: rotating compression test, strip-ironing test, ball-penetration test, and backward extrusion test. These results show that DLC-Si demonstrated high tribological properties in anti-galling and low friction. That means there is a high possibility for the application of DLC-Si on dies for cold metal forming.

1. Introduction

Recently, environmental protection and sustainable manufacturing have become hot issues. Concerning the environmental protection, resources, and energy conservation, manufacturing industries have started to develop new environmental friendly lubricants and long lasting machining tools [1–5]. Meanwhile, although the demand on metal forming of difficult-to-form metals, such as high strength steels, aluminium alloys, stainless steels, etc. has increased dramatically, the technology of preventing wear of tools and scratches formed on workpieces is still not mature enough. Regarding those problems, the industry tries to improve the tribological technologies. However, they are still not good enough to meet the expectation among the industry. In order to strengthen the tool materials in scratch resistance, instead of improving the properties of the materials like ceramics [6], there is a trend to develop and apply coating technologies because of their significant effective performances [7].

Conventional hard coatings, for example, VC which is formed by a thermal-reactive deposition and diffusion method, TiN and TiC which are formed by chemical vapor deposition, are widely used in cold metal working because of their excellent performances in anti-galling and anti-wear [8–10]. However, their friction coefficients are disappointingly high under severe frictional conditions in metal forming. As a result, the coating of diamond-like-carbon, DLC, which also has low friction coefficient and high wear resistance, becomes an outstanding coating for metal forming die [11]. The coatings of DLC and DLC containing chromium, titanium, tungsten, or silicon, etc. in the interface between the DLC and the base metal are applied on the moving parts and the cutting tools as well as dies for sheet metal forming. However, the DLC can only be applied to the dies that are limited to light forming conditions because of its poor adhesion to base metal [12–17]. Regarding this problem, Toyota Central R&D Labs., Inc. has developed a high adhesive coating of DLC containing Si, DLC-Si. The laboratories put the coating into practice on the automobile components of clutch discs [18,19]; however, it is not effective in forging due to the severe tribological conditions. Thus, the industry is looking forward to the improvement of DLC.

The tribological characteristics and the potential development of DLC-Si are evaluated by four tribo-simulators such as rotating compression test, strip-ironing test, ball-penetration test, and backward extrusion test.

2. Experimental details

2.1. DLC-Si coating

DLC-Si coating is coated on metals by a direct current plasma CVD method. This process to form DLC-Si was invented and its

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tribological characteristics were investigated by the researchers in Toyota Central R&D Labs., Inc. [18–21]. This coating method can provide mass production with relatively low production cost.

DLC-Si was formed through a process of direct current glow discharging by setting the external surface of vacuum chamber as anode and a specimen as cathode. During the vacuum-making process, the coating material is heated up to 500 °C by the direct current. The amount of Si in the coating depends on the composition of CH_4, $Si(CH_3)_4$ in the gas. The gas is provided to plasma CVD to form DLC-Si coating. The comparison between the composition of DLC-Si and DLC is shown in Table 1. The coating used in this research is composed with 10-at.% Si, 30-at.% H, 60-at.% C, where at.% means atomic percent. The coating thickness is 3 μm. The hardness values were obtained with nano-indentation measurements. The hardness is equivalent to the range of 1500–2000 HV in Vickers hardness numbers. The critical adhesion strength is 50–70 N according to the scratch test having the following conditions: the rate of loading 100 N min^{-1}, the scratching speed of 10 mm min^{-1}, the scratching length of 5 mm, and the tip of the same shape as Rockwell-C diamond with a radius of 200 μm [18].

A pre-activation surface process was treated before the DLC-coating. The pre-activation process consists of preliminary nitriding and succeeding ion bombardment. Plasma nitriding was carried out by DC discharge using gas of N_2 and H_2 at 600 MPa. The nitriding diffusion layer was 20 μm at 500 °C for 60 min. After plasma nitriding, H_2 gas and N_2 gas were admitted into chamber. The steel substrates were heated at 500 °C in the chamber. It was ion-etched with ion bombardment by H_2 ions and Ar ions at 300 V of bias voltage for 60 min. The surface of the steel was very finely roughened. According to cross-sectional TEM micrograph of the interface between the DLC-Si coating and the substrate nitriding, there was the entirely indented interface with a smaller roughness than about 10 nm. The critical load of DLC-Si coating was found to be higher than 50 N by the scratch test. Probably the fine roughness may promote an anchor effect between the substrate and the coating. Thus DLC-Si includes no intermediate layer between the coating and the substrate.

2.2. Ball-on-disk test for preliminary basic investigation

Ball-on-disk test was preliminarily conducted to determine the basic relationship between the friction coefficient, μ, and the amount of Si contained in the coating. In addition, a standard content of Si was also selected for the coating in this research. The coated disk is made by martensite stainless steel, SUS440C in JIS, which contains 1.1 mass% C and 17 mass% Cr. The disk has surface roughness of 0.01 μm Ra and the hardness of 650 HV. The ball is made by high chromium bearing steels, SUJ2 in JIS, which contains 1.0 mass% C, 0.25 mass% Si, and 1.5 mass% Cr. It has a diameter of 6.35 mm, surface roughness of 0.01 μm Ra, and the hardness of 800 HV. In the test, the load was set to be 10 N, which correspond to the maximum Hertzian contact stress of 1.3 GPa, the sliding speed was 0.2 m s^{-1}, and the sliding distance was 700 m. The test was conducted under 30–50% humidity condition without lubrication. After sliding for 700 m, the friction coefficient and the wear of the disk could be obtained [18]. They are affected by the Si content as shown in Figs. 1 and 2. The figures show that the friction coefficient of 2 at.% Si DLC-Si is similar to μ of DLC without Si. However, the friction coefficient of DLC-Si drops to 0.07 from 4 at.% in Si and it can reach as low as 0.05. Meanwhile, the wear volume of DLC-Si is proportional to the content of Si. When the Si content is lower than 4 at.% in Si, the wear depth is 0.2 μm which is close to that of DLC without Si. Referring to Fig. 2, the Si content at 10 at.% is the middle value of the range where the friction coefficient is constant. Therefore, the DLC coating with 10-at.% Si is selected as the standard DLC-Si coating in this study by using the following four tribo-simulators for metal forming.

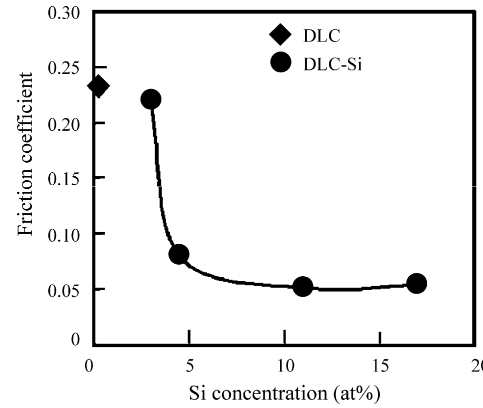

Fig. 1. Friction coefficient of DLC-Si coated on SUS440C against SUJ2 ball under dry condition.

The reasons for low friction coefficient of DLC have been explained by Mori et al. [18–21]. Although the corresponding friction coefficient is higher than 0.2 when the DLC contains 2-at.% Si in Fig. 1, no adhesion was observed on the ball by SEM, moreover no Si and no C were detected on the ball surface by X-ray photoelectron spectroscopy, XPS. Meanwhile, Si and no C were found on the ball surface even though there was low friction coefficient for the coating with 4-at.% Si. It suggests that the Si moved to surface of the ball. Moreover, no spectra changed on the sliding surface by analysis of visible light Raman micro-spectroscopy, and no spectra of σ-bond and π-bond changed on the surface by analysis of high-resolution transmission electron microscopy-electron energy loss spectroscopy, HRTEM-EELS. Therefore, the reason that graphite reduces the friction for the DLC-Si coating was eliminated. The XPS analysis showed that Si evenly distributes on the ball surface and O was surprisingly found; however the related bond due to Si and O was not clear by XPS and TOF-SIMS because the amount of product is too small to determine the group by XPS. Then, the surface was analyzed by a combination of XPS with substitution of Si-OH group into a compound including a fluoride. A derivative regent of tri-deca-fluoro-1, 1, 2, 2, 2-tetra-hydracrylic-octyl-dimethl-chlorosilane was used for the substitution. The amount of F increased with increasing Si in DLC-Si, so the existence of Si-OH group was found. Accordingly, the results conclude that the Si-OH group is effective in reducing friction. In

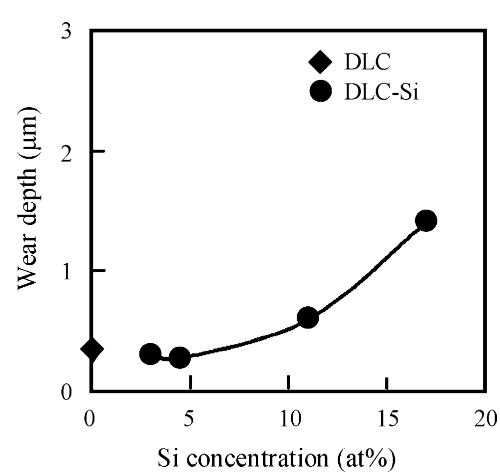

Fig. 2. Wear depth of DLC-Si coated on SUS440C against SUJ2 ball under dry condition.