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Investigation of DLC-Si coatings in large-scale production using DC-PACVD equipment

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Abstract

Probability of practical applications of the DLC-Si coating by DC-PACVD accompanied with the pre-treatment for improving adhesion strength was examined to numbers of small steel specimens with shapes of large-scale production parts. The whole surface of long narrow holes of 15 mm diameter 100 mm long were covered with the DLC-Si coating with enough thickness and high adhesion strength because of the uniform plasma-sheath formation at the inside holes. The coating was done on some hundreds of pins and rings with small spreads of coating thickness, silicon content, and adhesion strength. The process was concluded to have large probability for practical applications in the industry.

Keywords: Silicon containing diamond-like carbon; Direct-current glow discharge PACVD; Coating coverage; Adhesion; Friction

1. Introduction

Diamond-like carbon (DLC) coatings have excellent tribological properties such as high wear resistance and very low friction coefficient [1–4]. Moreover, the properties, such as high corrosion resistance, insulation resistance, gas diffusion resistance, and a high refractive resistance, are also excellent [5]. Therefore, DLC coatings have large possibility of wide use especially in automotive industry, where the DLC coatings having good productivity are highly desired from a viewpoint of ecology, if the coatings could be obtained easily on automotive components with complicated shapes in large numbers and also on production tooling.

Various DLC coating methods have been introduced up to now, magnetron sputtering [6], cathodic arc evaporation [7], ionized deposition [8], radio frequency (RF) PACVD [9], and direct-current glow discharge PACVD [10–12]. The last method, developed in Toyota Central Research and Development Labs. Inc. to coat DLC-Si coating, features very low friction coefficient, low equipment cost and easy scale up of the equipment for coating onto large numbers of parts in a loading. Furthermore, the adhesion strength of the DLC-Si coatings onto steel substrates by the method have been improved by the development of an activation pretreatment process so that the coatings can endure in severer servicing conditions [13]. The purpose of this paper is to show results of feasibility studies of the coating applications to a large number of small specimens well simulated some automotive components, without loss of the good characteristics of DLC-Si coatings recognized in the previous laboratory tests: low friction coefficient, high wear resistance, and strong adhesion, a part of them are summarized in Table 1 [12,13].

2. Experimental procedures

2.1. Specimen preparation

Four types of AISI 440C specimens, (1) 30 OD × 3 mm thick disk, (2) 6 OD × 55 mm long pins, (3) 25 OD, 15

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Table 1
Characteristics of the typical DLC-Si coatings obtained by PACVD

Structure	Amorphous
Si content	10 at.%
H content	30 at.%
Hardness by nanoindenter	20.7 GPa (maximum indentation depth 53 nm)
Adhesive strength—scratch test	50–60 N
—Rockwell C test	No spalling
friction coefficient—ball on disk test without lubricant	μ=0.05

ID × 100 mm long hollow cylinders, and (4) 100 OD, 94 ID × 2 mm thick rings were employed. The disk specimens were used for evaluation of tribological properties of the coatings and the hollow cylinder specimens for evaluation of the coating coverage onto the inside surfaces of the hollow specimens. The pins and rings were coated to evaluate feasibility of the process in mass-production. All specimens were quench-hardened to Hv 710 and ultrasonically cleaned in acetone after finished to surface roughness of 0.1 μm Rz (0.05 μm Ra).

2.2. Equipment and coating conditions

The used equipment for all coating operations was JPE-610-3S apparatus (Fig. 1) that was produced by NDK Inc., Japan, under license agreement with Toyota Central Research and Developments, Labs. Inc. It has a reaction chamber of inside diameter of about 800 mm and height of 1500 mm. Tetramethylsilane (TMS) and methane (CH_4) were added to hydrogen and argon as sources for silicon and carbon. The flow rates of each gas at the total pressure of 533–800 Pa were as follows: TMS 20–150 sccm, CH_4; 500 sccm, H_2; 300 sccm, and Ar; 300 sccm. Specimens on the negative electrode were heated to 500 °C by ion bombardment of DC glow discharge of 360–500 V discharge voltage and 0.2–0.6 mA/cm^2 current to make the DLC-Si coatings at the deposition rate of 3–5 μm/h on all types specimens. The apparatus is equipped with any mechanisms for neither revolution and rotation of loading devises and specimens nor internal or external heating systems. Prior to the coating, the surface activation pretreatment (preliminary nitriding followed by ion sputtering) shown in the previous paper [13] was applied to increase the adhesion strength of the coatings.

The hollow cylinders were placed on a disc of 550 mm diameter through small supporters of steel. Pins of 824 and rings of 500 were loaded as shown later together with results.

2.3. Evaluations of formed coatings

Hardness and thickness of the coatings were measured by a Nanoindenter (Hysitron Triboscope) and a spherical abrasion tester (CSM Calotester) with a ball of 10 mm diameter for hollow cylinders, 30 mm diameter for other samples. Electron Probe Micro-Analysis (EPMA) and Elastic Recoil Detection Analysis (ERD) determined contents of silicon and hydrogen in the coatings. X-ray diffraction (XRD) and Raman Spectroscopy were used for structure analysis of the coatings. Adhesion strength of the

Fig. 1. DC-PACVD equipment used for in this research (NDK JPE-610-3S).

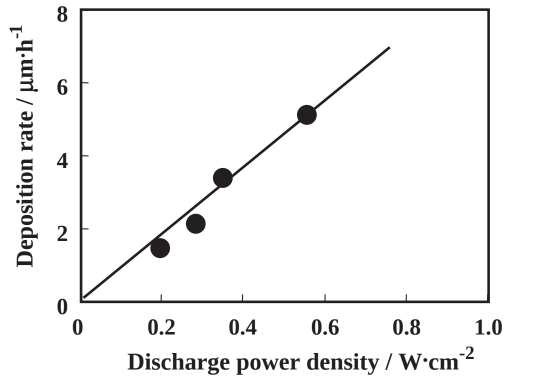

Fig. 2. Relation between discharge power density and deposition ratio of DLC-Si coatings.

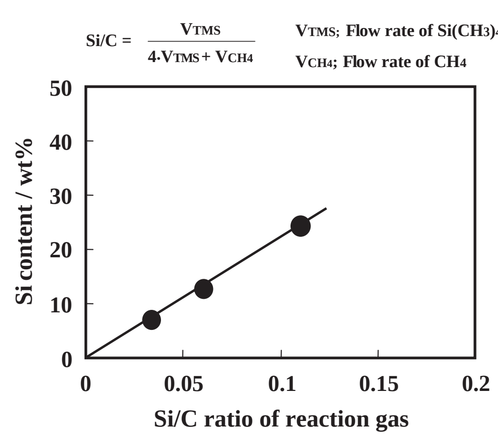

Fig. 3. Relation between Si/C ratio of reaction gas and Si content of DLC-Si coatings.