

Mass Density as Basis Parameter on Mechanical Properties under Diamond-Like Carbon Prepared in Wide Range of Conditions Using Variety of Methods

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Diamond-like carbon (DLC) films were prepared on steel substrates by eleven suppliers using different types of equipment utilized in each company's own coating formation process. The deposition methods include sputtering, electron cyclotron resonance chemical vapor deposition (ECR-CVD), plasma enhanced chemical vapor deposition (PECVD), arc ion plating (Arc), filtered arc deposition (FAD), and plasma-based ion implantation (PBII). Some correlation of mechanical properties seems to be valid in a narrow range of parameters produced by a particular deposition method. In order to investigate general correlations in a wide range of parameters, various DLC films were prepared by different suppliers on the same substrate, and were evaluated in exactly the same manner. Independent of deposition methods, there existed correlations of the hardness, elastic modulus and Raman shift with a wide range of mass density. © 2011 The Japan Society of Applied Physics

1. Introduction

Diamond-like carbon (DLC) shows excellent hardness, chemical inertness, durability, and low coefficient of friction, together with optical transparency. These mechanical properties are dependent on bond component (sp^2/sp^3 ratio), hydrogen (H) content, and amorphousness. The four main types of DLC are categorized into hydrogen-free tetrahedral amorphous carbon (ta-C), hydrogen-free amorphous carbon with lower tetrahedral fraction (a-C), hydrogenated ta-C (ta-C:H), and hydrogenated a-C (a-C:H).^{1,2} These DLCs have been prepared by various methods such as ion beam plating,³ magnetron sputtering,⁴ plasma chemical vapor deposition (PCVD),⁵ and vacuum arc deposition.⁶

Hardness significantly increases in ta-C films prepared by arc ion plating (Arc) method.⁷ Mass density and bond components correlate with the hardness of DLC films prepared by filtered arc deposition (FAD).⁸ The correlation of mechanical parameters are investigated on a series of samples prepared by a particular method under various deposition conditions. Some correlations of mechanical parameters can be valid in a narrow range of parameters produced by the same particular deposition method. Hardness, for instance, shows a correlation with mass density in a DLC film with a relatively lower mass density prepared by CVD method. In general, DLC films prepared by CVD show neither high mass density nor high hardness. The correlation can be valid at lower mass densities. It is unknown whether these relations are still valid on various DLC films prepared under different conditions by different methods with a wide range of variables.

In this study, DLC films were prepared by a variety of methods, which include both physical and chemical vapor depositions. Eleven companies using different types of equipment supplied 19 types of DLC film on common materials of bearing steel used in each company's own coating formation process. In order to investigate the basis factor for mechanical properties, the DLC films were evaluated in the same manner as the mechanical evaluation including Raman spectroscopy and an X-ray diffraction method.

2. Experimental Procedure

All the DLC films were deposited on substrates of bearing steel JIS SUJ2 (equivalent to AISI 52100), which is commonly used for actual sliding parts. The tempered steel showed a hardness of 60 HRC, and a roughness of $R_a \leq 0.01 \mu\text{m}$ was obtained on a mirror like finished surface using a profilometer. The steel substrate was a disk with a diameter of 33 mm and a thickness of 3 mm.

Eleven companies using different types of equipment supplied DLC films on common materials by their own deposition procedure. The deposition methods included sputtering, electron cyclotron resonance CVD (ECR-CVD), plasma-enhanced CVD (PECVD), arc ion plating (Arc), filtered arc deposition (FAD), and plasma-based ion implantation (PBII). These deposition methods were simply categorized into four types in this study, as mentioned below.

Each sample was evaluated in exactly the same manner. The nano indentation system, Hysitron TriboIndenter, was employed to evaluate the hardness and modulus of the DLC films. The applied load was adjusted to control the indenter penetration depth to be less than one tenth of the film thickness. A Berkovich indenter with a diamond tip was used in the indentation tests. The Raman spectroscopy and X-ray reflectivity (XRR) were used to evaluate bond component and mass density, respectively. JASCO NR-1800 and PANalytical X'pert MRD were employed for these purposes. In XRR measurement, X-ray irradiates a sample with a low angle of less than 1° , and the critical angle above which total incidence reflection occurs is related to the mass density of the target material.⁹ In addition to the mass density, XRR can be used to estimate film thickness and the roughness of thin films on a substrate. The thickness of the DLC films was out of range of XRR measurement in this study so that only the mass density was measured by XRR method. The hydrogen content was estimated by elastic recoil detection analysis (ERDA) with a 1.7 MV tandem accelerator. The films were irradiated with incident helium ions of 3.1 MeV and the recoil hydrogen atoms were measured. The adhesion between the film and the substrate was evaluated using a CETR scratch tester with a scratch speed of 10 mm/min and an applied loading rate of 100 N/min. A Rockwell C-scale indenter with a diamond stylus

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