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Evaluation of elastoplastic properties of DLC coating on SKD61 steel by optical indentation microscopy

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ABSTRACT

Diamond-like carbon (DLC) is well known as the unique properties of very hard, very low friction and low wear. That is why, the DLC coating has succeeded in commercializing on various field, including tools and/or dies' surface treatment. The life times of tools and dies are extended significantly. However, the mechanical properties of the DLC coating have not been understood well, yet. One of the authors (TM) has developed a novel method to measure the mechanical properties, such as Young's modulus, through the *in situ* determination of a true contact area during an optically transparent tip indentation by observing the contact image with CCD camera. The system, we called "Optical Indentation Microscopy" (OIM), enables one to determine true indentation stress-strain property. With this OIM, we have measured the mechanical properties of DLC coated and uncoated SKD61 (JIS or X40CrMoV5-1, ISO) steel samples. DLC coating made by our bipolar pulse PBII system. The thickness of DLC coatings were about 7 µm. Berkovich shape tip for usual indentation test and spherical-corn shape diamond indenter tip (0.4 mm in radius) for OIM were used. The maximum load and depth were about 2.5 N and 1 µm, respectively. Usual indentation test with Berkovich tip revealed that the DLC coated sample is harder than uncoated one, and the load-total penetration depth plots in logarithm shows the data of the DLC coated sample can be analyzed as if the tip shape is spherical. From the result of OIM, Young's modulus, yield stress and elastic limit of the DLC coated and uncoated SKD61 samples are 156 GPa and 201 GPa, 2.4 GPa and 2.6 GPa, and 2.2 GPa and 1.1 GPa, respectively. The DLC coating can improve the elastic limit of SKD61 by 100%. This effect of DLC coating is very useful to improve the lifetime of tools and dies.

1. Introduction

Diamond-like carbon (DLC) is well known as the unique properties of very hard, very low friction and low wear. That is why, the DLC coating has succeeded in commercializing on various field, including tools and/or dies' surface treatment. The life times of tools and dies are extended significantly. However, the mechanical properties of the DLC coating have not been understood well, yet.

Usually, indentation test with Vickers or Knoop tip is suitable to investigate mechanical properties of bulk materials. However, the test is not suitable for thin films because the effect of substrate is not negligible. So, instrumented indentation test, so-called nano-indentation test, which measures indentation load (P) and total penetration depth (h_t) simultaneously, is commonly used in order to evaluate mechanical properties of thin films.

Unfortunately, the height of real contact end between sample surface and indentation tip, which is directly related contact area (A_c), can not be measured with this method. In evaluation of hardness and Young's modulus from the instrumented indentation test, Oliver and Pharr approximation [1] is also commonly used, which is based on an elastic assumption for the P–h_t unloading path. However, many authors have pointed out that the assumption is not good for coating/substrate composite materials or ion implanted materials [2,3]. So, instead of measuring the penetration depth with indentation load, measuring the contact image, or contact area, through transparent indenter has been proposed and called "Optical Indentation Microscopy" (hereafter we call "OIM") [4].

The OIM can measure mechanical properties, such as Young's modulus, through the *in situ* determination of true contact area during an optically transparent tip indentation by observing the contact image with CCD video camera [2,4]. The system enables one to determine true indentation stress-strain property.

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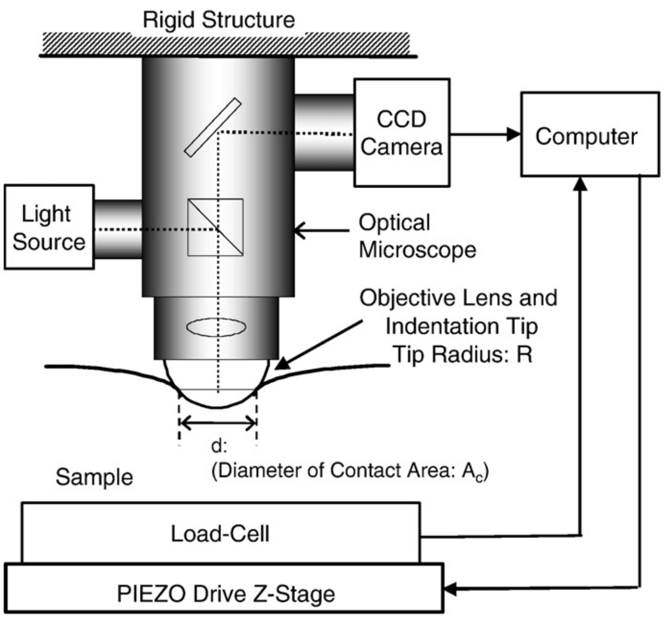

Fig. 1. Schematic illustration of "Optical Indentation Microscopy" (OIM) system.

In this paper we measured the mechanical properties of DLC coated and uncoated steel samples with the OIM, and made some comparison between the results obtained by a usual instrumented indentation test and our OIM system.

2. Experimental

DLC coating was carried out by our PBII system. The outline of our PBII system using bipolar pulses has been described in our previous paper [5]. Toluene ($C_6H_5CH_3$) was used as a precursor gas for DLC coating. The flow rate of toluene gas was set constant at 2.5 sccm, and the total gas pressure in the DLC coating process was set constant to 0.3 Pa.

A mirror polished SKD61 (JIS or X40CrMoV5-1, ISO) steel disk (25 mm ϕ x 5 mm) was used as a substrate. DLC coating process consists of: (1) Ar plasma sputter cleaning at −2 kV, (2) carbon ion implantation at −20 kV using CH_4 plasma, then (3) DLC deposition with +2 kV and −5 kV pulses with 4 kHz repetition under toluene plasma. Process times of (1), (2), and (3) were 30 min, 30 min and 4 h, respectively.

Raman spectroscopy was measured to determine the properties of coated DLC with Horiba, HR-800MX. Film thickness was evaluated by a laser microscope (Olympus, OLS3000) and it was about 7.1 μm.

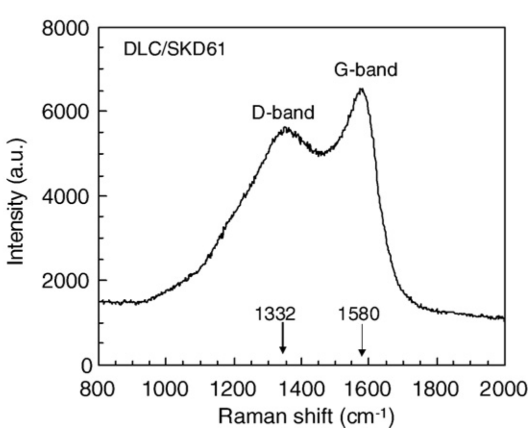

Fig. 2. Raman spectrum of the DLC coated SKD61 sample.

Table 1
Vickers hardness (H_v) of DLC coated and uncoated SKD61 steel.

Applied load (N)	DLC coated H_v (GPa)	Uncoated H_v (GPa)
9.8	6.5	5.3
49	5.8	5.6
98	5.7	5.6

Schematic of our OIM system is shown in Fig. 1. Spherical-conical shape (0.40 mm in radius at the end and 90° in angle) diamond tip was used as objective lens and indenter tip. Briefly, main body is optical microscope (Olympus, BX-51 M) and added with a computer controlled piezo-actuator in Z-axis, which applies displacement (or load) to the sample, and a load cell and a CCD camera, they were connected to the computer which evaluates indentation load and contact area. The details of OIM system were described in Miyajima and Sakai [4]. The maximum indentation load and depth were about 2.5 N and 1 μm, respectively. Using Berkovich shape diamond tip, usual instrumented indentation test was also performed by the same system added with a displacement sensor. Ordinal Vickers hardness tests with 9.8, 49 and 98 N loads were also performed.

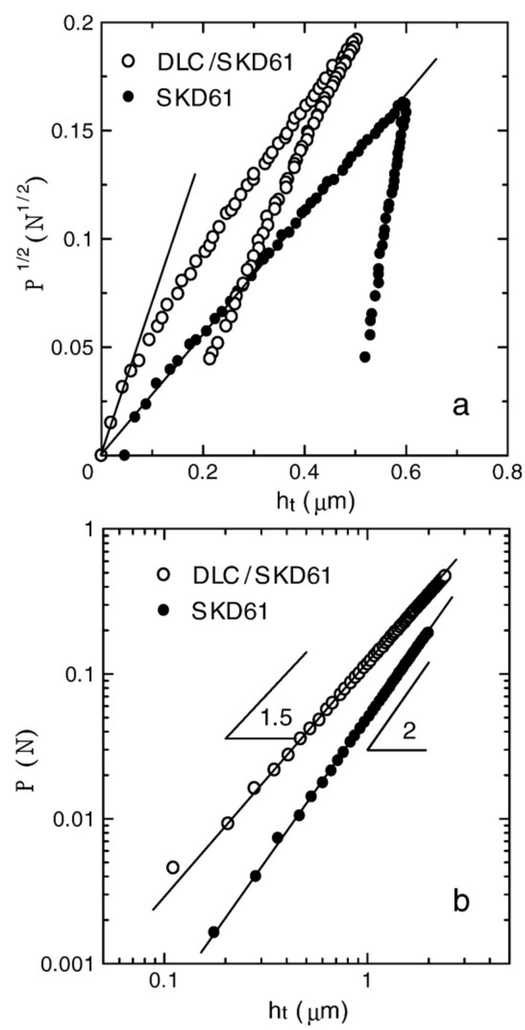

Fig. 3. Results obtained by usual instrumented indentation test with Berkovich shape diamond tip for DLC coated (shown as DLC/SKD61) and uncoated SKD61 samples. (a) $P^{1/2}$–h_t plots and (b) P–h_t plots in logarithm, where P and h_t represent indentation load and total penetration depth, respectively. Easy for eyes, lines are shown in the figures.