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Transparent Si–DLC coatings on metals with high repetition bi-polar pulses of a PBII system

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ABSTRACT

Diamond-like carbon (DLC) is widely used because of its good properties. However, the color of DLC is usually dark brown or black. Recently, we have made fairly transparent Si contained DLC (Si–DLC) coatings in visible light region. The fairly transparent Si–DLC was made by using our original bi-polar pulse type plasma based ion implantation (PBII) system, with recently introduced high slew rate pulse power supply. The colors of metal sample surface were uniformly changed as subdued red, yellow, subdued green and subdued blue or violet, with the change of Si–DLC coating's thickness. The colors come from the interference between reflected lights at the surface of the Si–DLC coatings and the surface of the metal samples. The colors were also changed with the angle of glancing.

Estimated refractive indexes show well agreements among almost all Si–DLC coatings, instead of the differences of coating conditions. Generally, the longer coating time or slower coating process makes the higher refractive index in near infrared region. Estimated band gap of a Si–DLC coating was about 1.5 eV. The developed Si–DLC coatings must be useful as not only protective but also decorative coatings.

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1. Introduction

Diamond-like carbon (DLC) is widely used because of its good properties. However, the color of DLC is usually dark brown or black, except for some DLC coatings for tools, their color are like rainbow [1]. The rainbow color is a character for almost hydrogen free DLC coatings they are made by sputtering [1] and pulsed laser ablation [2].

On the other hand, it is very difficult to make optically transparent hydrogen contained DLC coatings in visible light region, they are usually made by plasma CVD process or plasma based ion implantation (PBII) process. However, Abbas et al. [3] has revealed that Si incorporation into DLC lead the DLC films relatively optically transparent.

We have also been studying Si contained DLC (Si–DLC) coatings using our original PBII process [4] for several years [5–10]. In these studies, we have also found that Si–DLC coatings are relatively transparent than usual DLC coatings at the same thickness. And more recent introduction of high slew rate pulse power supply system to our PBII system, we can make fairly transparent Si–DLC coatings in visible light region. In this paper, we will show some results and discussions on the relatively transparent Si–DLC coatings on metals.

2. Material and methods

Mirror polished stainless steel (SUS304) and aluminum alloy (A5052) of 25 mm in diameter and 3 or 5 mm in thickness, Si wafer (for thickness measurement) and slide glass (for optical measurement) were used as substrates. Si–DLC coatings were performed with our bi-polar pulse type PBII system. The Si–DLC coating procedure is as follows; (1) Ar sputtering for 10 min with Ar gas flow of 3 sccm at the pressure of 0.3 Pa and +pulse: +2.0 kV, 0.25 μs, and −pulse: −5.0 kV, 1 μs, (2) CH_4 treatment for 10 min (this process has not been done in some cases), with CH_4 gas flow of 3 sccm at the pressure of 0.8 Pa, and +pulse: +1.5 kV, 0.25 μs, and −pulse: −5.0 kV (typ.), 1 μs, and (3) Si–DLC coating for 30, 60, or 160 min with tetramethylsilane (TMS, Si $(CH_3)_4$) gas flow of 3 sccm at the pressure of 1 Pa and +pulse: +1.5 kV, 0.25 μs, and −pulse: −4.0 kV, 1 μs. The values of voltage and time width of +pulse and −pulse shown above are setting values in our pulse control program. The real values in the vacuum chamber were different [10]. The repetition frequencies of the bi-polar pulses were set from 4 to 12 kHz as shown in Table 1 with other conditions, thickness and name of each coating.

Observation of samples was performed with a digital camera (Nikon D1 with PC-E Micro Nikkor 85 mm f/2.8D). Optical

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Table 1
Summary of coating conditions.

Rep. freq. (kHz)	CH_4 proc. time (min)	Coating time (min)	Thickness (nm)	Comment	Name of coatings
4	0	60	330		Si–DLC-c
4	0	160	850		Si–DLC-c
8	10	30	250	−2.0 kV in CH_4 proc.	Si–DLC-e
8	10	31	160	Pulse widths were twice	Si–DLC-a
8	10	30	185		Si–DLC-b
12	10	30	220		Si–DLC-d

measurements of reflection (5° off from normal) for all samples and optical transmittance for a slide glass sample were performed with an UV–Vis.–NIR spectrophotometer (Hitachi High-Tech., U-4100). Thickness of Si–DLC coatings on Si wafer substrates were measured with a profile micrometer (Keyence VF-7510). The measured thickness (d) of Si–DLC coatings are in the range of 160–850 nm as shown in Table 1.

3. Results and discussion

Surface images of A5052 and SUS304 samples are shown in Fig. 1 with the names of coatings and their thickness. Real images of these samples with eyes are much metallic than shown in Fig. 1, because these images were taken with scattered reflection lights from the sample surface, by using the camera with the shift lens.

To see Fig. 1, the colors of metal samples are uniformly changed from subdued red, yellow, subdued green and subdued blue or violet, with the change of Si–DLC coating's thickness. It is very clear that the color depends on the thickness of Si–DLC coating. The uniform color of each sample also shows the coated Si–DLC thickness was also uniform. In the case of Si–DLC-b and Si–DLC-c, the colors are resemble, however, the thickness are different. We will discuss this later.

The colors for SUS304 seem to shift to longer wave length (or red shift) a little, which means the coatings on SUS304 samples are thicker than those on A5052 samples. The colors of the thickest samples of Si–DLC-f (850 nm in thickness) on both metal substrates, were dark brown or almost black (not shown).

The images of Si–DLC-a on an A5052 sample with different angle of glancing are shown in Fig. 2. Apparently, the color of the film

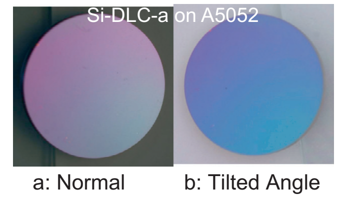

Fig. 2. Images of Si–DLC-a on an A5052 sample with different angle of glancing. 5° off from normal (left) and tilted angle (right).

is changed with the glancing angle. It means both of the colors of Si–DLC-a on A5052 shown in Fig. 2 are not an intrinsic color of the coating itself.

Results of reflection measurement for all samples in Table 1 are shown in Fig. 3. Every data show peaks and valleys at a regular interval in the wavenumber space, which means occurrence of optical interference. In visible light region (400–800 nm, within two dashed lines in each figure), there are one, two or three peaks according to the thickness.

The wavenumber or wavelength of the central reflectance peak in visible light region for Si–DLC-b and Si–DLC-c are almost same of around 19,000 cm^{-1} or 450 nm. In spite of the different thickness, almost same positions of the central peak lead the resemble colors of their colors as shown in Fig. 1.

In Fig. 3f of 850-nm thick coatings, the reflections in visible light region are very small, so the colors were dark brown or almost black, as mentioned above. The absorption of visible lights by the coatings increases with the thickness. At this moment, to get relatively brilliant colors, the thickness of coatings should be less than 600 nm. In other words, we can use the Si–DLC coatings up to 600 nm in thickness as decorative coatings on metals.

On thin-film light interference, there are well known conditions. They are represented as follows;

$$\text{Bright conditions,} \quad 2nd = (m + 1/2)\lambda, \quad (m = 0, 1, 2, \ldots) \tag{1}$$

$$\text{Dark conditions,} \quad 2nd = m\lambda, \quad (m = 1, 2, 3, \ldots) \tag{2}$$

where n: refractive index of film at a wave length of λ, and d: thickness of film.

Using these equations, we can estimate the value of n at each λ, where reflected light is bright or dark. The estimated refractive index profiles (as shown in Fig. 4) show well agreement among the Si–DLC coatings, instead of the differences of coating conditions.

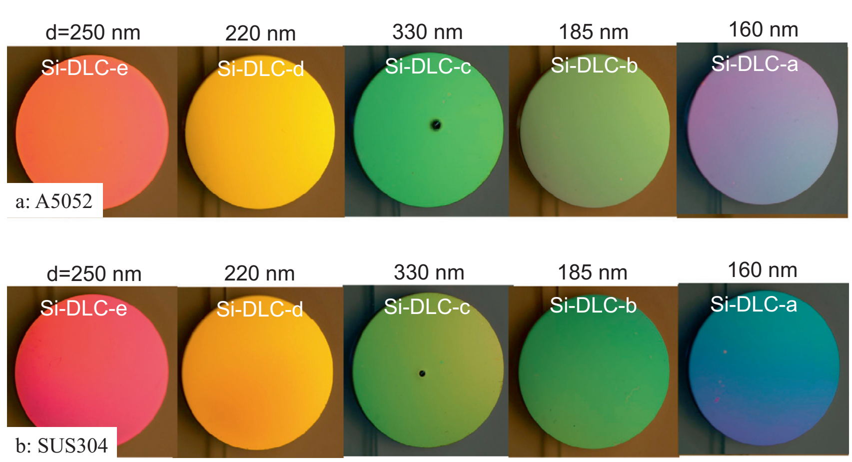

Fig. 1. Surface image of A5052 and SUS304 samples with the name and thickness of each coating. Dark circular images around the center of Si–DLC-c coatings on both A5052 and SUS304 substrates are traces of Rockwell HRC tests.

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