Study of optical dumpers used in high vacuum system of interferometric gravitational wave detectors

T. Tomaru¹, Y. Saito¹, T. Kubo¹, Y. Sato¹, M. Tokunari², R. Takahashi³, T. Suzuki¹, Y. Higashi¹, T. Shintomi⁴, Y. Naito⁵, N. Sato¹, T. Haruyama¹ and A. Yamamoto¹

¹High Energy Accelerator Research Organization (KEK), 1-1 Oho, Tsukuba, Ibaraki, 305-0801, Japan
²Institute for Cosmic Ray Research (ICRR), University of Tokyo, 5-1-5 Kashiwanoha, Kashiwa, Chiba, 277-8585, Japan
³National Astronomical Observatory of Japan (NAOJ), 2-21-1 Osawa, Mitaka, Tokyo, 181-8588, Japan
⁴Nihon University, 4-2-1-602 Kudankita, Chiyoda, Tokyo, 102-0073, Japan
⁵Anritsu Corporation, 1800 Onna, Atsugi, Kanagawa, 243-8555, Japan

E-mail: tomaru@post.kek.jp

Abstract. Outgassing velocities and optical absorption coefficients of black colored optical absorbers (UB-NiP, Phosblack II, Raydent, ECB+DLC and Alumite) were studied to reduce scattered and strayed light in interferometric gravitational wave detectors. The measured results showed that the UB-NiP had the largest optical absorption coefficient of 99.89% for 1064 nm wavelength of light. For the outgassing velocity, the ECB+DLC had the lowest value, which was $1 \times 10^{-8}$ Pa m$^3$/s/m$^2$ at 20 hours. This value was about two orders of magnitude smaller than that of the UB-NiP, however, its optical absorption coefficient was only 65%. Therefore, we concluded that the UB-NiP is suitable as an optical dumper with small area and high efficiency, and the ECB+DLC is suitable as large area coating such as a use for vacuum chamber walls.

1. Introduction

Strayed and scattered light in interferometric gravitational wave (GW) detectors can cause unidentified noises. In several GW detectors, metal baffles to block conduction of scattered light in beam ducts and multi-reflective beam dumpers to absorb undesirably reflected beam from optical components were introduced. For effective dumping of these undesirable light, black colored coatings are useful. However, there are few reports about black colored coatings possibly used in high vacuum system.

We investigated outgassing velocities and optical absorption coefficients for 1064 nm wavelength of light for five kinds of black colored coatings, which are ultra-black NiP (UB-NiP)[1, 2], Phosblack II, Raydent, ECB+DLC[3] and Alumite. The UB-NiP is a coating of nickel-phosphorus plating on a copper plate, developed by Anritsu Co. Since it is etched by nitric acid after the plating, it has extremely rough surface. It is known that its optical absorption coefficient is very large over wide range of wavelength from 488 nm to 1550 nm. The Phosblack
II is also a coating of nickel-phosphorus plating with smooth surface, manufactured by Asahi-precision Co. The Raydent is a kind of chrome oxide coating, manufactured by Raydent Co., and the alumite is a kind of aluminum oxide coating. The ECB+DLC is a diamond-like-carbon coating formed on electrochemical buffed steel (SS) surface. This coating is not black color, but has optical absorption around 1 μm wavelength of light.

2. Measurement procedure and experimental setup

Figure 1. Flowchart of measurement procedure

Figure 1 shows flowchart of measurement procedure. Firstly, we measured outgassing velocities and optical absorption coefficients for 1064 nm wavelength of light of the samples in the manufactured condition. Next, we baked the samples, and measured again those of the samples.

Outgassing velocity is defined as

\[ q = \frac{C \times P_{samp}}{S} \text{ Pa m}^3/\text{s/m}^2, \]  

where \( C \) is a conductance of the vacuum system, \( P_{samp} \) is a net outgassing pressure from the sample, and \( S \) is surface area of the sample. In this experiment, we used an orifice method and a conductance modulation method[4] to determine the conductance \( C \) of the system (Fig. 2). In the orifice method, which was used in outgassing velocity measurement for non-baked samples, the conductance of the vacuum system was calculated from conductance of the orifice. In the conductance modulation method, which was used for baked samples, the conductance was calculated by measuring pressure change in the vacuum chamber when conductance of the pumping was modulated. To obtain net outgassing pressure from the sample \( P_{samp} \), outgassing velocity of the vacuum chamber was firstly measured. Types of vacuum gauges were a spinning-rotor gauge and a Bayard-Alpert (BA) gauge for the apparatus of the orifice method, and a extractor gauge for the apparatus of the conductance modulation method. After the background measurement of outgassing velocity, samples were set in the chamber, and outgassing velocity of the samples were measured. By subtracting outgassing velocity of the chamber from that of the sample inserted, net outgassing velocity of the sample was calculated. Compositions of outgas were analyzed by mass spectrometers at regular intervals. Total measurement time for each sample was about 100 hours.

Figure 3 shows a measurement setup for optical absorption coefficient. In this apparatus, total integrated reflectivity \( R \) for 1064 nm wavelength of light was measured by using an integrating sphere, and optical absorption coefficient \( A \) was calculated from \( A = 1 - R \). The injected laser power, which was a few hundred mW in this experiment, was measured by a standard white plate with reflectivity of almost 1, to eliminate geometric errors in the integrating sphere measurement. Beam diameter on the sample surface was about 0.5 mm.
Figure 2. Measurement apparatus of outgassing velocity. (a) orifice method, (b) conductance modulation method.

Figure 3. Measurement apparatus of total integrated reflectivity \( R \) of a sample by using an integrating sphere. Optical absorption coefficient was derived from \( 1 - R \).

The baking condition for the UB-NiP was 250°C, 24 hours and \( 4 \times 10^{-4} \) Pa, which was decided from a preparatory baking test to investigate a condition with keeping a good performance for the optical absorption. The Raydent samples were baked in a condition of 150°C, 24 hours and in vacuum. Other samples were not baked.

3. Measurement result of outgassing velocity
Figure 4 shows measured results of outgassing velocities for the black colored coatings. Outgassing velocities for several materials were also shown in the graph as reference [5, 6]. The ECB+DLC had the smallest outgassing velocity, and its value was \( 1 \times 10^{-8} \) Pa m\(^3\)/s/m\(^2\) at 20 hours. This level is one order of magnitude smaller than that of the electrochemical buffed stainless steel surface (SUS ECB), which is a well known material with small outgassing velocity. The UB-NiP after baking had also small outgassing velocity comparable to the SS41 steal, however its value of \( 6 \times 10^{-7} \) Pa m\(^3\)/s/m\(^2\) at 20 hours was 60 times larger than that.

\(^{1}\) For the Phosblack II, stabilization of the coating by heating has been done in the manufacture process.
of the ECB+DLC. Outgassing velocities of the Raydent and the Phosblack II were levels of $10^{-6}$ Pa·m$^3$/s/m$^2$ at 20 hours, and their values were comparable to that of a machinable ceramics (MACOR). The outgassing velocities of these four black colored coatings were almost inversely proportional to the measurement time. This means that gasses were only absorbed on the sample surface. On the other hand, the alumite had large outgassing velocity and gentle curve. This can mean that outgas of the alumite came from inside of the coating.

One remarkable thing is that the outgassing velocity of the UB-NiP was reduced of two orders of magnitude by baking. Figure 5 shows outgas compositions of the UB-NiP at 20 hours, analyzed by mass spectrometers. Generally, main outgas composition of typical materials with small outgassing velocity is $H_2O$ ($m/e = 18$). Main outgas composition of the UB-NiP after baking was also $H_2O$ (Fig. 5 (b)), since $m/e = 1$ composition was caused by ionization of $H_2O$.

On the other hand, that of the UB-NiP before baking was $H_2$ ($m/e = 2$). Therefore, $H_2$ could be absorbed on the surface in the process of etching by nitric acid, and be released by baking.

From this result, we confirmed that baking of the UB-NiP is effective.

4. Measurement result of optical absorption coefficient

**Table 1.** Measured results of optical absorption coefficients of the black colored coatings. For the UB-NiP, averaged value between three samples after baking was adopted.

| Coating        | Optical absorption coefficient |
|----------------|-------------------------------|
| UB-NiP         | 99.89%                        |
| Phosblack II   | 85 %                          |
| ECB+DLC        | 65 %                          |
| Raydent        | 83 %                          |
| Alumite        | -                             |

Table 1 shows measured results of optical absorption coefficients of the black colored coatings for 1064 nm wavelength of light. The UB-NiP had extremely large optical absorption coefficient of 99.89%. Its large optical absorption coefficient can come from its complex surface structure. Figure 6 shows a surface image of the UB-NiP measured by a scanning electron microscope and a laser microscope. The sharp peaks on the surface were formed by nitric acid etching. Since optical absorption coefficient of the Phosblack II, which is also nickel-phosphorus plating but had smooth surface, is only 85%, the sharp peaks on the UB-NiP surface can cause multi-absorption of light. Optical absorption coefficient of the ECB+DLC was only 65% in a condition of perpendicular beam injection onto the surface and non-polarization of laser beam. However, there is a report that optical absorption coefficient of the ECB+DLC in the condition of 40° beam injection and p-polarization was 95%[3]. For the alumite, we obtained abnormal results such that detected reflectivity signals were over 1. Since a possibility that such signal was observed were that reflective light from the sample surface directly injected into a photodetector in the integrating sphere, we measured it again after setting an additional baffle in front of the photodetector, however, the reflectivity signal did not improve. Since we are under studying the reason, reflectivity of the alumite was eliminated from the Table 1.

5. Conclusion

We measured outgassing velocities and optical absorption coefficients for 1064 nm wavelength of light for the UB-NiP, the Phosback II, the ECB+DLC, the Raydent and the alumite. The UB-NiP had the largest optical absorption coefficient of 99.89%. It also had small outgassing velocity of $6 \times 10^{-7}$ Pa·m$^3$/s/m$^2$ at 20 hours, which was comparable to the SS41 steel. The ECB+DLC had smallest outgassing velocity of $1 \times 10^{-8}$ Pa·m$^3$/s/m$^2$ at 20 hours, which was one order of magnitude smaller than that of the SUS ECB and 60 times smaller than that of
Figure 4. Measured results of outgassing velocities of the black colored coatings (black lines). Dashed lines show outgassing velocities of several reference materials [5].
Figure 5. Outgas compositions of the UB-NiP after 20 hours pumping. (a): before baking (replotted from a chart record), (b): after baking.

Figure 6. Picture of the UB-NiP surface measured by (a) a scanning electron microscope and (b) a laser microscope.

the UB-NiP. However, its optical absorption coefficient in the condition of perpendicular beam injection onto the sample and non-polarization of light was only 65%. Therefore, we concluded that the UB-NiP is suitable as an optical dumper such that small area and high efficiency are required, and the ECB+DCL is effective for a large area coating such as the use for vacuum chamber walls.

Reference
[1] S. Kodama et al., IEEE Trance. Instrum. Meas., 39, Feb. (1990) 230.
[2] S. Kodato et al., Sensors and Actuators A, 28 (1991) 63.
[3] R. Takahashi et al., Vacuum, 73 (2004) 145.
[4] K. Terada et al., J. Vac. Sci. Technol. A7(3), (1989) 2397.
[5] T. Kubo et al., J. Vac. Soc. of Japan, 41, (1998) 217. (in Japanese)
[6] Y.Saito, Brazilian J. Vac. Applications, Vol.22 (2003) 39.