BL-3B/2016T002

XPS Study on the Thermal Stability of Oxygen-Free Pd/Ti Thin Film, a New Nonevaporable Getter (NEG) Coating

Tetsuya Miyazawa[1], Masashi Kurihara[2], Shinya Ohno[2], Takashi Kikuchi[3], and Kazuhiko Mase[1,3,*]
[1]SOKENDAI, 1-1 Oho, Tsukuba, Ibaraki 305-0801, Japan
[2]Yokohama National University, Yokohama, Kanagawa 240-8501, Japan
[3]Photon Factory, Institute of Materials Structure Science, High Energy Research Organization (KEK), 1-1 Oho, Tsukuba, 305-0801, Japan

1 Introduction

Nonevaporable getter (NEG) coating is a technique to coat the inner walls of vacuum chambers with NEG thin films that can be activated through baking under ultrahigh vacuum (UHV). The activated NEG films suppress outgassing from the chambers and evacuate residual gasses, and consequently maintain UHV in the range 10^{-8} Pa with small ion pumps. The most widely-used NEG coating is deposited TiZrV films that can be fully activated after baking at 180 °C for 24 h [1, 2]. However, NEG coatings have rarely been applied to beamlines and endstations in synchrotron radiation facilities. To expand the scope of NEG coating applications, we developed a new NEG coating using oxygen-free Pd/Ti film deposition [3,4]. In this paper we report thermal stability of oxygen-free Pd/Ti investigated by using X-ray photoelectron spectroscopy (XPS) [5].

2 Experiment

The oxygen-free-Pd/Ti was deposited on SS304L substrates under clean UHV conditions. XPS measurements were carried out using *p*-polarized SR with a photon energy (*hv*) of 110 eV and ARPES II (VSW, with HA54) at BL-3B of Photon Factory. The sample was irradiated at an incidence angle of 45° from the surface normal, and photoelectrons emitted in the surface normal direction were detected. The sample was annealed at 100, 120, 140, 160, 180, 200, 220, 240, 260, 280, 300, 320, 340, or 360 °C for 5–10 min by electron bombardment.

3 Results and Discussion

Figure 1 shows wide-scan XPS spectra of the oxygen-free Pd/Ti film as a function of annealing temperature in the range 100–360 °C. The Fermi level was taken as the origin of the binding energy (*BE*) axis. At room temperature, the Pd $4p_{1/2}$ and $4p_{3/2}$ peaks were observed at *BE*s of 51.1 and 49.4 eV, respectively, as shown in Fig. 2a. The Ti 3*s* and 3*p* peaks were not observed, as shown in Figs. 2a and 2b. This result indicates that the Pd film completely overcoated the Ti film. The Ti 3*p* peak appeared at *BE* of 33.9 eV when the sample was annealed at 280 °C, as shown in Fig. 2b. We assigned this Ti 3*p* peak to Ti^{1+}, because *BE* of Ti (Ti^0) was reported as 32.5 eV [6], while *BE*s of Ti_2O_3 (Ti^{3+}) and TiO_2 (Ti^{4+}) were reported as 36.2 eV [7] and 37.5 eV [8], respectively. The O 2*p* peak was not clear at 280 °C, but clearly appeared at *BE* of 9.3

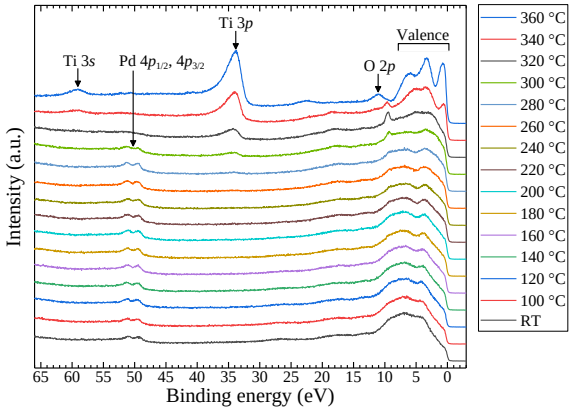

Fig. 1: Wide-scan XPS spectra of the oxygen-free Pd/Ti film. Reproduced from Ref. 5, with the permission of AIP Publishing.

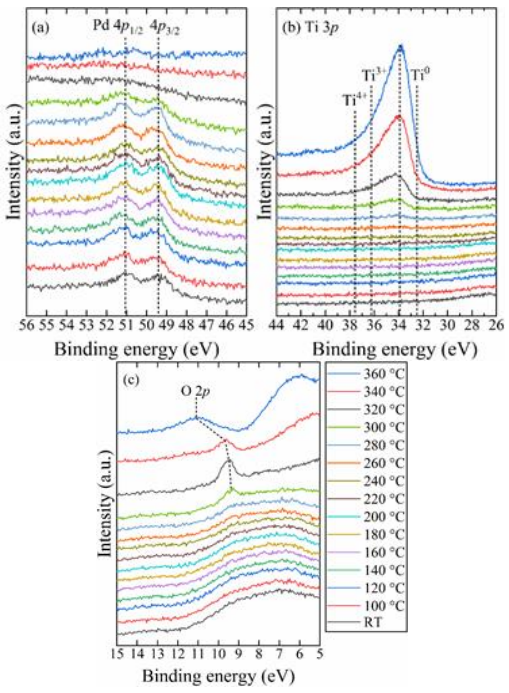

Fig. 2: Enlarged XPS spectra in the region of (a) Pd $4p_{1/2}$ (*BE* = 51.1 eV) and Pd $4p_{3/2}$ (*BE* = 49.4 eV), (b) Ti 3*p* (*BE* = 33.9 eV), and (c) O 2*p* (*BE* = 9.3–11.1 eV) peaks of the oxygen-free Pd/Ti film. Reproduced from Ref. 5, with the permission of AIP Publishing.

eV at 300 °C as shown in Fig. 2c. Because the vapor pressure of Pd is negligible in this temperature region [9], the results indicate that Ti segregates to the surface at 280–300 °C and forms Ti^{1+} such as Ti–OH and Ti_2O. On the other hand, the intensities of the Pd $4p_{1/2}$ and $4p_{3/2}$ peaks decreased at 300 °C and became negligible at temperatures greater than 320 °C, as shown in Fig. 2a. The Ti 3*p* and O 2*p* peaks grew at above 300 °C, as shown in Figs. 2b and 2c. These results indicate that Ti segregates to the surface and oxidizes forming Ti^{1+} at above 280–300 °C. Therefore, the maximum allowable temperature of oxygen-free Pd/Ti films is approximately 260 °C. This temperature has almost no limitations in its use in SR facilities because the maximum allowable temperature of beamlines and endstations is usually less than 260 °C.

Acknowledgement

The authors are grateful to Dr. Kenichi Ozawa (Tokyo Institute of Technology), Mr. Hiromu Nishiguchi (Baroque International Inc.), and Ms. Eriko Kazama (Baroque International Inc.) for their invaluable advice and support. This research was partly supported by a Grant-in-Aid for scientific research (JSPS KAKENHI Grant Number JP17K05067), TIA-Kakehashi grants (TK17-013 and TK18-014), and the Matching Planner Program from Japan Science and Technology Agency, JST.

References

[1] C. Benvenuti *et al*., *J. Vac. Sci. Technol. A* **16,** 148 (1998).
[2] C. Benvenuti *et al*., *Vacuum* **50,** 57 (1998).
[3] T. Miyazawa *et al*., *Vac. Surf. Sci.* **61,** 227 (2018).
[4] T. Miyazawa *et al*., *J. Vac. Sci. Technol. A* **36,** 051601 (2018).
[5] T. Miyazawa *et al*., *AIP Conf. Proc.* **2054,** 060045 (2019)
[6] A. Lebugle *et al*., *Physica Scripta* **23,** 825 (1981).
[7] F. Werfel and O. Brümmer, *Physica Scripta* **28,** 92 (1983).
[8] F. Lange *et al*., *J. Electron Spectrosc. Relat. Phenom.* **57,** 307 (1991).
[9] R. E. Honig and D. A. Kramer, *RCA Rev.* **30,** 285–305 (1969).

* mase@post.kek.jp