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# Oxygen-Free Palladium/Titanium Coating, a Novel Nonevaporable Getter Coating with an Activation Temperature of 133 °C

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## 1 Introduction

Nonevaporable getter (NEG) pumps are ideal for maintaining an ultra-high vacuum (UHV) owing to their high pumping speed for active residual gases (particularly hydrogen). NEG coating is a technique to coat the inner walls of vacuum chambers with NEG thin films that can be activated through baking under UHV [1, 2]. We recently developed a new NEG coating with an activation temperature of 185 °C using Ti vacuum sublimation followed by Pd vacuum sublimation (low-oxygen-content Pd/Ti coating) [3]. In the present paper, we report Pd/Ti coating with extremely low oxygen concentration (oxygen-free Pd/Ti coating) with an activation temperature as low as 133 °C [4].

## 2 Experiment

The apparatus used for the preparation of oxygen-free Pd/Ti samples for scanning transmission electron microscope (STEM), scanning electron microscope (SEM), and transmission electron microscope (TEM), and X-ray photoelectron spectroscopy (XPS) measurements is shown in Fig. 1. The apparatus used for the oxygen-free Pd/Ti coating for SS316L formed bellows is shown in Fig. 2. The oxygen-free Pd/Ti film was deposited on the SS304L substrates or inner walls of the formed bellows under clean UHV conditions. The concentration of oxygen in the Pd/Ti film was estimated to be less than 0.05%.

The apparatus used to measure pressure curves of oxygen-free-Pd/Ti-coated formed bellows is shown in Fig. 3. The UHV chamber is pumped with a TMP through a manual UHV gate valve. A B-A gauge is installed to measure the pressure in the UHV chamber. The oxygen-free-Pd/Ti-coated bellows, a nipple made of 0.2%-BeCu alloy, and a B-A gauge are connected to the UHV chamber via an all-metal valve. This system was pumped, and then successively baked at 133 °C for 12 h, 176 °C for 3.5 h, and 200 °C for 3.5 h. After each baking the pressure curves are measured with the B-A gauge.

## 3 Results and Discussion

STEM, SEM, and TEM images of oxygen-free Pd/Ti are shown in Figs. 4, 5, and 6, respectively. The Ti and the Pd films were almost uniform, and the Ti film was completely overcoated by the Pd film. The thicknesses of the Pd and Ti thin films were approximately 50 nm and 1.3 μm, respectively. The Pd/Ti surface had an uneven structure with irregularities of several tens to several hundreds of nanometers. XPS spectra of oxygen-free Ti and Pd/Ti films are shown in Fig. 7. Ti and oxygen were negligible on the Pd/Ti surface.

Figure 8 shows the summarized pressure curves after baking at 133 °C for 12 h, 176 °C for 3.5 h, and 200 °C for 3.5 h. After closing the all-metal valve, the pressure in the system reached values of $4.6 \times 10^{-6}$ Pa, $1.7 \times 10^{-7}$ Pa, and $6.1 \times 10^{-8}$ Pa, respectively. The pumping speeds of the coated bellows after baking at a maximum temperature of 133 °C for 12 h, 176 °C for 3.5 h, and 200 °C for 3.5 h were estimated to be 0.028, 0.23, and 0.23 L $s^{-1}$, respectively. The pumping speed did not decrease even after the oxygen-free-Pd/Ti-coated bellows were vented. The results indicated that the Pd film prevented the oxidation of the Ti film during venting.

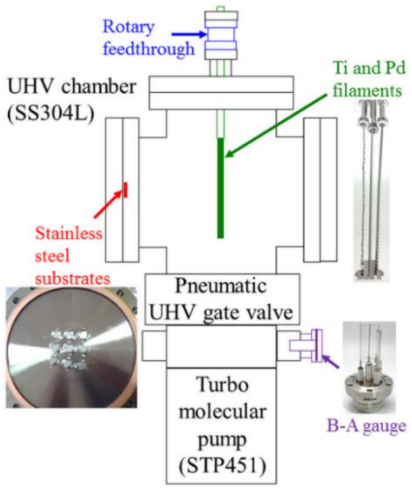


Fig. 1: Schematic of oxygen-free Pd/Ti and Ti coating apparatus for STEM, SEM, TEM, and XPS samples. Reproduced from Ref. 4, with the permission of AIP Publishing.

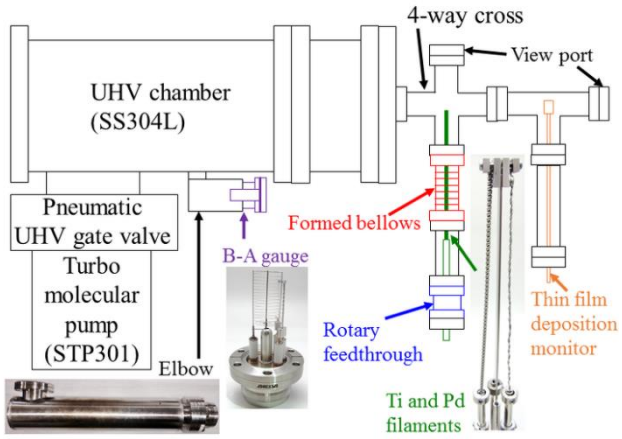


Fig. 2: Schematic of oxygen-free Pd/Ti coating apparatus for formed bellows. Reproduced from Ref. 4, with the permission of AIP Publishing.

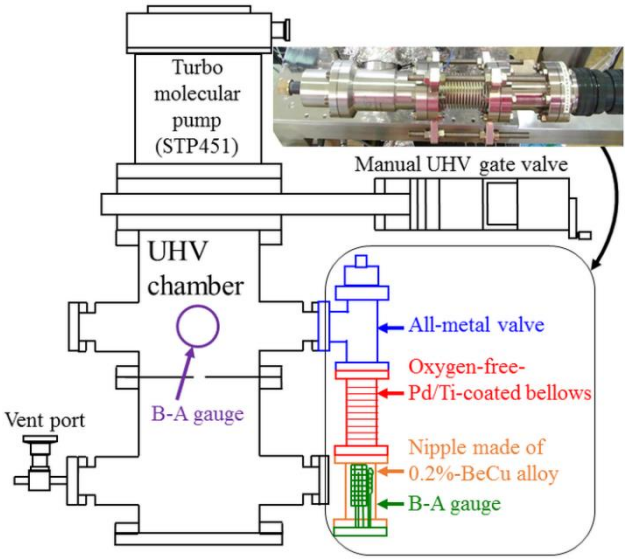


Fig. 3: Schematic of apparatus used to measure pressure curves of oxygen-free-Pd/Ti-coated bellows. Reproduced from Ref. 4, with the permission of AIP Publishing.

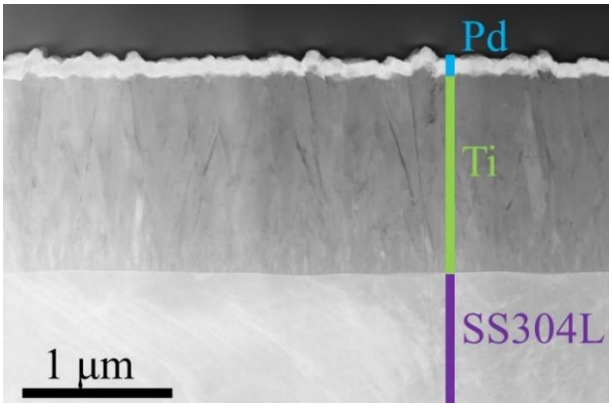


Fig. 4: STEM dark-field image of cross section of oxygen-free Pd/Ti film. Reproduced from Ref. 4, with the permission of AIP Publishing.

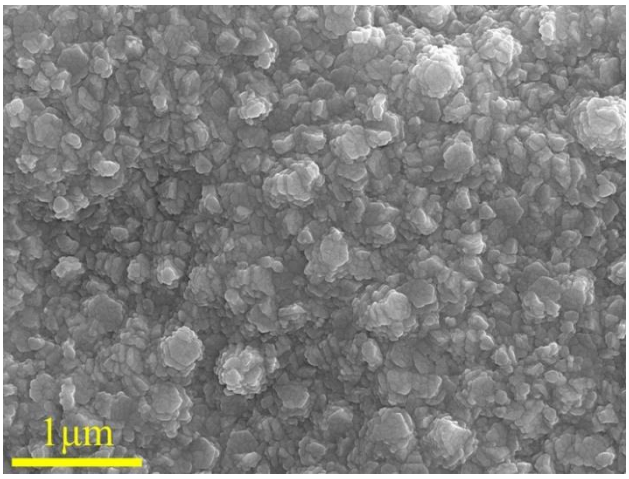


Fig. 5: SEM image of surface of oxygen-free Pd/Ti film. Reproduced from Ref. 4, with the permission of AIP Publishing.

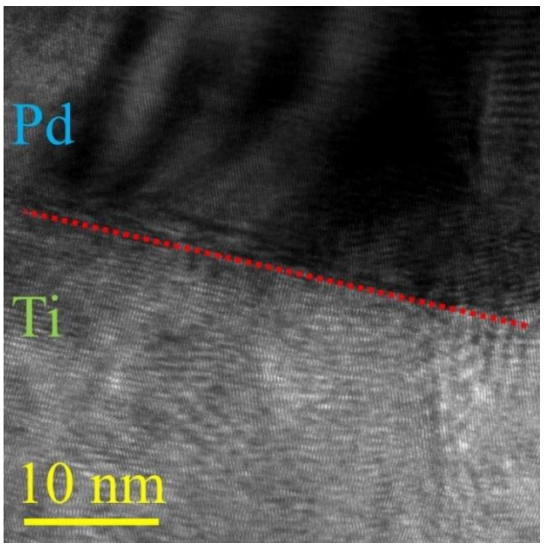


Fig. 6: TEM bright-field image of interface between Ti and Pd films of oxygen-free Pd/Ti. Reproduced from Ref. 4, with the permission of AIP Publishing.

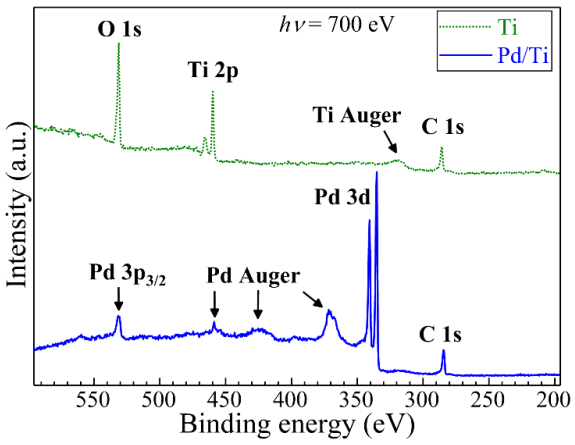


Fig. 7: XPS spectra of oxygen-free Ti and Pd/Ti films. Reproduced from Ref. 4, with the permission of AIP Publishing.

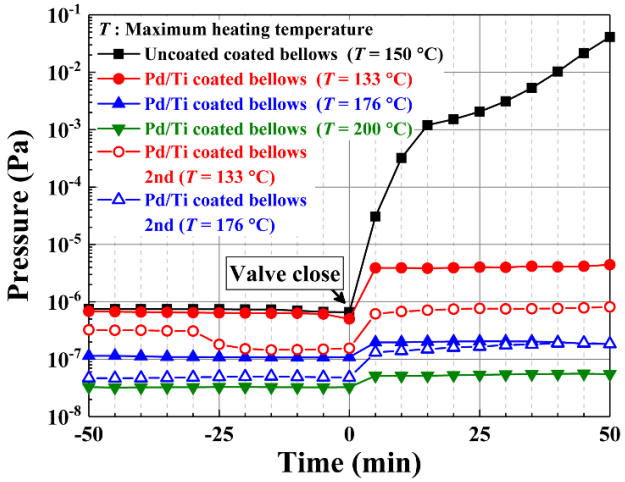


Fig. 8: Pressure curves of uncoated bellows and oxygen-free-Pd/Ti-coated bellows. Reproduced from Ref. 4, with the permission of AIP Publishing.

The advantages of the oxygen-free Pd/Ti coating are as follows: 1) it can be activated by baking at a maximum temperature of 133 °C for 12 h, 2) it is durable against repeated venting-baking cycles in principle, 3) it is economical, 4) it does not require skilled technicians, and 5) it can be applied to large chambers with a complicated structure. Therefore, the targets of this oxygen-free Pd/Ti coating will be expanded to include vacuum systems with Viton® O-rings.

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