

Development of a New Nonevaporable Getter (NEG) Named Oxygen-Free Palladium/Titanium; Surface Analysis by Synchrotron Radiation X-Ray Photoelectron Spectroscopy and Application for NEG Pump

We have developed a novel nonevaporable getter named oxygen-free Pd/Ti. After activation at 133°C, oxygen-free Pd/Ti evacuates H₂ and CO. Its pumping speeds do not decrease even after repeated venting–activating cycles. Surface analysis by synchrotron radiation X-ray photoelectron spectroscopy showed that carbon contamination decreased to an extent on heating in ultra-high vacuum, but decreased considerably on heating under an O₂ pressure. The pumping speeds of the oxygen-free Pd/Ti coated chamber improved remarkably after O₂ baking. The new NEG pump using oxygen-free Pd/Ti was commercialized in March 2019.

A nonevaporable getter (NEG) is a material that evacuates active residual gases when activated under clean ultra-high vacuum (UHV) conditions. Typical NEG materials are Al, Ti, V, Zr, Fe, and their alloys. Pumps using NEG (NEG pumps) are widely used in UHV apparatus because of their high pumping speeds for H₂ and active gases in the UHV region. However, the conventional NEG pump has the following drawbacks: 1) its pumping speeds decrease after repeated venting–activating cycles; 2) it requires relatively high activation temperature (typically 350°C for 24 h when ZrVFe alloy is used [1], and typically 180°C for 24 h when TiZrV film is used [2]); and 3) it requires a dedicated power supply and current feedthroughs. In order to overcome these disadvantages, we developed a new NEG with an activation temperature of 133°C using Ti sublimation followed by Pd sublimation under UHV, and named it oxygen-free Pd/Ti [3]. The activation and pumping mechanisms of oxygen-free Pd/Ti are shown in Fig. 1 [4]. During the activation of oxygen-free Pd/Ti, hydrogen atoms diffuse from the Ti film through the Pd overcoat to the surface and desorb as H₂ into vacuum leaving oxygen-free Pd/Ti available to H₂ adsorption. In the same activation process, CO desorbs from the Pd surface making the Pd surface available for CO adsorption. Therefore, hydrogen diffusion and desorption as well as CO desorption determine the activation temperature of oxygen-free Pd/Ti, which is as low as 133°C.

When oxygen-free Pd/Ti is exposed to air, the Pd

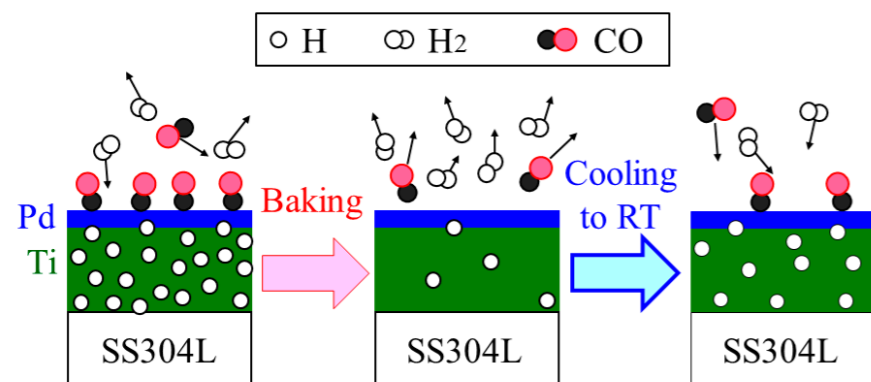


Figure 1: Schematic of the activation and pumping mechanisms of oxygen-free Pd/Ti coated on SS304L. Reproduced from Ref. 4, with the permission of AIP Publishing.

overcoat prevents the Ti thin film from oxidation, but the Pd surface becomes contaminated to some degree with carbon [3]. This contamination is supposed to reduce the pumping speeds of oxygen-free Pd/Ti for H₂ and CO [4]. To find an effective and simple method for removing carbon contamination, we investigated oxygen-free Pd/Ti samples heated in an UHV or under an O₂ pressure of 1.3×10^{-4} Pa by synchrotron radiation X-ray photoelectron spectroscopy (SR-XPS) at BL-13B [5]. The Pd 3d_{5/2} SR-XPS spectra shown in Fig. 2(a) suggested that the number of less-coordinated Pd atoms decreases after heating. This result is consistent with the secondary electron microscopy (SEM) images of the unheated and UHV-heated oxygen-free Pd/Ti samples [5]. On the other hand, the deconvoluted C 1s peaks indicated that the carbon contamination is mainly graphene and molecules containing carbon as shown in Fig. 2(b). The graphene is supposed to be generated by catalytic chemical reaction from hydrocarbons adsorbed on Pd. The graphene coverage of the unheated sample was estimated to be 0.9 monolayer (ML), whereas those of the UHV-heated and O₂-heated samples were estimated to be 0.3 and 0.04 ML, respectively. The pumping speeds of the oxygen-free Pd/Ti coated chamber for H₂ and CO were measured by using the orifice method, and were found to be improved after O₂ baking as shown in Fig. 3. Therefore, we concluded that removal of carbon contamination on oxygen-free Pd/Ti improves the pumping speeds for H₂ and CO.

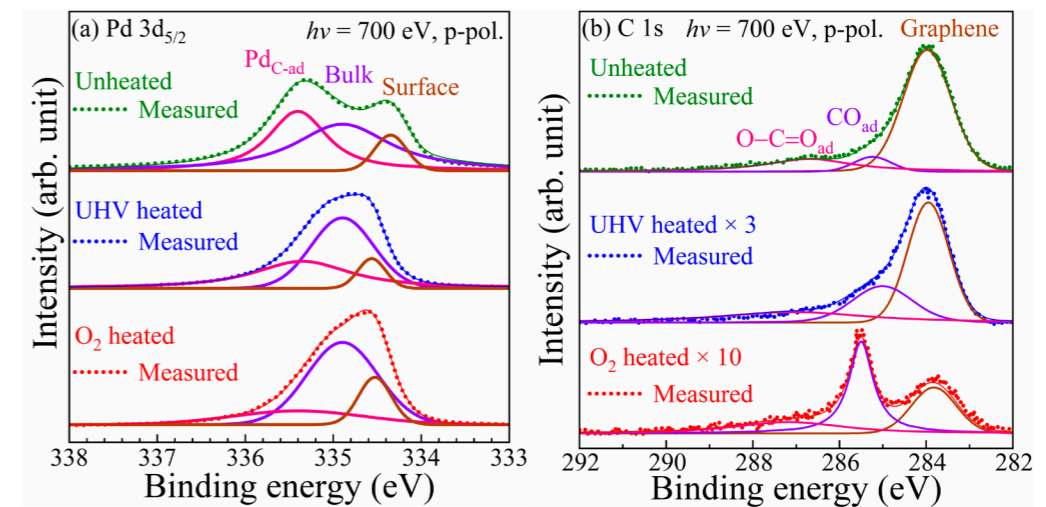


Figure 2: Measured, fitted, and deconvoluted peaks of (a) Pd 3d_{5/2} and (b) C 1s. Reproduced from Ref. [5], with the permission of AIP Publishing.

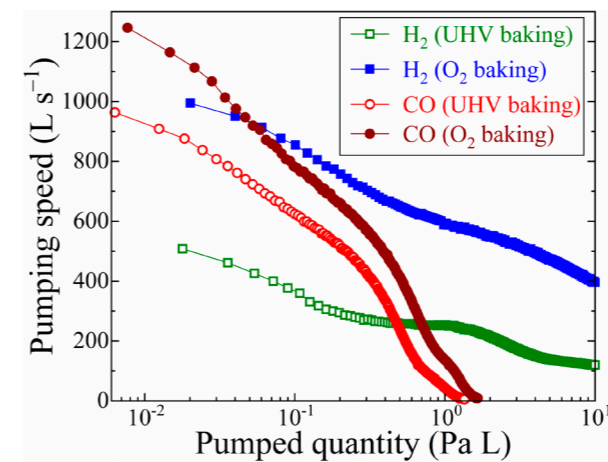


Figure 3: Measured pumping speeds of the oxygen-free Pd/Ti coated chamber with an inner diameter of 147 mm and inner length of 236 mm for H₂ or CO after UHV or O₂ baking at 150°C for 12 hours. Reproduced from Ref. 5, with the permission of AIP Publishing.

The new NEG pump using oxygen-free Pd/Ti was commercialized in March 2019. The advantages of the new NEG pump are as follows: 1) it can be activated by baking at 150°C for 6 h, and pumps H₂ and CO efficiently; 2) its pumping speeds do not decrease even after repeated venting–activating cycles; 3) there is no need for a dedicated power supply or current feedthroughs; and 4) the pumping speeds can be improved by O₂ baking. A comparison of the commercially-available ICF203 NEG pump [6] and the present new ICF203 NEG pump [7] is summarized in Table 1. Since we were able to quickly feed back the results obtained by SR-XPS to the oxygen-free Pd/Ti development, we could commercialize the new NEG pump in one year from publication of the first paper on oxygen-free Pd/Ti [3]. This is a good example of how the SR facility is making a major contribution to the research and development of new functional materials and products using them.

Table 1: Comparison of commercially-available ICF203 NEG pump [6] and the present new ICF203 NEG pump [7]

	Capaci Torr® D 2000 [6]	Present NEG pump [7]
Activation conditions	450°C, 10 min	150°C, 12 h
Gases that can be pumped	H ₂ , H ₂ O, O ₂ , N ₂ , CO, CO ₂	H ₂ and CO
Initial pumping speed for H ₂	2,000 L/s	2,200 L/s
Initial pumping speed for CO	1,000 L/s	1,500 L/s
Influence of repeated venting and activation cycles	Pumping speed decreases	Pumping speed does not decrease
Current feedthroughs	Necessary	Unnecessary
Dedicated power supply	Necessary	Unnecessary

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BEAMLINE

BL-13B

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