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ZERO-LENGTH CONFLAT FIN-TYPE NONEVAPORABLE GETTER PUMP COATED WITH OXYGEN-FREE PALLADIUM/TITANIUM

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Abstract

Nonevaporable getter (NEG) pumps are widely used in synchrotron radiation facilities because they are oil-free, vibration-free, space-saving, lightweight, and energy-saving. However, conventional NEG pumps have the following disadvantages: (1) a relatively high activation temperature (typically 300–450 ˚C for ZrVFe alloy); (2) the requirement for a dedicated power supply and electric feedthroughs; and (3) decreased pumping speeds after repeated cycles of activation and exposure to air. To overcome these disadvantages, we have developed a new zero-length conflat (CF) fin-type NEG pump with a DN 160 CF that uses oxygen-free Pd/Ti thin films as a new NEG material for evacuating residual H_2 and CO. The advantages of the new NEG pump are as follows: (1) it can be activated by baking at 150 ˚C; (2) no dedicated power supply or electric feedthrough is required; (3) pumping speeds do not degrade even after repeated cycles of activation and exposure to air; and (4) the unit is space-saving and lightweight.

INTRODUCTION

A nonevaporable getter (NEG) pump evacuates residual reactive gases at room temperature after it has been activated under clean ultrahigh vacuum (UHV) conditions [1-3]. However, the activation temperature of conventional NEG pumps is relatively high (typically 300-450 ˚C for ZrVFe alloy). In 1997, Benvenuti *et al.* proposed the idea of depositing a NEG thin film on the inner walls of a vacuum chamber to achieve a UHV after baking [4-6]. They named this method 'NEG coating'. A thin film of TiZr, deposited by direct-current (DC) magnetron sputtering, with an activation temperature of 250 ˚C, was proposed for use as the NEG coating [4–6]. Shortly thereafter, it was reported that TiZrV thin films deposited by DC magnetron sputtering could be activated by baking at 180 ˚C for 24 hours [7, 8]. This TiZrV coating were used with great success at CERN and has now been adopted in accelerator facilities around the world [9].

Another disadvantage of a conventional NEG is that repeated activation and exposure to air results in oxidation of the surface, degrading its pumping performance. To overcome this disadvantage, a method was developed involving the sequential deposition of TiZr or TiZrV and Pd by DC magnetron sputtering (Pd/TiZr or Pd/TiZrV) [10-13]. Pd surface is capable of dissociating H_2 molecules into their constituent H atoms at room temperature, and the resulting H atoms diffuse into the bulk of the Pd [14]. Pd surface is also capable of chemisorbing CO at room temperature [15]. Consequently, Pd/TiZr and Pd/TiZrV can evacuate H_2 and CO at room temperature after activation. Because Pd surface does not readily oxidize, its pumping performance should not deteriorate after repeated activation and exposure to air. In fact, Mura, and Paolini reported that Pd/TiZrV is activated by baking at 150 ˚C and that it pumps H_2 and CO at room temperature; moreover, the pumping speed for H_2 does not decrease even after 30 cycles of activation and exposure to air [12].

Recently Mase *et al.* developed a new method for depositing a Pd-overcoated NEG thin film without using sputtering [16, 17]. Their NEG was fabricated by sequential sublimation of Ti and Pd under UHV at 10^{-7} to 10^{-8} Pa. This new NEG was named 'oxygen-free Pd/Ti' because its oxygen content was estimated to be less than 0.05% [17]. Kikuchi *et al.* developed a prototype of a NEG pump with a DN 160 conflat (CF) flange that used oxygen-free Pd/Ti (Fig. 1) [18]. Here, we report the development of a zero-length CF fin-type NEG pump [19]. This NEG pump can be easily fabricated by using a ring to which fins are attached. The pumping speeds of the NEG pump for H_2 and CO were measured by the orifice method [20].

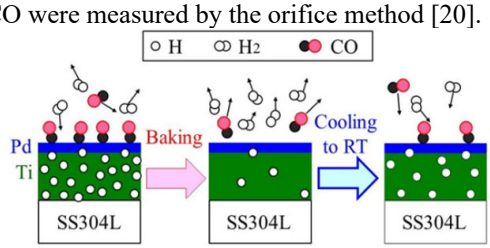

Figure 1: Schematic showing the activation and pumping mechanisms of oxygen-free Pd/Ti deposited on stainless-steel SS304L. Reproduced from Ref. [18].

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EXPERIMENTS

The vacuum vessel for the zero-length CF fin-type NEG pump (Fig. 2) consisted of a ring to which were attached 23 parallel fins (Fig. 3) and a DN 160 CF flange with a cylindrical space. Each fin was 0.2 mm thick and all the parts consisted of stainless steel 316L (SS316L). The vacuum vessel was placed on the side port of an apparatus for oxygen-free Pd/Ti deposition [18]. The Pd and Ti evaporator contained Pd and Ti filaments (Fig. 4). The fins were oriented perpendicularly to the Pd and Ti filaments, so that both sides of each fin were coated with oxygen-free Pd/Ti. H_2 and CO molecules entering between the fins are repeatedly adsorbed and desorbed in the spaces enclosed by the fins and the bottom, thereby increasing the probability of their sorption by the oxygen-free Pd/Ti thin film.

The oxygen-free Pd/Ti thin film was deposited on the vacuum vessel by the following procedure. The apparatus for oxygen-free Pd/Ti deposition was baked at 150 °C for 24 h. At the end of the baking period, the Ti and Pd filaments were degassed through heating by a direct current of 25 A for one hour for the former and at 19.5 A for 40 minutes for the latter. When the UHV chamber was cooled to room temperature, the pressure reached 1.7×10^{-8} Pa. The Ti filament was sublimated at about 47.5 A for three hours, and then the Pd filament was sublimated at about 33.0 A for five hours.

Pumping speeds of the NEG pump for H_2 and CO were measured by the orifice method [20]. Details of the pumping-speed measurements were similar to those reported in Ref. 18.

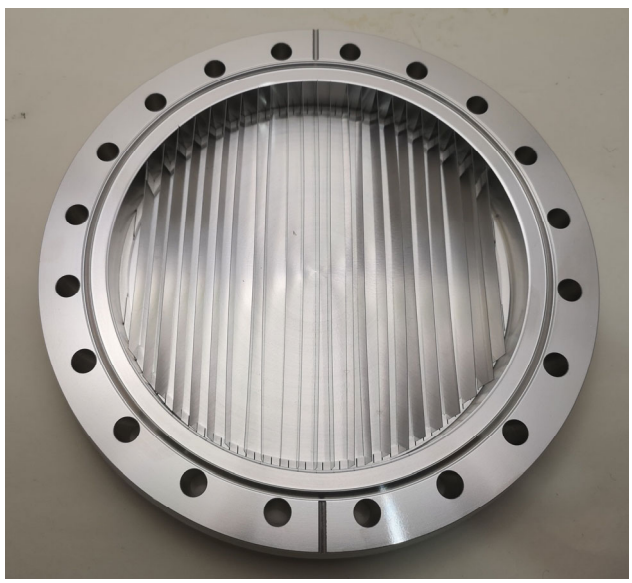

Figure 2: The vacuum vessel for the zero-length CF fin-type NEG pump before oxygen-free Pd/Ti deposition.

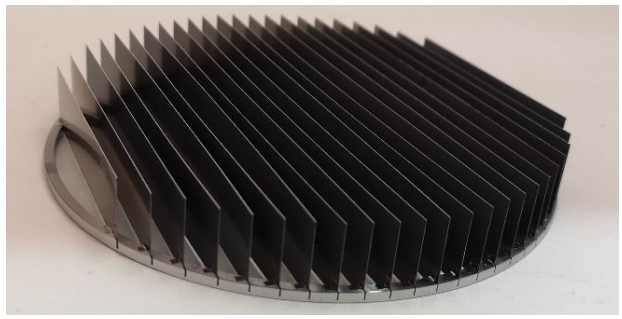

Figure 3: Ring with fins for the zero-length CF fin-type NEG pump before deposition of oxygen-free Pd/Ti. Reproduced from Ref. 19, with the permission of the Particle Accelerator Society of Japan (PASJ).

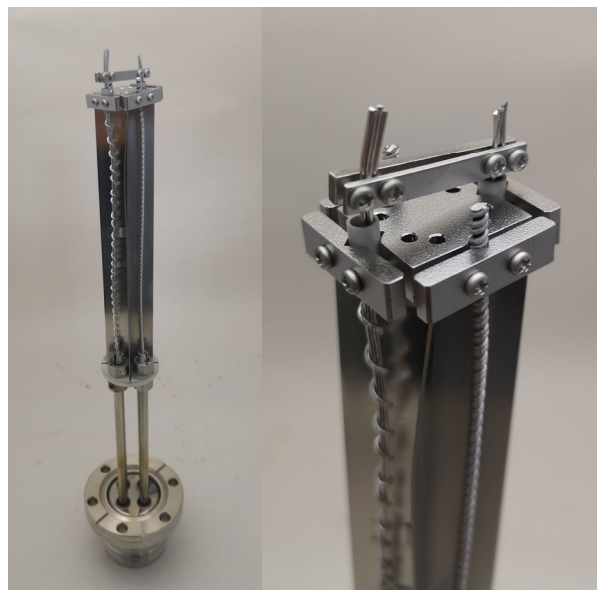

Figure 4: The Pd and Ti evaporator.

RESULTS AND DISCUSSION

The morphologies of oxygen-free Pd/Ti thin films on the fins and the bottom were examined by scanning electron microscopy, scanning transmission electron microscopy, and energy-dispersive X-ray spectroscopy. The Ti thin film was completely coated with Pd on the bottom, whereas the fins were covered by oxygen-free Pd/Ti nanostructures.

Figures 5 and 6 show the measured pumping speeds of the zero-length CF fin-type NEG pump for H_2 as a function of the pumped quantity after baking at 150 °C for 12 hours. The pumping speeds for H_2 were estimated to be about 2800, 1500, and 750 L s^{-1} for pumped quantities of 3×10^{-4}, 3×10^{-3}, and 10 Pa L, respectively. The pumping speed for H_2 decreased rapidly at about 200 Pa L. This suggests that saturation of the oxygen-free Pd/Ti nanostructures on the fins as a result of H sorption occurred when 200 Pa L of H_2 was absorbed. Figure 7 shows the measured pumping speeds of the zero-length CF fin-type NEG pump for CO after baking at 150 °C for 12 hours. The pumping speed for CO at a pumped quantity of 1×10^{-3} Pa L was about 1550 L s^{-1}. The pumping speed for CO dropped to almost zero at 20 Pa L. This result

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suggests that the oxygen-free Pd/Ti surface was almost completely covered with CO when 20 Pa L of CO was adsorbed.

This technology has been transferred to Baroque International Inc. and Irie Koken Co., Ltd.; as a result, more-sophisticated zero-length CF fin-type NEG pumps with superior pumping properties have become commercially available [21].

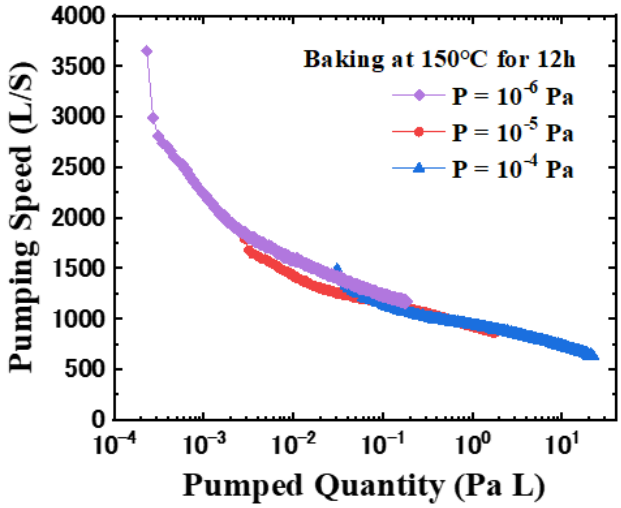

Figure 5: Pumping speeds of the zero-length CF fin-type NEG pump in various introduction pressures (P) of H_2 after baking at 150 °C for 12 hours. Reproduced from Ref. 19, with the permission of the PASJ.

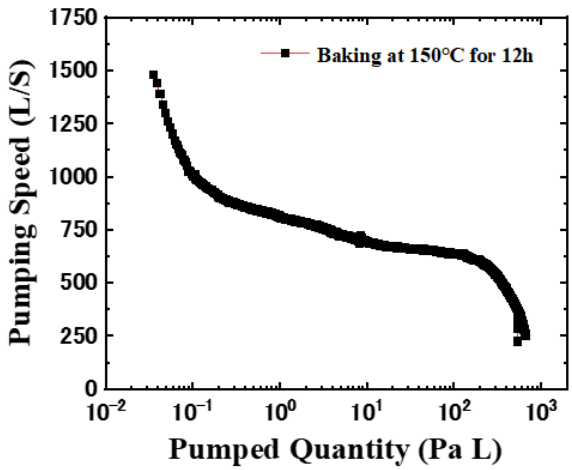

Figure 6: Pumping speeds of the zero-length CF fin-type NEG pump for H_2 after baking at 150 °C for 12 h, when the H_2 introduction pressure was 1×10^{-4} Pa.

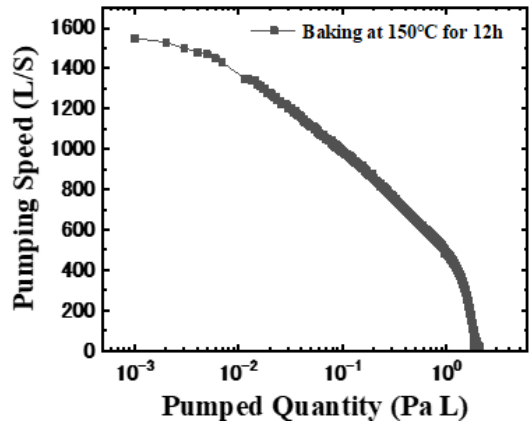

Figure 7: Pumping speeds of the zero-length CF fin-type NEG pump for CO after baking at 150 °C for 12 h The CO introduction pressure was 1×10^{-4} Pa. Reproduced from Ref. 19 with the permission of the PASJ.

CONCLUSION

We have developed a zero-length CF flange fin-type NEG pump and have evaluated its pumping speeds for H_2 and CO. The pumping speed for H_2 at a pumped quantity of 3×10^{-3} Pa L was estimated to be about 1500 L s^{-1}, whereas the pumping speed for CO at a pumped quantity of 3×10^{-3} Pa L was estimated to be about 1550 L s^{-1}. This NEG pump can be used for accelerators, beamlines, and endstations in SR facilities because it can be activated by baking at 150 °C for 12 hours. Research is being carried out to improve the pumping speeds further. More-sophisticated zero-length CF fin-type NEG pumps have since become commercially available [21].

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