ISBN: 978-3-95450-214-1 ISSN: 2673-5490 doi:10.18429/JACoW-IPAC2021-WEPAB337

SOME METHODS OF MAKING TITANIUM VACUUM CHAMBER ACT AS GETTER PUMP FOR UHV/XHV

J. Kamiya†, K. Takano, H. Yuza
Japan Atomic Energy Agency/J-PARC, Tokai, Naka, Ibaraki, Japan
K. Wada, Tokyo Electronics Co. LTD., Kokubunji, Tokyo, Japan

Abstract

In high-power beam accelerators, such as J-PARC, titanium has been used as the beam pipe and bellows material due to its low radioactivation characteristics and low outgassing rate. Titanium is also known as a getter material that adsorbs or absorbs the residual gas molecules in the vacuum chamber. However, the ordinal titanium surface has no getter function because it is covered with a titanium oxide film. If the surface oxide film is removed, the titanium vacuum chamber itself could act as a getter pump like a NEG-coated chamber. Some methods, such as baking or sputtering, for removing the surface oxide film have been investigated. The dependence of the surface chemical composition and the getter characteristics on the baking temperature has been measured. Also, by sputtering the inner surface of the titanium chamber, clear evidence that shows the chamber acts as a vacuum pump has been obtained. Furthermore, the NEG coating on the pure titanium surface can suppress the rapid decrease of the sticking probability by the repeated air purge and reactivation.

INTRODUCTION

The non-evaporable getter (NEG) coating, which has been developed in CERN, is a sophisticated technique that can make a beam pipe act as a UHV/XHV pump by coating the getter materials with the ability to adsorb/absorb gas molecules on the beam pipe surface [1, 2]. Alloys of titanium, zirconium, and vanadium have been selected as coating materials [1]. In this technique, the NEG coating can be activated by a low baking temperature such as 180 ℃. The NEG material sticks O_2, CO, etc. by forming the oxidized or carbonized NEG material on the surface. When the NEG is activated by baking, such oxide or carbide diffuses in the NEG film and the surface becomes fresh NEG material. The sticking probability on the NEG decreases when the ratio of the diffused impurity, i.e. the oxide or carbide, in the NEG coating increases by the repeated surface oxidation/carbonization and reactivations. In such a case, the baking temperature for reactivation should be higher, for example up to 300 ℃, to regain the initial sticking probability. Recently, a new NEG coating, which consists of oxygen-free palladium/titanium coating, has been developed to avoid the degradation of the sticking probability as well as to obtain lower activation temperature [3, 4]. When the palladium/titanium NEG coating is reactivated, the oxide or carbide on the palladium surface releases into the vacuum as O_2 or CO without diffusing into the coating. They reported the pumping speed for H_2 hasn't decreased after 6 times reactivation-venting cycles [3].

Titanium is used as the vacuum material especially in high-power beam accelerators due to its low radioactivation characteristic [5] and low outgassing rate [6]. For example, the beam pipes and bellows are made of titanium in J-PARC [7]. As described above, titanium is one of the getter materials. However, the ordinal titanium surface has no getter function because it is covered with titanium-oxide film. We've considered that the titanium vacuum chamber itself could become a getter pump like a NEG coated chamber if the surface oxide film is somehow removed. In this report, first, the effect of the baking temperature on the surface oxide film is evaluated. Next, the removal of the surface oxide film by the DC magnetron sputtering is performed and the getter characteristic is examined.

EFFECT OF BAKING

Oxide Profile Measurement by XPS

The depth profile of titanium oxide was measured by X-ray photoelectron spectroscopy (XPS) with Ar ion etching. Samples of pure titanium (JIS Class 2) with chemical polishing and pure water cleaning were prepared. Each sample size was 20 mm×10 mm. XPS Quantera SXM of ULVAC-PHI, Inc. was used for the measurement. The sample was irradiated with monochromatic Al Kα X-rays with a diameter of about 200 μm. Ar^+ ions with an acceleration voltage of 1 kV were induced for the sputtering. The sputtering rate was 2.5 nm/min as the SiO_2 equivalent value. At first, a sample is installed in the sample chamber and evacuated to around 1×10^{-5} Pa. Next, the sample was moved to the measuring chamber with the pressure of 3×10^{-7} Pa. Then, the depth profile was measured. After that, the sample was moved to the sample chamber and baking was performed for 22 hours. After the sample became the room temperature, it was again moved to the measuring chamber. Then, the depth profile after the baking was performed. The different spots of the same sample were measured before and after baking. All the process was performed in a vacuum. The baking was performed at 200, 300, 350, and 400 ℃.

Figure 1 shows the depth distribution of the O1s peak intensity. The representative data of a sample is plotted for the unbaked case because the data for the unbaked case was almost the same for each sample. The oxygen diffuses to the interior of the titanium bulk at a higher temperature. As a result, the oxygen on the surface is decreased. Figure 2 shows the photoelectron spectrum with the binding energy between 450 eV and 470 eV. The binding energy of the

† Junichiro.kamiya@j-parc.jp

ISBN: 978-3-95450-214-1 ISSN: 2673-5490 doi:10.18429/JACoW-IPAC2021-WEPAB337

photoelectron of $Ti2p_{3/2}$ and $Ti2p_{1/2}$ for pure titanium metal is 454 eV and 460 eV, respectively. The spectrum at bulk consists of peaks with these binding energies as shown in Fig. 2 (a). The binding energy of the photoelectron of $Ti2p_{3/2}$ and $Ti2p_{1/2}$ for titanium oxide (TiO_2) is 458.5 eV and 464.2 eV, respectively. The surface of the unbaked sample is consists of these peaks (Fig. 2 (b)). For higher baking temperature, the peak energy at the surface moved to the smaller binding energy (Fig. 2 (c-e)). The peak energy at the surface becomes almost the same as that in the bulk for the sample baked at 400 °C. These results suggest the surface oxide is diffused into the bulk and the pure titanium becomes exposed to the surface by raising the temperature.

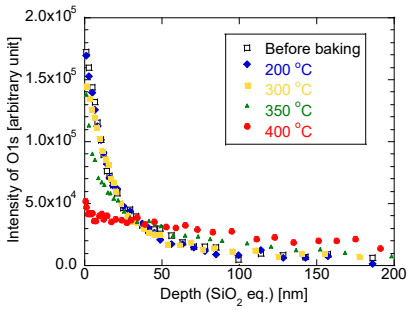

Figure 1: Depth distribution of O1s peak intensity for the samples without baking and baking at each temperature.

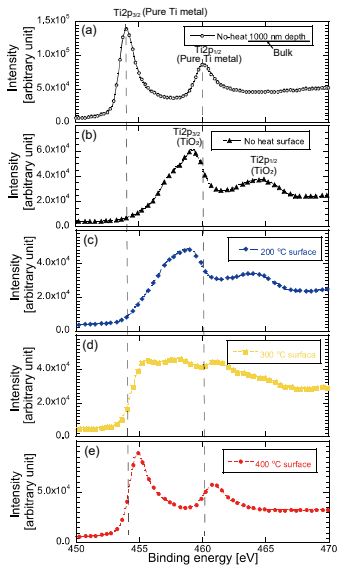

Figure 2: Photoelectron spectra of Ti2p. a) Bulk of the unbaked sample. b) Surface of the unbaked sample. c-e) Surface of the baked sample at 200, 300, and 400 °C.

Build-Up Test of a Titanium Vacuum Chamber

To examine whether a titanium vacuum chamber has a getter function by baking, the build-up test was performed. Figure 3 shows the setup. A titanium vacuum chamber is attached to the stainless steel (SUS) vacuum chamber, which is pumped by a turbomolecular pump (TMP), through a metal angle valve (MV). First, after the titanium chamber was evacuated by the TMP through the MV, the titanium chamber was baked at 200 °C for 3 hours. Then, the build-up test was performed at room temperature. After that, the chamber was baked at 300 °C. The same process was repeated to the build-up test after r the baking at 400 °C. In the build-up test, the total pressure and residual gas ion current were measured by an extractor gauge and a residual gas analyzer (RGA) for 2 hours in maximum. Figure 4 shows the result of the total pressure during the build-up. Figure 5 shows the ion current of each gas species after 1 hour build-up. Comparing with the SUS chamber for reference, the titanium chamber has partially been activated even by baking at 200 °C. The higher the baking temperature, the higher the degree of activation.

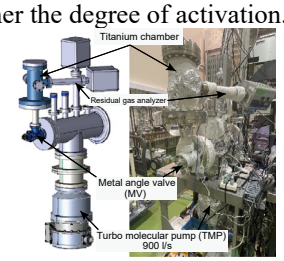

Figure 3: Setup of the build-up test for a titanium vacuum chamber.

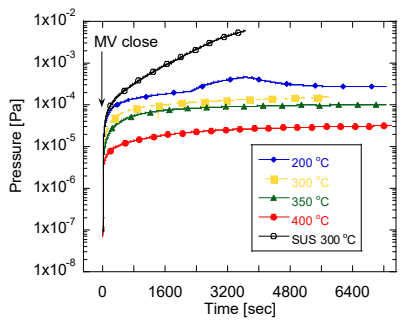

Figure 4: Build-up result of the total pressure after baking at each temperature. The result for the SUS chamber baked at 300 °C is also shown for comparison.

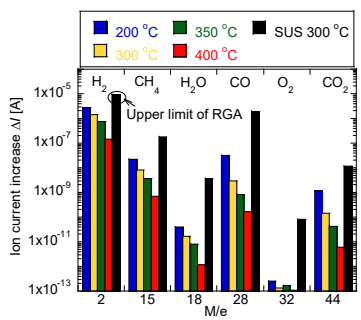

Figure 5: Ion current for each gas species after 1hour build-up. For the SUS chamber, the data after 40 min is plotted because the residual gas analyzer was stopped by the pressure interlock of RGA.

EFFECT OF SPUTTERING

Titanium Surface Oxide Removal

In most accelerator situations, the baking of the beam pipe at high temperatures has large risks, such as a leak and

ISBN: 978-3-95450-214-1 ISSN: 2673-5490 doi:10.18429/JACoW-IPAC2021-WEPAB337

breakage of the devices due to the heat expansion. This is the reason why the CERN has developed the NEG coating, which is activated at lower than 200 °C. Thus the sputtering has been verified as a method for removing a surface oxide film without rising the temperature. Figure 6 shows the experimental setup. A titanium chamber is connected with a SUS chamber through the orifice with a 5 mm diameter. The role of the orifice is both to limit the pumping speed and to compare the pumping ability with that of the TMP. A cylindrical electrode with a diameter of about 20 mm is inserted in the central axis of the titanium chamber. In the electrode, neodymium magnets are arranged to effectively make the plasma. Ar is used for sputtering gas. The voltage of +0.3 kV was applied in DC during the sputtering. The chamber was connected to the ground potential.

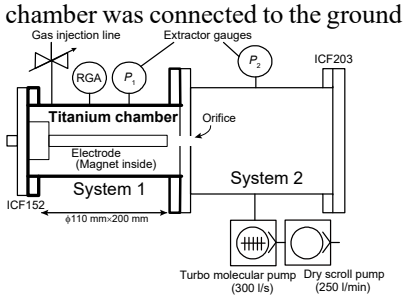

Figure 6: Setup for examining the surface oxide film removal by the sputtering. System 1 and 2 are connected with the orifice. Only system 2 is evacuated by the TMP.

Figure 7 shows the result of the pressure in the system 1 and 2. After the sputtering, the pressure of system 1 (P_1) has become smaller than that of system 2 (P_2). It means that the titanium chamber becomes a getter pump with a pumping speed larger than the TMP in system 2.

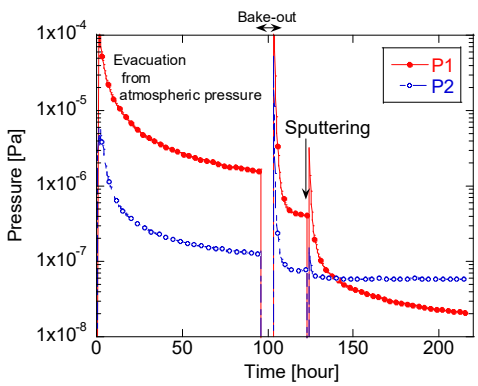

Figure 7: Effect of the surface titanium oxide film removal of the titanium chamber by the sputtering.

NEG Coating on the Pure Titanium

When the titanium chamber with a pure titanium surface is once purged by air, the surface immediately becomes oxidized and the getter function is lost. If the pure titanium surface is coated by the NEG, such as Ti-Zr-V, the NEG oxide which is formed on the surface by the air purge, could be partially diffused into the titanium bulk even at a low baking temperature such as 200 °C from the baking test results. Therefore the immediate decrease of the sicking probability by repeated saturations and reactivations could be suppressed. The experiment has been performed with a similar setup to Fig. 6. The electrode was covered by the NEG alloy (Ti-Zr-V) tube, which was electrically connected to the electrode. First, the titanium chamber surface was sputtered. Next, the NEG was coated on the titanium surface by sputtering the NEG electrode cover. In this stage, the electrode potential was changed to the minus to attract the Ar ions. After that, the electrode was removed and the repeated air purge and 200 °C baking were performed. Figure 8 shows the measured P_1 values. It is noticed that the lower pressure than that of the baked SUS chamber is achieved for the surface sputtered titanium chamber with NEG coating. Furthermore, the pressure keeps the constant value even after 13 times repeated air purge and reactivation. The NEG oxide may diffuse to the titanium bulk through the thin coating because there is no titanium oxide film barrier between the NEG coating and titanium bulk.

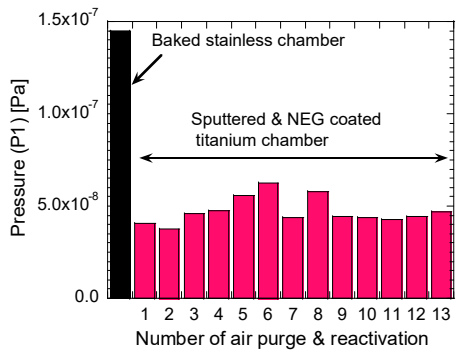

Figure 8: Pressure result after the repeated air purge and reactivation at 200 °C of the titanium chamber whose surface is coated by NEG after surface titanium oxide removal. The pressure of the stainless chamber baked at 200 °C is shown for comparison.

CONCLUSION

Methods for making a titanium chamber have a getter function have been examined. The XPS analysis shows the surface oxide diffuses into the bulk by the bakeout noticeably at higher than 300 °C. The buildup test showed that the titanium chamber had a getter function even at 200 °C baking. Both results showed that the surface oxide diffused deeper in the bulk at the higher temperature and pure titanium appears on the surface. This result is consistent with the measurement in CERN at the development stage of the NEG coating [8]. Surface oxide removal by sputtering is effective to make the titanium chamber act as a getter pump. The NEG coating on the pure titanium could suppress the rapid decrease of the sticking probability due to the increase of the diffused oxide in the thin coating because it could diffuse to the titanium bulk under the NEG coating.

ACKNOWLEDGEMENTS

The authors are grateful to Dr. Tanimoto of KEK for his technical support about NEG electrode. This work was supported by JSPS KAKENHI Grant Number JP18K11925.

ISBN: 978-3-95450-214-1 ISSN: 2673-5490 doi:10.18429/JACoW-IPAC2021-WEPAB337

REFERENCES

[1] C. Benvenuti *et al.*, "Vacuum properties of TiZrV non-evaporable getter films", *Vacuum*, vol. 60, pp. 57-65, 2001. doi:10.1016/S0042-207X(00)00246-3

[2] P. Chiggiato *et al.*, "Ti–Zr–V non-evaporable getter films: From development to large scale production for the Large Hadron Collider", *Thin Solid Films*, vol. 515, pp. 382-388, 2006. doi:10.1016/j.tsf.2005.12.218

[3] T. Miyazawa *et al.*, "Non-evaporable getter (NEG) coating using titanium and palladium vacuum sublimation", *Vacuum and Surface Science*, vol. 61, pp. 227-235, 2018. doi:10.1380/vss.61.227

[4] T. Miyazawa *et al.*, "Improved pumping speeds of oxygen-free palladium/titanium nonevaporable getter coatings and suppression of outgassing by baking under oxygen", *Journal of Vacuum Science & Technology A*, vol. 37, p. 021601, 2019. doi:10.1116/1.5074160

[5] J. Kamiya *et al.*, "Titanium alloy as a potential low radioactivation vacuum material", *Journal of Vacuum Science & Technology A*, vol. 33, p. 031605, 2015. doi:10.1116/1.4916934

[6] M. Minato *et al.*, "Vacuum characteristics of titanium", *Journal of Vacuum Science & Technology A*, vol. 13, pp. 540-544, 1995. doi:10.1116/1.579781

[7] N. Ogiwara, "Ultra high vacuum for high intensity Proton Accelerators", in *Proc. 2nd Int. Particle Accelerator Conf. (IPAC'11)*, San Sebastian, Spain, Sep. 2011, paper TUZB02, pp. 971-975.

[8] C. Benvenuti *et al.*, "A novel route to extreme vacua: the non-evaporable getter thin film coatings", *Vacuum*, vol. 53, pp. 219-225, 1999. doi:10.1016/S0042-207X(98)00377-7