EXPERIMENTAL STUDY OF STAINLESS STEEL VACUUM CHAMBER WITH TiN FILM COATING

Yong Wang*, Bo Zhang, Jianping Wang, Le Fan, Wei Wei, Yaofeng Zhang, Zuping Liu
National Synchrotron Radiation Laboratory, USTC, Hefei, Anhui 230029 China

Abstract

TiN coating has been widely applied in surface treatments of particle accelerator vacuum chambers because of its characteristics such as good electrical conductivity, stability of performance, ability to block hydrogen permeation, low SEY, etc.[1-3]. With DC sputtering, TiN film has been coated on the inner face of a stainless steel pipe vacuum chamber, 86 mm in diameter and 2300 mm in length. The vacuum performances testing of the coated chamber has also been done, including thermal outgassing rate measurement, Photon stimulated Desorption (PSD) measurement, and Secondary Electron Yield (SEY) measurement of samples. Compared with those of uncoated stainless steel chamber, the results show that coating TiN film is a very effective method of the treatment of particle accelerator vacuum chamber.

TIN (TITANIUM NITRIDE) COATING AND PARAMETERS OF THE FILM

TiN Coating

TiN film was coated by DC sputtering on the inner face of a stainless steel vacuum chamber which is 86 mm in diameter and 2300 mm in length, the same as the straight pipe chambers used in the storage ring in NSRL (National Synchrotron Radiation Laboratory). The schematic diagram of the coating setup is shown as follows:

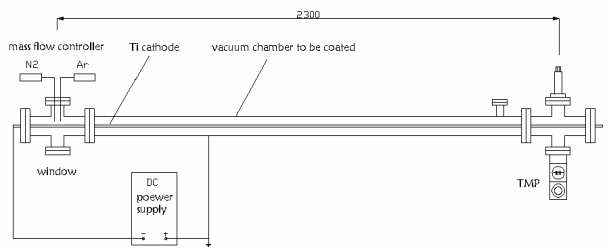

Figure 1: Schematic diagram of DC sputtering setup.

The setup in Fig. 1 can be divided into 6 parts, which are the straight vacuum chamber to be coated, left and right auxiliary chambers, TMP pumping system, a mass flow controlling system, a cathode and its DC power supply. The left auxiliary chamber is used to mix N_2 and Ar gases and to install the connecting device between the cathode and feedthrough, and the right auxiliary chamber is used to install the devices for the measurement and the control of the sputtering. The cathode is 45 mm in diameter and made of Ti with 99.99% purity. To achieve uniformly distributed TiN coating, the cathode is in the coaxial position of the chamber, and both tips of the cathode are in the auxiliary chambers to make sure that the deposition of TiN coating can hardly be affected by the tips, which may lead to high deposition rate. Glass and stainless steel samples, which had been set in suitable positions in the chamber, were to be used to test and evaluate the coating film after sputter coating. With coating experiment of 4 hours, the chamber was coated on the inner face with TiN film of 500 nm thickness.

Test of Chemical Compositions of the Film Coating

One of the samples was investigated with an XPS survey scan, resulting in Fig. 2 shown as follows:

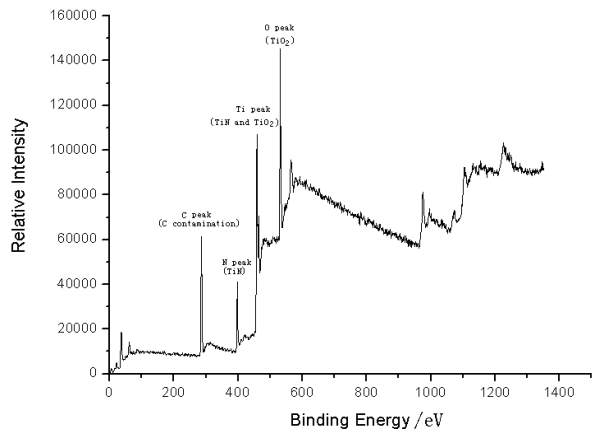

Figure 2: XPS survey spectra of the SS sample.

From Fig. 2, the survey found 4 elements, C, N, O and Ti in the coating, which form TiN, TiO_2 and contaminants of hydrocarbons [4]. XPS results show that the number ratio of N atoms to Ti atoms is 16.4 to 19.8, that is 1:1.2. The formation of TiO_2 is due to two reasons. One is that the O_2 desorbed from the heated chamber react with Ti atoms from the cathode; the other is O_2 in the air reacted with Ti atoms deposited on the sample before the XPS test. For the former, mixing of small flux H_2 into gas mixture can help to inhibit the oxidation of Ti during sputtering; for the latter, suitable temperature of substrate and proper concentration of the reacting gas can enhance the efficiency of Ti atoms' nitridation during sputtering, so as to reduce the probability of Ti atoms' oxidation in the air. Therefore, we are planning to improve our TiN coating experiments in the near future.

*Corresponding author: ywang@ustc.edu.cn

OUTGASSING RATE TESTS OF THE COATED CHAMBER AND THE UNCOATED CHAMBER

For an all-metal vacuum system, bakeout is a very effective method to achieve ultra high vacuum rapidly. Outgassing rates were measured by little orifice method to check whether the rate of TiN coated chamber is improved. The results are as follows:

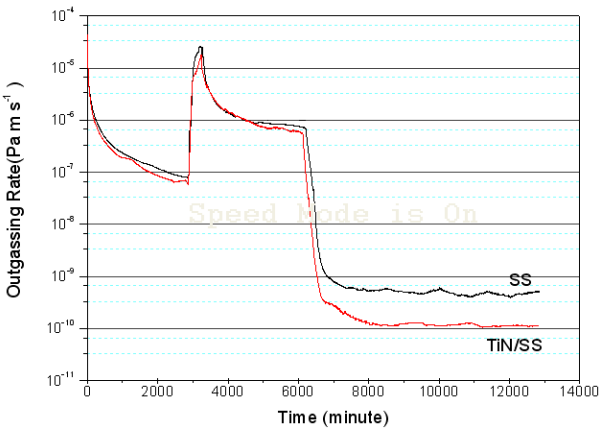

Figure 3: Compare of the outgassing rate for SS chamber and TiN coated SS chamber.

As shown in Fig. 3, 'SS' and 'TiN/SS' denote respectively the data of thermal outgassing rates of the stainless steel chamber before and after TiN film coating. The coated chamber had almost the same outgassing rates as those of the uncoated chamber in the process of pumping before baking, which achieved 1.0×10^{-7}Pa·m ·s^{-1} when the pumping time arrived at 48 hours and the baking began. In the process of baking, they still had the same outgassing rates; after baking ending, the outgassing rates of the coated and uncoated chambers both decreased quickly, but the curve of 'TiN/SS' decreased even more rapidly. The outgassing rates of the coated and uncoated chambers reached 1.2×10^{-10}Pa·m ·s^{-1} and 5.4×10^{-10}Pa·m ·s^{-1} respectively, when the pumping time arrived at 24 hours after baking ending, and they reached 1.17×10^{-10}Pa·m ·s^{-1} and 5.0×10^{-10}Pa·m ·s^{-1} respectively versus pumping time of 48 hours. We can see that coating with TiN film executes an active role in lowering the outgassing rate of stainless steel chamber.

PSD TESTS OF THE COATED CHAMBER AND THE UNCOATED CHAMBER

PSD Test Results for the Uncoated Chamber

Before the chamber was coated, a PSD test for the chamber was done. The results are shown in Fig. 4:

In Fig. 4, 'D' denotes the accumulated number of photons, 'η'denotes the molecular desorption yield. H_2O, H_2, CO, CO_2, CH_4, Ar and O_2 constitute major parts of the desorption gas. During the first test, total accumulated number of photons arrived at 1.77×10^{21}photons m^{-1}, and the desorption yield of H_2O, H_2, CO and CO_2 decreased from order of -3 to -4, while the desorption yield of CH_4, Ar and O_2 decreased from the order of -4 to -5. Then, after the chamber was baked out for 48h at 200℃, PSD tests were performed on the chamber many times. Finally, when total accumulated number of photons arrived at 13.34×10^{21}photons m^{-1}, the desorption yield of every

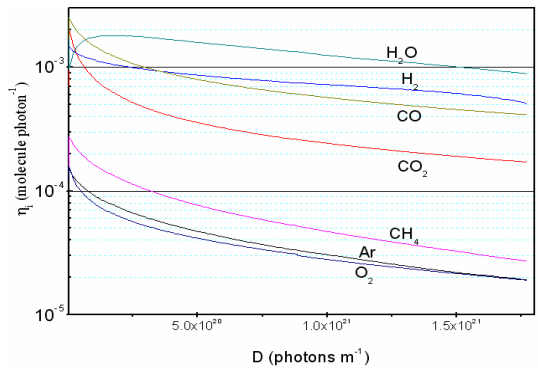

Figure 4: Result of the first PSD test for the chamber.

residual gas remained stable, which was of the order of -4 for CO, -5 for H_2O, CO_2 and H_2,and -6 for O_2, Ar, CH_4, as shown in Fig. 5.

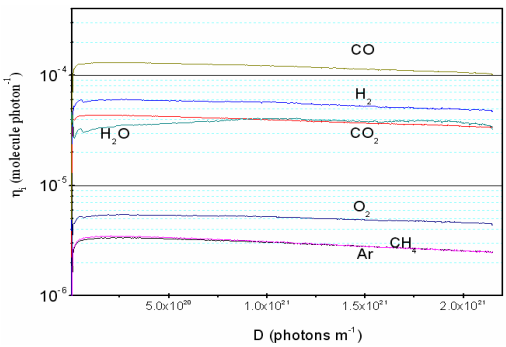

Figure 5: Final PSD test result for the chamber after baking.

PSD Test Results for TiN Coated Chamber

We process PSD tests after the chamber described in paragraph 3.1 has been coated with TiN film. The test results are shown in Fig. 6.

Fig. 6 shows the results of the first test after the vacuum chamber was coated, in which the total accumulated number of photons was about 2.5×10^{21}photons m^{-1}. Compared with the results of the uncoated chamber under the same condition, the coating process has distinct effect to reduce the desorption of H_2 and H_2O. As shown in Fig. 6, the desorption yield of H_2 of the coated chamber reduced to 4×10^{-5} rapidly after the accumulated number of photons arrived at 2.5×10^{21}photons m^{-1}, while that of the uncoated chamber reached just 5×10^{-5} after several tests. And the desorption yield of H_2O in Fig. 6 reduced to 2×10^{-4} in the end, while that of the uncoated chamber is 9×10^{-4}. The coating has certain effect to reduce the desorption of CH_4, Ar and O_2, but the effect is not evident as to CO and CO_2.

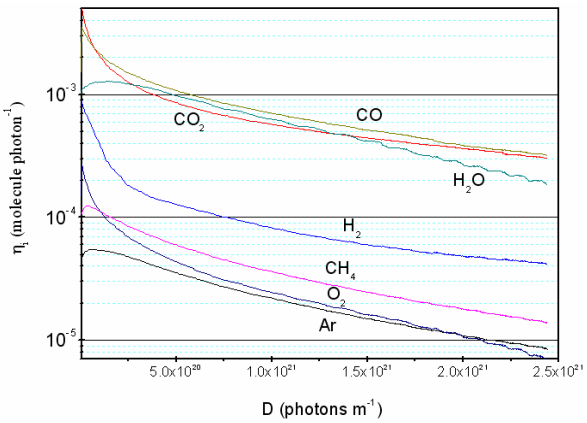

Figure 6: The first PSD test result for TiN coated vacuum chamber before baking.

Then, a series of PSD tests were performed for the coated chamber which had been baked out for 48h at 200℃. Final results are shown in Fig. 7. Compared with the results for the uncoated chamber in Fig. 5 under the same test condition, we could draw the conclusion that coating the chamber with TiN film could reduce the desorption of H_2 and H_2O distinctly. The desorption yield of H_2 reached 1.5×10^{-5} for the coated chamber in contrast to 5×10^{-5} for the uncoated chamber under the same condition, and that of H_2O nearly down to 0 for the coated chamber. The process can also inhibit the desorption of CO, CH_4, O_2 and Ar, but the effect is uncertain for CO_2.

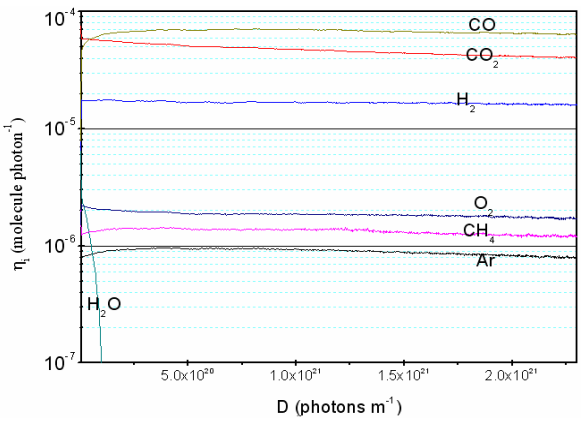

Figure 7: Final result of PSD test for TiN coated vacuum chamber after baking.

SEY TESTS FOR STAINLESS STEEL SAMPLES BEFORE AND AFTER TIN WAS COATED

TiN film coating is an effective way to reduce the SEY value of vacuum material, which is related to electron cloud instability effect. So SEY tests for stainless steel samples before and after TiN coating have also been performed, the results are presented in Fig. 8.

In Fig. 8, 'SS', 'TiN/SS(1)' and 'TiN/SS(2)' denote SEY test results for the uncoated sample, TiN coated sample exposed to the air for 10 days and TiN coated sample exposed to the air for 10 minutes, respectively. Once a sample is exposed to the air, it will be contaminated by hydrocarbons. The conclusion can be made that TiN coating is an effective method to reduce SEY of stainless steel material. For the coated samples, higher SEY result implies that the corresponding sample has been contaminated more seriously. It is believed that uncontaminated sample with TiN coating will shows even lower SEY test result.

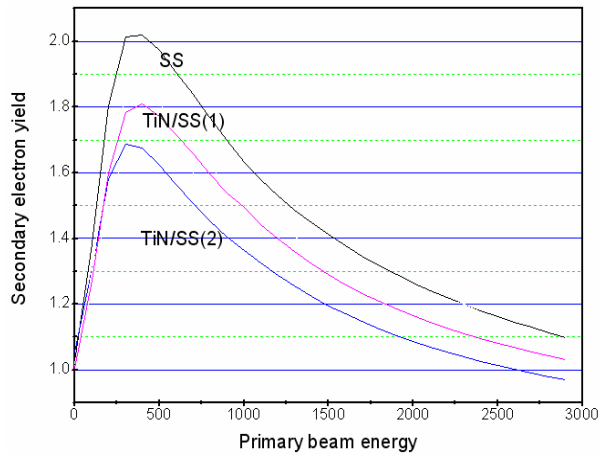

Figure 8: SEY results of stainless steel and TiN coated stainless steel samples under different condition.

CONCLUSION

A series of tests including thermal outgassing rate, PSD and SEY tests have been done for a stainless steel vacuum chamber before and after it was coated with TiN film by DC sputtering. The test results show that coating with TiN film is an effective method to improve the performances of vacuum chamber, especially in thermal outgassing rate, PSD and SEY.

ACKNOWLEDGEMENT

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