EXPERIMENTAL STUDY OF STAINLESS STEEL VACUUM CHAMBER WITH TiN FILM COATING

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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.](image)

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 N₂ 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 sputtering 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.](image)

From Fig. 2, the survey found 4 elements, C, N, O and Ti in the coating, which form TiN, TiO₂ 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 TiO₂ is due to two reasons. One is that the O₂ desorbed from the heated chamber react with Ti atoms from the cathode; the other is O₂ in the air reacted with Ti atoms deposited on the sample before the XPS test. For the former, mixing of small flux H₂ 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.

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Accelerator Technology - Subsystems

T14 - Vacuum Technology
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:

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 $1.0\times10^{-7}\text{Pa}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$ 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 $1.2\times10^{-10}\text{Pa}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$ and $5.4\times10^{-10}\text{Pa}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$ respectively, when the pumping time arrived at 24 hours after baking. 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, ‘$\eta$’ denotes the molecular desorption yield. H$_2$O, 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 $1.77\times10^{21}\text{photons m}^{-2}$, and the desorption yield of H$_2$O, 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°C, PSD tests were performed on the chamber many times. Finally, when total accumulated number of photons arrived at $13.34\times10^3\text{photons m}^{-1}$, the desorption yield of every residual gas remained stable, which was of the order of -4 for CO, -5 for H$_2$O, CO$_2$ and H$_2$, and -6 for O$_2$, Ar, CH$_4$, as shown in Fig. 5.

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 $2.5\times10^{21}\text{photons m}^{-1}$. 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$_2$O. As shown in Fig. 6, the desorption yield of H$_2$ of the coated chamber reduced to $4\times10^{-5}$ rapidly after the accumulated number of photons arrived at $2.5\times10^{21}\text{photons m}^{-1}$, while that of the uncoated chamber reached just $5\times10^{-5}$ after several tests. And the desorption yield of H$_2$O in Fig. 6 reduced to $2\times10^{-4}$ in the end, while that of the uncoated chamber is $9\times10^{-4}$. 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$_2$.
Then, a series of PSD tests were performed for the coated chamber which had been baked out for 48h at 200°C. 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 H₂ and H₂O distinctly. The desorption yield of H₂ reached $1.5 \times 10^{-5}$ for the coated chamber in contrast to $5 \times 10^{-5}$ for the uncoated chamber under the same condition, and that of H₂O nearly down to 0 for the coated chamber. The process can also inhibit the desorption of CO, CH₄, O₂ and Ar, but the effect is uncertain for CO₂.

**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**

Financial supports for this work were provided by the National Nature Science Foundation of China under Grant Nos. 10245002.

**REFERENCES**