Experimental study on TiN coated racetrack-type ceramic pipe *

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Abstract: TiN film was coated on the internal surface of a racetrack-type ceramic pipe by three different methods: radio-frequency sputtering, DC sputtering and DC magnetron sputtering. The deposition rates of TiN film under different coating methods were compared. The highest deposition rate was 156 nm/h, which was obtained by magnetron sputtering coating. Based on AFM, SEM and XPS test results, the properties of TiN film, such as film roughness and surface morphology, were analyzed. Furthermore, the deposition rates were studied with two different cathode types, Ti wires and Ti plate. According to the SEM test results, the deposition rate of TiN/Ti film was about 800 nm/h with Ti plate cathode by DC magnetron sputtering. Using Ti plate cathode rather than Ti wire cathode can greatly improve the film deposition rate.

Key words: TiN, ceramic vacuum chamber, Ti cathode

PACS: 29.20.-c **DOI:** 10.1088/1674-1137/39/11/117005

1 Introduction

In the past few decades, TiN thin film has attracted much interest because of its low secondary electron yield (SEY), good electrical conductivity, stability of performance, ability to block hydrogen permeation, [1–3] etc. The properties and coating process of TiN thin film have been studied at laboratories including KEK [1], NSRL [4], DESY [5] and BNL [6]. TiN coated ceramic vacuum chamber is a key component of the electron storage ring injection system at the Hefei Light Source II (HLS II). However, for some irregular type ducts, such as the racetrack type (Fig. 1) ceramic chamber in accelerators, the shape of the ceramic pipeline will introduce considerable new technological difficulties for the uniformity of TiN coating, which is important for vacuum and beam stability in the pipeline. Therefore, it is of great importance to get high-quality film which meets the requirements of the physical design of the storage ring, such as mitigating the electron cloud instability [7].

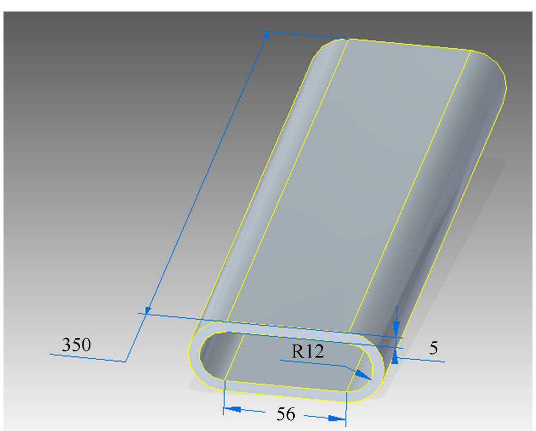

Fig. 1. (color online) Ceramic vacuum pipe.

In this study, TiN film was deposited on ceramic pipe with different cathode structures and various film coating methods. The TiN film properties were investigated by atomic force microscope (AFM), scanning electron microscope (SEM) and X-ray photoelectron spectroscopy (XPS).

2 Apparatus and methods

2.1 Coating system

TiN films were deposited onto the interior wall of a ceramic vacuum pipe as shown in Fig. 1. The deposition system, which is shown in Fig. 2, consists of an observation window, 300 l/s turbo molecular pump, power supply, vacuum gauge, coating vacuum chamber and gas flow control system. Argon gas and nitrogen are introduced into the sputtering system through an adjustable leak valve. There are two types of cathode structure, one consisting of three Ti wires and one consisting of a Ti plate of size 2 mm×350 mm×56 mm. Figure 3 shows

Received 16 January 2015, Revised 31 May 2015

∗ Supported by National Nature Science Foundation of China (11075157)

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the positions of silicon samples on the inner surface of the ceramic pipe. In addition, the silicon samples were used to do SEM tests conveniently.

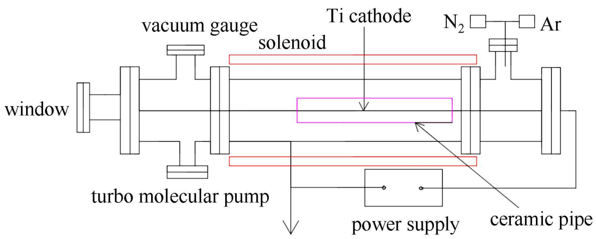

Fig. 2. (color online) Schematic diagram of sputtering coating system.

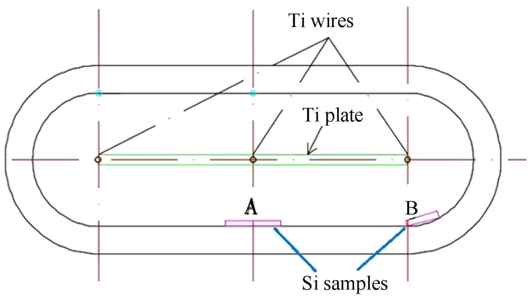

Fig. 3. (color online) Positions of silicon samples on the ceramic pipe inner surface, three Ti wire cathode and Ti plate cathode.

2.2 Characterization method

Film thickness was measured using a Sirion 200 Schottky field scanning electron microscope (SEM). The basic SEM parameters used in the experiments are as follows: 5 kV accelerating voltage, 5.4 mm working distance, 91.53K magnification. Material surface and internal compositional data were taken with a Thermo ESCALAB 250 X-ray photoelectron spectroscopy (XPS). The spectrometer was equipped with a hemispherical analyzer and a monochromator, with a beam spot size of 500 μm. All XPS data were measured with Al Kα X-rays with ($h\nu$=1486.6 eV), operated at 150 W and with an analyzer at 45 degrees. Surface morphology was observed through an Innova atomic force microscope (AFM) at room temperature. The micrographs were taken at 5 kV to keep the images surface sensitive.

2.3 Cathode structure

The cross-section of the shaped ceramic vacuum chamber in the system is a special racetrack type with a small aperture. In order to obtain a uniform thin film, a few titanium wires were used in the experiment, horizontally mounted as a cathode target. Because of the rotational asymmetry, the coated film obviously could not be uniform with only one or two titanium wires as cathodes, so three cathodes were considered. Due to the complexity of the calculation process, the analysis of sputtering rates (S) on different locations of the inner surface of the ceramic vacuum chamber was performed using the Matlab software.

In Fig. 4 the left side of the ceramic vacuum pipeline was simulated according to the symmetry characteristics. S will decrease with the increase of e for point F. In the case of $0\leqslant b\leqslant c$, for point D, $S=S_1+S_2+S_3$

$$S_1=\frac{\pi Kd}{2(b^2+d^2)},\ S_2=\frac{\pi Kd}{2[(c-b)^2+d^2]},\ S_3=\frac{\pi Kd}{2[(2c-b)^2+d^2]}$$

where K is constant. S take a large value when $b=c$ for point B, and S increases with c for point A. Point E is the most appropriate location for the left Ti cathodes considering the homogeneity of the film thickness. On the basis of the computation, the ratio of the maximum and the minimum film thickness is about 1.4:1 for c=28. Meanwhile the average thickness of the TiN film at points A and B is 20.6 nm and 27.9 nm respectively in DC sputtering experiments, which means that the average thickness ratio is 1:1.35. Hence the results are in good agreement with the experimental data. In order to improve the sputtering efficiency, a titanium plate cathode was then used. Furthermore, CST software was used to simulate the distribution of the electric field under

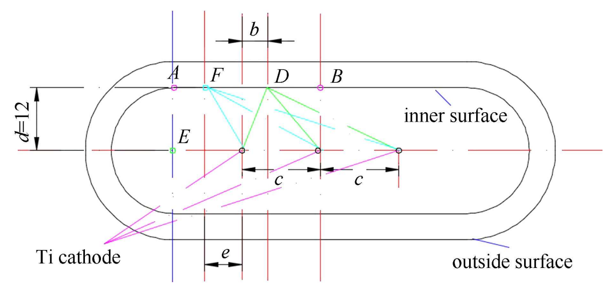

Fig. 4. (color online) Sputtering rate analysis of one point on the inner surface of a ceramic vacuum pipeline, where c is the distance between the Ti cathodes, b is the distance along the horizontal direction between the left Ti cathode and point D which is on the right of the left Ti cathode in the horizontal direction, and e is the distance along the horizontal direction between the left Ti cathode and point F.

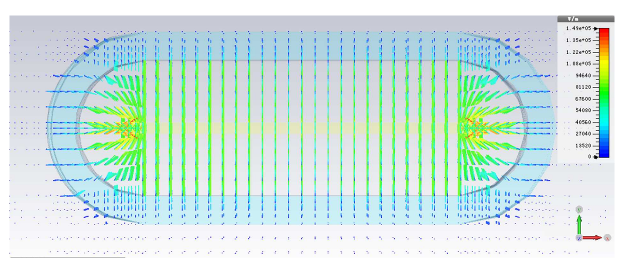

Fig. 5. (color online) Electric field distribution in the case of Ti plate with a size of 66 mm×2 mm× 350 mm.

different sizes of titanium plate. According to the simulation result, a titanium plate size with 66 mm×2 mm×350 mm can meet the requirements of film uniformity, because the electric potential is basically the same on the edges of the ceramic (Fig. 5).

3 Results and discussion

3.1 DC sputtering

During the experiment, the background vacuum pressure was below 10^{-4} Pa, then mixed gases of nitrogen and argon were injected, controlled by D08-3B/ZM gas mass flow. The typical sputtering parameters are: −900 V cathode voltage, 66 Pa gas pressure, 3 h deposition time, 5:1 nitrogen and argon gas flow ratio and 30 mA sputtering current. The film thickness was tested by selecting two points on each sample at positions A and B in Fig. 3. The average thickness of the film was 51.0 nm. The approximate deposition rate is about 17 nm/h. Figure 6 shows the cross-sectional SEM morphology of TiN film samples prepared by DC sputtering.

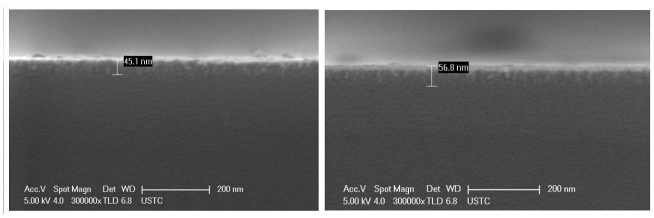

Fig. 6. Cross-sectional SEM morphology of TiN film sample prepared by DC sputtering with three Ti wire cathodes.

3.2 Radio-frequency sputtering

TiN film thickness was 750–800 nm, as can be seen in Fig. 7, and the deposition rate was about 80 nm/h by radio-frequency sputtering.

3.3 DC magnetron sputtering

The typical sputtering parameters are: −542–−503 V cathode voltage, 20–30 Pa working pressure, 3 h deposition time, 2.5 Sccm nitrogen and 5 Sccm argon, 200 G magnetic field and 0.5 A sputtering current. The film deposition rate and thickness with different cathode types are shown in Table 1. Figure 8 shows the cross-sectional SEM morphology of a TiN film sample prepared by DC magnetron sputtering with the Ti plate cathode. In Fig. 9, the roughness of TiN films at positions A and B in Fig. 3 which were prepared by DC magnetron sputtering with the Ti plate cathode, were 157.7 nm and 233.3 nm respectively within a 5 μm range. The film deposition rate can be improved significantly using the Ti plate cathode by DC magnetron sputtering, as shown in Table 1.

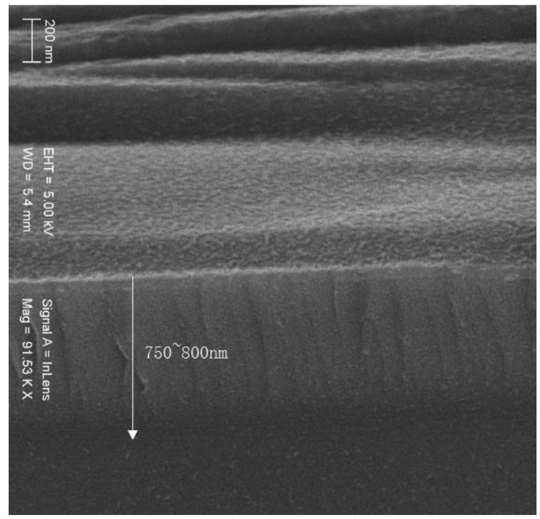

Fig. 7. Cross-sectional SEM morphology of TiN film sample prepared by radio-frequency sputtering with three Ti wire cathode, 10 h deposition time.

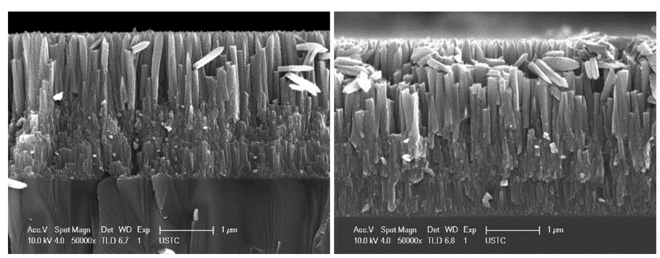

Fig. 8. Cross-sectional SEM morphology of TiN films at position A (left) and B (right) prepared by DC magnetron sputtering with Ti plate cathode.

Table 1. Film deposition rates and thicknesses with different cathode types under various deposition methods.

deposition method	deposition rate/(nm/h)	thickness at position A/nm	thickness at position B/nm	cathode type
radio-frequency sputtering	80	800	750	three Ti wires
DC sputtering	17	56.8	45.1	three Ti wires
DC magnetron sputtering	156	780	535	three Ti wires
DC magnetron sputtering	800	3200	2720	Ti plate

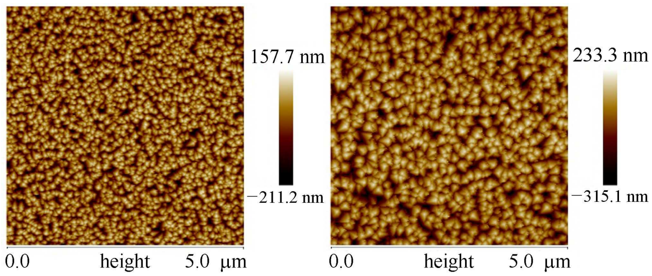

Fig. 9. (color online) AFM morphology of TiN films at position A (left) and B (right) prepared by DC magnetron sputtering with Ti plate cathode.

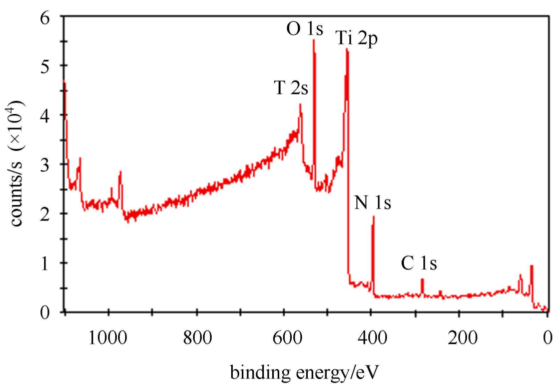

Fig. 10. (color online) Wide scan XPS spectrum of an as-received TiN film deposited by magnetron sputtering, showing oxygen and carbon contamination on the surface of the sample.

A typical XPS spectrum is shown in Fig. 10, illustrating that the average composition of TiN samples deposited was Ti 56 at% and N 44 at%, under typical deposition parameters. A large amount of oxygen and a little carbon contamination on the TiN surface appears in the XPS spectrum. The test depth of the TiN sample was 1–3 nm for XPS, and the film is easily polluted by active gases, like oxygen, in the process of transferring the TiN sample, so this large amount of oxygen appears in the XPS spectrum.

4 Conclusion

For non-conductive ceramic materials, TiN film deposition rate can be effectively improved by the DC magnetron sputtering method，compared to the DC sputtering and RF sputtering methods. In addition, the cathode installation position, and shape and size of the cathode material, can influence the uniformity of film thickness to a certain extent. TiN film deposition rate with a three-Ti-wire cathode was about 20 nm/h by DC sputtering, while it was 156 nm/h by DC magnetron sputtering. Besides, DC magnetron sputtering combined with Ti plate cathode can greatly improve the film deposition rate, which was found to be 800 nm/h on the basis of cross-sectional SEM morphology. AFM test results shows that the roughness of TiN was about 200 nm within the 5 μm range, which meets the requirements of engineering in accelerator fields.

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