VACUUM PUMPING PERFORMANCE COMPARISON OF NON-EVAPORABLE GETTER THIN FILMS DEPOSITED USING ARGON AND KRYPTON AS SPUTTERING GASES*

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Abstract

Owing to the outstanding vacuum performance and the low secondary electron yield, non-evaporable getter (NEG) thin film deposited onto interior walls has gained widespread acceptance and has been incorporated into many accelerator vacuum system designs. In this paper, the titanium-zirconium-vanadium NEG thin films were deposited onto interior wall of stainless pipes via DC magnetron sputtering method using argon and krypton gas respectively as the sputtering gas. Vacuum pumping evaluation tests were carried out to compare vacuum pumping performances of the Ti-Zr-V NEG thin films deposited using the two rare gases. The results showed much higher initial pumping speed for the Kr-sputtered NEG film than the Ar-sputtered film, though both films have similar activation behavior. Films were also deposited onto silicon wafers under the same experimental conditions, and Rutherford Backscattering Spectrometry (RBS) analysis was carried out to obtain information of the film composition and thickness. The RBS analysis showed that the concentration of argon is significantly higher that of krypton. Insight of the textures of both thin films will be pursued for better understanding of the cause of their difference in pumping performances.

INTRODUCTION

The technique of depositing a NEG film onto the inner wall the vacuum chamber has been developed in the past decade and begun to be widely used especially in the particle accelerators and storage rings, in meeting the challenges of higher vacuum requirement and often limited spatial access for lumped pump installation. Among NEG films with various compositions that have been studied, Ti-Zr-V film is one of the most favored due to its excellent vacuum performance and low activation temperature. As part of the Energy Recovery Linac (ERL) project at Cornell, we have studied the coating procedure and tested the vacuum performance [1]. In this paper, we report the comparison study of the NEG coating using argon and krypton respectively as discharge gas.

EXPERIMENTAL

The experimental setup for the NEG film coating is a DC magnetron sputtering system, which is similar to that reported before [1], and only a brief description is given here. The stainless steel (SST) tube to be coated is 12 inches long with a 3.88 inch inner diameter. The sputtering cathode was formed by twisting 1-mm-diameter titanium, zirconium, and vanadium wires. The flow rate of discharge gas was adjustable and controlled by a leak valve. The pressure in the chamber was adjusted by a valve between the chamber and the turbo pump, so that a stable discharge with desired current was maintained. A solenoid outside the SST tube supplied 200-G magnetic field. Typical conditions were 2 - 3 mTorr discharge pressure, -700 V cathode voltage, and 25 mA sputtering current. Fan cooling was applied on the solenoid, and the SST wall temperature was estimated to be around 60 ºC during the deposition. The typical film thickness was 2 µm.

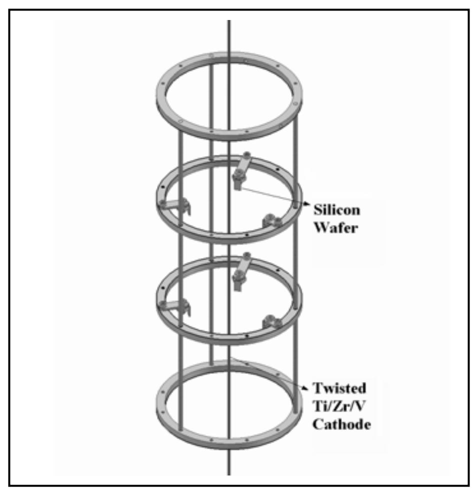

Figure 1: Silicon wafer sample holder for NEG film deposition. The assembly is mounted to the SST tube.

NEG thin film was deposited onto silicon samples for composition analysis by RBS, under deposition conditions same as for the SST tubes. The silicon wafers (3 x 5 mm) were glued using ultra-high-vacuum compatible epoxy (TorrSeal®, Varian) onto SST nuts and mounted to the sample support (see figure 1). The distance form the cathode to the wafers ranges from 1 in. to 1.3 in. Very thin films (10 – 40 nm thickness) were deposited in order for RBS to get high enough resolution. The RBS analysis was carried out at Cornell Center for Material Research, and the spectra were simulated using RUMP program [2].

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The vacuum pumping performance of the NEG thin film deposited using Kr as sputtering gas was compared with that using Ar as sputtering gas. The vacuum pumping speed of the NEG films was measured using throughput method, with CO as testing gas. The setup was same as the one described in reference [1].

RESULTS AND DISCUSSION

Pumping Performance

The pumping performance of Kr-sputtered and Ar-sputtered NEG films are summarized in figure 2. Whereas the pumping capacities are similar and show similar activation temperature dependence using the two discharge gases, the initial pumping speeds S_0 (after each activation) show significant difference. The measured

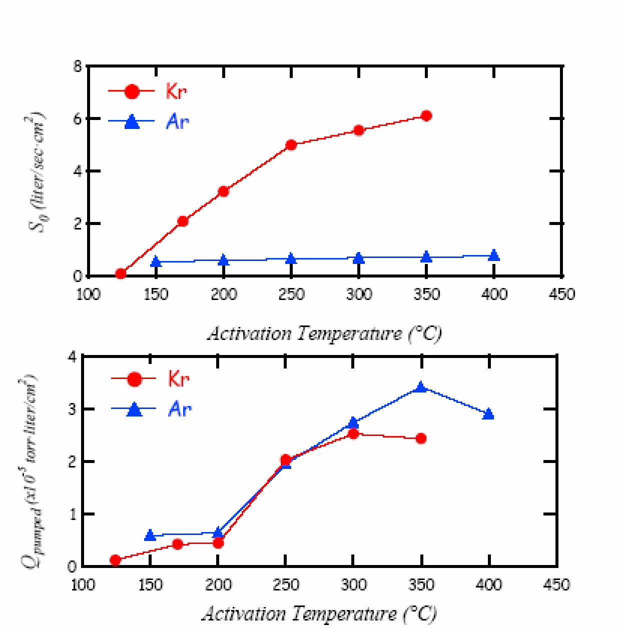

Figure 2: Dependence of the initial pumping speed (S_0) (top) and the pumping capacity (bottom) on the activation temperature for both Ar- and Kr-sputtered films.

initial pumping speed after activations at various temperatures for both films coated using argon and krypton respectively as discharge gas is shown in the top plot. The S_0 of the Kr-sputtering film is five times as high as that of the Ar-sputtering film after 200 ºC activation, is eight times after 350 ºC activation. The dependence of the pumping capacity on the activation temperature for both films is shown in the bottom plot. The NEG film texture difference is most likely the reason for the much enhanced pumping performance of the Kr-sputtered NEG film over the Ar-sputtered film. Further investigation should be carried out.

NEG Film Composition

NEG films deposited on silicon wafers were studied by RBS to analyze the film composition. Particular attention is paid to the embedment of the sputtering gas, as large amount of the embedded Ar or Kr may affect the film quality and may be source of outgassing. Typical RBS spectra are shown in figure 3, and the compositions of more samples are summarized in table 1 for convenience

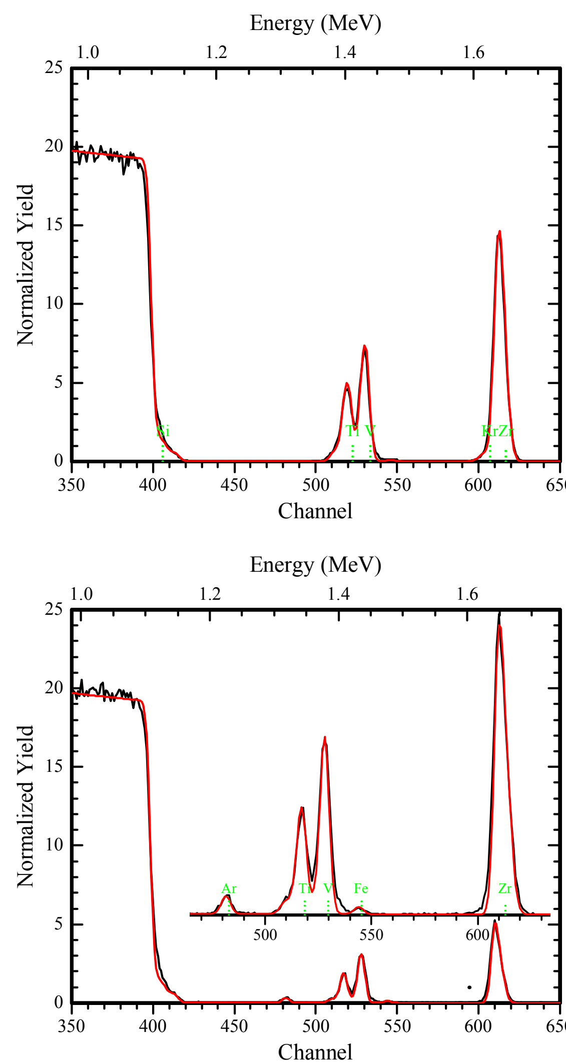

Figure 3: RBS spectra of Ti-Zr-V films using krypton (top) and argon (bottom) respectively as sputtering gas.

of comparison. The concentration of Ar in the film is much higher that that of Kr in the Kr-sputtered film. The atomic concentration of Kr is around 1 percent, while that of Ar is between 3 and 6 percent, varying with the film thickness. The numbers for Ar have very small error bar, for the Ar peak is completely resolved from others; the concentration reported here for Kr, however, should only be considered as its upper limit, because the Kr peak overlaps with the tail of the much stronger Zr peak in the RBS spectrum.

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Table 1: NEG Film Compositions

Sputtering Gas	Thickness	Composition Ti/Zr/V/Fe/(Kr or Ar)
Kr	39 nm	0.35/0.30/0.33/0.01/0.012
Kr	23 nm	0.34/0.29/0.35/0.003/0.012
Ar	19 nm	0.33/0.28/0.35/0.007/0.033
Ar	11 nm	0.31/0.26/0.36/0.012/0.059

The fact that the argon trapping in the film is more severe is actually not surprising because previous studies by Benvenuti et al [3, 4] showed more remarkable difference between the two, although the concentrations are lower by orders of magnitude than this study. It is believed that this rare gas trapping in the film results from the bombardment to the NEG film being coated by the energetic rare gas atoms which are neutralized on arriving at the cathode and then backscattered. Therefore, the mass ratio of the rare gas atom relative to the target atom (in the cathode) is a very important factor in determining the probability of the backscattering and the fraction of the kinetic energy it carries. From this aspect, trapping of argon in the film is obviously favored than Kr. However, there are many other factors which are also crucial, such as the average free path (and therefore the pressure), the distance between the cathode and the sample, and the temperature of the substrates. The rare gas trapping obviously does not just simply modify the composition of the desired Ti-Zr-V alloy, but probably more importantly has a strong effect on the texture of the film because the bombardment of the sputtering gas cause more efficient mixing of the coating elements and with the substrate, which in turn influence the pumping performance. The texture is going to be studied.

The concentrations of both Ar and Kr are higher by at least one order of magnitude than that measured by Amorosi et al [4] using laser ablation and residual gas analyzer (RGA) probe. Both measurements are able to provide quantitative composition information with appropriate calibration for the laser ablation method; therefore we believe both measurements are correct, and the dramatic difference can result from many other experimental factors mentioned above. It is necessary to point out that the concentration of the sputtering gas could be much higher than expected according to our RBS measurement.

CONCLUSIONS

The vacuum performance of Ti-Zr-V NEG films using argon and krypton respectively as sputtering gas have been investigated. While the pumping capacities are similar, the kr-sputtered film supplies initial pumping speed several times higher than that of Ar-sputtered film. RBS analysis shows that the upper limit of the krypton concentration is about 1 percent and the argon concentration is 3 to 6 percent, which are higher by orders of magnitude than reported in literature.

REFERENCES

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