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ELECTRON STIMULATED DESORPTION FROM TITANIUM TUBE

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Abstract

Titanium is one of the materials that used for production of accelerator vacuum chamber and components. In this paper we report the results of vacuum properties evaluation measurements of titanium vacuum chamber. The sample was produced from 40-mm inner diameter tube made of titanium and equipped with CF40 flanges at both ends. The electron stimulated desorption (ESD) was measured after 24-h bakeout to 80, 150, 180 and 250 °C. H_2 and CO initial sticking probabilities were measured after bakeout before the ESD measurements. After ESD measurements, the initial H_2 and CO sticking probabilities were measured again together with CO sorption capacity. These measurements provide the results for ESD as a function of electron dose baked to different temperatures and demonstrate the efficiency of electron stimulated activation of titanium vacuum chamber.

INTRODUCTION

Titanium and its alloys were investigated in the past as materials for accelerator vacuum chamber that can provide low thermal, photon, electron and ion stimulated desorption yields [1-3]. However, the information on the desorption yields after different treatments and for different conditions is still very limited.

Ti is a well-known getter material which is used in sputter ion pumps (SIP) and evaporable getter (titanium sublimation pumps – TSP). Our earlier studies of non-evaporable getter films demonstrated that a Ti film with a columnar structure shows pumping properties after heating to ≥ 200 °C for 24 hours. Dense Ti structures would require higher activation temperature.

The aims of this work were: (1) to check is any pumping properties of pure Ti material would be measured after heating to 150-300 °C (typical range of temperatures applied for vacuum chamber bakeout), (2) to measure ESD yield from non-baked Ti and after bakeout to 150, 180 and 250 °C, (3) to check if there are effects of electron bombardment stimulated activation similar to one for NEG coatings [4, 5].

SAMPLE

The sample is a tube with an inner diameter of 40 mm and a length of 50 cm, and was produced from titanium tube welded to CH40 flanges made of 316 LN stainless steel at Scanwel Ltd. (Bala, Gwynedd, UK).

The received sample was cleaned at Daresbury Laboratory following standard cleaning procedure [6].

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ESD FACILITY

A facility for ESD measurement from tubular samples is described in [5]. This facility provides electron bombardment from a hot Thoria coated filament placed along the tube axis. The filament is heated with a current (up to I_f=8 A) from the floating power supply, biasing the filament is possible up to 6.5 kV allowing electron incident energies in the range $0 \le E_e \le 6.5$ keV. The experiments described in this paper were performed with the electron incident energy of E_e = 500 eV and the electron current in the range of $1 < I_e < 30$ mA. The sample temperature was maintained at room temperature. The gas injection line allows the injection of H_2, CH_4, CO, CO_2 and N_2 for RGA calibration, sticking probability, pumping speed and capacity measurements.

EXPERIMENTAL PROCEDURE

After installing the Ti tubular sample on the ESD measurement facility, the test vacuum chamber was pumped out and, after 12 hours pumping, set for a bakeout. In this study, to minimise a possible contamination of the Ti sample, the bakeout procedure was the same as used for the NEG coated samples [1].

The Run 1 procedure consists of the following steps:

a. Initially, all parts of test chamber were heated to 200 °C with exception of the Ti tube heated to 80 °C.
b. After the 24-h bakeout all other parts of test chamber were cooled to 150 °C, while Ti tube remains heated to 80 °C.
c. Thoria filament switched on and degassed, then extractor gauge and RGAs switched on and degassed.
d. After that all parts of test chamber including the sample were cooled to room temperature.
e. The ESD measurement were started after ~12 hours at room temperature.
f. Pumping properties were checked before irradiation started and after irradiation completed.
g. Venting to air for 30 minutes.

The Ti sample temperature of 80 °C is a compromised value allowing us to minimise two effect: it should be sufficiently high to avoid re-condensation of gas species desorbed from other parts of test chamber during their bakeout and, from another side, it should be low in comparison to usual bakeout temperatures (150-300 °C) and it should be sufficiently low to avoid activation of the Ti as a getter material.

The Run 2-4 procedure was different and consists of the following steps:

a. Initially, same as in Run 1, all parts of test chamber were heated to 200 °C with exception of the Ti tube heated to 80 °C.

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b. After the 24-h bakeout all other parts of test chamber were cooled to 150 °C, while Ti tube was heated to 150 °C.
c. Thoria filament switched on and degassed, then extractor gauge and RGAs switched on and degassed.
d. After 1 h the temperature of the Ti tubular sample was set to a selected temperature (150 °C in Run 2, 180 °C in Run 3, 250 °C in Run 4).
e. 2 hours after reaching the desired sample temperature all other parts of test chamber were cooled to room temperature.
f. The Ti sample remains at the desired sample temperature for 24 h then it cooled down to room temperature.
g. The ESD measurement were started after ~12 hours at room temperature.
h. Pumping properties were checked before irradiation started and after irradiation completed.
i. Venting to air for 30 minutes

In Run 5 the Ti sample was heated to 300 °C to check pumping properties only.

RESULTS

In all five runs no pumping properties were measured neither before nor after electron bombardment.

The ESD yields (η) were calculated as described in [5] for non-sorbing surface. They are shown in Figs. 1-5 as a function of accumulated electron dose after bakeout to 80, 150, 180 and 250 °C for 24 hrs for H_2, CH_4, CO, CO_2 and H_2O, respectively.

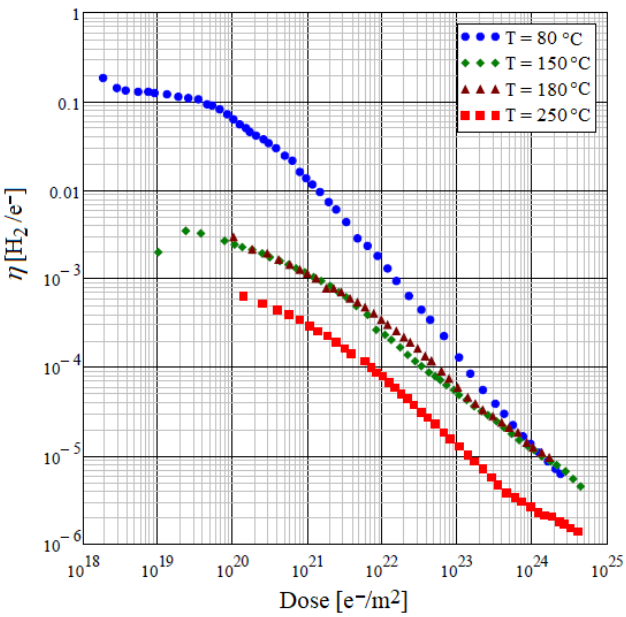

Figure 1: H_2 ESD yields as a function of accumulated electron dose after bakeout to 80, 150, 180 and 250 °C for 24 hrs.

Run 1 bombardment started with a very low current (<1 mA) allowing to measure ESD at very low electron accumulated dose of 2×1018 e-/m2. For low doses 2×1018 ≤ D ≤ 4×1019 e-/m2, the initial ESD yields for H2 and CH4 changes within factor 2, reduces by factor 5 for CO and factor 10 for CO2, however it is increasing by factor 5 for H2O. For higher doses (D > 4×1020 e-/m2) the ESD yields reduce exponentially: η(D) ∝ D–a where a ≈1 for H2, CH4 and CO, while the H2O and CO2 ESD yields reduce quicker.

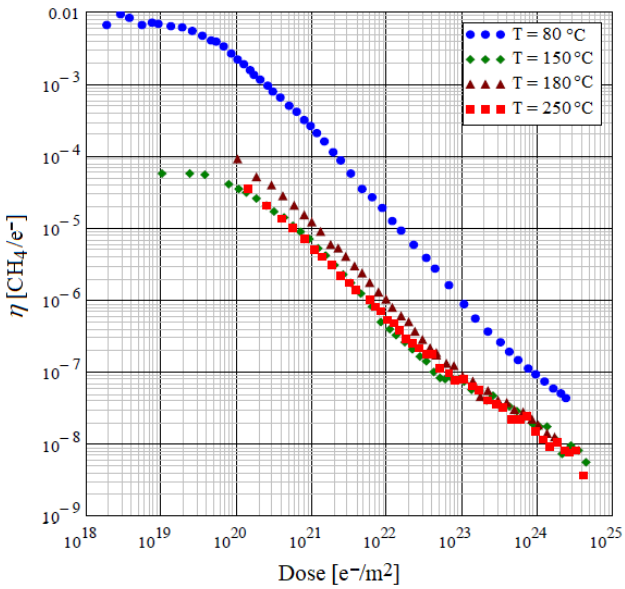

Figure 2: CH_4 ESD yields as a function of accumulated electron dose after bakeout to 80, 150, 180 and 250 °C for 24 hrs.

After bakeout to 150 °C in Run 2, the initial ESD yields measured at $D = 1\times10^{19}$ e-/m^2 are much lower than in Run 1, however the effect of conditioning with a dose is slower. The higher bakeout temperature of 180 °C in Run 3 results in ESD yields similar to ones in Run 2. The bakeout temperature of 250 °C in Run 4 results in further reduction of the ESD yields for H_2 and insignificant difference for other gas species.

DISCUSSION

The 80 °C bakeout in Run 1 was used to avoid contamination from other parts of vacuum chamber during their bakeout, this bakeout is unlikely affect the ESD yields of any measured gas species apart of H_2O, so this sample can be considered as unbaked. In comparison to Run 1, the bakeout to 150 °C in Run 2 reduces ESD yields by a factor from 30 to 1 for H_2, 60 to 5 for CH_4, 120 to 3 for CO, 500 to 9 for CO_2, and 2×10^3 to 2 for H_2O, correspondingly measured at accumulated electron doses of $D = 10^{20}$ and 10^{24} e$^-$/m^2.

Results obtained after bakeout to 180 °C in Run 3 are quite comparable to ones in Run 2: the same for H2, by up to a factor 3 higher for CH4 and CO, and by up to a factor 20 higher for CO2 and H2O. There is no explanation for the ESD yields in Run 3 higher than in Run 2, however they significantly lower than in Run 1.

The bakeout to 250 °C in Run 4 provide significant reduction of ESD yields in comparison to Run 1: by a factor

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from 100 to 5 for H2, 60 to 10 for CH4, 300 to 10 for CO, 3×103 to 10 for CO2, and 2×104 to 5 for H2O, correspondingly measured at D = 1020 and 1024 e–/m2.

It must be noted that bakeout in all runs provides a more significant improvement for low doses and less significant at high doses. However, this is exactly what is required for the machine operation: significant reduction of pressure at the starting operation of machine and reduced time for vacuum conditioning.

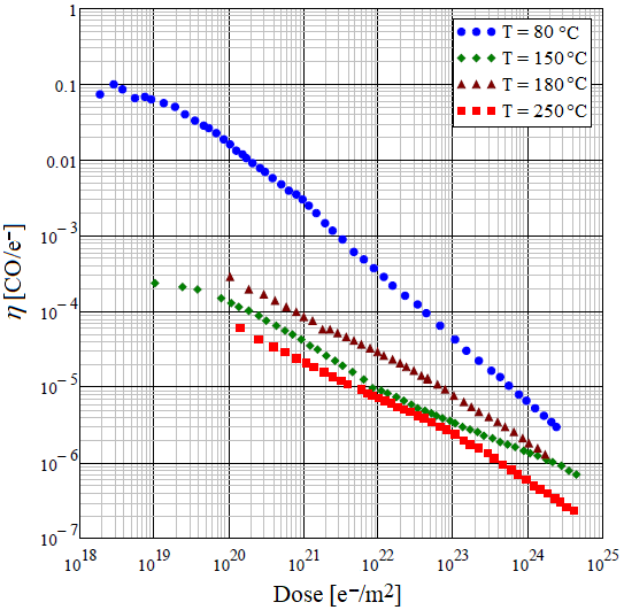

Figure 3: CO ESD yields as a function of accumulated electron dose after bakeout to 80, 150, 180 and 250 °C for 24 hrs.

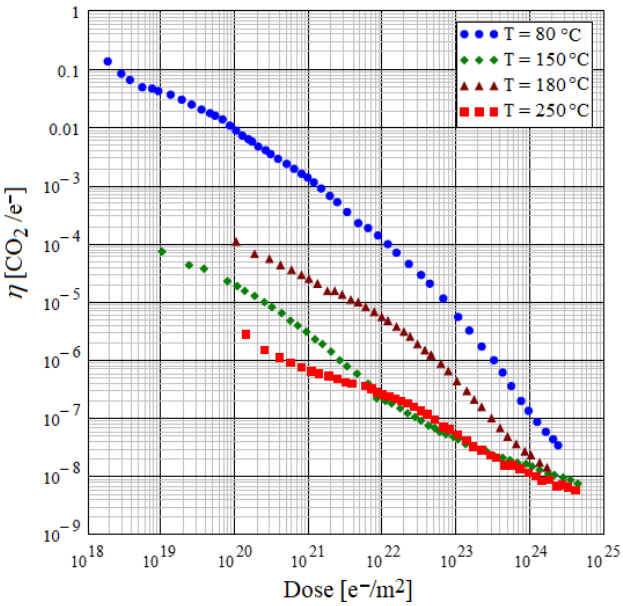

Figure 4: CO_2 ESD yields as a function of accumulated electron dose after bakeout to 80, 150, 180 and 250 °C for 24 hrs.

The ESD result reported above can also be compared to the ones obtained on the same test facility for a 316LN stainless steel tube [7] baked to 250 °C for 24 hrs. The ESD yields for Ti sample baked to T = 80 °C are comparable to ones for 316LN stainless steel tube within a factor 2, while the ESD yields for Ti sample baked to $T \geq 150$ °C are much lower than ones for 316LN stainless steel tube. This demonstrates the advanced vacuum properties of vacuum chamber made of Ti in comparison to one made of 316LN stainless steel. An unbaked vacuum chamber made of Ti can provide the same vacuum specifications as 316LN stainless steel baked to o 250 °C for 24 hrs.

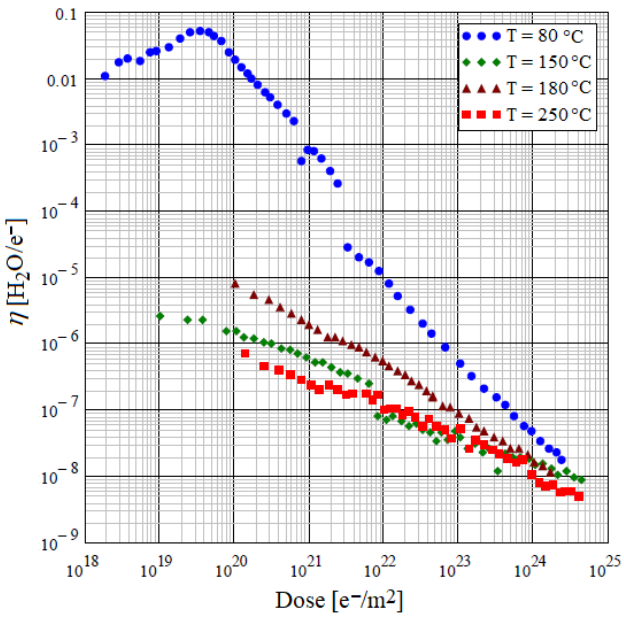

Figure 5: H_2O ESD yields as a function of accumulated electron dose after bakeout to 80, 150, 180 and 250 °C for 24 hrs.

CONCLUSION

The ESD yields from Ti sample have been measured for various gas species as a function of accumulated electron dose after bakeout to 80, 150, 180 and 250 °C.

Unbaked (bakeout to 80 °C) Ti sample provides ESD yields comparable to 316LN stainless steel baked to 250 °C for 24 hrs.

Baking to 150-180 °C reduces ESD yields from Ti sample by a factor 30 for H_2 and by approximately two orders of magnitude for other gas species at low doses, at higher doses this factor reduces to a value between 1 and 10. Baking to 250 °C reduces ESD yields even further by approximately two orders of magnitude for H_2, CH_4 and CO, and by approximately four orders of magnitude for CO_2 at low doses, at higher doses this factor reduces to a value between 5 and 10.

Ti sample demonstrated no pumping properties either after baking up to 300 °C or after electron bombardement.

These results clearly demonstrate the advantage of Ti vacuum chambers for accelerator beam chambers.

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REFERENCES

[1] A. G. Mathewson, "Ion induced desorption coefficients for titanium alloy, pure aluminium and stainless steel", CERN-ISR-VA/76-5, Mar. 1976.

[2] H. Kurisu, K. Ishizawa, S. Yamamoto, M. Hesaka, and Y. Saito, "Application of Titanium Materials to Vacuum Chambers and Components", *J. of Physics: Conference Series*, vol. 100, p. 092002, 2008.

[3] E. Miyauchi, S. Okamura, T. Taguchi, S. Goto, and T. Hisatsugu, "Photon Stimulated Desorption from Finely Polished Metals Irradiated with Synchrotron Radiation". *Jap. J. Appl. Phys*., vol. 30, 2B, pp. 313-315, 1991.

[4] V. V. Anashin *et al.*, "Comparative study of photodesorption from TiZrV coated and uncoated stainless steel vacuum chambers", *Vacuum*, vol. 75, pp. 155-159, 2004.

[5] O. B. Malyshev, A.P. Smith, R. Valizadeh and A. Hannah, "Electron stimulated desorption from bare and NEG coated stainless steel", *J. Vac. Sci. Technol. A*, vol. 28, pp. 1215-1225 2010.

[6] O. B. Malyshev. *Vacuum in Particle Accelerators*, Willey, 2020, pp. 102-105.

[7] O. B. Malyshev and C. Naran, "Electron stimulated desorption from stainless steel at temperatures between –15 and +70 °C", *Vacuum*, vol. 86, pp. 1363-1366, 2012.