



## **Secondary Electron Yield Measurements of TiN Coating and TiZrV Getter Film**

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Abstract: In the beam pipe of the positron Main Damping Ring (MDR) of the Next Linear Collider (NLC), ionization of residual gases and secondary electron emission give rise to an electron cloud which can cause the loss of the circulating beam. One path to avoid the electron cloud is to ensure that the vacuum wall has low secondary emission yield and, therefore, we need to know the secondary emission yield (SEY) for candidate wall coatings. We report on SEY measurements at SLAC on titanium nitride (TiN) and titanium-zirconium-vanadium (TiZrV) thin sputter-deposited films, as well as describe our experimental setup.

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## Abstract

In the beam pipe of the positron Main Damping Ring (MDR) of the Next Linear Collider (NLC), ionization of residual gases and secondary electron emission give rise to an electron cloud which can cause the loss of the circulating beam. One path to avoid the electron cloud is to ensure that the vacuum wall has low secondary emission yield and, therefore, we need to know the secondary emission yield (SEY) for candidate wall coatings. We report on SEY measurements at SLAC on titanium nitride (TiN) and titanium-zirconium-vanadium (TiZrV) thin sputter-deposited films, as well as describe our experimental setup.

## 1 Introduction

Beam-induced multipacting, which is driven by the electric field of successive positively charged bunches, arises from a resonant motion of electrons that were initially generated or by gas ionization or by secondary emission from the vacuum wall. These electrons then bounce back and forth between opposite walls of the vacuum chamber. The electron cloud density depends on characteristics of the positively charged circulating beam (bunch length, charge and spacing) and the secondary electron yield and spectrum of the wall surface from which the starting electrons arise. The electron cloud effect (ECE), due to multipacting, has been observed or is expected at many storage rings [1]. The space charge of the cloud, if sufficient, can lead to a loss of the beam or, at least, to a drastic reduction in bunch luminosity.

In order to minimize the electron cloud problem which might arise in the NLC, we are looking to a solution involving surface coating of the secondary electron emitting vacuum wall. The SEY of technical surfaces has been measured in the past at SLAC [2] [3], at CERN Fig.1 [4] [5] and in other labs [6]. In this paper we present measurements of the SEY of materials previously measured and known to have low SEY [7] [8] [9] like titanium-nitride thin film (TiN) and titanium-zirconium-vanadium getter film (TiZrV).

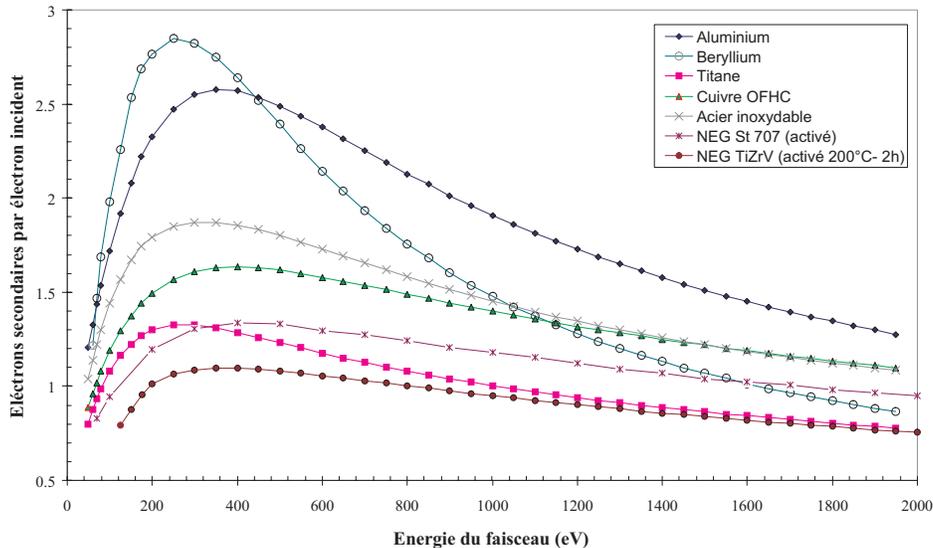


Figure 1: SEY of baked technical surfaces. 350°C for 24hr [10]

## 2 Experiment Description

The system used to measure SEY is composed of two coupled stainless steel (S/S) UHV chambers where the pressure is in the low  $10^{-10}$  Torr scale in the measurement chamber and high  $10^{-9}$  Torr scale in the "load lock" chamber, Fig.2. Samples individually screwed to a carrier plate, are loaded first onto an aluminium transfer plate in the load lock chamber, evacuated to a low  $10^{-8}$  Torr scale, and then transferred to the measurement chamber.

The measurement chamber has two electron guns and a soft (1.49 keV) x-ray source. One electron gun (energy, 1-20 keV) is used for Auger electron spectroscopy (AES) light element surface contamination analysis. The x-ray source is used to excite photoelectrons for surface chemical valence analysis, called ESCA (Electron Spectroscopy for Chemical Analysis). TiN stoichiometry is measured by ESCA technique which is also called XPS (X-ray Photoelectron Spectroscopy).

The principle of XPS is to collect photoelectrons ejected by x-rays of known energy near the surface (1 - 5 nm information depth). The emitted electrons have an energy  $E_k$  which is given by equation 1

$$E_k = h\nu - E_b - \Phi \quad (1)$$

where  $h\nu$  is the energy of the incident photon,  $E_b$  the binding energy of the electron relative to the Fermi level of the material and  $\Phi$  the spectrometer work function. The spectrum of the measured kinetic energy gives the spectrum of the binding energy of the photoelectrons.

The x-ray source is also used for exciting secondary X-ray Fluorescence (XRF) for thickness measurement of the deposited TiN overlayers. The second electron gun (0-3 keV) is used measure the SEY, and can also be used to electron condition the surface.

An ion gun is available for cleaning the samples by sputtering and for ion conditioning surfaces.

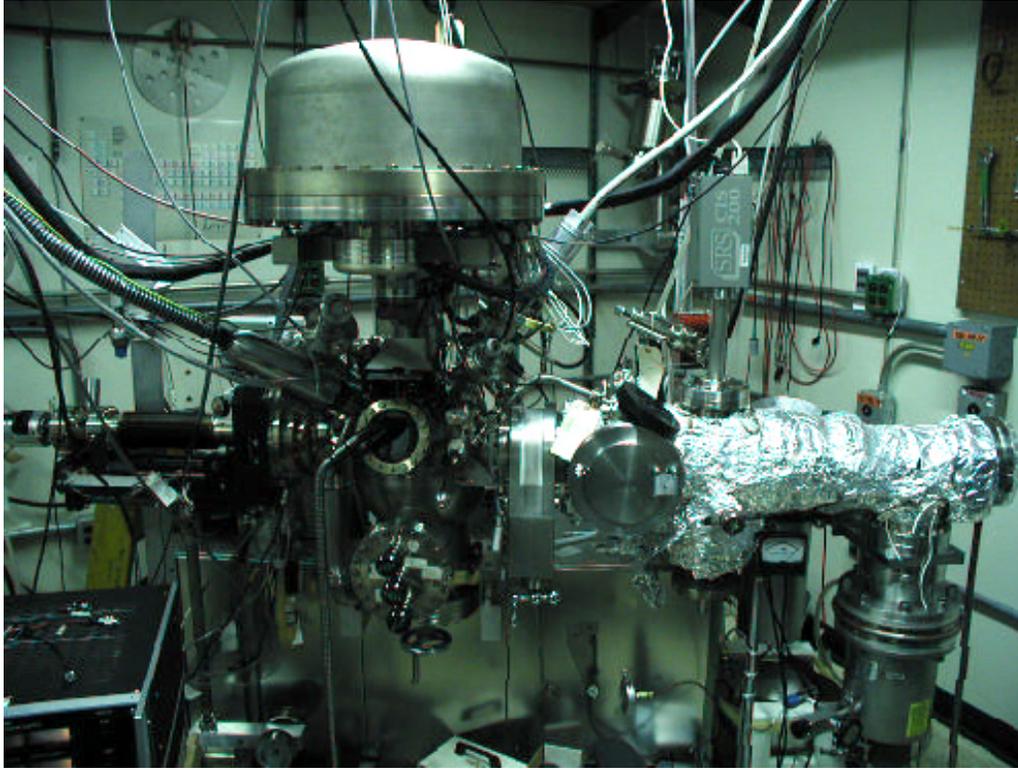


Figure 2: Experimental system used for surface analysis

After all samples (up to ten or so) are transferred into the measurement chamber, one sample at a time is loaded, on its individual carrier plate, onto a manipulator arm (Vacuum Generators "Omniac"). The Omniac<sup>®</sup> carrier plate holder design is shown in Fig.4. Two thermocouples are installed on this holder plate as well as heating filament and sample connection wires. Samples can be heated via a tungsten wire filament, Fig.3, by radiation or by electron bombardment. Electron bombardment is achieved by biasing the filament negatively. The Omniac sample holder is insulated from ground via an alumina ceramic and by several thermal shield plates insulated via four sapphire balls, Fig.3 and Fig.4.

The sample carrier plates are made of molybdenum (for attaining the highest temperatures) or stainless steel, Fig.5. This plate will support one sample which is held by corner screws onto its surface. This design allows the plate to then slide into the rails of the Omniac plate holder, cf Fig.3. An example of an aluminium prototype carrier plate is shown in Fig.6 and Fig.7. Note that on Fig.6 the holes for the screws are not drilled. In this configuration, the thermocouples are not attached directly to the carrier plate but indirectly through the holder plate. Sample temperatures are compared to the thermocouple temperatures by using a black body-calibrated infrared pyrometer ( $0.8 \mu\text{m}$  -  $1.3 \mu\text{m}$  bandpass) which is corrected for absorption by the 7056 glass viewport of the measurement chamber and the sample emissivity ( $\epsilon$ ) of 0.2 - 0.4.

A good way to monitor the activation process of the TiZrV non-evaporable getter

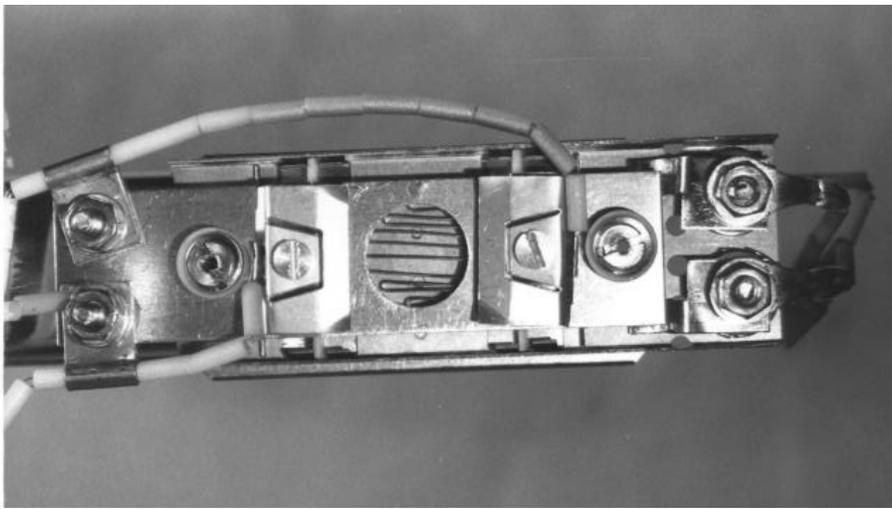


Figure 3: Omniax sample holder plate with heating filament visible

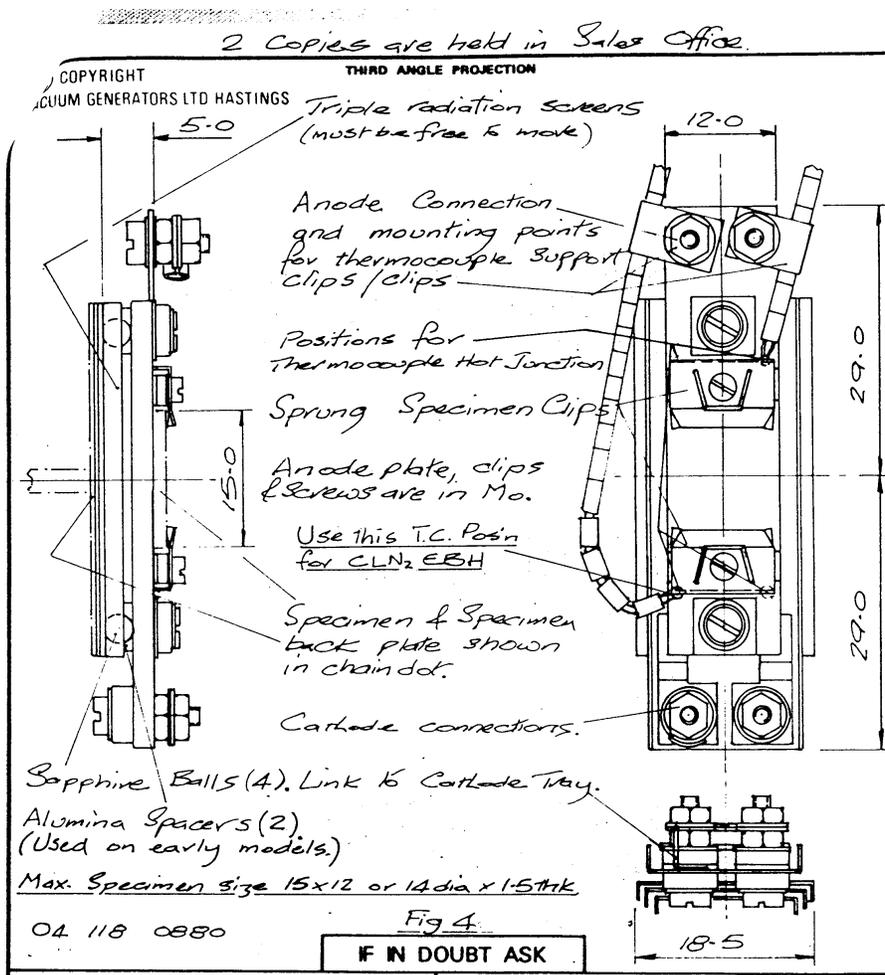


Figure 4: Drawing of the Omniax<sup>®</sup> Sample Holder plate without the heating filament.

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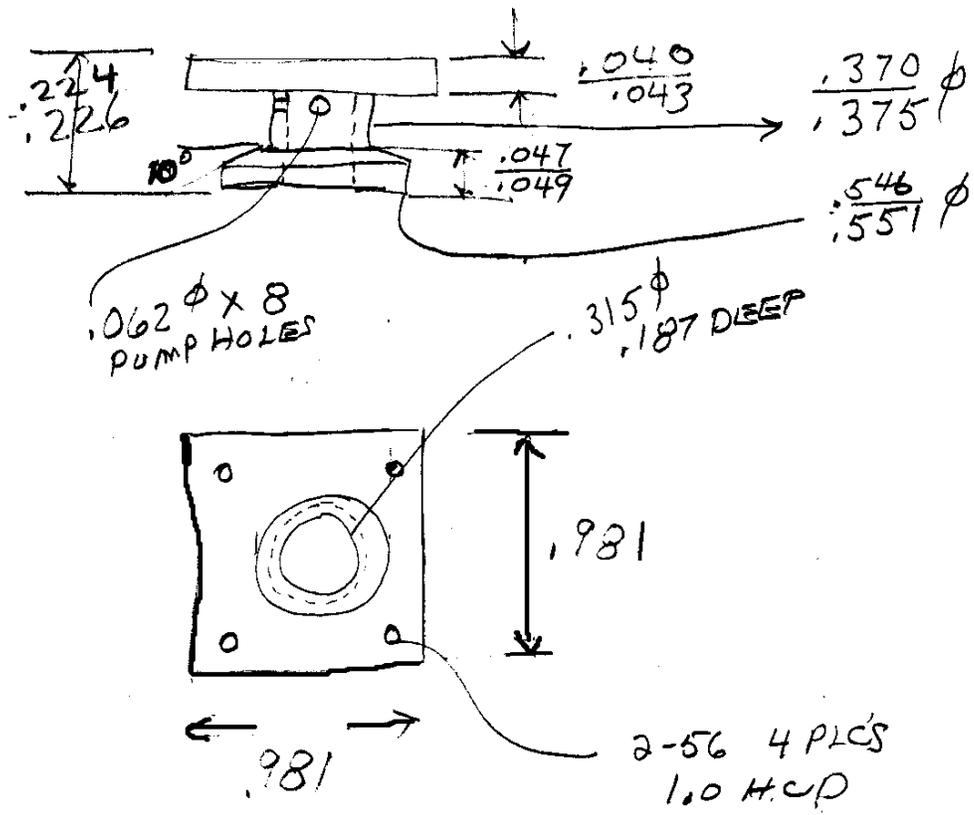


Figure 5: Sketch of the Sample carrier plate made in stainless steel. Dimensions are in inches

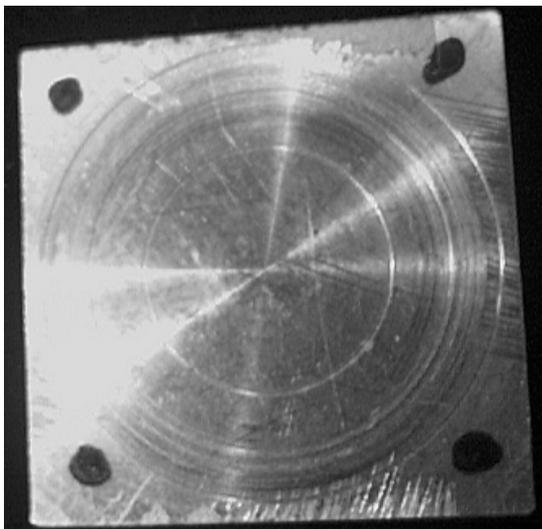


Figure 6: Sample holder prototype in aluminium. The black dots are the location of the screw sample mounting holes

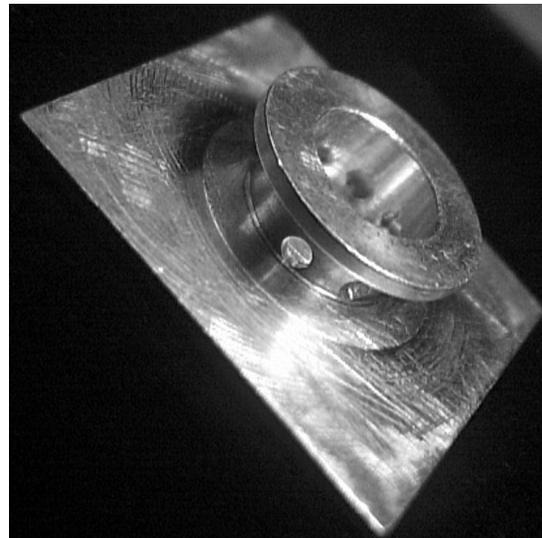


Figure 7: Bottom side of the sample holder

(NEG) is to record the decrease of the surface oxygen concentration with XPS. During the NEG activation, the surface goes from an oxidized state to a partially metallic state. The backside of the NEG was heated, directly via a hole in the plate holder, by electron bombardment. The minimum temperature needed to activate this NEG is 180°C [8] [11]. The thermocouples displayed a temperature of 216°C for 2 hours. In order to determine the temperature of the NEG, a S/S sample holder ( $\epsilon \simeq 0.31$ ) was heated, with a hot plate, to various temperature with a thermocouple spot welded to it, with and without 7056 glass viewport correction. Matching the reading of the pyrometer of this experimental calibration with the reading recorded during an in situ heating of the same S/S sample holder allowed us to determine the actual temperature of the NEG, assuming the same emissivity,  $T_{NEG} = 232^\circ\text{C}$ . An in-situ temperature measurement with the black body calibrated pyrometer gives for the same S/S surface 201°C when the thermocouples on the Omniax carrier plate holder read 216°C.

The experimental electronic circuit is identical to the one presented in Fig. 8 [3]. The computer controlled electron beam coming from the gun is decoupled from the target measurement circuitry. However, the ground is common to both. In short, the target is attached to a biasing voltage source and an electrometer connected in series to the data gathering computer Analog Digital Converter (ADC).

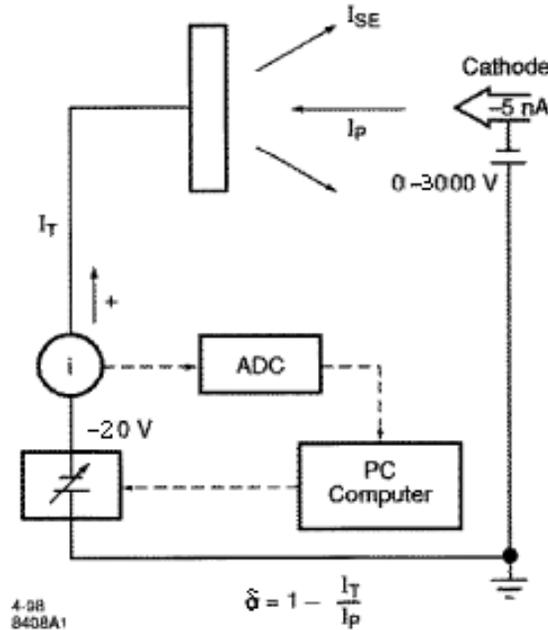


Figure 8: Electronic circuitry used to measure the secondary emission yield

### 3 SEY measurement methodology

In order to characterize our system, we have measured the SEY in-situ of inert gas-ion cleaned materials: carbon, copper and gold. Results are not presented here but agree with widely published results for ion sputter cleaned sample of these materials. After confirming the SEY from these reference materials, we proceeded to measure a TiN-coated

aluminium samples provided by BNL and a TiZrV sputter deposited film on stainless steel substrate, obtained from CERN, Fig.9.

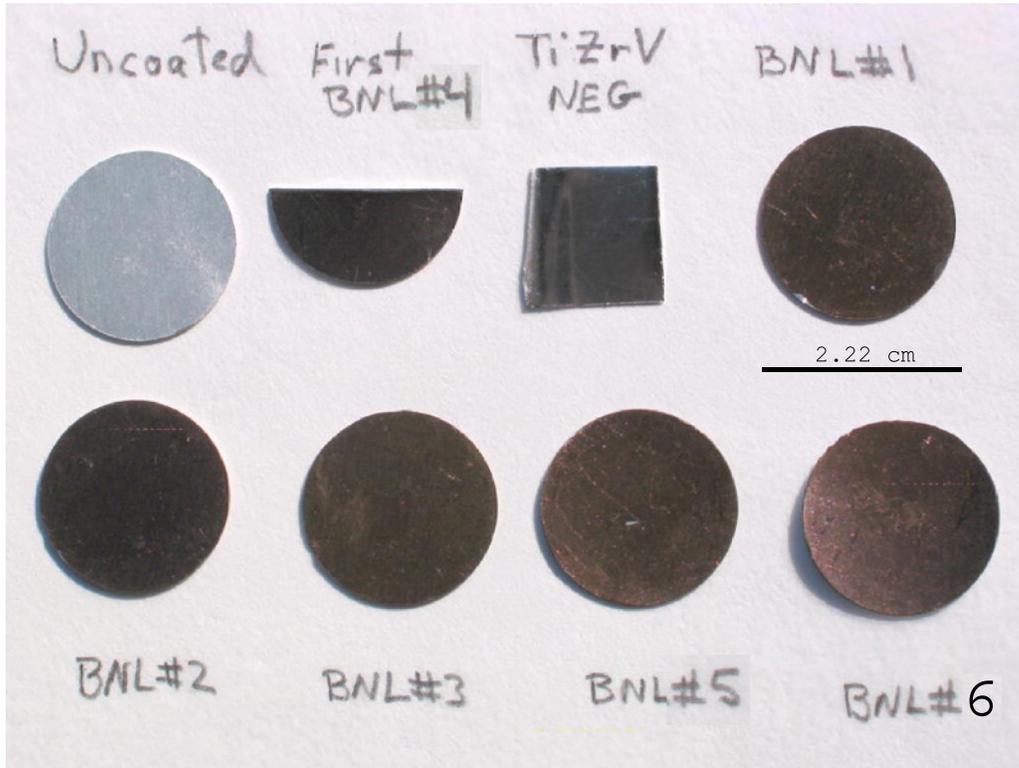


Figure 9: Samples measured thus far.

SEY ( $\delta$ ) definition is given in equation 2. In practice equation 3 is used because it contains parameters measured directly in the experiment.

$$\delta = \frac{\text{Number of electrons leaving the surface}}{\text{Number of incident electrons}} \quad (2) \qquad \delta = 1 - \frac{I_T}{I_P} \quad (3)$$

Where  $I_P$  is the primary current or the current leaving the electron gun and impinging on the surface of the sample and  $I_T$  is the total current measured on the sample ( $I_T = I_P + I_S$ ).  $I_S$  is the secondary electron current leaving the target.

The spectrum of secondary electron current leaving the target is composed of true secondaries (0 eV to 40 eV, by convention), re-diffused primary electrons exiting after suffering losses in the sample (40 eV to  $E_p$ ) and from incident primary electrons ( $E_p$ ) elastically reflected from the surface, Fig.10. The majority of the electrons leaving the surface are true secondaries.

In order to measure the primary current leaving the electron gun, the sample is biased at +150 V. The bias voltage prevents all re-diffused and secondary electrons of less than 150 eV from leaving the sample. Elastically reflected electrons are not collected and could strike nearby surfaces, creating secondary electrons that are then collected by the sample bias. This effect is small because the reflectivity at 100 eV is a few per cent. We estimate the error in primary beam current measurement to be small (1-2%) because the loss of elastics is balanced by the gain of nearby secondaries. The SEY around 100 eV for baked

stainless steel is close to 1.1, Fig.1. With regard to the gun current as a function of energy, it starts at zero for zero energy and smoothly increased to its saturation value at 70 eV. We measure the magnitude and functional dependence of the beam current up to somewhat higher value(100 eV) and use a constructed lookup table of the beam current for SEY calculations. Not biasing the sample with a high enough voltage will lead to an underestimation of the beam current. This is easily understood from the secondary spectrum, Fig.10. The selected 2 nA gun current is measured for a gun energy of 0-100 eV by energy steps of 10 eV (0-3000 eV range) or 2 eV (0-300 eV range).

Actual measurement of the SEY is done by biasing the sample to -20 V. This retarding field repels most secondaries from adjacent parts of the system that are excited by the elastically reflected primary beam. SEY measurements are done twice, once between 0 eV to 3000 eV with 10 eV steps, then between 0 eV to 300 eV, with 2 eV steps. The final energy of the primary electrons is respectively 2980 eV and 280 eV because of the bias. The primary beam current function is measured and recorded each time before an SEY measurement, and with the same step in energy for the electron beam. A fresh current lookup table is created with each measurement.

The purpose of the second measurement, 0 eV to 300 eV, is to try to understand the structure of the SEY curve at very low energy. Several points are important, though.

1. Because of the negative sample bias, primary electrons near 0 eV at the sample are assured to be leaving the gun (20 eV departure) and arriving at (20 eV -20 V) eV.
2. Because of the algorithm used to calculate the SEY from the primary and sample currents at 0 eV incident energy, a divide by 0 blowup occurs. To avoid this problem, the first point at 0 eV is forced to value one. The first "true" data point is at 2 eV for 300 eV range and 10 eV for 3000 eV range.
3. The uncertainty in the sample electrometer current reading is set by the input operational amplifier bias leakage,  $\pm 20$  pA.
4. Using sample current to determine SEY excludes the elastically reflected electrons from the calculation (they would need to be collected by an external to the sample grid structure). That serves to increase the SEY (less sample current) by 1-3%. This is fortuitously balanced by the fact that the -20 V bias does not repel 100% of nearby surface secondaries.

The consequence of all of these points is that the SEY measurements are just that, "secondary". Which means that it does not include the elastics. The SEY measurements are however, accurate. It is important to not look at the SEY at low primary energy and try to conclude something about elastic reflectivity. Data below 20 eV comes from a band structure and are a combination of diffraction from the crystalline structure and energy absorption by the material [12]. Surface effects such as roughness can also change the SEY.

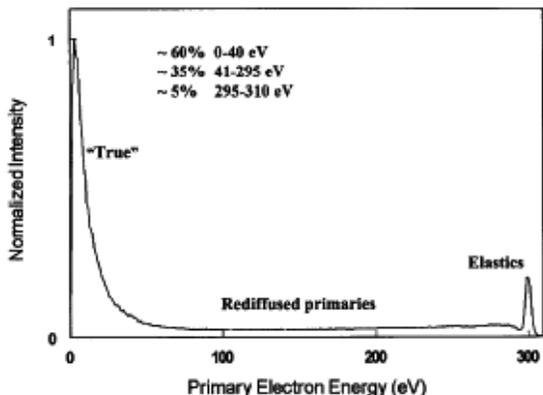


Figure 10: Spectrum of a secondary electron beam from a 300 eV incident primary beam impinging on a TiN on Al substrate sample [13]

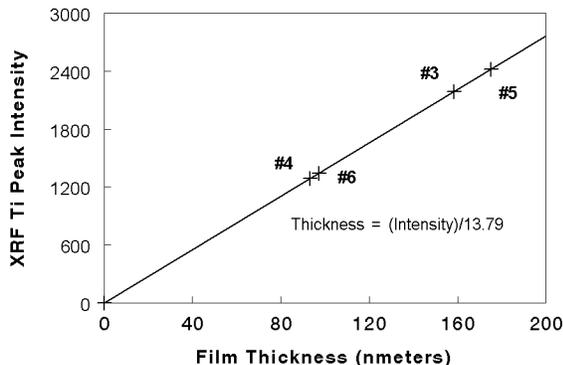


Figure 11: TiN thickness measured by x-ray fluorescence of the Ti K-line of four BNL samples

## 4 Results and Comments

The TiN coating, made at Brookhaven National Laboratory (BNL), was deposited onto aluminium alloy substrates, following the same recipe described in [9]. For the Spallation Neutron Source (SNS) project, the coating was done on S/S. According to BNL, the expected film thickness is around 1000 Å. We measured the actual sample thicknesses using XRF, Fig.11.

The principle of XRF is to collect secondary x-rays generated and exiting the sample when bombarded by primary incident x-rays. The secondary fluorescence yield is highest somewhat above the K-absorption edge of Ti, so primary x-rays of 7 keV are used for the excitation of the Ti- $K_{\alpha}$  line (4.51 keV). A TiN film of similar known thickness (by Rutherford backscatter spectrometry, performed at an outside lab) is used to calibrate the technique.

The height of the measured Ti- $K_{\alpha}$  line, using a Si Li-drifted x-ray detector, is linearly proportional to the number of Ti atoms in the film. Results are shown for a few samples in Fig.11.

SEY measurements results of six different "as received" TiN samples are displayed in Fig.12 and Fig.13. The electron beam impinging onto the surface is of the order of 2 nA over an area of less than a  $\text{mm}^2$ . Typically the beam size is between 0.2 mm to 0.4 mm in diameter. The low current is necessary in order to avoid surface conditioning during SEY measurement. The size of the beam can be checked by using a fluorescent screen, or is inferred from secondary electron microscopical imaging (available on the measurement system and used to precisely choose the point of SEY measurement).

The SEY of the samples varies from 1.5 to 2.5, with the thickest film samples displaying the lower SEY. However, we have no data concerning the roughness, and the roughness can be a factor which can change the SEY. The commonly accepted hypothesis is that, for a given chemical surface, the rougher surface has a lower SEY than a smoother one [4]. The irregularity in the SEY, at near maximum, for sample TiN#4 and TiN#6 can be due to a non uniform spot emitting secondaries with two different yields. The result

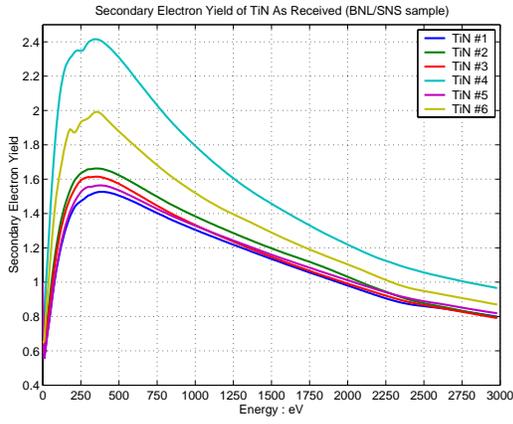


Figure 12: SEY of different TiN sample for electron energy between 0-2980 eV

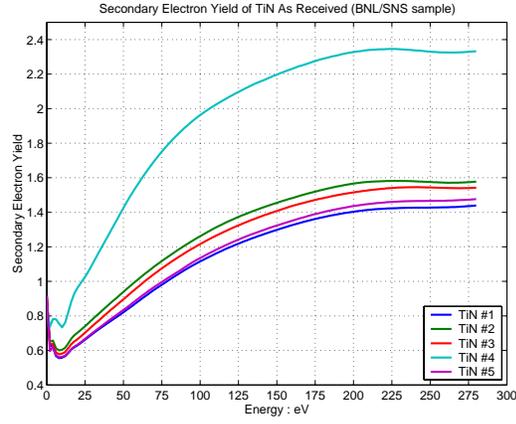


Figure 13: SEY of different TiN sample for electron energy between 0-280 eV.

could be the superposition of the two SEY curves. As the gun appears very reliable in repetitive measurements we do not believe the irregularity is an artifact due to the system. Other measurements of the SEY for unbaked S/S ( $\delta \simeq 2$  at 250 eV) or an as received NEG, Fig.14, do not show these shoulders. XPS survey of TiN#6 shows presence of magnesium. Its expected influence on the SEY is not known.

Finally, an alternative coating to TiN is sputter-deposited TiZrV getter ( $\sim 2\mu\text{m}$  thickness). This NEG, when activated, shows a drastic reduction of its SEY, Fig.14 and Fig.15. These results confirm what has been investigated elsewhere [8]. It is also interesting to follow the behaviour of the SEY curves when the sample is just exposed to only a residual gas background of  $\sim 3.10^{-10}$  Torr for an extended period of time. The SEY of the TiZrV goes up with time when exposed to even such good vacuum. Interestingly enough, it is claimed in [8] that the influence in the SEY of the NEG after exposure to 30 000 L ( $1\text{L} = 10^{-6}$  Torr.s) of CO or CO<sub>2</sub> is rather small.  $\delta_{max}$  will increase from 1.1 (CERN fully activated NEG) to 1.35 (max) while in UHV [8].  $\Delta(\text{SEY})$  is 0.25 using CERN results and is 0.3 for ours,  $1.3 \leq \delta_{max} \leq 1.6$ , Fig.14.

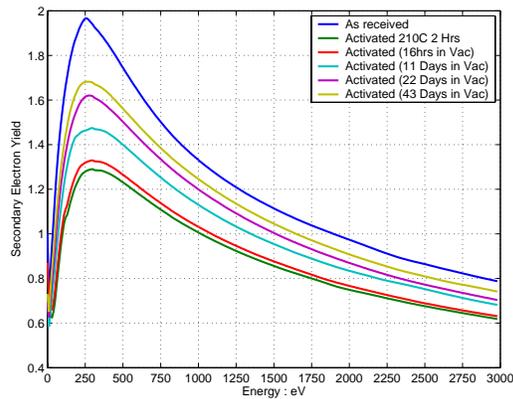


Figure 14: SEY of TiZrV after different process, electron energy between 0-2980 eV

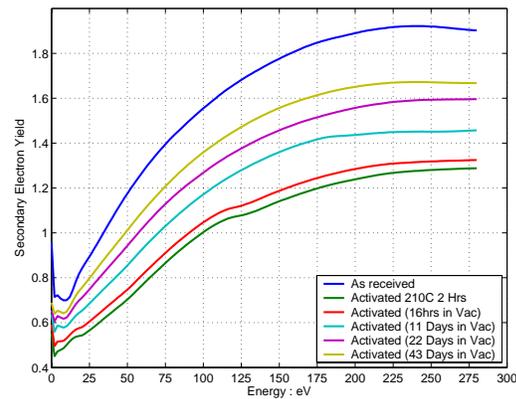


Figure 15: SEY of TiZrV after different process, electron energy between 0-280 eV

If we consider, at first, that our SEY measurement is errorless, one can argue that our activation was not complete. Hence, some initial air formed oxide is still present and the surface chemistry is not identical to a fully activated NEG, hence explaining our  $\delta$  after activation of 1.3. In this case the SEY should still not increase above 1.35 [8] as the getter does not have many pumping sites left. However, this hypothesis might have to be discarded as the minimal temperature we are certain to have achieved is at least 201°C. As a result of a power outage of an hour, during the eleven days of monitoring, an ion gauge, a residual gas analyser and the ion pump were turned off. At the switch on of the gauges, hot filaments release gases in the system before being pumped away by the ion pump. The recorded pressure in the system was at the switch on of the gauges  $\sim 6.10^{-10}$  Torr. This kind of incident also can happen in an accelerator, and it is interesting to see that this effect leads to the recontamination of the getter. Between the days eleven and days twenty, the system was used to XPS other samples. Due to the transfer of samples from the load lock chamber to the measurement chamber, the pressure rose up momentarily to  $\sim 2.10^{-9}$  Torr.

If CO or CO<sub>2</sub> exposure do not seem to affect the increase of  $\delta$ , air exposure does [8]. It is possible that opening our baked measurement chamber ( $P \sim 3.10^{-10}$  Torr) to the load lock chamber ( $P \sim 9.10^{-9}$  Torr), which is frequently open to air, can be consider as an air exposure. Hence, there is no contradiction between CERN [8] and our results.

XPS analysis was carried out to observe the evolution of the carbon chemistry during this 20 days and compared to the XPS spectrum taken after the end of the activation. The XPS spectrum shows a slight rise of the oxygen peak and a double valency carbon 1s peak. The elemental carbon peak has an energy of  $\sim 285$  eV, the oxidized carbon peak has an energy of  $\sim 288$  eV, and the a typical metal-carbide peak is around  $\sim 283$  eV. The oxidized carbon peak after the end of the activation at 288 eV was barely present, Fig.16, blue plot. Only The C peak and Ti-C peak is present, the NEG being at 180°C. A cleanly-scraped carbon surface, measured at room temperature, will only present a peak at 285 eV. After sixteen hours of pumping, green plot, the oxidized state of the carbon shows up, and the carbide peak is gone. This state, oxidized, becomes dominant after eleven days of pumping and keep increasing after 20 days, gray curve. In the last cases the NEG was at room temperature. The plots presented in Fig.16 are a fit of the actual data. This increase of the C-O peak implies that the SEY should also increase, as is usually the case for oxidized metal surfaces.

The roughness (R) of the TiZrV film is unknown. This roughness affects not only the SEY [4] but also the pumping capacity and speed of the NEG. TiZrV deposited on S/S is relatively smooth ( $R \simeq 1$ ) and is much rougher on an aluminium substrate [11]. Let's assume that our system was for twenty days at  $\sim 3.10^{-10}$  Torr, the CO being present at 15% of the total spectrum, and that the sticking coefficient  $\sigma$  for CO is 0.4. The sticking coefficient decreases when the TiZrV NEG has pumped almost half of a monolayer ( $1 ML \sim 10^{15}$  molecules-cm<sup>-2</sup> for a smooth surface) by a factor 10 after reaching 1 ML, and by a factor 100 after 10 ML [11]. A monolayer of a surface (ML<sub>s</sub>) can be defined as :  $ML_s = ML \times R$  [10]. The frequency of collision  $\nu$  (molecules.s<sup>-1</sup>.cm<sup>-2</sup>) equation 4 and the total amount of CO per cm<sup>-2</sup> pumped by the NEG in one day is given by equation 5.

$$\nu = 3.5123 \cdot 10^{22} \frac{P}{\sqrt{M T}} \quad (4) \quad N_{CO} = \nu \times 86400 \times \sigma \quad (5)$$

where P is the pressure in Torr, M the atomic mass and T is the temperature in K.

After one day of pumping the NEG would have pumped  $\sim 0.6$  ML. Assuming that

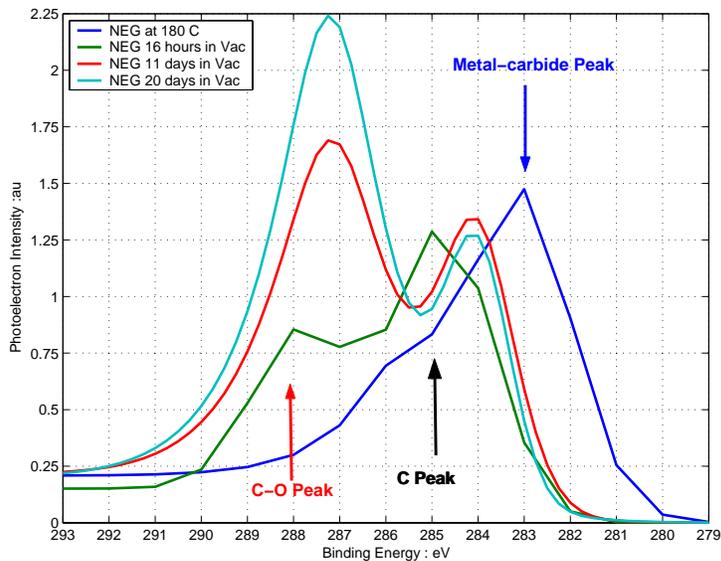


Figure 16: XPS C peak of a TiZrV film after the end of activation

for ten days the sticking coefficient is 0.04, the NEG would have pumped an additional  $\sim 0.6$  ML. According to the rough calculation for the total amount of CO pumped by the NEG, the XPS of 11 days and 20 days should be similar, as the NEG is basically saturated. This increase of the C-O peak, Fig.16, suggests that the surface still had some remaining pumping speed after eleven days of vacuum exposure. Hence having a roughness  $R > 1$ . NEG provides a nice solution for a distributed pumping inside a vacuum chamber, when activated. Our small sample, a few  $\text{cm}^2$ , being at 300 K pumped all of the residual gas in the chamber except  $\text{CH}_4$ . In a few meters long NEG coated chamber the contamination comes from outgassing surfaces outside this chamber, since the NEG film is a diffusion barrier for outgassing molecules of the substrate. Depending of the average vacuum in the machine and the length of the NEG chambers, the recontamination of the surface might take longer than for our sample. Hence, the SEY might not increase as rapidly as measured here, Fig.14 and Fig.15.

Also, it has to be taken into account that the pumping lifetime of thin film NEG depends on its thickness and the number of activation cycles [11]. The influence of the thickness of the film should also be taken into account, when calculating the impedance for the image current on the vacuum chamber wall, due to a passing particle beam. The requirement on the conductivity and the good mechanical performance after an in-situ bake of the substrate is of importance.

For the main damping rings, current design will use aluminium-alloy chamber. Aluminium loses mechanical strength when heated above  $150^\circ\text{C}$ . An adequate substrate for TiN or TiZrV which fulfills the mechanical requirements is the alloy, Al 6061, or Al 6060 which is easier to extrude. Their conductivity is  $\sim 1.4$  times lower than for pure aluminium. SEY measurements on a TiN film deposited on Al 6061 has already been carried out at SLAC [13].

## 5 Conclusion

We have presented a report on the status of the SEY experiment carried out at SLAC. Description of our experimental system has been presented.

First results on as-received TiN sample and on an as-received TiZrV getter have been shown. In the case of the getter, the influence of the activation and recontamination by its pumping action were investigated. The maximum SEY  $\delta$  increased from  $\sim 1.3$  to  $\sim 1.5$  after eleven days and to  $\sim 1.6$  after twenty-two days of exposure to a vacuum of  $\sim 3.10^{-10}$  Torr. Our SEY results seem to disagree with CERN [8]. First of all, our starting  $\delta_{max}$  is 1.3 compare to 1.1 [8]. Second, we have an increase in  $\delta_{max}$  above the CERN-predicted 1.35 [8]. This matter should be investigated further as the implication to electron cloud development is of importance for a positively charged beam running in an accelerator.

Values of  $\delta$  for energies below 20 eV should be used carefully if plugged into simulation. It is planned to investigate further this part of the SEY curve, as has been done at CERN [14].

Additionally, we will study the influence of various treatments, such as the in-situ bakeout of TiN, electron conditioning applied for the NLC case, and also the influence of ion conditioning. By conditioning we mean bombarding the surface with a given spectrum in energy of electrons and ions. Different species of ions can also be investigated. It has to be stressed that conditioning (dose effect) is a very efficient way of lowering the SEY of any technical surfaces to almost the same value ( $\delta = 1.2$ ); and thus independently of the initial  $\delta$  [4]. However, such measurements are time consuming and might not be relevant to the operation of an accelerator, depending on the flux of electrons associated with post-commissioning production operation.

## 6 Acknowledgments

We would like to thank P. He and H.C. Hseuh at BNL for providing the TiN samples and the EST group from C. Benvenuti at CERN for the TiZrV sample. We also thank A. Wolski at LBNL for shepherding the production of sample plates, and in the near future, for thin film samples coming from LBNL. Most valuable was the work of G. Collet and E. Garwin, SLAC, for converting and baking the XPS system for use on SEY measurements.

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