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K. Kennedy, B. Harteneck, G. Millos  
Lawrence Berkeley National Lab, Berkeley, CA 94720

M. Benapfl  
Lawrence Livermore National Lab, Livermore, CA 94550

F. King and R. Kirby  
Stanford Linear Accelerator Center, Stanford University, Stanford CA 94309

### Abstract

The PEP-II Low-Energy Ring will operate at a nominal energy of 3.1 GeV with a positron beam current of 2.1 A. Design parameters for vacuum components are 3.5 GeV at 3 A. The arc vacuum system is based on an aluminum antechamber concept. It consists of 192 pairs of 2 m long magnet chambers and 5.5 m long pumping chambers. Titanium nitride coating of the entire positron duct is needed in order to suppress beam instabilities caused by multipactoring and the "electron-cloud" effect. An extensive R&D program has been conducted to develop coating parameters that give proper stoichiometry and a suitable thickness of TiN. The total secondary emission yield of TiN-coated aluminum coupons has been measured after the samples were exposed to air and again after electron-beam bombardment. A coating facility has been built to cope with the large quantity of production chambers and the very tight schedule requirements.

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The PEP-II Low-Energy Ring will operate at a nominal energy of 3.1 GeV with a positron beam current of 2.1 A. Design parameters for vacuum components are 3.5 GeV at 3 A. The arc vacuum system is based on an aluminum antechamber concept. It consists of 192 pairs of 2 m long magnet chambers and 5.5 m long pumping chambers. Titanium nitride coating of the entire positron duct is needed in order to suppress beam instabilities caused by multipactoring and the "electron-cloud" effect. An extensive R&D program has been conducted to develop coating parameters that give proper stoichiometry and a suitable thickness of TiN. The total secondary emission yield of TiN-coated aluminum coupons has been measured after the samples were exposed to air and again after electron-beam bombardment. A coating facility has been built to cope with the large quantity of production chambers and the very tight schedule requirements.

## 1 Introduction

An instability was observed at the Photon Factory [1] which was later analyzed [2] and attributed to trapped "electron clouds." The origin of these clouds is thought to be the amplification of photo-electrons by secondary electron multiplication at the surface of the vacuum chamber. The tendency for this amplification can be predicted from the total secondary electron yield (SEY) which, for most metals (when averaged over primary energies of 0 to 1000 volts), is slightly above 1. The yield for aluminum, with its native oxide, tends to be much greater than 1 (typically 3-5 at peak yield). The PEP II Low Energy Ring (LER) will have 3 A of positrons with aluminum vacuum chambers in the arcs, conditions which are expected to show the cloud effect.

A SEY-lowering coating is needed on the chamber walls, if electron clouds are to be avoided. This coating will constitute the inner surface of the vacuum system and must have properties consistent with that role. These include: 1) low vapor pressure, 2) moderately good electrical conductivity, 3) good thermal stability and, 4) low ion-induced gas desorption yield. Titanium nitride (TiN) has the needed properties.

## 2 Titanium Nitride

TiN has a boiling point above 3000K and an electrical conductivity above that of titanium. The nitrogen is covalently-bonded to the titanium without destroying electrical conductivity. TiN has been used previously in storage rings, in places where a low SEY is necessary such as rf cavities. TiN can have a SEY that averages below one.

A thin coating of TiN must be put on all surfaces of the arc vacuum chambers that are effected by the electric field of the positrons. The coating must be thin because the thermal expansion of TiN is one third that of aluminum. A thick coating would create high stress between the TiN and the aluminum. The coating must, however, be thick enough to resist "20 years of ion bombardment". A 50 nm TiN film has been calculated to withstand such primarily hydrogen-ion bombardment, using conservative assumptions.

Thermal dissociation of TiN makes an evaporative coating process impractical; however, titanium itself can be deposited by sublimation or sputtering and reacted with nitrogen gas to form a TiN deposit. Sputtered titanium ions arrive with some kinetic energy at the wall and adhere better than thermal energy atoms. This is the preferred choice for a stable coating.

## 3 Vacuum Chambers

The LER vacuum system has six arc regions made of two types of 6063 aluminum alloy vacuum chambers. The profiles are shown in Figure 1. The magnet chamber is 2 m long with a 1.875° bend and the pumping chamber 5.5 m long. The positrons circulate in the elliptical chamber, with their electrical field the largest on the minor axis dropping off rapidly in the antechamber.

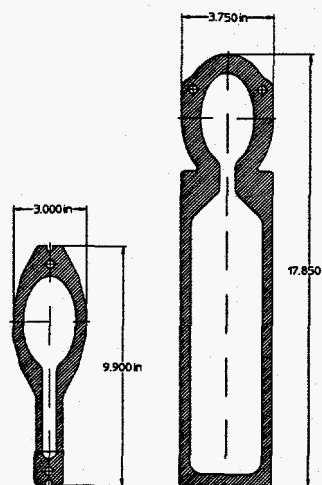


Figure 1: Magnet and Pumping chambers

## 4 Sputtering

### 4.1 Selection Process

The sputtering method chosen needed to be able to readily coat 5.5 m-long chambers with 100 nm or so of TiN with

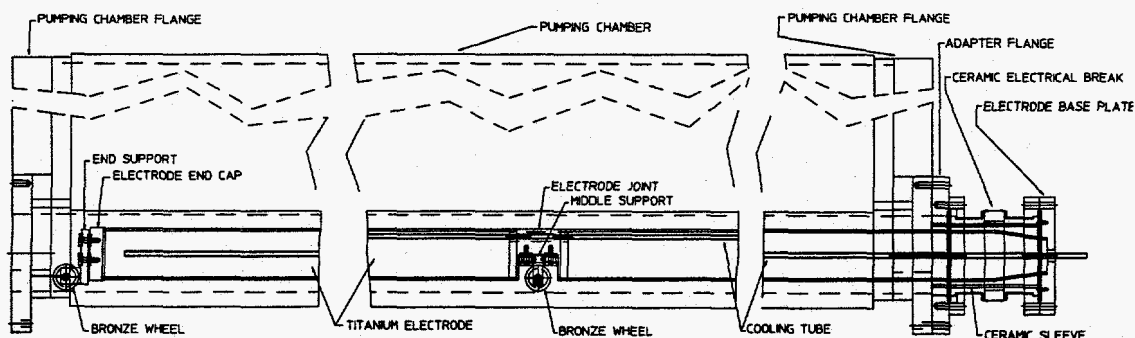


Figure 2: Titanium cathode shown in vacuum chamber

the sputtering source being easily reproducible for high production rate use and with reliable operating parameters. DC diode sputtering with a shaped-crosssection cathode met the requirement. The cathode can be made virtually any length needed provided it can be centered, supported and insulated. The shape of the cathode determines the deposition rate ratio for the major and minor axis.

#### 4.2 Cathode design and fabrication

The sputtering cathode has to reflect the shape of the substrate being coated. To get a uniform coating thickness, however, the perfect cathode shape would take into account the effect of radius of curvature on electric field. A constant gap between the cathode and inside surface of the beam duct gives adequate thickness uniformity and was adopted for our purposes.

The cathode was made by forming a round 48 mm titanium tube in a closed die to make an elliptical tube that would create a uniform annulus with the vacuum chamber. The space left for a sputtering plasma, 15 mm, is very small and created the need to run at relatively high pressure. To increase the gap between the cathode and the vacuum chamber wall by only 5 mm would decrease minor diameter of the cathode to an unusable 15 mm.

The 2 meter cathode could be supported at the ends and have an acceptable deflection in the center. The vacuum feedthrough created one of the insulating supports and an insulating wheel at the end of the electrode was the other support. The 5.5 meter electrode for the pumping chamber needed an additional center support as shown in Figure 2.

#### 4.3 Vacuum system

The vacuum chambers had to be baked before and possibly after coating, so coating and baking facilities were combined. Processing multiple chambers is required to achieve the production goal of 8 chambers per week. The vacuum system was built around a manifold that could accommodate 6 chambers and was pumped by a 250 l/s turbo pump and a 400 l/s ion pump. Attached to the manifold was an ion gauge, an RGA and a capacitance manometer as shown in Figure 3. Up to 6 chambers can be installed over permanently attached electrode. The process gas is introduced to each vacuum chamber at the end opposite the vacuum manifold. The process gas is regulated by a piezoelectric valve controlled by the

capacitance manometer. After the piezoelectric valve reduces the process gas pressure to 250 mTorr the gas passes through a liquid nitrogen trap to remove any water.

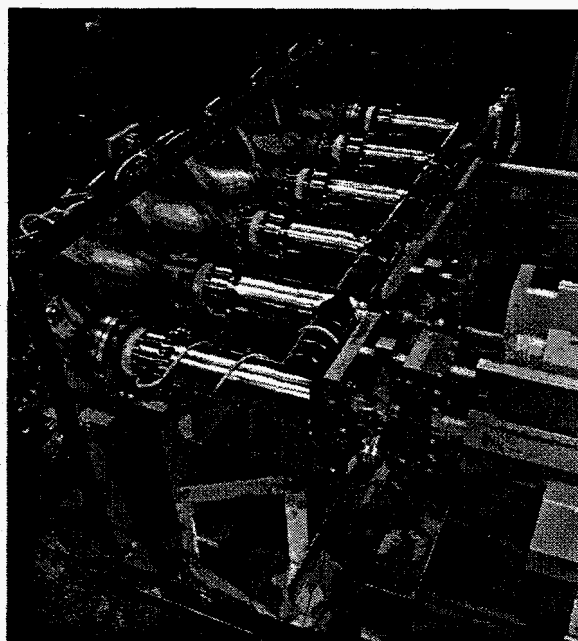


Figure 3: TiN coating oven

#### 4.4 Operation

The vacuum chambers are installed in the coater/bakeout oven, baked into the turbo for 4 hrs and baked into the ion pump for 60 hr. The system is then pumped by the turbo through a 2 mm orifice, the pressure controller is set for 224 mTorr, the premixed 20% N<sub>2</sub>-80% Ar is turned on and liquid N<sub>2</sub> is added to the trap. When pressure equilibrium is established, the electrodes are energized. A single variable-voltage 1500 V power supply powers the multiple electrodes. Each electrode has a 250  $\Omega$  resistor that limits the current from arcs. The power supply voltage is adjusted until the current to each electrode averaged 1.5 A. The current distribution to the electrodes was uniform to about  $\pm 5\%$ . After 16 hours the process gas is turned off and the system pumped by the ion pump to remove any argon and verify that the chamber is leak free.

### Process Parameters

Process Gas Composition	80%A-20% N <sub>2</sub>
Current Density [A/cm <sup>2</sup> ]	2x10 <sup>-4</sup>
Cathode Potential [V]	1000
No. of N Atoms Entering [/sec/cm <sup>2</sup> ]	2.7x10 <sup>+13</sup>
No. of Molc of TiN Formed [/sec/cm <sup>2</sup> ]	1.2x10 <sup>+13</sup>
No. of Ions [/sec/cm <sup>2</sup> ]	1.2x10 <sup>+15</sup>
No. of N <sub>2</sub> Molc. Landing [/sec/cm <sup>2</sup> ]	1.0x10 <sup>+20</sup>
Time to Form a 100 nm Film [sec]	4.3x10 <sup>+4</sup>

## 5. Film Characterization

### 5.1 Stoichiometry

Measurements of the film stoichiometry, thickness and secondary yield were done with sectioned pieces of coated chamber and, routinely, aluminum witness coupons mounted in the beam-position-monitor ports of the chambers being processed/coated. Stoichiometry was measured by x-ray photo-electron spectroscopy (XPS) [3] with Ti/N ratios consistently in the 0.9-1.1 range. The information depth for this method is a few nanometers.

### 5.2 Film Thickness

Film thickness was determined in the same analytical unit by low-energy x-ray fluorescence spectrometry (LEXRF), a technique originally developed [4] to measure ultra-thin silicon oxide layers on silicon. In the present application of this method, the exciting x-ray energy is tuned to just above the absorption energy edge of the titanium K-alpha x-ray emission line, thereby maximizing its fluorescence yield. The absorption length of the now-excited titanium x-ray is large compared to the film thickness and, if suitable TiN thickness standards are available, a linear calibration plot of fluorescence intensity vs. film thickness can be constructed. Then, measuring an unknown film thickness under identical conditions readily gives a non-destructive measurement. Coating runs of 16 hrs gave thicknesses in the range of 90-140 nm, depending on the coupon location in the chamber and the accuracy with which the cathode was radially located. Thicknesses above 30 nm were sufficient to completely obscure the aluminum substrate to XPS.

### 5.3 SEY Measurements

After the as-coated and exposed-to-air non-surface-modifying measurements described above, the coated pieces were transferred to a second system where the SEY measurements [3] were made. The total SEY was measured, for normal incidence, from 50-1000 eV primary energy at very low current density (to minimize surface modification effects). The as-deposited /air-exposed very low-electron-dose yield (Figure 4, inset) is determined by the presence of an adventitious surface water layer. Electron bombardment of this layer, similar to the initial processing expected during machine-commissioning,

desorbs the layer and also causes electron-induced adsorption of carbon from carbon-containing residual-gas molecules. The water desorption and small amount of carbon deposition serve to reduce the peak SEY to near 1 (Figure 4, graph). This yield is near the expected in-situ as-deposited value [3].

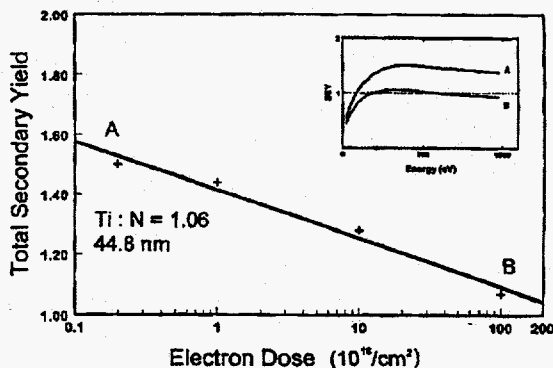


Figure 4: Secondary electron yield as a function of electron dose and primary energy

## REFERENCES

- [1] M. Izawa, Y. Sato and T. Toyomasu, Phys. Rev. Lett. **74**, 5044 (1995).
- [2] K. Ohmi, Proc. EPAC96, Barcelona, Spain, June 10-14, 1996, p. 1042.
- [3] E.L. Garwin, F.K. King, R.E. Kirby and O. Aita, J. Appl. Phys. **61**, 1145 (1987).
- [4] R.E. Kirby, D. Wherry and M. Madden, J. Vac. Sci. Technol. **A11**, 2687 (1993).