

Reduction of Multipactor in RF Ceramic Windows Using a Simple Titanium-Vapor Deposition System.¹

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Abstract

A brief description of multipactor is followed by a discussion of design parameters for a titanium sublimation system. Efforts to correlate operating parameters (time, temperature, etc.) with thickness of coated titanium are reported. Rutherford backscattering thickness measurements are described and reported.

I. INTRODUCTION

The term "multipactor" is commonly used to describe a phenomena which occurs in vacuum, with a radio frequency field and suitable configurations and types of surfaces. If the surfaces have high secondary emission coefficient (especially if > 1), secondary electrons can move synchronously with the rf, typically arriving at a surface one-half a period after leaving a surface, releasing increasing numbers of secondaries which repeat the process. The secondary electron yield is a function of the primary electron energy and incidence angle and varies with the surface material; moreover, is sensitive to temperature and surface contamination [1]. The CRC Press publishes secondary electron emission properties for many elements and compounds, including "crossover" points. At extremely low or extremely high energies, secondary emission coefficients fall below unity; hence, crossover points exist where the secondary emission function (σ) crosses unity. Of particular interest to coupler windows for the Advanced Photon Source (APS) are secondary emission coefficients of alumina and copper, which are listed at $\sigma_{\max} = 2$ to 9 and 1.3, respectively [2].

Since it is possible to dissipate large amounts of power at microwave frequencies and thereby generate destructive thermal-stress gradients, multipactor is clearly undesirable. Furthermore, this power may be deposited in localized regions, leading to increased outgassing or evaporation (and subsequent sputter deposition) of the material. Ultimately, the multipactor loading may become so great that the desired rf voltage cannot be reached or may cause significant reflection. Unfortunately, few elements have low secondary emission characteristics; moreover, many of these are unsuitable for vacuum and/or rf applications. Titanium, with $\sigma_{\max} = 0.9$, and familiarity from its use in sublimation pumps, has become the multipactor-reduction material of choice.

II. SYSTEM DESIGN

Generally, vacuum evaporation is accomplished by heating a small amount of material under vacuum. With

sufficient heating (e.g., using an electron beam), source material will melt, evaporate, then immediately condense on any cool (i.e., room temperature) surface. Elements, such as titanium, that sublime constitute a special case of vacuum evaporation for thin-film production. Materials that sublimate have a vapor pressure sufficiently high that deposition can be achieved without melting. Titanium sublimation lends itself to the simple approach of resistance heating a wire centered in, and coaxial with, our cylindrical alumina window. Accordingly, we chose sublimation over (potentially more complex) sputtering system designs.

Early difficulties with borrowed filaments, reported to be pure titanium, lead us to conclude that a small amount of molybdenum ($< 1\%$) alloyed with titanium would allow the filament to support its own weight while in the sublimation-temperature range. Molybdenum's vapor pressure is sufficiently low that only titanium will evaporate in our system. Interestingly, lowering the pressure decreases the spread between the titanium and molybdenum sublimation temperatures. Increasing the pressure increases the spread, but at the expense of approaching the melting point for titanium. We targeted our operating pressure at 1×10^{-7} Torr, meant to be an optimum in the sense that the sublimation temperature of molybdenum is approximately equal to the melting point of titanium. Hence, our sublimation temperature spread is as large as possible while we are assured that titanium will melt before molybdenum sublimates [3].

Titanium's melting point is 1941 K. At 10^{-7} Torr, its sublimation temperature is approximately 1400 K. Hence, by passing a current through the titanium-molybdenum alloy filament itself, we must heat to at least 1400 K, but no more than 1941 K. The sublimation rate is strongly affected by the operating temperature of the filament. For the purposes of system design, we based subsequent calculations on a filament temperature of 1700 K.

In order to estimate the power supply requirements, an energy balance was performed. Combining equations for resistance as a function of temperature and radiative heat transfer, while ignoring end effects (esp. conduction to filament holders—which will lead to a slightly parabolic temperature profile over the length of the filament) and latent heat of sublimation, the required current is given by:

$$I = \left[\frac{\pi r L \epsilon \sigma (T_2^4 - T_1^4)}{R_{T_1} \{1 + \alpha(T_2 - T_1)\}} \right]^{1/2}$$

I = Required filament current

r = Filament radius

L = Filament length

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- α = Temperature coefficient of electrical resistance
- ϵ = Emissivity
- σ = Stefan-Boltzmann constant
- T_1 = Temperature of surroundings
- T_2 = Filament temperature
- R_{T_1} = Resistance temperature of surroundings

For a 21.6-cm-long x 0.193-cm-diameter titanium-wire filament at a uniform temperature of 1700 K, the required current is calculated to be 34.6 amps. Of course, this is a lower bound; nevertheless, is useful in selection of a constant-current power supply.

Determining the desired titanium thickness involves a trade-off: more titanium is better to suppress multipactor, while too much titanium will result in excessive resistance heating. Saito reports that the effective loss tangent (serving as a metric for excessive heating) of the coating increases “almost exponentially” with the coating thickness [4]. Preist looked at the penetration depth of electrons in the multipactor discharge and found the depth (with electron energy in the range required for $\sigma > 1$) is “of the order of 100 Å” [1]. Elsewhere, the literature reports desired thickness in the range of 15 to 150 Å [4,5,6,7]. We targeted a thickness of 30 Å.

Combining the Langmuir equation for the rate of evaporation [8] with the authors’ derivation of required deposition mass, leads to an estimate of coating time as a function of filament temperature:

$$\tau = \frac{m^*}{G^*} = \frac{t\pi D_c M}{d^3 f_{Nest} f_{Stack} N_A} \left[0.0583 \frac{(\text{gram} \cdot \text{Kelvin})^{1/2}}{\text{cm}^2 \cdot \text{sec} \cdot \text{torr}} \right] \gamma \pi 2r \left(\frac{M}{T} \right)^{1/2}$$

- τ = Time required for coating
- m^* = Mass required per unit length
- G^* = Evaporation rate per unit length
- t = Coating thickness
- M = Molecular weight of filament material
- D_c = Diameter of surface to be coated (I.D. of window)
- d = Covalent diameter of titanium atom
- f_{Nest} = Nesting factor for rows of spheres
- $f_{Stacking}$ = Stacking factor for layers of spheres
- N_A = Avogadro’s constant
- T = Filament temperature
- P = Vapor pressure (at filament temperature)
- γ = Sticking coefficient (=1 for most metals)

For a 30-Å titanium coating on a 10.16-cm diameter surface with the filament at a uniform 1700 K, coating time is estimated to be 99 seconds (neglecting warm-up and cool-down of filament). We considered this to be in an ideal range; that is, is slow enough for control of thickness by adjusting the coating time (without an investment in a sophisticated control system), yet fast enough that contamination will be insignificant. The result is a function of filament temperature not only by way of the temperature term itself, but also due to the associated vapor pressure term. Relatively small changes in temperature of the titanium-molybdenum filament lead to orders of magnitude changes in

the associated vapor pressure. Increasing the filament temperature has an extraordinary impact on coating time; for example, filament temperatures of 1400 or 1800 K lead to calculated coating times of 25 hours or 17 seconds, respectively.

Finally, we planned a bakeable, all-metal system where we could pumpdown to well below 10^{-7} Torr, then bleed in pure nitrogen purge gas. Following a pumpdown to the 10^{-9} -Torr range, the purge gas at 10^{-7} Torr overwhelms any other remaining vapors; hence, we coat in a nearly pure nitrogen environment. Initially, we believed it was important to produce a nearly pure TiN coating. However, the literature indicates that oxygen may be not only unavoidable, but desirable as well. Isagawa reports that such coatings are more precisely described as TiN_xO_y ; furthermore, TiN_xO_y is a “best possible” coating material. Very high resistivity is ensured by the oxygen-rich grain boundary layers of TiN_xO_y [5]. Discussion with colleagues confirms that resistivity increases upon removal of a coated component from its coating system; presumably as atmospheric oxygen combines with the TiN. Nyaiesh describes how, upon exposure to air, the TiN oxidizes 2 to 3 to monolayers of TiO_2 . This TiO_2 layer is subject to decomposition on heating and, since the secondary electron emission coefficient is very different than for TiN, results in a widely varying σ during klystron processing [6].

Our system, shown in Figure 1, was assembled from (primarily) on-hand equipment: knife-edge-flanged vacuum fittings, flange-mounted electrical feed-through, quartz viewport, variable leak valve, magnetic-bearing turbo (without isolation valve), an oil-free roughing system, and borrowed power supply.

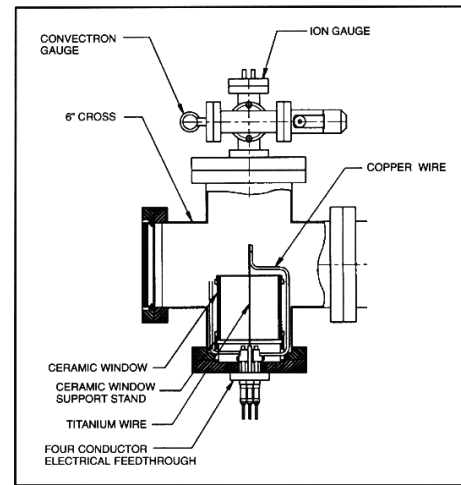


Figure 1: Titanium Sublimation Coating System

III. THICKNESS MEASUREMENT

During early use of the system, a 1-cm-square sample accompanied each window to be coated. These samples were then subjected to a Rutherford backscattering (RBS) analysis for determination of titanium thickness. Using results from

the RBS, current and time were adjusted in a trial and error process.

RBS involves bombarding a sample with high-energy particles (typically ^4He) and measuring the energy of the resulting backscattered particles. Only a small fraction of the incident particles actually have a “collision” with nuclei of the sample; the remainder end up “implanted” in the sample’s space between nuclei. The energy of a backscattered particle, at a given angle, depends on both the loss of energy due to transfer of momentum to the target atom, and losses during transmission through the material before and after “collision.” Because there is a greater change in energy when transferring momentum to a lighter particle, RBS offers greater resolution (spread between energies) for light elements, but gives a stronger signal (higher energy) for heavy elements [9].

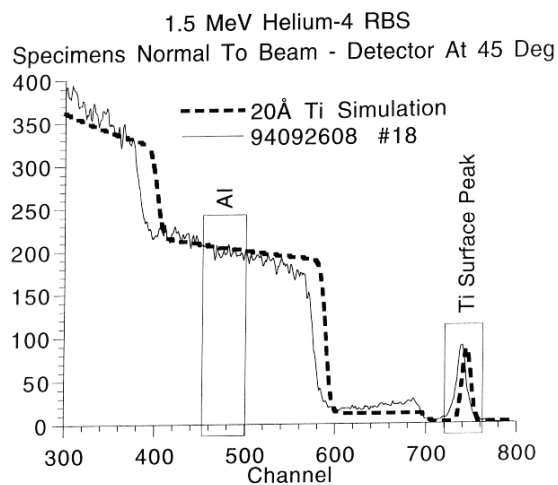


Figure 2: Rutherford Backscattering Plot with Simulation Fit for 20-Å-thick Titanium on Alumina. Vertical axis is number of detected, backscattered particles; hence, is proportional to each elements’ concentration.

For our situation, where a heavier element (Ti) is coated over a lighter one (Al), an RBS plot can be “read” from right to left with a correspondence to the sample surface (however, this is not always the case with RBS). In Figure 2, the peak near 750 keV corresponds to titanium at the surface, while its width corresponds to 20 Å (this is said to be accurate within ± 5 Å and is determined from simulation and/or experience of the RBS scientist). The larger rise, centered about 500 keV, corresponds to the aluminum in the ceramic, with a larger energy spread due to varying losses of energy during transmission to/from random depths.

Additionally, we monitored resistance during coating, with the idea that it might be possible to use a determined value of resistance as the cut-off point where a desired thickness had been reached. Whereas starting resistance was a constant $3 \times 10^{14} \Omega$, measured resistance after coating, but still under vacuum with nitrogen purge, varied from 1.5×10^{11} to $2 \times 10^{12} \Omega$. Accordingly, we concluded that feedback from the RBS measurements, though requiring a longer loop time, was more meaningful.

IV. RESULTS

For coating our coupler windows, we are using a 21.6-cm-long x 0.193-cm-diameter titanium-molybdenum wire with 44.5 amps for 65 seconds. Voltage is nearly constant (despite consuming a small amount of the filament) at 5.1 volts. Measured thickness varies from 20 to 28 Å. To date, we have coated 34 coupler windows. Seventeen couplers have been conditioned up to 100 kW; twelve of those have been subjected to rf power with beamloading; all without difficulty. Each pumpdown requires approximately 20 hours; hence, the system can be operated on a daily cycle—sufficient for our demand. We bake the system only occasionally; specifically, when the ultimate pumpdown pressure begins to degrade..

The system has been in operation for sixteen months, coating (along with coupler windows) rf cavity ceramics for the APS positron accumulator ring, rf field probe cups, beam current monitors, and [chromox ceramic] fluorescent screens with thickness requirements over the range of 10 to 300 Å. In many cases, coating has been requested for prevention of static charge build-up (and possible arcing); rather than protection against multipactor.

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