# METALLIZATION OF SNS RING INJECTION KICKER CERAMIC CHAMBERS\*

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#### Abstract

Ceramic chambers will be used in the pulsed kicker magnets for the injection of H into the Spallation Neutron Source (SNS) accumulator ring, to avoid shielding of a fast-changing external magnetic field by metallic chamber walls and to reduce eddy current heating. The inner surfaces of the ceramic chambers will be coated with a conductive layer, possibly titanium (Ti) or copper (Cu) with a titanium nitride (TiN) overlayer, to reduce the beam coupling impedance, provide passage for beam image current and to reduce the secondary electron yields. This paper describes the development of sputtering method for the 0.83m long 16cm inner diameter (ID) ceramic chambers. Coatings of Ti, Cu and TiN with thickness up to 10µm were produced by means of DC magnetron sputtering. The difficulty of coating insulators was overcome with the introduction of an anode screen. Films with good adhesion, uniform longitudinal thickness, and conductivity were produced.

# 1. INTRODUCTION

The SNS ring injection system is composed of conventional window-frame ferrite-core kicker magnets. The ferrite yokes are mounted around ceramic vacuum chambers metallized on the inside surface. Ceramic chambers are used to allow the external time-varying field to penetrate the vacuum chamber, while a thin metallic coating is required to carry beam-induced image currents.

The ceramic chambers, totaling six for the eight injection kicker magnets, have a 16cm ID and are 0.83m long. Alumina (Al<sub>2</sub>O<sub>3</sub>) ceramic was chosen as vacuum chamber because of its good mechanical strength, excellent UHV properties and ease of brazing to metal flanges. The coating must have a minimum conductivity to avoid attenuation and deformation of the magnetic field. A homogenous and well adherent layer is required to avoid destructive sparks due to the high voltage pulse induced by the pulsed magnetic field. Either Ti or Cu with a TiN overlayer (to reduce the secondary electron yield), have been chosen as the potential coating materials for their high melting point, high sputtering efficiency, low resistivity and good adhesion to the alumina. This paper presents the development of the coating process and the properties of these coatings.

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# 2. COATING METHOD

Magnetron sputtering is currently being used to coat the 4m long SNS half-cell chambers with TiN [1]. A cathode made of Ti tubing of 38mm in diameter was used to produce TiN films of good uniformity and composition. Commercially *Alnico* magnets 32mm diameter and 51mm in length were inserted into the Ti tubing. A 13mm hole in the center of the magnets allowed for the water-cooling of the cathode. The magnets were stacked with opposing poles using non-ferrous 13mm spacers resulted in a looping magnetic field of sufficient strength to project from the cathode surface. This "low cost" cathode powered with a 10 kW DC power supply produce the satisfactory field (0.08T~0.1T) and discharge plasma as shown in Figure 1.

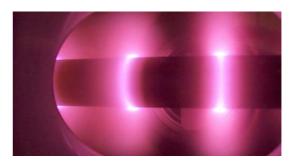


Figure 1. Discharge characteristics of DC magnetron with bright rings at the locations of the magnets.

The uniformity of sputtered films was calculated by treating each plasma ring as a point source. The thickness distribution on a flat surface (i.e. chamber wall) located 8.3 cm away was calculated using a point source cosine distribution [2]. Based on a plasma ring spacing of 6.6cm, the superposition of the individual thickness distributions resulted in an overall uniformity ± 10% (Figure 2). This cathode design was used to coat Cu, Ti or TiN. The coating development was done in a 20cm diameter test chamber (Figure 3) allowing insertion of bare (without end flanges) glass or ceramic tubes.

#### 3. COATING DEVELOPMENT

# 3.1 TiN coating on ceramic tube

The first phase of the development involved coating of a bare ceramic pipe with 6  $\mu$ m of TiN. The 16 cm ID 83cm long pipe was positioned at the midpoint of the test

chamber. The chamber was evacuated and baked to 250  $^{\circ}$ C for 48 hours to remove water vapor and other surface contaminants and maintained at 250  $^{\circ}$ C during the sputtering process to minimize oxide formation on the substrate. Following residual gas analysis to ensure the absence of contaminants, very high purity Ar and N<sub>2</sub> were fed into the test chamber. The flow rates were adjusted using the mass flow controllers and were 13.5 and 7.5 std.cc/minutes for N2 and Ar, respectively. Total pressure in the chamber was  $^{\circ}$ 5 mTorr. The discharge was current limited at 10A. The discharge voltage was  $^{\circ}$  340 VDC. The deposition rate of TiN was  $^{\circ}$  0.07 nm/sec as measured by a quartz crystal thickness monitor.

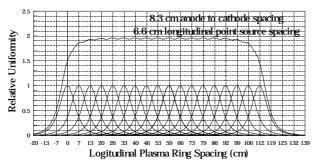


Figure 2. Calculated longitudinal film uniformity based on point source cosine distribution.

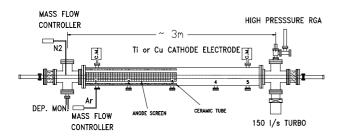


Figure 3. Test chamber with vested anode screen and ceramic tube.

The reactive process was controlled using partial pressure feedback of nitrogen gas [3]. Mass flow controllers were used to introduce both argon and nitrogen, while a high-pressure residual gas analyzer was used to monitor nitrogen partial pressure. The nitrogen was introduced through a 6mm diameter Ti tube attached to the cathode. Small (i.e. ~ 0.2mm diameter) holes located every 15cm provided even distribution of reactive gas, which was necessary for consistent color and composition throughout the length of the chamber.

# 3.2 Difficulties with coating insulators

Initial measurement of TiN coatings on ceramic tube revealed less than ideal thickness uniformity along the length of the tube. The resistance of the film, which is proportional to the coating thickness, was measured using a four-point probe. The thickness distribution derived using a resistivity value of 45 $\mu\Omega$ -cm for TiN, as shown in Fig. 4, was not uniform. It was suspected that a charge buildup on the ceramic had distorted the electrical field and inhibited the discharge within the tube.

To verify this hypothesis, a second run was made using two glass tubes, which were more readily available than the ceramic tubes. The use of glass tubes also enabled viewing of the discharge pattern through view ports located along the test chamber. One tube had two well-grounded 25mm wide aluminum strips running along the inner wall of the glass tube acting as an anode trap. The second tube was installed without these grounding strips. With Ar as plasma medium, only Ti was laid down in this run. With the discharge initiated, there was a familiar, well formed, discharge in the tube with the anode trap, but no visible discharge in the other.

The longitudinal resistance was again measured using the four-point probe. The thickness distribution was derived using a thin film resistivity of 95  $\mu\Omega\text{-cm}$  for Ti. As shown in Figure 4, the thickness distribution of the glass tube without Al grounding stripes, is very similar to that of the ceramic tube. The glass tube with Al strips has much better longitudinal thickness uniformity. This verified our hypothesis that the electric field builds up inside the insulating tube and affects the discharge, consequently the thickness uniformity.

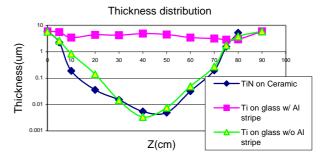


Figure 4. Thickness variation as a result of the charge buildup during coating within the ceramic and glass tubes.

# 3.3 Copper on glass pipes

The overall resistance of the coating on the kicker chamber is to be  $\sim 0.04\Omega$  which requires a thickness of 40  $\mu m$  for Ti or an impractical 160 hours of coating time with a 0.07 nm/s deposition rate. Instead, only a few-hour sputtering is sufficient if Cu is deposited due to its low resistivity. The drawback is that a TiN coating is needed afterwards requiring bleedup and installing a Ti cathode.

A 1.5" diameter OFHC copper tube was used as the cathode in this test. A grounded anode screen made of 304 stainless steel mesh (Figure 5) was inserted in the tube to ensure the uniform distribution of the DC field and enable the conduction of electrical charges. Copper was sputtered in two runs, on to both smooth and etched glass tubes 17 cm in diameter and 91 cm in length. The 10cm diameter anode screen was positioned concentric with the Cu cathode and glass tube. A 90cm magnet strings was centered longitudinally in the glass tube. The discharge was current limited at 5 amps with a discharge voltage of ~ 400 VDC. The deposition rate was ~ 0.56 nm/s. The thickness distribution of the copper film on the glass tubes was found to be within +/- 50% as shown in Fig. 6.



Figure 5. Anode screen shown with Cu cathode, magnet string and coated glass tube.

# 3.4 Copper and TiN on ceramic tube

After successful coating of Cu on the glass tube, 1 $\mu$ m Cu with 0.1 $\mu$ m TiN overlayer was deposited on a ceramic tube of 15cm ID and 50cm in length using the same anode screen shown in Fig.5. The thickness distribution was again measured by a four-point probe and shown in Fig.6. The flange-to-flange resistance is ~ 0.051 $\Omega$ .

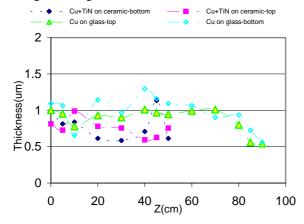


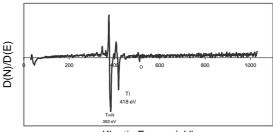
Figure 6. Thickness distribution of Cu and Cu/TiN measured around the circumference of glass and ceramic tubes with anode screen in the tubes.

# 4. FILM PROPERTIES

#### 4.1 TiN Composition

Film stoichiometry and thickness of the coatings were analyzed by Auger electron spectroscopy (AES) using stainless steel witness coupons mounted in the bottom ports along the test chamber. The AES system used is a modified PHI4300 Scanning Auger Microprobe with incident electron beam of 10 keV and 10 nA. Survey scans for all elements (Li to U) were acquired on the asreceived surfaces. Depth profiles were obtained by alternating an acquisition cycle with a sputtering cycle. The sputtering cycles remove material from sample surface using a 4 keV Ar<sup>†</sup> source rastered over a 3x3 mm<sup>2</sup> area. To eliminate crater-wall effect, the data were acquired from a smaller region in the center of the sputtered area. Typical results of AES analysis are shown in Figure 7.

#### **AES Survey Spectra**



Kinetic Energy (eV)

Figure 7. AES analysis of TiN sample, showing little oxygen or carbon contamination and correct stoichiometric ratio of Ti to N.

#### 4.2. Adhesion

The coating adhesion to the substrate surface is one of the most important properties. Two methods have been used for adhesion test. The first one is the simple "scotchtape test" in which adhesive tape is attached to the film and pulled quickly in an attempt to remove the film. The second more demanding test requires scribing a grid of finely spaced cuts through the film down to the substrate, typically a 4x4 mm² grid. This portion of the film is then subjected to the tape test. Both methods were applied to the ceramic tubes and the glass tubes with good adhesion to the ceramic tube and etched glass tube and poor adhesion to the smooth glass surface. It was also observed that adhesion is reduced as thickness increases.

# 5. SUMMARY

DC magnetron sputtering has been developed to coat the SNS ring injection kicker ceramic vacuum chamber with TiN, Ti and Cu. The difficulties in producing a film with uniform thickness within a long insulating chamber have been overcome with the development of a unique cathode with internal magnet string and an anode screen.

# 6. ACKNOWLEDGEMENTS

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# 7. REFERENCES

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