

# CRITICAL ISSUES IN ENSURING REPRODUCIBLE AND RELIABLE DEPOSITION OF NEG COATINGS FOR PARTICLE ACCELERATORS

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## Abstract

Non Evaporable getter (NEG) coating technology, developed at CERN in the late 90s', is an effective pumping solution for conductance limited vacuum chambers. It reduces thermal out-gassing and provides distributed pumping ability, allowing the achievement of very low pressure.

NEG films do show additional interesting features, like low secondary electron yield and low gas de-sorption rates under ions, electrons and photons bombardment.

For these reasons, large scale adoption of NEG coated chambers is now a reality and several leading edge machines will soon benefit from it.

A critical issue for the successful application of this technology is the ability to deposit NEG coatings in a reproducible and reliable way all along a pipe. This is particularly important for narrow-gap or specially shaped chambers which pose severe challenges in term of film thickness distribution, chemical composition and sorption properties.

A dedicated study was carried out to fully understand the deposition process as a function of the sputtering parameters and the chamber geometry. Results obtained do allow optimizing the coating process.

## INTRODUCTION

Hydrogen outgassing from the internal walls of a UHV vacuum system is the major factor limiting the final achievable pressure. Such kind of problem is particularly important for the case of long pipes and vacuum chambers like those of particle accelerators. The use of a new distributed pumping concept in pipes based on Non Evaporable Getter (NEG) films started in 1995 at CERN for the LHC project [1]. Thin film getter coating lowers the pressure in a vacuum system changing the vacuum chamber nature from a gas source to a pump. CERN developed and patented the deposition technique which is now commercially available through the SAES Getters Group under the brand name IntegraTorr. Even if the basic properties of the sputtered getter films were extensively studied [2], only few papers [3,4] described the pumping behaviour and thickness distribution of real pipes or tubulation coated with the TiZrV getter film, where strongly anisotropic conditions are imposed.

In the past works [5,6], a new mathematical approach, based on the angular coefficient method [7], and a new experimental set-up, based on the transmission factor method, for the evaluation of the pumping properties of the whole object (the coated tubulation) have been presented. This measurement method allows the optimization of the process to obtain the best morphology

of the film and then the best pumping speed and capacity. The optimization of the process parameters depends on the particular geometry of the pipe section and must take into account the constraint to obtain uniform deposition on the film. In this work, we will present an analysis of electrical characteristics that can highlight non-optimal conditions and check if the process parameters are enough good to ensure a homogeneous film distribution. A multivariate analysis that uses this method as observable variable allows knowing how the coating uniformity depends on the process parameters and geometry section. Of course, this statistical approach is linked to a robust deterministic interpretation.

## MAGNETRON SPUTTERING CHARACTERISTICS

It is known in the literature [8] how it is important to set the process parameters (pressure  $p$  and magnetic field  $B$ ), in order to run the magnetron system in *abnormal glow* conditions.

In this way, the whole cathode is subject to ion bombardment so that the sputtering process can take place in a more uniform way. In fact:

If the DC discharge is in *normal glow* conditions, only parts of the cathode tend to be emissive or, in the worst case, only diffused spot emissive zone starts to sputter material. In the first case, the deposition shows a continuous meaningful variation of the deposited material along the axial direction. In the second case, lack of homogeneity can be observed, with no particular trend.

If the DC discharge is in *arc condition*, the cathode starts to get hotter and evaporation occurs. Then, the benefic capability of control for the composition and for the low thickness, typical of the sputtering phenomena, is lost.

In the abnormal glow conditions a DC magnetron obeys the current-voltage relation [9]:

$$I = kV_{DC}^n \quad (1)$$

Mapping the conditions around those recommended by CERN ( $1\div 2 \cdot 10^{-2}$  mbar of plasma gas and  $500\div 600$  gauss of magnetic field), we observe that the typical characteristic is actually represented by equation (1), as shown in Fig.1, referred to a 3 mm cathode and a 100 mm diameter pipe.

The exponent  $n$  is in the range  $5\div 15$ .

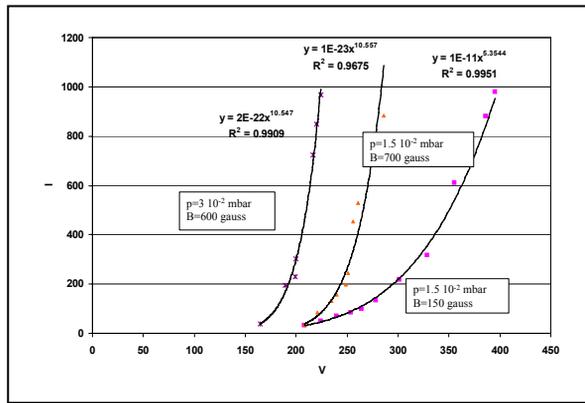


Figure 1: Typical magnetron characteristic.

In this vision, high pressure and high magnetic field should improve deposition uniformity, until arc conditions are achieved or excessive heating of the coating does not affect the sorption properties.

### THE CHARACTERISTIC INSTABILITY

The condition of abnormal glow is not the only one to be considered. It is sufficient only for a long cylindrical pipe.

Otherwise, instability problems can appear although the condition of abnormal glow is achieved. This means that little perturbation in the field or in the geometry can generate big anisotropy in the sputtering process. Example of little perturbation can be a deformation of the cathode or a not perfect relative position between cathode and pipe.

There are two main conditions where instability can appear:

- next to strong variation of the diameter
- if more than one cathode is used

In the first case, fixing the current, the electric field can have a non-uniform distribution and part of the cathode can go back to normal characteristic conditions.

In the second case, although the cathodes are arranged quasi-symmetrically, a non-symmetric solution can be obtained, causing a strong asymmetry for the deposition profile in the section itself.

For sake of example, we consider the case of strong variation of diameter.

A pipe made up by joining different sections has been considered to generate a 218 cm long bi-pipe composed by a part of 105 cm with a diameter of 100 mm and by a part of 113 cm with a diameter of 63 mm. The pieces are joined by flanging. Only one cathode is used just in the center of the pipe.

Fig.2 shows the instability effect at a Kr process pressure of  $1.5 \cdot 10^{-2}$  mbar and 0.1 mbar.

With a coil current of 30 A, the I-V characteristic seems to have a normal shape.

This does not happen with a coil current of 70 A, where the strange curve is the symptom of unstable characteristics. One can observe, in the case of a pressure of about  $1.5 \cdot 10^{-2}$  mbar, three characteristics: between 0-200 mA

between 400-600 mA  
between 800-1000 mA

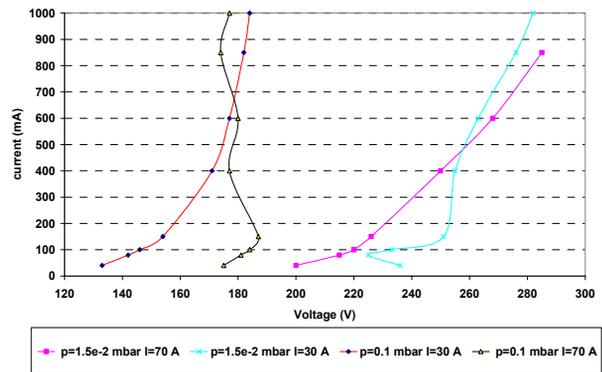


Figure 2: Instability effect at a pressure of  $1.5 \cdot 10^{-2}$  mbar . See text for detail

These are three different characteristic curves joined together. This is not acceptable. In fact, as explained, this shape depends too much on the details of the chamber and process conditions.

Another example is the instability related to the use of more than one cathode. In this case, the process parameters must be optimized not only to consider the diameter variation, but also to control the sputtered material source flux in the different cathodes, that can be non-uniformly distributed. In the same pipe described before, we have measured the I-V characteristics considering three cathodes, as shown in Fig.3.

The three cathodes are placed one in the center and the other two at a distance of 20 mm in symmetric position. The instability is visible at higher magnetic field and lower pressure. The currents in the three cathodes appear also different in the non-optimized case.

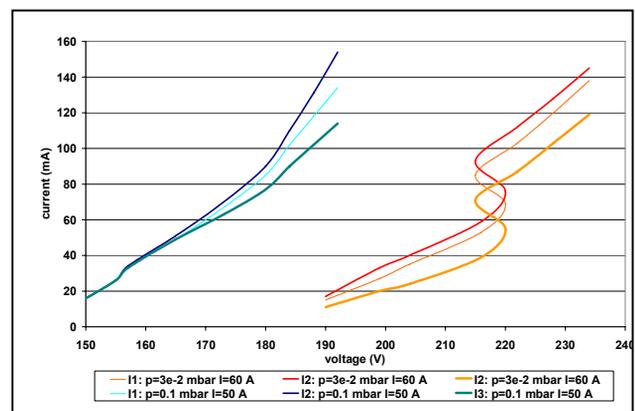


Figure 3: Instability phenomena in the characteristic when three cathode are used

The phenomenon described above is the symptom of a non-uniform sputtering source along the cathode and between the three cathodes.

A multivariate analysis of the I-V characteristic and the deposition profiles allows finding, for every geometry, the best condition of deposition, not only from the abnormal glow point of view.

In order to show that the conditions of abnormal glow are not sufficient to guarantee the absence of instability problems, two depositions have been performed on the pipe with a diameter variation described before with the conditions indicated in table 1.

We have previously checked that the condition of test 1 is of abnormal glow for both pipes with constant diameter 63 mm and constant diameter 100 mm.

Fig. 4 shows the characteristics related to the different process parameters.

Test 1 shows instability while test 2 seems to be optimized for this geometry, because of the better I-V characteristic.

Table 1: Deposition conditions to check uniformity with two different situations.

	Pressure (mbar)	I coil (A)	I cathode (mA)
Test 1	$3 \cdot 10^{-2}$	60	500
Test 2	$10^{-1}$	30	500

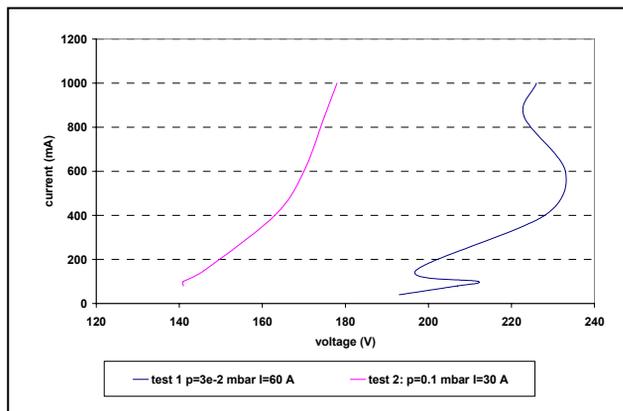


Figure 4: I-V characteristic for the two different tests of Table 1.

Figure 5 shows how test 2, that has better I-V characteristics, produces a uniform sputtering source and therefore a uniform thickness distribution (the thickness is inverse proportional to the diameters).

This experiment shows that the abnormal glow condition is not sufficient to ensure uniform sputtering source as instability phenomena can occur.

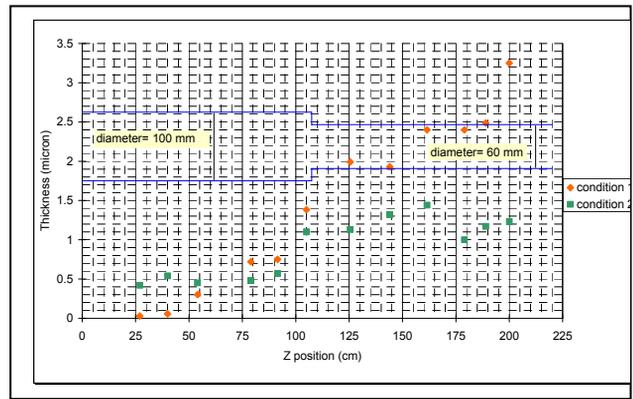


Figure 5: Measured thicknesses on the double pipe, in the different conditions of Table 1.

### CONCLUSIONS

Optimization of the NEG coating for particle accelerators pipes must be set from the point of view of both pumping speed and coating uniformity. Experimental analysis have been performed to evaluate the pumping speed in situ (Transmission Factor Method) and to evaluate the uniformity of the sputtering source. Results demonstrate that abnormal glow conditions are not sufficient to guarantee the uniformity of the source. Instability problems can occur and must be avoided. Multivariate analysis allows to select the best parameters for any different geometry.

This methodology is currently applied by SAES Getters as a part of its quality procedures.

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