Abstract

The approximately 6 km total length of the Large Hadron Collider (LHC) warm sections consist of about 1000 chambers. Most of these chambers, of various length and cross sections, are made of copper, but different lengths in stainless steel, mumetal, aluminium and beryllium will also be used. All the chambers will be internally coated with a thin film of TiZrV, to reduce both the secondary electron yield and the photon and electron stimulated desorption, with the further advantage of providing a huge additional pumping speed. In order to cope with the tight production schedule, a new dedicated coating facility was created, which allows four chambers to be coated per working day. This coating facility and its operating mode are described, together with the TiZrV film characteristics (structure, morphology) and performance (pumping speed, ultimate pressure, discharge gas outgassing).
1 INTRODUCTION

The search for an effective solution to the problem of pumping conductance-limited vacuum systems, as those for particle accelerators, lead, at the end of 1995, to the start of the development of Non Evaporable Getters (NEG) thin film coatings at CERN. Coating the whole inner surface of a vacuum chamber, after dissolving the NEG oxide layer by heating (activation), transforms it from a source of gas into a pump providing very high pumping speed per unit length [1]. The clean surface obtained after thermal activation also reduces significantly the secondary electron yield [2] and the photon [3] and electron induced desorption yield [4]. These features have a capital role on the beam stability and life time of the LHC and of many other accelerators [5].

The heating temperatures for activation of these coatings should be lower than to those allowed by using traditional construction materials, i.e. lower than 400 °C for stainless steel, 250 °C – 300 °C for copper and 200 °C for aluminum. The lowest activation temperature (180 °C for 24 h heating) was found for the TiZrV ternary alloy over a broad range of compositions. This behavior allows best quality TiZrV film coatings to be produced by sputtering from low cost cathodes made of intertwisted wires of the constituent elements.

Because of its favourable characteristics, TiZrV will be extensively applied in the LHC warm sections and in the experiments, as described below.

2 THE TiZrV NEG FILM CHARACTERISTICS AND PERFORMANCE

TiZrV film are produced by cylindrical DC magnetron sputtering [9], with a thickness of about 2 µm and average elemental composition, as assessed by energy dispersive X-ray analysis (EDX), of Ti 30 %, Zr 30 % and V 40 % (at), with an acceptable variation of about ± 20 %.

A nanocrystalline structure (3-5 nm grain size) is obtained, which is correlated with the low activation temperature observed in the TiZrV NEG system [10]. This is presumably due to a large density of grain boundaries which may facilitate the transport of oxygen from the surface to the bulk of the film during activation.

The film surface morphology depends mainly on the substrate surface roughness and on coating substrate temperature [9]. As seen by secondary electron microscopy (SEM), at coating substrate temperatures lower than 250 °C the film is compact and homogeneous, while for higher temperatures an intrinsic roughness appears (see Fig.1). Roughness plays an important role both on surface capacity (which increases with increasing the surface area) and sticking probability (which increases due to multiple molecular surface collisions).

Fig. 1: TiZrV film coated at: a) 100 °C and b) 300 °C
Typical values for the pumping speed per unit area are in the order of 0.5 l s\(^{-1}\) cm\(^{-2}\) for H\(_2\) and of 8 l s\(^{-1}\) cm\(^{-2}\) for CO, with surface capacity for CO of about one monolayer (depending on surface roughness, from 3 \times 10^{14} to about 10^{16} molecules/cm\(^2\) for CO). During the activation process, the oxygen present in the passivation layer is diffused into the bulk of the NEG film, increasing its oxygen concentration, which may induce a progressive degradation of the film performance. This degradation can be counteracted by the increase of the activation temperature. Fig. 2 shows the variation of the hydrogen pumping speed per unit of length of a chamber (diameter of \(\phi = 80\) mm) as a function of the number of activation-air venting cycles.

![Variation of the H\(_2\) pumping speed of a TiZrV NEG film as a function of the number of activation-air venting cycles. The activation temperature is applied during 24 h.](image)

The oxide free surface obtained after the NEG activation presents a secondary electron yield as low as 1.1, low enough to avoid resonant electron multiplication (multipacting) phenomena in the LHC. The photon and electron induced desorption are also strongly attenuated due to the extremely clean NEG surface (about two orders of magnitude).

During the coating process, discharge gas energetic neutrals are implanted in the film. The desorption of this gas, not pumped by NEG materials, may spoil the advantages of NEG films and, therefore a dedicated series of measurement was carried-out. Very low outgassing rates, below \(10^{-17}\) Torr l s\(^{-1}\) cm\(^{-2}\) was measured. The same results were obtained for methane (also not pumped by NEG materials) [11].

NEG films, as for NEG materials in general [12], do not present ultimate pressure intrinsic limitations. Experiments performed at CERN show that if methane and rare gases are removed, the pressures measured down to the low \(10^{-15}\) Torr range are originated by gauge degassing.

### 3 APPLICATION TO LHC

The beam vacuum system interposed between the cryogenic modules of the LHC, called long straight sections (LSS), will operate at room temperature. In order to achieve the optimal operating conditions, most of its components will be coated with TiZrV films. It is the case for the 700 LSS drift space chambers, designated here as LSS standard, and the 285 warm magnet chambers (MQW, MCBW, MBW, MSI, MSD and MBWMD), designated as LSS non-standard.

Some additional chambers (about 50) for the experimental areas of ATLAS, CMS and ALICE will also be coated. These chambers present a wide range of lengths and cross sections, and are made of stainless steel, aluminum alloy and beryllium brazed to stainless steel sleeves. The LSS non-standard chambers also present a wide spread of lengths (from 1 m to 5 m), with circular and elliptical cross sections, and are made of copper, mumetal or stainless steel. With a diameter of 80 mm and lengths from 0.3 m and 7.0 m, and made of copper, the LSS standard chambers represent most of the
long straight sections and are assembled in sectors up to 28 m long and connected to the cryogenic components by an interface comprising valves, vacuum gauges and ion pumps (for the pumping of non reactive gases, as methane and noble gases). The valves permit the isolation of the cold regions in case of air venting of the warm zones and are interlocked with the vacuum gauges for their opening and to the ion pumps for closing. Fig. 3 shows the layout of a typical sector.

At the end of 2002, a work package was attributed to the EST division for the manufacturing of the LSS standard chambers. The construction of a chamber [14] is done in five basic steps: the two stainless steel flanges are each vacuum brazed to two OFE copper stubs which are then welded to an OFS copper tube by using argon arc without filler metal, and leak checked. Finally, the chamber is degreased, about 70 µm of the inner surface are chemically etched to remove a zone damaged during extrusion, and NEG coating is carried out by DC magnetron sputtering in cylindrical configuration. After coating, the chamber is sealed, filled with dry nitrogen and stored. Certificates of each manufacturing step are introduced into the EDMS “traveler” and attached to the chamber’s MTF form. A non conformity in one of the steps generates a non conformity report and the corresponding action is decided case by case.

4 TOWARDS SERIES PRODUCTION

4.1 Coating facilities

For series production, a new coating facility with three coating units was created. One unit is dedicated to the coating of the LSS non standard and experimental chambers, and the other two to the LSS standard chambers. Each unit consists on a vacuum pumping system, a manifold, a base support and a vertical solenoid (see Fig. 4).
Four chambers to be coated are assembled on a cradle. The vacuum pumping system, driven by a rotary-turbomolecular pumping station, is equipped with a discharge gas injection line (Kr as discharge gas), a dry air venting injection line, a pumping by-pass, two cold cathode gauges, and a residual gas analyzer (RGA). An electropneumatic gate valve isolates the system in case of power failure, keeping it under vacuum.

A manifold, supported by a stainless steel structure, allows the pumping of the chambers and provides a rigid mechanical connection to the cradle. Each chamber can be isolated from the rest of the system by a valve. The supports and electrical feedthroughs for the cathodes are located on top of the manifold and are fed by four 1.5 kW DC plasma power supplies (one per cathode). At the other extremity of the cradle, a mechanical support allows the centering of the assembly inside the solenoid. The solenoid is 8 m long, has a 600 mm internal diameter, provides a magnetic field of 150 Gauss and is powered by three 6 kW DC power supplies operated in master-slave configuration. The unit dedicated to the coating of the experimental and LSS non-standard chambers is equipped with a special solenoid providing a magnetic field up to 300 Gauss (powered by four 6 kW DC power supplies also in master-slave configuration and a fifth power supply to compensate for end effects). It offers the possibility of coating independently chamber sections 1.33 m long.

A 16 m long assembling bench allows the horizontal insertion of the cathodes in the chambers. The whole structure can then be lifted up with a crane and inserted into the solenoid, installed inside a 6.3 m deep pit. The vacuum pumping system is installed on a platform located at the top of the solenoid and is connected to the manifold by a bellows.

All the coating parameters, (pressures, residual gas analysis, currents and voltages), are computer monitored, giving a real time view of the global process to the operator.

### 4.2 The coating procedure

To obtain a 2 µm thick film in one working day, a deposition rate of 0.2 µm h⁻¹ has been chosen; it is obtained by imposing a cathode current of 1.5 A and a voltage of -500 V at a pressure of about 5 x 10⁻³ Torr and a magnetic field of 150 Gauss. The chambers are baked overnight at 200 °C and maintained at 100 °C during coating. Pressures on the low 10⁻⁸ Torr are attained at this stage. Each coating unit is able to coat four chambers in 2 working days and the operation of two coating units in parallel allows a maximal production rate of four chambers per working day.

A coating cycle requires a day to mount four chambers and pump down, and one day for coating. Then the chambers are vented with dry air, dismounted, pumped with an independent turbomolecular pumping station, filled with dry nitrogen and stored. In parallel, four new chambers are installed and a new cycle is started. Table 1 shows the working plan of the two units in order to obtain a daily production of four chambers.

<table>
<thead>
<tr>
<th>Table 1. Production plan for the parallel operation of two coating units.</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Coating unit 1</strong></td>
</tr>
<tr>
<td>Day 1</td>
</tr>
<tr>
<td>Check residual gases after coating, dry air venting, dismount chambers, pump down and store under N₂ atmosphere.</td>
</tr>
<tr>
<td>Mount new chambers, pump down and start bakeout.</td>
</tr>
<tr>
<td>Bakeout 8h at 200°C during night</td>
</tr>
<tr>
<td>Day 2</td>
</tr>
<tr>
<td>Check residual gases before coating, inject Kr, start coating.</td>
</tr>
<tr>
<td>Follow up coating parameters, stop coating.</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
</tbody>
</table>

### 4.3 Quality assessment

Before assembling on the coating unit, the quality of the internal surface of each chamber is visually inspected and recorded. If not satisfactory, the chamber is sent back to the surface treatment for further
cleaning. Another visual inspection takes place after the coating and the final state of the surface is described in a coating report.

To ascertain the coating quality, a 15 x 20 mm² copper sample, (submitted to the same surface treatment as for the chambers), is coated with each chamber. For every batch of 4 chambers, one is used for composition (by EDX) and thickness (by SEM) measurements, another to check the sample activation and the surface composition by X-ray photoelectron spectroscopy, and the remaining two are stored for future analysis if requested.

Once a week, (about every 20 chambers), two 25 cm long, 80 mm diameter, stainless steel tubes are coated together with two chambers and then used to check the pumping speed performance of the NEG film. In addition, once per moth of production, one chamber is fully characterized (pumping speed, surface capacity, CH₄ and Kr outgassing) in a dedicate test bench.

Reports of all these tests are introduced into the EDMS and attached to the relevant chamber MTF form.

4.4 State of the production

After production and validation of a pre-series of 10 chambers, the production process was started at the end of February 2004. Figure 5 shows the evolution of the coating production. During the first 8 weeks of production, 113 chambers were coated, representing 1 % of the total.

Fig.5: Evolution of the LSS standard chambers production. The installation is foreseen to start in January 2005.

A coating efficiency (number of coated chambers/(4 chambers per working day)) of 88.3 % was attained. The remaining 11.7 % were due for: 6.7 % to non-optimal internal surface (sent back to surface treatment and re-inserted in the production); 1.6 % were considered non conform to the geometry specification and 3.9 % were delayed for other causes (problems concerning the coating units, etc.).

5 CONCLUSIONS

The NEG film coatings will assure the main pumping for the LHC warm sections and experimental beam pipes. After several years of development, the production phase is started and a maximal rate of about 20 standard LSS chambers is obtained by means of a new coating facility. At present, the production proceeds smoothly at the rate foreseen by the project. The production of the standard chambers is expected to be completed by the end of 2004.

6 REFERENCES


