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**Ultimate pressures achieved in TiZrV sputter-coated
vacuum chambers**

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Abstract

Two metre long, cylindrical vacuum chambers of diameters ranging from 34 to 100 mm, coated with TiZrV getter films by sputtering, have been baked for about 24 h at temperatures from 120 to 250 °C. The ultimate pressures achieved after bakeout were found to correspond to the ratio of the pressure gauge degassing to the effective pumping speed provided by the chamber at the location of the gauge. The results covering a pressure range from 10^{-11} Torr down to 10^{-13} Torr are presented and discussed.

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1. INTRODUCTION

In the framework of the Large Hadron Collider (LHC) project, at the end of 1995 an activity was started at CERN with the aim of producing non-evaporable getter thin films. Within a few months many coatings of different composition were produced, offering activation temperatures lower than 400 °C^{1,2}. At the end of 1997 a ternary alloy of titanium, zirconium and vanadium was found to display full activation after 24 h heating at 200 °C³.

The composition of this alloy, produced by sputtering from a composite cathode made by intertwisting elemental wires, is Ti 30 % Zr 20 % V 50 %. Since its discovery, this alloy has become the object of detailed studies to obtain a complete understanding of its properties. A review of the results achieved so far is presented elsewhere in this conference⁴.

The present report is limited to the ultimate pressures obtained inside TiZrV coated chambers after bakeout and activation of the getter coating.

2. EXPERIMENTAL SET-UP AND PROCEDURES

The experimental set-up consists of a main vacuum system to which the coated test chamber is connected via an orifice of known conductance (25 ls⁻¹ for H₂). The position and the size of the orifice are defined to minimize the gas flowing from the system to the chamber, while providing a sufficient (and known) pumping speed for gases not pumped by the getter. A standard chamber length of 2 m has been adopted with 3 different (internal) diameters, namely 34, 58 and 100 mm.

The vacuum system is pumped by a titanium sublimation pump (SU) cooled by liquid nitrogen and by a sputter-ion pump (SP) of 400 ls⁻¹ pumping speed. During bakeout additional pumping is provided by a turbomolecular pumping station, which is valved off after baking. A leak valve allows the injection of gas for pumping speed evaluation.

The total pressure in the system is measured by means of a Bayard-Alpert gauge (BAG), while a CERN type Helmer gauge⁵ is installed on the chamber extremity opposite to the orifice. This gauge allows pressures down to the 10⁻¹⁴ Torr range⁶ to be measured. Partial pressures are measured by means of a Residual Gas Analyser (RGA) of quadrupole type, usually installed on the main system so as not to upset the chamber vacuum by its high degassing. The RGA was mounted on the chamber on one occasion to obtain a more precise evaluation of the gas release during chamber heating (see section 3).

After installing a new chamber the system is pumped and baked for 24 h (main system at 300 °C, measuring instruments at 350 °C, chamber at 120 °C). The chamber ultimate pressure is measured at room temperature at least 24 h after the end of the bakeout. Then another bakeout is carried out (without exposing the system to air) during which the temperature of the chamber is increased to 150 °C. Ultimate pressure measurements and bakeouts at increasing chamber temperature are repeated, usually up to 250 °C. After each cycle, the measurement of the ultimate pressure is followed by short H₂ injections in order to measure the pressure drop across the chamber and to extract from it the H₂ sticking factor on the getter surface by a Monte Carlo simulation programme. Additional controls are also carried out, as discussed below.

The ultimate pressure measured in the coated chamber may result from many contributions, namely degassing from the getter surface, gas back-streaming from the vacuum system through the orifice, and gauge degassing.

Whatever gas originates from the chamber coating, its degassing should increase with increasing chamber temperature. After measuring the ultimate pressure, the temperature of the chamber is increased sufficiently to evaluate the temperature dependence of the pressure and its

composition. Extrapolating the pressure increase back to room temperature allows a rough estimate of the contribution of the getter degassing to the measured ultimate pressure to be obtained.

As shown by gas injection, hydrogen entering the chamber through the connecting orifice undergoes a pressure attenuation of 2 to 3 orders of magnitude (depending on chamber diameter) on its way to the Helmer gauge. Even larger pressure drops are experienced with heavier gases, such as CO and CO₂. Since the vacuum system pressure is about 10⁻¹² Torr, this contribution to the ultimate pressure is negligible in the context of this study. The effect of back-streaming CH₄ and rare gases, not pumped by the getter coating, may be evaluated by cooling the SU pump to liquid N₂ temperature. Cooling increases the available pumping speed by an amount which may be measured by injecting these gases in turn with the pump cold and at room temperature. By combining the variation of the pumping speed and of the ultimate pressure measured by the Helmer gauge when cooling the pump, a reasonably accurate evaluation of the contribution of these gases to the ultimate pressure may be obtained.

Powering a pressure gauge filament to produce the required ionizing electrons results in enhanced degassing due to heating and bombardment (by electrons and X-rays) of the surrounding surfaces. This effect may be quantified by switching off the filament and measuring the pressure variation under known pumping speed conditions. To minimize this effect, the Helmer gauge is equipped with a thoria coated filament operated at low emission current (3 mA). However, even if the filament is not powered, the stainless steel gauge housing and the transitions required to connect the gauge to the chamber are a steady source of H₂. This gas source may be estimated by multiplying the surface area of these components by the specific degassing rate of the stainless steel used for their construction.

3. RESULTS AND DISCUSSION

The influence of the baking temperature on the ultimate pressure measured for chambers of different diameter is shown in Fig. 1. Complete activation of the getter should result in a constant pumping speed and the data indicate that this is indeed the case at temperatures above 200 °C for all three tubes, but these values are spread over about one order of magnitude. This spread is apparently not correlated to the getter film quality, because the H₂ sticking factors measured for the three chambers after full activation are very similar, i.e. they all fall in the interval $(8.5 \pm 1.5) \times 10^{-3}$.

When heating the 58 mm diameter chamber after its complete activation, the partial pressures vary as shown in Fig. 2. No pressure increase is observed up to 120 °C, then the hydrogen peak starts to increase followed by krypton at 140 °C (krypton is the discharge gas used for the coating), and by CH₄ at 170 °C. Extrapolating these degassing curves down to room temperature allows estimating a contribution of the chamber degassing to the ultimate pressure of the order of 10⁻¹⁸ Torr. Even if the applicability of the linear extrapolation of Fig. 2 is questioned, the room temperature contribution of the chamber degassing is certainly lower than our present measuring limit. Similar results are obtained when applying this same procedure to chambers of different diameter.

Methane or rare gases entering the chamber through the connecting orifice cannot be the cause of the ultimate pressure spread either. These gases, not pumped by getters, should produce the same effect for any chamber diameter. This is also shown by cooling the SU pump with liquid nitrogen. Cooling results in a pumping speed increase of a factor 1.7 for CH₄ and Ar, and produces in the best case a decrease of about 10⁻¹⁴ Torr on the Helmer gauge reading. Hence, backstreaming of CH₄ and Ar give a contribution to the ultimate pressure (measured with the SU pump cold) of about 2×10^{-14} Torr, i.e. negligible in this context.

The measured average degassing rate of the Helmer gauge at 3 mA electron emission current is $(1.1 \pm 0.15) \times 10^{-10}$ Torr ls⁻¹, and consists mainly of H₂. The stainless steel gauge

housing and connecting transitions provide additional H₂ degassing which brings the total to $(1.25 \pm 0.25) \times 10^{-10}$ Torr ls⁻¹ (these values are N₂ equivalent). The larger uncertainty now accounts also for the different surface areas of the different connecting transitions, of which the estimated specific degassing rate is 10^{-13} Torr ls⁻¹ cm⁻². The degassing molecules are pumped with a speed given by the conductance of the chamber aperture multiplied by a pumping probability which depends on the H₂ sticking factor. This dependence, calculated by Monte Carlo simulation, is shown in Fig. 3. Note that the chamber geometry provides large pumping probabilities even for low sticking factors: for H₂, with a sticking factor of about 10^{-2} , 20 % of the molecules entering the chamber are pumped.

Since the sticking factors for H₂ are obtained by gas injection after each baking cycle, it is possible to calculate the chamber pumping speed at the gauge location for each of the measured ultimate pressures. This allows plotting the ultimate pressures versus the corresponding pumping speeds, as shown in Fig. 4. In this same figure are also indicated the contributions to the ultimate pressures produced by the degassing of the Helmer gauge. The close agreement between the two sets of data provides strong evidence that the degassing of the Helmer gauge is fully responsible for the measured ultimate pressures. This conclusion substantiates an hypothesis formulated years ago to justify the ultimate pressure limitation experienced in a similar situation ⁶.

4. CONCLUSIONS

The base pressures measured inside getter coated chambers of various diameter, down to 1×10^{-13} Torr, are an instrumental artefact resulting from the degassing of the measuring instrument, which is however among the best in this respect. If an ideal, non degassing gauge was available, an ultimate pressure of about 2×10^{-14} Torr would be obtained inside chambers of any diameter, due to non reactive gases entering through the connecting orifice in our present experimental set-up. If also this gas source could be eliminated, unmeasurably low pressures would be obtained.

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Figures

Figure 1 - Ultimate pressures measured in chambers of different diameters (D) after 24 h baking at various temperatures. The baking cycles are carried out without intermediate air venting.

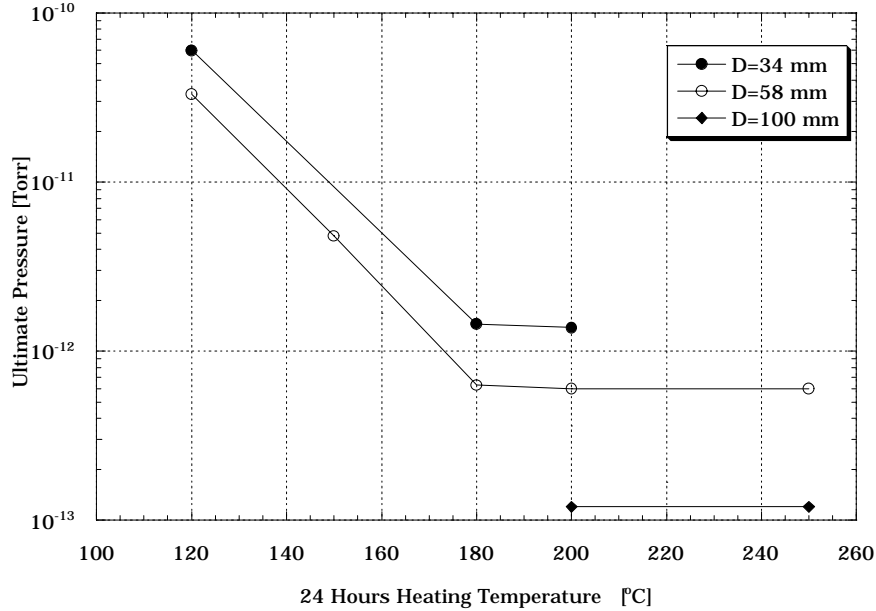


Figure 2 - Partial pressures evolution measured while heating an activated getter-coated chamber (internal diameter 58 mm). The full lines correspond to the measured pressure range, the dotted ones represent the extrapolation to room temperature. The rate of temperature rise is 50 °C/hour. For these measurements the mass spectrometer is installed on the chamber, which is linked to the main vacuum system via an orifice of 25 ls⁻¹ conductance for H₂.

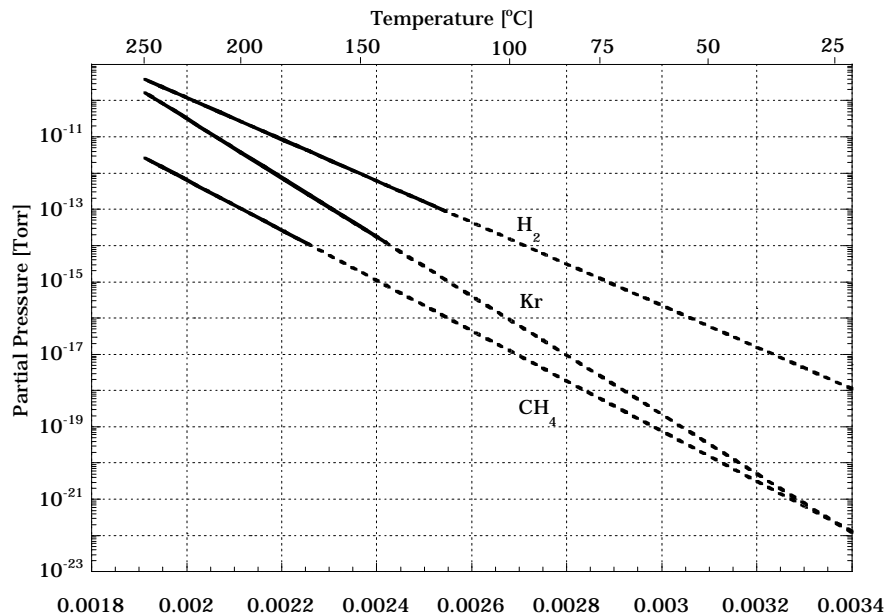


Figure 3 - Variation of the pumping probability as a function of the sticking factor for molecules produced by the pressure gauge and pumped by getter coated chambers. These data are valid only for a chamber length to diameter ratio larger than 20.

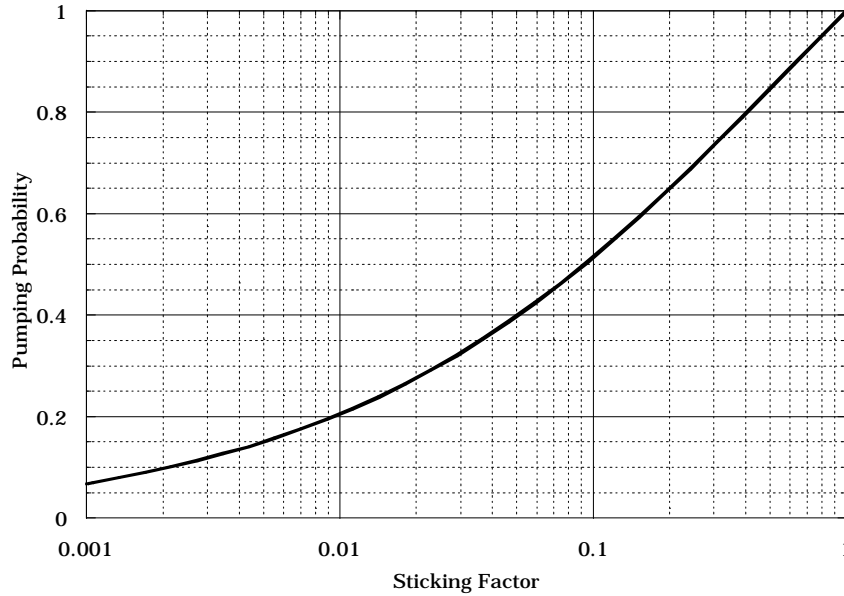


Figure 4 - Variation of the measured ultimate pressures as a function of the H₂ pumping speed provided by the coated chamber at the location of the measuring gauge. The ultimate pressures are measured by the same gauge on 2 m long chambers of different diameter D after different activation procedures. The measuring uncertainty is $\pm 20\%$ for gauge sensitivity calibration and $\pm 3 \times 10^{-14}$ Torr for the gauge residual current. The pumping speeds are calculated by a Monte Carlo simulation programme starting from the H₂ sticking factor measured after each bakeout. The full lines represent the upper and lower limits of the contribution due to the gauge degassing.

