

VACUUM CHARACTERISATION OF A WOVEN CARBON FIBER CRYOSORBER IN PRESENCE OF H₂

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Abstract

Some of the cold bores of the Large Hadron Collider (LHC) will operate at 4.5 K. In these elements, the desorbed H₂ pressure will rapidly reach the saturated vapour pressure, 3 orders of magnitude larger than the design pressure. Therefore, the use of cryosorbers is mandatory to provide the required pumping capacity and pumping speed. The behaviour of a woven carbon fiber to be potentially used as a cryosorber has been studied under H₂ injection. The pumping speed and capacity measured in the range 6 to 30 K are described. Observations made with an electron microscope are shown. A proposed pumping mechanism and the implications for the LHC are discussed.

INTRODUCTION

In the LHC, most of the elements operate at cryogenic temperature. The proton beam stimulates the desorption of molecules by photon or electron irradiation. The design molecular density ensures that the beam life time due to nuclear scattering equals to 100 h. This design density is equivalent to a H₂ pressure at 300 K of 10⁻⁸ Torr.

In the arcs, the vacuum envelope is a cold bore (CB) operating at 1.9 K. A beam screen (BS) is inserted into the CB to intercept the heat load produced by the beam. The perforations inside the BS ensure the vacuum stability and provide an adequate pumping speed. When desorbed or recycled into the gas phase, the molecules are pumped through the slots onto the CB. With the exception of He, at 1.9 K all the gases have a negligible vapour pressure, for this reason, the CB acts as a perfect cryopump.

In the long straight sections (LSS), some CB operate at 1.9 K and others at 4.5 K. After a few days of operation, a few monolayers of H₂ are desorbed and condensed onto the CB. Since the saturated vapour pressure of H₂ at 4.5 K is by far too large as compared to the design pressure, cryosorbers shall be installed onto the BS of the elements operating at 4.5 K. Such cryosorbers shall ensure a minimum pumping speed and a hydrogen capacity compatible with the LHC operation. The required pumping speed is determined by the sticking probability. The required capacity is defined as the surface coverage at which the vapour pressure equals 10⁻⁸ Torr at the operating temperature.

This paper presents the results of the vacuum characterisation of a woven carbon fiber cryosorber.

EXPERIMENTAL SET UP

The woven carbon fiber cryosorber was made in collaboration by the Institute of Solid State Chemistry and Mechanochemistry (ISSCM) and The Budker Institute of

Nuclear Physics (BINP). A sample of 5 cm diameter was glued onto a Cu support. Epoxy glue was chosen for its good thermal conductivity at cryogenic temperature. The sample is screwed onto the head of a cryopump. An indium foil is inserted between the sample and the head to ensure a good thermal contact. The system is connected to a Fisher-Mommsen dome [1]. Figure 1 shows a schematic of the experimental system. The pressure in the vacuum chamber 1, P₁, is measured by a calibrated Bayard-Alpert gauge (BAG). The pressure in the vacuum chamber 2, P₂, is measured by a calibrated BAG and a calibrated Pirani-Penning gauge. A calibrated conductance separates the vacuum chamber 1 and 2. Before the cool down of the cryopump, the system is evacuated within a day to 10⁻⁹ Torr by a turbomolecular pump.

The H₂ is injected in the vacuum chamber 1. The pressure P₂ is defined by the ratio of the injected flow to the cryosorber pumping speed. The flow of injected H₂ is given by the product of the conductance times the pressure difference between the 2 vacuum chambers. The amount of injected H₂ is derived from the integral of the H₂ flow or the reduction of pressure in a reservoir of known volume.

The temperature of the sample is controlled by a heating system. The baffles and a thermal screen minimise the heat load onto the head of the cryopump. The system operates from 6 K to 150 K. Finally, a sample screen is inserted between the second and the first stage of the cryopump. It allows a good definition of the pumping area thereby avoiding any parasitic pumping.

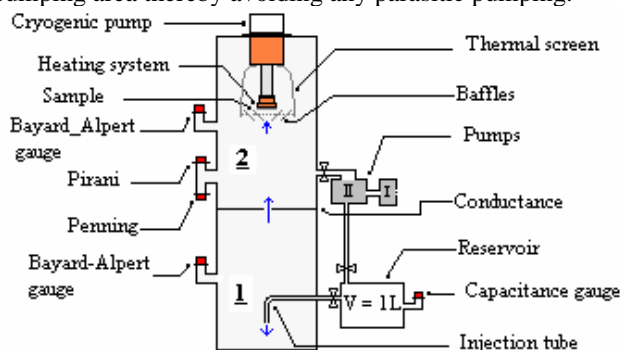


Figure 1 : Fischer-Mommsen dome

The cryosorber is made of carbon fibers, which are weaved altogether. Figure 2 shows two electronic microscope photographs of such a cryosorber. A carbon fiber wire is ~ 1 mm diameter. These wires are weaved. The fibres, which composed the wire, are ~ 10 μm diameter. Each fibre has pores which ranges from 50 to 500 nm diameter. The pumping speed and the capacity are provided by the trapping of the molecules within these pores, which represents about 15 % of the total surface.

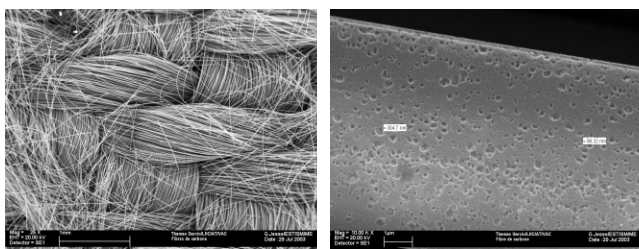


Figure 2 : Photograph of the woven carbon fiber cryosorber. Left x 25, right, x 10000.

RESULTS

Adsorption isotherms

The adsorption isotherm of the carbon fiber was measured for different temperatures in the range 6 to 30 K. For each measurement, a known amount of H₂ is injected into the system while the sample is held at 6 K. At low pressure, a waiting time of ~ 10 h is required to reach the equilibrium pressure defined as the vapour pressure. Due to the higher collision rate of the molecules at higher pressure, this waiting time reduces to a few minutes when the pressure reach 10⁻⁶ Torr. When the equilibrium pressure is reached, the temperature of the sample is slowly increased step by step. Again, some waiting time is required to reach the vapour pressure.

Figure 3 shows the H₂ adsorption isotherm in the 6 to 30 K temperature range. The saturated vapour pressure is reached at surface coverage larger than 10²⁰ H₂/cm². The cryosorber capacity decreases when increasing the temperature. It equals 2 10¹⁸ H₂/cm² at 6K and decrease to 6 10¹⁶ H₂/cm² at 30 K.

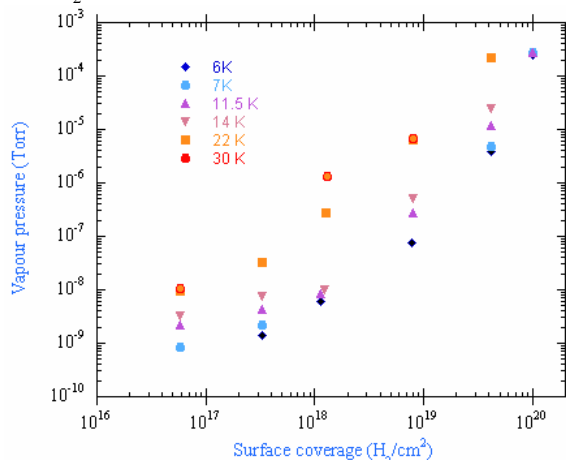


Figure 3 : H₂ adsorption isotherms in the 6 to 30 K temperature range.

Sticking probability

The sticking probability of the carbon fiber was measured for H₂ in the 6 K to 30 K temperature range. At low surface coverage, the sticking probability was measured while injecting permanently H₂. At a surface coverage larger than 10¹⁶ H₂/cm², the sticking probability was measured after the equilibrium pressure was reached.

The sticking probability, σ , is given by equation (1) where S is the measured pumping speed and S_{\max} is the ideal pumping speed. The ideal pumping speed of the cold surface equals 31 l.s⁻¹.cm⁻² for H₂ at 300 K.

$$\sigma = \frac{S}{S_{\max}} \quad (1)$$

The pumping speed is measured while injecting H₂ into the system. It is given by the equation (2) where C is the conductance and ΔP_1 , ΔP_2 are respectively the pressure increase in the vacuum chamber 1 and 2.

$$S = \frac{C(\Delta P_1 - \Delta P_2)}{\Delta P_2} \quad (2)$$

Figure 4 shows the measured sticking probability of H₂ onto the carbon fiber as a function of the surface coverage. At 6 K, initially, the value is close to 1 and decreases to 0.2 at a surface coverage of 10¹⁶ H₂/cm². At larger surface coverage, the sticking probability equals 0.2 in the 6 to 30 K temperature range.

When the cryosorber starts to pump, both the plane surface and the pores of the carbon fibres are pumping. However, for a surface coverage larger than 10¹⁶ H₂/cm², the plane surface stops to pump effectively and the remaining pumping speed is ensured by the pores. This assumption is supported by the fact that the measured sticking probability at large surface coverage is close to the pore surface occupation (15 %). Finally, the pumping speed decrease to zero when the pores start to be filled with H₂.

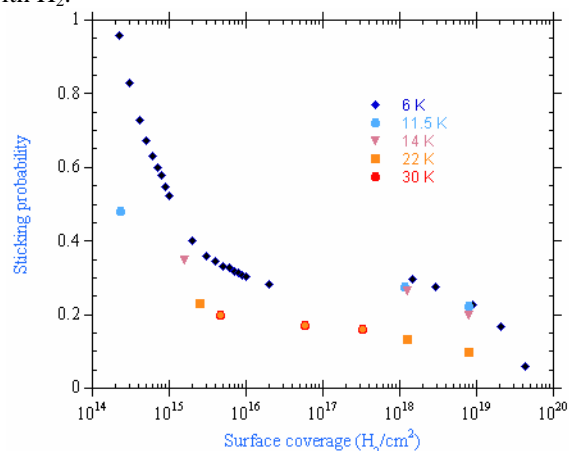


Figure 4 : Sticking probability of the carbon fiber cryosorber, in the 6 to 30 K temperature range, as a function of the H₂ surface coverage

Regeneration and activation energy

During the LHC operation or the machine shutdown, the regeneration of the cryosorbers will be required. Figure 5 shows the H₂ desorption from the cryosorber while its temperature was increasing by 0.5 K/min. Prior to the measurement, 1.4 10¹⁶ H₂/cm² was condensed onto the cryosorber. While the temperature is increasing, the pressure measurement, P₂, made by the BAG is taken over by the Penning gauge. Figure 5 shows that the H₂ desorbs at ~ 80 K from the carbon fiber. So, a

regeneration of the cryosorber implies a control of the BS temperature to ~ 90 K.

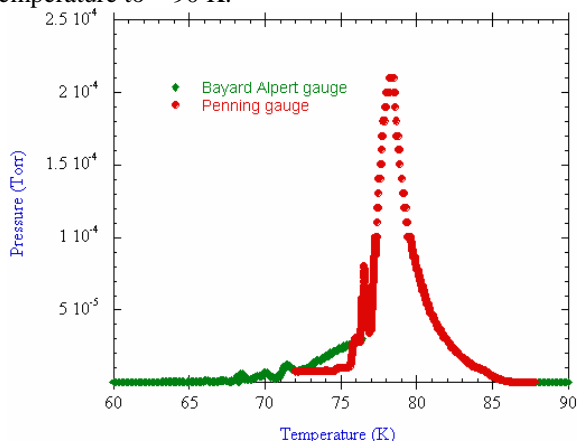


Figure 5 : Thermal desorption spectroscopy of the H_2 condensed onto the carbon fiber cryosorber.

The activation energy for desorption, E_d , can be computed following the first order desorption model which assumes that there is no interaction between the adsorbed molecules during the desorption process. The model applies for physisorbed and non-dissociated chemisorbed molecules. When the heating rate, β , is constant, the activation energy is given by equation (3) with the peak temperature of desorption, T_p , the Frenkel period of vibration, τ_0 ($\tau_0 = 10^{-13}$ s), and the Boltzmann constant, k . The activation energy for desorption of H_2 from the carbon fiber cryosorber is found to be 236 meV. However, the activation energy of cryosorbers varies with the surface coverage and the temperature at which the molecules are pumped [2].

$$\frac{E_d}{kT_p^2} = \frac{1}{\tau_0 \beta} \exp\left(-\frac{E_d}{kT_p}\right) \quad (3)$$

IMPLICATIONS FOR THE LHC

The capacity and the pumping speed provided by the cryosorber shall be within the LSS vacuum requirements [3]. The vacuum performance of the LSS cryosorber will depend on the total quantity of installed material and the material itself. A technological limitation will come from the available space between the BS and the CB. Assuming the installation of carbon fibres onto the top and the bottom of the BS, about $200 \text{ cm}^2/\text{m}$ of cryosorber could be installed. In such a configuration, the minimum sticking probability is above 15 % and the minimum capacity above $10^{18} \text{ H}_2/\text{cm}^2$.

In the LSS, the installed pumping speed is expected to be large enough to provide a similar pumping speed as in the arcs. During the first year of LHC operation without electron cloud, the gas load will be well within the carbon fiber capacity. Therefore, no regeneration is expected. It has been estimated that $3 \cdot 10^{20} \text{ H}_2/\text{m}$ per year will be desorbed when the LHC will operate in the electron cloud regime. If the BS of the LSS will operate above 15 K, several reconditioning of the carbon fiber cryosorber will

be required. Operation of the BS above 20 K will require more than 3 reconditioning per year. Moreover, similarly to the arcs case, a beam conditioning will be required to ensure the design life time.

The above prediction assumes that the cryosorber performance will not be altered by the quality of the thermal anchoring of the carbon fiber onto its support. The thermal conductivity of the proposed attachment as compared to the glue will be probably lower. However, it is believed that, due to the surrounding CB, no major consequences are to be expected.

Figure 2 showed that the cryosorber is made of thousands of fibres of a few micrometer diameter. Its manipulation leaves the fingers black. While inserting the BS or during the ageing of the cryosorber some dust might be produced. As a consequence, the reliability of the valves or the ion pumps might be altered [4]. The presence of dust in a machine is a delicate question, which might have significant consequences. As an example, it is known that the dust in a superconducting cavities acts as a field emitter thereby reducing its performance [5].

CONCLUSIONS

An experiment has been set up to measure the vacuum performance of LHC cryosorber candidates. The woven carbon fiber produced by a collaboration between the ISSCM and BINP was tested.

The H_2 isotherm and the pumping speed were measured in the 6 to 30 K temperature range. The activation energy for desorption was found to be 236 meV. In the case of a regeneration of the carbon fiber cryosorber, a control of the BS temperature to 90 K is required.

The vacuum performance of this cryosorber was found to be within the LHC vacuum requirements. It has been estimated that when the LHC operates in the electron cloud regime, a few regeneration per year will be required in the case the BS operates above 15 K.

The potential presence of dust released by the cryosorber and the associated reliability of some vacuum components might be an obstacle to the installation of this material in the LHC LSS beam vacuum system.

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