# SUMMARY OF RESENT STUDIES OF CRYOSORBERS FOR LHC LONG STRAIGHT SECTIONS\*

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#### Abstract

The vacuum chamber inside some cryogenic elements in the Long Straight Sections of Large Hadron Collider (CERN, Switzerland) will have cold bore at 4.5 K and a beam screen at temperature between 5 and 20 K. In such chamber the desorbed molecules of  $H_2$  will be pumped through pumping holes on the beam screen onto a cryosorber placed between the cold bore and beam screen. The search of perspective cryosorber for collider is described in this paper.

#### **INTRODUCTION**

The major part of vacuum chamber inside some cryogenic elements in the Long Straight Sections (LSS) of Large Hadron Collider (LHC) will have a cold bore (vacuum envelope) at 4.5 K and a so-called "beam screen" maintained at a temperature between 5 and 20 K by a flow of gaseous helium. A beam screen (BS) will be inserted into the cold bore (CB) to protect the CB against synchrotron radiation, electrons and ions exposure [1]. The gas molecules desorbed due to photons and electrons will pass through the holes on the BS to the shadowed part between the CB and BS (Fig. 1). All desorbed gases except  $H_2$  could be adsorbed on the CB and BS but a cryosorber is required to pump  $H_2$ . Cryosorber should be mounted on the shadowed (i.e. outer) surface of the BS.



Figure 1: Cross section of LHC cold vacuum chamber.

The most of the published information about cryosorber for  $H_2$  concerns the studies of the cryosorption properties of standard materials at the temperature of boiling liquid nitrogen (~77K), liquid hydrogen (~20K) and liquid helium (~4.2K). There is a requirement to find suitable cryosorbers with large adsorption capacity and certain pumping speed for  $H_2$  in the temperature range between 5 and 20 K. The cryosorber material for LHC must have the low regeneration temperature, the size in order to fit into the limited space available and it should be easy to mount.

The geometric estimation indicates that four 5-mm wide strips of cryosorber could be installed onto the BS.

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So,  $H_2$  adsorption capacity of cryosorber should be no less than  $10^{17}$  molecules/cm<sup>2</sup> for LSS LHC [2]. It imposes the strong restrictions to choice of cryosorber material. To evaluate the gas density inside such vacuum chamber it is very important to know the dynamic pressure behavior,  $H_2$  capacity and  $H_2$  pumping speed of the BS with cryosorber in the real LHC LSS beam pipe configuration.

This paper includes the summary results of resent studies of cryosorbers to use them for distributed pumping in the cold parts of LHC LSS vacuum chamber. These studies were performed in the Budker Institute of Nuclear Physics (BINP) in the framework of collaboration with CERN. With aim of search of perspective cryosorbers for use inside the collider vacuum chamber, the new types of anodized aluminum, porous copper and charcoal-based materials were developed and studied to cryopump H<sub>2</sub> at temperatures between 10 and 20K. The advantages and disadvantages of cryosorbers and technological problems of development of new similar cryosorbers were defined. The vacuum parameters of LHC vacuum chamber prototypes with charcoal and two types of carbon fibre cryosorbers were measured.

A main result is that woven carbon fibre cryosorber meets the LHC requirements and can be proposed as cryosorber for LHC. The dynamic pressure behavior at BS temperature oscillations was also studied for BS with woven carbon fibre to predict the dynamic pressure at nonstandard or transient regimes of the LHC operation.

# SEARCH OF A PERSPECTIVE CRYOSORBING MATERIAL

### Survey of the well-known cryosorbers

Charcoal is a well-known cryosorber and has quite a high adsorption capacity. However, charcoal has some disadvantages for use inside the vacuum chamber of an accelerator: it is space-consuming, dusty and difficult to bond to surfaces and fit shapes. Cryosorber on basis of charcoal with platinum requires regeneration at 300°C.

The zeolites and silica gel filters are the less effective adsorbent than charcoal and have the bad handling abilities. Moreover, zeolites have the additional disadvantage, which is the great dependence of sorption capacities on cleanness of the zeolite surface porous (especially water is the danger). Therefore they are needed to bake at high temperature during long time.

Non-evaporated getter films require the activation at high temperatures. The sorption capacity of the stainless-steel sample coated with TiZrV getter film is little for use at low temperature [3].

It was reported that up to 200 monolayers of  $H_2$  could be adsorbed on anodized aluminum at 4.3 K [4]. However other experimental study of the different cryosorbing materials at 4.2 K [3] showed that the capacities of metallic foams, filters, anodized aluminum and some other materials are low for the LHC and only the charcoal capacity is high enough. The large sorption capacity for anodised aluminium measured by Rao et al. [4] could be explained by the very low  $H_2$  injection rate. However the temperatures of samples in those studies were lower than expected temperature of cryosorber in the LHC.

### Development of the new types of cryosorbers

New types of anodized aluminum, porous copper and charcoal-based materials were developed in collaboration with a number of research institutes and were studied to cryopump  $H_2$  at temperatures between 10 and 20K. These studies are described in detail in ref. [5].

Porous copper and anodized aluminum were chosen due to their good properties for vacuum use. Several plate aluminum samples were anodized using the microplasma method because this method results in a thick  $Al_2O_3$  film. The electrolytic anodization combines with plasma anodization in this method. The formation process of the porous film made by this method is not well understood due to a number of variable process parameters.

Several porous copper samples with different pore (grain) sizes were also made from dendritic structure copper powder with use of advanced technology [5].

Mold pressed charcoal was chosen to make a cryosorber with the defined geometric shape and the good sorbing properties of charcoal. Several charcoal samples were made by mould pressing of a mixture of powders from stamped charcoal and different stickers.

A carbon fibre cryosorber was proposed to meet the requirements of a cryosorber for the LHC vacuum chamber [6]. Manufacturing of carbon fibre based on regenerated cellulose similar to rayon. Cryosorber can be made as a non-woven fabric as well as a woven material.

The results of specific H<sub>2</sub> pumping speed measurements are shown in Fig. 2 at different temperatures as a function of the number of adsorbed molecules in terms of surface density (molecules/cm<sup>2</sup>) for anodized aluminum (AA3), porous copper (Cu1), charcoal with the high temperature superconductor ceramics sticker (C+SCC) and nonwoven carbon fibre fabric (CF) covered special stainless steel net (array pitch of 100-mkm and optical transparency of 50%) for fast cooling of the sample under laboratory conditions. Sample temperatures were chosen in the range between 5 and 20 K. Here the pumping speeds S, m<sup>3</sup>/s, in cold chamber are multiplied by a Knudsen factor  $(T_{RT}/T_{crvo})^{1/2}$  $(T_{RT}, K$  – room temperature of gas near the gauge,  $T_{cryo}, K$ - temperature of gas inside the cold chamber). Reduction of pumping speed defines the adsorption capacity.

The results are shown only for one of anodised aluminium samples and for one of porous copper samples. However, these results characterize the obtained adsorption capacities of these materials.

Only charcoal with the high temperature superconductor ceramics (SCC) sticker was studied for adsorption properties. The SCC was added into the sample as an adhesive element between the particles of charcoal powder for fast cooling of the sample under laboratory conditions. The adsorption capacity of same sample depends on percentage of charcoal in the sample.



Figure 2: Dependences of pumping speeds of studied cryosorbers on number of molecules of adsorbed  $H_2$ .

Comparing all the results one can see that the specific adsorption capacities for  $H_2$  were measured as more than  $10^{17}$  molecules/cm<sup>2</sup> for the charcoal and carbon fibre samples and equal or less than  $10^{17}$  molecules/cm<sup>2</sup> for the other samples (taken at the same pumping speed as the charcoal sample). Thus, only charcoal and carbon fibre meet to LHC capacity requirement.

Presumably, the pumping parameters of anodized aluminium and porous copper may be improved. The growth of  $Al_2O_3$  film becomes slower after attainment of the critical film thickness during electrolytic anodization. It is sequent of exothermic reaction of anodizing because the energy liberation increases with increasing of ohmic resistance of film. The anodizing process allows a number of parameters to be varied, for example changing the chemical composition of the electrolyte, the current density, sample temperature, etc. There are up to 40 process parameters of microplasma anodizing. Variation of any of these could affect the surface performance and alter the cryosorbing properties.

The thin surface layer on the porous copper samples is formed during the pressing process and contains small pores. The low diffusion from this layer into the bulk gives rise to the increase of gas pressure above sample surface. It is required to exclude the formation of same layer during the sample production.

Use of other stickers (for example,  $Al_2O_3$ ) for charcoal samples is difficult because samples with sufficient percentages of stickers adhere to the molds after pressing. The special demolding technology of sample from the mold or advanced search of other stickers is required.

## DISTRIBUTED CRYOPUMPDOWN

These studies and the studied designs of LHC LSS vacuum chamber prototype are described in detail in Ref. [6, 7, 8]. The CB and BS parameters are shown in Fig. 1. The total area of BS holes is 2% of area of BS surface. The studied cross-section designs of 1-m long LHC LSS vacuum chamber prototypes with charcoal crumb and two types of carbon fibre cryosorbers (CF) are shown in Fig.



Figure 3: LHC LSS vacuum chamber prototype. a) – design with charcoal, b) – design with carbon fibre (woven or nonwoven), c) – design with nonwoven carbon fibre fabric, d) – design with woven carbon fibre material.

3a-3d. The adsorption areas of cryosorbers are  $200 \text{ cm}^2$  for designs shown in Fig. 3a, 3b, 3d and  $120 \text{ cm}^2$  for design shown in Fig. 3c.

The comparison of dependences of dynamic pressures  $\Delta P$  inside the test chambers during H<sub>2</sub> injection on number of adsorbed molecules N is shown in Fig. 4.  $\Delta P$ , Pa, was measured by gauge placed at room temperature T<sub>RT</sub>, K. The dynamic pressure inside cold chamber is  $\Delta P_{cryo}=\Delta P \cdot (T_{cryo}/T_{RT})^{1/2}$  (T<sub>cryo</sub>, K – temperature of gas inside the cold chamber). These measurements were performed at close temperatures of BS with cryosorbers (T<sub>BS</sub>). The temperature of CB (T<sub>CB</sub>) is chosen as T<sub>CB</sub>=78 K for these experiments to exclude the adsorption of H<sub>2</sub> on the CB. Pressures are normalized to the gas flux Q=10<sup>14</sup> H<sub>2</sub>/sec.



Figure 4: Dynamic pressures inside the different designs of the LHC vacuum chamber prototypes,  $Q=10^{14}$  H<sub>2</sub>/sec.

The increase of the  $T_{BS}$  and  $T_{CB}$  at nonstandard or transient regimes of the LHC operation [9] will release the physisorbed gas into the gas phase. To predict the dynamic pressure in the LHC vacuum chamber after magnet quench, the dynamic pressure behavior during  $T_{BS}$  oscillations at  $T_{CB}$ =4.3 K was studied for BS with woven carbon fibre material (Fig. 3d). The measurements were performed without H<sub>2</sub> injection. 10<sup>20</sup> molecules of H<sub>2</sub> were adsorbed on the BS with cryosorber at  $T_{BS}$ =19K before beginning of measurements. Then  $T_{BS}$  was increased up to 50 K for 15 minutes to remove H<sub>2</sub> from carbon fibre to cold bore (simulation of quench case). Then the  $T_{BS}$  was kept in the range from 40 to 50 K

during 20 minutes. Then the BS was again cooled down to 19 K during 30 minutes. Then the equilibrium pressure was waited during 3 hours at  $T_{BS}$ =19K. Results show that after  $T_{BS}$  excursion and following cool down, the pressure level reaches acceptable for LHC value (which was before  $T_{BS}$  oscillation) during about 3 hours.

#### DISCUSSION AND CONCLUSION

The measured dynamic pressure  $\Delta P$  inside 1-m long test chamber with woven carbon fibre cryosorber corresponds to H<sub>2</sub> density in the cold part of vacuum chamber n≤10<sup>15</sup> H<sub>2</sub>/m<sup>3</sup> at gas flux Q=10<sup>14</sup> H<sub>2</sub>/sec and adsorbed gas dose up to more than N=2·10<sup>20</sup> molecules, that meet to LHC requirements [1, 2] (see Fig. 4). The regeneration temperature of carbon fibre cryosorber was experimentally defined as T<sub>reg</sub>~80K [8]. It is also very important that this material does not crumble and makes a convenient fixation onto the BS in comparison to charcoal. However, fluff can come off the carbon fibre at the edges, or, when the material is cut. To avoid this disadvantage a woven carbon fibre material can be made with specially treated edges.

Thus, the woven carbon fibre material can be proposed as perspective cryosorber for LHC and other colliders.

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