Characterization of ZAO sintered getter material for use in fusion applications

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The use of non-evaporable getter (NEG) pumps is common in many UHV applications, and the properties of the new sintered getters based on the ZAO® alloy make them appealing for applications dealing with large fluxes of hydrogen and its isotopes, as typically occurs in fusion research. In particular, the use of NEG pumps is interesting for the vacuum system of negative ion-based neutral beam injectors, which require a huge deuterium gas throughput. The key advantages of a NEG pumping solution are the high specific pumping speed and capacity, the ability to withstand a large number of adsorption/desorption cycles, the ease of integration. In addition, NEG do not release hydrogen unless power is supplied to heat them, addressing an important safety issue related to uncontrolled release in case of power outage or subsystem failures.

In this paper we report the experimental characterization of ZAO® sintered getters, in pressure regimes and sorption amounts relevant for the use in the next generation NBI. Discs and pumps were characterized in different conditions of H2/D2 pressure between 10^−4 and 10^−1 Pa and operating temperatures up to 150°C. In particular, the feasibility of getter regeneration within time windows acceptable for a high-availability NBI system was demonstrated.

Keywords: Neutral Beam Injector, Non-Evaporable Getter, ZAO alloy, Vacuum pump.

1. Introduction

Non-evaporable getter (NEG) technology has been considered for application in fusion research since 40 years ago, mainly by virtue of the high affinity of Zr-based alloys towards hydrogen and its isotopes [1]. Since then, significant steps forward have been made in the NEG field, in particular with the recent development of sintered pumping elements based on the ZAO® alloy [2]. At the same time, requirements as reliability, power efficiency and safety are acquiring more and more importance for components of next-generation fusion experiments, including the pumping system. In particular, the possible use of large NEG pumps in negative ion-based neutral beam injectors (NBI) is being studied [3]. The main advantages are related not only to the high specific pumping speed and capacity, but also to engineering and safety aspects. ZAO® pumping elements are very robust against repeated adsorption/desorption cycles and can work at room temperature or moderate temperatures like 150°C, without problems related to the freezing of components, radiative heat exchange or stray electrons, and with low power consumption. Thanks to the highly modular structure, NEG pumps allow for scalability and the development of tailor-made solutions, including compatibility with remote-handling. Then, by their nature NEGs are safe against unwanted release of hydrogen, since desorption is a process requiring adequate energy supply.

The conceptual design and the test of a relatively large mock-up NEG pump is a mandatory intermediate step towards a full-scale pump for NBI application. In order to provide a set of basic data for this task and support the development of appropriate models, single getter elements and small pumps were tested in conditions relevant for NBI operation.

2. Characterization of ZAO® getter elements

2.1 Effect of pressure and H2/D2 concentration on the pumping speed

Getter elements chosen for this application are in the form of highly porous sintered disks, about 24.3 mm in diameter and 2 mm thick. Then the disks are stacked onto a proper support and can be grouped in cartridges depending on the most suitable design. In a NBI, getter pumps are required to work at pressures ranging from the HV range up to 10^−2 Pa and to sorb loads of hydrogen isotopes by far larger than what is commonly done in UHV. Therefore, the effect of pressure and H2/D2 concentration on the pumping speed is one of the key features to be investigated. After activation at 550°C for one hour, ZAO® disks have been tested at constant pressure according to the ASTM F798-97 standard. Fig. 1 reports the relative initial pumping speed of the getter for different pressures (right panel) and loads of hydrogen or deuterium (left panel). The speed is normalized to the maximum value, obtained in the 10^4 Pa range or lower.
and at low and uniform concentration, respectively. The decrease of pumping speed for increasing pressure is due to the onset of the diffusion-limited pumping regime [4];

![Graph showing relative pumping speed vs. pressure for different concentrations.](image)

Fig. 1. Relative initial pumping speed of single ZAO® getter elements at room temperature for different pressure and hydrogen/deuterium concentration. Tests at different concentration were performed at a pressure of 4e-4 Pa.

...the ZAO® alloy exhibits a good ability to diffuse D₂, retaining about 80% of the maximum pumping speed in the 10⁻² Pa range. The results are satisfactory also with respect to the effect of H₂/D₂ concentration, considering that the limit of validity of the Sieverts’ law, about 1.3 Pa·m³/g, is not expected to be exceeded in real applications.

![Equilibrium isotherms for H2 and D2 of ZAO® sintered material.](image)

Fig. 2. Equilibrium isotherms for H₂ and D₂ of ZAO® sintered material.

### 2.2 Equilibrium pressure isotherms

The equilibrium pressure of hydrogen isotopes at the surface of getter materials obeys the well-known Sieverts’ law, which can be usefully expressed in the following way:

\[
\log(P) = A + 2 \log(c) - B/T
\]  

(1)

where A and B are constants, c is the concentration and T the temperature in K. The equilibrium isotherms of the ZAO® material have been measured for hydrogen and deuterium and are shown in Fig. 2. The difference between hydrogen and deuterium equilibrium pressure is within 20%. Most important, a set of measurements at 400°C and 500°C for H₂ was repeated 5 times showing the same results within the experimental uncertainty, thus ruling out the presence of any hysteresis effect during repeated absorption/desorption cycles.

### 2.2 Mechanical properties

In view of application in NBIs, long-term operation without maintenance is required. Therefore, the NEG material resistance to repeated adsorption/desorption cycles is an essential parameter. The production process of ZAO® getter disks results in a significant improvement of mechanical properties with respect to previous getter solutions. In sharp contrast with compressed getter elements, i.e. non-sintered, ZAO® pumping solutions exhibit an extremely low particle release and have been accepted for use in RF cryogenic cavities, i.e. one of the most demanding applications in terms of particle contamination [5,6].

A dedicated setup was developed to test the embrittlement limits of the NEG material, allowing to dose and extract hydrogen in a controlled way on single disks or small stacks of five units. The possibility of vibrating the disk in between each gas dose was provided too (33 Hz, 5 mm stroke). The main outcomes can be resumed as follows:

- ZAO® getter disks can adsorb up to 13 Pa·m³/g of hydrogen before showing a noticeable particle loose or cracking under vibration;
- Stacks have been subjected to 1000 adsorption/desorption cycles between 0.13 and 1.85
Pa·m³/g, showing no fatigue degradation (deformation, cracking or particle loose).

According to present application scenarios, a maximum concentration of 1.0 Pa·m³/g has been taken into consideration, thus providing a suitable safety margin for the disk robustness. The number of cycles performed during the test corresponds to nearly 20 years of operation in case of weekly regeneration (i.e., H₂/D₂ extraction).

![Graph showing absorption tests](image)

**Fig. 3.** Absorption tests at a constant pressure of 0.02 Pa on a NEG pump assembled with 31 ZAO® pumping elements.

3. Small-scale NEG pumps testing

3.1 Hydrogen and deuterium absorption tests

The performances of a NEG pump are determined by the single disk properties, the stack geometry (spacing, number of disks) and the pump geometry itself (number of stacks and their relative position). Results obtained on a single-stack pump of 31 disks with 1 mm spacing are shown in Fig. 3. Absorption test at a pressure of 0.02 Pa for hydrogen and deuterium have been performed at different temperatures. The pumping speed for hydrogen is about 30% larger than for deuterium. For concentrations larger than about 0.2 Pa·m³/g the pumping speed drops rather slowly, and a suitable operation window with limited speed variation is available. The effect of temperature (measured by a thermocouple welded on a getter disk) is mainly to promote the diffusion of hydrogenic species in the getter bulk, thus increasing the pumping speed at any concentration. A temperature as moderate as 150°C already results in a significant improvement of the performances, while maintaining the equilibrium pressure low enough to be still compatible with UHV conditions.

![Graph showing pressure evolution](image)

**Fig. 4.** Pressure evolution during the regeneration tests at different temperature. The auxiliary pumping speed is 5.5 l/s except for the dotted curve. The initial gas load is 0.7 Pa·m³/g except for the orange curve (1.1 Pa·m³/g).

![Graph showing time trend of hydrogen concentration](image)

**Fig. 5.** Time trend of hydrogen concentration during the regeneration tests.

3.2 Regeneration tests

The process of extraction of hydrogen isotopes from the getter material is referred to as “regeneration”, and it consists in heating the getter under pumping, usually at temperatures in the range 450-650°C. The time and the size of the auxiliary pumps needed for the NEG regeneration process are among the main design parameters to be taken into account for the elaboration of realistic NEG working scenarios in NBI application. In order to collect data to study the regeneration process, after each absorption test the NEG pump prototype was regenerated with different parameters; in particular, three temperatures (450 – 550 – 650°C) and two different auxiliary pumping speeds (5.5 – 139 l/s for H₂) were used.

**Fig. 4 and Fig. 5 show the results of selected H₂ regeneration runs, in particular the time trend of pressure and concentration, respectively. All shown curves have been collected with an auxiliary pumping speed of 5.5 l/s for hydrogen, except for the black dotted curve (139 l/s). The desorbed quantities match the absorbed ones, within uncertainties given by the initial concentration which may vary by about 0.05 Pa·m³/g. The expected general
The behavior of the regeneration process is confirmed by the experiments: first, the regeneration is much slower at 450°C and significantly faster at 650°C, due to the exponential dependence of the equilibrium pressure on temperature. Then, shortening of the process can be obtained also increasing the auxiliary pumping speed: as visible in Fig. 5, a very similar regeneration process was obtained either at 650°C and 5.5 l/s pumping or with a pumping speed 25 times larger but a temperature 100°C lower.

The initial conditions of the getter in terms of concentration have a not so large influence on the required regeneration time when the final target concentration is below 0.25 Pa·m³/g, as suggested by the regeneration curves at 550°C and 5.5 l/s starting from different concentrations (near 1.1 and 0.7 Pa·m³/g). It is interesting to note that these processes show a different evolution of pressure with time, but superimpose when pressure is plotted as a function of concentration, as shown in Fig. 6. This indicates that the process follows the same rules connecting temperature, concentration and pressure, regardless of the initial absorbed quantity. It was also found that the regeneration occurs in identical way when the absorption takes place at room temperature or 150°C. It was verified that regeneration at 5.5 l/s occurs in quasi-stationary conditions, and the experimental curves are well reproduced with a model based on the Sieverts law if an effective temperature is used, which for all test is given by the experimental one multiplied by a factor in the range 0.88 – 0.90. This may be related to non-uniformity of the getter cartridge temperature.

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4. NEG mock-up pump

In view of application in next generation NBI, these results have been used to design and build a relatively large-scale prototype NEG pump to demonstrate the concept feasibility and validating scaling models. Detailed information on the pump prototype design features will be described elsewhere. The expected speed for deuterium at 0.02 Pa is about 45 m³/s and the getter mass is 32 Kg. In the same way, pumps of about 500 m³/s with a getter mass of less than 350 Kg can be envisaged. The experimental results suggest that regeneration down to 0.2 Pa·m³/g is feasible in a few hours with an auxiliary speed of 5.5 l/s, i.e. 0.05 l/s per each gram of getter material (see Fig. 4). This means that NEG pumps delivering very large pumping speed on the order of several hundred m³/s can likely be regenerated with auxiliary pumps of a few tens m³/s, which sounds reasonable based on recent developments in pumping systems for fusion technology [7].

5. Conclusions

Thanks to its properties, the ZAO® getter material opens new perspectives for NEG application in next-generation fusion facilities. The material has been characterized in conditions relevant for NBI operation, giving useful and promising results. A relatively large NEG mock-up pump will be tested in the near future at KIT, for validation of the pumping concept and adopted technological solutions.

Acknowledgments

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