

# Design and fabrication of a Permeator Against Vacuum prototype for small scale testing at Lead-Lithium facility

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Tritium recovery is one of the major issues of a future DEMO reactor, in order to accomplish with the requirement of tritium self-sufficiency. The EUROfusion Programme has considered the permeation against vacuum (PAV) technique as baseline for those blankets which use PbLi as breeder.

A conceptual design of a squared multi-channel PAV for its implementation in an experimental PbLi loop, under construction at CIEMAT, has been produced. Previous studies have shown the feasibility of using vanadium, tantalum or niobium as PAV membrane due to their good permeability and compatibility with PbLi. In order to save costs and time a preliminary  $\alpha$ -Fe membrane was considered for the prototype, in spite of its low permeability. However, due to mechanical problems related to the use of this material, a change in the design was produced in order to afford the use of vanadium.

Structural calculations are also presented and evaluated to develop the best way to implement the manufacturing process, paying special attention to the interface between the membranes and the main structure in order to avoid leakages. Other important aspects such as keeping an adequate vacuum level and its arrangement system have also been considered.

Keywords: DEMO, permeation against vacuum, membrane, prototype

## 1. Introduction

The Dual Coolant Lithium Lead (DCLL) is one of the blanket concepts which are being considered for DEMO. It uses liquid metal (eutectic PbLi) as primary coolant, tritium breeder, tritium carrier and neutron multiplier [1].

One of the most important functions of the blanket is to achieve tritium self-sufficiency [2], consequently the development of tritium recovery systems from the breeder is mandatory. Permeation Against Vacuum (PAV) has been selected as first candidate for tritium extraction from PbLi in the DCLL [3]. Its working principle is based on the diffusion of tritium through a permeable membrane in contact with the liquid metal and its extraction by a vacuum pump which drives it to the tritium plant. However, this technique has not been experimentally validated, and some efforts are being performed in Europe with the purpose of demonstrating its capabilities.

The present work provides a small scale prototype of PAV based on the conceptual design presented in [4]. A description of the PbLi loop driving the PAV design is presented in Section 2; the geometrical characteristics of the device are shown in Section 3; main issues concerning its fabrication are presented in Section 4; and the PAV auxiliary systems are described in Section 5. Finally, conclusions are drawn in Section 6.

## 2. Facility for tritium extraction from PbLi at high velocity

Within the EUROfusion R&D programme different experimental works related to advanced tritium extraction techniques are being performed. Thus, PAV

has been considered as the most promising technique for extracting tritium from flowing PbLi [3]. Its operational conditions depend on the blanket concept which is being considered, in the case of a DCLL these conditions can be found in [4]. The construction of a facility for demonstrating the PAV technique at high PbLi flows is being implemented at CIEMAT. The aim of this PbLi loop is to:

- Test hydrogen/deuterium permeation in flowing PbLi at DCLL conditions of temperature, velocity and tritium partial pressure.

- Perform different measurements in relevant ranges of these conditions.

- Test different permeator concepts or configurations for efficiency assessment.

- Test different materials to be used as membrane of the PAV.

The loop will be divided into two sections with different temperatures: a cold leg working at 300°C which owns an electromagnetic pump and a flowmeter with lower operational requirements in terms of temperature; a hot leg (550°C) where the test section is installed. Table 1 depicts the main parameters of the loop.

Table 1: PbLi loop main parameters

| Parameter                     | Value      |
|-------------------------------|------------|
| Temperature [5]               | 300-550 °C |
| PbLi mass flow rate [5]       | 2-39 kg/s  |
| PbLi pressure [5]             | 1-3 bar    |
| Max. PbLi velocity in the PAV | 1 m/s      |
| Space for test section        | 1.5 m      |

### 3. PAV design optimization

Following [4], a small scale prototype of PAV has been developed, being conditioned by the PbLi loop parameters presented in section 2. Although tests will be done at different PbLi velocities, the target value, relevant for a DCLL DEMO, is 1 m/s [4]. For this reason, the study of the influence of each parameter to optimize the design has been performed by fixing that velocity.

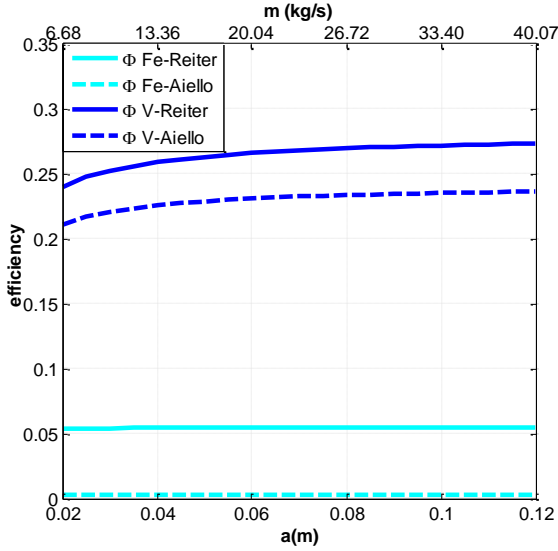


Fig. 1. Efficiency dependence with channel width. ( $v = 1$  m/s;  $h = 5 \times 10^{-3}$  m;  $z = 1 \times 10^{-3}$  m;  $N = 7$ ;  $T = 823$  K;  $\Phi_{Fe} = 1.75 \times 10^{-10}$  mol/m/s/Pa $^{0.5}$ ;  $\Phi_V = 1.52 \times 10^{-7}$  mol/m/s/Pa $^{0.5}$ )

Taking into account the available space for the permeator implementation, i.e. 1.5 m in the experimental room, the membrane has to be limited to 1 m length ( $L$ ). The main parameter affecting the PAV design is the PbLi mass flow rate ( $m$ ) which will determine the number of channels ( $N$ ) and their width ( $a$ ) [4]. Another important parameter is the channel height ( $h$ ). Low  $h$  values lead to high pressure drops, and regarding the range of mass flow rate, the height has been maintained at  $5 \times 10^{-3}$  m. It is easy to follow that a decrease in the membrane thickness ( $z$ ) results in an improvement of efficiency due to the reduction of the permeation barrier. Regardless, the mechanical resistance of the membrane should be taken into account, therefore  $z$  is set to  $1 \times 10^{-3}$  m.

Best materials for the PAV fabrication are vanadium, niobium and tantalum due to their good permeability ( $\Phi$ ) [6] and compatibility with PbLi in static conditions [7]. But in order to save costs and due to the availability of materials, the first PAV prototype was thought to use  $\alpha$ -Fe membranes. In spite of its low permeability [8], this material can lead to enough extraction capability in order to demonstrate the permeation technique.

It is important to note that there is some dispersion in measured hydrogen solubility in PbLi. Depending on the methodology followed for its obtainment and the eutectic grade of the alloy it can change up to two orders of magnitude. For this reason Reiter [9] and Aiello [10] Sievert's constants have been used for the study as the most optimistic and pessimistic cases, respectively.

Figs. 1 to 3 show the difference between PAV efficiencies for both V and Fe membranes. Fig. 1 shows the relation between the efficiency and the permeator width ( $a$ ) using the two values of hydrogen solubility in PbLi [9, 10]. A slight increase of the efficiency with growing values of  $a$  is observed. In order to fix this value the mass flow rates in the loop have been considered. Thus, the channel width is fixed to 0.085 m and the number of PbLi flowing channels to 7 (therefore 8 vacuum channels) to allow the variation of the mass flow providing a range of velocities able to test the technique.

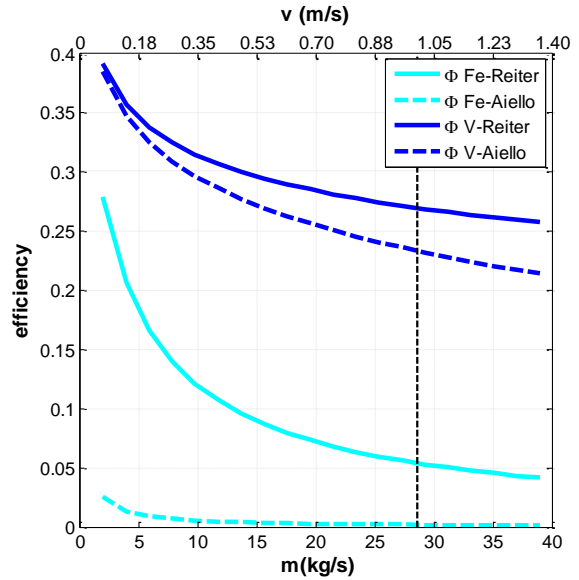


Fig. 2. Efficiency dependence with mass flow rate. ( $h = 5 \times 10^{-3}$  m;  $z = 1 \times 10^{-3}$  m;  $a = 0.085$  m;  $N = 7$ ;  $T = 823$  K;  $\Phi_{Fe} = 1.75 \times 10^{-10}$  mol/m/s/Pa $^{0.5}$ ;  $\Phi_V = 1.52 \times 10^{-7}$  mol/m/s/Pa $^{0.5}$ )

The dependence of the efficiency with the PbLi mass flow in the loop is presented in Fig. 2. As expected, an increase on the mass flow rate, with the subsequent increase on velocity, implies a decrease on the efficiency following the exponential relation showed in [4]. In the case of V membranes (higher permeability) the permeation flux is driven by mass transport (i.e. tritium transport in the PbLi). Hence, a change in the solubility is less accentuated. On the contrary, when using Fe the permeation is limited by membrane processes, causing higher impact on the PAV efficiency.

An interesting situation arises when the temperature is varied, Fig. 3, where the mass flow is fixed to 26.4 kg/s (corresponding to 1 m/s in the PAV channels). Although hydrogen solubility in PbLi increases with temperature (faster for Aiello; disfavoring the extraction process), the increase of the diffusivity in the alloy is faster and hence the efficiency is enhanced. However, hydrogen permeability through vanadium decreases with temperature (contrary to what happens in Fe) and for temperatures higher than 825 K the huge solubility given by Aiello diminishes the efficiency.

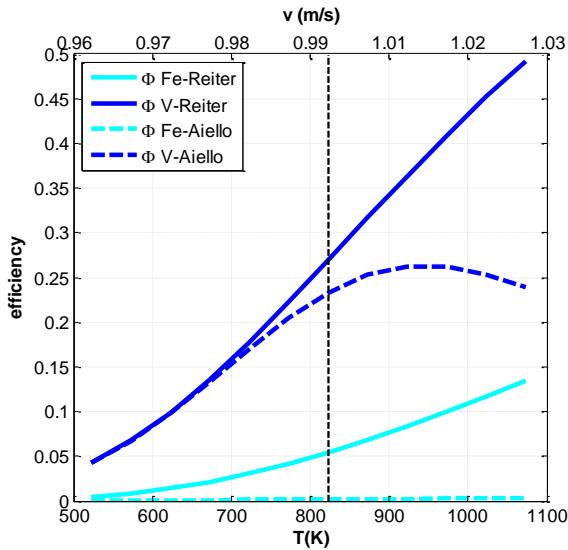


Fig. 3. Efficiency dependence with temperature. ( $m = 26.4$  kg/s;  $h = 5 \times 10^{-3}$  m;  $z = 1 \times 10^{-3}$  m;  $a = 0.085$  m;  $N = 7$ ;  $T = 823$  K;  $\Phi_{Fe} = 1.77 \times 10^{-8} \exp(-31600/R/T)$  mol/m/s/Pa $^{0.5}$ ;  $\Phi_V = 4 \times 10^{-9} \exp(24900/R/T)$  mol/m/s/Pa $^{0.5}$ )

According to all these results the main parameters for the permeator prototype design are summarized in Table 2.

#### 4. Manufacturing

Regarding the manufacturing process, structural calculations and assembly possibilities have been performed when considering two different approaches (Fe or V). The range of efficiencies achieved with these materials is also presented as a function of the mass flow rate and the hydrogen solubility in PbLi.

##### 4.1. $\alpha$ -Fe membrane

According to the mass flow rate limits (Table 1), the proposed design shows an efficiency for  $\alpha$ -Fe membranes ranging from 4 to 27%, considering Reiter's solubility. When Aiello's solubility is applied, the efficiency ranges from 0.1% to 0.4%, Table 3.

Table 2. PAV prototype main parameters.

| Parameter          | Value       |
|--------------------|-------------|
| Channel width      | 0.085 m     |
| Channel height     | 0.005 m     |
| Membrane length    | 1 m         |
| Membrane thickness | 0.001 m     |
| Number of channels | 7 (15)      |
| Membrane area      | 1.26 m $^2$ |
| PbLi volume        | 3 dm $^3$   |

For the permeator construction the most feasible option seems to be the stacking of plates in a lateral structure, also made of Fe, to define the channels. There are needed stiffening elements located inside vacuum channels in order to avoid membrane deformations. Fig. 4 shows preliminary elastic analyses performed in order to find a suitable arrangement of the stiffeners. The resulting Von Mises equivalent stresses are under the reference yield strength for iron (150 MPa). However, there are some issues related to the use of this material. The ferritic structure of  $\alpha$ -Fe with low content on carbon complicates the mechanization due to its softness and magnetic properties. Therefore, the possibility of using other membranes, in spite of the price or availability, has been explored.

##### 4.2. V, Nb, Ta membrane

As stated, V, Nb and Ta have great permeability properties. The efficiency achieved with these three materials is the same, ranging from 38-39% to 21-26% depending on the hydrogen solubility in PbLi, see Table 3. The huge difference between Fe and V/Nb/Ta permeabilities leads to a different relation between the coefficient of solubility used and the efficiency provided by each material, as it was explained in section 3.

The price of these materials is rather high and in order to reduce the total amount, a change on the assembly design has been introduced: only the membranes are made of V/Nb/Ta while the supporting structure is fabricated on stainless steel.

It must be underlined that the involvement of two materials with different coefficients of thermal expansion ( $\sim 16 \mu\text{m/m}$  and  $8.3 \mu\text{m/m}$  for austenitic steels and V, respectively) can cause intolerable thermal stresses when the PAV is heated up to  $550^\circ\text{C}$ . Before the definitive selection of the structural material, a preliminary elastic analysis has been carried out considering V as the unique material. Maximum Von Mises equivalent stress is 129.5 MPa, fig. 5, close to the lowest value indicated by the supplier (range between 124 and 172 MPa). The maximum deformation along the vertical axis is 0.03 mm and will not affect the PbLi flow.

Due to the huge melting temperature of Nb and Ta (higher than  $2500^\circ\text{C}$ ) the welding with stainless steel is not straightforward, it is needed an interface. This complication is avoided with the use of V membranes which have a melting temperature near to that of the steel. Therefore, TIG welding can be used for the fabrication of the PAV.

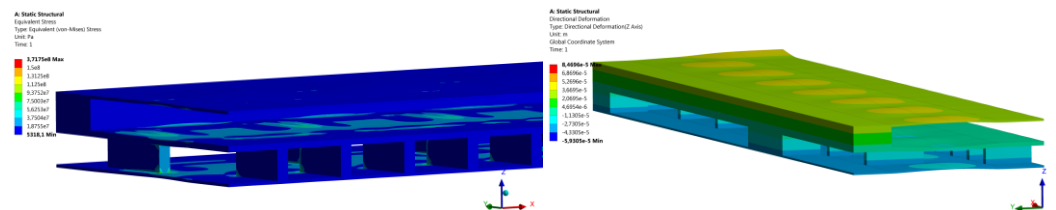


Fig. 4. Von Mises stress (MPa) and total deformation (mm) for  $\alpha$ -Fe .

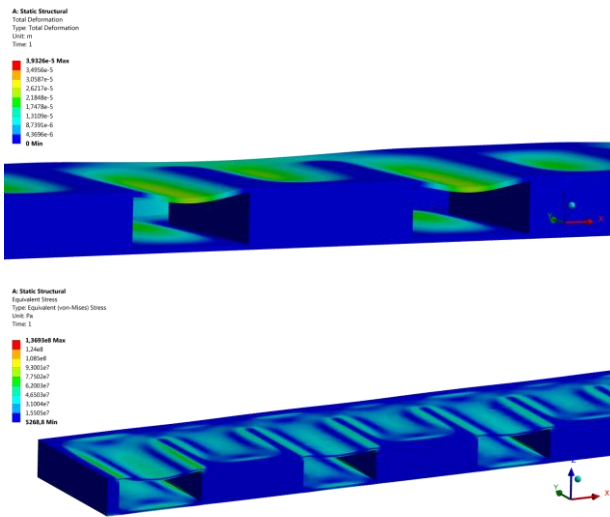


Fig. 5. Von Mises stress (MPa) and total deformation (mm) for V

## 5. PAV final design

From the two manufacturing designs presented, the V-based PAV has been selected since it solves issues related to marching and structural resistance.

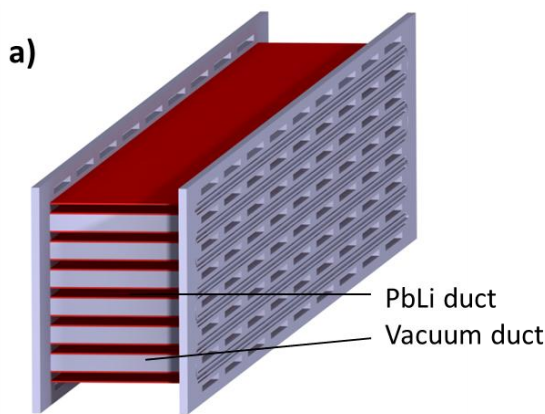
The final design is shown in Fig. 6-a. A supporting structure of stainless steel with splines to allocate the 1 mm thick, 1m long, V membrane was designed. There are needed 14 sheets of 90 mm width to conform 7 PbLi flowing ducts and 8 vacuum channels. The structure has some lateral holes for vacuum extraction.

To close the structure and integrate the vacuum system a box containing the flange and feedthroughs for the vacuum pump, thermocouples, pressure sensors and heating elements has been designed (Fig. 6-b). The connection to the PbLi circuit is made through a round to square diffuser to distribute the flow over the PbLi ducts.

With all these integrations the final dimensions of the PAV are: 1.4 m length, 28.4 cm width (including the vacuum flange connection) and 20 cm height.

Different auxiliary systems must be implemented in the PAV to proceed with the experimental phase.

### a. Vacuum system



Due to the high mass flow, the tritium partial pressure in a DCLL-BB is really low, about 110 mPa. Therefore, as shown in Table 1, the range of hydrogen pressure to be tested starts at 0.1 Pa. Since tritium permeation flux is driven by the pressure gradient generated between the two sides of the membrane, it is needed to achieve a good vacuum in order to accomplish a high extraction rate.

Table 3. PAV efficiency.

| Mass flow | Material       | Reiter | Aiello |
|-----------|----------------|--------|--------|
| 2 kg/s    | $\alpha$ -iron | 28%    | 2%     |
|           | Vanadium       | 39%    | 38%    |
| 39 kg/s   | $\alpha$ -iron | 4%     | 0.1%   |
|           | Vanadium       | 26%    | 21%    |

For the PAV presented in Fig. 6 the vacuum volume is about 16 liters. A high vacuum level is accomplished by the use of a turbomolecular pump. The *HiPace 500@* gives a pumping speed up to 445 l/s for H<sub>2</sub>, enough for this application.

### b. Heating system

Since the experiments should be performed at 550°C, integration of a heating system is essential. A mineral insulated electrical resistance, from *Thermocoax*, will be used for that purpose. It is made of an Inconel alloy sheath with a nickel/chromium core apt for working in a high vacuum environment and up to 1000°C. It will be installed in the lateral face of the PAV structure, where a zig-zag slot will host the cable.

### c. Instrumentation

Additional instrumentation is needed to control the fluid temperature and vacuum pressure:

- Type N- Microbell D sheath thermocouples, from *TCDirect*, installed into vacuum channels to measure the internal temperature of the permeator along its length.
- Pirani/cold cathode full range gauge, from *Pfeiffer-Vacuum*, to control the vacuum level and disposed in the opposite face of the pump connection.

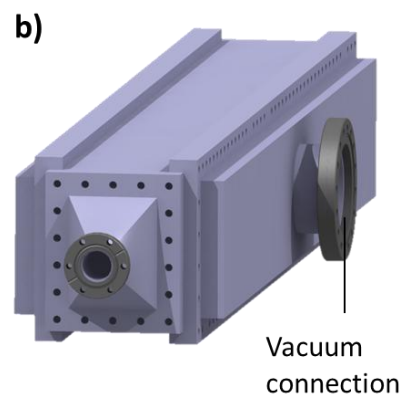


Fig. 6. a) PAV design based on vanadium sheets (red) embedded on a stainless steel structure (grey); b) PAV general view with vacuum devices and circuit connection.

## 6. Conclusions

The manufacturing design has been completed to fabricate the PAV prototype for testing the hydrogen extraction from PbLi capability of V membranes.

Specific requirements for manufacturing, assembly and testing drove some design changes. Initially, the use  $\alpha$ -Fe membrane was envisioned. However, due to structural and machining issues a rearrangement was introduced in order to simplify and improve the design with the use of more adequate materials.

The implementation of auxiliary systems has also been included in order to keep an adequate vacuum level and the required temperature both controlled with the corresponding sensors.

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