



## Hydrogen permeability in pure Fe metal and FeC alloy

I. Peñalva<sup>a,\*</sup>, G. Alberro<sup>a</sup>, J. Aranburu<sup>a</sup>, F. Legarda<sup>a</sup>, R. Vila<sup>b</sup> and C.J. Ortiz<sup>b</sup>

<sup>a</sup> University of the Basque Country (UPV/EHU), Department of Nuclear Engineering & Fluid Mechanics, E.T.S.I. Bilbao, Alda. Urquijo s/n, 48013 Bilbao, Spain  
<sup>b</sup> CIEMAT, Avda. Complutense 22, 28040, Madrid, Spain

### ABSTRACT

Samples of pure Fe metal and FeC alloy with controlled chemical alloying element contents and microstructure, supplied by the European Fusion Development Agreement (EFDA), were experimentally analysed in the facilities located at the University of the Basque Country (UPV/EHU) in collaboration with CIEMAT.

In this work, the hydrogen transport parameter of permeability was experimentally measured in pure Fe metal and FeC alloy by means of the gas evolution permeation technique. The experimental temperature range explored was from 423 K to 823 K and the high purity hydrogen loading pressures from  $10^3$  Pa to  $1.5 \cdot 10^5$  Pa. We observed that the permeability obtained for the two materials in this temperature range follows an Arrhenius law. The resultant diffusive permeability for pure Fe metal and FeC alloy were found to be  $\Phi_{Fe} = 3.88 \cdot 10^{-8} \exp(-35.9 \text{ (kJ/mol)} / RT)$  and  $\Phi_{FeC} = 9.10 \cdot 10^{-8} \exp(-37.4 \text{ (kJ/mol)} / RT)$ , respectively. The resulting activation energies turn to be very close, whereas the permeation prefactor increases for the FeC alloy, in comparison to pure Fe. These results are in good agreement with values found in the literature for pure Fe metal and for low carbon content FeC alloys. According to the results, the influence of the metallurgical composition of C in Fe alloys in the diffusion of hydrogen is discussed.

### EXPERIMENT

**PERMEATION FACILITY** - A single run of the experiment consists of holding one surface of a thin membrane of the material at a certain high H pressure level, forcing the H permeation through the specimen to a low pressure region (Figures 1 and 2 and Photograph). Two capacitance manometers (P1 and P2) record the pressure increase in the low pressure volume and allow obtaining the transport parameters at different temperatures achieved with a resistance furnace (F).

**MATERIAL PREPARATION** - Rods provided by EFDA with controlled chemical alloying element contents and microstructure: **pure Fe** and **FeC alloy**. Initially, samples were cut to a thickness of about 1.3 mm. After that, the specimens were prepared with a fixed grinding sequence using progressively finer abrasive grits. Then, samples were polished with diamond paste and heating at 353 K before cleaning them in different ultrasonic baths and drying of the specimen in hot air. Finally, samples were heated under UHV conditions up to 823 K inside the permeation facility before the first experimental run.

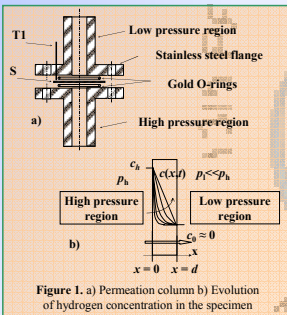


Figure 1. a) Permeation column b) Evolution of hydrogen concentration in the specimen

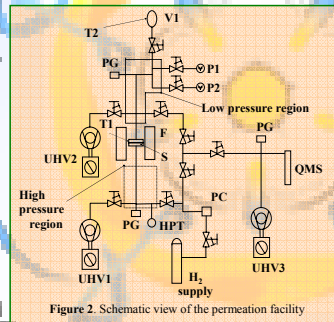


Figure 2. Schematic view of the permeation facility



### THEORY

The effective transport parameters can be evaluated for each experimental  $T$  and  $p_h$ , by modelling the permeation tests (Figure 3). The theoretical expression for the pressure increase with time in the low pressure region in a permeation test, as a function of the transport parameters, is obtained from the solution of Fick's second law in an infinite slab (Figure 1.b):

$$p(t) = \frac{RT_p}{V_p} \left[ \frac{DS}{d} A_1 - \frac{Sd}{6} A_2 - \frac{Sd}{3} A_3 \sum_{n=1}^{\infty} \frac{(-1)^n}{n^2} \exp\left(-D \frac{n^2 \pi^2}{d^2} t\right) \right]$$

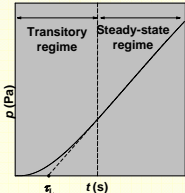


Figure 3. Experimental permeation curve: transitory and steady-state permeation regime

The permeated flux under diffusive regime for every temperature depends on: the thickness of the sample, the values of the loading pressure and the PERMEABILITY of the gas ( $\Phi$ ). This transport parameter defines the gas-material interaction.

### RESULTS

Permeability was calculated for both samples at different temperatures, following in both cases an Arrhenius law (Figure 4):

$$\Phi_{Fe} = 3.88 \cdot 10^{-8} \exp(-35.9 \text{ (kJ/mol)} / RT); \quad \Phi_{FeC} = 9.10 \cdot 10^{-8} \exp(-37.4 \text{ (kJ/mol)} / RT).$$

In both cases the diffusive regime has turned out to be prevailing, with variation of the flow with loading pressures ( $J$  vs.  $p_h$ ) following a potential relation with powers close to 0.5 for every temperature (Figure 5).

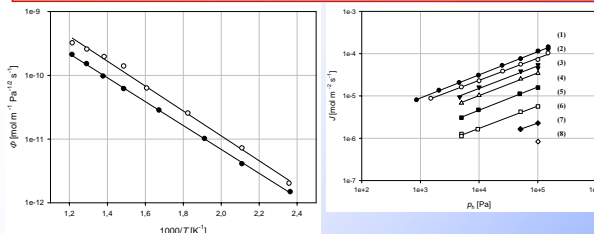


Figure 4. Experimental hydrogen permeability for EFDA samples: pure Fe (black dots) and FeC alloy (white dots)

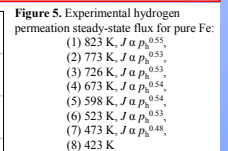


Figure 5. Experimental hydrogen permeation steady-state flux for pure Fe:

- (1) 823 K,  $J$  vs  $p_h^{0.55}$
- (2) 773 K,  $J$  vs  $p_h^{0.53}$
- (3) 726 K,  $J$  vs  $p_h^{0.53}$
- (4) 673 K,  $J$  vs  $p_h^{0.54}$
- (5) 598 K,  $J$  vs  $p_h^{0.54}$
- (6) 523 K,  $J$  vs  $p_h^{0.53}$
- (7) 473 K,  $J$  vs  $p_h^{0.48}$
- (8) 423 K

### CONCLUSIONS

The gas permeation technique has been used with samples of pure Fe and FeC alloy in a broad range of temperatures (423 K to 823 K) to obtain its hydrogen permeability values. The analysis of steady-state permeation fluxes at each experimental temperature and different loading pressures has agreed on a diffusion-limited regime. The resulting activation energies for both samples are similar with an increase of the permeation prefactor for the FeC.

This work was funded by the UPV/EHU, EFDA and the Spanish Ministry of Science and Innovation. The authors would like to thank the FEMaS-CA for the support in fusion research.

