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STRESS EFFECTS ON HYDROGEN PERMEATION THROUGH TUBULAR MULTILAYER MEMBRANES

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Abstract. *Hydrogen permeation in metals is a subject of great relevance in several technological applications. For instance, metallic membranes have been used with success for hydrogen purification and separation processes. Palladium-based membranes are particularly suited to this purpose due to their high permeability and good surface properties. Despite their advantages, palladium-based membranes are usually too expensive for large-scale applications. This has led to the development of multilayer membranes, where a low-cost material such as tantalum, niobium and vanadium, is coated with palladium. This work deals the formulation and numerical simulation of a continuum model for investigating the effects of the stress on hydrogen permeation in multilayer tubular membranes. In this case, the stress affects not only hydrogen transport and ingress/egress, but also the continuity of the hydrogen content across the interface between two materials. The novelty here is the treatment of this interface condition and its corresponding numerical implementation. The numerical simulation is implemented by using COMSOL Multiphysics.*

Keywords: *Multilayer membranes, Hydrogen diffusion, Stress-induced diffusion, Finite element*

1. INTRODUCTION

Gibbs (1993) introduced the idea of a solid which also contains fluid components, whereby a fluid can enter into, move independently through and distort the solid, which otherwise is conserved and behaves elastically. As indicated by Li *et al.* (1966) a genuine example of this kind of body is provided by interstitial solid solutions at sufficiently low temperatures, with host and interstitial species playing the role of solid and fluids, respectively. Additional examples can be found in the fields of geology, polymer science and metallurgy, as pointed out by Larché and Cahn (1973), who extended Gibbs' idea by allowing solid diffusion. This extension is basic for the modern understanding of equilibrium and diffusion kinetics in solid solutions under stress (see Larché and Cahn (1982); Larché *et al.* (1985)), as well as for the unified treatment of atomic transport given by Fried and Gurtin (1999).

The aforementioned works set the foundations for investigations concerning the role played by stress during hydrogen permeation in metals. See, for instance, Baranowski (1989), Adrover *et al.* (2003), Wang *et al.* (2002), and Lee *et al.* (2000), in the case of single-material membranes. It is worth mentioning that metallic membranes have been used with success for hydrogen purification and separation processes. In particular, palladium-based membranes are particularly suited to this purpose due to their high permeability and good surface properties. Despite their advantages, palladium-based membranes are usually too expensive for large-scale applications. This has led to the development of multilayer membranes, where a low-cost material such as tantalum, niobium and vanadium, is coated with palladium (Buxbaum and Kinney (1996)). So, it seems natural to investigate the role played by stress on the permeation in composite materials. This is the purpose of the present contribution.

This paper is organized as follows. In Section 2 we present continuum equations, including interface and boundary conditions, that govern solute diffusion and solid deformation within a small deformation setting. These equations are specialized for the case of a hollow cylinder in Section 3, which also contains the essential ingredients of the numerical model. Results are presented in Section 4.

2. PRELIMINARIES

We consider a material body $\mathcal{B}$ composed of an elastic solid and a one-component interstitial solute (solute for short). This body, from now on identified with the region occupied by its solid component in a fixed reference configuration, is

the stage of two interdependent phenomena, namely solid deformation and solute diffusion. The state of $\mathcal{B}$ at time t is defined in terms of solid displacement $\mathbf{u}(\cdot, t)$ and solute concentration $c(\cdot, t)$, which respectively assign to each material point $\mathbf{x}$ in $\mathcal{B}$ the corresponding displacement and solute content per unit reference volume at time t. Within a small deformation setting, a relevant quantity is the small strain tensor $\mathbf{E}$, which is given by the symmetric part of displacement gradient $\nabla\mathbf{u}$, that is,

$$\mathbf{E} = \frac{1}{2}\left(\nabla\mathbf{u} + \nabla\mathbf{u}^\top\right). \tag{1}$$

Henceforth $\mathbf{A}^\top$ denotes the transpose of the tensor $\mathbf{A}$.

2.1 Bulk equations

We now present bulk equations that govern solute diffusion and solid deformation within a small deformation setting. This set of equations is comprised by the additive decomposition of the small strain tensor $\mathbf{E}$ into two contributions, one due solute change with no change in stress and one due stress, $\mathbf{E}_c$, and one due to stress with no solute change, $\mathbf{E}_e$, and two field equations describing the mechanical force and solute content balances. These equations are conveniently organized as follows:

- Additive decomposition:

$$\mathbf{E} = \mathbf{E}_e + \mathbf{E}_c, \qquad \mathbf{E}_c = \hat{\mathbf{E}}_c(c). \tag{2}$$

- Force balance:

$$\operatorname{div}\mathbf{T} = \mathbf{0}, \qquad \mathbf{T} = \frac{\partial\hat{\psi}(\mathbf{E}_e, c)}{\partial\mathbf{E}_e}. \tag{3}$$

- Solute conservation:

$$\frac{\partial c}{\partial t} = -\operatorname{div}\mathbf{h}, \quad \mathbf{h} = -\hat{\mathbf{M}}(\mathbf{E}_e, c)\nabla\mu, \quad \mu = \frac{\partial\hat{\psi}(\mathbf{E}_e, c)}{\partial c} - \mathbf{T}\cdot\frac{\mathrm{d}\hat{\mathbf{E}}_c(c)}{\mathrm{d}c}. \tag{4}$$

Here and henceforth: div denotes the divergence operator; $\hat{\mathbf{E}}_c$, $\hat{\psi}$ and $\hat{\mathbf{M}}$ are the constitutive response functions determining the solute-induced strain, the free-energy density and the solute mobility; $\mathbf{T}$ is the stress tensor; $\mathbf{h}$ is the solute flux relative to the solid; and μ is the chemical potential of the solvent in the solid. Notice that deformation and diffusion are coupled via the solute-induced strain $\hat{\mathbf{E}}_c$ and that additional couplings may also arise through the response functions $\hat{\psi}$ and $\hat{\mathbf{M}}$.

Throughout this paper, we shall concentrate on a simple theory for the coupling between mechanics and diffusion in isotropic solids. This theory is based on the following assumptions:

- The solute-induced strain is purely dilatational and given by

$$\hat{\mathbf{E}}_c(c) = \eta\,(c - c_0)\mathbf{I} \tag{5}$$

  where $\eta > 0$ is constant and c_0 is the solute concentration in the reference configuration used to measure strain. Notice that $\eta = \upsilon/3$, where υ is the volume occupied by one solute atom in the solid.

- The free-energy response $\hat{\psi}$ describes two contributions, elastic $\hat{\psi}_e$ and chemical $\hat{\psi}_c$, that is,

$$\hat{\psi}(\mathbf{E}_e, c) = \hat{\psi}_e(\mathbf{E}_e) + \hat{\psi}_c(c), \tag{6}$$

  with

$$\hat{\psi}_e(\mathbf{E}_e) = \frac{1}{2}(\lambda|\mathrm{tr}\mathbf{E}_e|^2 + 2G|\mathbf{E}_e|^2) \quad \text{and} \quad \hat{\psi}_c(c) = \mu_0 c + k_B T c(\ln c - 1), \tag{7}$$

  where, λ and G are the Lamé moduli, μ_0 is the reference chemical potential, k_B the Boltzman constant, and T the temperature.

- The mobility response is linear in c, independent of $\mathbf{E}_e$ and given by

$$\hat{\mathbf{M}}(\mathbf{E}_e, c) = \frac{Dc}{k_B T}\mathbf{I}, \tag{8}$$

  where D is the diffusion coefficient of the solute in the solid.

The aforementioned choices imply that

$$\begin{aligned} &\mathbf{T} = \lambda\,(\mathrm{tr}\mathbf{E}_e)\mathbf{I} + 2G\mathbf{E}_e, \\ &\mu = \mu_0 + k_B T \ln c - \eta\,\mathrm{tr}\,\mathbf{T} \quad \text{and} \quad \mathbf{h} = -D\left(\nabla c - \frac{\eta}{k_B T} c\,\nabla \mathrm{tr}\,\mathbf{T}\right). \end{aligned} \tag{9}$$

2.2 Boundary and interface conditions

The aforementioned governing equations must be supplemented by boundary conditions that involves the interactions between the solid and the environment. For instance, if one supposes that $\mathcal{B}$ is immersed in an environment wherein the chemical potential of the solute is constant and equal to μ_a and assume that at the interface $\partial\mathcal{B}$ between the solid and the environment a traction $\mathbf{s}$ is prescribed and chemical equilibrium prevails, the attaining boundary conditions are

$$\mathbf{T}\mathbf{n} = \mathbf{s}, \qquad \mu = \mu_a, \tag{10}$$

where $\mathbf{n}$ is the unit normal to $\partial\mathcal{B}$.

If the body is immersed in gaseous environment composed of pure H_2, we have

$$\mu_a = \frac{1}{2}\mu_{H_2} \quad \text{with} \quad \mu_{H_2} = \mu^0_{H_2} + k_B T \ln \frac{p}{p_0}, \tag{11}$$

where $\mu^0_{H_2}$ is the chemical potential of the gaseous hydrogen at the reference pressure p_0. In this case, the boundary condition $\mu = \mu_a$ gives that

$$c = K\sqrt{p} \qquad \text{where} \qquad \ln K = \frac{\frac{1}{2}\mu^0_{H_2} - \mu^0 + \eta \mathrm{tr}\mathbf{T}}{k_B T} - \frac{1}{2}\ln p_0. \tag{12}$$

This relation, called Sieverts Law, accounts for elastic effects via the presence of $\mathrm{tr}\mathbf{T}$ into the definition of K.

If $\mathcal{B}$ is composed of different materials separated by sharp interfaces, additional conditions must be supplemented. For instance, suppose that $\mathcal{B}$ is separated by a fixed interface $\mathcal{S}$ into complementary subregions $\mathcal{B}_\alpha$ and $\mathcal{B}_\beta$ occupied by the materials α and β. We write $\mathbf{n}$ for the unit normal on $\mathcal{S}$ directed outward from $\mathcal{B}_\alpha$. In this case, a basic assumption of theory is that the chemical potential μ and the displacement $\mathbf{u}$ are continuous across the interface, that is,

$$[\mu] = 0, \qquad [\mathbf{u}] = \mathbf{0}, \tag{13}$$

where $[f]$ denotes the jump in a field f across the interface. The additional interfacial conditions represent the force and mass balances localized at the interface

$$[\mathbf{T}]\mathbf{n} = \mathbf{0}, \qquad [\mathbf{h}] \cdot \mathbf{n} = 0. \tag{14}$$

Notice the continuity of the chemical potential at the interface is equivalent to following relation between c_α and c_β at the interface

$$c_\alpha = K_{eq} c_\beta \qquad \text{with} \qquad \ln K_{eq} = \frac{1}{k_B T}(\mu^0_\beta - \mu^0_\alpha - \eta_\beta \mathrm{tr}\mathbf{T}_\beta + \eta_\alpha \mathbf{T}_\alpha), \tag{15}$$

where K_{eq} can be written in terms of the Sieverts constants. In fact, it can be seen that if K_α and K_β are the Sieverts coefficients for each material. Then it follows from $(12)_2$ that

$$K_{eq} = \frac{K_\alpha}{K_\beta}. \tag{16}$$

3. HOLLOW CYLINDER

Let us consider a hollow cylinder, with inner radius A and outer radius B, subjected to an internal pressure p_a and an external pressure p_b. The chemical potentials μ_a and μ_b are prescribed at the inner and outer surfaces of the cylinder, respectively. The cylinder is long enough so that permeation in the longitudinal direction is neglected and the condition of plane strain prevails.

Therefore, the deformation and diffusion problem is axially symmetric and all the relevant quantities depend on the radial coordinate r and time t. In particular, the displacement $\mathbf{u}$ is totally described by the radial displacement u. This implies that the only relevant components of $\mathbf{E}$ are the radial and circumferential strains ϵ_r and ϵ_θ, which by their turn are related to u by the relations

$$\epsilon_r = \frac{\partial u}{\partial r}, \qquad \epsilon_\theta = \frac{u}{r}. \tag{17}$$

Notice that the longitudinal strain ϵ_z is equal to zero thanks to the plane strain assumption. The stress tensor $\mathbf{T}$ is defined in terms of the radial stress σ_r, the circumferential stress σ_θ, and the longitudinal stress σ_z, which, in view of $(9)_1$ and the plane strain condition, are written as

$$\begin{aligned}
\sigma_r &= \lambda(\epsilon_r + \epsilon_\theta) + 2G\epsilon_r - (3\lambda + 2G)\eta(c - c_0),\\
\sigma_\theta &= \lambda(\epsilon_r + \epsilon_\theta) + 2G\epsilon_\theta - (3\lambda + 2G)\eta(c - c_0),\\
\sigma_z &= \lambda(\epsilon_r + \epsilon_\theta) - (3\lambda + 2G)\eta(c - c_0).
\end{aligned} \tag{18}$$

The flux $\mathbf{h}$, defined by the radial flux J and using $(9)_3$, is given by

$$J = -\frac{Dc}{k_B T}\frac{\partial \mu}{\partial r} \tag{19}$$

with

$$\mu = \mu_0 + k_B T \ln c - \eta(\sigma_r + \sigma_\theta + \sigma_z). \tag{20}$$

The equations (17)-(18), along with the force $(3)_1$ and solute $(4)_1$ balances, simplifies to

$$\begin{aligned}
&\frac{\partial \sigma_r}{\partial r} + \frac{\sigma_r - \sigma_\theta}{r} = 0,\\
&\frac{\partial c}{\partial t} = -\frac{1}{r}\frac{\partial (Jr)}{\partial r}
\end{aligned} \tag{21}$$

supplemented by the following boundary conditions

$$\sigma_r(A,t) = -p_a, \quad \sigma_r(B,t) = -p_b, \quad \mu(A,t) = \mu_a, \quad \mu(B,t) = \mu_b. \tag{22}$$

In the case of a compound cylinder, these conditions must be supplemented by the continuity across the interface between different materials of σ_r, u, J and μ.

3.1 Weak form

The the theoretical model presented in the previous section has been implemented and subsequently solved numerically in the finite element software COMSOL Multiphysics (COMSOL (2013)). To implement the model we have used the Weak Form PDE mode, and the governing equations have to be recast in weak form as follows: find u and c, and boundary concentrations $c_a = c(A,t)$ and $c_b = c(B,t)$, such that, for any variables $\tilde{u}$, $\tilde{c}$ and $\tilde{\mu}$, it holds

$$\begin{aligned}
&\int_A^B \Big(\frac{\partial \tilde{u}}{\partial r}\sigma_r + \frac{\tilde{u}}{r}\sigma_\theta\Big) r\, dr - \tilde{u}(B)p_b + \tilde{u}(A)p_a = 0,\\
&\int_A^B \Big(\tilde{c}\,\frac{\partial c}{\partial t} - \frac{\partial \tilde{c}}{\partial r}J\Big) r\, dr - \tilde{c}(B)J(B) + \tilde{c}(A)J(A) = 0,\\
&\tilde{\mu}(A)\,\big(\mu(c_a) - \mu_a\big) = 0 \quad \text{and} \quad \tilde{\mu}(B)\,\big(\mu(c_b) - \mu_b\big) = 0.
\end{aligned} \tag{23}$$

Notice that the boundary conditions involving the chemical potential have been written in weak form. Further, they take the form of an implicit Dirichlet condition for the concentration c, being the balance of solvent written in terms of this state variable, and the concentration c_a and c_b cannot be assigned directly, cf. Lucantonio *et al.* (2013). So that, as in Lucantonio *et al.* (2012), we treat the boundary concentration c_a and c_b as additional state variables defined on the boundary where the chemical potential is assigned and employ a separate Physics Interface, that is, Weak Form PDE contribution on the boundary, for c_a and c_b, respectively, which is then computed by solving the algebraic equation $(23)_{3,4}$.

If the cylindrical membrane body is composed of different material separated by sharp interfaces, we need treating material discontinuities at the interfaces. Based on the interface conditions described above, the continuity of the displacement u renders the interface coherent, whereas the continuity of the chemical potential μ is the assumption of local chemical equilibrium. Thus, we have only one displacement u on both sides of the interface between regions, that is, $u_\alpha = u_\beta$. However, continuity of the chemical potential (i.e., $\mu_\alpha = \mu_\beta$) prevents continuity of the solute concentration. We note that Comsol program assume continuity of both flux and dependent variables as its default condition. As Plawsky (2014) observes, to handle interface conditions like $(14)_2$ and (15) we need to resort to a trick. In COMSOL, we must actually specify two separate solute concentrations one in each layer.

Granting the above, the weak form (23) applied to split layers may be written now as: find u and c such that

$$\begin{aligned}
&\int_{A_{(k)}}^{B_{(k)}} \Big(\frac{\partial \tilde{u}_{(k)}}{\partial r}\sigma_{r_{(k)}} + \frac{\tilde{u}_{(k)}}{r}\sigma_{\theta_{(k)}}\Big) r\, dr - \tilde{u}(B)p_b + \tilde{u}(A)p_a = 0,\\
&\int_{A_{(k)}}^{B_{(k)}} \Big(\tilde{c}_{(k)}\,\frac{\partial c_{(k)}}{\partial t} - \frac{\partial \tilde{c}_{(k)}}{\partial r}J_{(k)}\Big) r\, dr - \tilde{c}_{(\alpha)}J_c|_{\mathcal{S}_{\alpha\beta}} + \tilde{c}_{(\beta)}(-J_c)|_{\mathcal{S}_{\beta\alpha}} + \tilde{c}(B)J(B) - \tilde{c}(A)J(A) = 0,\\
&\tilde{\mu}(A)\,\big(\mu(c_a) - \mu_a\big) = 0 \quad \text{and} \quad \tilde{\mu}(B)\,\big(\mu(c_b) - \mu_b\big) = 0.
\end{aligned} \tag{24}$$

for all variables $\tilde{u}$ and $\tilde{c}$, with the summation implicit ($k = \alpha, \beta$). The flux $J_c = -K(c_\alpha - K_{eq}c_\beta)$ is enforced at the interface $\mathcal{S}$ between layers. We note that K is a positive constant chosen sufficiently large to enforce continuity of the potential. Here, we anticipate that this constant was identified by numerical experiments. The fields u, c, $\tilde{u}$ and $\tilde{c}$ belong to functional spaces that must be carefully selected, which are approximated by finite dimensional spaces by using the finite element method.

In all simulations, we have used one-dimensional quadratic Lagrange elements to interpolate the displacement u and the solute concentration c. For the time discretization we employed second-order backward differentiation formula (BDF), with the time steps controlled by the numerical solver during the computations. To solve the discrete system resulting from the discretization at each time step, we have used a MUMPS solver with residual tolerance levels of 10^{-4}. This was verified to be sufficient since similar solutions were obtained at lower tolerance levels.

4. NUMERICAL RESULTS

We employ the finite element model implemented into COMSOL Multiphysics in two numerical examples: the cylinder consisting of palladium material only and the cylindrical membrane composed of two materials. We set the inner cylinder diameter 8.0 mm and membrane thickness equal to 0.5 mm, corresponding to the geometry tested in laboratory by Baranowski (1989). We have considered the following hydrogen permeation experiment, cf. Adrover *et al.* (2003). The system is left to evolve until, after some transient, the pressure equalization to the constant pressure value p_0 at both sides of the cylindrical membrane (pressure of hydrogen at equilibrium with the metal surface), the pressure at the outer surface at radius B is suddenly increased to a value p_b greater than p_0. Hydrogen pressure at inner surface radius A is maintained constant, $p_a = p_0$. Importantly, the equilibrium conditions is assumed set instantaneously at the metallic membrane surface. That is, the hydrogen chemical potential at the metallic membrane surface equals the chemical potential of gaseous hydrogen in contact with the surface, as it was previously discussed. Here, we assumed that the tubular membrane is under a prescribed constant pressure $p_0 = 3.2$ kPa and $p_b = 4p_0$ at the temperature of 833 K. The values of the material parameters of the model used in the numerical examples are shown in Tab. 1. We denoted with L the membrane thickness, and the dimensionless time is defined as the ratio $t/(L^2/D)$.

Table 1. Numerical values of the parameters of the model.

Parameter	Palladium	Niobium
Young's modulus E (GPa)	121	105
Poisson's ratio ν	0.39	0.40
Diffusivity D (m^2/s)	5×10^{-8}	3×10^{-8}
Coefficient η (m^3/mol H)	1.73×10^{-6}	1.73×10^{-6}
Permittivity PM (mol/m s $Pa^{1/2}$)	121	105

First, we have considered the problem of a tubular membrane consisting of palladium material only. The results obtained were in agreement with those presented in the literature (see Androver *et al.*, 2003). For the sake of saving space, the results are neither presented or discussed here.

The second benchmark problem that we consider involves a tubular multilayer membrane composed of two materials, namely palladium and niobium. In this case, as is shown in Fig. 1, the hydrogen concentration undergoes a stress-dependent jump across an interface between two materials, a result that follows from the continuity of the chemical potential. Figure 2 shows the dimensionless stress profiles for multilayer membrane composed of palladium-niobium-palladium. The time behavior of hydrogen flux at the inner wall surface is shown in Fig. 3.

5. CONCLUSIONS

This paper addressed the issue of stress effects on hydrogen permeation through tubular multilayer membranes by using modeling and numerical simulations. The analysis is carried out using continuum equations, including interface and boundary conditions, that govern solute diffusion and solid deformation within a small deformation setting. These

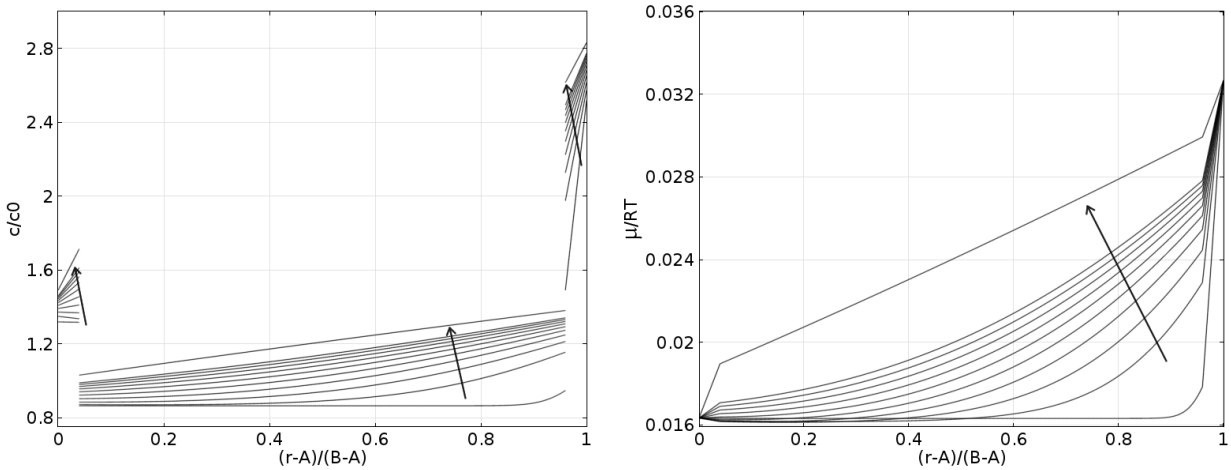

Figure 1. Hydrogen permeation in a tubular multilayer membrane. (left) hydrogen concentration, (right) continuity of the chemical potential across the interfaces between materials.

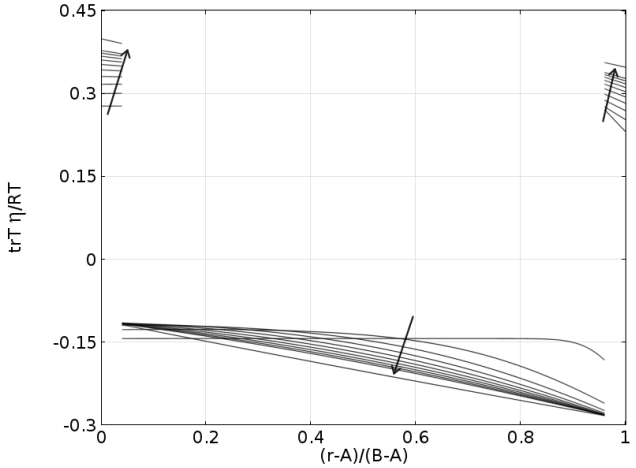

Figure 2. Dimensionless stress profiles.

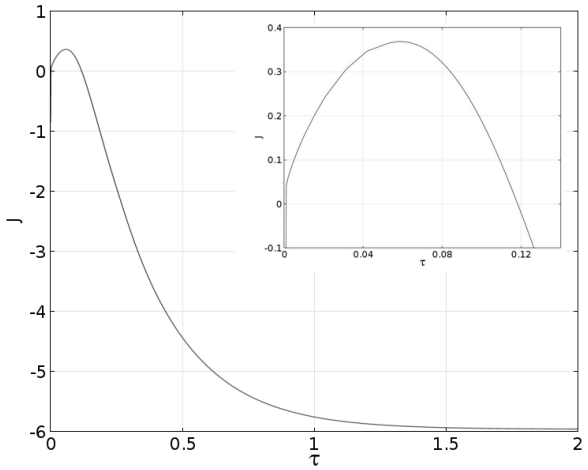

Figure 3. Time behavior of the hydrogen flux at the inner wall surface for hydrogen permeation in a tubular multilayer membrane.

equations are specialized for the case of a hollow cylinder, which also contains the essential ingredients of the numerical model. It was that hydrogen concentration undergoes a stress-dependent jump across an interface between two materials, palladium and niobium, a result that follows from the continuity of the chemical potential. A finite-element model, in which hydrogen concentration is approximated by discontinuous functions and the corresponding interface condition accounted for in a weak manner corresponding, was developed and implemented using COMSOL Multiphysics. Results

showed a great potential of the theory and the numerical approach to describe stress effects on hydrogen permeation through tubular multilayer membranes.

6. ACKNOWLEDGEMENTS

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