



Effect of residual stresses on hydrogen permeation in iron

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ABSTRACT

The effect of residual stresses on electrochemical permeation in iron membrane was investigated. Four thermal and mechanical treatments were chosen to obtain different surface states in relation to the residual stresses.

Residual stresses were determined by X-ray diffraction (XRD) using the Macherauch and Müller method. The results were completed by the microhardness measurements. For all iron membranes, compressive residual stresses were obtained.

Electrochemical permeation experiments using a Devanathan and Stachurski cell were employed to determine the hydrogen permeation behaviour of the various iron membranes. The latter was charged with hydrogen by galvanostatic cathodic polarization in 0.1 M NaOH at 25 °C. The experimental results revealed that hydrogen permeation rate increases with increasing residual stresses introduced in iron membranes.

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1. Introduction

The absorption of hydrogen in metals is a serious problem for many electrochemical processes such as electroplating of metals or storing of hydrogen in tanks at high pressure for automotive industry. This absorption leads to hydrogen embrittlement of metals, and therefore modifications of the mechanical properties of the material with occasional stress corrosion cracking [1]. The diffusion of hydrogen in metals and more particularly in iron has been extensively discussed in previous papers [2–7]. Several papers deal with hydrogen permeation through metal using the Devanathan two-compartment cell, mostly with the aim of determining the hydrogen diffusion coefficient [1]. Stresses introduced by surface treatments such as shot-peening or mechanical abrading have been shown to affect the hydrogen diffusion and distribution in iron base alloys and their subsequent embrittlement [8]. The resulting delay in the hydrogen permeation can also depend on the level of the residual stresses. Nevertheless, many authors use mechanically abraded metal membranes in hydrogen permeation studies without taking into account the effect of residual stresses [3,4,6,7,9].

The purpose of this study was to investigate the effect of residual stresses on the hydrogen permeation in iron membrane. The residual stresses introduced by surface treatments were quantified by the well-known XRD $\sin^2\psi$ method using a diffractometer with

ψ -goniometer geometry. This method established by Macherauch and Müller has been fully described in 1961 [10]. Amongst all stress measurement techniques, X-ray diffraction is one of the most popular methods because it enables a non-destructive evaluation of surface stresses of crystalline materials [11,12] and does not require special specimen preparation in contrast with conventional bending beam techniques.

2. Experimental

2.1. Sample preparation and characterization

Membranes were cut from a rod iron with purity of 99.99% and 2.5 cm diameter. The samples were first mechanically abraded and then heat treated to remove residual stresses. Four treatments were chosen to obtain different surface states in relation to the residual stresses.

- (1) *Specimen S₁*: This sample was mechanically abraded with Carbimet-SiC grinding paper down to 1200 grit then rinsed with distilled water and cleaned with acetone. This sample preparation is widely employed for membranes used to hydrogen permeation study.
- (2) *Specimen S₂*: This sample was heat treated, then mechanically abraded with Carbimet-SiC grinding paper down to 1200 grit, rinsed with distilled water and cleaned with acetone. The heat treatment was conducted at 600 °C for 9 h under an argon atmosphere.

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- (3) *Specimen S₃*: This sample was heat treated as in (2) then rolled at 30% of thickness and mechanically abraded with Carbimet–SiC grinding paper down to 1200 grit, rinsed with distilled water and cleaned with acetone.
- (4) *Specimen S₄*: This sample was heat treated as in (2) and electrochemically polished. The electrochemical polishing was conducted with stirring as shown in Table 1.

The microhardness of iron membranes was measured by using a Shimadzu microhardness tester equipped with a Vickers diamond indenter and 25 g of load.

The roughness of iron membranes was measured by three-dimensional profilometry recordings.

The residual stresses were studied by X-ray Diffraction (XRD) using a D8 advance Bruker with CuK α radiation ($\lambda = 1.5418 \text{ \AA}$) by means of two methods: (1) qualitatively by analyzing the shift and width of the diffraction peaks and (2) quantitatively by using the $\sin^2\psi$ method.

XRD diagram was performed on the specimen S₂ (with Bragg-Bretano configuration). The 2θ range of 40° – 140° was recorded at scan speed of $0.05^\circ \text{ s}^{-1}$ with 0.005° step (Fig. 1).

Qualitative analysis of residual stresses was carried out around the peak registered between 44° and 46° which is the most intense. The 2θ range of 44° – 46° was recorded at scan speed of $0.001^\circ \text{ s}^{-1}$ with 0.005° step.

For the quantitative analysis; the X-ray diffraction (XRD) techniques exploit the fact that when a metal is under stress, applied or residual, the resultant elastic strains cause the atomic planes in the metallic crystal structure to change their spacings. XRD is capable of directly measuring this interplanar atomic spacing, and from this quantity, the total stress on the metal can be obtained [11–15].

In this study, we used the (2 2 2) lattice plane, corresponding to $2\theta \approx 137^\circ$, to determine the residual stresses in iron membranes since the X-ray stress measurement at high angle 2θ is highly precise.

The $\theta/2\theta$ scans were measured around the (2 2 2) Bragg diffraction peak at tilt angles ψ between -63 and $+63^\circ$ for all samples. By

Table 1
Operating condition for the electrochemical polishing.

Electrolyte:	(500 mL HClO ₄ + 500 mL ethanol + 5 mL H ₂ O)
Current density:	40 A dm ⁻²
Time:	1 min
Temperature:	Room temperature

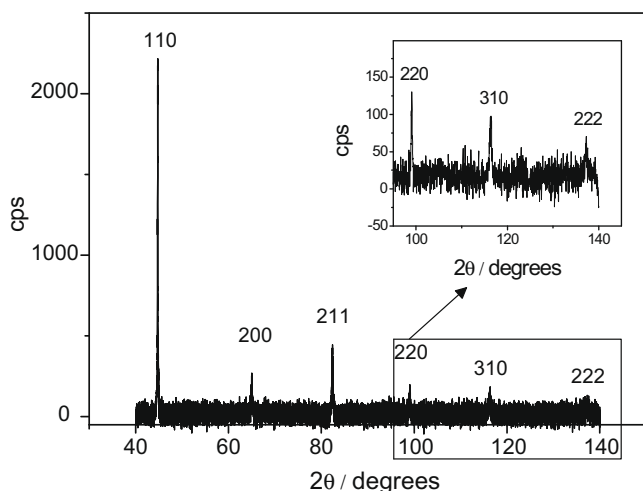


Fig. 1. X-ray diffractogram of specimen S₂.

fitting the (2 2 2) X-ray curves with a double Gaussian, the central peak positions belonging to the CuK α ₁ line were obtained for all ψ -values. The strain value ϵ_ψ was then calculated and plotted as a function of $\sin^2\psi$. ϵ_ψ is related to internal stress σ_r following the expression

$$\epsilon_\psi = \frac{1 + \nu}{E} \sigma_r \sin^2 \psi \quad (1)$$

where ψ is the angle through which the sample is tilted, E is the Young's modulus, ν is the Poisson's ratio.

2.2. Hydrogen permeation tests

2.2.1. Experimental procedure

In the course of electrochemical permeation, hydrogen atoms were first absorbed at the entry surface, then diffused through the metallic membrane, and were finally desorbed from the exit surface. On the entry surface, the production of hydrogen could be controlled galvanostatically or potentiostatically. On the exit surface, it is common to apply a constant potential to ensure that all hydrogen atoms could be ionized, ensuring that the measured current density was the hydrogen permeation flux. The instrumentation of electrochemical hydrogen permeation was composed of an electrolytic cell with two compartments; cathodic and anodic sides. A detailed discussion of the instrument used in this study is given elsewhere [6,7]. The experimental conditions are summarized in Table 2.

The permeation curves obtained after interrupting the cathodic charging were compared for the different residual stresses. The passivation time before charging was strictly controlled and was the same for all the tests. At least five permeation tests were carried out in each case.

2.3. Data analysis

The flux of hydrogen through the membrane measured in terms of the steady-state current density, I_p^∞ (A m⁻²), and converted to the hydrogen permeation flux, J_∞ (mol m⁻² s⁻¹), according to the following equation and resulted directly from the Fick's first law:

$$J_\infty = \left(-D \frac{\partial C}{\partial X} \right)_{X=L} = I_p^\infty / nF \quad (2)$$

where D is the hydrogen diffusion coefficient (m² s⁻¹), I_p^∞ indicates the steady-state permeation current density, n the number of electrons transferred, F the Faraday's constant, L (m) the membrane thickness and J_∞ the flux [3,7,16,17]. According to Eq. (2), permeation flux J_∞ increase proportionally to permeation current density I_p^∞ .

Table 2
Experimental procedure for hydrogen permeation tests.

Thickness of samples:	1.3 mm
Useful surface:	1.76 cm ²
Preparation of samples:	See sample preparation
Cathodic and anodic solutions:	0.1 M NaOH de-aerated by argon bubbling 1 h before testing and throughout the test.
Cathodic charging current density:	-2 mA cm^{-2}
Imposed potential at the detection side:	$-220 \text{ mV vs. reference electrode}$
Temperature:	$25 \pm 0.5^\circ \text{ C}$
Passivation time before cathodic charging:	23 h
Permeation time before interruption of cathodic charging:	16 h
Reference electrode:	Hg/HgO/NaOH 0.1 M
Counter electrode:	Platinum wire

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