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Effect of cyclic loading on hydrogen diffusion in low carbon steels

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STUDY OF HYDROGEN DIFFUSION AND EMBRITTLEMENT IN LOW-ALLOYED STEELS UNDER CATHODIC PROTECTION

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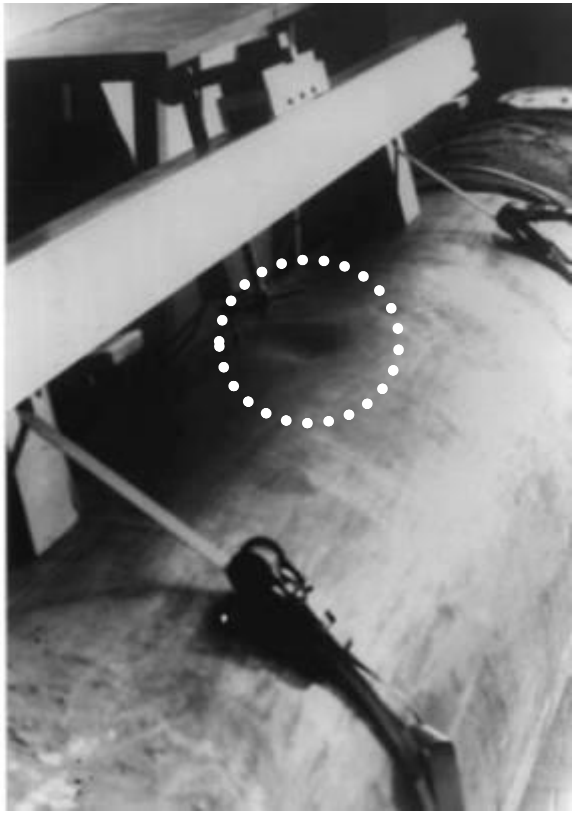
Susceptibility to HE of HSLA steels

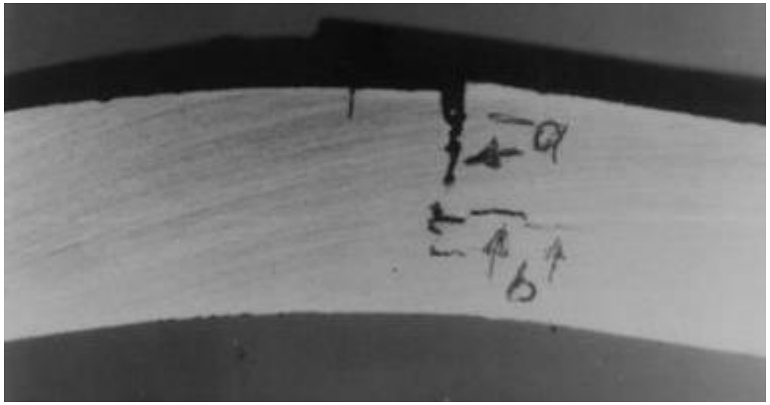
- Pipeline steels are **immune** to HE under cathodic protection also in the presence of:
  - Very low potentials (over-cathodic protection)
  - Very high constant load (higher than yield strength)

- Possible occurrence of HE phenomena on pipeline steels under cathodic protection in the presence of **dynamic loading conditions**

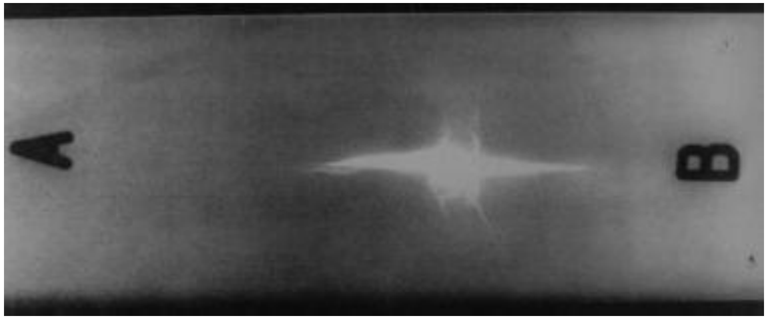
Case history of HE failure of a buried pipeline due to landslides



Internal surface of pipe

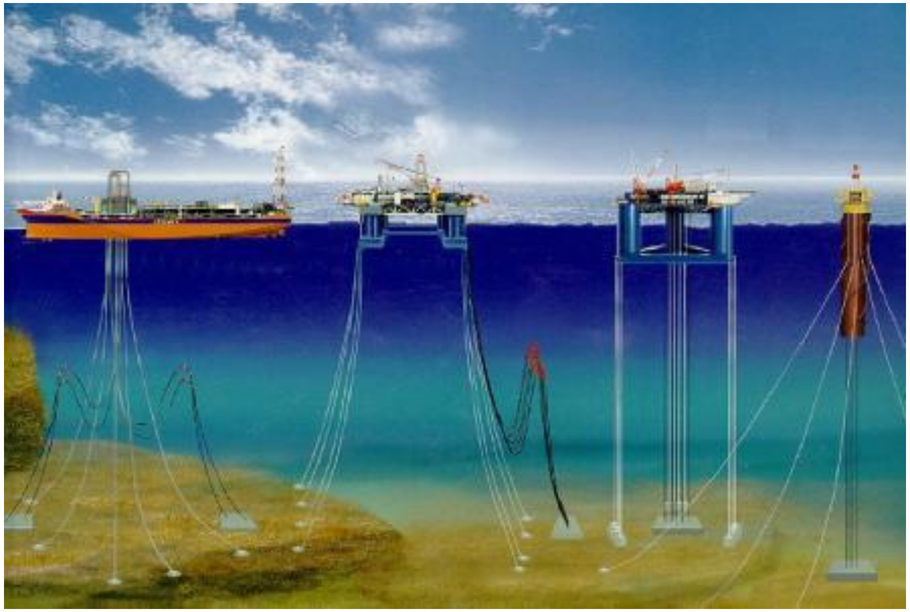


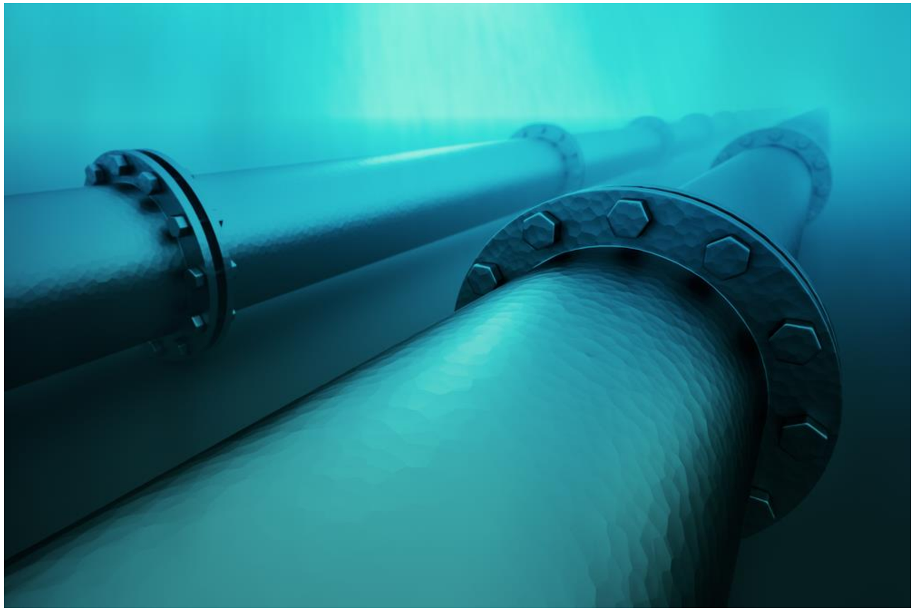
External surface of pipe



M. Cabrini et al., "Unique HSC scenario leads to gas line failure", OIL GAS J, 98(10), 2000, pp. 61-65

Corrosion-fatigue and HE in marine structures





Regulatory requirements for HE prevention in carbon steels and low-alloyed steels

ISO 19902:2007

→ HE susceptibility increases with the yield strength for steels with a SMYS in the range 460 ÷ 500 MPa

EN 12473:2014

→ C-Mn and low-alloyed steels (SMYS $\leq$ 550 MPa)

E_P = -1.10 V vs Ag/AgCl/SW

→ High strength steels (SMYS > 550 MPa)

E_P = -0.83 V ÷ -0.95 V vs Ag/AgCl/SW

Regulatory requirements for HE prevention in carbon steels and low-alloyed steels

ISO 15589-1:2015

→ SMYS ≤ 550 MPa

E_P = -0.85 V and -0.95 V vs SCE

(Additional limit of E_P = -1.20 V vs SCE reported only for the prevention of coating disbonding/blistering)

→ SMYS > 550 MPa

Necessity to determine experimentally the **critical limit** for the prevention of the negative effects of hydrogen development on the surface

⬇

No specific test or evaluation criterion specified for this limit

Regulatory requirements for HE prevention in carbon steels and low-alloyed steels

ISO 7539-11:2013

«A fundamental question is how long should a laboratory test be to ensure that hydrogen uptake is sufficient in reflecting behavior in service, for which exposure times are of the order of years»

«… pre-*charging may be pertinent to ensure that uptake of hydrogen is significant. For some circumstances, such as cathodic polarization, it is relatively straightforward to obtain an approximate estimate of the time evolution of the hydrogen concentration, using Fick's second law with an effective diffusivity»*

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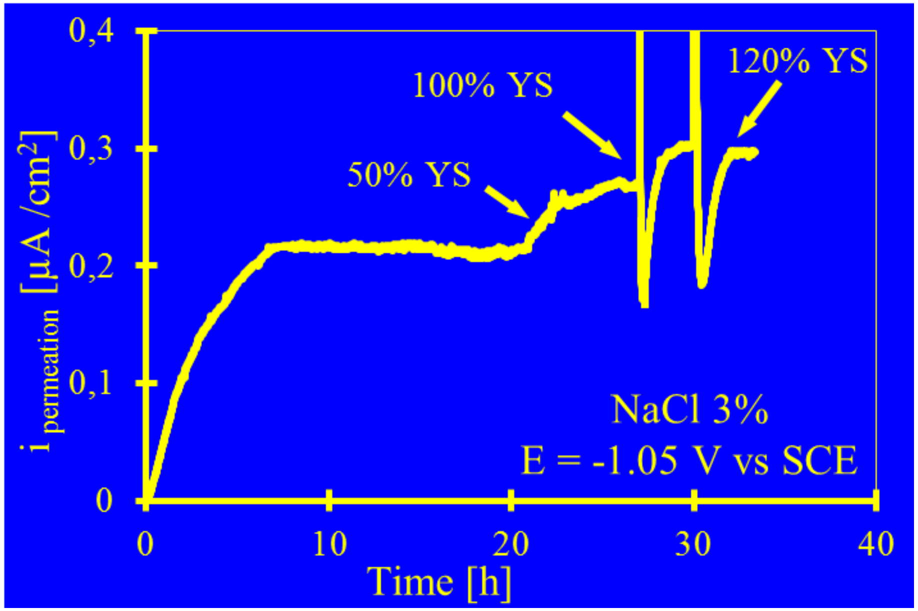
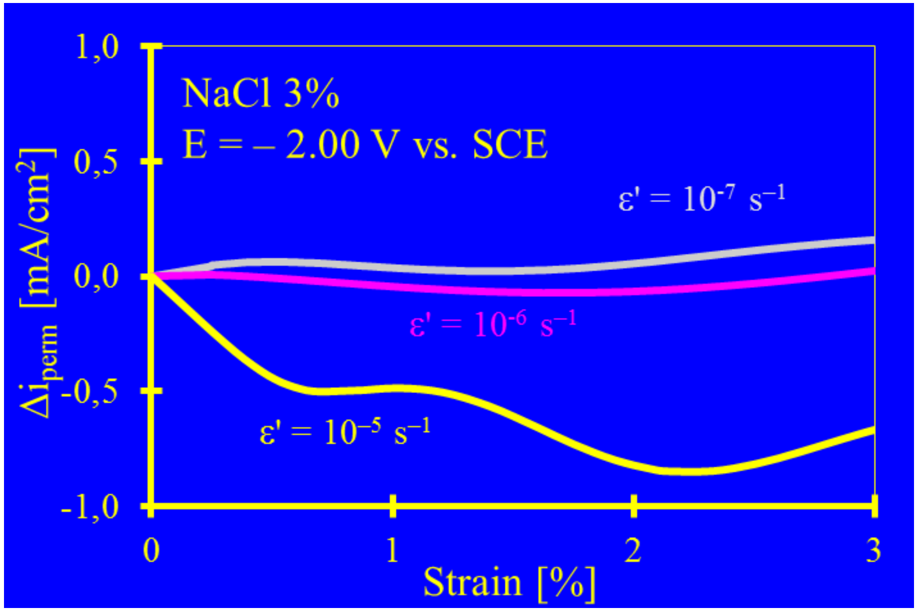
Hydrogen permeation flux depends on the loading conditions

Example of the effect of loading condition on hydrogen diffusion in X60 steel

Constant load



Slow strain rate



M. Cabrini et al., CORROSION REVIEWS, Vol. 33, 2015, pp. 529-545

Aim of the research activity during the PhD program

Study of hydrogen diffusion and embrittlement in high strength low-alloyed steels under cathodic protection:

- Analysis of hydrogen permeation process, by means of a electrochemical permeation technique through thin steel foils, in the presence of static and dynamic loading, in the elastic and plastic field

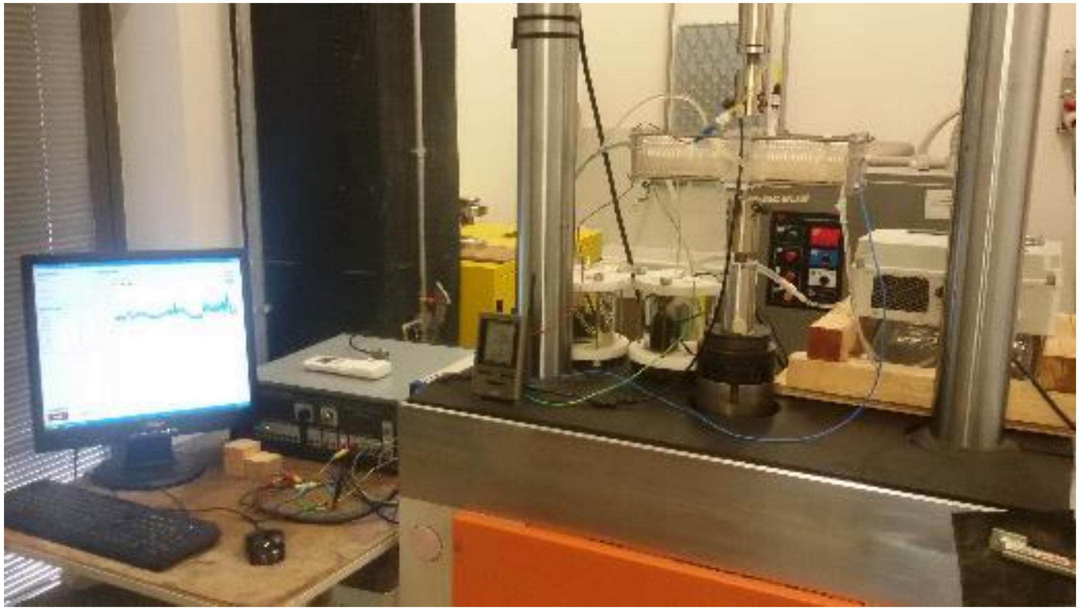
Experimental tests

Test conditions:

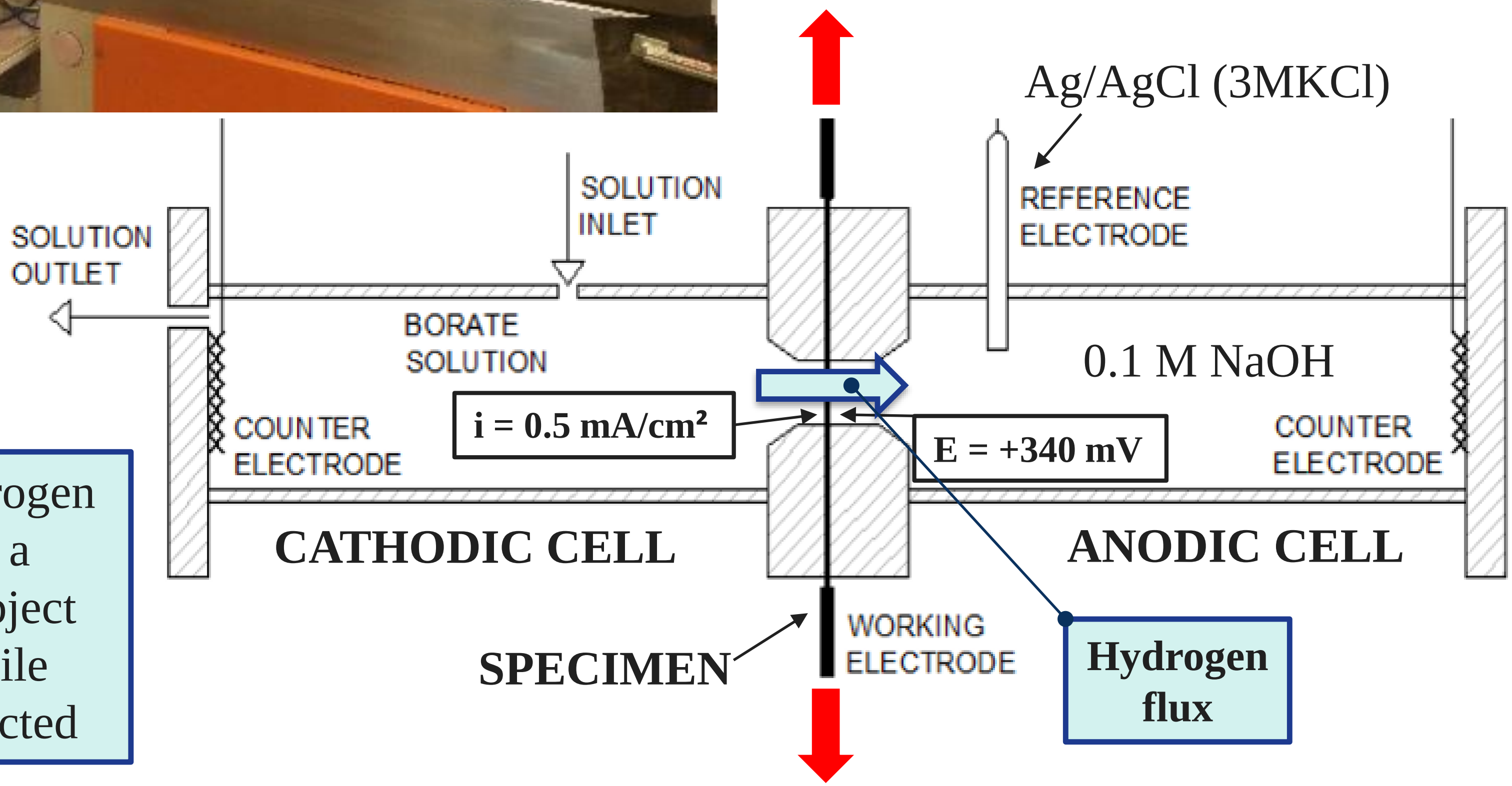
- In the absence of loading:
  - As-received material without pre-straining → **BM**
  - Pre-strained (tensile) material at 100/110% TYS → **PT**
  - Pre-strained (compressive) material at 100/110% TYS → **PC**
  - Pre-strained (tensile/compressive) material at 110% TYS → **PTC**

- In the presence of tensile loading:
  - Material subject to cyclic load, sine waveform, maximum load 55÷110% TYS, amplitude ±10/20% TYS, frequency $10^{-2} \div 1$ Hz → **C**
  - (Material subject to incremental step load, according to the ASTM F1624-12, both for the as-received and thermally treated material → **S**)

Permeation test under loading
(ISO 17081:2014)

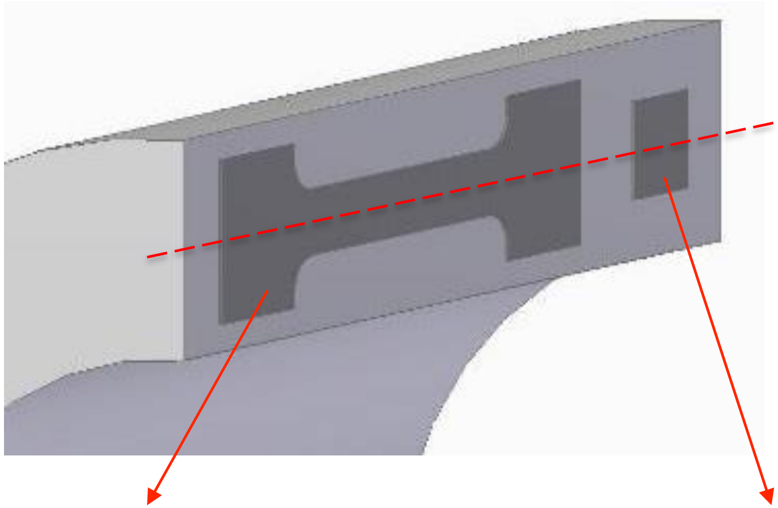


$$P = P_0 + \Delta P \cos(2\pi f t)$$



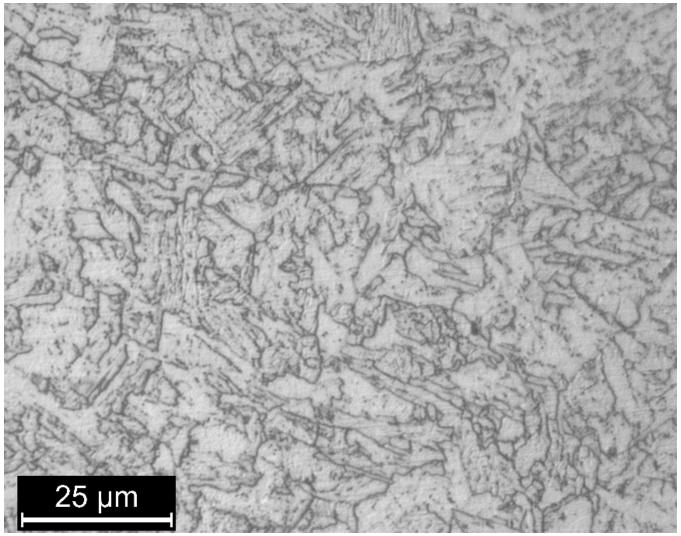
Detection of hydrogen diffusing through a thin steel foil, subject to a dynamic tensile loading, was detected

Specimens for permeation tests

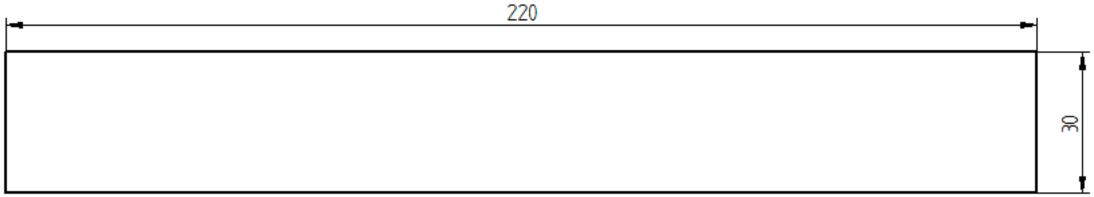


Tests on pre-strained (tensile) material, or under cyclic tensile loading, (or under incremental step loading)

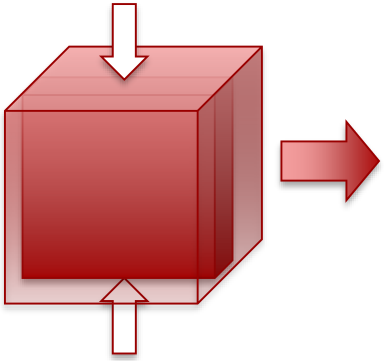
Tests on as-received material

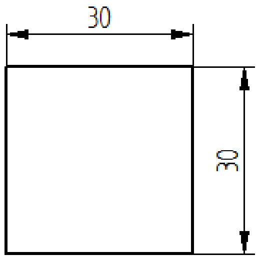


Ferritic-bainitic microstructure of the X65 grade steel



Specimen for tests on pre-strained (tensile/compressive) material





Thin foils (obtained from a prismatic specimen) for tests on pre-strained (compressive) material

Permeation curves processing

Evaluation of the apparent diffusivity (D_{app})

Interpolation of the experimental curves by means of the pure diffusion model through a steel foil

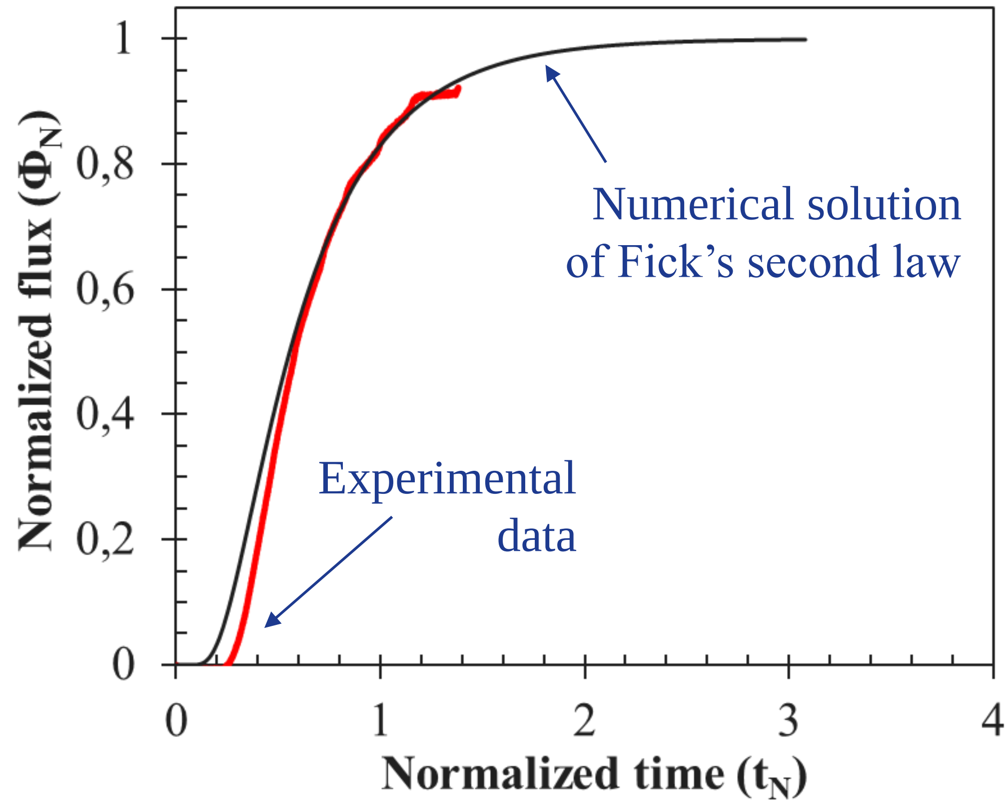
Determination of:

- Average apparent diffusivity, D_{app}
- Total sub-surface hydrogen concentration:

$$C_{0,tot} = \frac{s \cdot \emptyset_\infty}{D_{app}}$$

- Estimate of the apparent diffusivity variation depending on the adimensional time

$$D_{app} = f(t_N)$$



$$\emptyset_N = \frac{\emptyset(t)}{\emptyset_\infty} = \frac{i(t)}{i_\infty} \qquad t_N = \frac{4D_{app} \cdot t}{s^2}$$

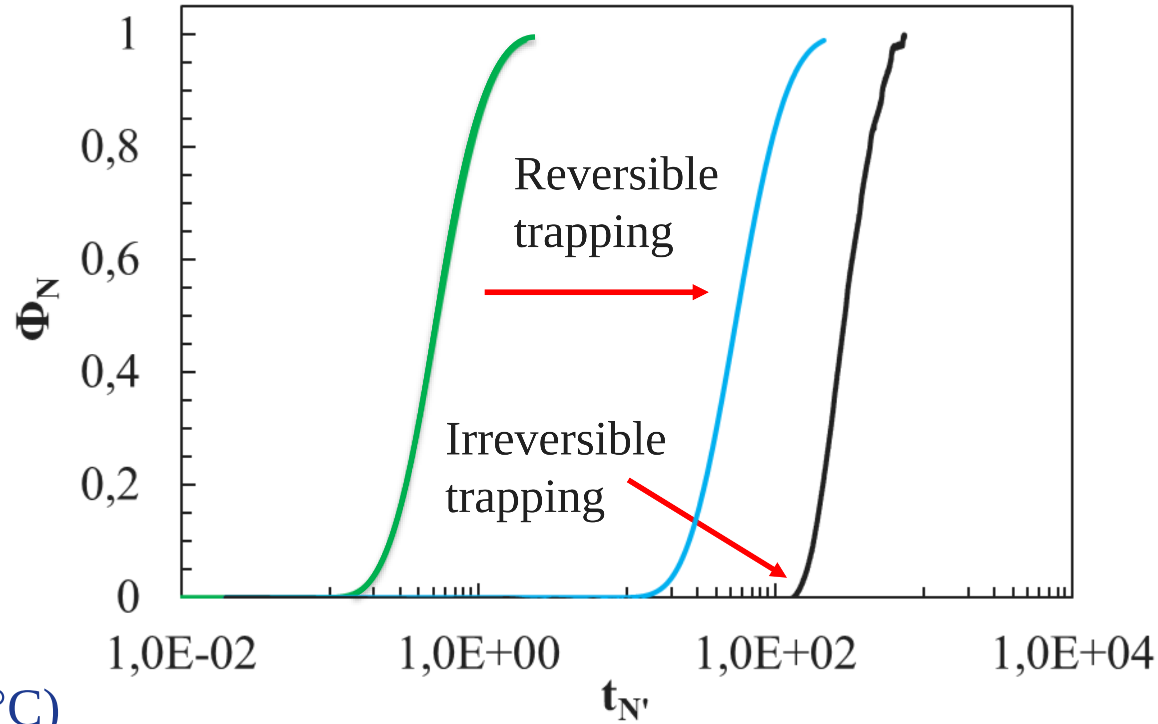
Normalization of the permeation curves

Expressed as a function of the normalized flux and the normalized time ($t_{N'}$)

$t_{N'}$ = normalized time to the lattice diffusivity

$$t_{N'} = \frac{4D_l \cdot t}{s^2}$$

Systematic variations of the experimental permeation curve with respect to the pure diffusion curve in the ferrite



D_l = lattice diffusivity (bcc-iron)
= $7.17 \cdot 10^{-9}$ [m^2/s] (T=23°C)

(K. Kiuchi and R.B. McLellan)

1) As-received material → ***BM***

2) Cyclically loaded material → ***C_110***

Permeation curves processing

Evaluation of the apparent diffusivity (D_{app})

R. Oriani, "The diffusion and trapping of hydrogen in steel", ACTA METALLURGICA, vol. 18, no. 1, pp. 147-157, 1970

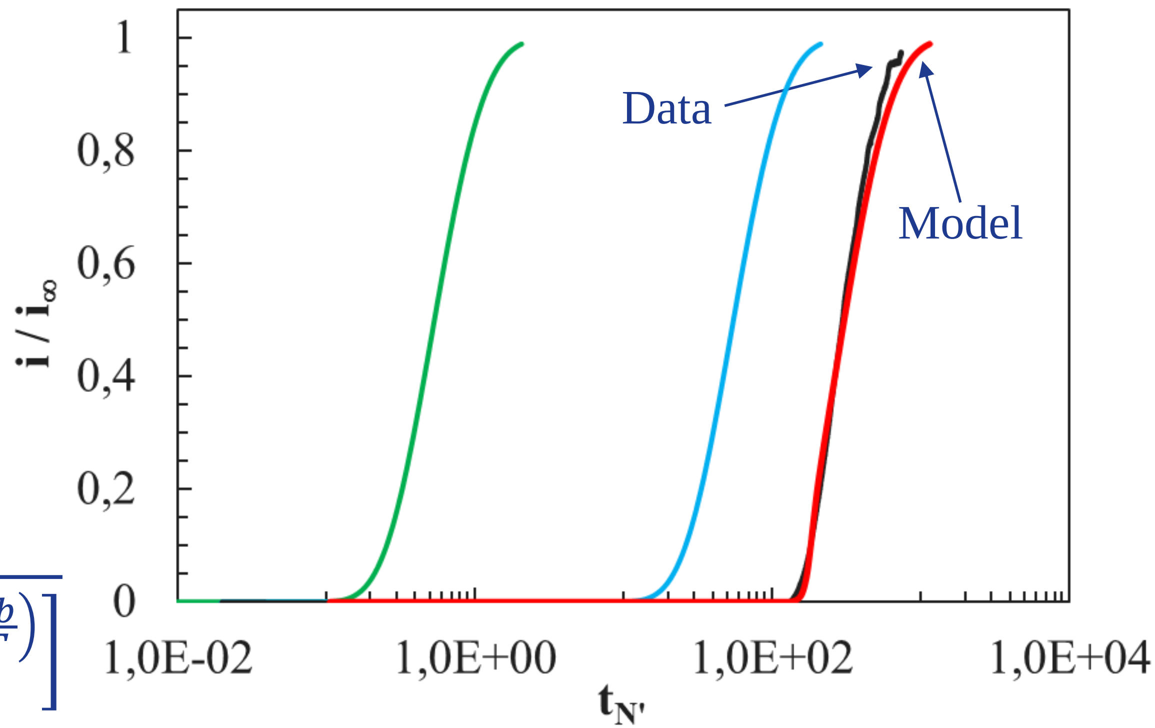
$t_{N'}$ = normalized time to the lattice diffusivity

$$t_{N'} = \frac{4D_l \cdot t}{s^2}$$

Modification to Oriani's model (presence of only reversible traps) to take into consideration the effect of **irreversible traps**

$$D_{app}[m^2/s] = \frac{D_l}{\left[1 + \frac{N_r}{N_l} \cdot e^{\left(\frac{-E_b}{R \cdot T}\right)}\right]}$$

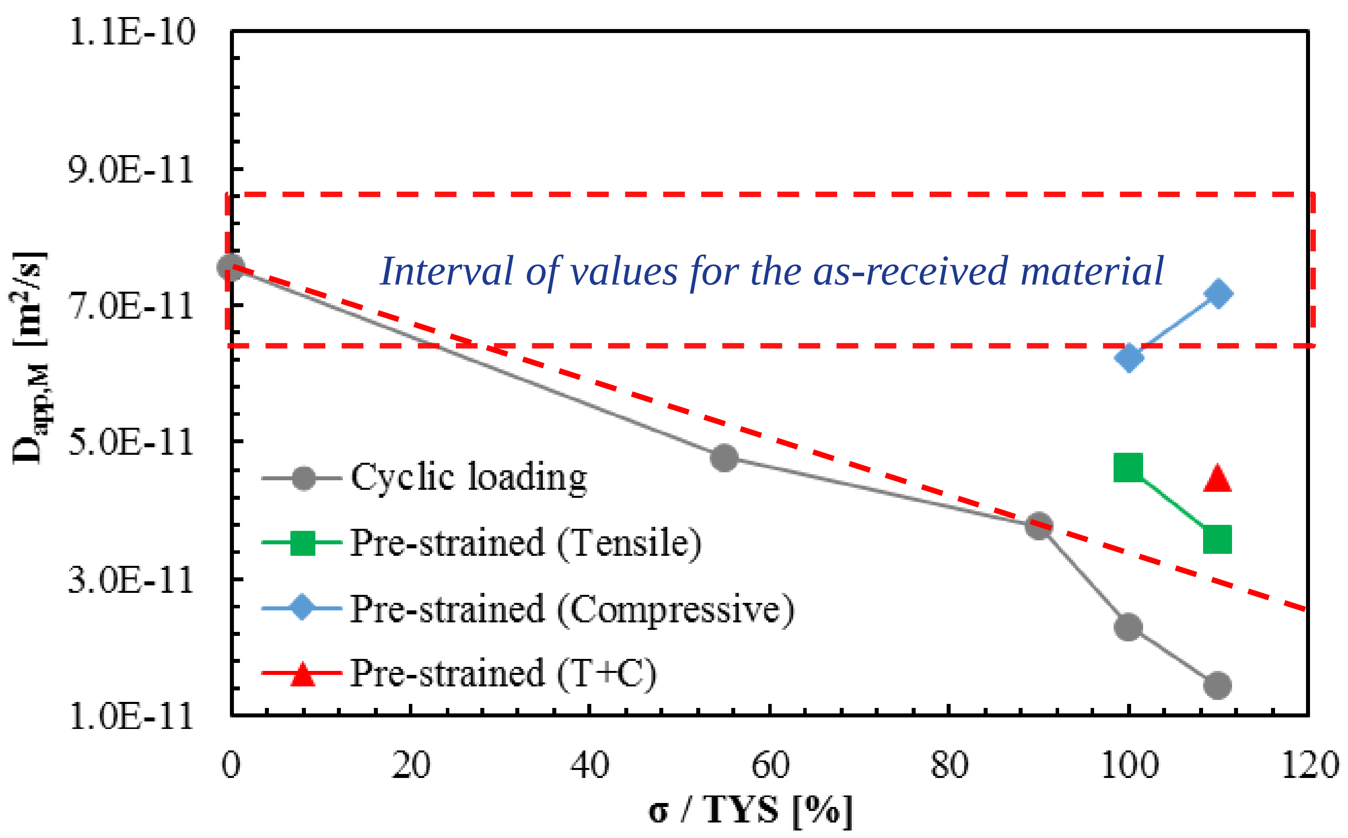
$$N_{t,r}\left[\frac{sites}{cm^3}\right] = \frac{D_l}{D_{app}} \cdot N_l \cdot e^{\left(-\frac{E_b}{R \cdot T}\right)}$$



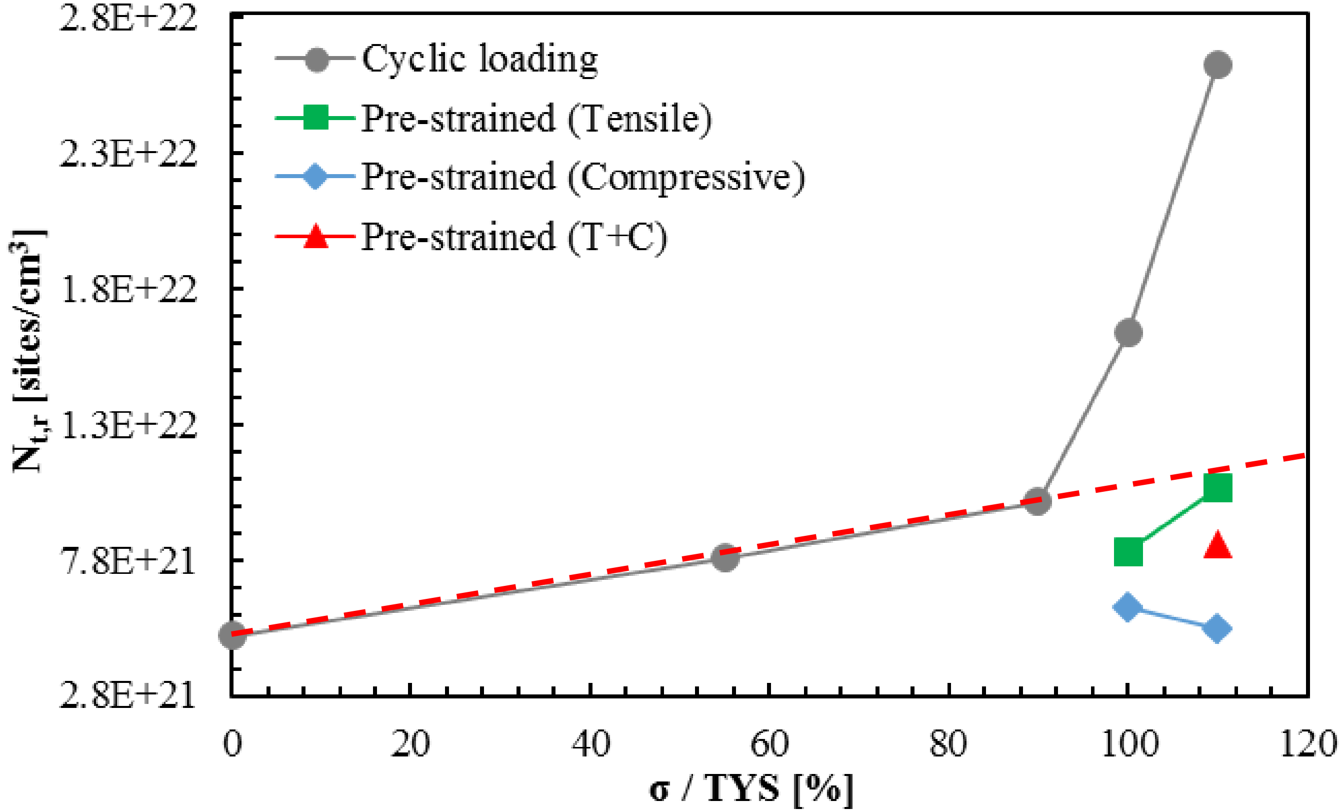
*1) As-received material → **BM***

*2) Cyclically loaded material → **C_110***

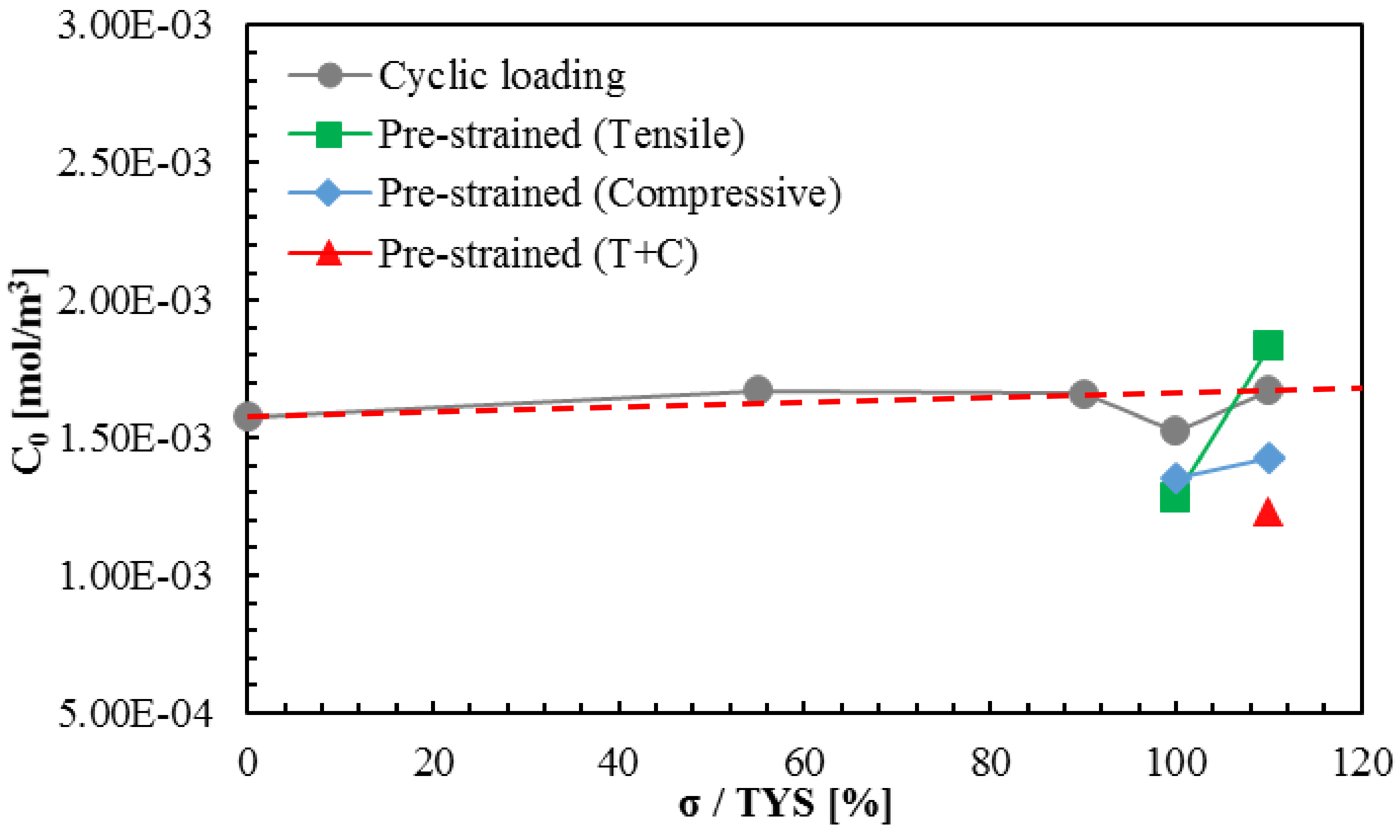
Effect of the maximum stress on the apparent diffusivity (D_{app})



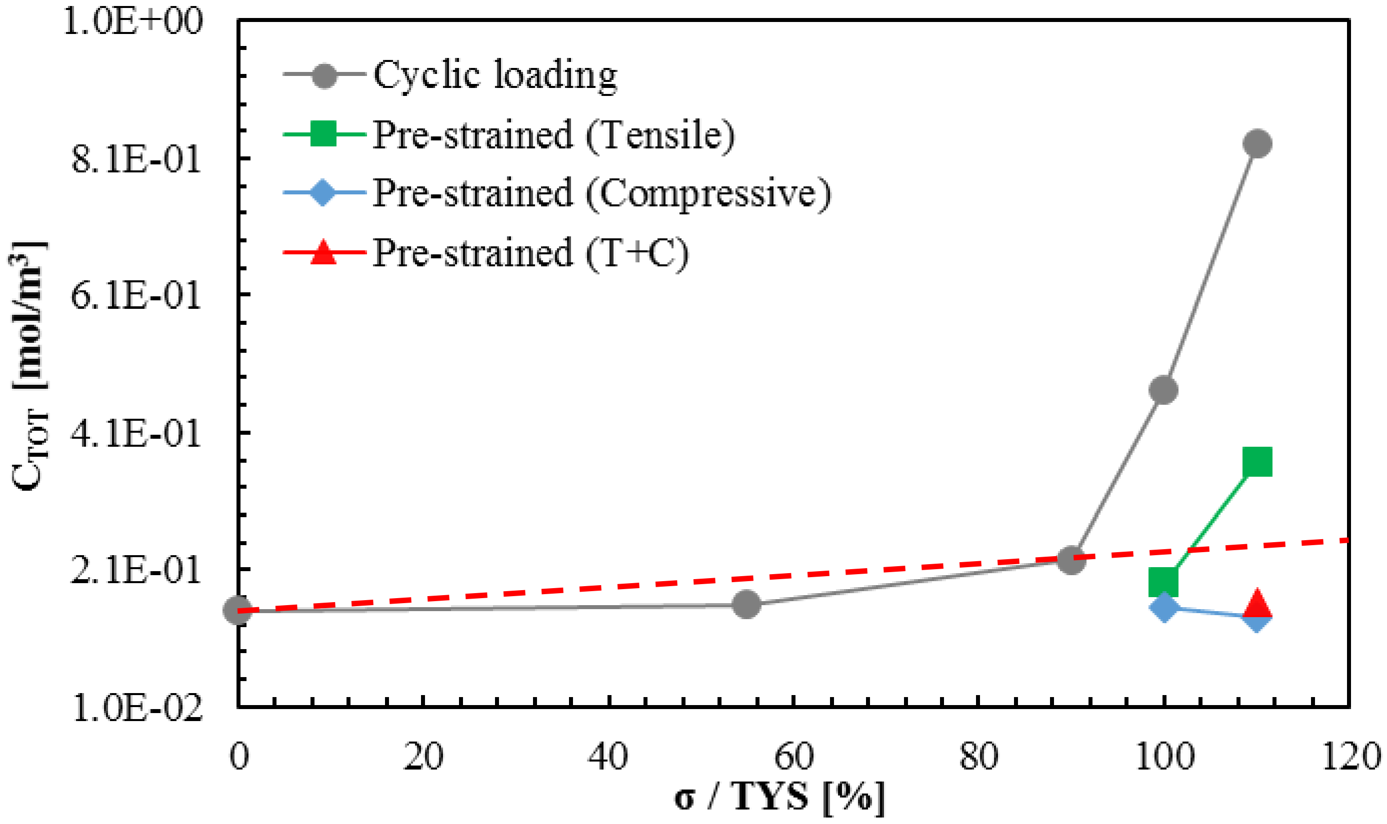
Effect of the maximum stress on the number of reversible traps ($N_{t,r}$)



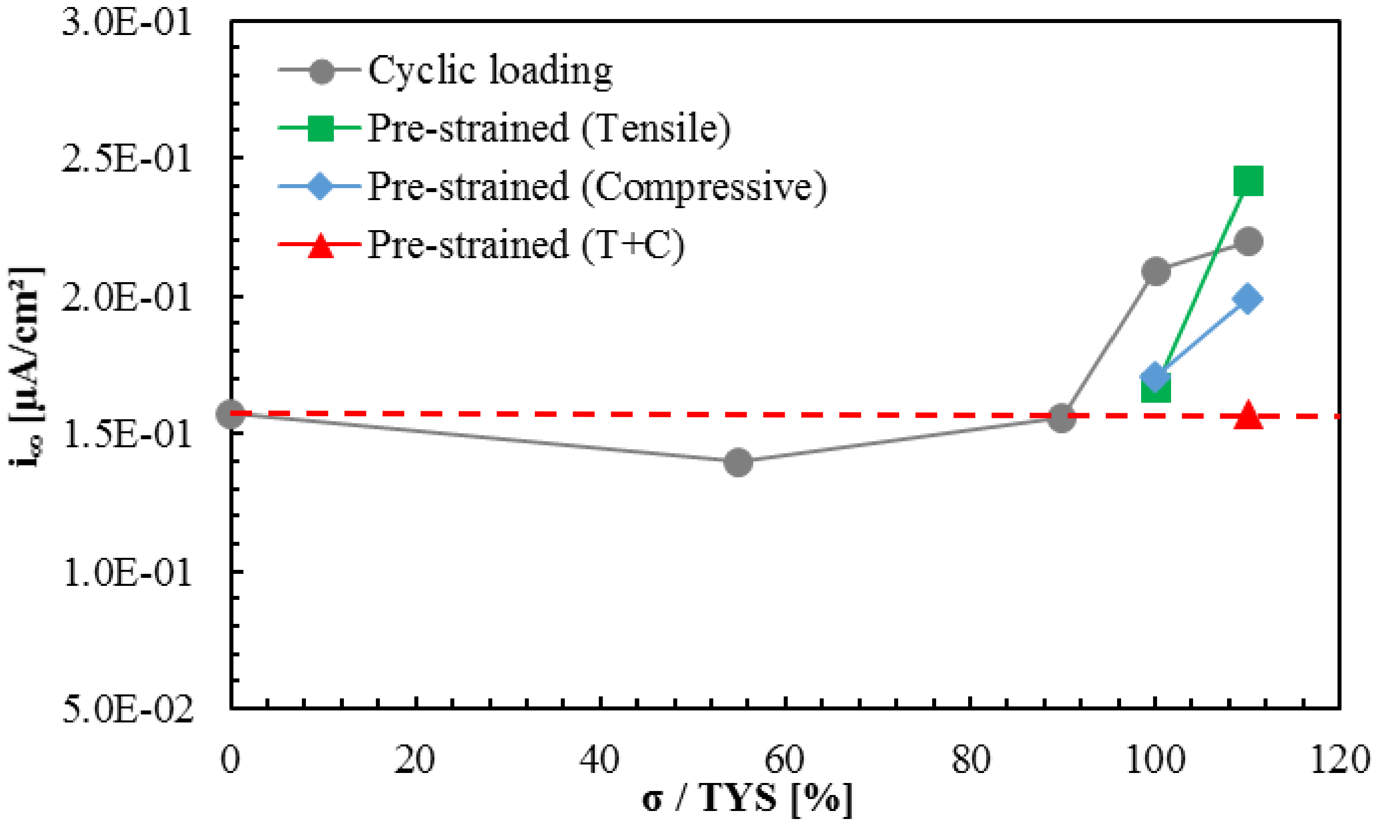
Effect of the maximum stress on the sub-surface hydrogen concentration in interstitial lattice (C_0)



Effect of the maximum stress on the total sub-surface hydrogen concentration (C_{TOT})



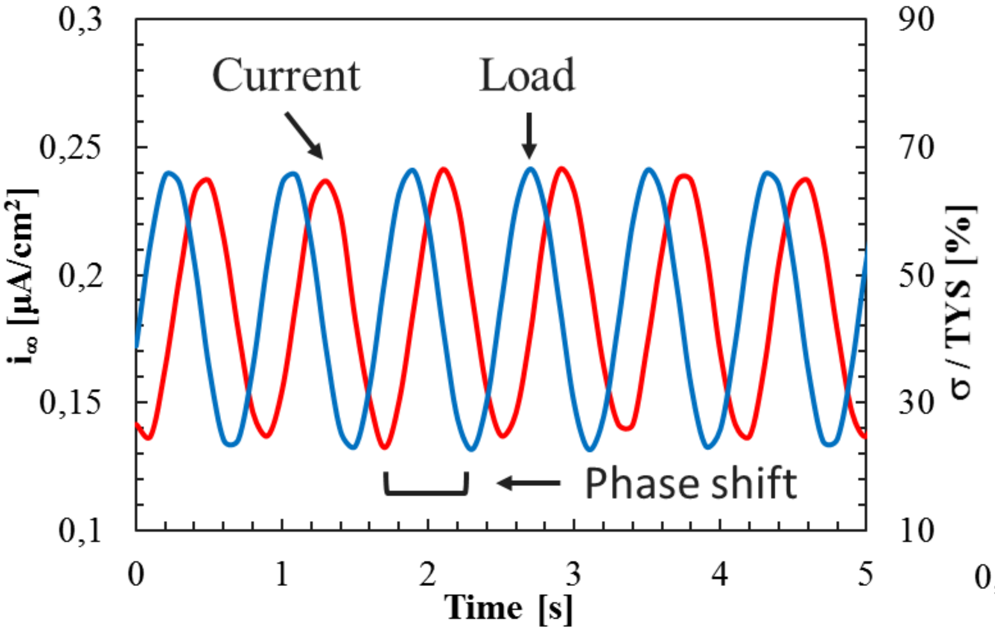
Effect of the maximum stress on the steady-state current (i_∞)



Effect of the alternate component of the load on steady-state current (i_∞) and passivity current (i_P)

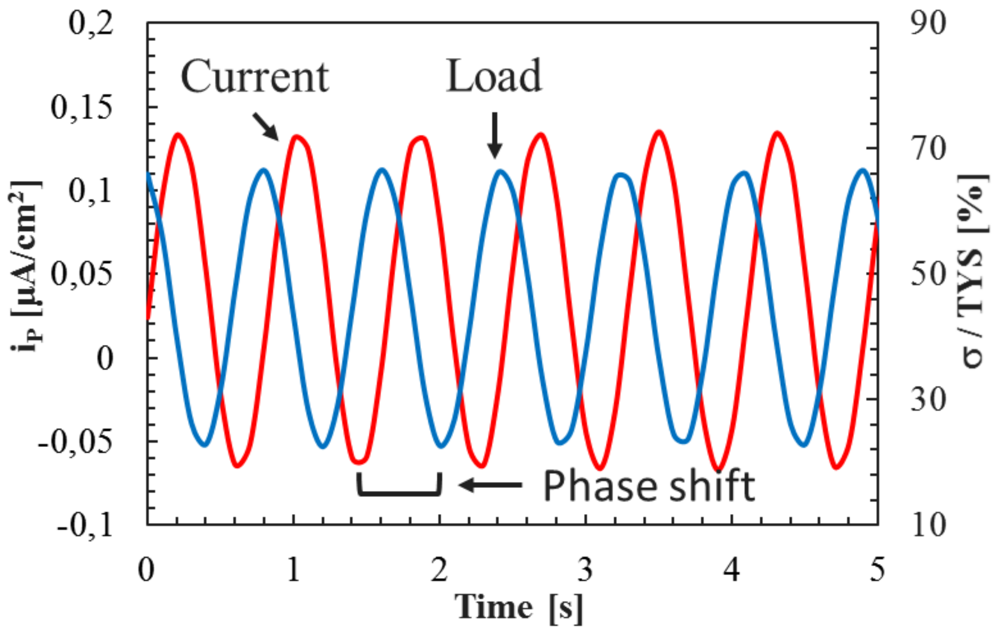
Amplitude = ±20% TYS

Frequency = 1 Hz

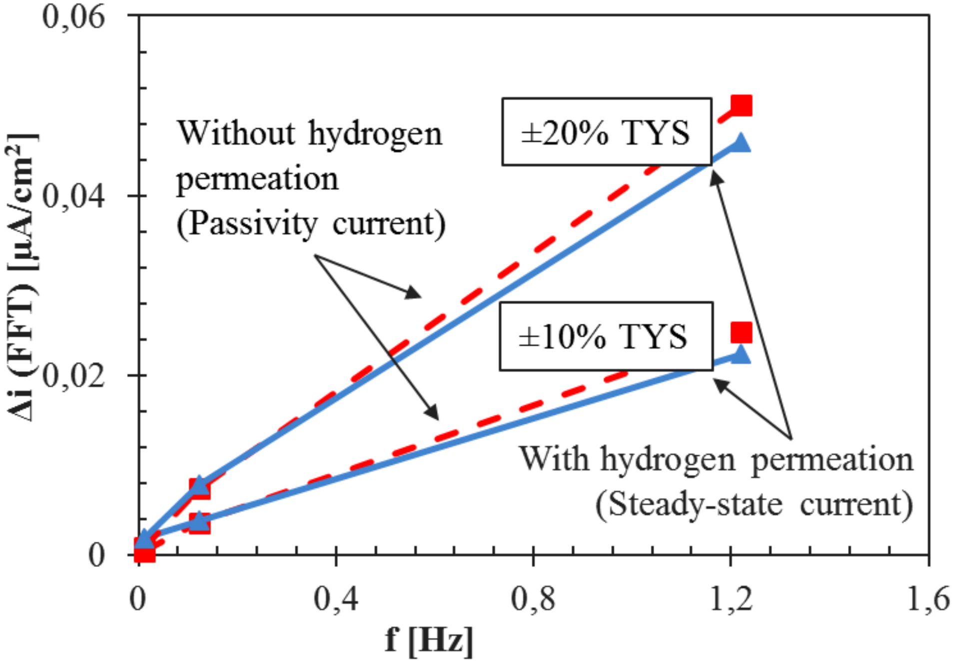


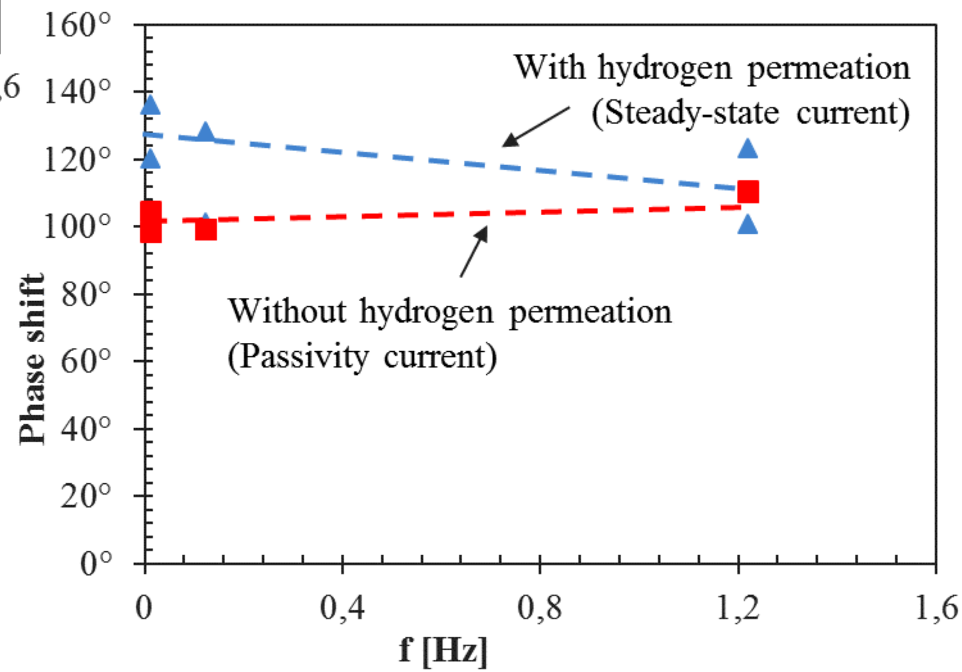
Steady-state current (i_∞)

Passivity current (i_P)

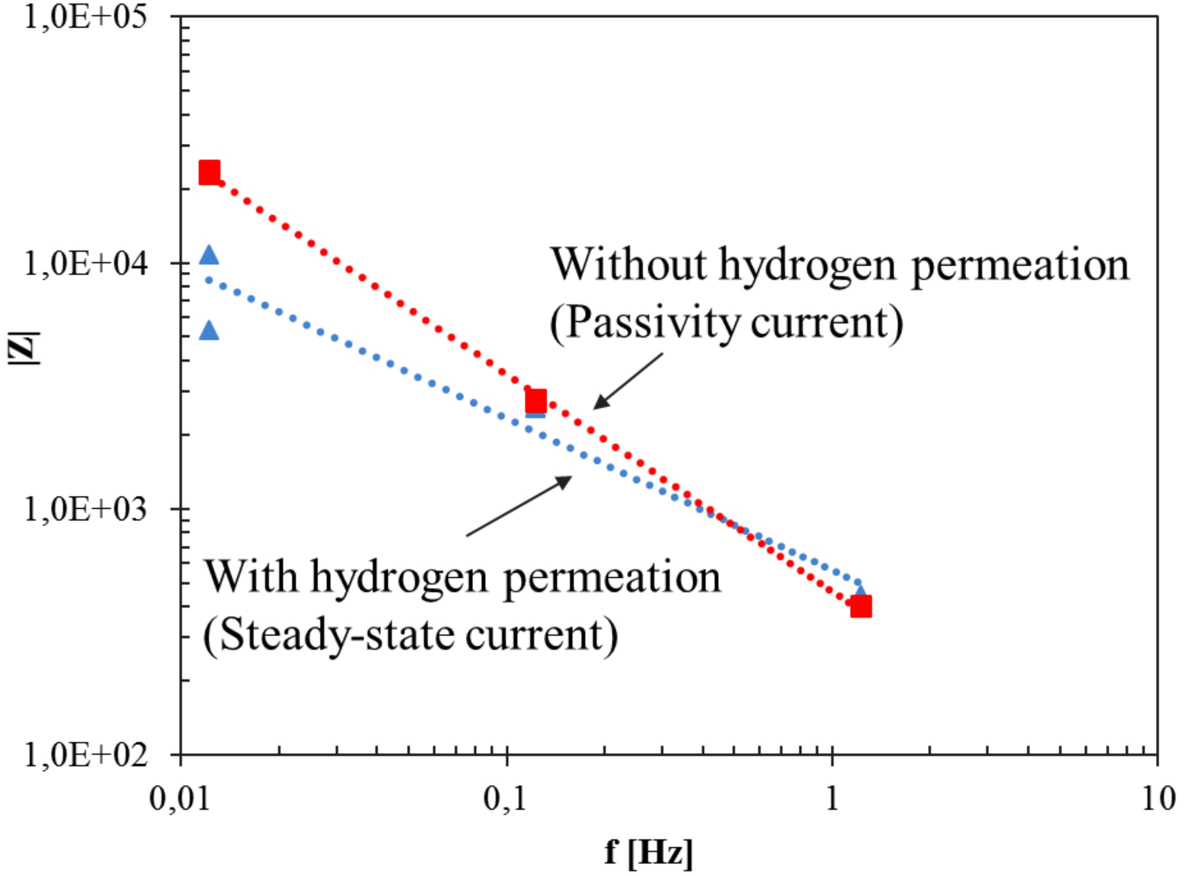


Effect of the alternate component of the load on steady-state current (i_∞) and passivity current (i_P)





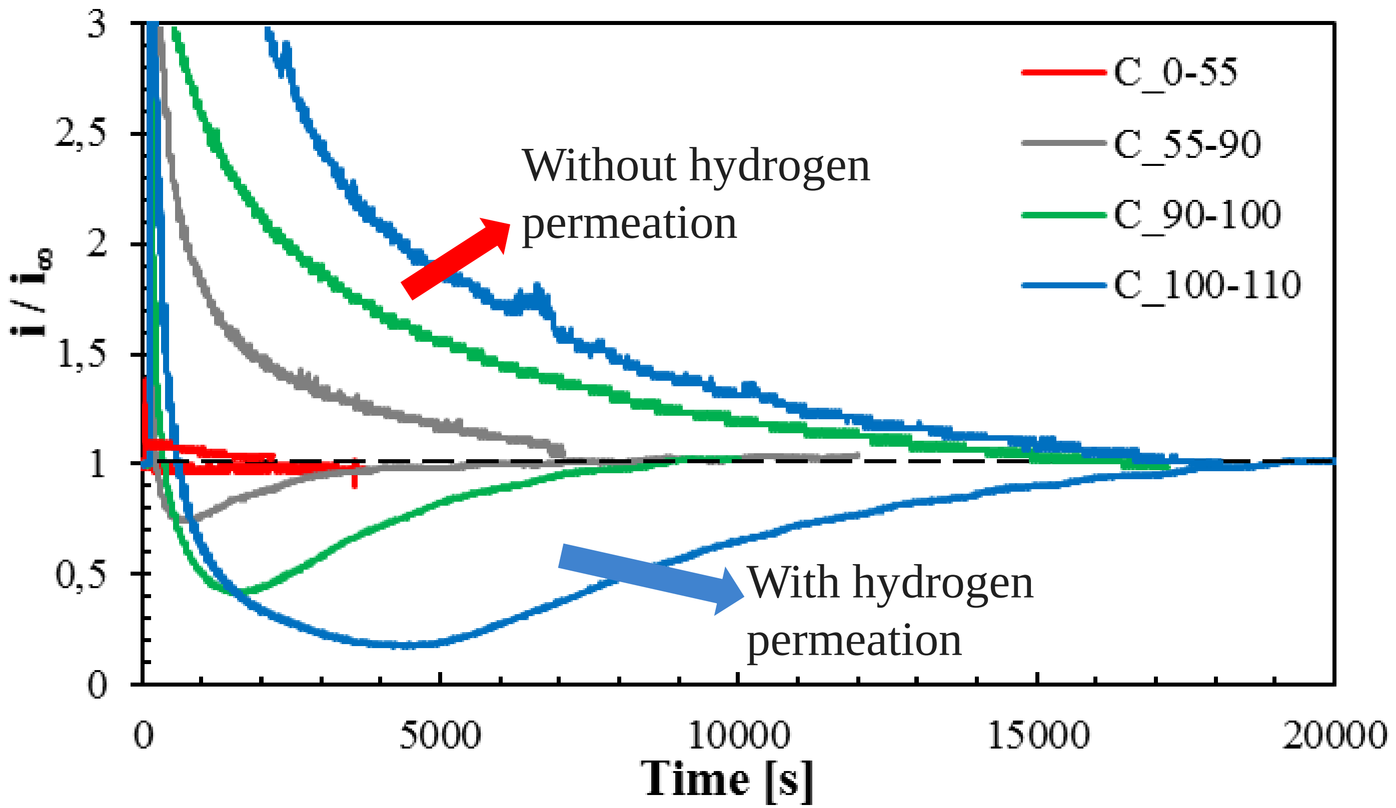
Effect of the alternate component of the load on steady-state current (i_∞) and passivity current (i_P)



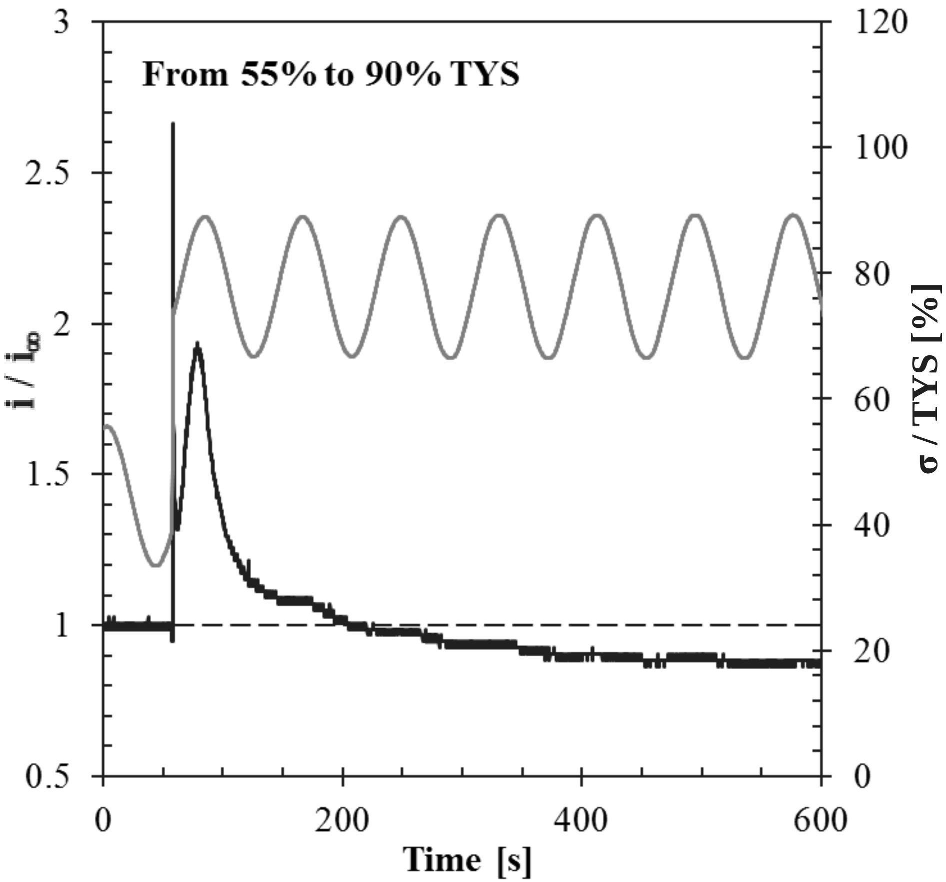
Effect of the variation of the maximum stress on the steady-state permeation current (i_∞)

Amplitude = ±10% TYS

Frequency = 10^{-2} Hz

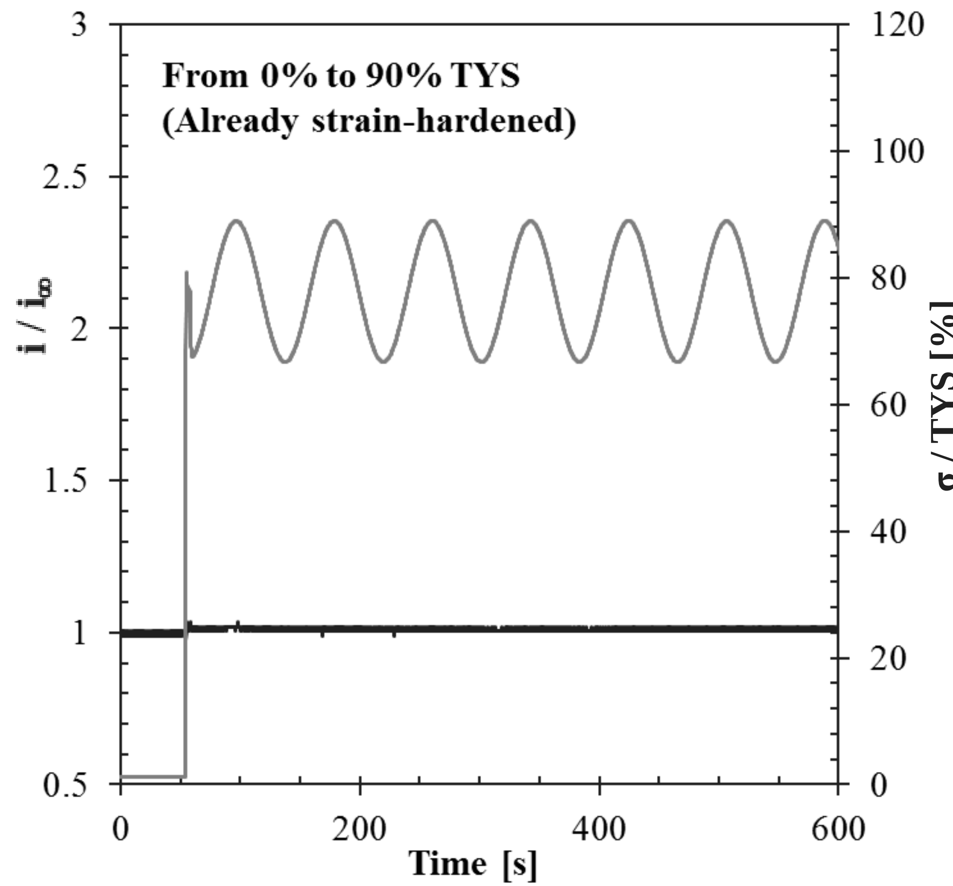


Response to subsequent variations of the maximum stress (locally strain-hardened steel)

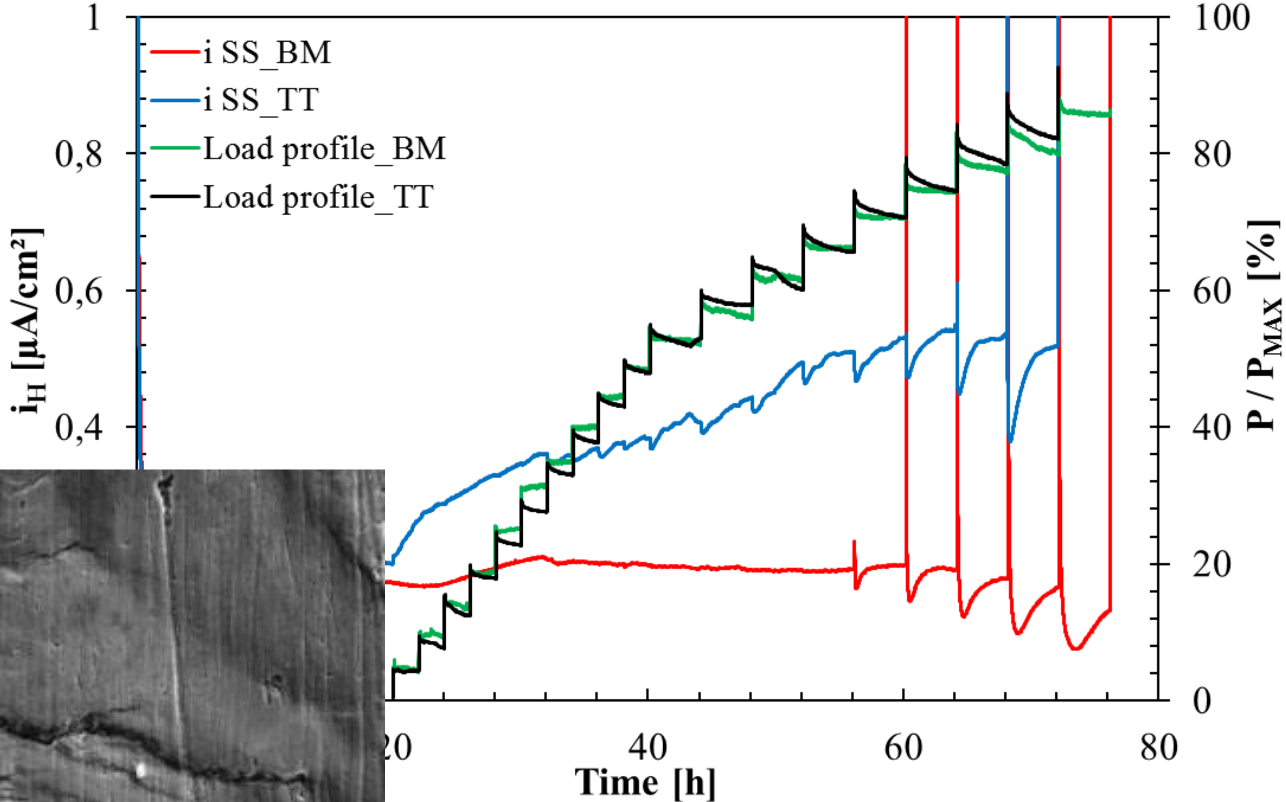


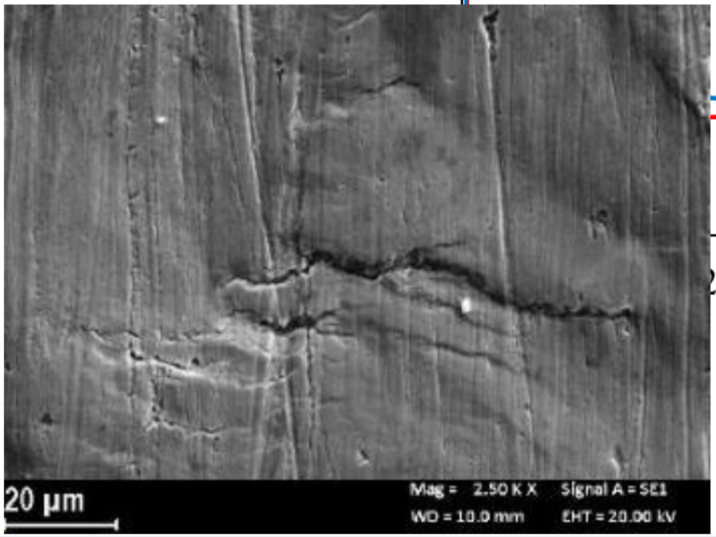
Amplitude = ±10% TYS

Frequency = 10^{-2} Hz



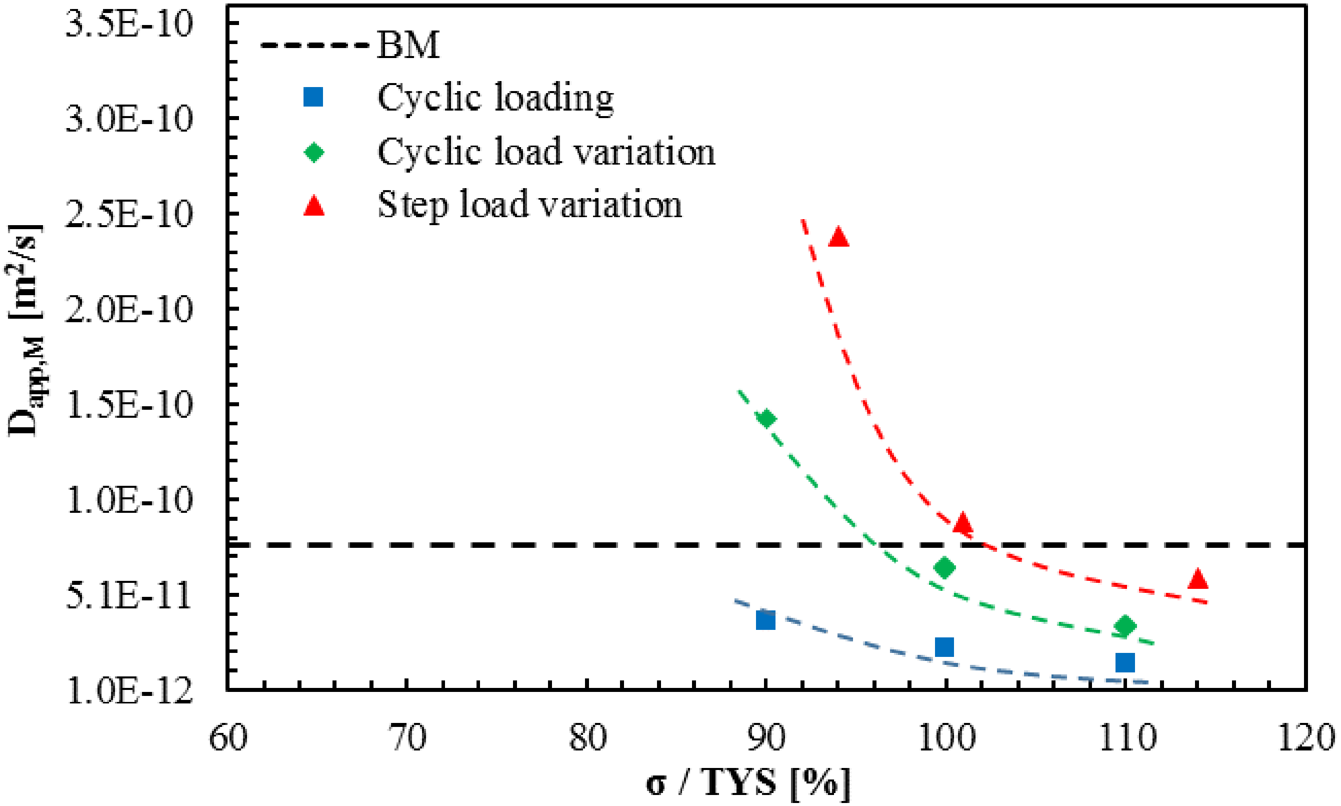
Comparison of the thermal treatment on the steady-state current (i_∞) during the incremental step loading



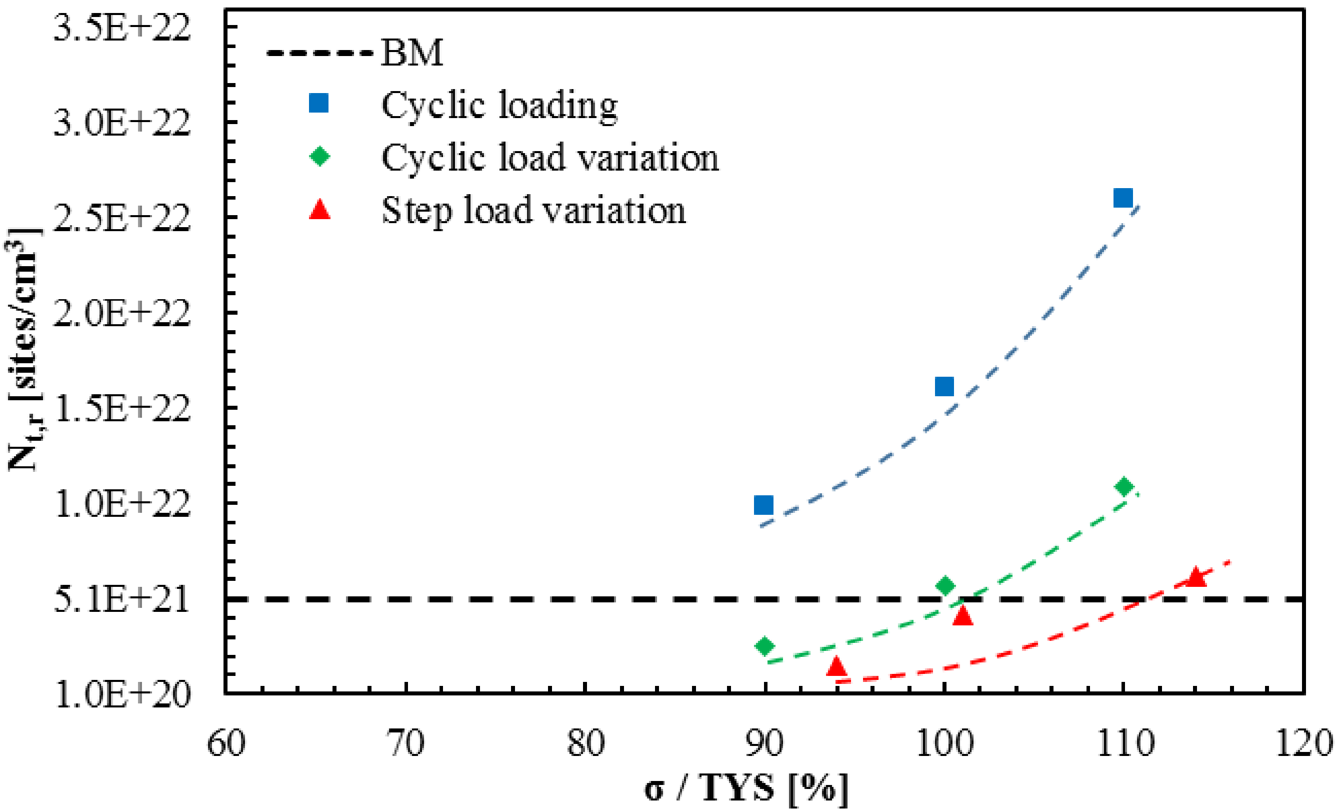


Cathodic surface of the as-received material (BM)

Comparison between first permeation transient and after load variation (cyclic and step load condition)



Comparison between first permeation transient and after load variation (cyclic and step load condition)



Conclusions (1/2)

- Apparent diffusivity showed a drastic reduction for loads beyond the yield limit
- Total hydrogen concentration exhibited a relevant increase for loads beyond the yield limit
- Reversible and irreversible trapping increase significantly under cyclic loading conditions compared to pre-straining (tensile) ones. Trapping seems to be absent, however, in pre-straining (compressive) conditions
- In the pre-straining (tensile/compressive) conditions, the influence of newly generated traps on hydrogen diffusivity and concentration due to the tensile stress seems to be strongly mitigated by the consequent compressive stress

Conclusions (2/2)

- Current response induced by the alternate component of the load, even at high frequencies, is substantially identical to that induced on the passivity current, with comparable phase shifts

- Load variation causes a temporary reduction in the permeation flow, which increases for load variations closer to the yield limit, and due to an instantaneous reduction of the mobile hydrogen concentration in the lattice

- Response to subsequent load variations on the same specimens, already locally strain-hardened, does not imply a modification in the permeation flow

- Higher diffusivity values estimated during a load variation (cyclic and step) are probably determined by the geometric distortion of the lattice

Thank you for your kind attention