

# **Autolab Application Note COR07**

# Corrosion Part 7 – Hydrogen permeation experiments with PGSTAT302F

## Keywords

Corrosion; Hydrogen permeation; PGSTAT302F

## Summary

Hydrogen permeation is a phenomenon that limits the practical use of high tensile strength steel alloys by introducing cracking hazards due to hydrogen penetration and hydrogen accumulation in the bulk of the alloy.

Hydrogen permeation is usually encountered during electrochemical processes that involve hydrogen evolution, such as electroplating, corrosion or cathodic protection. Hydrogen tends to accumulate in areas of high stress and can reach critical concentration causing cracks to occur which in turn compromises the mechanical integrity of the structure.

#### Hydrogen permeation measurements

Characterization of hydrogen permeation in stainless steel samples can be carried out by using two PGSTAT302F instruments operated in floating mode (see Figure 1).



Figure 1 – Autolab PGSTAT302F instrument

The electrochemical cell used for this type of experiments is the so-called Devanathan-Stachurski permeation cell, represented schematically in Figure 2.

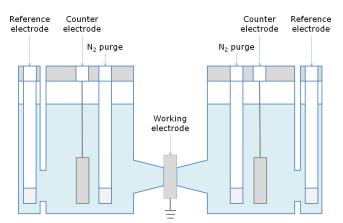


Figure 2 - Schematic overview of the Devanathan-Stachurski cell

The Devanathan-Stachurski cell is comprised of two individual electrolytic cells (see Figure 2), filled with electrolyte solution, separated with a membrane made from the material to be investigated. In one cell, hydrogen is produced electrochemically by cathodic polarization (cathodic compartment). The hydrogen is then adsorbed on the sample surface and atomically absorbed into the bulk of the material of the membrane. In the other cell (anodic compartment), the hydrogen diffusing though the sample is oxidized at constant potential and the anodic current is recorded. The oxidation current is directly proportional to the amount of hydrogen penetrating through the membrane with time.

Since two instruments need to be connected to the sample (the membrane), floating instruments need to be used. For this application, the Autolab PGSTAT302F can be used, since it offers the possibility to be operated both in normal and in floating mode.

#### Experimental

The data shown in this application note was obtained using the cell described in Figure 2. Two Autolab PGSTAT302F instruments, operating in floating mode, were connected to the anodic and the cathodic cells. The working electrode was physically connected to ground.



The working electrode was a sheet of 304 stainless steel. Before the measurements, both sides of the working electrode were mechanically polished. The exposed surface area was  $3.14 \text{ cm}^2$  on both sides.

The cathodic cell was filled with a 0.1 M NaOH solution spiked with 10 mg/L of  $As_2O_3$ , which acts as a hydrogen adsorption promoter. The anodic cell was filled with a 0.1 M NaOH solution. Both cells were deareated before the experiment by purging N<sub>2</sub> during 15 minutes.

The reference electrodes used are Ag/AgCl 3M KCl Metrohm (6.0726.100). The reference electrodes were positioned with respect to the surface of the membrane by Luggin-Haber capillaries. The counter electrode was a sheet of platinum on both sides.

All measurements were carried out at room temperature.

The cathodic hydrogen charging was carried out by applying a constant charging current of 1 mA/cm<sup>2</sup>. The anodic hydrogen oxidation was carried out at a constant potential of 0.3 V with respect to the reference electrode. The oxidation current was monitored during the experiment.

Figure 3 and Figure 4 show the recorded hydrogen oxidation transients for two separate carbon steel samples. Two transients are recorded for each sample.

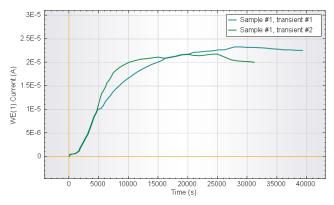


Figure 3 - Hydrogen oxidation transients recorded for sample #1

Autolab Application Note COR07 Corrosion Part 7 – Hydrogen permeation measurements with PGSTAT302F

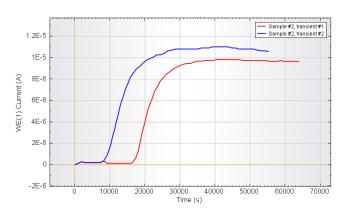


Figure 4 - Hydrogen oxidation transients recorded for sample #2

The first sample exhibits a significantly higher current density for the hydrogen oxidation, indicating a larger diffusivity of the hydrogen in the sample. The second sample exhibits a delay between the first and second transient, indicating a substantial delay in the hydrogen transport.

# Conclusions

This application note has illustrated the use to floating potentiostat/galvanostat instruments for the determination of hydrogen permeation. The PGSTAT302F is ideally suited for these measurements since it can be toggled at any time between normal operation mode and floating mode.

More information about Hydrogen permeation measurements can be found in ASTM G148-97.

#### References

• ASTM G148-97(2003) Standard Practice for Evaluation of Hydrogen Uptake, Permeation, and Transport in Metals by an Electrochemical Technique

# Date

1 March 2012