Application Note AN-EC-028

Measuring hydrogen permeation according to ASTM G148

How VIONIC powered by INTELLO can be used to study hydrogen permeation

In aqueous solution, hydrogen atoms are produced on the sample surface by electrochemical reactions. Hydrogen formation is encountered in different situations including electroplating, corrosion, and cathodic protection. The hydrogen atoms formed can permeate inside the sample material and accumulate in defective sites, causing cracks, inducing corrosion, and diminishing the mechanical properties of the sample.

It is therefore important to measure the hydrogen permeation in samples by studying hydrogen evolution and successive diffusion in samples.

In this Application Note, hydrogen permeation experiments are conducted following the procedure described in the ASTM standard G148.

EXPERIMENTAL SETUP

The experiment was carried out in a H-cell composed of two chambers separated by the working electrode shared between the compartments.

In one side (i.e., the «charging side»), hydrogen is formed at the working electrode surface. The hydrogen is detected in the other side (i.e., «oxidation side» or «detection side») [1].

The hydrogen permeation experiment was conducted with two VIONIC powered by INTELLO instruments from Metrohm Autolab (Figure 1).

Figure 1. VIONIC powered by INTELLO.

Since both instruments shared the same working electrode, they were set in floating mode – working electrode grounded. The working electrode was not physically connected to the ground.

One instrument was used to produce hydrogen on the sample by applying a negative current in galvanostatic mode. The other instrument was used in potentiostatic mode to oxidize the permeated hydrogen and to measure the resulting permeation current.

The working electrode was a 316L stainless steel foil of 50 µm thickness. The area exposed to each side of the H-cell was 1.8 cm^2. The charging side was filled with an acidic solution composed of 1 mol/L HCl in deionized water, where 0.25 g/L of disodium hydrogen arsenate heptahydrate (Na_2HAsO_4 $7H_2O$) was added to promote the hydrogen formation and permeation [2]. A Metrohm PT.SHEET was used as counter electrode. No reference electrode was used.

The oxidation side was filled with an aqueous solution of 0.1 mol/L NaOH (alkaline solution). A Metrohm Ag/AgCl 3 mol/L KCl reference electrode and a Metrohm PT.SHEET platinum electrode were used as reference and counter electrode, respectively. None of the solutions were deaerated.

The procedures consisted of the following steps:

1. The alkaline solution is added to the oxidation cell, together with the electrodes.
2. The procedure to detect hydrogen is started at the oxidation cell. In potentiostatic mode, +300 mV vs. OCP (open circuit potential) is applied. The recorded current decreases due to the relaxation of the polarization.
3. When the oxidation current is stable, the acidic solution is added to the charging side of the cell and the charging procedure is started. In galvanostatic mode, a negative (reductive) current of -1 mA/cm2 is applied to the working electrode. In this experiment, a current of -1.8 mA was applied.

RESULTS AND DISCUSSION

Figure 2 shows the plot of oxidation current vs. time which is related to the polarization in the oxidation side.

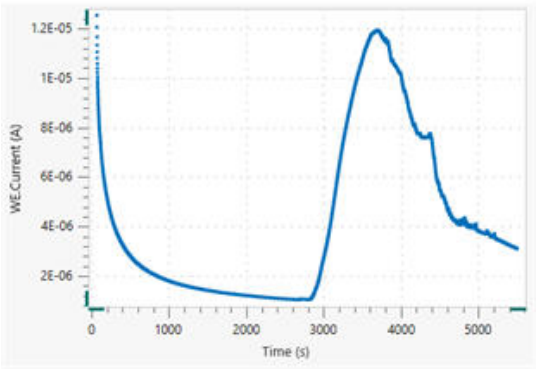

Figure 2. Plot showing oxidation current vs. time.

At the beginning of the recording, the current decreases due to the relaxation of polarization. The polarization is induced by applying 300 mV vs. OCP to the working electrode.

At approximately 2700 s, a small bump in the graph is visible. At that time, the charging side was filled up with the acidic solution and the production of hydrogen started.

The hydrogen permeation started to be detected at approximately 2800 s. Here, the current increased again due to the hydrogen becoming oxidized at the detection side.

CONCLUSIONS

In this Application Note, the study of hydrogen permeation according to ASTM G148 across a 316L stainless steel sample was presented.

This work was possible with the use of two VIONIC powered by INTELLO instruments, both in floating mode – working electrode grounded.

An H-cell was used for this study where the working electrode was shared between two compartments. One side was used to generate hydrogen with an acidic solution, and the other side was used to detect hydrogen with an alkaline solution.

The hydrogen permeation was triggered using disodium hydrogen arsenate heptahydrate, added in the acidic solution.

REFERENCES

1. ASTM G148 - Standard Practice for Evaluation of Hydrogen Uptake, Permeation, and Transport in Metals by an Electrochemical Technique.
2. Charca, S. M. Study of Hydrogen Permeation and Diffusion in Steels: Predictive Model for Determination of Desorbed Hydrogen Concentration - ProQuest, University of Puerto Rico, Mayaguez, 2006.

FOR MORE INFORMATION

More information about this Application Note and the associated INTELLO software procedure is available from your local Metrohm distributor. Additional instrument specification information can be found at: VIONIC powered by INTELLO

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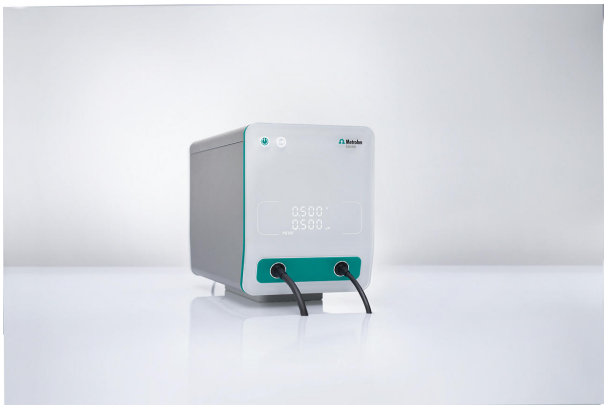
CONFIGURATION



VIONIC

VIONIC is our new-generation potentiostat/galvanostat that is powered by Autolab's new INTELLO software.

VIONIC offers the **most versatile combined specifications of any single instrument** currently on the market.

- Compliance voltage: ± 50 V
- Standard current ± 6 A
- EIS frequency: up to 10 MHz
- Sampling interval: down to 1 µs

Also included in VIONIC's price are features that would usually carry an additional cost with most other instruments such as:

- Electrochemical Impedance Spectroscopy (EIS)
- Selectable Floating
- Second Sense (S2)
- Analog Scan

Platinum sheet electrode

This electrode consists of a thin sheet of platinum embedded into a glass shaft. This electrode can be used as a counter electrode for most electrochemical measurements. The surface area is approximatively 1 cm2.

Ag/AgCl reference electrode

Silver / silver chloride reference electrode with c(KCl) = 3 mol/L as reference electrolyte.

This reference electrode is suitable for aqueous applications and the SGJ 14/15 enables easy assembly.