



Aalto University
School of Engineering

Laboratory Case Study: Hydrogen charging testing

Nguyen Xuan Binh (887799)

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1. Introduction

The objective of this laboratory exercise is to investigate the susceptibility of metallic materials to hydrogen embrittlement via a hydrogen charging test. This test helps us understand how materials behave under the influence of hydrogen, particularly in environments where hydrogen exposure is common, such as in fuel cells and pipeline transportation. By analyzing how hydrogen interacts with the material structure, we can predict potential failures and improve structural and material design for better durability and safety.

Hydrogen embrittlement can lead to catastrophic failures in metals; thus, testing for this phenomenon allows researchers and engineers to identify and mitigate risks associated with hydrogen-induced material degradation. By charging the specimen with hydrogen and subsequently performing a tensile test, the impact of hydrogen on the mechanical properties of the material can be quantified, highlighting potential weaknesses in material design.

2. Introduction

Overview of the approach

The approach includes preparing a hydrogen-saturated solution, exposing the specimen to this solution to absorb hydrogen, and then measuring the mechanical properties of the hydrogen-charged material through a tensile test. This procedure helps determine the decrease in ductility and tensile strength caused by hydrogen.

First, we would charge solution for one time charging: 1,25 L of 1N H_2SO_4

- 1216 ml H_2O
- 34 ml H_2SO_4
- 25 mg Thiourea (20 mg/l)

We need to use the proper equipment to measure the amount of solution correctly, such as 10:500 and 1:100 beaker.



Preparing the specimen

1. Polishing: First, the specimen is polished in the longitudinal direction with 800 grit sandpaper, then polished in the transverse direction with 1200 grit sandpaper.

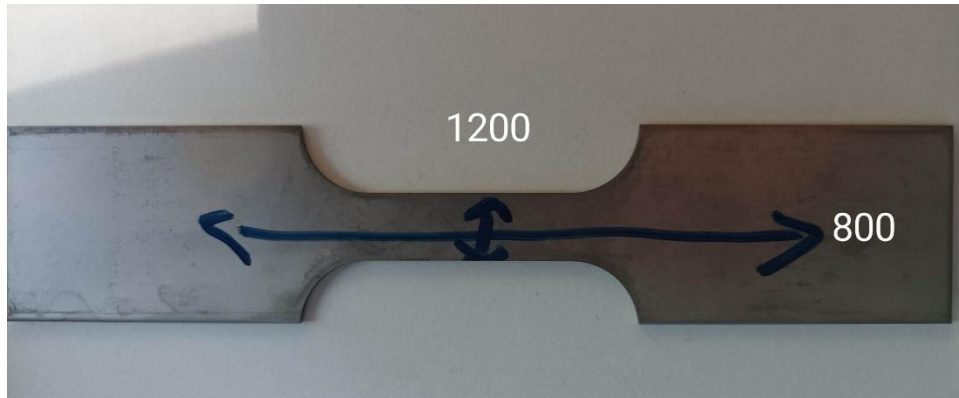


Figure 1 Polish the specimen in grit 800 direction first and then grit 1200 direction

2. Cleaning: After polishing, the specimen is thoroughly cleaned with water followed by ethanol to remove residual contaminants.
3. Protection: The specimen is then taped to protect specific areas from exposure to the charging solution, except for the notch which is left exposed.



Figure 2 Prepared specimen

Characterization of the specimen

Qualitative: visual inspection for surface uniformity

Quantitative: Dimensions and mass are recorded pre- and post-test to track any physical changes. Material composition and crystalline structure may also be analyzed using X-ray diffraction or similar techniques before the test.

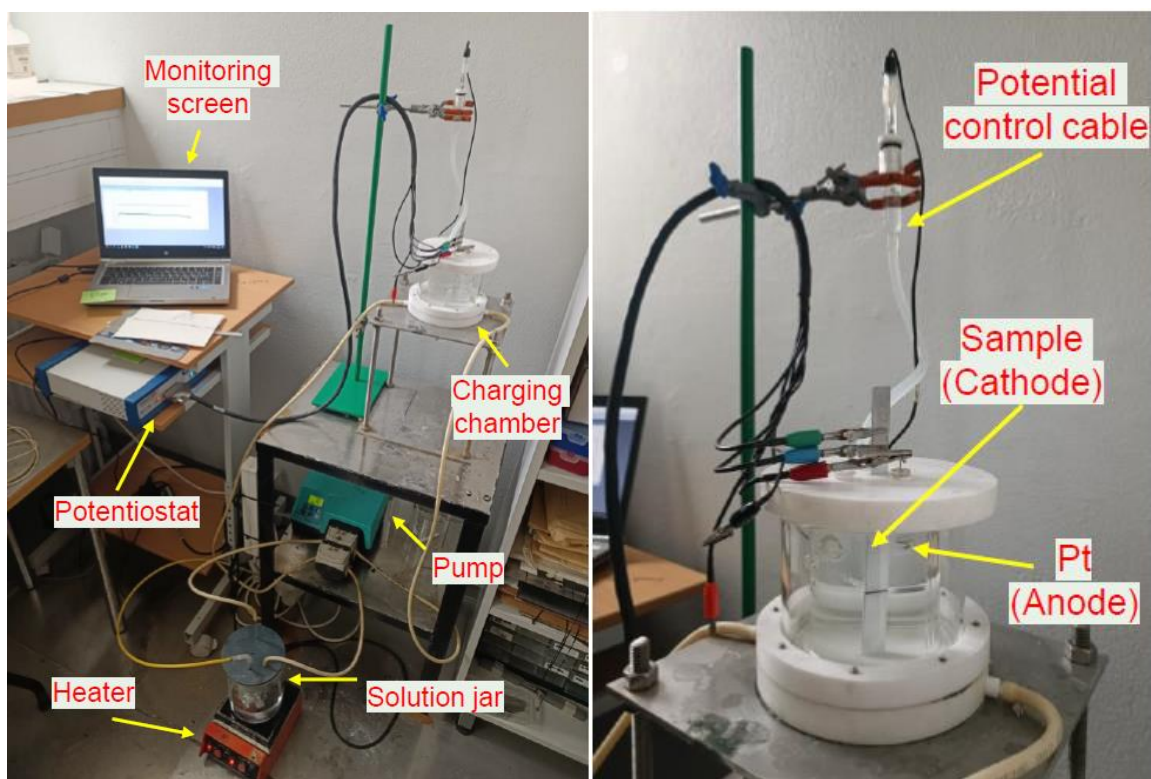
Physical quantities to be measured

Tensile Strength: Measured using a tensile test machine that evaluates the force required to break the specimen. We have already discussed this in the third report, Standard Tensile Testing using extensometer

Ductility: It is measured by recording the elongation of the specimen at break.

Conducting the hydrogen charge

The set up in the lab room



We can now put a beaker on the magnetic stirrer and heating plate. Next, we pour the solution into the beaker.



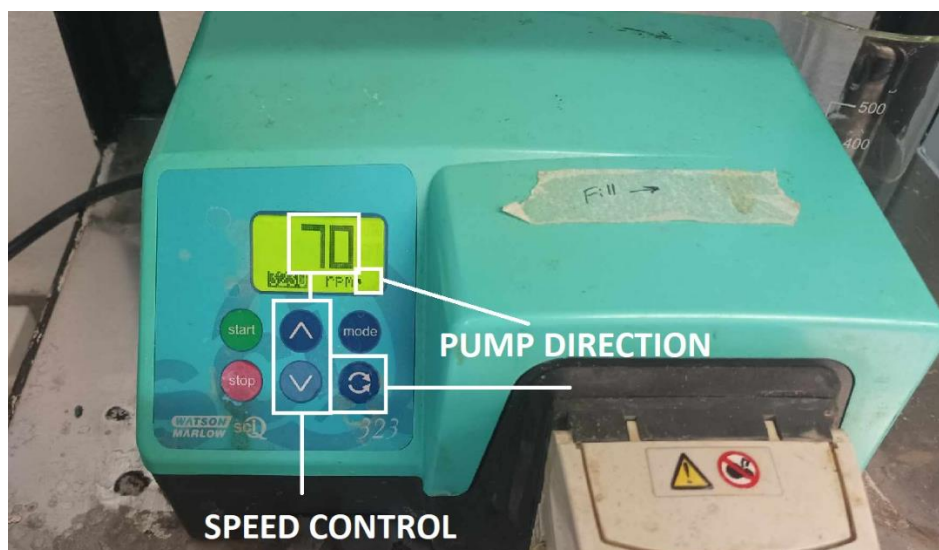
We set the heating plate power such that the temperature of the solution inside the beaker is consistently 50 Celsius degrees.



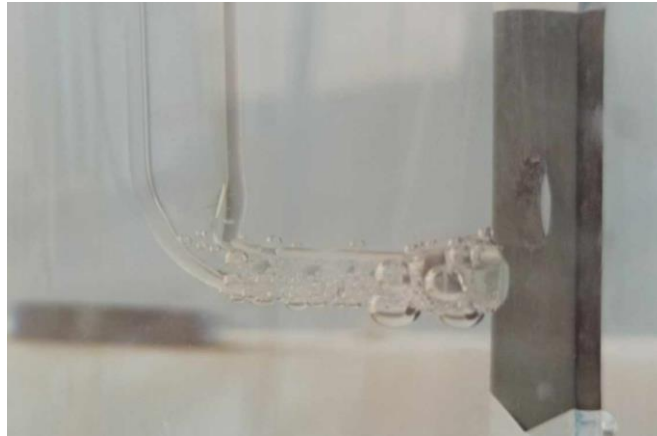
Now, we set the specimen inside the charging cell, use the gasket so that there is no leakage. Then, the pump should be connected with the beaker and the charging cell.



Next, we turn on the pump and set the liquid's direction so that it is transferred into the charging cell. Put the other beaker under the charging cell in case of leakage/ overflow.



Next, we check the tube on the right to see if the pump is working correctly. The tube should have the solution poured in. Additionally, we should examine the tube inside the cell and rotate so that the hydrogen gas is sucked through the tube. Clear the tube if there is any bubble stuck inside the tube, as the process will mainly corrode the specimen if it is clogged. Refill the upper tube with K_2SO_4 if needed.



Plug in the wire: blue and green to the cathode (the material) and red to anode (platinum wire)



We start the program with: Experiment -> Potentiostatic -> Choose name+ potential (use a difference of 0.0001 in order to use 1 value of potential. For example, -0.966 and -0.9661 to set -0.966)

The electrolysis program should show the blue dot. If the red dot appears, there might be some error in the process. Contact the technicians.

Collecting the specimen

1. Stop the electrolysis process by double click skip in the monitor program.
2. Reverse the pump, pulling all the solution out of the charging cell. Press stop.
3. Take out the specimen, discard the tape and clean the specimen with **cold water** and ethanol.
4. Conduct the tensile test as soon as possible in order to reduce the diffusion of hydrogen.
5. We discard the solution in a plastic container in the lab, mark the solution on the container and when it is full, the container will be emptied by the technician.

Safety precautions

Chemical Handling: Proper gloves and goggles should be worn when handling acids and solvents.

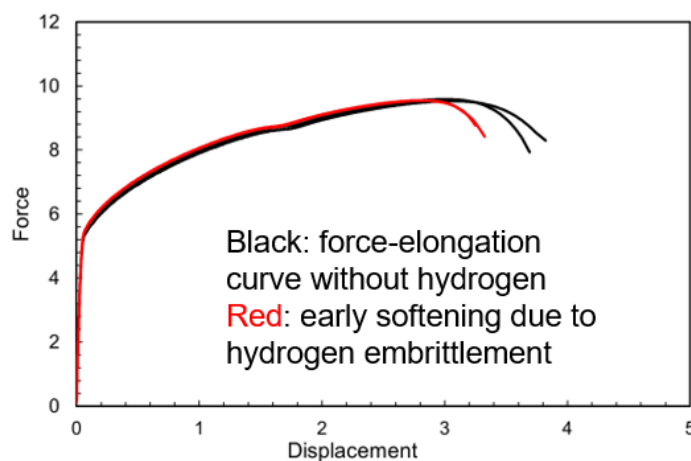
Equipment Operation: Only trained personnel should operate tensile machines and heating equipment.

Analysis of test results

Results will be analyzed by comparing the tensile strength and ductility before and after hydrogen exposure. Changes in these properties indicate the level of susceptibility to hydrogen embrittlement.

Evaluation of Accuracy

Calibration of instruments and standardized test procedures ensure accuracy. Deviations in expected results might necessitate re-evaluation of the experimental setup or specimen preparation.

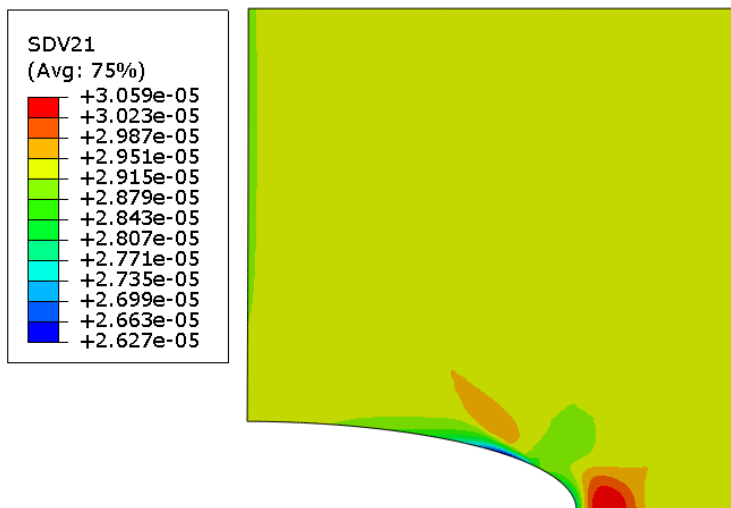


As we can see, hydrogen greatly changes the material properties of metals, causing it to fracture prematurely compared to conditions where it is not subject to hydrogen

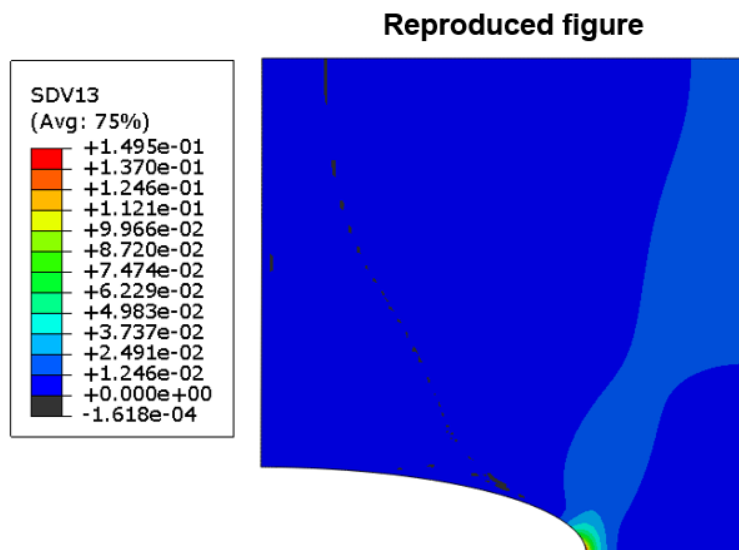
Simulation of hydrogen diffusion coupled with mechanical loading

The result of hydrogen charged specimen can be used to verify the fracture models coupled with the HELP (hydrogen-enhanced localized plasticity) and HEDE (Hydrogen-enhanced decohesion) mechanism. For example, below is a simulation of 2D plate where it is subject to both hydrogen diffusion and yield strength softening, both of which influences each other

The two main features of the HELP mechanism are (a) material softening and (b) localization of plastic flow in those regions where the hydrogen content is high. This should lead to the embrittlement of the material, especially metals.



Distribution of hydrogen at lattice sites



Distribution of equivalent plastic strain

Additionally, there is another species of hydrogen, which traps hydrogen at defects or dislocation sites. Its concentration depends on lots of factors, but its influencing origins are

1. Equivalent plastic strain, which tells how hydrogen are trapped at dislocation densities
2. Lattice hydrogen concentration C_L , because according to Oriani's equilibrium theory, lattice hydrogen should be in equilibrium with the trapped hydrogen
3. Hydrostatic stress gradient, since its gradient tells how lattice hydrogen diffuses

On the other hand, trapped hydrogen concentration has no effects on either lattice hydrogen or hydrostatic stress.

This simulation above is one of the preliminary results on how we can use hydrogen charged experiment to validate our material model, where both mechanical loading and hydrogen diffusion process influence each other.