Final protocol for material testing

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Revision History

Revision	Revision date	Summary of changes
0	30 March 2021	Initial version

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Name	Institute	Date
Gregor Mori WP4 Leader	Montanuniversitaet Leoben	23 March 2021
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1. Introduction

The ultimate goal of WP4: Materials and Corrosion of project "Hystories" is to determine what material (steel grade) can be used for underground hydrogen storage under which conditions.

Moreover, the most critical environment should be selected in a conservative approach. Conditions are:

- Temperature variation in underground depleted fields or aquifers could vary between 40 and 100°C. At the wellhead the temperature can reach room temperature.
- Pressure in storage facilities can oscillate between 50 and 200 bar.
- Humidity is about 100 %.
- Salinity of the brine can oscillate between 1 (condensate water) and 200 g/l NaCl (formation water in some very saline aquifers).

Beside H_2, the gas can contain CH_4, H_2S and CO_2. Since methane is not considered to be corrosive, it is not included into the testing program.

2. Test matrix

The following types of tests will be done:

- Autoclave testing,
- Permeation testing and
- Thermal Desorption Spectrometry.

Below the different experiments are described in more detail.

<u>Autoclave testing:</u>
For autoclave testing laboratory autoclaves made of Alloy 625 will be used consisting of a tube with two threaded lids. One lid contains a valve (Figure 1). Two specimens are placed in each autoclave: a tensile specimen that is stressed at 90 % of yield strength via a spring and a 6x6x30 mm coupon to determine hydrogen uptake after the experiment. To avoid movement of specimens in the autoclave during testing, the specimens are fixed inside the autoclave with PTFE distance holders (Figure 2).

Figure 3 shows the tensile specimens, the ceramic nuts and the spring in detail.

Figure 1: Autoclave

Figure 2: Specimen arrangement inside autoclave

Figure 3: Geometry of the tensile specimen, spring and nuts

Tests will be carried out for 720 hours. When the autoclaves have to be rotated (1 rpm) during testing they will be mounted on two horizontally rotating shafts in a heating cabinet (Figure 4).

After testing, the tensile specimens will be investigated with regard to cracking and to corrosive attack. Corrosion rate, if any, will be determined gravimetrically. The corrosion speed is determined by diameter loss of the tensile specimens. The cuboid specimen will be immediately cooled in liquid nitrogen, cleaned from corrosion products and its hydrogen uptake will be determined by hot extraction in the equipment shown in Figure 5.

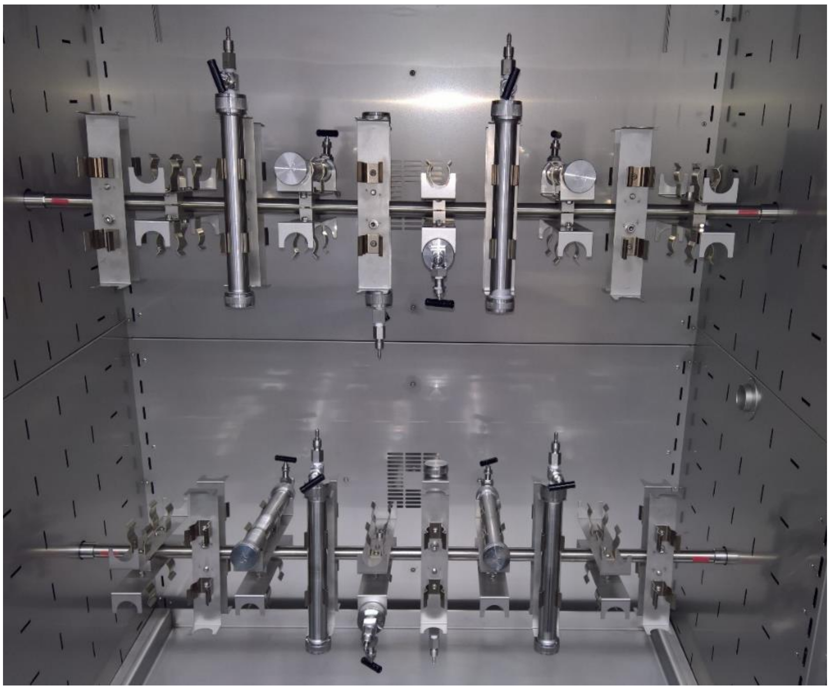

Figure 4: Heating cabinet with two horizontal rotary shafts and autoclave holders

Figure 5: Hydrogen analysis by hot extraction method

Together with the consortium leader and members of the Advisory Board the following gas compositions have been selected (Table 1).

Table 1: Partial pressures of the test gases A to D [bar]

Gas component	A	B	C	D
H_2	120	120	120	120
CO_2	0	15	0	15
H_2S	0	0	1	1

Tests with gas A will be performed at a total pressure of 120 bar, for gas B of 135 bar, for gas C of 121 bar and tests with gas D will be performed at a total pressure of 136 bar. By this way the hydrogen partial pressure remains constant through the test matrix and CO_2 and H_2S partial pressures are also equal. This will be done to maintain the same partial pressure of gases within the test matrix.

The autoclaves will be filled with or without electrolyte. In case electrolyte is added a quantity of 60 ml is used to maintain an electrolyte level of 50 % of the height of the autoclave. Autoclaves and electrolytes are carefully degassed with Argon before being filled.

Table 2: Conditions of electrolyte

Quantity of electrolyte in autoclave [ml]	Salt content of water [g/l]	Rotation of autoclave [rpm]
0	0	0
60	1*	0
60	200*	0
60	1	1
60	200	1

* … 2 specimens: 1 specimen immersed, 1 specimen in gas phase

Tests will be done at room temperature and at 120 °C. In total for a full investigation of one material, 4 gases x 7 conditions x 2 temperatures = 56 tests have to be performed. This full program will be done for material K55, L80 and the welded K55.

In addition, all other materials and surface conditions that have been chosen will be investigated under the most severe conditions. It is expected that most severe conditions will be gas D with 60 ml of electrolyte at 200 g/l NaCl with rotation at room temperature. In case different results are obtained for K55 and L80, these other conditions will be applied. In case cracking occurs more tests will be performed.

Permeation testing:

By performing permeation tests, the effective hydrogen diffusion coefficient is determined which as information about the trapping behaviour of the material. The permeation test will be conducted according to standard ISO 17081 [1]. It will be carried out in a Devanathan-

Stachursky-Cell [2] (principle in Figure 6 and real test setup in Figure 7). By multiple charging and decharging of the material it can be determined whether deep or flat hydrogen traps are present in the material and which type prevails.

At least the three basic materials K55, L80 and K55 in welded condition will be characterised. Other materials will be characterised when unexpected results are obtained.

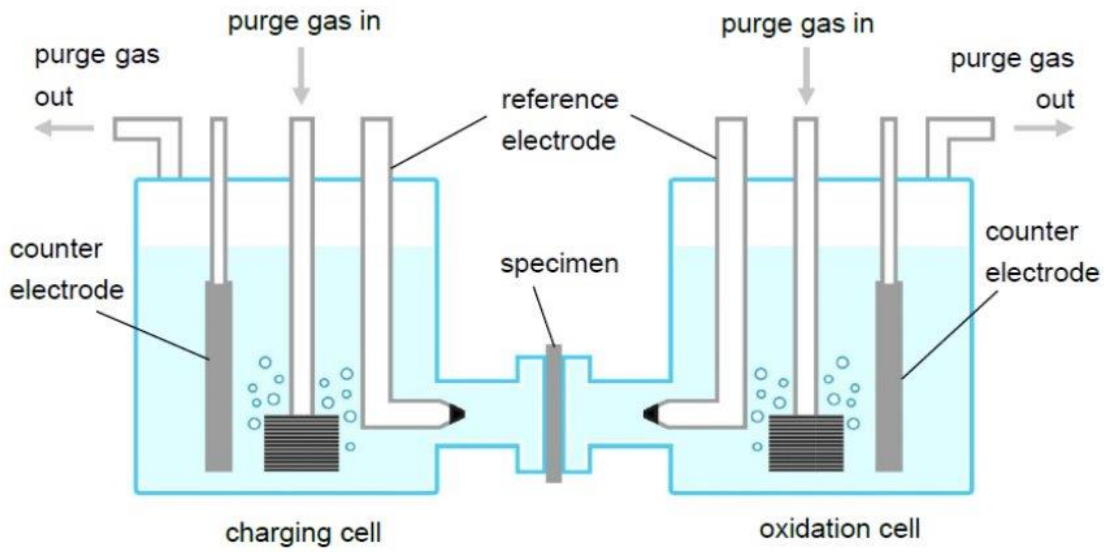

Figure 6: Principle of the permeation test [3]

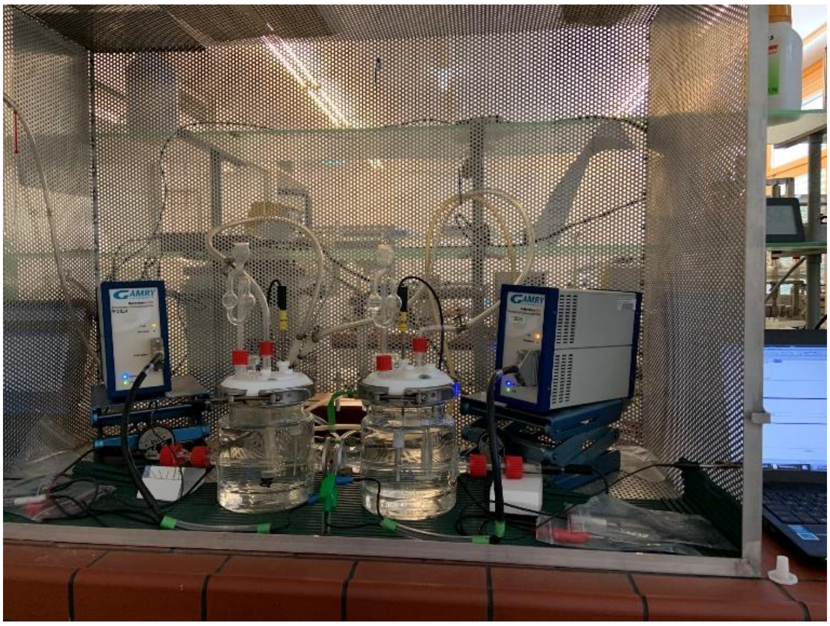

Figure 7: Permeation test setup

Thermal Desorption Spectrometry:

In addition, the three materials K55, L80 and K55 welded condition, will be loaded cathodically with atomic hydrogen and subsequently analysed in a Thermal Desorption Spectroscopy (TDS). The system is shown in Figure 8. By increasing temperature linearly and simultaneously analysing the amount of hydrogen leaving the material, one can determine the amount of hydrogen that is trapped in each single lattice defect of the steel such as:

- Lattice hydrogen,
- dislocation hydrogen,
- grain boundary hydrogen and
- hydrogen trapped at precipitates.

Figure 8: Thermal Desorption Spectrometry Bruker Galileo G8

<u>Other investigations</u>
All materials will be characterised with respect to chemical composition, mechanical properties and microstructure.

3. References

[1] International Organization for Standardization, ISO 17081:2014, Method of measurement of hydrogen permeation and determination of hydrogen uptake and transport in metals by an electrochemical technique, Geneva, Switzerland, 2014.

[2] M. A. V. Devanathan, Z. Stachursky, The Adsorption and Diffusion of Electrolytic Hydrogen in Palladium. D62T, Proceedings of the Royal Society A: Mathematical, Physical and Engineering Science 270, 1962, p.90-102.

[3] W. Siegl, G. Mori, W. Ecker, J. Klarner, G. Kloesch, A. Drexler, G. Winter, H. Schnideritsch, Hydrogen Trapping in Heat Treated and Deformation Armco Iron, Nashville, Tennessee, USA, NACE International, 2019, paper 13083.

Hystories project consortium

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