

# Summary report on all investigated steels

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## **1.Introduction**

In the course of work package WP4 of the Hystories project, the testing was carried out of selected materials in D4.2 (list of steel grades to be investigated) were carried out in autoclave tests by the Chair of General and Analytical Chemistry at Montanuniversitaet Leoben.

The three carbon steel grades K55, L80 and the welded J55 were tested in the full test program. Other selected materials were tested under certain conditions.

The full test program includes:

- Autoclave tests including
  - time to failure,
  - hydrogen content,
  - SEM investigation of surface layer and
  - permeation tests.

Experiments are described in detail in report "D4.1\_Final protocol for material testing". In this report only results are described.

Table 1 shows the chosen conditions for selected materials (were marked with a red X) and the main materials K55, L80 and welded J55 (were marked with an orange X). This table is filled in completely with all conditions when the full test programme is carried out at room temperature and at 120  $^{\circ}$ C.



Table 1: Test matrix of steel grades at 25 °C and 120 °C in, a) gas A (120 bar  $H_2$ ), b) gas B (120 bar  $H_2$  + 15 bar  $CO_2$ ), c) gas C (120 bar  $H_2$  + 1 bar  $H_2$ S) and d) gas D (120 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2$ S), marked with an orange x for the materials L80, K55 and the welded J55 and those marked with a red X were for the selected materials.

condition		Immersion in								
		dry gas	humid gas (1 g/l)	electrolyte (1 g/l)	gas + electrolyte (1 g/l)	humid gas (200 g/l)	electrolyte (200 g/l)	gas + electrolyte (200 g/l)		
	gas A	ХХ	X	X	X	X	X	ХХ		
room temperature	gas B	x	Х	X	Х	х	X	хх		
	gas C	X	Х	X	X	Х	X	X X		
	gas D	X	X	X	X	Х	X	XX		
120 °C	gas A	X	X	X	X	Х	X	X		
	gas B	X	Х	X	X	Х	X	X		
	gas C	X	X	X	X	Х	X	X		
	gas D	X	X	X	X	X	X	X		



## 2.Investigation of a quenched steel

#### 2.1. Time to failure of Constant Load Tests (CLT)

Steel L80 has been austenitized and quenched in water. Due to its quenched properties, this material is very sensitive to hydrogen embrittlement and can easily crack under stress if hydrogen is present. This steel grade is not commercialized however, the presence of cracks in this quenched material allows to check the reliability of the testing method.

For Constant Load Tests, tensile specimens are stressed at 90 % of their yield strength. The test is static and lasts for 720 h.

Three tests were performed according to the graph (Figure 1). Two of them resulted in a failure. The time to failure cannot be given since the autoclave are only opened after 720 h. A fracture occurred under dry hydrogen (120 bar  $H_2$ ) conditions at room temperature and under the more severe conditions in gas D (120 bar  $H_2$  + 15 bar CO<sub>2</sub> + 1 bar  $H_2$ S) with an electolyte. This is shown in Figure 1. No failure occurred unter dry hydrogen (120 bar  $H_2$ ) conditions at 120 °C.



Figure 1: Time to failure of quenched material after testing in gas A (120 bar  $H_2$ ), no electrolyte, at room temperature and 120 °C & in gas D (120 bar  $H_2$  + 15 bar CO<sub>2</sub> + 1 bar  $H_2$ S), 200 g/l NaCl, immersion in gas + electrolyte, 120 °C.

#### 2.2. Hydrogen content

Figure 2 illustrates the hydrogen uptake at 25 °C and 120 °C in dry gas A and in gas D which contains hydrogen sulfide H<sub>2</sub>S, with the addition of an electrolyte with 200 g/l NaCl and rotation of the autoclave. The hydrogen uptake at room temperatures and dry hydrogen gas A (120 bar H<sub>2</sub>) is 0.1 ppm and at 120 °C is 0.36 ppm. The hydrogen uptake in dry H<sub>2</sub> gas A can be compared with the blank value (0.2 ppm), this is slightly higher, but this can be considered as a scattering of the values The hydrogen uptake in gas D (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub> + 1 bar H<sub>2</sub>S), 200 g/l NaCl, immersion in gas + electrolyte, 120 °C is 0.86 ppm.





Figure 2: Hydrogen uptake of quenched material after testing in gas A (120 bar  $H_2$ ), no electrolyte, at room temperature and 120 °C & in gas D (120 bar  $H_2$  + 15 bar CO<sub>2</sub> + 1 bar  $H_2$ S), 200 g/l NaCl, immersion in gas + electrolyte, 120 °C.

#### 2.3. SEM investigation of fracture surface

Figure 3 shows a fracture image of a quenched steel. The test condition was the more aggressive one in gas D ( $H_2 + CO_2 + H_2S$ ) with an electrolyte of 200 g/l sodium chloride NaCl at 120°C. Under these severe conditions a fracture occurred. It did not make sense to make a SEM examination with this fracture surface, because the surface is covered by a thick layer of corrosion products. The hydrogen content in the specimen was 0.86 ppm after the experiment.



Figure 3: Fracture surface of quenched steel tested in gas D (120 bar  $H_2$  + 15 bar CO2 + 1 bar  $H_2$ S), 200 g/l NaCl, immersion in gas + electrolyte, 120 °C.



Figure 4 shows a fracture image of the quenched steel tested under milder conditions. The test condition was dry gas A ( $H_2$ ) at room temperature. Under these conditions also a fracture occurred. At the 12 o´clock position the fracture origin is observed. A hydrogen amount of only 0.10 ppm was measured using TDS.



Figure 4: Fracture surface of quenched steel tested in gas A (120 bar H<sub>2</sub>), no electrolyte, 25 °C, 30x.

Figure 5 shows the different fracture zones in a SEM. In Figure 5 a), in the light blue frame from Figure 4, a clear hydrogen fracture is present. There are ductile and brittle zones present. Figure 4 b) is a higher magnification. There is a cleavage like fracture with river patterns and some fine pores typical for a hydrogen induced fracture. In Figure 5 c) the green area shows a brittle fracture as well. In the dark blue area from Figure 4 and magnified in Figure 5 d), the transition area between brittle hydrogen crack and a ductile forced fracture is visible.





Figure 5: Details of fracture surface of quenched steel a) hydrogen fracture 300x, b) river patterns at hydrogen fracture 3000x, c) hydrogen fracture 300x d) transition zone between hydrogen fracture (left) and ductile forced fracture (right), 300x.

Because fractures occurred with this brittle quenched material that is very sensitive to hydrogen embrittlement, it confirms that the testing method is reliable to evaluate the steel grades compatibility for  $H_2$  application.



## 3.Investigation of steel K55

### 3.1. Time to failure of Constant Load Tests (CLT)

Figure 6 shows the time to failure of ferritic perlitic steel K55 for all gas compositions. Gases without  $H_2S$  (non-sour gas) are gas A (120 bar  $H_2$ ) and gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ). Gases with  $H_2S$  (sour gas) are gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ). No fracture at all occurred under any condition. Figure 6 below shows as an example of time to failure in gas D. No cracks at all occurred in specimens of steel K55. The same happened for gases A, B and C and at room temperature and at 120 °C.



Figure 6: Time to failure of ferritic perlitic steel K55 in gas D: 120 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2S$ , 25 °C.

#### 3.2. Hydrogen content

Figure 7 shows the hydrogen uptake of steel K55 in four test gases A to D at room temperature (25 °C) in comparison to a higher temperature (120 °C).

In this autoclave tests with steel K55 at room temperature, the lowest hydrogen value is 0.07 ppm for gas A and the highest value is 2.33 ppm for immersion in gas C. Gases A and B show no increased hydrogen uptake. Gases C and D with H<sub>2</sub>S show increased values whenever an electrolyte is present. When no electrolyte is present, H<sub>2</sub>S has almost no effect on hydrogen uptake. In gases C and D higher hydrogen contents were generated with the presence of an electrolyte. However, no failure occurred. In the autoclave tests with the steel K55 at 120 °C, the lowest value is 0.19 ppm in gas B and the highest value is 0.49 ppm in gas D, in comparison, the blank value is 0.22 ppm. The hydrogen content was measured by thermal desorption spectroscopy (TDS).





Figure 7: Hydrogen content after immersion of steel K55 in various gases, electrolytes and salinities at different temperatures for 30 days; a) and b) are without  $H_2S$ ; c) and d) are with  $H_2S$ ; a) gas A (120 bar  $H_2$ ), b) gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), c) gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and d) gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ).

On the contrary, the addition of  $H_2S$  to the gas resulted in fast sulfide layer formation at 120°C and consequently in a reduction of the absorbed hydrogen content compared to the tests conducted at room temperature.

In all conditions no specimen failed.

Table 2 below compares the hydrogen uptake for different conditions at room temperature and at 120  $^\circ\text{C}.$ 



condition		Hydrogen content [ppm] after immersion in								
		dry gas	humid gas (1 g/l)	electrolyte (1 g/l)	gas + electrolyte (1 g/l)	humid gas (200 g/l)	electrolyte (200 g/l)	gas + electrolyte (200 g/l)		
	gas A	0.07	0.13	0.12	0.18	0.26	0.21	0.21		
room temperature	gas B	0.11	0.14	0.15	0.21	0.20	0.23	0.21		
	gas C	0.17	0.78	1.00	2.33	0.36	0.38	0.24		
	gas D	0.17	1.88	1.41	1.30	1.24	0.77	0.63		
120 °C	gas A	0.37	0.30	0.26	0.30	0.26	0.35	0.28		
	gas B	0.20	0.20	0.19	0.33	0.26	0.24	0.22		
	gas C	0.28	0.23	0.32	0.27	0.24	0.21	0.30		
	gas D	0.26	0.49	0.44	0.37	0.45	0.34	0.41		

Table 2: Hydrogen uptake of steel K55 in autoclave tests at 25 °C and 120 °C in, a) gas A (120 bar  $H_2$ ), b) gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), c) gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and d) gas D (120 bar  $H_2 + 15$  bar CO2 + 1 bar  $H_2S$ ).

g/l = g/l NaCl

Slightly more hydrogen was absorbed at higher temperatures in H<sub>2</sub>S free conditions.

In the following the effect of different hydrogen uptake at different temperatures was investigated by characterizing the surface layers formed at specimens with a scanning electron microscope (SEM).



#### 3.3. SEM investigation of surface layers

Figure 8 a) and b) show a cross-section of the ferritic and pearlitic steel K55 in the SEM at a magnification of 300x in gases without  $H_2S$  (non-sour gas). Figure 8 c) and d) show the cross section of the steel K55 but at gases with  $H_2S$  (sour gas). Figure 9 shows the same conditions as Figure 8 but at higher temperature.



Figure 8: SEM analysis of the surface of steel K55 at room temperature after 720 hours of autoclave testing with 200 g/l NaCl in a) and b) are without  $H_2S$ ; c) and d) are with  $H_2S$ ; a) gas A (120 bar  $H_2$ ), b) gas B (120 bar  $H_2$  + 15 bar CO2), c) gas C (120 bar  $H_2$  + 1 bar  $H_2S$ ) and d) gas D (120 bar  $H_2$  + 15 bar CO2 + 1 bar  $H_2S$ ).





Figure 9: SEM analysis of the surface of steel K55 at 120 °C after 720 hours of autoclave testing with 200 g/l NaCl in a) and b) are without  $H_2S$ ; c) and d) are with  $H_2S$ ; a) gas A (120 bar  $H_2$ ), b) gas B (120 bar  $H_2 + 15$  bar CO2), c) gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and d) gas D (120 bar  $H_2 + 15$  bar CO2 + 1 bar  $H_2S$ ).

On Figure 8 d) a certain localized attack can be seen starting from the surface of the sample. This attack has a depth of 15  $\mu m.$ 

## 3.4. Corrosion rate

Figure 10 a) and b) show the corrosion rate of the ferritic - pearlitic steel K55 in gases without  $H_2S$  (non-sour gas). Figure 10 c) and d) show the corrosion rate of this steel in Gases with  $H_2S$  (sour gas).





Figure 10: Corrosion rate of steel K55 in autoclave tests at different temperatures a) and b) are without  $H_2S$ ; c) and d) are with  $H_2S$  in; a) gas A (120 bar  $H_2$ ), b) gas B (120 bar  $H_2$  + 15 bar CO2), c) gas C (120 bar  $H_2$  + 1 bar  $H_2S$ ) and d) gas D (120 bar  $H_2$  + 15 bar CO2 + 1 bar  $H_2S$ ).

Corrosion rate are mainly below 0.1 mm/y for most conditions. Some experiments yielded to a higher corrosion rate up to 0.4 mm/y. However there are no preferential conditions that resulted in a higher corrosion rate, such as higher temperature, higher salinity, presence of  $H_2S$  or immersion of the specimens in the electrolyte. The higher corrosion rates are randomly distributed among the results and therefore it is believed that there is some scatter.

#### 3.5. Permeation tests

By performing the permeation tests, the effective hydrogen diffusion coefficient has been determined, which gives information about the trapping behaviour of the material. The permeation test was conducted according to standard ISO 17081 in a Devanathan-Stachursky-Cell. 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 predominates.

The oxidation current was measured over the time to determine the effective diffusion coefficient. Results from hydrogen permeation tests on steel K55 can be seen in Figure 11. In



Figure 11 a) the permeation current as function of time for 2 consecutive loadings can be seen, in Figure 11 b) a detail of the permeation current as function of time for first loading with evaluation is shown, in Figure 11 c) a detail of the permeation current as function of time for the second loading with evaluation is given.



Figure 11: Hydrogen permeation current as function of time for 2 consecutive loadings for steel K55, Oxidation cell with 0.1M NaOH and the charging cell with 3.5%NaCl + 1 g/l Thiourea a) overview of two charging cycles, b) detail of 1<sup>st</sup> charging, c) detail of 2<sup>nd</sup> charging.

The first loading resulted in an effective diffusion coefficient  $D_{eff}$  of  $6.22 \cdot 10^{-6} \frac{cm^2}{s}$ , seen in Table 1 and the second loading gave an  $D_{eff}$  value of  $7.96 \cdot 10^{-6} \frac{cm^2}{s}$ , see Table 3. There is a small amount of deep traps since the second loading is slightly faster than the first one. The literature confirms, that the value is within the range for carbon steels. The results show diffusion coefficients for non-sour gas resistant steels as expected from the literature review as well.



#### Table 3: Effective diffusion coefficient of hydrogen in steel K55 at room temperature

# loading	$D_{\mathrm{eff}}\left[\frac{cm^2}{s}\right]$			
1	6.22 · 10 <sup>-6</sup>			
2	7.96 · 10 <sup>-6</sup>			



## 4. Investigation of steel L80

#### 4.1. Time to failure of Constant Load Tests (CLT)

Figure 12 shows the time to failure of tempered martensite steel L80 for all gas compositions. No fracture occurred under any condition. Figure 12 shows as an example time to failure in gas D at room temperature. No cracks at all occurred for the steel L80. The same happened for gases A, B and C.



Figure 12: Time to failure of a tempered martensitic steel L80 in gas D: 120 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2S$ , 25 °C.

#### 4.2. Hydrogen content

Figure 13 illustrates the hydrogen uptake at room temperatures and at 120 °C in a) gas A and b) gas B, both gases do not contain hydrogen sulfide  $H_2S$ , c) shows the uptake in gas C and d) shows the hydrogen content after exposure to gas D. In the autoclave tests with the steel L80 at room temperature, the lowest value is 0.09 ppm for gas A and the highest value is 1.03 ppm for gas C, the amount of hydrogen was measured using also TDS. In comparison, the blank value is 0.22 ppm, which is sometimes slightly higher than in the dry tests. There is a certain hydrogen uptake in gases C and D at 25 °C, when an electrolyte is present. This is due to the lowering of the solution pH by H<sub>2</sub>S. In the autoclave tests with the steel L80 at 120 °C, the lowest value is 0.15 ppm for gas A and the highest value is 0.32 ppm for gas D. There is almost no hydrogen uptake in the autoclave tests. Even H<sub>2</sub>S-containing conditions result in low H concentrations in the steel compared to tests at room temperature.





Figure 13: Hydrogen content after immersion of steel L80 in various gases, electrolytes and salinities at different temperatures for 30 days; a) and b) are without  $H_2S$ ; c) and d) are with  $H_2S$ ; a) gas A (120 bar  $H_2$ ), b) gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), c) gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and d) gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ).

This is the same effect as with the steel K55, on the contrary, the addition of  $H_2S$  to gas at 120°C led to a rapid formation of sulphide layers and consequently to a reduction in the absorbed hydrogen content compared to the tests carried out at room temperature.

In all conditions, no specimen failed.

Table 4 below compares the hydrogen uptake for all different conditions at room and at high temperatures.



condition		Hydrogen content [ppm] after immersion in								
		dry gas	humid gas (1 g/l)	electrolyte (1 g/l)	gas + electrolyte (1 g/l)	humid gas (200 g/l)	electrolyte (200 g/l)	gas + electrolyte (200 g/l)		
	gas A	0.09	0.20	0.19	0.14	0.19	0.10	0.23		
room	gas B	0.11	0.20	0.18	0.17	0.19	0.12	0.16		
temperature	gas C	0.14	0.51	0.47	0.38	0.77	0.83	1.03		
	gas D	0.17	0.82	0.96	0.34	0.82	0.46	0.67		
	gas A	0.36	0.20	0.20	0.16	0.19	0.15	0.20		
120 °C	gas B	0.16	0.23	0.21	0.16	0.18	0.25	0.25		
	gas C	0.18	0.16	0.16	0.26	0.28	0.18	0.17		
	gas D	0.17	0.17	0.30	0.19	0.17	0.32	0.21		

Table 4: Hydrogen uptake of steel L80 in autoclave tests at 25 °C and 120 °C in, a) gas A (100 bar  $H_2$ ), b) gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), c) gas C (100 bar  $H_2 + 1$  bar  $H_2S$ ) and d) gas D (100 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ).

g/l = g/l NaCl

The tempered martensitic material L80 has similar hydrogen uptake than the ferritic perlitic steel K55 with lower strength. Nevertheless, the 22 HRC criterion for resistance to  $H_2S$  is met and this is confirmed by the fact that no cracking at all did occur.



#### 4.3.SEM investigation

Figure 14 a) and b) show a cross-section of the tempered martensitic steel L80 in the SEM at a magnification of 300x in gases without  $H_2S$  (non-sour gas). Figure 14 c) and d) also show the cross section of the steel L80 but at gases with  $H_2S$  (sour gas). Figure 15 shows the cross section after testing at higher temperature.



Figure 14: SEM analysis of the surface of steel L80 at room temperature after 720 hours of autoclave testing with 200 g/l NaCl in a) and b) are without  $H_2S$ ; c) and d) are with  $H_2S$ ; a) gas A (120 bar  $H_2$ ), b) gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), c) gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and d) gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$   $H_2S$ ).





Figure 15: SEM analysis of the surface of steel L80 at 120 °C after 720 hours of autoclave testing with 200 g/l NaCl in a) and b) are without  $H_2S$ ; c) and d) are with  $H_2S$ ; a) gas A (120 bar  $H_2$ ), b) gas B (120 bar  $H_2 + 15$  bar CO<sub>2</sub>), c) gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and d) gas D (120 bar  $H_2 + 15$  bar CO<sub>2</sub> + 1  $H_2S$ ).

There are no obvious defects in any of the cross-sections, except one. The cross-section of steel L80 in Figure 15 d) shows localized attack on the surface, at higher pressures and when hydrogen sulphide is present. The approximate maximum depth of attack is 68  $\mu$ m, although it cannot be excluded that deeper attack may be found at another point on the sample. The material seems to be durable under tested conditions and is suitable for use in pure hydrogen applications. In presence of high content of H<sub>2</sub>S (partial pressure of 1 bar), localized defects might appear. Nevertheless, according to the standard ISO 15156-2, L80 is applicable for H<sub>2</sub>S service provided that it is type 1.

#### 4.4. Corrosion rate

Figure 16 a) and b) show the corrosion rate of the tempersteel L80 in gases without  $H_2S$  (non-sour gas). Figure 16 c) and d) show the corrosion rate of the steel L80 but at Gases with  $H_2S$  (sour gas).





Figure 16: Corrosion rate of steel L80 in autoclave tests at different temperatures in a) and b) are without  $H_2S$ ; c) and d) are with  $H_2S$  in; a) gas A (120 bar  $H_2$ ), b) gas B (120 bar  $H_2$  + 15 bar  $CO_2$ ), c) gas C (120 bar  $H_2$  + 1 bar  $H_2S$ ) and d) gas D (120 bar  $H_2$  + 15 bar  $CO_2$  + 1  $H_2S$ ).

The highest corrosion rate of steel L80 at 120 °C was measured in gas A, B and C with a rate of 0.12 mm/a. The highest rate at room temperature was obtained also in gas B with a rate of 0.54 mm/a. Under all different environments and conditions that occurred in the autoclaves, the gases  $CO_2$  and  $H_2S$  probably had the biggest impact on the observed corrosion rates. Especially at room temperature, including the presence of an electrolyte, with a content of 1 g/l and 200 g/l sodium chloride, increased corrosion rates were observed in the gas environments containing  $H_2S$ . But also the presence of gaseous  $CO_2$  in combination with an electrolyte, with both chloride contents, increased corrosion rates at room temperature. The corrosion rate for dry gases without electrolyte was 0 mm/a.

#### 4.5. Permeation tests

The oxidation current was measured as function of time to determine the effective diffusion coefficient. Results from hydrogen permeation tests on the tempered martensite steel L80 can be seen in Figure 17. In Figure 17 a) the permeation current as function of time for two consecutive loadings can be seen, in Figure 17 b) a detail of the permeation current as function



of time for the first loading including the evaluation is shown, in Figure 17 c) a detail of the permeation current as function of time for the second loading with the evaluation is presented.



Figure 17: Hydrogen permeation current as function of time for 2 consecutive loadings for steel L80, Oxidation cell with 0.1M NaOH and the charging cell with 3.5%NaCl + 1 g/l Thiourea a) overview of two charging cycles, b) detail of 1st charging, c) detail of  $2^{nd}$  charging.

The first loading gave an effective diffusion coefficient  $D_{eff}$  of  $2.68 \cdot 10^{-6} \frac{cm^2}{s}$  and the second loading  $D_{eff}$  resulted in a value of  $3.98 \cdot 10^{-6} \frac{cm^2}{s}$  (Table 5). These values are comparable to those found in the literature. The steel L80 has more traps, as this steel is also more deformed than the ferritic-pearlitic steel. Consequently the diffusion coefficients are slightly lower than on K55.



Table 5: Effective	diffusion coe	fficient of hydr	ogen in steel L80	) at room temperature.
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# loading	$D_{eff}\left[\frac{cm^2}{s}\right]$
1	2.68 · 10 <sup>-6</sup>
2	3.98· 10 <sup>-6</sup>



## 5.Investigation of a welded steel J55

It is admitted that hydrogen embrittlement can be enhanced in the presence of stress concentrations, such as in welding areas<sup>1</sup>. Therefore, it was decided in the program to test a steel grade on a welding area.

A weld with a V-joint shape was performed on a J55 steel grade. Preheating temperature was set to 120 °C and the interpass temperature was between 120 and 170 °C. There was no Post Welding Heat Treatment (PWHT). Therefore, it can be considered that local stresses were not reduced, the hardness mapping according to Vickers [HV1] can be seen in Figure 18. The mapping shows hardness values from 162 up to 458 HV1, the highest vaule is located in the top layer. It is noticed that measured hardness exceeds the recommended value of 250 HV for H<sub>2</sub>S applications based on NACE Standard MR0175. Nevertheless, due to sample manufacturing the first layer which had the highest hardness was removed and was not tested.



Figure 18: Hardness mapping according to Vickers HV1 for the welded steel J55.

Test pieces for all the following tests were manufactured on the weld area.

#### 5.1. Time to failure of Constant Load Tests (CLT)

Figure 19 shows the time to failure of a ferritic pearlitic welded steel J55 for all gas compositions. No fracture occurred under any condition. Figure 19 below shows as an example time to failure in gas D at room temperature. No cracks at all occurred for the welded steel J55. The same happened for gases A, B and C at any condition that was tested.

<sup>&</sup>lt;sup>1</sup> ASME-STP-PT-006, « Design Guidelines for Hydrogen Piping and Pipelines", 2007





Figure 19: Time to failure of a ferritic pearlitic welded steel J55 in gas D: 120 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2S$ , 25 °C.

#### 5.2. Hydrogen content

Figure 20 illustrates the hydrogen uptake at room temprature in a) gas A and b) gas B, both gases do not contain hydrogen sulfide H<sub>2</sub>S. Figure 20 c) shows the uptake in gas C and Figure 20 d) shows the hydrogen content after exposure to gas D. The lowest hydrogen content to be found utilizing autoclave tests with the steel J55 at room temperature is 0.12 ppm for gas A and the highest value for gas D is 2.95 ppm, the absorbed hydrogen amount was measured by using TDS. In the autoclave tests with the steel J55 at 120 °C, the lowest value is 0.35 ppm for gas A and the highest value is 2.08 ppm for gas C. For the autoclaves containing gases A and B, almost no hydrogen uptake was observed at low temperatures, compared to the blank value of 0.65 ppm. However, higher hydrogen uptake is indicated at higher temperatures under same gaseous conditions. Measurements with gas C show a significant hydrogen uptake for both, ambient temperature and elevated temperature conditions. Due to the decreasing of the pH value by adding H<sub>2</sub>S to the solution, also gas D presents higher absorbed hydrogen content at ambient temperature. Figure 20 shows the results after testing steel J55 for all conditions.





Figure 20: Hydrogen content after immersion of a welded steel J55 in various gases, electrolytes and salinities at different temperatures for 30 days; a) and b) are without  $H_2S$ ; c) and d) are with  $H_2S$ ; a) gas A (120 bar  $H_2$ ), b) gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), c) gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and d) gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ).



The Table 6 below compares the hydrogen uptake for different conditions at room and at high temperatures.

condition		Hydrogen content [ppm] after immersion in						
		dry gas	humid gas (1 g/l)	electrolyte (1 g/l)	gas + electrolyte (1 g/l)	humid gas (200 g/l)	electrolyte (200 g/l)	gas + electrolyte (200 g/l)
room temperature	gas A	0.23	0.19	0.16	0.18	0.12	0.15	0.19
	gas B	0.18	0.19	0.35	0.71	0.26	0.22	0.25
	gas C	0.19	0.81	0.79	0.52	0.98	0.74	0.93
	gas D	0.80	2.29	1.62	1.45	0.65	2.95	1.38
120 °C	gas A	0.35	0.61	0.69	0.51	0.83	0.87	0.83
	gas B	0.57	0.64	1.13	1.07	1.49	1.86	1.01
	gas C	0.98	0.93	0.62	1.01	1.26	1.08	2.08
	gas D	0.23	0.75	0.29	0.30	0.36	0.81	0.27

Table 6: Hydrogen uptake of the welded steel J55 in autoclave tests at 25 °C and 120 °C in, a) gas A (100 bar  $H_2$ ), b) gas B (120 bar  $H_2$  + 15 bar  $CO_2$ ), c) gas C (100 bar  $H_2$  + 1 bar  $H_2S$ ) and d) gas D (100 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2S$ ).

#### g/l = g/l NaCl

It is globally observed that the ferritic pearlitic welded material J55 has higher hydrogen uptake than the ferritic pearlitic without welding. It could indicate that welded material is more susceptible to hydrogen embrittlement than same metal with lower stress.



#### 5.3. SEM investigation

Figure 21 a) and b) show a cross-section of the ferritic-pearlitic steel J55 in the SEM at a magnification of 300x in gases without  $H_2S$ . Figure 21 c) and d) show the cross section of the welded J55 with gases with additional  $H_2S$ . Figure 22 shows the same conditions as Figure 21 but at higher temperature.



Figure 21: SEM analysis of the surface of a welded steel J55 at room temperature after 720 hours of autoclave testing with 200 g/l NaCl in a) and b) are without  $H_2S$ ; c) and d) are with  $H_2S$ ; a) gas A (120 bar  $H_2$ ), b) gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), c) gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and d) gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$   $H_2S$ ).





Figure 22: SEM analysis of the surface of a welded steel J55 at 120 °C after 720 hours of autoclave testing with 200 g/l NaCl in a) and b) are without  $H_2S$ ; c) and d) are with  $H_2S$ ; a) gas A (120 bar  $H_2$ ), b) gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), c) gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and d) gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$   $H_2S$ ).

The cross-section of steel J55 in Figure 21 and Figure 22 shows that there is no localized damage attack at the surface. The material is resistant under testing conditions and seems to be suitable for the use of hydrogen applications.



#### 5.4. Corrosion rate

Figure 23 a) and b) show the corrosion rate of the welded ferritic pearlitic steel J55 in gases without  $H_2S$ . Figure 23 c) and d) show the corrosion rate of the welded J55 in gases with  $H_2S$ .



Figure 23: Corrosion rate of steel J55 welded in autoclave tests at different temperatures a) and b) are without  $H_2S$ ; c) and d) are with  $H_2S$  in; a) gas A (120 bar  $H_2$ ), b) gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), c) gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and d) gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 15$  bar  $CO_2 + 15$  bar  $H_2S$ ).

The highest corrosion rate at steel J55 at 120 °C was measured in gas D with a rate of 0.36 mm/a. The highest corrosion rate was measured at room temperature also in gas A with a rate of 0.6 mm/a. Under all different environments and conditions in the autoclaves, the presence of  $CO_2$  and  $H_2S$  probably had the highest impact on the observed corrosion rates, except for the one high value for gas A, which could very likely also be considered as an outlier. Especially at room temperature, including the presence of an electrolyte, with a content of 1 g/l and 200 g/l sodium chloride, increased corrosion rates were observed in the gas environments containing  $H_2S$ . But also the presence of gaseous  $CO_2$  in combination with an electrolyte, with both chloride contents, increased corrosion rates at room temperature. The corrosion rate for dry gases without electrolyte was again 0 mm/a. Corrosion rates of welding specimens are globally in the same order of magnitude than the specimens without welding.


## 5.5. Permeation tests

Results from hydrogen permeation tests on the welded steel J55 can be seen in Figure 24. In Figure 24 a) the permeation current as function of time for two consecutive loadings can be seen, in Figure 24 b) a detail of the permeation current as function of time for the first loading including the evaluation is shown, in Figure 24 c) a detail of the permeation current as function of time for the second loading with the evaluation is presented.



c)

Figure 24: Hydrogen permeation current as function of time for 2 consecutive loadings for the welded steel J55, Oxidation cell with 0.1M NaOH and the charging cell with 3.5%NaCl + 1 g/l Thiourea a) overview of two charging cycles, b) detail of 1st charging, c) detail of  $2^{nd}$  charging.

The first loading results in an effective diffusion coefficient Deff of  $1.69 \cdot 10-6 \frac{cm^2}{s}$  and the second loading Deff gave a value of  $2.36 \cdot 10-6 \frac{cm^2}{s}$  (Table 7). These values are comparable to those found in the literature. The welded ferritic pearlitic steel J55 has more traps, as this steel



is also more deformed than the ferritic-pearlitic K55. Consequently the diffusion coefficients of the weld are lower.

# loading	$D_{eff}\left[\frac{cm^2}{s}\right]$
1	1.69 · 10 <sup>-6</sup>
2	2.36 · 10 <sup>-6</sup>

Table 7: Effective diffusion coefficient of hydrogen in a welded steel J55 at room temperature.



# 6.Investigation of a welded steel K55

As for J55 steel grade, a weld was performed on a K55 steel grade, with a V-joint shape. Preheating temperature was set to 110 °C and the maximum interpass temperature was set to 235 °C. There was no Post Welding Heat Treatment (PWHT). Therefore, it can be considered that local stresses were not reduced, the hardness mapping according to Vickers [HV1] can be seen in Figure 25. The mapping shows hardness values from 171 up to 316 HV1. It is noted that the measured hardness exceeds the recommended value of 250 HV for H<sub>2</sub>S applications according to NACE standard MR0175. Due to specimen manufacturing from the centre of the weld, the outer layers, which had the highest level of hardness, were removed and not tested.



Figure 25: Hardness mapping according to Vickers HV1 for the welded steel K55.

Test pieces for all the following tests were manufactured on the weld area.

#### 6.1. Time to failure of Constant Load Tests (CLT)

Figure 26 shows the time to failure of welded ferritic pearlitic steel K55. No fracture occurred under any condition. Figure 26 below shows the time to failure in gas A (120 bar H<sub>2</sub>), gas B (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub>), gas C (120 bar H<sub>2</sub> + 1 bar H<sub>2</sub>S) and gas D (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub> + 1 bar H<sub>2</sub>S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar H<sub>2</sub>). All tests were performed at room temperature.





Figure 26: Time to failure of steel K55 welded in autoclave tests at room temperarture in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

#### 6.2. Hydrogen content

Figure 27 illustrates the hydrogen uptake at 25 °C in gas A and gas B, both gases do not contain hydrogen sulfide H<sub>2</sub>S. Figure 27 also shows the hydrogen uptake in gas C and gas D after autoclave tests with the welded steel K55 at room temperature. The lowest value is 1.03 ppm for gas A and the highest value is 2.69 ppm for immersion in gas C, the blank hydrogen amount is 0.49 ppm. All amounts of hydrogen were measured using TDS.



Figure 27: Hydrogen uptake of steel K55 welded in autoclave tests at RT in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

Table 8 below shows the results of hydrogen uptake after testing the welded steel K55 in mentioned conditions at room temperature.



Table 8: Hydrogen uptake of steel K55 welded in autoclave tests at 25 °C in, a) gas A (100 bar  $H_2$ ), b) gas B (120 bar  $H_2$  + 15 bar  $CO_2$ ), c) gas C (100 bar  $H_2$  + 1 bar  $H_2$ S) and d) gas D (100 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

condition		Hydrogen content [ppm] after immersion in	
		dry gas	gas + electrolyte (200 g/l)
	gas A	1.03	1.68
room	gas B		1.74
temperature	gas C		2.69
	gas D		1.98
g/l = g/l NaCl			

The ferritic pearlitic welded material K55 has a higher hydrogen content than the ferritic pearlitic steel K55 without welding. Nevertheless, the 22 HRC criterion for resistance to  $H_2S$  is met and this is confirmed by the fact that no cracking at all occured.

## 6.3. SEM investigation

Figure 28 shows the cross-section of pearlitic ferritic steel welded K55 at a magnification of 300x.



Figure 28 SEM analysis of a welded steel K55 with sour gas in autoclave tests at RT in gas D (120 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2S$ ).

Considering the cross-section of Figure 28, we can observe that there has been no localized deformation. For this reason, there is a good applicability of the welded steel K55. The welded steel K55 is no susceptible to HE.



#### 6.4. Permeation tests

The oxidation current was measured as function of time to determine the effective diffusion coefficient. Results from hydrogen permeation tests on of a welded ferritic perlitic steel K55 can be seen in Figure 29. In Figure 29 a) the permeation current as function of time for two consecutive loadings can be seen, in Figure 29 b) a detail of the permeation current as function of time for the first loading including the evaluation is shown, in Figure 29 c) a detail of the permeation is presented.



Figure 29: Hydrogen permeation current as function of time for 2 consecutive loadings for steel K55, Oxidation cell with 0.1M NaOH and the charging cell with 3.5%NaCl + 1 g/l Thiourea a) overview of two charging cycles, b) detail of 1st charging, c) detail of  $2^{nd}$  charging.



The first loading resulted in an effective diffusion coefficient  $D_{eff}$  of  $4.57 \cdot 10^{-7} \frac{cm^2}{s}$  and in the second loading  $D_{eff}$  was  $6.5 \cdot 10^{-7} \frac{cm^2}{s}$  (Table 9 ). These values are comparable to those found in the literature. The welded steel K55 has more traps, as this steel is also more deformed than the unwelded ferritic-pearlitic steel. Consequently the diffusion coefficients are lower.

Table 9: Effective diffusion coefficient of hydrogen in steel K55 welded at room temperature.

# loading	$D_{eff}[rac{cm^2}{\mathrm{s}}]$
1	4.57 · 10 <sup>-7</sup>
2	6.50 · 10 <sup>-7</sup>

#### 6.6. Corrosion rate

Figure 30 shows the corrosion rate of a welded ferritic pearlitic steel K55. Corrosion rate of welded steel K55 in autoclave tests at 25 °C in, a) gas A (100 bar H<sub>2</sub>), b) gas B (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub>), c) gas C (100 bar H<sub>2</sub> + 1 bar H<sub>2</sub>S) and d) gas D (100 bar H<sub>2</sub> + 15 bar CO<sub>2</sub> + 1 bar H<sub>2</sub>S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar H<sub>2</sub>).



Figure 30: Hydrogen uptake of steel K55 welded in autoclave tests at RT in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2$  + 15 bar  $CO_2$ ), gas C (120 bar  $H_2$  + 1 bar  $H_2S$ ) and gas D (120 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

The corrosion rate of welded steel K55 was in all gases below 0.1 mm/a. The corrosion rate was 0 mm/a in dry gas A without electrolyte.



## 7.Investigation of a steel P110

#### 7.1. Time to failure of Constant Load Tests (CLT)

Figure 31 shows the time to failure of tempered martensitic steel P110 for all gas compositions. No fracture occurred under any condition. Figure 31 below shows the time to failure in gas A (120 bar H<sub>2</sub>), gas B (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub>), gas C (120 bar H<sub>2</sub> + 1 bar H<sub>2</sub>S) and gas D (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub> + 1 bar H<sub>2</sub>S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar H<sub>2</sub>).



Figure 31: Time to failure of a tempered martensitic steel P110 at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2$  + 15 bar  $CO_2$ ), gas C (120 bar  $H_2$  + 1 bar  $H_2S$ ) and gas D (120 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

#### 7.2. Hydrogen content

Figure 32 illustrates the hydrogen uptake at 25 °C in gas A and gas B, both gases do not contain hydrogen sulfide  $H_2S$ . Figure 32 also shows the hydrogen uptake in gas C and gas D after autoclave tests of P110 at room temperature. The lowest value is 0.09 ppm for gas A and the highest value is 1.65 ppm for gas D. All amounts of hydrogen were measured using TDS. An addition of  $H_2S$  resultend in a significant hydrogen uptake.





Figure 32: Hydrogen uptake of steel P110 in autoclave tests at RT in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar H2S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

Table 10 below shows the results of the hydrogen uptake after testing steel P110 in in the autoclave tests, the lowest value is 0.09 ppm for gas A and the highest value is 1.65 ppm for gas D, in comparison the blank value is 0.49 ppm. There is almost no hydrogen uptake in  $H_2S$  free conditions.  $H_2S$  containing conditions resulted in a significant hydrogen uptake.

Table 10: Hydrogen uptake of steel P110 in autoclave tests at 25 °C in, a) gas A (100 bar  $H_2$ ), b) gas B (120 bar  $H_2$  + 15 bar  $CO_2$ ), c) gas C (100 bar  $H_2$  + 1 bar  $H_2$ S) and d) gas D (100 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

condition		Hydrogen content [ppm] after immersion in	
		dry gas	gas + electrolyte (200 g/l)
	gas A	0.09	0.22
room	gas B		0.22
temperature	gas C		0.94
	gas D		1.65
	gas D		1.65

g/l = g/l NaCl

The tempered martensitic material P110 has the same order of magnitude of hydrogen content as the tempered martensitic steel L80 with lower strength.

It is much more susceptible to hydrogen embrittlement than the tempered martensitic steel L80 with lower strength, because of the higher strength of the P110.

## 7.3. SEM investigation

Figure 33 shows the cross-section of tempered martensite steel P110 at a magnification of 300x.





Figure 33: Cross-section of steel P110 at magnification 300x, with Gas D containing 200g/I NaCl at RT.

Considering the cross-section of Figure 33, it was observed that there is a pronounced localized attack in gas D The approximate maximum depth of attack is 55  $\mu$ m, although it cannot be excluded that deeper attacks may be found at another point on the sample. For this reason, there is limited applicability of the material P110 under the influence of hydrogen sulphide. Based on ISO 15156, the steel P110 should be employed for H<sub>2</sub>S application only if temperature is above 80°C. Therefore, at room temperature and with gas D, it was out of its application domain.

## 7.4. Corrosion rate

Figure 34 shows the corrosion rate of a tempered martensitic steel P110. Corrosion rate of steel P110 in autoclave tests at 25 °C in, a) gas A (100 bar H<sub>2</sub>), b) gas B (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub>), c) gas C (100 bar H<sub>2</sub> + 1 bar H<sub>2</sub>S) and d) gas D (100 bar H<sub>2</sub> + 15 bar CO<sub>2</sub> + 1 bar H<sub>2</sub>S) with an electrolyte 200 g/l NaCl rotating.



Figure 34: Hydrogen uptake of steel P110 in autoclave tests at RT in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating.



The highest corrosion rate of steel P110 was measured in gas B and C with a rate of 0.30 mm/a. The lowest value was measured for gas D with a value of 0.12 mm/a. The corrosion rate for dry gases without electrolyte was 0 mm/a.



# 8. Investigation of a steel 20MnV5

#### 8.1. Time to failure of Constant Load Tests (CLT)

Figure 35 shows the time to failure of a ferritic pearlitic steel 20MnV5. No fracture occurred under any tested condition. Figure 35 shows the time to failure in gas A (120 bar H<sub>2</sub>), gas B (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub>), gas C (120 bar H<sub>2</sub> + 1 bar H<sub>2</sub>S) and gas D (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub> + 1 bar H<sub>2</sub>S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar H<sub>2</sub>).



Figure 35: Time to failure of a ferritic pearlitic steel 20MnV5 at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

#### 8.2. Hydrogen content

Figure 36 illustrates the hydrogen uptake at 25 °C in gas A and gas B, both gases do not contain hydrogen sulfide  $H_2S$ . Figure 36 also shows the hydrogen uptake in gas C and in gas D. In the autoclave tests with the steel 20MnV5 at room temperature, the lowest value is 0.09 ppm for dry gas A. In comparison, the blank value is 0.11 ppm, which can be considered as identical. The highest value is 2.71 ppm for gas C with 200 g/l NaCl, the amount of hydrogen was measured using TDS. There is a significant hydrogen uptake in gases C and D when an electrolyte is present. This is due to the lowering of the solution pH by  $H_2S$ .





Figure 36: Hydrogen content of a ferritic pearlitic steel 20MnV5 at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2$  + 15 bar CO<sub>2</sub>), gas C (120 bar  $H_2$  + 1 bar  $H_2$ S) and gas D (120 bar  $H_2$  + 15 bar CO<sub>2</sub> + 1 bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

Table 11 below compares the hydrogen uptake for different conditions at room and at high temperatures.

Table 11: Hydrogen uptake of a ferritic pearlitic steel 20MnV5 at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2$  + 15 bar CO<sub>2</sub>), gas C (120 bar  $H_2$  + 1 bar  $H_2$ S) and gas D (120 bar  $H_2$  + 15 bar CO<sub>2</sub> + 1 bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry conditions with gas A (120 bar  $H_2$ ).

condition		Hydrogen content [ppm] after immersion in	
		dry gas	gas + electrolyte (200 g/l)
	gas A	0.09	0.42
room	gas B		0.37
temperature	gas C		2.71
	gas D		2.65

g/l = g/l NaCl



## 8.3.SEM investigation



Figure 37 shows the cross-section of ferritic pearlitic steel 20MnV5 at a magnification of 300x.

Figure 37: Cross-section of steel 20MnV5 at magnification 300x, with Gas D containing 200g/l NaCl at RT.

The cross-section of the steel 20MnV5 shows that there are no cracks on the surface. The material seems to be resistant under the tested conditions and is appropriate for hydrogen applications.

## 8.4. Corrosion rate

Figure 38 shows the corrosion rate of a ferritic pearlitic steel 20MnV5. Corrosion rate of steel 20MnV5 in autoclave tests at 25 °C in between 0.18 and 0.54 mm/a.



Figure 38: Hydrogen uptake of steel 20MnV5 in autoclave tests at RT in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating.

The highest corrosion rate of steel 20MnV5 was measured in gas B with a rate of 0.54 mm/a. The lowest value was measured in gas D with a value of 0.18 mm/a. The corrosion rate for immersion in dry gases without electrolyte was 0 mm/a.



## 9. Investigation of a Duplex steel 2205

#### 9.1. Time to failure of Constant Load Tests (CLT)

Figure 39 shows the time to failure of Duplex steel 2205 consists of ferrite and austenite, with an average austenite content of  $39.5 \pm 3.1$  %. One fracture occurred under gas D where H<sub>2</sub>S is present. The tests were carried out at room temperature in five different atmospheres, dry gas A (120 bar H<sub>2</sub>) as well as gas A, gas B (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub>), gas C (120 bar H<sub>2</sub> + 1 bar H<sub>2</sub>S) and gas D (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub> +1 bar H<sub>2</sub>S) with an addition of electrolyte (200 g/l NaCl) were rotated during the test.



Figure 39: Time to failure of a Duplex steel 2205 consists of a ferrite and austenite microstructure at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2$  + 15 bar CO<sub>2</sub>), gas C (120 bar  $H_2$  + 1 bar  $H_2$ S) and gas D (120 bar  $H_2$  + 15 bar CO<sub>2</sub> + 1 bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

#### 9.2. Hydrogen content

Figure 40 illustrates the hydrogen uptake of Duplex steel at 25 °C in gas A (120 bar H<sub>2</sub>), gas B (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub>), gas C (120 bar H<sub>2</sub> + 1 bar H<sub>2</sub>S) and gas D (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub> + 1 bar H<sub>2</sub>S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar H<sub>2</sub>). In the autoclave tests with Duplex 2205 at room temperature, the lowest value is 4.08 ppm for immersion in dry gas A and the highest value is 7.02 ppm for immersion in gas D, the amount of hydrogen was measured using TDS. In comparison, the blank value is 4.72 ppm, which can be seen as scatter. There is a slight inctrease hydrogen uptake in all gases, in case an electrolyte is present.





Figure 40: Hydrogen uptake of a Duplex 2205 microstructure at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

The following Table 12 compares the hydrogen uptake for different conditions at room temperature.

Table 12: Hydrogen uptake of a Duplex 2205 at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating and one dry conditions with gas A (120 bar  $H_2$ ).

condition		Hydrogen content [ppm] after immersion in	
		dry gas	gas + electrolyte (200 g/l)
	gas A	4.08	4.68
room	gas B		6.30
temperature	gas C		5.57
	gas D		7.02

g/l = g/l NaCl

#### 9.3. SEM investigation of fracture surface

Figure 41 shows a fracture image of the Duplex 2205 tested in gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  Bar  $H_2S$ ), 200 g/l NaCl, room temperature with a magnification of 20. Under these conditions a fracture occurred. On the upper side of the fracture surface, at the 12 o'clock position, the starting point of the fracture can be seen. The area at the upper side is a brittle hydrogen fracture. The ductile area is visible in the lower area of the fracture surface. A hydrogen concentration of 7.02 ppm was measured using the TDS.





Figure 41: Fracture surface of Duplex steel 2205 austenite tested in gas D (120 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2S$ ), 200 g/l NaCl, room temperature, 20x.

Figure 42 shows in detail the different fracture zones of the Duplex steel in SEM images. The fractured surface of a hydrogen crack can be separated into two parts. In Figure 42 a), in the yellow frame, a clear hydrogen fracture with river patterns can be observed. The fracture mode in this area is dominated by hydrogen induced brittle quasi cleavage fracture. Figure 42 b) the same fracture area in the yellow frame is shown with a higher magnification, of 3000. Figure 42 c) and d) document show the ductile fracture surface at a magnification of 1000x (c)) and 3000x (d)).





Figure 42: Details of fracture surface of Duplex 2205 in a) and b) brittle fracture area, c) and d) shows the ductile fractured area.

The Duplex steel 2205 consisting of ferritic and austenitic grains shows a high susceptibility to hydrogen embrittlement. As per ISO 15 156-3, Duplex steel 2205 should be used for  $H_2S$  application, only if partial pressure of  $H_2S$  is below 2 kPa (0.02 bar). Therefore, with 1 bar of  $H_2S$ , the tests were performed out of its application domain.



# 10.Investigation of a Alloy 625

#### 10.1. Time to failure of Constant Load Tests (CLT)

Figure 43 shows the time to failure of a welded Alloy 625 with a dendritic microstructure. No fracture occurred under any condition. Figure 43 shows the time to failure at room temperature in gas A (120 bar H<sub>2</sub>), gas B (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub>), gas C (120 bar H<sub>2</sub> + 1 bar H<sub>2</sub>S) and gas D (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub> + 1 bar H<sub>2</sub>S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar H<sub>2</sub>).



Figure 43: Time to failure of a welded Alloy 625 with a dendritic microstructure at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2$  + 15 bar CO<sub>2</sub>), gas C (120 bar  $H_2$  + 1 bar  $H_2$ S) and gas D (120 bar  $H_2$  + 15 bar CO<sub>2</sub> + 1 bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

#### 10.2. Hydrogen content

Figure 44 illustrates the hydrogen uptake at 25 °C in gas A (120 bar H<sub>2</sub>), gas B (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub>), gas C (120 bar H<sub>2</sub> + 1 bar H<sub>2</sub>S) and gas D (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub> + 1 bar H<sub>2</sub>S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar H<sub>2</sub>). In the autoclave tests with the Alloy 625 at room temperature, the lowest value is 1.13 ppm for immersion in dry gas A and the highest value is 6.86 ppm for immersion in wet gas D. The measured blank value is 0.78 ppm, which is below the dry condition, all amounts of hydrogen was measured using TDS. There is an increased hydrogen uptake in gases C and D when an electrolyte is present. This is due to the lowering of the solution pH by H<sub>2</sub>S.





Figure 44: Hydrogen uptake of a welded Alloy 625 with a dendritic microstructure at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2$  + 15 bar  $CO_2$ ), gas C (120 bar  $H_2$  + 1 bar  $H_2$ S) and gas D (120 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

There is a low to moderate hydrogen uptake in the autoclave tests in conditions without  $H_2S$ .  $H_2S$  containing conditions lead to high hydrogen contents in the steel.

The Table 13 shows the hydrogen uptake of welded Alloy 625 at room temperature in gas A (120 bar H<sub>2</sub>), gas B (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub>), gas C (120 bar H<sub>2</sub> + 1 bar H<sub>2</sub>S) and gas D (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub> + 1 bar H<sub>2</sub>S) with an electrolyte 200 g/l NaCl rotating and one dry conditions with gas A (120 bar H<sub>2</sub>). The values are taken from Figure 44.

Table 13: Hydrogen uptake of a welded Alloy 625 is dendritic microstructure at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2$  + 15 bar  $CO_2$ ), gas C (120 bar  $H_2$  + 1 bar  $H_2S$ ) and gas D (120 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating and one dry conditions with gas A (120 bar  $H_2$ ).

condition		Hydrogen content [ppm] after immersion in	
		dry gas	gas + electrolyte (200 g/l)
	gas A	1.13	1.41
room	gas B		2.03
temperature	gas C		4.49
	gas D		6.86

g/I = g/I NaCl



## 10.3. SEM investigation

Figure 45 shows the cross-section of Alloy 625 with a dendritic microstructure at a magnification of 300x.



Figure 45: Cross-section of Alloy 625 at magnification 300x, with Gas D containing 200g/l NaCl at RT.

The cross-section of the Alloy 625 shows that there is no localized attack at all on the surface. The material seems to be highly resistant under test conditions and is appropriate for hydrogen applications. According to the standard ISO 15156-3, it is applicable in  $H_2S$  environment at any temperature or partial pressure.



# 11.Investigation of a stainless steel 316L (supplier 1)

#### 11.1. Time to failure of Constant Load Tests (CLT)

Figure 46 shows the time to failure of a stainless steel 316L from supplier 1. No fracture occurred under any condition. Figure 46 shows as an example the time to failure at room temperature in gas A (120 bar H<sub>2</sub>), gas B (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub>), gas C (120 bar H<sub>2</sub> + 1 bar H<sub>2</sub>S) and gas D (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub> + 1 bar H<sub>2</sub>S) with addition of an electrolyte 200 g/l NaCl cyclic wetting (rotating autoclave) and in comparison one dry condition with gas A (120 bar H<sub>2</sub>).



Figure 46: Time to failure of a stainless steel 316L from supplier 1 at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

#### 11.2. Hydrogen content

Figure 47 illustrates the hydrogen uptake at 25 °C in the 4 test gases with addition of an electrolyte with 200 g/l NaCl under rotating autoclave conditions and in dry conditions with gas A (120 bar H<sub>2</sub>). In the autoclave tests with the stainless steel 316L at room temperature, the lowest value is 1.70 ppm for immersion in gas A and the highest value is 2.04 ppm for immersion in gas D. The measured blank value is 1.94 ppm, which is slightly above of the dry condition in gas A There is a slightly higher hydrogen uptake in gases C and D when H<sub>2</sub>S dissolves in the electrolyte.





Figure 47: Hydrogen uptake of a stainless steel 316L from supplier 1 at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2$  + 15 bar  $CO_2$ ), gas C (120 bar  $H_2$  + 1 bar  $H_2S$ ) and gas D (120 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

Table 14 shows the hydrogen uptake of a stainless steel 316L from supplier 1 at room temperature in 4 different gases.

Table 14: Hydrogen uptake of a stainless steel 316L from supplier 1 at room temperature in gas A (120 bar H2), gas B (120 bar  $H_2$  + 15 bar  $CO_2$ ), gas C (120 bar  $H_2$  + 1 bar  $H_2S$ ) and gas D (120 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating and one dry conditions with gas A (120 bar  $H_2$ ).

condition		Hydrogen content [ppm] after immersion in	
			gas + electrolyte (200 g/l)
	gas A	1.70	1.83
room	gas B		1.92
temperature	gas C		1.95
	gas D		2.04

g/l = g/l NaCl



## 11.3. SEM investigation

Figure 48 shows the cross-section of stainless steel 316L from supplier 1 at a magnification of 300x.



Figure 48: Cross-section of a stainless steel 316 L (supplier 1) at magnification 300x, with gas D containing 200g/l NaCl at RT.

There is no corrosive attack at the surface. Stainless steel 316L from supplier 1 can be used for hydrogen storage at the tested conditions. As per information, based on the standard ISO 15156-3, austainitic satinless steels 316L can be used for  $H_2S$  application provided that the partial pressure is lower than 10.2 kPa (0.1 bar). It is noticed that the material was used out of its application domain with 1 bar of  $H_2S$ , nevertheless, it still seems applicable. The stainless steel is not susceptible to hydrogen embrittlement under the tested conditions.



# 12.Investigation of a stainless steel 316L (supplier 2)

#### 12.1. Time to failure of Constant Load Tests (CLT)

Figure 49 shows the time to failure of stainless steel 316L from supplier 2. Again no fracture occurred under any condition. Figure 46 shows the time to failure at room temperature in the 4 gases. All gases were additionally cyclic wetting with an electrolyte (200 g/l NaCl) and in comparison one dry condition.



Figure 49: Time to failure of a stainless steel 316L from supplier 2 at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

#### 12.2. Hydrogen content

Figure 50 and Table 15 illustrates the hydrogen uptake. In the autoclave tests with the stainless steel 316L at room temperature, the lowest value is 3.17 ppm for immersion in gas A and the highest value is 4.53 ppm for immersion in gas C, the amount of hydrogen was measured using TDS. The blank value in the material is 3.20 ppm, which is the same value as in dry condition (gas A) There is no preferential hydrogen uptake in sour (H<sub>2</sub>S containing) conditions. The presence of an electreolyte results in a certain (low) hydrogen uptake compared to dry conditions.





Figure 50: Hydrogen uptake of a stainless steel 316L (supplier 2) at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating and one dry conditions with gas A (120 bar  $H_2$ ).

Table 15: Hydrogen uptake of a stainless steel 316L (supplier 2) at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating and one dry conditions with gas A (120 bar  $H_2$ ).

condition		Hydrogen content [ppm] after immersion in	
		dry gas	gas + electrolyte (200 g/l)
	gas A	3.17	4.08
room	gas B		4.48
temperature	gas C		4.53
	gas D		4.05
// //			

g/l = g/l NaCl

The stainless steel is not susceptible to hydrogen embrittlement and hydrogen uptake is low (for a Corrosion Resistant Alloy) due to the low diffusion coefficient of the steel. It is observed that the hydrogen content is higher than the one with 316L steel from supplier 1. This point highlights the uncertainty that can be observed between two steels of the same grade but manufactured by different suppliers.



## 12.3. SEM investigation

Figure 51 shows the cross-section of stainless steel 316L from supplier 2 at a magnification of 300x.



Figure 51: Cross-section of a stainless steel 316L (supplier 2) at magnification 300x, with Gas D containing 200g/l NaCl at RT.

There is no attack at the surface. According to the information based on the standard ISO 15156-3, 316L stainless steel can be used for  $H_2S$  applications, provided the partial pressure is below 10.2 kPa (0.1 bar). The steel can be used under tested pressure hydrogen conditions.



# 13.Investigation of a 13%Cr steel

#### 13.1. Time to failure of Constant Load Tests (CLT)

Figure 52 shows the time to failure of a tempered martensitic 13% Cr-steel. No fracture occurred under any condition. Figure 46 shows the time to failure at room temperature in the 5 testing conditions.



Figure 52: Time to failure of a tempered martensite 13% Cr-steel at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2$  + 15 bar  $CO_2$ ), gas C (120 bar  $H_2$  + 1 bar  $H_2$ S) and gas D (120 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

#### 13.2. Hydrogen content

Figure 53 illustrates the hydrogen uptake at the tested conditions. At room temperature, the lowest hydrogen value is 0.42 ppm for immersion in dry gas A and the highest value is 7.02 ppm for immersion in gas D. The blank The measured blank hydrogen value is 0.52 ppm. There is a certain hydrogen uptake in gases C and D when  $H_2S$  and electrolyte is present.



Figure 53: Hydrogen uptake of a tempered martensite 13% Cr-steel at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2$  + 15 bar CO<sub>2</sub>), gas C (120 bar  $H_2$  + 1 bar  $H_2$ S) and gas D (120 bar  $H_2$  + 15 bar CO<sub>2</sub> + 1 bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).



Table 16 shows the results from Figure 53 as a Table.

Table 16: Hydrogen uptake of a 13% Cr-steel at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar CO<sub>2</sub>), gas C (120 bar  $H_2 + 1$  bar  $H_2$ S) and gas D (120 bar  $H_2 + 15$  bar CO<sub>2</sub> + 1 bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry conditions with gas A (120 bar  $H_2$ ).

condition		Hydrogen content [ppm] after immersion in	
		dry gas	gas + electrolyte (200 g/l)
	gas A	0.42	0.53
room	gas B		2.57
temperature	gas C		6.13
	gas D		7.02
$\alpha /   = \alpha /   N   \alpha C  $			

g/l = g/l NaCl

It is observed that hydrogen uptake of the tempered martensite 13 % Cr is higher than the austentic 316 L, when H<sub>2</sub>S and electrolyte is present.

## 13.3.SEM investigation

Figure 54 shows the cross-section of 13% Cr-steel at a magnification of 300x.



Figure 54: Cross-section of a tempered martensite 13% Cr-steel at magnification 300x, with Gas D containing 200g/l NaCl at RT.

There is a certain uniform attack in sour conditions. This was not the case for  $H_2S$  free conditions.



## 13.4. Corrosion rate

Figure 55 shows the corrosion rate of the 13% Cr-steel for the tested conditions.



Figure 55: Hydrogen uptake of a tempered martensite 13% Cr-steel in autoclave tests at RT in gas A (120 bar H2), gas B (120 bar  $H_2$  + 15 bar  $CO_2$ ), gas C (120 bar  $H_2$  + 1 bar  $H_2$ S) and gas D (120 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating.

The corrosion rate of 13% Cr-steel was in all cases with electrolyte between 0.3 and 0.48 mm/a. The corrosion rate for dry gas without electrolyte was 0 mm/a.



# 14.Investigation of steel K55 precorrosion

In the frame of conversion of underground storage, casing and completion that would be used for hydrogen service, might be already covered with rust due to uniform corrosion during their operation. It was wondering if this uniform corrosion at the surface of the steel could enhance or slowdown hydrogen impacts. Therefore, pre-corroded specimens were tested.

## 14.1. Time to failure of Constant Load Tests (CLT)

Figure 56 shows the time to failure of ferritic perlitic steel K55 in the pre-corroded condition. The pre-corrosion was done in an rotating autoclave with 200 g/l NaCl at 15 bar  $CO_2$  for one week prior to the CLT, a pre-corroded sample can be seen in Figure 57. No fracture occurred under any condition. Figure 56 shows the time to failure.



Figure 56: Time to failure of a ferritic perlitic pre-corroded steel K55 at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2$ S) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).



Figure 57: Pre-corroded sample after one week in a autoclave with 200 g/l NaCl at room temperature with 15 bar CO<sub>2</sub>.



## 14.2. Hydrogen content

Figure 58 illustrates the hydrogen uptake. In the autoclave tests the lowest hydrogen value is 0.67 ppm (which is most likely an outlier) and the highest value is 3.64 ppm for immersion in gas B.



Figure 58: Hydrogen uptake of a ferritic perlitic pre-corroded steel K55 at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

Table 17 shows the hydrogen uptake as a table.

Table 17: Hydrogen uptake of a ferritic perlitic pre-corroded steel K55 at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2$  + 15 bar  $CO_2$ ), gas C (120 bar  $H_2$  + 1 bar  $H_2$ S) and gas D (120 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

condition		Hydrogen content [ppm] after immersion in	
		dry gas	gas + electrolyte (200 g/l)
	gas A	1.57	1.95
room	gas B		3.64
temperature	gas C		0.64
	gas D		3.20

g/l = g/l NaCl

It is observed that hydrogen uptake in pre-corroded K55 sample was higher than in standard K55 specimen. Therefore, the corroded surface of the steel increases the hydrogen content inside the material.



## 14.3.SEM investigation

Figure 59 shows the cross-section of pre-corroded steel K55 at a magnification of 300x.



Figure 59: Cross-section of steel K55 pre-corroded at magnification 300x, with Gas D containing 200g/l NaCl at RT.

The steel K55 pre-corroded is not sensitive to hydrogen embrittlement. There has been no visible damage to the surface.

## 14.4. Corrosion rate

Figure 60 shows the corrosion rate of the pre-corroded steel K55. Corrosion rate of precorroded steel K55 in autoclave tests at 25 °C in, a) gas A (120 bar H<sub>2</sub>), b) gas B (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub>), c) gas C (120 bar H<sub>2</sub> + 1 bar H<sub>2</sub>S) and d) gas D (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub> + 1 bar H<sub>2</sub>S) with addition of electrolyte with 200 g/l NaCl and rotating the autoclave and one dry condition with gas A (120 bar H<sub>2</sub>).



Figure 60: Hydrogen uptake of a ferritic perlitic pre-corroded steel K55 in autoclave tests at RT in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2$ S) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

The corrosion rate of pre-corroded steel K55 is between 0.05 and 0.12 mm/a. The corrosion rate for dry gases without electrolyte was again 0 mm/a.



# 15. Investigation of steel K55 with notch

It is admitted that localized defects as notches can enhance hydrogen embrittlement. It was decided to tests specimens with v-notch in order to highlight any additional impacts.

## 15.1. Time to failure of Constant Load Tests (CLT)

Figure 61 shows the time to failure of notched steel K55. The sample was v-notched with a 0.5 mm indentation to create sharper conditions (thickness loss of 33 %), can be seen in Figure 62. No fracture occurred under any condition. Notching did not result in a sufficient stress increase during testing to embrittle the steel.



Figure 61: Time to failure of a ferritic perlitic steel K55 with a notch at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).



Figure 62: Sample with a v-Notch with a 0.5 mm indentation.

#### 15.2.Hydrogen content

The hydrogen content was not measured, as the hydrogen sample did not change, and therefore the results are the same as in chapter 3.2.



## 15.3. SEM investigation

Figure 63 shows the cross-section of steel K55 with a notch at a magnification of 300x.



Figure 63: Cross-section of steel K55 notched at magnification 300x, with Gas D containing 200g/l NaCl at RT.

There is visible localized attack of the notch. The approximate maximum depth of localized attack is limited to 30  $\mu$ m, although it cannot be excluded that deeper attack may be found at another point of the notch along its circumference.



# 16.Investigation of steel L80 precorroded

#### 16.1. Time to failure of Constant Load Tests (CLT)

Figure 64 below shows time to failure of tempered martensitic pre-corroded steel L80. The pre-corrosion was done in rotating autoclave semifilled with electrolyte with 200 g/l NaCl at 15 bar  $CO_2$  for one week. No cracks at all occurred for the pre-corrdoded steel L80.



*Figure 64:* Time to failure of a tempered martensite steel pre-corroded L80 at room temperature in gas A (120 bar H<sub>2</sub>), gas B (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub>), gas C (120 bar H<sub>2</sub> + 1 bar H<sub>2</sub>S) and gas D (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub> + 1 bar H<sub>2</sub>S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar H<sub>2</sub>).

## 16.2. Hydrogen content

Figure 65 illustrates the hydrogen uptake. The lowest value is 1.51 ppm after immersion in gas A and the highest value is 3.55 ppm for immersion in gas B. The results contain certain scatter. No significant higher hydrogen uptake was obtained in  $H_2S$  containing conditions.




Figure 65: Hydrogen uptake of a tempered martensite steel pre-corroded L80 at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2$  + 15 bar  $CO_2$ ), gas C (120 bar  $H_2$  + 1 bar  $H_2S$ ) and gas D (120 bar  $H_2$  + 15 bar  $CO_2$  + 1 bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

Table 18 shows the hydrogen uptake as a table in a different form.

Table 18: Hydrogen uptake of a tempered martensite steel pre-corroded L80 at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2$  + 15 bar CO<sub>2</sub>), gas C (120 bar  $H_2$  + 1 bar  $H_2$ S) and gas D (120 bar  $H_2$  + 15 bar CO<sub>2</sub> + 1 bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

condition		Hydrogen content [ppm] after immersion in		
		dry gas	gas + electrolyte (200 g/l)	
	gas A	1.51	1.69	
room	gas B		3.55	
temperature	gas C		2.88	
	gas D		2.83	

```
g/l = g/l NaCl
```

It is observed that hydrogen uptake in pre-corroded L80 sample was higher than in standard L80 specimen. Therefore, the corroded surface of the steel increases the hydrogen content inside the material.

#### 16.3. SEM investigation

Figure 66 shows the cross-section of pre-corroded steel L80 at a magnification of 300x.





Figure 66: Cross-section of steel L80 pre-corr. at magnification 300x, with Gas D containing 200g/l NaCl at RT.

The maximum depth of attack depth is 15  $\mu$ m, although it cannot be excluded that deeper sites may be found at another point on the sample. No pronounced localized attack was present.

## 16.4. Corrosion rate

Figure 67 shows the corrosion rate of the pre-corroded steel L80.



Figure 67: Hydrogen uptake of a tempered martensite pre-corroded steel L80 in autoclave tests at RT in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating.

The highest corrosion rate of the pre-corroded steel L80 was measured in gas C with a rate of 0.12 mm/a. The other values are measured in gases A, B and D with a value of 0.05 or 0.06 mm/a. The corrosion rate for dry gases without electrolyte was 0 mm/a.



## 17. Investigation of steel L80 with notch

#### 17.1. Time to failure of Constant Load Tests (CLT)

Figure 68 below shows time to failure of v-notched steel L80 (with a 0.5 mm indentation). No cracks at all occurred for the notched steel.



*Figure 68:* Time to failure of a tempered martensite steel L80 notched at room temperature in gas A (120 bar H<sub>2</sub>), gas B (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub>), gas C (120 bar H<sub>2</sub> + 1 bar H<sub>2</sub>S) and gas D (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub> + 1 bar H<sub>2</sub>S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar H<sub>2</sub>).

#### 17.2.Hydrogen content

The hydrogen content was not measured, as the hydrogen sample did not change so that it had the same uptake as in chapter 4.2.

#### 17.3. SEM investigation

Figure 69 shows the cross-section through the notch of steel L80 at a magnification of 300x.



Figure 69: Cross-section of steel L80 at magnification 300x, with Gas D containing 200g/l NaCl at RT.

The approximate maximum depth of attack is limited to  $35 \mu$ m. Attack is like for the other notched material, that has been investigated (K55), localized.



## 18.Investigation of welded steel J55 precorroded

#### 18.1. Time to failure of Constant Load Tests (CLT)

Figure 70 below shows as time to failure of a ferritic perlitic welded pre-corroded steel J55. Specimens are corroded in a rotating autoclave with 200 g/l NaCl at 15 bar  $CO_2$  for one week. No cracks at all occurred for the pre-corroded steel J55 during the subsequent constant load autoclave testing. Figure 70 shows the time to failure at room temperature in gas A (120 bar H<sub>2</sub>), gas B (120 bar H<sub>2</sub> + 15 bar  $CO_2$ ), gas C (120 bar H<sub>2</sub> + 1 bar H<sub>2</sub>S) and gas D (120 bar H<sub>2</sub> + 15 bar  $CO_2$  + 1 bar H<sub>2</sub>S) with addition of electrolyte with 200 g/l NaCl and one dry condition with gas A (120 bar H<sub>2</sub>).



Figure 70: Time to failure of a ferritic perlitic welded pre-corroded steel J55 at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2$ S) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

## 18.2. Hydrogen content

Figure 71 illustrates the hydrogen uptake of these specimens. The lowest value is 1.07 ppm for immersion in dry gas A and the highest value is 3.32 ppm for immersion in gas D, the amount of hydrogen was measured using TDS.





Figure 71: Hydrogen uptake of a of a ferritic pearlitic welded pre-corroded steel J55 at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2$  + 15 bar CO<sub>2</sub>), gas C (120 bar  $H_2$  + 1 bar  $H_2$ S) and gas D (120 bar  $H_2$  + 15 bar CO<sub>2</sub> + 1 bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

Table 19 shows the numbers of the hydrogen uptake of pre-corroded steel J55. Despite the scatter of results there is a certain increase of hydrogen uptake, when an electrolyte is present.

Table 19: Hydrogen uptake of a ferritic pearlitic welded pre-corroded steel J55 at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2$  + 15 bar CO<sub>2</sub>), gas C (120 bar  $H_2$  + 1 bar  $H_2$ S) and gas D (120 bar  $H_2$  + 15 bar CO<sub>2</sub> + 1 bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

condition		Hydrogen content [ppm] after immersion in		
		dry gas	gas + electrolyte (200 g/l)	
	gas A	1.07	1.51	
room	gas B		3.08	
temperature	gas C		1.67	
	gas D		3.32	

g/l = g/l NaCl

It is noticed that hydrogen uptake of J55 welded and pre-corroded material is similar as the one in th pre-corroded steel K55. The welding does not seem to bring an additional effect on pre-corroded samples, whereas it had an effect on standard samples.

#### 18.3.SEM investigation

Figure 72 shows a cross-section of the pre-corroded steel J55 after the autoclave test. There is a homogenous sulfide layer with a thickness of  $20 - 30 \,\mu\text{m}$ . The layer consists of two sublayers, where the inner one is more heterogeneous, while the outer is more homogeneous.





Figure 72: Cross-section of a welded pre-corroded steel J55at magnification 300x, with Gas D containing 200g/l NaCl at RT.

## 18.4. Corrosion rate

Figure 73 shows the corrosion rate of the welded pre-corroded steel J55.



Figure 73: Hydrogen uptake of a welded pre-corroded steel J55in autoclave tests at RT in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2S$ ) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2S$ ) with an electrolyte 200 g/l NaCl rotating.

The highest corrosion rate of of welded pre-corroded steel J55 are measured in gases C and D. The lowest values are measured in gases A and B with 0.05 mm/a. The corrosion rate for dry gas without electrolyte was 0 mm/a.



# 19.Investigation of welded steel J55 with notch

#### 19.1. Time to failure of Constant Load Tests (CLT)

Figure 74 below shows time to failure of welded steel J55 with a notch, the notch was located in the weld metal. The weld was always in the gauge length of the tensile specimen, but the notch may not necessarily have been in the middle of the weld. No cracks at all occurred for the pre-corrdoded steel L80. Figure 74 shows the time to failure at room temperature in gas A (120 bar H<sub>2</sub>), gas B (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub>), gas C (120 bar H<sub>2</sub> + 1 bar H<sub>2</sub>S) and gas D (120 bar H<sub>2</sub> + 15 bar CO<sub>2</sub> + 1 bar H<sub>2</sub>S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar H<sub>2</sub>).



Figure 74: Time to failure of a ferritic pearlitic welded steel J55 with a notch at room temperature in gas A (120 bar  $H_2$ ), gas B (120 bar  $H_2 + 15$  bar  $CO_2$ ), gas C (120 bar  $H_2 + 1$  bar  $H_2$ S) and gas D (120 bar  $H_2 + 15$  bar  $CO_2 + 1$  bar  $H_2$ S) with an electrolyte 200 g/l NaCl rotating and one dry condition with gas A (120 bar  $H_2$ ).

#### 19.2.Hydrogen content

We have not measured the hydrogen content, as the hydrogen sample has not changed, and therefore has the same recordings as in chapter 5.2.

#### 19.3. SEM investigation

Figure 75 shows a cross-section of steel J55 with a notch at a magnification of 300x.





Figure 75: Cross-section of steel J55 welded at magnification 300x, with Gas D containing 200g/l NaCl at RT.

The welded notched steel J55 shows a 40 -  $50\mu$ m thick layer of sulfides on its surface. There is no localized attack. Therefore, the welding does not seem to amplify any localized attacks on the tip of the notch.



## 20.Summary

## 20.1. Overview on the applicability of investigated steels in hydrogen atmospheres

In Figure 76 and Table 20 an overview of diffusion coefficients derived from permeation measurements of the investigated steel grades is shown.

For the ferritic pearlitic steel K55, there is a small amount of deep traps since the second loading is only slightly faster than the first one.

The steel L80 has more traps, as this tempered martensitic steel is also more deformed than the ferritic-pearlitic steel K55. Consequently the diffusion coefficients are slightly lower. These results show diffusion coefficients for these steels as expected from the literature review as well.

The welded ferritic pearlitic steel J55 has more traps. Consequently, the diffusion coefficient of the welded grade is even lower.

The welded steel K55 contains most traps and the diffusion coefficients are the lowest. The literature confirms that the values are within the typical range for carbon steels.

Usually a low diffusion coefficient is favourable for hydrogen applications since a large number of deeper traps hinders hydrogen to diffuse to zones with high stresses. So low hydrogen diffusion coefficients represent more hydrogen resistant steels for carbon steels.



Figure 76: Overview of all investigated steel grades from which permeation measurements have been carried out



Material	D <sub>eff</sub> [cm²/s]
K55 1 <sup>st</sup> loading	$6.22 \cdot 10^{-6}$
K55 2 <sup>nd</sup> loading	$7.96 \cdot 10^{-6}$
L80 1 <sup>st</sup> loading	$2.68 \cdot 10^{-6}$
L80 2 <sup>nd</sup> loading	$3.98 \cdot 10^{-6}$
J55 welded 1st loading	$1.69 \cdot 10^{-6}$
J55 welded 2 <sup>nd</sup> loading	2.36·10 <sup>-6</sup>
K55 welded 1 <sup>st</sup> loading	$4.57 \cdot 10^{-7}$
K55 welded 2 <sup>nd</sup> loading	$6.50 \cdot 10^{-7}$

Table 20: Overview of the values from the permeation measurements

With respect to corrosion rate in general, one can say that gases containing  $H_2S$  and  $CO_2$  have the highest impact on corrosion rates. An electrolyte has to be present, so that the gases can dissolve and lower its pH value. There are some outliers in the corrosion rates for some materials (e.g. for the steel K55 at each gas, for the welded J55 at RT and 120 bar  $H_2$ , and for steel L80 at RT and 120 bar  $H_2$  + 15 CO<sub>2</sub>). A temperature increase from 25 °C to 120 °C had no significant influence on corrosion rates.

Table 21 shows the summary of results of localized corrosion attacks and cracking respectively for investigated materials.



		Material	max H-uptake / blank value	Depth of localized corrosion attack*	cracking (no/yes)
		20MnV5	2.71 / 0.11	≤ 5 μm	no
		welded J55	2.95 / 0.65	≤ 5 μm	no
	th	welded J55 pre- corroded	3.32 / 0.65	≤ 5 μm	no
	reng	welded J55 with notch	-	≤ 5 μm	no
	d st	K55	2.33 / 0.22	15 µm	no
	g yiel	K55 pre- corroded	3.64 / 0.22	≤ 5 μm	no
	Ising	K55 notched	-	30 µm	no
	crea	welded K55	2.69 / 0.49	≤ 5 μm	no
	.⊑	L80	1.03 / 0.21	68 µm	no
		L80 pre- corroded	3.55 / 0.21	15 µm	no
		L80 notched	-	35 μm	no
		P110	1.65 / 0.23	55 µm	no
		quenched material	0.86 / 0.20	fracture	yes, in gas A and gas D
	2	13%Cr	7.02 / 0.52	≤ 5 μm	no
	g allo	316L supplier 1	2.04 / 1.94	no attack	no
	asing	316L supplier 2	4.53 / 3.20	no attack	no
	crea	Duplex 2205	7.02 / 4.72	fracture	yes, in gas D
4	, <u> </u>	Alloy 625	6.86 / 0.78	no attack	no

Table 21: Summary of results of localized corrosion rates in Constant Load Tests for investigted materials

\* low...≤ 5 μm/mt

\* medium...5 – 30 μm/mt

\* high...≥ 30 μm/mt

\*Table 21 shows the depth of localized corrosion attacks and cracking in a colour code as well. Green represent harm less or not significant localized attack (low or no danger of HE), while



red and orange represent either cracking or deep localized attack that rather easily might result in failure (either by ongoing localized corrosion or by HE). The deepest local corrosive attack was found in gas D consisting of  $H_2$  and additionally  $H_2S$  and  $CO_2$  atmosphere. In Table 22 an overview of the applicability of all investigated steels for use in hydrogen storage is given.



Table 22: Applicability of investigated steels according to results of Constant Load Tests

		Material	Damage	Application with H <sub>2</sub> S based on ISO 15156	Applicability in H <sub>2</sub> environment
		20MnV5	no damage	Not specified	well applicable
	increasing yield strength	welded J55	no damage	Acceptable for H <sub>2</sub> S application for all temperatures	well applicable
		welded J55 pre- corroded	no damage		
		welded J55 with notch	no damage		
		K55	no damage	Acceptable for H <sub>2</sub> S application for all	well applicable when localized corrosion is not an issue
		K55 pre- corroded	no damage		
		K55 with notch	some localized damage	temperatures	
		welded K55	no damage	Acceptable for H₂S application if hardness ≤ 22 HRC	well applicable
		L80	deep localized damage	Acceptable for H <sub>2</sub> S application for all temperatures	applicable when localized corrosion is not an issue
		L80 pre- corroded	some localized damage		
		L80 with notch	some localized damage	is type 1	
	•	P110	deep localized damage	Acceptable for H <sub>2</sub> S application only if T° > 80°C	applicable at RT when no H <sub>2</sub> S is present
		quenched material	failure in H <sub>2</sub>	Not applicable	not applicable
		13%Cr	no damage	Acceptable if pH <sub>2</sub> S < 10.2 kPA	well applicable
	increasing alloy content	316L supplier 1	no damage	Acceptable if	well applicable
		316L supplier 2 no damage		pH <sub>2</sub> S < 10.2 kPA	well applicable
		Duplex 2205	failure in (H <sub>2</sub> + CO <sub>2</sub> + H <sub>2</sub> S)	Acceptable if pH <sub>2</sub> S < 2 kPA	not applicable
Ţ		Alloy 625	no damage	Acceptable for H <sub>2</sub> S application for all temperatures	well applicable

**no damage	**localized damage	**failure	
**well applicable in hydrogen environment	**applicable in hydrogen environment when localized corrosion is not an issue	**not applicable in hydrogen environment	
hystories	D.4.6-0 – Summary report on all investigated steels	8	

investigated steels

\*\* The applicability is indicated by a colour code in Table 22. Materials showing no cracking in constant load testing and nor or very minor localized corrosive attack are considered as "Applicable". Materials showing localized corrosive attack although no cracking occurred in Constant Load Testing are considered as "Applicable with limitations". Finally materials that failed during Constant Load Testing are considered as "Not applicable". The tests were performed with different gas compositions and also at different temperatures. The tests with sour gas composition (gas C and D) were carried out with 1 bar of  $H_2S$  at 121 bar or 136 bar in total = 0.83 % or 0.75 %, which can be seen as very severe compared to the ISO 15156 standard.

#### Applicable according to Constant Load Tests

Although the absorbed hydrogen content is very high compared to the blank value on some steels, no local damage occurred.

The research work done throughout the Hystories project (c.f. reports D4.1, D4.2, D4.3, D4.4, D4.5 and D4.6) supports that the following steels can be used without any identified restrictions for hydrogen service, and in presence of wet environment and  $CO_2$  (up to 15 bar) and in their application domain in presence of H<sub>2</sub>S as specify in ISO 15156 :

#### CRAs

- Alloy 625
- 316L
- 13% Cr

#### **Carbon steels**

- K55
- 20MnV5
- welded J55
- welded K55
- K55 pre-corroded
- welded J55 pre-corroded

#### Applicable with limits according to Constant Load Tests

In these materials, more significant localized attacks were observed, especially under  $H_2S$  environments.

#### **Carbon steels**

- L80
- L80 pre-corroded
- P110



#### Not applicable according to Constant Load Tests

The research work done throughout the Hystories project (c.f. reports D4.1, D4.2, D4.3, D4.4, D4.5 and D4.6) supports that it is highly recommended not to use the following materials in hydrogen:

#### CRAs

• Duplex 2205

#### **Carbon steels**

• quenched material (not commercial)



## Hystories project consortium













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