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Materials for Geothermal Steam Utilization at Higher Temperatures and Pressure

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ABSTRACT

Materials used in high temperature/pressure geothermal steam can be subjected to corrosion due to the aggressiveness of the geothermal fluid and non-condensable gasses such as hydrogen sulfide (H_2S) and carbon dioxide (CO_2), chloride ions (Cl^-) and hydrogen fluoride (HF). Icelandic Deep Drilling well (IDDP-1) in the Krafla area in NA Iceland gave well head temperature of 452°C at 140 bar pressure flowing 12 ks/s of superheated steam. This well alone could produce 25-35 MWe, but due to extreme conditions it is not currently operational. This paper focus on learnings from the material testing performed during the lifetime of the well and reveal several promising material choices for utilizing geothermal steam in extreme conditions like in IDDP1. Work is now ongoing in designing the next Icelandic Deep Drilling (IDDP-2) well in Iceland and expectations are to get 400-500°C steam with well head pressure over 100 bar. This will put several constrains on materials normally suitable for high temperature geothermal steam and calls for redesigning of materials, both down hole and above surface. Safety issues will be considered and direct or indirect use of the steam for electricity production will be discussed. Materials are evaluated based on research from the first IDDP well and possible changes in chemical composition of the geothermal fluid and gas contents are discussed.

1. INTRODUCTION

The Icelandic Deep Drilling Project (IDDP) is a research and development project, aiming at deeper geothermal wells reaching into supercritical conditions. The first well of planned three wells in Iceland was drilled in the Krafla caldera in north east of Iceland in 2008-2009 by the National Power Company in Iceland - Landsvirkjun. This well was designed to be cemented down to 3500 m with a slotted liner down to 4500 m (Thórhallsson Vol 49, January 2014.).

When reaching the depth of 2.1 km the drilling was stopped due to stuck drill bits and after three attempts were two sidetracks were drilled, it was realized that the drill had reached a magma chamber with temperatures > 900°C. This was not the only well to enter magma in the Krafla geothermal region, the well KJ-39 drilled in 2006-2009 intersected magma at 2.5 km (A.K.Mortensen 2010) (Karlsdottir.S.N 2012)

Although the well was much shorter than designed, temperatures/pressures were above superheated conditions and therefore it was decided to test the well and look for possible utilization both with traditional direct use of the steam for steam turbines but as well in a binary process through heat exchangers. The well gave unique opportunities for research on corrosion and degradation of metallic materials in high enthalpy steam. This paper will give overview of the main findings in the material research in the IDDP-1 steam and discuss material selection and safety concerns for future IDDP wells

2. IDDP-1 WELL DESIGN

The IDDP-1 well was designed to reach a depth of 4.5 km to intersect supercritical conditions that were anticipated to exist below 3500 m depth. Five cemented casing strings of different diameters and depths were to exclude the sub-supercritical fluids above 3500 m and offer safety during drilling and later flow-testing. From 3500 m a slotted leader were planned down to 4500 m (Thórhallsson Vol 49, January 2014.) The material selection for the casing was API K-55 except that for the anchor casing reaching to 2400 m (the main pressure barrier).There API T95 steel grade was selected for the top most 290 m for its improved creep resistance at high temperatures. The premium casing connections selected is Hydril/Tenaris 563 with double dovetail thread profiles, except for the 7" holed liner which has API Buttress connections. The 90 m long surface casing is welded together and is API grade X56 line pipe. The casing depths and program had to be modified in the IDDP-1 well and the 13 3/8" anchor casing landed short to 1949 m due to well stability problems. Once the magma was intersected at 2100 m the 9 5/8" production casing was run to 2072 m with the deepest 137 m of it holed to allow fluid entry into the well. Then the section of the production casing above 1640 m was cemented back to surface. The production casing is considered as „sacrificial" casing as far as corrosion and casing damage is concerned as the next casing string, the anchor casing, is the main pressure barrier and carries the wellhead. To minimize possible corrosion from sour gases and dew-point condensates the expansion spool and master valve were factory cladded with welding ANSI 309 material. The master valve was ANSI 1500 with ANSI 2500 flanges of the expanding gate type.

3. IDDP-1 GEOTHERMAL STEAM

The geothermal steam from the IDDP-1 well was superheated with a temperature of 450°C and a pressure of 120 bar at the wellhead. Several samples were taken from the steam to measure the chemical composition during discharging and before the pilot

testing of the IDDP-1 well and has been reported (Armannsson, Geothermics 2014; Hauksson et al., Geothermics 2014). **Table 1** summarizes the chemical composition and characterization of the IDDP-1 steam (Hauksson et al., 2014).

Table 1. Chemical composition of the IDDP-1 superheated steam (Hauksson et al, Geothermics 2014)

Temperature	(°C)	240-270
pH	-	2.7
CO_2	(mg/kg)	339
H_2S	(mg/kg)	732
H_2	(mg/kg)	10
Cl	(mg/kg)	93
F	(mg/kg)	5.0
Fe	(mg/kg)	8.4
B	(mg/kg)	1.0

The amount of H_2S, CO_2 and H_2 is relatively small compared to nearby wells in the Krafla geothermal area. NH_3 was measured in the steam but in very little amount (0.14 mg/kg) (Hauksson et al., 2014). The IDDP-1 steam also contained HCl and HF which made the condensate acidic with pH level ranging from 2.6-3.5 (Armannsson, 2014; Hauksson et al., 2014). Very little NH_3 was measured in the steam, only 0.14 mg/kg. The steam contained both dissolved silica (SiO_2) and silica. Iron (Fe) was measured in rather high concentration 8.4 mg/kg, most likely resulting from corrosion of the casings in the well during heating. The concentration of other minerals was low (Hauksson et al., 2014).

4. IDDP-1 PILOT TESTING

Several experimental projects were done to evaluate the possibility of utilizing the superheated steam from the IDDP-1 well. These experiments were considered essential for future deep drilling projects in general as well as for future utilization of the deep acidic reservoir in Krafla. Material research was included in all the experimental projects, which were the following: wet scrubbing experiments with the superheated steam (Hauksson et al., Geothermics 2014); in-situ erosion-corrosion testing with high velocity of the superheated steam (Karlsdottir et al. Geothermics, 2014); testing of pipes to simulate heat exchanger environments at high temperature and pressure (Karlsdottir et al. NACE 2014); and corrosion testing in the superheated steam (Karlsdottir et al. Geothermics accepted 2014).

The wet scrubbing experiment was successful, i.e. the superheated steam with the acid gas (HCl and HF) can be effectively scrubbed from the steam with brine, condensate or cold water (Hauksson et al., Geothermics 2014). But sulfur (S) that the superheated geothermal steam was found to contain 80 – 100 mg/kg in gaseous form could only be scrubbed from the steam with alkaline water. Also the SiO_2 dust and the dissolved SiO_2 in the steam, which precipitated out of the steam as particles when the pressure was reduced, were washed from the steam with the wet scrubbing. The main concern then was that the scrubbing water then contains SiO_2 particles that could deposit in scrubbing equipment and clog the injection wells if it is mixed with injection brine. When a condensate sample was analyzed after wet scrubbing test it contained 94 mg/l of suspended matter which was found to be solid elemental sulfur (S) and SiO_2 particles (Hauksson et al., 2014). The origin of S was not determined by Hauksson et al. but the hypothesis was that it must have been in gaseous form in the saturated steam and sparingly soluble in water. Possibly in the form of disulfide (HS_2H) or polysulfide (HS_nH) gas. The sulfides could though not be identified in the steam in the wet scrubbing experiment (Hauksson et al., 2014).

The wet scrubbing equipment was inspected for corrosion and scaling and the results from the inspection has been reported (Karlsdottir et al, NACE 2013). The temperature and pressure was on average 282°C and 13 bar when entering the wet scrubbing experimental equipment. The material used for the spray chamber of the wet scrubbing equipment, where the steam was mixed with water, was the nickel based alloy UNS N06625. Minimum corrosion damage was found in the N06625 material; small pits were found at high magnification, see Fig. 1.

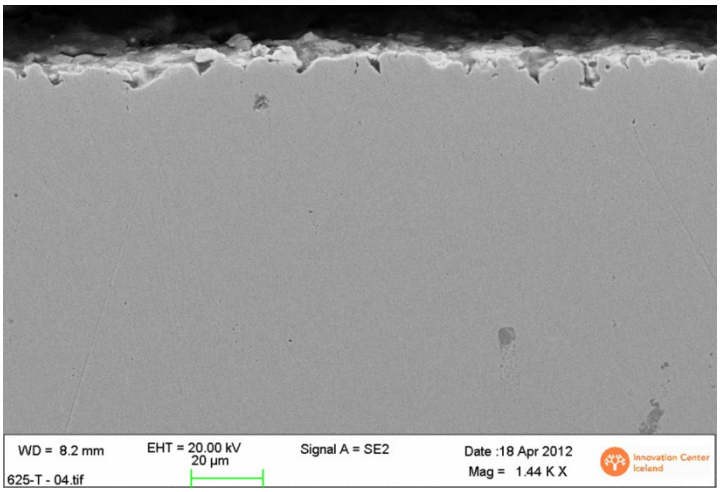

Figure 1. Small pits visual on the surface of UNS N06625 from wet scrubbing experiment in IDDP-1 steam

The pipe connected to the spraying chamber is the mixing zone, made out of the austenitic stainless steel UNS S31254, it consists of a 6 m long pipe spool of narrow diameter, causing increased steam velocity and favorable flow conditions for steam-water contact. No corrosion damages were detected in the S31254 pipe. Carbon steel pipes, type P235GH, were also part of the wet scrubbing experimental equipment and served the role of simulating the gathering system in the steam field. Corrosion was visible in these pipes, both uniform corrosion and pitting corrosion. The carbon steel pipe (type P235GH) connecting the N06625 alloy spraying chamber and the inlet carbon steel steam pipe was heavily corroded. This was reported to be due to the short distance of the connector from the mixing zone in the spraying chamber. Steam and moisture separators were part of the experimental equipment, connected to the carbon steel pipes (gathering system). Corrosion was visible in the separators, both uniform and pitting, and the surfaces were largely covered with a corrosion film, with crystals rich in iron (Fe) and sulfur (S) (Karlsdottir, et al. NACE 2013). Surface films like these are common on carbon steel in geothermal environment and can be protective against further corrosion but any disruption/shut-offs will cause breakage in the film that will accelerate the corrosion rate.

To evaluate whether the superheated steam could be used directly for the evaporator in a binary power production system without prior removal of the acid gases (HF, HCl), silica and sulfur impurities from the steam an experimental project was carried out. The results have recently been reported (Karlsdottir et al., NACE 2014). This project was set up to try answering the question what material would be best for such heat exchanger units and also to give insight on whether silica or sulfur scaling would be a problem. The test unit composed of 6 m long pipes from eight different materials; the stainless steels S31254, S32707 and N08028, the nickel based alloys N06625, and N08825, and the titanium alloys R50400 and R52400, and the carbon steel P265GH for a reference. The inlet temperature and pressure were 330-350°C and 52-62 bar and the steam condensed in the tubes with an outlet temperature of about 270°C. It was reported that severe problems occurred when steam traps were used at the outlet end of the tubes due to extensive clogging of silica. The traps had to be replaced with orifice plates so the material testing could continue. The results showed very little silica scaling. The high pressure and temperature and possibly the acid condensate removing deposited silica from the pipes was thought to be the cause. No elemental sulfur was found in the test pipes but Fe and S rich deposits were found in the pipes. The P265GH carbon steel pipe was heavily corroded after only 2 days of testing which confirms the high corrosion rate in the acid condensate. The S31254 pipe showed little evidence of uniform or pitting corrosion but extensive cracking was observed. The cracking was identified to be intergranular stress corrosion cracking (SCC) caused by the high temperature, acid chloride and sulfide environment and stresses caused by thermal and pressure fluctuation formed in the multiple shut-downs (due to scaling problems in the orifices and the steam traps). Figure 2 shows the cross-section view of the S31254 pipe where the SCC cracks can be seen clearly.

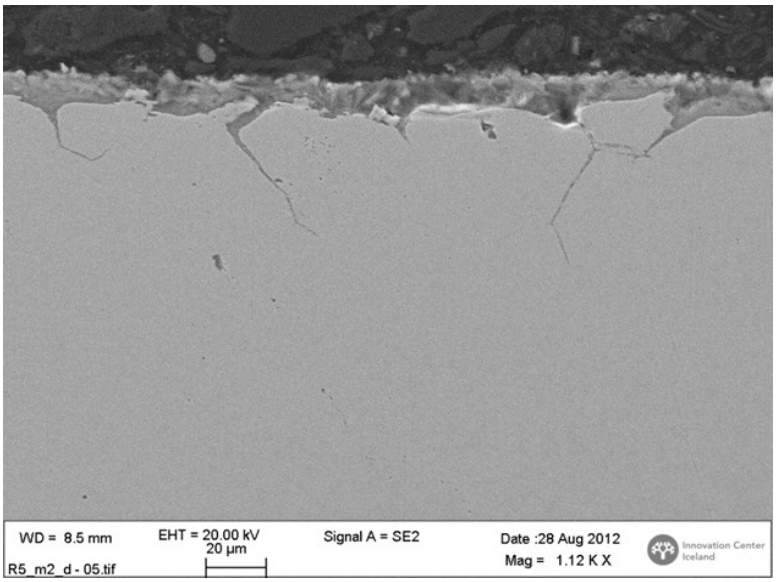

Figure 2. Intergranular stress corrosion cracks in the S31254 material from heat exchanger test

The SCC cracks formed both under a chromium oxide surface film rich in S and under the Fe and S rich deposits. The S32707 was reported to show the best results in the microstructural analysis but the authors stated that it is not recommended by the manufacturer for service at temperatures above 250°C for prolonged periods due to possible microstructural changes. Large pits and cracks were seen in the microstructure of the N08028 after the tests, thus the N08028 was not considered as a potential candidate for use in the IDDP-1 superheated steam at these temperatures. The titanium alloy R50400 was effected by both corrosion and cracking which was not unexpected since pure titanium is not particularly resistant to HCl acid. Fluoride (F) was detected in the corrosion product of the R50400 pipe but it is well known that titanium is particularly prone to corrosion in the presence of fluorides, even the small concentration of HF gas in the IDDP-1 steam seems, therefore, to have an effect. The Pd added titanium alloy R52400 pipe performed very well; there was little evidence of corrosion or cracking. No evidence of hydrogen embrittlement was observed for the R52400 which can be a concern in environments with temperature above 80°C and with low pH value. The nickel based alloy N08825 was affected by both corrosion and cracking while the higher alloyed N06625 showed little signs of pitting corrosion or cracking. The N06625 was reported to have performed the best along with the titanium alloy R52400 of the materials tested in the heat exchanger experiment. The results from the heat exchanger experiment show that the construction of a heat exchanger for direct use of the IDDP-1 steam appears to be a possibility as regards to corrosion and possibly also as regards to scaling in the tubes (Karlsdottir et al, Nace 2014).

A corrosion testing experiment was also setup at the IDDP-1 site to investigate the corrosion behavior of various materials in direct contact to the superheated geothermal steam (Karlsdottir et al, Geothermics accepted 2014). The specimens were exposed to the steam at 360°C for a total of 113 days. Both stressed (U-bend) and unstressed (coupons) specimens were tested to investigate the uniform corrosion rate and the susceptibility to SCC of the metals in the IDDP-1 environment. The samples that were tested are shown in Table 2

Table 2. Materials tested in IDDP-1.

		Yield str.	Blank	A5	Blank	C%	Si%	Mn%	Cr%	Ni%	Mo%	Ti%	N%	Fe%	Other
	Material	[Mpa]	[Mpa]	[%]	[%]										
Carbon	K55	379-552	379	>19	30	nd	nd	nd	nd	nd	nd	nd		Bal	
Steel	TN95	655-759	691	nd	20	nd	nd	nd	1	nd	0,45	nd		Bal	
	P265GH	>265	nd	>15	26	0,2	0,4	0,8-1,4	0,3	0,3	0,08	0,3	0,02	Bal	Al<0,02; CU=0,3; Nb=0,02; V=0,02
	13 Cr	552-655	592	>15	26				13					Bal	
Stainless	S31254	>310	339	>35	56	0,02	0,8	1	20	18	6,2	nd	0,2	Bal	Cu=0,7
Steel	S32507	>530	641	>20	41	0,02		<1,2	25	7	4		0,27		
	S32707	>700	nd	>25	nd	0,03	0,5	1,5	27	6,5	4,8	nd	0,4	Bal	C0=1,0
	N08028	>760	837	>11	19	0,02	0,6	2	27	31	3,5	nd		Bal	Cu=1
Nickel	N08825	440	380	>35	42	0,05	0,5	1	19,5-23,5	38-46	2,5-3,5	0,6-1,2		<22	
Alloys	N06625	490	453	>49	55	0,1	0,5	0,5	20-23	60-65	8,0-10,0	0,4		<5	
Titanium	R50400	275-410	nd	>20	nd	0,08	nd	nd	nd	nd	nd	99,2	0,03	0,3	H=0,015; O=0,25
	R52400	>276	nd	nd	nd	0,08	nd	nd	nd	nd	nd	99	0,03	0,3	H=0,015; O=0,25; Pd=0,2

Severe scaling occurred during the testing and the average corrosion rate was extremely low; < 0.1 mm/year for all the metals tested. This was explained by combined effect of the high superheat in the steam preventing any condensation and the protectiveness of the silica scale on the samples. Despite the low corrosion rate the most corrosion resistant materials tested, the N06625 and the R52400 showed indications of localized corrosion damages after the test. This indicates that despite the covering of the silica the IDDP-1 superheated steam promotes localized corrosion and cracking. The API TN95 and K55 carbon steel casing materials were also tested and were reported to have the highest corrosion rates among the materials tested. Pitting corrosion was also evident, more in the TN95 than in the K55 sample but no cracking occurred for either sample. Thus the low amount of chromium (Cr) 1wt% and molybdenum (Mo) 0.5wt% in TN95 does not seem to help in preventing localized corrosion. Under-deposit corrosion was also found on some of the materials tested including the N08028. The UNS N06255 nickel based alloy used in sour and high temperature oil and gas wells shows pitting but no sign of crack formation. No damages were reported due to hydrogen embrittlement. For the stressed specimens only the low alloy stainless steels S30403, S31603 and S31803 had SCC cracks after the test, figure 3 shows the cross-section view of the S30403 U-bend specimen stressed to 12.5% true strain.

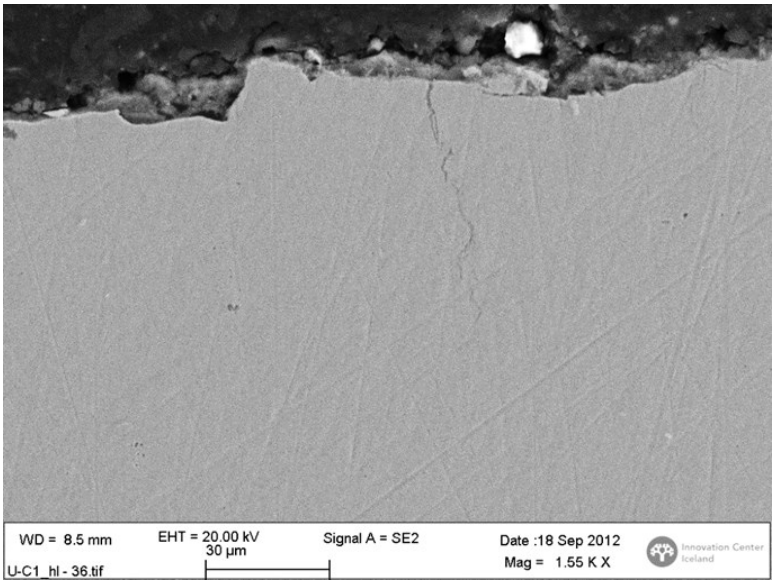

Figure 3. Cross-section view of the S30403 U-bend specimen stressed to 12.5% true strain

Of the specimen tested the austenitic stainless steels UNS S31254 and N08904; the duplex stainless steels UNS S32750, S32707; and the nickel alloy UNS N06255 were described to be the most promising materials in the study (Karlsdottir et al, Geothermics accepted 2014).

In the in-situ erosion-corrosion experiment the IDDP-1 superheated steam (at 320-350°C and 12-13 bar) at high velocity (~100-120 m/s) caused erosion-corrosion in a 3.4 mm thick carbon steel pipe bend, such that it started leaking after only 14 days of testing (Karlsdottir et al, Geothermics 2014). The dry superheated steam was supersaturated with silica which caused trouble in running the test. After three weeks of testing the test was stopped due to extensive silica scaling which closed the inlet pipe. Carbon steel pipe with a larger pipe radius (~50 m/s) was also tested for a comparison. The pipe experienced erosion-corrosion indicated by decrease in the wall thickness and corrosion pits. Thus for utilization of the superheated steam care must be taken in the selection of flow

rate for the pipelines and the equipment. Small erosion damages were in the bends of the stainless steels S31254, S32707 and the N08028 which were also tested with velocity of around 100 m/s. Figure 4 shows an example of these, in the S32707.

Thin hard silica scale covered these damages which stopped further erosion damages. Cracks or very narrow pits also formed in the N08028 and S32707 stainless steel pipes but not in the S31254 pipe. Figure 5 shows a crack or a narrow pit in the inner surface of the S32707 stainless steel pipe.

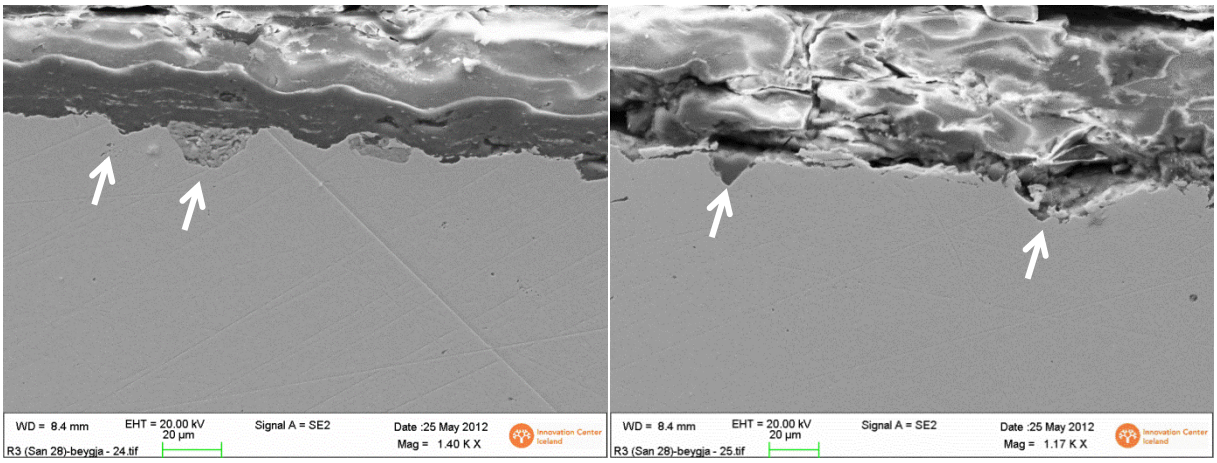

Figure 4. Small erosion damage found in the S32707 duplex stainless steel.

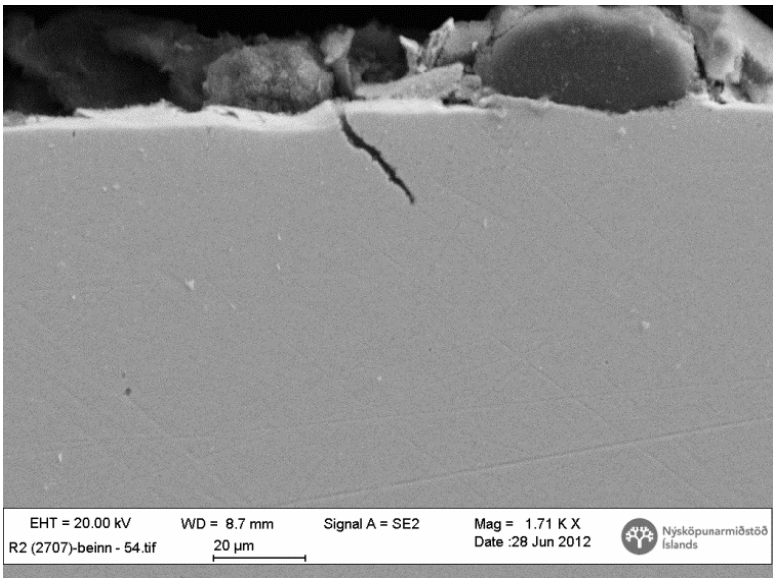

Figure 5. Crack in the inner surface of the S32707 duplex stainless steel

The corrosion damages were described to be possibly caused by condensation of the water vapor and acid gases which could have diffused through the silica scale to the metal surface due to the pipes not be insulated at the outside and the insulating effect of the silica layer. But because similar corrosion damages were observed in the corrosion coupon testing in the IDDP-1 steam where condensation is very unlikely the enhanced corrosion effect of HCl, HF and possibly gaseous sulfur at higher temperatures (>300°C) cannot be ruled out (Karlsdottir et al, Geothermics 2014).

5. MATERIALS SELECTION AND CONCERNS FOR FUTURE IDDP WELL DESIGN.

Materials tested in the IDDP-1 steam were of four types, low alloy carbon steels, Stainless steels, Nickel based alloys and Titanium alloys. Not all of the steel types were tested in all test setups described in this paper, relevant material types were selected for each setup. Based on the findings from testing in the IDDP-1 steam together with findings the authors have got from other high temperature geothermal wells, following remarks regarding material selection can be made

Carbon Steel. Carbon steels or low alloy steels are the dominating material used for geothermal wells, for pipes and equipment above ground. In general this material has shown to be both economic and long lasting for the geothermal wells mainly used is API K55 grade. API TN95 has been used were higher strength and creep resistance is required. In general carbon steel has shown long lasting properties in geothermal use, but with the exception were the pH level has dropped and when condition has allowed condensate on the material surface. This is also the experience from the corrosion testing experiment where the carbon steel

specimens in direct contact to the superheated IDDP-1 steam at 360°C measured with corrosion rates below 0.1 mm/year. The low amount of additives in the TN95 does not help in corrosion resistance and the pitting corrosion was actually quite extensive for the TN95 in the IDDP-1 steam testing (Karlsdottir et al 2014). Above ground in diverse equipment the carbon steels has suffered from erosion-corrosion problems as can be seen from the erosion-corrosion test with the IDDP-1 steam (Karlsdottir NACE2014). Carbon steels are although good candidate for use in controlled conditions where condensate can be avoided and where erosion-corrosion is not a problem.

Stainless Steels. Stainless steels have a wide use in applications in the geothermal power production, mainly in diverse equipment's and piping. They have not been used in geothermal wells in Iceland. In general this material group can be divided into two major groups when used in geothermal fields, Austenitic stainless steels and Duplex stainless steels. Both groups have long history of use in geothermal fields in Iceland and have been proven as a proper material for wide variety of applications. For higher temperatures above 250°C it is not recommended to use the Duplex type due to its structural change, shown as change in the material tensile strength above that temperature. Testing in IDDP-1 steam has shown erosion-corrosion damage for S32707 (Karlsdottir et al 2014). Below 250°C the Duplex types S32507 and S32707 have both excellent resistance to corrosion and during testing in IDDP-1 practically none problems in use were detected. The Austenitic types can in fact been divided into two types, the lower alloyed types AISI 304 and AISI 316 and then the higher alloyed S31254, N08904 and N08028. The 304/316 types has shown to have limited resistance to corrosion and suffer from diverse corrosion forms in high temperature steam such as in IDDP-1. These results indicate limited use for these lower alloyed austenitic stainless steels in such conditions. The higher alloyed austenitic steels have shown better performance, especially the S31254 type. In one instance, the heat exchanger test, evidence of intergranular stress corrosion cracking was found (Karlsdottir, Nace 2014) in this material which was the only case, where problems were found with the S31254 material. In general the stainless steels especially the higher austenitic grades can be considered as good candidates materials for use in high temperature geothermal steam with temperatures above 250°C.

Nickel based alloys. Nickel based alloys has long tradition for being good materials for diverse harsh environments. These alloys have not been widely used in geothermal steam in Iceland so limited practical experience has been obtained. Testing in IDDP-1 high temperature geothermal steam where N08825 and N06625 nickel alloys were tested revealed that even these corrosion resistant materials have some concerns in use. The N08825 type suffered from corrosion pitting after testing in the IDDP-1 steam and even the higher alloyed N06625 type showed evidence of small corrosion damage in the form of narrow pitting in the heat exchanger test, the corrosion testing in the IDDP-1 steam at 360°C and in the wet scrubbing experiments. Further testing of these materials is needed for better clarification of their use in high temperature geothermal steam.

Titanium alloys. Two types of titanium alloys were tested in the IDDP-1 steam. Type R50400 and R52400. The R50400 type suffered from narrow pitting but the R52400 type alloyed with Pd showed more resistance. There are some concern with regard to HF containing steam like in IDDP-1 for the R50400 type were the R50400 has more resistance to HF containing steam due to the Pd alloying. In general the concern of strength for Titanium alloys can limit their use at higher temperatures (>400°C). The possibility of hydrogen embrittlement is also concern for low pH environment at higher temperatures (>80°C).

The second IDDP well IDDP-2 is already planned and designed for the Reykjanes peninsula geothermal field. The drilling has not yet been decided by the responsible partner HS Orka, the energy company operating at the Reykjanes geothermal field. This IDDP-2 well is designed based on the findings from IDDP-1 and will be built with the same materials. Due to findings in the IDDP-1 work and as well other sour wells (Karlsdottir.S 2013) (Karlsdottir. S 2012) in Iceland the liner has been designed to be closed for the first 200 m and then slotted, this is to avoid the risk for condensate inside the production casing. The carbon steels used has shown to be reliable with regard to corrosion if condensate can be avoided. By having the liner un-slotted for the upper most 200 m the risk for condensate and mixing inside the production casing will be minimized, and thereby give more protection against corrosion inside the production casing. The production casing reaching down to over 2000 m will be cemented to bottom and therefore risk for outside corrosion on the outer part of the casing will be minimized. Liner and product casings are designed to be from API K55, Anchor casing from API T95 for the uppermost part and API K55 for the lower part, the API T95 chosen for its creep strength at higher temperatures. These materials will be in direct contact with the steam and are therefore in risk for corrosion and scaling due to the contents of the steam, mainly non-condensable gasses and chlorides. Other casings like intermediate and surface casings will be used but they are not in direct contact with the steam and therefore not subjected to this paper. The design for the IDDP-3 has not yet been started.

6. CONCLUSION.

The material testing in the IDDP-1 pilot testing has been reviewed and the main results reported and discussed to evaluate the possibilities in utilizing superheated steam from a deep high temperature/pressure geothermal well (IDDP-1). The main results are that the corrosion rate in the IDDP-1 superheated steam where silica scaling is present is below 0.1 mm/year which are good news. On the down site the results from the tests indicate that the superheated steam nevertheless promotes localized corrosion in some extent in all the materials tested ranging from carbon steel, stainless steels to nickel based and titanium alloys. Of the materials tested the N06625 based nickel alloy and the R52400 titanium alloy seem to be the least effected by corrosion. The austenitic stainless steel S31254 is a promising candidate material and performs well in the majority of the test except in the heat exchanger experiment where intergranular SCC was observed. Main concern for the future IDDP-2 and IDDP-3 wells, both the well, pipes and equipments will be to avoid condensate and to effective scrubbing of gasses, chlorides and other harmful contents of the steam

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