

Borehole Integrity of Austenitized and Annealed Pipe Steels Suitable for Carbon Capture and Storage (CCS)

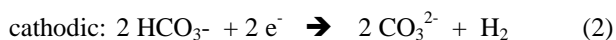
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Abstract—Properties of pipe steels for CCS (carbon capture and storage) technology require resistance against the corrosive environment of a potential CCS-site (heat, pressure, salinity of the aquifer, CO₂-partial pressure). The influence of austenitizing in heat treatment routines of two different injection pipe steels (1.4034, X46Cr13 and 1.4021, X20Cr13) was evaluated. Steel coupons were austenitized at different temperatures (900–1050 °C) for different lengths of time (30–90 min) before quenching and annealing prior to long term corrosion experiments (60 °C, 100 bar, artificial brine close to a CCS-site in the Northern German Basin, Germany). In general, fewer pits are found on X46Cr13. Comparing steels with 13% chromium each the higher carbon content of X46Cr13 (0.46% C) results in a lower number of pits compared to X20Cr13 (0.20% C). It is found that neither the carbon content of the steels nor austenitizing temperature has much influence, but local corrosion behaviour is most susceptible towards austenitizing time.

Index Terms—Corrosion, CCS, carbon storage, aquifer, austenitizing.

I. INTRODUCTION

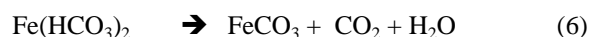
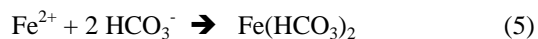
Engineering a geological on-shore saline aquifer CCS-site (CCS Carbon Capture and Storage [1]–[3]) corrosion of injection pipe steels may become an issue when emission gasses, e.g. from combustion processes of power plants, are compressed into deep geological layers [4]–[12]. CO₂-corrosion is sensitively dependent on alloy composition, contamination of alloy and media, environmental conditions like temperature, CO₂ partial pressure, flow conditions and protective corrosion scales [6]–[8], [13]–[27]. Because the solubility of FeCO₃ in water is low ($p_{Ksp} = 10.54$ at 25 °C [28], [29]) generally steels, that are exposed to CO₂-environment, precipitate slow growing surface layers mainly comprised of FeCO₃ (siderite) [4], [8], [30], which is also found in pits of locally corroded samples [21], [23]:



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Possibly the initial formation of Fe(OH)₂ [7], [30] leads to an increase of the local pH near the hydroxide film resulting in the precipitation of an internal and external ferrous carbonate film, Han *et al.* [29]. Localized corrosion may then start especially at grain boundaries when the ferrous hydroxide film is locally damaged. The ferrous carbonate film is exposed to the brine, dissolves and is locally depassivated. As a consequence the carbonate film detaches (Fig. 1).

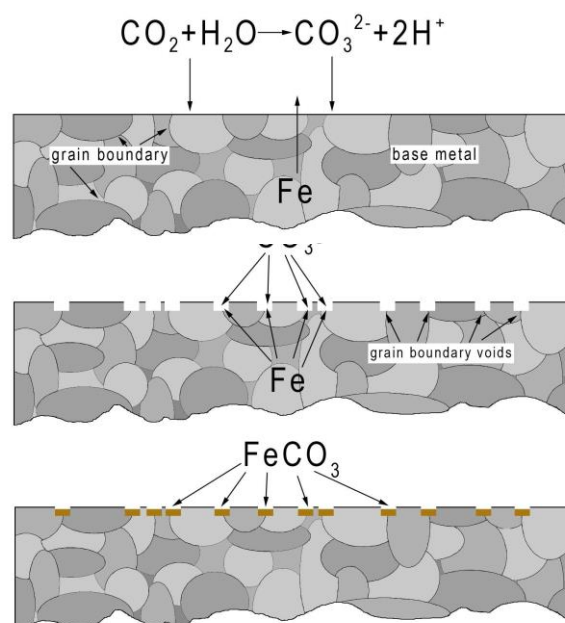


Fig. 1. Pit precipitation on steels initiated at grain boundaries (schematic).

The influence of heat treatment, that is: temperature and time of austenitizing, cooling rate as well as temperature and time of annealing, has been shown by various authors. The presence and amount of retained austenite as a microstructural component resulting from the heat treatments applied has a beneficial effect on the pitting corrosion resistance of 13%-chromium steels (13CrNiMo) [31]. A higher Ni and Cr content in the heat treated steels improve the corrosion resistance [31], [32]. Hou *et al.* introduce a method of empirically calculating the influence of alloying elements in heat treated steels [33]. Cvijović and G. Radenković showed that the corrosion resistance of duplex steels with chromium contents even as high as 22–27% varied with solidification mode and annealing condition [34]. In general raising the annealing temperature lowers the pitting potential of lean duplex stainless steels [34]–[36]. The lowest potentials, corresponding to the transition from metastable to stable

pitting, are observed for annealing at 900 °C while a maximum improvement of corrosion stability can be achieved by annealing at 1200 °C [34]. The better corrosion resistance of martensitic stainless steels with 13% Cr at higher austenitizing temperature (980-1050 °C) is related to the dissolution of carbides [36]-[38]. The precipitation of Cr-rich $M_{23}C_6$ and M_7C_{12} carbides reduced the resistance of passive film and pitting corrosion [36] and has high impact on mechanical properties due to secondary hardening [37]. The influence of heat treatment on the microstructure and mechanical properties is well known [36], [39], [40]. However for C-Mn (carbon) steels in a H_2S -containing NaCl solution the martensitic microstructure has the highest corrosion rate up to two orders of magnitude higher than ferritic or ferritic-bainitic microstructures due to the fact that martensitic grain boundaries are more reactive [41].

II. EXPERIMENTAL PROCEDURE

The steel qualities for laboratory experiments X20Cr13 (1.4021, AISI 420 J) and X46Cr13 (1.4034, AISI 420 C) are used as injection pipe. Exposure tests in CO_2 -saturated aquifer brine were carried out using samples made of thermally treated specimen of the steels with 8 mm thickness, 20 mm width, 50 mm length. Austenitizing prior to exposure was done at 950 °C, 1000 °C and 1050 °C for 30 min, 60 min and 90 min. Following usual heat treatment protocols the specimen were then annealed at 650 °C for 30 min to gain martensitic microstructure with sufficient hardness and toughness. A hole of 3.9 mm diameter was used for sample positioning. Samples of each base metal were positioned within the vapour phase and within the liquid phase. The brine (as known to be similar to the Stuttgart Aquifer [42]: Ca^{2+} : 1760 mg/L, K^{2+} : 430 mg/L, Mg^{2+} : 1270 mg/L, Na^{2+} : 90,100 mg/L, Cl^- : 143,300 mg/L, SO_4^{2-} : 3600 mg/L, HCO_3^- : 40 mg/L) was synthesized in a strictly orderly way to avoid precipitation of salts and carbonates. Flow control (3 NL/h) of the technical CO_2 (purity 99,995 vol.-%) into the brine at ambient pressure was done by a capillary meter GDX600_man by QCAL Messtechnik GmbH, Munic. The exposure of the samples between 700 h to 4000 h was disposed in reaction vessels according to the conditions at the geological site at 60 °C at ambient pressure – each material in a separated reaction vessel [21] (Fig. 2).



Fig. 2. Reaction vessels and experimental set up.

Before corrosion tests the surfaces of the steels were activated by grinding with SiC-Paper down to 120 μm under water. After the corrosion tests, the samples were cut partly for scale analysis with the corrosion layer and prepared partly for kinetic analysis after the scale was etched. Descaling of

the samples was performed by exposure to 37% HCl. Then parts of the samples were embedded in a cold resin (Epoxicure, Buehler), cut and polished first with SiC-Paper from 180 μm to 1200 μm under water and then finished with diamond paste 6 μm and 1 μm .

Different light optical and electron microscopy techniques were performed on specimens to investigate the layer structures and morphology of the samples. X-ray diffraction was carried out in a URD-6 (Seifert-FPM) with $CoK\alpha$ -radiation with an automatic slit adjustment, step 0.03 ° and count 5 sec. Phase analysis was performed by matching peak positions automatically with PDF-2 (2005) powder patterns. Mainly structures that were likely to precipitate from the steels were chosen of the ICSD and refined to fit the raw-data-files using POWDERCELL 2.4 [43] and AUTOQUAN ® by Seifert FPM. To characterise the pitting corrosion, 3-D-images were realized by the double optical system Microprof TTV by FRT. Kinetics of the corrosion were determined by the corrosion rates which were calculated via mass change of the samples before and after corrosion testing according to DIN 50 905 part 1-4 and using the semi-automatic analyzing program Analysis Docu ax-4 by Aquinto.

III. RESULTS AND DISCUSSION

The inhomogeneous corrosion layer specimen surfaces after 700 h, 2000 h and 4000 h of exposure to CO_2 -saturated saline aquifer water on consists of either a non-consistent carbonate layer or carbon precipitates covering pits precipitated on the samples surface. X46Cr13 and X20Cr13 steels are known for susceptibility towards local corrosion. Therefore pitting will be taken into account more closely-even if thin surface corrosion layers occurred after long exposure times (Fig. 3).

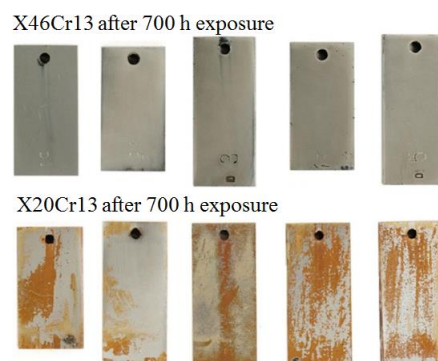


Fig. 3. Sample surfaces after 700 h of exposure.

As a measure of quality the hardness was analyzed prior to exposure. Longer austenitizing times lead to lower hardness and higher austenitizing temperatures result in lower hardness due to grain growth processes at longer austenization time at higher temperatures. Larger grains within the microstructure result in longer dislocation paths allowing for plastic deformation. In general the hardness of X46Cr13 was higher by a factor of 2 compared to X20Cr13 due to the higher C-content and accompanying carbide precipitation. Reason is the precipitation of iron and chromium carbides that act as dislocation barriers reducing

plastic deformation.

To evaluate the influence of the heat treatment on the surface and local corrosion behavior the samples were examined via light optical methods to predict the number of pits and pit depths. Kinetics was obtained via weight loss according to DIN 50 905 after exposure to the CO₂-saturated aquifer water. Fig. 4 demonstrates that the surface corrosion behavior of X46Cr13 and X20Cr13 is comparable after long exposure times. Here the results of all heat treatment parameters have been combined to evaluate the influence of the carbon content. Even if X46Cr13 has a slightly higher corrosion rate after only 700 h of exposure the corrosion rate of both steel qualities is approximately 0.01 mm/year after 4000 h of exposure.

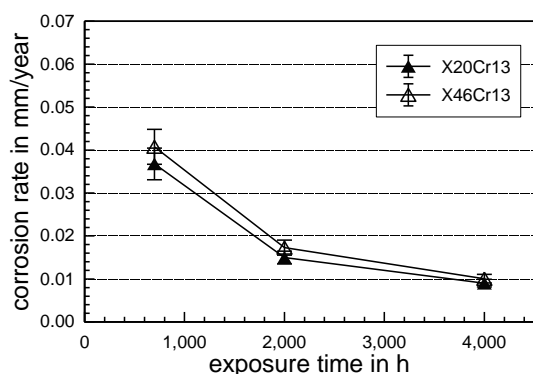


Fig. 4. Combined surface corrosion rates of X46Cr13 and X20Cr13 without regard to austenitizing.

Similar results are obtained when focusing on the austenitizing temperature; after 4000h of exposure. The corrosion rate is about 0.01 mm/year no regard to the temperatures 950 °C, 1000 °C or 1050 °C (Fig. 5).

The influence of exposure time is more significant as demonstrated in Fig. 6. Austenitizing for only 30 min at either one of the three different austenitizing temperatures gives lowest corrosion rates after short exposure times of X20Cr13 and X46Cr13 (700 h) to CO₂-saturated saline aquifer and after long exposure times (4000 h). The corrosion rate is approximately 0.005 mm/year and therefore lower by a factor of 2.

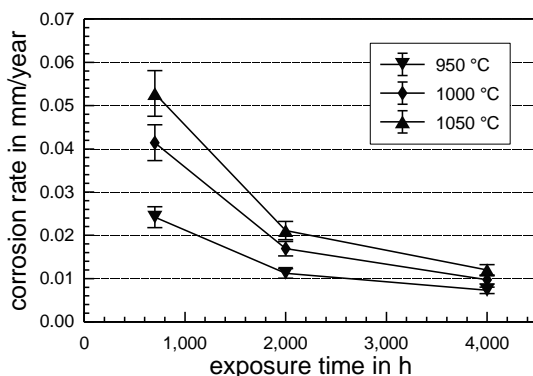


Fig. 5. Surface corrosion rate of X46Cr13 and X20Cr13 combined as a function of austenitizing temperature after 4000 h of exposure to CCS environment.

In general, the corrosion resistance increases with shorter austenitizing time and lower austenitizing temperature. The austenitizing time has the greatest influence on the surface

corrosion rate (Fig. 6), which scales with the elongation of austenitizing, but is neglectable when evaluating local corrosion phenomena [44]. Specimens heated to 950 °C and annealed for 30 minutes exhibit the lowest corrosion rates. The highest corrosion rate is found for X20Cr13 austenitized at 1050 °C for 60 minutes. After 700 h the corrosion rates have the highest values, but as time proceeds the rates decrease.

Comparing local corrosion of X20Cr13 and X46Cr13 Fig. 7 shows that X20Cr13 shows a better pit corrosion resistance. X20Cr13 exhibits an average of 3508814 pits per m², whereas the X46Cr13 averages 9622 pits per m² after 700 h of exposure. Here for both steel qualities austenitizing at 1000 °C seems to give better results as austenitizing at 950 °C or 1050 °C.

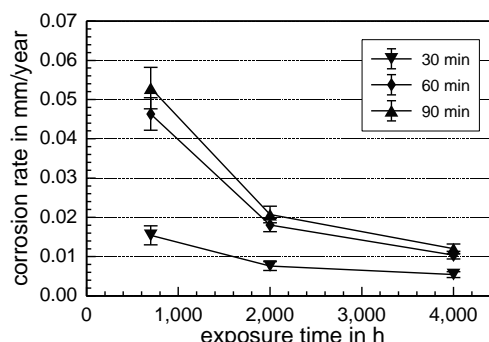
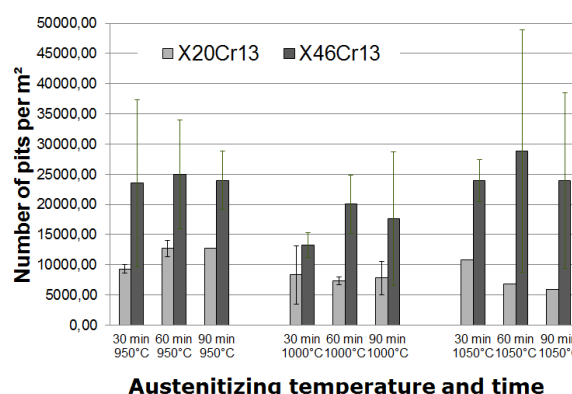


Fig. 6. Surface corrosion rate of X46Cr13 and X20Cr13 combined as a function of austenitizing time after 4000 h of exposure to CCS environment.



Austenitizing temperature and time

Fig. 7. Number of pits on X20Cr13 and X46Cr13 after applying different austenitizing routines and annealing at 650 °C for 30 min. prior to exposure to CO₂-saturated saline aquifer water for 700 h [45].

In the beginning the corrosion rates are significantly higher at higher austenitizing temperatures and longer austenitizing times, but after 4000 h the corrosion rates are of not much difference. Most likely the coarse grain structure is corrosively attacked at the grain boundaries, but once a sufficient corrosion layer has precipitated the mutual diffusion of ionic species towards the metal surface and into the surface is significantly slowed down with increasing thickness of the corrosion layer. In addition the grain growth process is limited at shorter austenitizing temperature giving the equilibration as little time as possible with additionally short austenitizing time. If grain growth is slow the small grain sizes within the microstructure will result in lower corrosion rates.

X20Cr13 also shows a better local corrosion resistance compared to X46Cr13 when taking into account pit diameters.

The average pit on X46Cr13 (diameter average = 249 μm) is 5 times larger as the average pit on the X20Cr13 (49 μm) [46]. Most pits on both steel qualities have a diameter of 51 μm to 100 μm after 700 h of exposure – with the number of pits of X46Cr13 higher than X20Cr13 by a factor of 3 (Fig. 8).

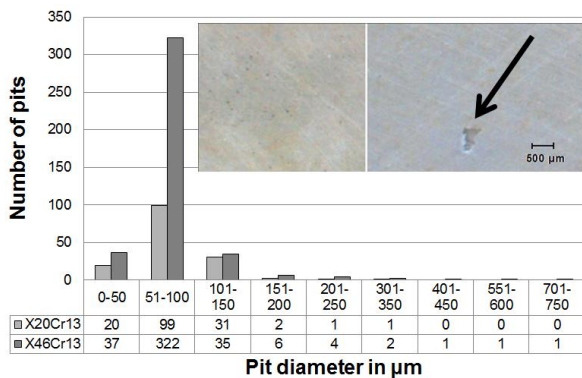


Fig. 8. Number of pits precipitated on X20Cr13 and X46Cr13 divided in diameter groups after applying different austenitizing routines and annealing at 650 °C for 30 min. prior to exposure to CO₂-saturated saline aquifer water for 700 h.

Even if the maximum pit diameter of pits precipitated on X46Cr13 succeed those precipitated on X20Cr13 by a factor of 3.5 (Fig. 9) for steel coupons austenitized at 950 °C for 90 minutes the average pit diameter of both steel qualities does not differ significantly. Furthermore there is no significant influence on the austenitizing routines prior to exposure to CCS environment. The average pit diameter does not succeed 100 μm after 700 h of exposure. Notice, critical parameter to assess the influence of pit precipitation is preferably not the diameter, but depth of pits. But, after 700 h to 4000 h the depth of pits were shown to be quite comparable and these will be evaluated more closely in future.

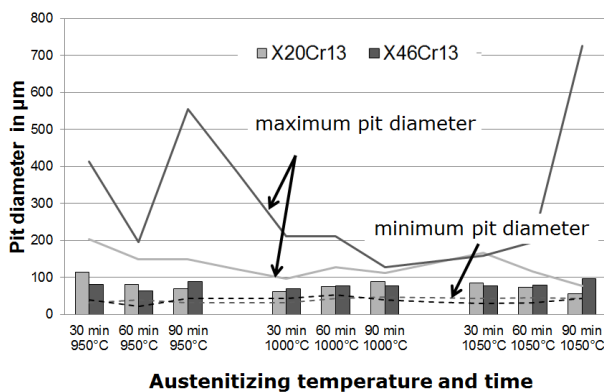


Fig. 9. Diameter of pits precipitated on X20Cr13 and X46Cr13 after applying different austenitizing routines and annealing at 650 °C for 30 min. prior to exposure to CO₂-saturated saline aquifer water for 700 h.

Fig. 10 shows results from combined DoE-Analysis performed after Klein [45], [46] with respect to alloy composition and carbon content, austenitizing time and austenitizing temperature. This method does not allow for the evaluation of dependent parameters such as the combination of both, carbon content and austenitizing time. Still, the results prove that the corrosion resistance regarding surface corrosion is only dependent on the austenitizing time, but neither the austenitizing temperature nor the carbon content of the base material [23].

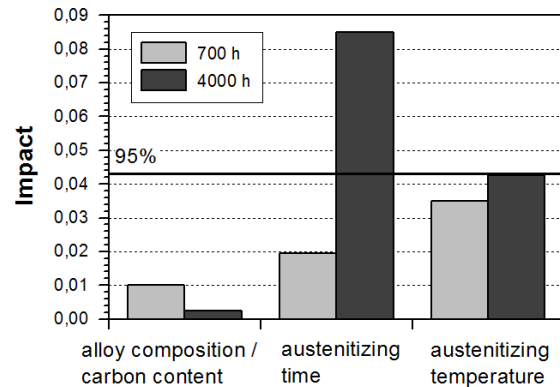


Fig. 10. The impact of the experimental parameters on the surface corrosion rate after 700 h and 4000 h of exposure time with 95%-confidence interval.

The fact that the carbon content exhibits the biggest impact on the local corrosion behaviour is shown in the results of the DoE analysis revealed by Fig. 11. But, since none of the parameters are within the liability regime the results here prove a tendency but do not show significant dependence of carbon content on the local corrosion behavior [45].

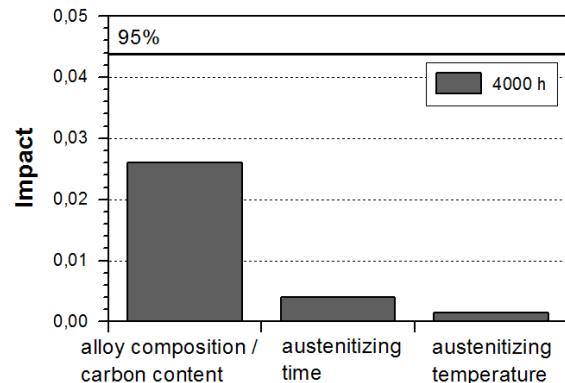


Fig. 11. Impact of the experimental parameters on the local corrosion behaviour of X20Cr13 and X46Cr13 after 4000 h of exposure time with 95%-confidence interval.

The austenitizing time is the only parameter during heat treatment procedure that shows significant influence on surface corrosion behavior of 13%-chromium steels. Local corrosion behavior is not influenced by either austenitizing time or temperature or alloy composition. Most likely pit precipitation is a phenomenon of local elemental decomposition, depleting the alloy matrix of chromium leading to localized degradation and pit growth. Still, in order to prevent severe corrosion during CCS steels should be austenitized as short as possible and pit depth should always been taken into account, even if pit diameters remain small after long exposure times.

IV. CONCLUSION

When engineering a CCS-site CO₂ is injected into deep geological layer supposedly in its liquid or supercritical phase. As a worst case scenario during carbon capture and storage is that intermissions of CO₂ injection may lead to a rising the water level within the injection pipe. As a consequence of this three-phase boundary (liquid brine, supercritical or gaseous CO₂ and solid injection pipe steel) corrosion products precipitate and pits are formed. In general, the shorter

austenitizing times and lower austenitizing temperatures of the steel qualities prior to exposure to CCS environment exhibit the lowest corrosion rates. Still, the austenitizing time has no significant impact on the number of pit and pit sizes. The following facts could be summarized acting as an aid to heat treatment of steels used in CCS-technology. Most important finding are:

- 1) Shorter austenitizing time at lower austenitizing temperature results in better corrosion resistance regarding surface corrosion. Especially the austenitizing time has a significant impact.
- 2) A higher carbon content of the steels (X20Cr13 < X46Cr13) increases the number of pits.
- 3) After 4000 h of exposure there is no difference in surface corrosion rates comparing X20Cr13 and X46Cr13
- 4) The alloy composition as well as the austenitizing procedure has no impact on pit diameters.

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