

IN-SITU STRESS-STRAIN TESTING OF STEELS IN CO₂-CONTAINING SALINE SOLUTIONS

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Abstract: In CCS environment (carbon capture and storage) pipes and in geothermal power plants the materials used in pumps are loaded cyclically and exposed constantly to the highly corrosive hot thermal water. The lifetime reduction of AISI630 (X5CrNiCuNb16-4, 1.4542) is demonstrated in in-situ-laboratory experiments ($T=60$ °C, geothermal brine: Stuttgart Aquifer flow rate: 9 l/h, CO₂). S-N plots, micrographic-, phase-, fractographic- and surface analysis were applied to obtain sustainable information on the corrosion fatigue behavior.

Keywords: CORROSION FATIGUE, HIGH CYCLE FATIGUE, STEEL, CCS, CO₂-STORAGE, GEOTHERMAL ENERGY

1. Introduction

Materials in geothermal power plants are loaded cyclically under pressure and exposed constantly to the highly corrosive hot thermal water (up to ca. 200 °C, ca. 100 bar, ca. 20 % salinity of the geothermal water) where fluid properties may differ strongly [1]. This leads to corrosion fatigue and thus inevitably to the reduction of the lifetime of these components. The influence of frequency, temperature and chloride concentration on the corrosion fatigue behaviour is very well known in literature [2]. In general corrosion processes with or without applied mechanical stress are enhanced, especially in steels with low chromium content [3], with the presence of chloride [4], hydrogen sulfide (H₂S) [5] and CO₂ [6]. The endurance limit [7] will decrease with increasing temperature, increasing mechanical load and decreasing pH for high alloyed steels. But increasing chromium content of steels as well as internal compressive stress in surface regions will increase the endurance limit [8].

This work was carried out to assess the influence of corrosive media on the mechanical behaviour of stainless steels such as AISI630 (X5CrNiCuNb16-4, 1.4542) in geothermal energy production.

2. Corrosion Chamber for Corrosion Fatigue Testing

The objective was to simulate in-situ conditions (temperature up to 100 °C, corrosive environment) of a material exposed to dynamic mechanical stress and corrosive gas- saturated saline aquifer environment, such as components in geothermal power plant. Highlight is the corrosion chamber fixed directly onto the sample leaving the resonant testing machine unaffected (figure 1). During mechanical stress-strain tests a magnetically driven gear pump (3) constantly pumps the corrosive media from the reservoir (4) to the corrosion- and temperature-resistant corrosion chamber (1) surrounding the test specimen (2). Heating is realized by two independent heating elements (5,6). The ratio of sample surface to volume of the corrosive media after DIN 50905 Part 1 (10 ml/cm²) is greater than required. The connecting of the chamber onto the specimen via clamping collar creates a force-fit process ensuring enough force to the corrosion chamber at high frequencies to keep it firmly on the test specimen (figure 2). The corrosion chamber is sealed in the area of restraint over O-rings made of Viton. In order not to impede the change in length occurring during the experiment, the corrosion chamber has a membrane as a motion-compensating element.

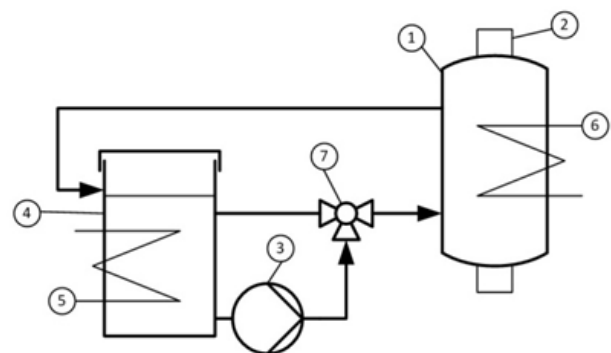


Figure 1: Schematic set-up of operating corrosion chamber for in-situ corrosion fatigue testing



Figure 2: Experimental set-up: corrosion chamber applied to resonance testing machine

The corrosion chamber is also equipped with measurement technique to gain electrochemical data during the mechanical tests. For measurement of the electrochemical potential a sensor (figure 2) is placed in the chamber. The sensor which is used is a silver-silver chloride electrode. Because of its method of construction this electrode is shock resistant and hence optimized for use inside the corrosion chamber under cyclically load. The silver wire (position 1) is fixed in a channel made of Teflon (position 2).



Figure 3: Silver-silver chloride sensor optimized for the corrosion chamber

3. Dynamic in-situ corrosion experiments at ambient pressure (HCF)

The corrosion fatigue strength of stainless steel with 16% chromium (1.4542, hardened and tempered with martensitic microstructure, surface roughness $R_z=4$) is examined in dynamic stress-strain tests ($R=-1$, ~ 30 Hz) in CO_2 -saturated aquifer (Stuttgart Aquifer [9]) at 60°C . Without corrosive environment the fatigue strength of the material (theoretically an infinite number of load cycles without failure) is and has a relatively smooth slope. The decrease of the fatigue limit line of 1.4542 samples with increasing number of cycles (Wöhler-exponent of $k = 3,59$) is much larger in corrosive environment than in air (tensile strength in air: 1078 MPa, largest number of cycles in corrosive environment ($0,6 \times 10^7$ at 200 MPa). As for 1.4034 [9] no typical fatigue strength of 1.4542 exists as shown in a log-log plot (figure 4). The coefficient of correlation $r^2 = 0,33$ from the regression is so small that doubt on the hypothesis of a (linear) relationship of the considered variables exist. In addition, the scattering range $TN = 1:34,4$ is disproportionately large.

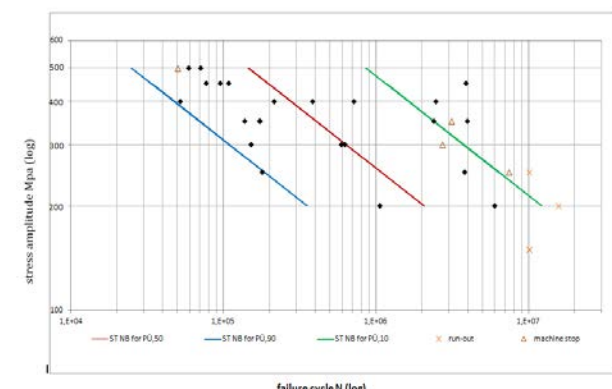


Figure 5: S-N-curve, corrosion chamber, sample and fracture surface 1.4542 exposed to flowing saline aquifer [10] and CO_2 .

Generally multiple cracks are found throughout the entire sample area and not only within the sample area that is mechanically loaded highest. Once the crack opens the crack-flank surfaces corrode continuously during cyclic load (figure 5). Crack propagation is then perpendicular to the direction of load. Localized corrosion (pits: 0.1 mm deep and 0.2 mm diameter) is accompanied with cracks, but not necessarily identified as the cause of crack initiation and failure under mechanical load.



Figure 4: Sample Surface after Testing.

4. Conclusion

- A highly flexible corrosion chamber allowing for electrochemical testing, O_2 -partial pressure or gas partial pressure measurement was designed to support stress-strain loaded corrosion fatigue experiments by enabling an in-situ corrosive environment which may be used up to 100°C at ambient pressure.
- The corrosion fatigue behaviour of AISI630 (X5CrNiCuNb16-4, 1.4542) is described by statistical crack initiation but characteristic crack propagation and fracture surfaces for one stress amplitude. A typical fatigue strength of the S-N-curve does not exist under CCS corrosive conditions. The fatigue strength of the material in non-corrosive conditions of under cyclic rotation of 620 MPa is reduced significantly due to corrosion. Note, that cyclic rotation does not accurately compare to compression-tension testing and that reference measurements are due in future experiments.

5. Acknowledgement

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7. Literature

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