Examples of testing methods focussed on evaluation of corrosion influence on steel structures

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Abstract. The paper presents methodologies and results of two case studies dealing with corrosion damage in steels. Specifically, it is concerned with corrosion fatigue and stress corrosion cracking. Description is given of preparing test specimens, special devices and loading frames that have been used to obtain significant experimental results. These are referred to influence of three different chemical solutions on the fatigue life of specimens taken from L485MB steel crude oil pipeline IKL. In the second part of the paper negative effects of stress corrosion cracking on the fracture toughness of structural steel CSN411353 were investigated and main results of this investigation are presented.

1. Introduction

It is well known that many civil engineering structures are subjected to degradation processes caused by various chemical substances. Degradation processes take place when protecting coats and technological anticorrosion barriers are damaged and water solutions can run into the structures. In many cases this is due to technological imperfections. These processes can also be of a natural origin and they cannot be avoided at the structure, so that it is necessary to account them in the stage of designing the structure. It is estimated that corrosion losses in industrially developed countries reach 3–5% of the gross domestic product. This means that in the Czech Republic we lose approximately 150 billions CZK per year [1].

The word corrosion is derived from the Latin word „corrodere“, which means to nibble, to gnaw away. The mechanism of rise and effectiveness of corrosion impacts are influenced by a number of factors which are dependent first of all on the aggressivity of the environment, then on the stress state of material, the quality of insulation, the state of the surface of material, etc. The main dividing of corrosion is on the basis of the principle of the corrosion process itself. There is a chemical, biological, and electrochemical corrosion [2]. According to its volumetric size and significance on the limitation of the life of a steel structure corrosion can be divided into general corrosion, pitting, stress corrosion cracking, intercrystalline, transcrystalline corrosion and selective corrosion [3]. The issue of corrosion is very important and still a timely subject which becomes a motive of many research projects.

This paper brings about two case studies which deal with corrosion problems of steels. In the first study a procedure and results of testing of the effect of corrosion on the fatigue resistance of steel are described. The motivation for this research were petrochemical industry demands for pipeline operational reliability. It is well known that separated water in crude oil can reduce steel fatigue life [4]. Our point of interest was a comparison of the fatigue properties of the concrete pipe steel in air, in crude oil, in separated water and in alkaline solution. The latter study aimed at the description of potentialities of a laboratory simulation of a very dangerous type of corrosion damage which is the stress corrosion.
It is usually associated with hydrogen embrittlement [5] which is very topical subject e.g. in the development of Tokamak fusion power plants and other specific parts of industries.

2. Corrosion fatigue

Owing to the fact that (i) operational loading of many civil engineering structures is not constant but it varies during operation, and moreover, (ii) the steel comes frequently into contact with aggressive environment, one of the factors which could reduce the life of a structure is corrosion fatigue [6]. A research of fatigue behaviour of steel in an aggressive environment appears therefore as very desirable and useful. Even though these fatigue tests should naturally be carried out at conditions close to real ones, especially as far as frequency of loading is concerned, it is often not possible to realize such tests in a real time. Therefore, in such cases the effect of the length of time of chemical action of the environment on corrosion damage is held back. However, it is advantageous to carry out at least current fatigue tests at resonant frequencies for the specimens used. These fatigue tests, of course, cannot represent the real resistance of material against corrosion fatigue damage but the results obtained can be used as informative data for indication of the relative fatigue resistance of the material. We can encounter corrosion fatigue at bridges when mechanically loaded steel components come into contact with saline or at transport systems, power engineering as well as chemical and petrochemical industry. As an example of the research into the effect of corrosion environment on the fatigue life of steel an experimental work is described hereunder on a pipe segment taken from crude oil pipeline IKL (Vohburg an der Donau – Nelahozeves) after it had been in operation for 13 years.

2.1. Corrosion fatigue tests

Fatigue tests of both types of specimens were carried out in zero-to-tension loading in detail described in [3] using a resonant-type electromagnetic machine with the nominal force capacity 100 kN. The resonance frequency was approximately 130 Hz, $F_{\text{max}}$ varied from 23.4 kN up to 39.9 kN. Reference tests of IKL specimens were realized in air whilst main fatigue tests were realized in liquid media: in crude oil and in water separated from the crude oil. Fatigue tests of MIT specimens were performed in 1N water alkaline solution of Na$_2$CO$_3$ with NaHCO$_3$ in the ratio 1:1 with a pH value of 9.325 [7].

A leak-proof chamber, developed in co-operation with RCP company Prague, was used for fatigue tests in liquid media. A section layout of the chamber is shown in figure 2 and a general view of the specimen with a chamber in the grips of the machine is presented in figure 3.
The number of cycles to fracture, \( N \), i.e. the life of a specimen, was determined as a number of stress cycles which the resonant type fatigue machine was able to make out without any changes in pre-set parameters. A relative crack size at the instant of automatic switching off the machine was \( 0.25–0.4 \times \) the width of a specimen in its smallest cross section. Fatigue tests in the water phase separated from crude oil were, to some extent, specific, because this environment is a residue of the separated water which is present in small amounts in distributed crude oil, say either in emulsified form or as dissolved (in very small amounts). Chemical composition of the separated water is shown in table 1. Before starting the fatigue tests the separated water was in contact with air in order to be saturated with oxygen and \( \text{CO}_2 \) in the temperature range \( 18^\circ \text{C}–23^\circ \text{C} \).

### Table 1. The results of analysis of the water phase.

| Units              | Magnitudes |
|--------------------|------------|
| pH                 | 7.0        |
| specific conductivity | mS.m\(^{-1}\) | 2000 |
| dissolved substances | g.dm\(^{-3}\) | 14   |
| neutralizing capacity (KNK\(_{4.5}\)) | mmol.dm\(^{-3}\) | 11   |
| \( \Sigma (\text{Ca}+\text{Mg}) \) | mmol.dm\(^{-3}\) | 22   |
| chlorides (Cl\(^-\)) | g.dm\(^{-3}\) | 8.1  |
| sulphates (SO\(_2^2\)-) | mg.dm\(^{-3}\) | 31   |
| oxygen (15–25°C)   | mg.dm\(^{-3}\) | 9.5–8.5 |

### 2.2. Results of fatigue tests

All results are presented in figure 4. Besides fatigue curves relating to IKL specimens there is also shown a fatigue curve for MIT specimens. The fatigue limit in zero-to-tension loading for IKL specimens is demonstrated by a horizontal dashed line. According to [8] it is taken as \( 0.6 \times \text{R}_m = 366 \text{ MPa} \).
Figure 4. Summarized results of fatigue tests.

It can be seen in the figure that on the top of the set of all experimental points there are green colour triangles corresponding to the crude oil and also dark blue colour diamonds corresponding to the alkaline solution which was applied for MIT specimens. At the bottom of the set of experimental points red colour diamonds are situated with a large distance from other experimental points. These red colour points are related to the separated water. Slightly under green colour triangles there are pink colour squares which represent results obtained in air. Respective fatigue curves were obtained by smoothing experimental points with power function between stress and number of cycles to fracture. The results show that in relation to fatigue properties of IKL steel in air the effect of crude oil appears as non-aggressive and the effect of alkaline solution on MIT steel appears similarly, i.e. non-aggressive. On the other hand the separated water exhibits a very negative effect on corrosion fatigue properties of IKL steel.

If the fatigue curve for air is considered as a reference curve, then, according to this diagram, the crude oil environment will not reduce the fatigue strength of the steel investigated up to $2 \times 10^6$ cycles. It is worthy to note that the slope of the fatigue curve for crude oil is greater than for air. As it is noticeable from the position of fatigue curve for the separated water in the diagram the presence of the separated water in crude oil leads to a considerable deterioration of fatigue properties of the steel under investigation.

If we express the fatigue curve by a power function of the type (1)

$$
\sigma_{max} = \frac{A}{(N_f)^b}
$$  \hspace{1cm} (1)

we shall get the following magnitudes of the constants $A$, $b$ (these parameters were determined by least square methods from experimental data) for various environments (table 2):

| Environment                  | Steel | $A$     | $b$   |
|------------------------------|-------|---------|-------|
| air (5 pc.)                  | IKL   | 1147.4  | 0.0813|
| crude oil (6 pc.)            | IKL   | 1803.1  | 0.1129|
| separated water (5 pc.)      | IKL   | 1188.3  | 0.0971|
| alkaline solut. (6 pc.)      | MIT   | 2098.3  | 0.1239|
If we identify the fatigue limit with the maximum stress in a cycle for the life \( N_f = 2 \times 10^6 \) cycles we shall receive the following most probable magnitudes of the fatigue limit in the zero-to-tension cycle:

- For IKL steel it is \( \sigma_f = 353 \) MPa for air, \( \sigma_f = 350 \) MPa for crude oil and \( \sigma_f = 290 \) MPa for separated water;
- For MIT steel it is \( \sigma_f = 348 \) MPa for alkaline solution.

2.3. Discussion

Experimental results showed that crude oil has no adverse effect on fatigue properties of IKL steel. This finding is in an abrupt contrast to the effect of separated water which shows corrosion aggressivity towards this steel. Corrosion aggressivity of the separated water is caused by its saturation with oxygen and by a high content of chlorides [9]. On the other hand the presence of acid carbonates in high concentrations reduces its aggressivity [10]. At crude oil conditions the separated water can contain only a limited amount of oxygen but at conditions of a long time storage of crude oil the separated water can saturate with air [11].

This environment can be considered to be the most aggressive among those which are really likely [12]. In other words: as a result of a low value of pH factor and of a high ratio \( [c(\text{Cl}) + c(\text{SO}_4)]/c(\text{HCO}_3) \) the separated water becomes an environment with a high chemical corrosion activity. The effect of crude oil on fatigue properties of the steel investigated is comparable to the effect of an inert environment for that it expresses itself by locking the surface to air oxygen (fatigue tests in air). The composition of the alkaline solution (its alkalinity and presence of the system \( \text{HCO}_3^- / \text{CO}_3^{2-} \)) leads to the passivation of the steel which results in a lower aggressivity of the solution.

3. Stress corrosion cracking

This part of our work was aimed at the application of fracture mechanics approach to assessment of the reliability of structures containing stress corrosion (SC) cracks. Stress corrosion cracking is very dangerous type of corrosion damage. It can be found in power industry, chemical industry and other constructions in chemical and petrochemical industry (pipelines, storage tanks, etc.). The main damaging factors are (i) formation of crack networks and (ii) material embrittlement. The synergy of these factors creates a big danger for the integrity of structures.

Stress corrosion cracking may occur by several mechanisms; one of them is the hydrogen mechanism leading to hydrogen embrittlement (HE). In the HE process hydrogen atoms from the environment enter the metal and diffuse in the lattice. They tend to be attracted to regions of high triaxial tensile stress where the metal structure is dilated. Thus, the hydrogen atoms are drawn to the regions ahead of crack tips or sharp notches that are under stress. They can occupy the interstitial sites in the metal lattice, as shown by neutron diffraction in many cases [13].

The main point of this research consists in an attempt to clear up a widely discussed problem of a higher degree of dangerousness of SC cracks than fatigue (F) cracks on the integrity of structures. Seeing that character of damage is diffusive it is natural to suppose that odd atoms coming from the aggressive corrosion environment diffuse to the structure of material at the crack tip. It is also likely that due to this mechanism the fracture toughness will be reduced in comparison with a „clean“ F crack.

The task of the work was therefore to compare both magnitudes of the fracture toughness and to elucidate eventual differences.

The procedure of work to fulfil the main aim was divided into four steps:
- choosing a suitable aggressive environment, test material and the shape of specimens,
- design and building up of a testing equipment,
- test methods and experimental work (including specimens with F cracks),
- evaluation and analysis the results obtained.

3.1. Methods

For generating SC cracks it was necessary to develop a special equipment (figure 5). The equipment ensured the initiation and growth of SC cracks.
Figure 5. A Photo with schematic arrangement of the SC crack generator.

It is basically a horizontal cylindrical container made of linear polyethylene, inside which there is a corrosive solution with immersed compact tension (CT) specimens fixed in a loading frame. Specimens in the generator were loaded by a constant force induced by means of two pistons in a pneumatic cylinder connected in series with maximal force capacity $F_{\text{max}} = 15$ kN. It transmits a constant force to CT specimens with recording the deformation during the chemical process. Force transmission is via pin joints and the maximum piston displacement is 30 mm. This equipment is registered as a utility model. It was developed in our institute.

Experimental testing was carried out on CT specimens made from a commonly used structural steel CSN 411353. Three groups of specimens (A, B, C) were tested. Specimens A were provided with a $F$ crack, whilst specimens B and C contained SC cracks which were generated in the SCC generator after the specimens were precracked by fatigue. Specimens B were tested after fortnight and specimens C immediately after taking them out of the generator. The generating of SC cracks was carried out in an acidic solution with its composition defined by the standard NACE [14] and with $\text{H}_2\text{S}$ bubbling through. Displacement of the pistons indicated indirectly the SC crack extension on CT specimens.

The fracture resistance of the material tested (steel CSN 411353) was determined on the basis of the so called R curve with the $J$ integral as the elasto-plastic fracture parameter. The primary step to obtain the fracture toughness was determination of the „force – force point displacement“ dependence for all three groups of CT specimens, i.e. A, B, and C. The procedure for fracture toughness determination is standardized and proceeds according to the standard [15]. The $J$ integral was introduced as a line integral [16], given for a cracked body by a general relation, equation (2):

$$J = \int_{\Gamma} W \cdot dy - \int_{\Gamma} T \cdot \frac{\partial u}{\partial x} \cdot ds$$

where: $W$ is the strain energy density, $T$ is the traction vector, $u$ is the displacement vector, $ds$ is a length increment along the arbitrary contour $\Gamma$ around the tip of a crack.

3.2 Results

A comparison of the results obtained for specimens of the group A, B, and C clearly shows that the stress corrosion process significantly influences the fracture toughness in the negative direction. This keeps not only for material in which the SC process proceeds (group C) but also for material for which the cause of SC process had been removed (group B).

All three R curves obtained for the material tested (CSN 411353) are presented on figure 6 as dependences „$J$ integral – $\Delta a$“. Altogether 7 specimens were tested for each group.
Figure 6. R curves for groups A, B and C with marked values of the J integrals $J_{in}$, $J_{0.2}$ a $J_m$.

From this figure it can readily be seen the effect of stress corrosion on the fracture toughness of material. While the R curve for F crack specimens (group A) lies in the highest position and exhibits high magnitudes of the J integral the fracture properties of B group specimens are lower. This is caused by a “residual” hydrogen occurring in a certain bulk of material ahead of the crack tip even after a 14 days pause in normal atmospheric conditions. Owing to diffusion the hydrogen atoms in the structure of material can recombine into molecules at certain suitable places inducing thus extremely high local stresses in the structure (hundreds of MPa). This causes a local or a total loss of plastic properties of the steel.

The R curve for C group specimens lies in the lowest position in figure 6 and has the flattest characteristics. The magnitudes of the J integral ($J_{in}$, $J_{0.2}$ a $J_m$) exhibit approximately 80% decrease of the fracture toughness in comparison with the magnitudes referring to F cracks (group A). The results are summarized in table 3.

Table 3. Magnitudes of the J integral for A, B, and C group specimens from steel CSN 411353.

| Group | $J_{in}$ [N/mm] | $J_{0.2}$ [N/mm] | $J_m$ [N/mm] |
|-------|----------------|-----------------|--------------|
| A     | 331.8          | 446.4           | 360.2        |
| B     | 82.3           | 265.8           | 279.2        |
| C     | 51.7           | 82.0            | 80.3         |

3.3 Discussion

The radical reduction of fracture toughness of C group specimens proves how dangerous the occurrence of SC cracks in a structure can be. The cause of the reduction of fracture toughness is the diffusion of odd atoms from aggressive medium to the structure of material ahead of the crack. These atoms induce a high local stresses and lead to a reduced plastic behavior of material. The results obtained unambiguously demonstrate an increased threat of stress-corrosion cracks in structural components. It is shown that the fracture resistance of a component depends not only on the material of the component and on the crack tip constraint but also on the origin of the crack (fatigue, stress corrosion) and thus on the corresponding crack growth mechanism.

4. Conclusions

Results of two case studies proved very detrimental effects of corrosion in either corrosion fatigue or...
stress corrosion cracking in connection with pipeline steel and structural steel. From three different
corrosion media, namely crude oil, alkaline solution, and separated water, it was found that the most
detrimental effect on fatigue properties of the pipeline steel L485MB was that of the separated water.
In contrast, the effect of crude oil as well as that of alkaline solution appear to have no detrimental
effect up to the fatigue life $2 \times 10^6$ cycles as compared to fatigue properties of the steel in air (reference
S-N curve).

As for the other case study it was found that stress corrosion of CSN 411353 steel under constant
load in acidic solution, according to NACE standard, with bubbling hydrogen sulphide through
the solution reduced the J - based fracture toughness of the steel by a factor of six. The methodology
of these specific tests proved itself as effective and functional.

Acknowledgement
The authors acknowledge kind support from the Technological Agency of the Czech Republic (Project
No. TE02000162).

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