INVESTIGATIONS INTO THE INFLUENCE OF THE MECHANICAL CONDITIONS AT THE CRACK TIP ON SCC TEST RESULTS

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ABSTRACT

A great deal of the work of the Technical Committee 10, "Environmentally Assisted Cracking", of the European Structural Integrity Society (ESIS) is focused on methodical aspects of stress corrosion cracking (SCC) testing of structural materials. In this paper the results of a comparison of different SCC test methods, performed as part of the ESIS TC10 activities, are presented. A main point of concern was a possible variation of the mechanical conditions at the crack tip caused by the presence of a corrosive environment. The effect of intensive crack branching, typical for the investigated austenitic steel AISI 316H when tested in aqueous solutions, on the mechanical situation at the crack tip was evaluated using a specific procedure. This procedure is based on the estimation of the fracture toughness of specimens with a sharp fatigue pre-crack and of those having a corrosion affected crack. It is found that the retarded yielding of material at crack tip plays an essential role with respect to the corrosion crack growth resistance. This phenomenon particularly affects the data obtained from rising displacement tests and constant displacement tests.

The results of this investigation may be used for a further development of methodical recommendations pertaining to SCC tests.

INTRODUCTION

In the damage tolerant design it is assumed that cracks or defects already exist in a structure or component, and fracture mechanics assessment methods are applied. This, in turn, requires test data from pre-cracked specimens. A primary objective of fracture mechanics based investigations of environmental cracking is to evaluate a threshold, K_{Iscc} , below which stress corrosion cracking (SCC) is unlikely to occur. In the framework of the linear elastic fracture mechanics approach, which in the past has almost exclusively been used to study SCC, the stress intensity factor K in the opening mode (mode I), K_{I} , characterises the mechanical driving force, which is responsible for the initiation and subsequent growth of an environmentally assisted crack from an initial defect. The threshold value of this parameter with respect to the onset of SCC, K_{Iscc} , is used to characterise the susceptibility of materials to SCC, and serves as a guide to develop and/or select materials which exhibit a higher resistance to SCC.

Guidance for SCC tests which are aimed at determining K_{Iscc} can be found in the international standard ISO 7539 - Part 6 [1], the identical British Standard 6980 [2], and the ASTM standard E 1681-95 [3]. In the former Soviet Union the Methodical Recommendations were developed, which address SCC testing, including the use of pre-cracked specimens [4]. According to these documents K_{Iscc} usually is obtained from constant load or constant deflection tests. Recommended tests durations range from 100 to 10 000 hours, depending on the material studied. Despite of such long testing times it often remains uncertain whether the K-value measured in these tests really represents the threshold of the material. Another shortcoming of SCC tests following current standards lies in the fact that testing is performed under static loading conditions, while in reality often time-dependent loading (increasing plastic deformation) is required to generate SCC.

In an attempt to overcome the afore-mentioned problems, the Technical Committee 10 "Environmentally Assisted Cracking", of the European Structural Integrity Society (ESIS) has proposed an SCC test procedure which makes use of a time-dependent loading test method, i.e. rising displacement tests [5]. This test method is comparable to the techniques used in the so-called rising load K_{Iscc} test [6] and in the slow strain rate test [7]. Like these, it is a potentially accelerating technique in which pre-cracked specimens are increasingly deformed in the corrosive environment until they fail. According to this method, specimens containing fatigue pre-cracks are subjected to a slowly and steadily increasing displacement while they are exposed to the environment of interest. In principle, this technique resembles the fracture toughness tests in air. The major difference, however, lies in the fact that the displacement rates which are to be used for SCC tests generally have to be significantly lower than those used for fracture toughness tests in air. In rising displacement rate is a key variable which is directly related to the crack growth kinetics and/or the crack tip displacement/strain rate [8, 9].

It is known that the geometry of corrosion cracks often is different from that of the fatigue pre-cracks. The corrosion cracks can be curved and branched. Compared to these, a fatigue pre-crack can be considered as a straight line and having a sharp tip. In addition to this, stress corrosion cracks often are more blunted than fatigue cracks. This can be explained by two reasons: Firstly, the maximum of stress intensity factor, K_{max} , at pre-cracking should be lower than the K_i level at the beginning of the succeeding SCC test. Secondly, the existence of the reverse plastic zone at the crack tip causes a sharper fatigue crack even at equal load in fatigue and SCC ($K_{\text{max}} = K_i$). Thus, the effect of the corrosion environment on the SCC resistance of structural materials manifests itself not only in a change of the material's resistance to deformation and fracture but also in a change of the mechanical situation near the crack tip.

Another factor which needs to be taken into account in SCC tests is the retarded (creep-like) yielding of the material in the crack tip region taking place at ambient temperature. This phenomenon causes additional blunting due to deformation [10].

This paper continues the investigation [11] and focuses on the problem of branching and retarded yielding at SCC tests of the stainless steel A316H in water containing 100 ppm Cl (added as NaCl) at 85 °C.

EXPERIMENTAL PROCEDURE AND RESULTS

The specimens were cut out from plate-like pieces in the rolling direction (L-T orientation). The SCC experiments were performed using three different test methods, i.e. constant load tests (CLT); constant displacement tests (CDT); and constant extension rate tests (CERT).

Compact tension specimens with a thickness of 25 mm (1T CT) were used for the CLT and CERT methods. In the case of CLT, the specimens were loaded to the required load level and were then kept under constant loading during the entire time of the test. Recording of the load line displacement V_{LL} was performed similarly to the method used for *J*-integral measurements. However, the recording of V_{LL} was not conducted only during the ramp loading of the specimen but was continued under the subsequent

sustained loading. This was necessary for a verification of the occurrence of the retarded yielding. For the CERT experiments two load-line displacement rates dV_{LL}/dt were used, i.e. $0.95 \mu m/hour$ and $9.5 \mu m/hour$.

Double cantilever beam (DCB) specimens with a thickness of 25 mm were used for the CDT experiments. These specimens were loaded by bolts to different initial levels of the stress intensity factor, K_i .

All specimens were pre-cracked by fatigue at a stress ratio of R = 0.05 and a frequency of 10 Hz. The K_{max} level during the fatigue pre-cracking did not exceed 10 MPa \sqrt{m} for the final 0.5 mm of propagation of the fatigue crack.

Figs. 1 and 2 refer to SCC results obtained from CLT. It should be noted that, except for the case of $K_i = 61 \text{ MPa}\sqrt{\text{m}}$ all other levels of K_i correspond to the linear range of specimen loading (Fig. 1). This confirms that linear elastic fracture mechanics criteria can be applied.



Figure 1: Load F vs. displacement V_{LL} diagram for a specimen of the steel A316H



Figure 2: Dependencies of the displacement V_{LL} on time *t* for different levels of K_i : 1 -61 MPa· \sqrt{m} ; 2 - 31 MPa· \sqrt{m} ; 3 - 20 MPa· \sqrt{m}

After the load had been applied still a further increase of the value of V_{LL} was observed (Fig. 2). This obviously was caused by retarded yielding of the material at the crack tip. The intensity of this process decreased with the time of exposition leading to a certain stabilisation. This can particularly be seen for the lower levels of loading (curves 2 and 3, Fig. 2). At the onset of SCC a subsequent further increase of V_{LL} was observed.

In the Fig. 3 a typical corrosion crack in the steel A316H is shown. The intensive crack branching is inherent to the boundaries of the austenitic grains. Evidently, these separate cracks developed in all planes.



Figure 3: Stress corrosion crack under CLT in the steel A316H, initial load level $K_i = 61 \text{ MPa} \sqrt{\text{m}}$

It is proposed to evaluate the effect of branching on the change of the mechanical conditions at the crack tip by determining an effective stress intensity factor, K_{Leff} , using the following coefficient of relaxation, α_K , [12]:

$$\alpha_K = \frac{K_{\rm Lc}}{K_{\rm Lc}^c}.\tag{1}$$

Here, K_{Ic} and K_{Ic}^c are the fracture toughness of specimens with a usual fatigue crack and a corrosion (branched) crack, respectively. The fracture toughness tests for obtaining these K_{Ic} and K_{Ic}^c data were performed at low temperatures using liquid nitrogen in order to reduce the size of the plastic zone during loading as far as possible. Otherwise, the intensive crack blunting would have shaded the effect of branching on stress relaxation. This method is based on the concept that the fracture toughness is a material characteristics and that hence differences between K_{Ic} and K_{Ic}^c are caused by variations in the tip geometry of the fatigue and corrosion cracks. It is further assumed that the fracture of the specimens with branched corrosion cracks will initiate from that branch which corresponds to the maximum level of K_I as compared to the other branches of the corrosion crack. The actual value of the critical stress intensity factor at the tip of this branch should be equal to the value of K_{Ic} which is obtained from the fracture toughness test of a specimen with a sharp fatigue pre-crack. However, the relaxation effect of neighbouring branches cause an increase of the critical level of load and, correspondingly, of the nominal level of the stress intensity factor from K_{Ic} to K_{Ic}^c . Therefore, according to the method proposed the K_{Ieff} level can be calculated as

$$K_{\text{leff}} = \alpha_K K_{\text{I}} \,. \tag{2}$$

Even in liquid nitrogen the tests of ductile steels will not always result in brittle fracture, i.e. the application of linear fracture mechanics is not always appropriate. In these cases it is proposed to use the *J*-integral approach instead by:

$$\alpha_J = \sqrt{\frac{J_{\rm Lc}}{J_{\rm Lc}^c}},\tag{3}$$

or

$$\alpha_J = \frac{K_{\rm Ic}(J)}{K_{\rm Ic}^c(J)}.\tag{4}$$

Here, J_{Ic} and J_{Ic}^{c} are the critical values of the *J*-integral which correspond to crack initiation under active loading of specimens having a fatigue and a corrosion crack, respectively.

The load *F* vs displacement V_{LL} diagrams that were obtained during the fracture toughness tests of A316H steel specimens with a fatigue crack (1) and a corrosion (2-4) crack are presented in Fig. 4. The specimens 2 to 4 were preliminary tested for SCC under constant load at initial stress intensity factors, K_i , to achieve a corrosion crack increment, Δa_c . Further analysis showed that in active load tests the crack initiation corresponded roughly to the maximum load levels F_{max} . Hence, the parameters K_c , K_{lc}^c , J_{lc} and J_{lc}^c were determined based on this level of F_{max} .



Figure 4: Load F vs. displacement V_{LL} diagram of A316H steel specimens having a fatigue crack (1) or corrosion cracks (2-4)

As can be seen from the data given in Table 1 the coefficient of relaxation α_J lies within the range of 0.60 to 0.65, i.e. K_{Leff} is about 35 percent lower than the nominal stress intensity factor calculated without taking into account the specific geometry of the corrosion crack.

The threshold values determined from crack arrest tests at DCB specimens having an initial crack a_i were heavily affected by the severe crack branching which is typical of the environmental cracking of this steel in chloride-containing environments at elevated temperatures (Table 2). The scatter of K_{Iscc} data determined from crack arrest ranges from 13 to 22 MPa \sqrt{m} . As a general rule it can be concluded from these results that the K_{Iscc} value increases when the initial level of K_i is increased. This may be explained by the fact that the crack branching is more intensive at higher levels of K_i .

Concerning the CERT experiments, two types of loading were used, i.e. constant loading rates $dV_{LL}/dt = 0.95$ and 9.5 µm/h, respectively, and a combined loading with $dV_{LL}/dt = 60$ mm/h up to $K_I = 12$ MPa \sqrt{m} and $dV_{LL}/dt = 0.95$ µm/h for the final stage.

TABLE 1

RESULTS OF FRACTURE TOUGHNESS TESTS IN LIQUID NITROGEN OF A316H STEEL SPECIMENS WITH THE FATIGUE (1) and corrosion cracks (2-4)

Specimen	K_i ,	Δa_c , mm	$K_{c,}$	α_{K}	$J_{\mathrm{I}c,}$ N/mm	$K_{\mathrm{I}c}(J),$	α_J
	$MPa\sqrt{m}$		$MPa\sqrt{m}$			$MPa\sqrt{m}$	
1	-	-	92	-	80	137	-
2	57,5	1,3	103	0.89	193	213	0.64
3	19,2	6,5	147	0.63	214	224	0.61
4	28	3,5	99	0.93	203	221	0.63

TABLE 2

RESULTS OF SCC TESTS OF THE STEEL A316H USING CONSTANT DISPLACEMENT TESTS

Specimen	K_i ,	K_{Iscc} ,	a_i ,	Δa_c ,	Test time <i>t</i> ,	
	$MPa\sqrt{m}$	MPa√m	mm	mm	hours	
1	35.5	22	23.87	0.38	3600	
2	15	13	19.85	0.00	3600	
3	26	16	20.67	0.76	3600	
4	13	15	18.63	2.86	3600	

The tests results show that in the first case the initiation of stress corrosion crack growth occurred at $K_{\rm I} > 18 \text{ MPa} \cdot \sqrt{\text{m}}$ and at $K_{\rm I} > 24 \text{ MPa} \sqrt{\text{m}}$ for $dV_{LL}/dt = 0.95$ and 9.5 µm/h, whereas for the combined loading it occurred at $K_{\rm I} = 25 \text{ MPa} \sqrt{\text{m}}$.

When analysing the effect of the loading rate on the K_i level at crack initiation, it appears that this behaviour can be related to a retarded yielding of the material at the crack tip. In order to verify this, K_I vs. V_{LL} diagrams are plotted (Fig. 5). It appears that for the ambient value of K_I , the displacement V_{LL} significantly depends on the loading rate. The value of V_{LL} may be presented in the form:

$$V_{LL} = V_{LL}^{a} + V_{LL}^{ry}.$$
 (5)

Here, V_{LL}^a is an active component which can be determined from a plot obtained at a high loading rate, and V_{LL}^{ry} is a component which is determined from the intensity of the retarded yielding of the material at the crack tip. This relation means that the intensity of the retarded yielding is increasing when the loading rate is decreased. Therefore, it may be concluded that the true value of K_{Lscc} can be obtained at a rate of loading, $(dV_{LL}/dt)^*$, for which the retarded yielding process is most pronounced. For rates $dV_{LL}/dt \leq (dV_{LL}/dt)^*$ no change in the intensity of the retarded yielding process should occur.

Those points on the K_{I} vs V_{LL} diagram, which correspond to the corrosion crack initiation are indicated by arrows in Fig. 5. Since the effect of the retarded yielding of the material can be evaluated qualitatively using the *J*-integral approach, an attempt was made to estimate the value of J_{I} which corresponds to the initiation of the corrosion crack:

$$J_{\rm I} = J_{\rm I}^{e} + J_{\rm I}^{ry}.$$
 (6)

Here, the elastic part was estimated using the equation:

$$J_{\rm I}^{e} = \frac{K_{\rm I}(1 - v^2)}{E},\tag{7}$$

and the plastic part J_1^{Iy} was calculated. As a result, the following values of J_I were obtained: 6.4 and 8.9 kJ/m² for the loading rates of $dV_{LL}/dt = 0.95$ and 9.5 µm/h, and 8.3 kJ/m² for the combined loading. This indicates that a conservative level was reached at the lower of the two constant loading rates. It should be pointed out that the highest value of J_i was not measured under the combined loading, which started off at a very high initial loading rate, but at the higher one of the two constant loading rates. This in turn would mean that taking into account the retarded yielding phenomenon can to a certain extent yield a means of correction when using different techniques for measuring stress corrosion crack initiation. However, the *J* integral, which describes the deformation state at the crack tip, can not comprehensively characterise the conditions under which the corrosion crack starts from the tip of fatigue pre-crack. Instead, the time dependence of this parameter has to be taken into consideration.



Figure 5: Applied stress intensity factor K_{I} vs. displacement V_{LL} for specimens of A316H steel under rising load at different rates of dV_{LL}/dt : 1 -60 mm/h; $2 -9.5 \mu$ m/h; $3 -0.95 \mu$ m/h; 4 - combined: 60 mm/h for the initial stage and 0.95 μ m/h for the final stage

CONCLUSIONS

The SCC of the stainless steel A316H in chloride containing environments at elevated temperatures is accompanied by intensive crack branching and the occurrence of retarded yielding at the crack tip. Therefore, these phenomena should be taken into consideration when assessing SCC tests at this material/environment combination.

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References

- 1. ISO 7539, (1989). International organization for standardization. Corrosion of Metals and alloys stress corrosion testing, Part 6: Pre-cracked specimens. ISO, Geneva.
- 2. BS 6980, (1990). British standards institution. Stress corrosion-testing. Part 6: Method for the preparation and use of pre-cracked specimens. BSI Gr.6, London.
- 3. ASTM Standard E 1681-95. (1995). Test method for determining a threshold stress intensity factor for environment-assisted cracking of metallic materials under constant load. Annual Book of ASTM Standards.
- 4. M ethodical recommendations]] 185-86. (1986). Analyses and tests of sensitivity of steels and alloys to stress corrosion cracking in liquid environments. VNIINMASH, Moscow.
- 5. ESIS P4-92 D. (1992). European Structural Integrity Society. ESIS recommendations for stress corrosion testing using pre-cracked specimens. ESIS, Delft.
- 6. McIntyre, P. and Priest, A.H. (1972). Accelerated test technique for the determination of K_{Iscc} in *steels*. British Steel Corporation Report MG /31/71, London.
- 7. Parkins, R.M. (1979). In: ASTM STP 665, (G.M. Ugiansky and J.H.Payer, eds.), ASTM, Philadelphia. pp.5-25.
- 8. Strieder, K., Daum, K.-H., Dietzel W. and Müller-Roos J. (1994). In: *Structural Integrity: Experiments Models Applications, Proc. 10-th European Conf. on Fracture*. Berlin, pp. 715-720.
- 9. Dietzel, W. and Ghosal, S.K. (1997). *Physicochemical Mechanics of Materials*, pp. 97-106.
- 10. Nykyforchyn, H.M., Dietzel, W., Tsyrulnyk, O.T. and Bassarab, A.I. (1999). In: *Environmental Degradation of Engineering Materials, Proc. Intern. Conf.* Gdansk Scientific Society, Gdansk, v.1, pp. 54-65.
- 11. Dietzel, W., Gabetta, G., Brozova, A., Sajdl, P. and Nykyforchyn H. (1998). In: Problems of Corrosion and Corrosion Protection of Materials, Proc. 4-th Int. Conf. Karpenko Physico-Mechanical Institute, Lviv, pp. 418-420.
- 12. Romaniv, O.M. and Nykyforchyn H.M. (1984). In: Proc. Int. Congress on Metallic Corrosion. Toronto, v. 4, pp. 498 505.