STANDARDIZATION OF FRACTURE MECHANICS BASED SCC TESTING

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The technique specified in the first draft of the ESIS recommendations for stress corrosion testing using pre-cracked specimens (ESIS document P4-92 D) is a dynamic stress corrosion cracking (SCC) test procedure which aims at providing both threshold and crack propagation data within an acceptable amount of time. Pre-cracked fracture mechanics specimens are loaded at a constantly rising displacement during exposure to a chemically aggressive environment. Elastic-plastic fracture mechanics test and evaluation techniques are optionally included in the test concept. The proposed procedure could become a supplement to existing standards or form the basis for a new SCC test standard.

INTRODUCTION

Although stress corrosion cracking (SCC) has long been recognized as a major cause for failure of structures, the basic mechanisms of this environmentally assisted cracking (EAC) are only partly known. There is no general theory of SCC and it is not possible to truly predict which combinations of alloys and environments will result in SCC. Thus, at the present time all guidelines for avoiding environmentally induced cracking during service are based either on past experience or on laboratory tests.

Unfortunately, the usefulness of SCC tests is restricted by a number of problems such as

- the complexity of environmentally assisted cracking, which is influenced by alloy microstructure, the chemistry of the environment, and the form of the mechanical stress-strain conditions;
- the large number of variables that may affect the behaviour of each combination of material and environment;

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- the uncertainties associated with "incubation" times;
- the large amount of data scatter and sometimes poor correlation between test results and service experiences;
- the lack of generally accepted standard test methods.

From the designers viewpoint, the synergism between metallurgy, chemistry and mechanics which is characteristic for SCC makes it necessary to account for a wide range of variables which often appear to be unrelated. Thus, designing against environmentally induced cracking is much more difficult than designing against purely mechanical fracture.

To reduce the likelihood of unexpected failure caused by SCC an appropiate strategy in relation to design, material selection, environment control, crack detection and quantitative risk assessment is required. The development of standardized and practicable methods of testing is a key element in this strategy in order to provide a unified reference framework for generating reliable measurement data. The test times required in SCC tests are a hindrance to the development of these data. Therefore, the need exists for accelerated test methods, provided that these can yield reliable results which can be interpreted for long-term cracking problems, since most of the critical issues in SCC involve the service life of components over many years, decades, or even centuries.

FRACTURE MECHANICS BASED SCC TEST STANDARDS

In the damage tolerance approach which is increasingly applied to problems of environmentally induced failure, cracks or defects are assumed to already exist in a given structure. Use is made of fracture mechanics analyses, which in turn require test data from cracked specimens including both the commencement of crack growth from an initial crack or defect and the kinetics of the crack growth process. Since the fracture mechanics assessment of SCC susceptibility is usually based on linear elastic fracture mechanics (LEFM), the elastic stress intensity factor K_I is used to characterize the mechanical driving force for the commencement and subsequent propagation of environmentally assisted cracking from an initial crack or defect. The parameters determined from this LEFM approach to SCC are the threshold value of the stress intensity factor, K_{Iscc} (or K_{EAC}), below which environmentally assisted cracking in pre-cracked specimens does not occur, and the crack growth velocity, da/dt, as a function of K_I (Fig. 1).

The application of fracture mechanics to environmentally assisted fracture dates back almost thirty years (1-3). However, the number of standard test methods that provide guidelines for carrying out fracture mechanics based SCC tests is very limited. So far, only the International Standard ISO 7539 - Part 6 (identical to British Standard BS 6980-6) and the NACE Standard TM 0177-90 - Section 9 provide guidelines for the use of pre-cracked samples in stress corrosion tests aimed at the determination of K_{Iscc} and $da/dt = f(K_I)$ (4-6). According to these standards

K_{Iscc} is evaluated either in constant load or in constant deflection experiments. The duration of the tests is generally "left open to the parties concerned". However, minimum testing times are recommended. In the ISO standard 7539-6 (the NACE Standard pertains only to the special case of sulfide stress cracking in H_2S environments) these recommended testing times range from 100 hours for titanium alloys to 10 000 hours for aluminium alloys, and for lower strength and high alloy steels.

Standard procedures for fracture mechanics based stress corrosion cracking tests are also in preparation by two ASTM Committees. Here too, testing will be based on static loading, i.e. on constant load and on constant deflection experiments, and the test durations will be essentially the same as in ISO 7539-6.

Apart from their merits - the test are easy to carry out and they require a minimum amount of laboratory equipment - the existing standards and drafts have a number of drawbacks:

- The recommended testing times for determining K_{Iscc} in static SCC tests are extremely long; yet there remains uncertainty whether the K-value measured at test termination really represents the threshold value for a corrosion system. Interlaboratory tests programs which were conducted in the past by using static loading procedures and in which nominally identical material/environment systems were investigated have revealed high degrees of scatter between the results obtained in different laboratories (7-9).

- They are based on static loading, i.e. constant load or constant deflection. However, it appears that in certain material/environment systems dynamic loading (increasing plastic deformation) is required to cause SCC (10).

- The specimen sizes have to satisfy the limitations of linear elastic fracture mechanics which, for lower strength and/or more ductile materials, may result in large specimen dimensions. None of the existing standards or drafts takes into account the use of elastic-plastic fracture mechanics methods which would help to reduce the size of the test specimens.

PROBLEMS WITH SCC TESTING

The main reason for the difficulties in developing generally accepted fracture mechanics based SCC test standards lies not in a shortcoming of the underlying fracture mechanics approach but in the complexity of the parameters which influence the environmentally assisted failure and in their interdependence (11, 12).

Mechanical Driving Force

In the existing standards and drafts for SCC testing of precracked specimens the crack tip stress intensity factor K_I is used to characterize the mechanical driving force. The result of this is a disparity of the crack size requirements for fracture mechanics based SCC testing and the size of cracks typical of practical problems of environmentally induced cracking. In practice SCC can occur under conditions that deviate significantly from plane-strain conditions.

The applicability of LEFM to SCC is based on the assumption of small scale yielding which, for lower strength alloys and for alloys with high resistance to SCC, is not justified. Moreover, sufficient plasticity may occur that neither plane-strain nor linear elastic conditions are satisfied. In these cases LEFM cannot be applied and K is no longer a meaningful parameter. Instead, elastic-plastic approaches such as the J-integral or CTOD parameter should be used as the crack driving force. In the case of large scale yielding additional changes in the interaction between the environment and the deformed microstructure may have to be taken into account.

Other important mechanical parameters can be the degree of prestressing of components, the amount of residual stresses, corrosion product wedging, or the portion of mode II and mode III loading at the crack tip in cases of large scale yielding. The results of SCC tests are furthermore dependent on the loading rate and on the shape of the loading ramp. Finally, the potential influence of stress or strain gradients resulting from different loading conditions (e.g. tension vs bending) on crack growth needs to be considered, since these parameters may affect the access of the corrosive environment to the crack tip.

Besides the loading configuration, the location, size and shape of a defect can play an important role. The same holds for crack branching, which also influences the crack growth conditions and, in crack arrest tests, the measured threshold.

Environmental Variables

Environmental variables such as temperature, pH, electrochemical potential, concentration or partial pressure control the kinetics of the electrochemical process responsible for the environmentally induced fracture. Further complication arises from the fact that the crack tip chemistry can be considerably different from the electrochemical conditions outside the crack. Therefore a precise characterization of the environmental conditions inside the crack is essential for the achievement of realistic test conditions. SCC test recommendations often avoid this problem by defining "standard" environmental conditions.

Metallurgical Variables

Apart from composition, a number of metallurgical variables such as heat treatment (temper) conditions, inhomogeneity of the microstructure, grain size and orientation with respect to the mechanical loading can influence the SCC susceptibility of metallic materials. Further complications may arise from welded components and structures. Care therefore has to be taken to ensure that the metallurgical conditions of the test material really represent those of the material in the actual service situation.

Consequences for Standardizing SCC Tests

As a consequence of this complex problem the test conditions for various material-environment combinations can be considerably different. It therefore appears extremely difficult (if not impossible) to define generally valid test conditions. A catalogue of requirements for developing a rational test methodology for fracture mechanics based SCC tests would therefore comprise a list of parameters that especially need to be looked at, i.e.:

- loading situation,
- test duration,
- criteria for crack initiation or crack arrest,
- state of stress and plasticity effects,
- small size defects,
- environmental control,
- metallurgical parameters.

Prerequisite for an exact treatment of these problems would be a comprehensive understanding of the underlying processes. Since this understanding still remains incomplete, decisions about test conditions have to be based on experimental experience gained from tests with a wide variety of corrosion systems and will have to be adjusted in the course of time.

ESIS PROCEDURE P4-92 D

The first draft of "Recommendations for Stress Corrosion Testing Using Pre-Cracked Specimens" (ESIS P4-92 D (13), the letter D stands for "draft") which has been prepared by the Technical Committee 10 ("Environmentally Assisted Cracking") of the European Structural Integrity Society ESIS is an attempt to solve at least some of these problems. It comprises a dynamic SCC test concept which is based on a rising load or a rising displacement technique. The recommended test procedure partly makes use test techniques which are specified in the ESIS procedures P1-92 (14) and P2-92 (15) for determining the fracture behaviour of materials in air. Specimens containing a sharp fatigue pre-crack are subjected to a constantly increasing displacement, and the load and displacement are measured during the test. The onset and extent of crack growth is monitored via indirect crack length measuring techniques such as the potential drop technique or unloading compliance. Contrary to fracture toughness measurements in air the specimens are exposured to a chemically aggressive environment while being loaded and considerably low displacement rates are applied.

Rising load and rising displacement tests have been used in various studies of environmentally assisted cracking over the last two decades (16-29). A good deal of this work made use of linear elastic fracture mechanics; yet in some more recent investigations elastic-plastic fracture mechanics methods were applied (19-27). Comparisons between results obtained in rising displacement tests with those from static SCC tests have revealed different amounts of correspondence (27-29).

In Figs. 2 - 4 a comparison is shown for the high strength aluminium alloy 2024 T 351 tested in sodium chloride solution. Fig. 2 contains two da/dt-K curves which were generated with different test methods: One curve, starting at high K levels and running in the direction of decreasing K, was measured using a double cantilever beam (DCB) specimen which was constantly deflected via bolt loading. The second curve which starts at the left hand part of the diagram was measured in a rising displacement test of a compact type tension (CT) specimen. In both cases the threshold K_{Iscc} and the plateau value of the crack velocity da/dt are nearly identical.

The significant difference between these two test methods becomes obvious from Figs. 3 and 4, where the increase in crack lengths measured with time is plotted. Fig. 3 reflects the results from periodical measurements at the DCB specimen. In the first part of the test the crack has grown considerably fast, and the plateau value for da/dt could be determined within two to three weeks. After approximately 200 days the curve asymptotically approached the crack arrest value. From the final crack length K_{Iscc} was calculated. The total duration of this test was about one year.

Fig. 4 is an original plot from DC potential drop measurements during the rising displacement test of the CT specimen. This test was performed at a displacement rate of 0.2 µm/h and the DCPD signal was measured with a pulsed technique specially designed for long term SCC measurements (29). In the first part of the test the potential drop remains more or less stable. After about 15 days the DCPD signal starts to increase, indicating that the crack has initiated and that K_{Iscc} is exceeded. After a test duration of 27 days or less than one month the crack has grown by about 2 millimeters and da/dt is in the plateau region. Thus, despite of the extremely low displacement rate it took significantly less time to obtain the same results than in a static SCC test.

Although the accelerating nature of dynamic SCC testing has often been pointed out in the past, no attempts so far have been made to standardize this test technique. The use of an increasing load for accelerated testing is already mentioned in ISO 7539-6. Yet, the standard contains no information about an appropiate procedure for this type of test; especially not about the key parameter, i.e., the loading or displacement rate which should be applied in order to obtain reliable results.

Test Procedure

In the procedure specified in the ESIS document P4-92 D guidance is provided for the determination of the displacement rates which are suitable for rising displacement SCC tests. The procedure consists of a multiple specimen method in which several specimens made of the same material are loaded at different displacement rates. Tests are performed in the corrosive environment of interest and in an inert environment.

Displacement rate. It is assumed that the displacement rate, $(dq/dt)_{scc}$, at which a rising displacement test in a corrosive environment has to be performed in order to determine K_{Iscc} for a material/environment system, can be estimated from the ratio of the measured crack growth velocity in a rising displacement test in an inert environment, $(da/dt)_{inert}$, and the crack growth velocity in the plateau region for environmentally induced cracking, $(da/dt)_{scc}$, by

$$(dq/dt)_{scc} < \frac{(da/dt)_{scc}}{(da/dt)_{inert}} \cdot (dq/dt)_{inert} \quad . \tag{1}$$

As a first approach ESIS P4-92 D recommends that

$$(dq/dt)_{scc} \leq 0.5 \cdot \frac{(da/dt)_{scc}}{(da/dt)_{inert}} \cdot (dq/dt)_{inert} \quad . \tag{2}$$

The value of $(da/dt)_{scc}$ can often be obtained within reasonable time from a number of test techniques which avoid long incubation periods by applying high stress intensity levels. For example, constant displacement tests on CT, DCB or WOL (wedge opening load) specimens which are terminated after a sufficient amount of crack propagation can be suited for this purpose (27). The plateau velocity for SCC may also be measured in a stepwise loading procedure (30, 31). Even average crack velocity data from conventional slow strain rate tests on smooth specimens can serve as a lower bound value for the calculation (21, 32, 33).

Test steps. A possible procedure for determining the value of K_{Iscc} for a material/environment system is illustrated in Fig. 5. In principle, the test procedure comprises the following steps:

- the ratio of $(da/dt)_{inert}/(dq/dt)_{inert}$ is determined in an R curve test in air or in an inert environment (step I);
- $(da/dt)_{scc}$ is measured with a suitable technique, e. g., a constantly deformed DCB specimen which is exposed to the corrosive environment (step II); this test is terminated after the crack has grown for several millimeters;

- a pre-cracked specimen is loaded in the corrosive environment using the displacement rate $(dq/dt)_{SCC}$ calculated according to relationship (2); the test is performed until crack initiation and subsequent crack growth are observed (step III);

- step III is repeated at least once - either at the same or at a displacement rate which differs by a factor of 10 - in order to verify the measurement (step IV).

After termination of each test the specimens are broken open and the initial and final crack lengths are determined from the fracture surfaces. The crack growth velocity da/dt is evaluated.

Data Analysis. In the first draft of ESIS P4-92 D the data analysis is based on the assumption that cracking takes place under conditions which justify the application of linear elastic fracture mechanics. Thus the fracture parameter used in this procedure is the stress intensity factor, K_I . The susceptibility of the material to stress corrosion cracking is then characterized by the threshold stress intensity factor, K_{Iscc}, and by the crack propagation velocity, da/dt, as a function of the applied stress intensity factor K_I.

Although the extent to which the presence of the corrosive environment reduces the amount of plastic deformation associated with the onset of cracking is not known, it is recommended in this first draft that the specimen dimensions should be sufficient to maintain predominantly triaxial (plane strain) conditions in which plastic deformation is limited to the vicinity of the crack tip. This means that a test is valid, i.e., the provisionally determined value K_{Qscc} corresponds to K_{Iscc}, if

$$a\,,\,B > 2.5 \cdot \left(\frac{K_{Qscc}}{\sigma_y}\right)^2 \qquad (3)$$

In cases, where plastic deformation exceeds the assumptions of linear elastic fracture mechanics, elastic-plastic fracture parameters such as the J-integral or the crack tip opening displacement (CTOD) are recommended instead of the stress intensity factor K. Procedures for the evaluation of the J-integral and/or the CTOD will be included in the second draft of ESIS P4-92 D.

DISCUSSION

Currently the first draft of ESIS P4-92 D is discussed within ESIS TC 10 and in the corresponding ISO working group. One major argument against this attempt to specify a dynamic SCC test procedure is concerned with the way of determining the displacement rate. The cracking mechanisms in air or inert environment usually are completely different from the mechanisms of environmentally induced failure. It therefore appears at least doubtful whether crack velocities measured in air can be used for determining displacement rates for SCC tests. Other arguments are related to the problem of specifying one unified test procedure for the various phenomenons of environmental degradation such as anodic dissolution and hydrogen embrittlement.

To further assess the feasability and reliability of the recommended test procedure and to learn more about its requirements an interlaboratory test programme has been initiated in which more than 20 laboratories will be generating data for different corrosion systems using various test techniques. A final version of ESIS P4 will be formulated on completion of the programme according to the experience which has thus been obtained. Depending on the outcome of this project it will be decided whether the recommended test procedure is suited for forming a new part of ISO 7539 which would entirely be related to dynamic SCC testing and can serve as a basis for a corresponding CEN standard.

SYMBOLS USED

a = crack length

B = specimen thickness

K = stress intensity factor

K_{Iscc} = threshold stress intensity factor for the onset of stress corrosion cracking

K_{Qscc} = provisional value of K_{Iscc}

q = crack opening displacement (general)

σ_y = yield strength

REFERENCES

(1) Steigerwald, E. A., Proc. Am. Soc. Test. Mat. Vol. 60, 1960, pp. 750-760

(2) Progress in Measuring Fracture Toughness and Using Fracture Mechanics; Fifth Report of a Special ASTM Committee; Mat. Res. Stds. 4, 1964, pp. 107-119

(3) Brown, B. F., Mat. Res. Standds. Vol. 6, 1966, pp. 129-133

(4) International Standard ISO 7539: Corrosion of Metals and Alloys - Stress Corrosion Testing; Part 6: Pre-Cracked Specimens, ISO, Geneva, 1989

(5) British Standard 6980, Stress CorrosionTesting - Part 6. Method for the Preparation and Use of Pre-Cracked Specimens; BSI (Gr. 6), London, 1990

(6) NACE Standard TM 0177-90: Standard Test Method Laboratory Testing of Metals for Resistance to Sulfide Stress Cracking in H_2S Environments, 1990

(7) Wei, R. P. and Novak, S. R., J. Test.. Eval. Vol. 15, 1987, pp. 38-75

(8) Yokobori, T., Watanabe, J., Aoki, T., and Iwadate, T., ASTM STP 945, 1988, pp. 843-866

(9) Domack, M. S., ASTM STP 1049, 1990, pp. 391-409

(10) Erlings, J. G., de Groot, H. W., and Nauta, J. Corrosion Sc., Vol. 27, No. 10/11, 1987, pp. 1153-1167

(11) Characterization of Environmentally Assisted Cracking for Design, NMAB - 326, National Materials Advisory Board, Washington, 1982

(12) Judy, R. W. Jr., King, W. E. Jr., Hauser, J. A. H., and Crooker, T. W., ASTM STP 1049, 1990, pp. 410-422

(13) ESIS P4-92 D: ESIS Recommendations for Stress Corrosion Testing Using Pre-Cracked Specimens (1st Draft), ESIS, Delft, 1992

(14) ESIS P1-92, ESIS Recommendations for Determining the Fracture Resistance of Ductile Materials, ESIS, Delft, 1992

(15) ESIS P2-92, ESIS Recommendations for Determining the Fracture Behaviour of Materials, ESIS, Delft, 1992

(16) McIntyre, P. and Priest, A. H., Br. Steel Corp. Rep. MG/31/71, London, 1972

(17) Clark, W. G., Jr. and Landes, J. D., ASTM STP 610, 1976, pp. 108-127

(18) Hirano, K., Ishizaki, S., Kobayashi, H., and Nakazawa, H.; J. Test. Eval. Vol. 13, 1985, pp. 162-168

(19) Mayville, R. A., Warren, T. J., and Hilton, P. D.; J. Test. Eval. Vol. 17, 1989, pp. 203-211

(20) Dietrich, M. R., Caskey, G. R., and Donovan, J. A., in: Hydrogen Effects in Metals, I.M. Bernstein and A. W. Thompson (Eds.), New York, 1980, pp. 634-643

(21) Anderson, D. R. and Gudas, J. P., ASTM STP 821, 1984, pp. 98-113

(22) Abramson, G., Evans, J. T., and Parkins, R. N., Met. Trans. A, Vol. 16, 1985, pp. 101-108

(23) Kawakubo, T. and Hishida, M., Trans. A.S.M.E. Vol. 107, 1985, pp. 240-245

(24) Hackett, E. M. Moran, P. J., and Gudas, J. P., ASTM STP 905, 1986, pp. 512-541

(25) Mayville, R. A., Warren, T. J., and Hilton, P. D., Trans. A.S.M.E. Vol. 109, 1987, pp. 188-193

(26) Dietzel, W., Schwalbe, K.-H., and Wu, D., Fatigue Fract. Engng. Mater. Struct., Vol. 12, No. 6, 1989, pp. 495-510

(27) Dietzel, W., Ph. D. Thesis, Technische Universität Hamburg-Harburg, 1989, Rep. GKSS 91/E/27, Geesthacht, 1991

(28) Mayville, R. A., Warren, T. J., and Hilton, P. D., ASTM STP 1020, 1989, pp. 605-614

(29) Dietzel, W. and Schwalbe, K.-H., Z. Materialprüfung/Materials Test., No. 28, 1986. pp. 368-372

(30) Raymond, L. and Crumly, W.R., in: Proceedings First Hydrogen Problem in Steel, Washington, D.C., A.S.M.E., Ohio, 1982, pp. 477-480

(31) Christensen, C. and Hill, R. T., Corrosion Sc. Vol. 27, 1987, pp. 1137-1144

(32) International Standard ISO 7539: Corrosion of Metals and Alloys - Stress Corrosion Testing; Part 7: Slow strain rate testing, ISO, Geneva, 1989

(33) Strieder, K., Master Thesis, Ruhr-Universität Bochum, 1993

(34) Brown, B. F. Metallurgical Rev., Vol. 13, 1968, pp. 171-183

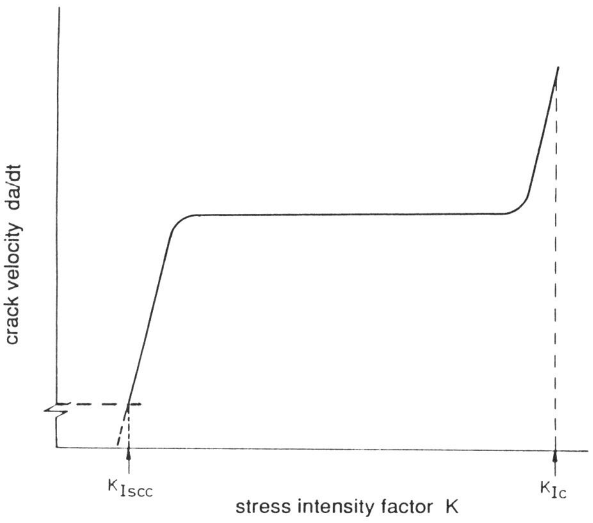

Figure 1 Relationship between the stress intensity factor K and the crack growth velocity da/dt ("v-K curve"), schematic after Brown (34)

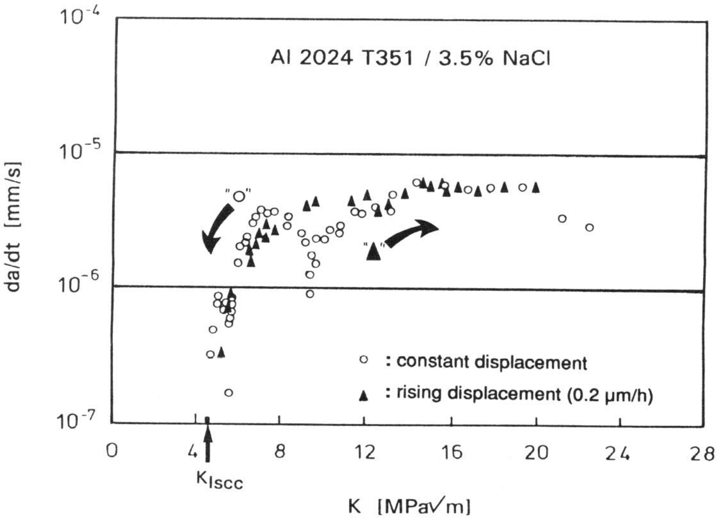

Figure 2 Experimental v-K curves obtained in SCC tests under conditions of constant and rising displacement

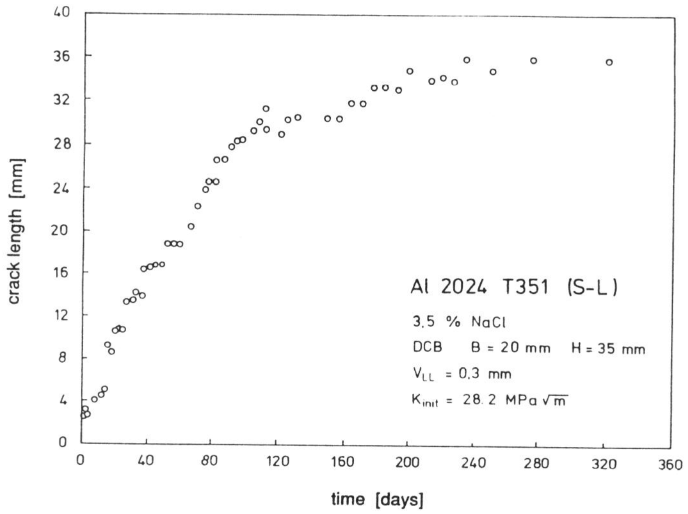

Figure 3 Crack extension as a function of test duration measured on a bolt loaded DCB specimen

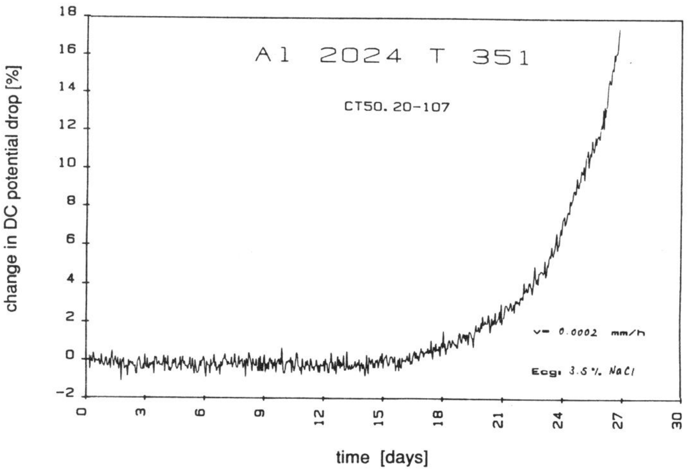

Figure 4 Change in DC potential drop with test time measured on a CT specimen in a rising displacement test (dq/dt = 0.2 μm/h)

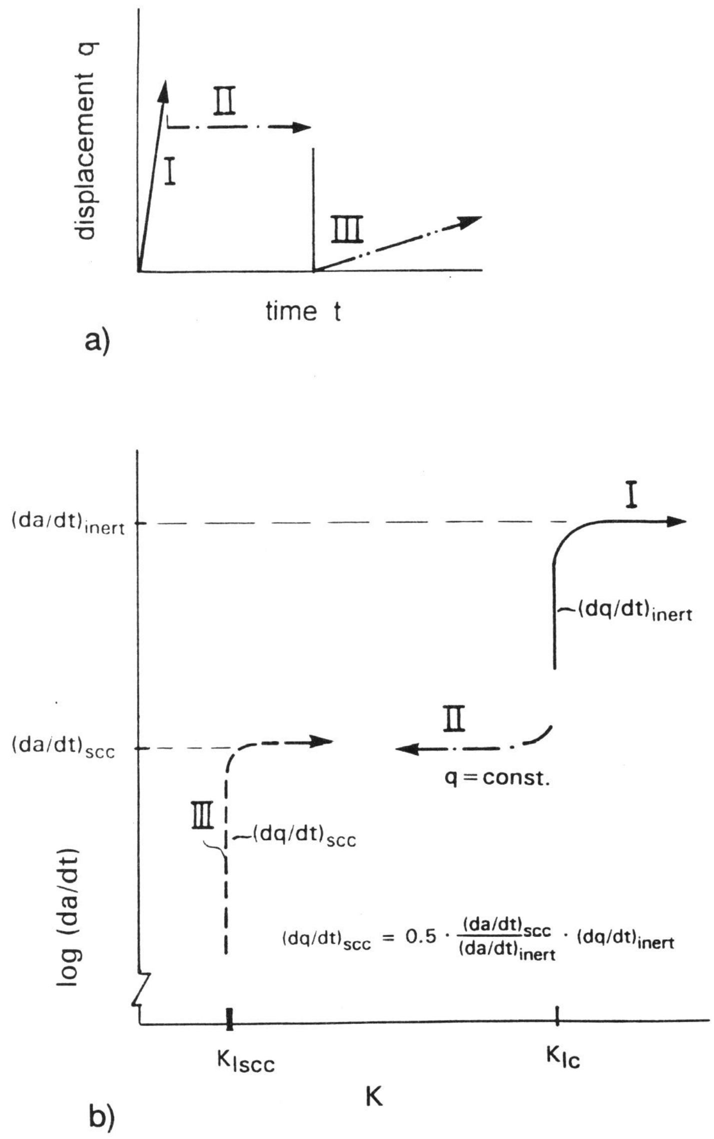

Figure 5 Illustration of the test procedure proposed in ESIS P4-92 D
a) displacement vs time b) v-K curve obtained in steps I to III (schematic)