# 11.03 Stress Corrosion Cracking

### W. DIETZEL

GKSS-Forschungszentrum Geesthacht GmbH, Geesthacht, Germany A. TURNBULL National Physical Laboratory, Teddington, UK

11.03.1 INTRODUCTION	44
11.03.2GENERAL ASPECTS OF SCC TESTING11.03.2.1SCC Test Philosophies11.03.2.2Specimen Configuration and Loading Mode11.03.2.3Environment Control11.03.2.4Time-Dependent Issues	46 46 47 48 49
11.03.3NON-PRECRACKED SPECIMENS11.03.3.1Constant Load Tests11.03.3.2Constant-Displacement Tests11.03.3.3Slow Strain Rate Tests11.03.3.4Breaking Load and Linearly Increasing Stress Tests	50 50 51 52 54
<ul> <li>11.03.4 PRECRACKED SPECIMENS – THE FRACTURE MECHANICS APPROACH TO SCC</li> <li>11.03.4.1 Linear Elastic Fracture Mechanics</li> <li>11.03.4.2 Requirements of Fracture Mechanics SCC Testing</li> <li>11.03.4.3 Loading Mode</li> <li>11.03.4.4 Constant-Load Tests</li> <li>11.03.4.5 Constant-Displacement Tests</li> <li>11.03.4.6 Rising-Load and Rising-Displacement Tests</li> </ul>	54 55 56 57 57 58 59
11.03.5 CRACK GROWTH MEASUREMENT 11.03.5.1 Electrical Resistance Measurement Methods 11.03.5.1.1 DC potential drop method 11.03.5.1.2 AC potential drop method 11.03.5.2 Compliance Methods	61 61 63 63
11.03.6 LIMITATIONS OF THE LEFM APPROACH TO SCC 11.03.6.1 The J-Integral 11.03.6.2 Crack-Tip Opening Angle/Displacement 11.03.6.3 Shallow Cracks	63 64 65 66
11.03.7 THE USE OF SCC DATA         11.03.7.1 Predicting the SCC Behavior of Materials and Structures         11.03.7.2 Studies of SCC Mechanisms	67 67 67
<ul><li>11.03.8 GUIDE TO SELECTION OF MECHANICAL SCC TEST METHOD</li><li>11.03.9 CONCLUDING REMARKS</li></ul>	69 72
11.03.10 REFERENCES	73

#### NOMENCLATURE

a	crack size, half length of a center
	crack, crack depth for a surface crack
A	elongation to fracture
$a_0$	initial crack size
В	specimen thickness
С	half crack length for a surface crack
$\mathrm{d}\delta_5/\mathrm{d}t$	applied deformation rate measured
_	at the tip of the fatigue precrack
$D_{\rm eff}$	(effective) diffusivity of hydrogen
_	atoms
<i>E</i>	modulus of elasticity
E'	modulus of elasticity for constraint
_	conditions
$E_{f}$	fracture energy
$F_{-}$	applied force
$F_{\mathbf{Y}}$	yield load
J	J-integral
$J_{0.2}$	value of $J$ at 0.2 mm of stable crack
_	extension
$J_{SCC}$	value of J at initiation of environ-
	mental crack extension
K	linear elastic stress intensity factor
$K_1$	linear elastic stress intensity factor,
	crack opening mode I
$K_{\rm lc}$	plane-strain fracture toughness
K <sub>ISCC</sub>	threshold value of the stress inten-
	sity factor for the onset of
••	environmental cracking
K <sub>mat</sub>	fracture resistance in terms of K
$K_{\rm r}$	ratio of $K_1$ and $K_{mat}$
$L_{\rm r}$	ratio of applied load to yield load,
	$F/F_{\rm Y}$
q	load point displacement (general)
R <sub>el</sub>	elastic limit of a steel with a Luders
	distance behind the creak tip for
r <sub>m</sub>	distance benind the crack up for $\frac{1}{2}$
D	measuring $o_c$
Λ <sub>m</sub> D	viold strength aquivalent to 0.2 per
<b>Л</b> <sub>Р0.2</sub>	sent proof stress
ta	time to failure
4 V	load-line displacement
	crack-mouth opening displacement
7	reduction in area
$\tilde{\lambda}_{\epsilon}$	crack-tip opening displacement
~ 3	(CTOD)

$\delta_{5c}$	critical crack-tip opening
- 50	displacement
$\Delta a$	average crack extension
ε	strain
$\varepsilon_{\rm el}$	elastic strain
$\varepsilon_{\rm pl}$	plastic strain
$\varepsilon_{\rm tot}$	total strain
ν	Poisson's ratio
$\sigma$	stress
$\sigma_{ m init}$	initial stress
$\sigma_{\rm v}$	yield strength
$\psi$	crack-tip opening angle
$\psi_{ m c}$	critical value of $\psi$ at initiation of

#### **ABBREVIATIONS**

#### **Specimen Types**

C(B)	cantilever bend
C(T)	compact
DCB	double-cantilever beam
SE(B)	single-edge notch
WÒĹ	wedge open loaded

crack extension

#### Acronyms

CERT	constant-extension rate tensile
	(= slow strain rate)
CMOD	crack-mouth opening
	displacement
COD	crack opening displacement
CTOA	crack-tip opening angle
CTOD	crack-tip opening displacement
EAC	environmentally assisted
	cracking
IGSCC	intergranular stress corrosion
	cracking
LIST	linearly increasing stress test
LVDT	linear variable displacement
	transducer
RA	reduction in area
SCC	stress corrosion cracking
SEM	scanning electron microscope
SSRT	slow strain rate test
TGSCC	transgranular stress corrosion
	cracking

#### 11.03.1 INTRODUCTION

Stress corrosion cracking (SCC) describes the initiation and growth of cracks caused by the synergistic combination of a nominally static, or slowly increasing, tensile stress, and an environment, which is usually corrosive. The stresses are typically below the macroscopic yield strength but sufficient to induce localized microscopic plastic strain, a key component in the initiation and propagation process. They may include or entirely consist of residual and/or fit up stresses.

The nominally static or slowly increasing monotonic nature of the stresses distinguishes SCC from corrosion fatigue in which the loading is predominantly cyclic (see Chapter 4.04). Although there are considerable similarities between the two processes at the mechanistic level, SCC tends to be more environment specific than corrosion fatigue. Accordingly, SCC is not an all-pervasive failure process. Nevertheless, most structural materials have been demonstrated to have susceptibility in certain environment-temperature combinations as highlighted in **Table 1** (Cottis, 2000). Besides, the table discusses as follows:

1. The table presents the systems for which SCC problems are well established and of practical importance. The absence of a metal-environment combination from this table does not mean that SCC has not been observed.

2. There are rarely well-defined temperature or concentration limits for SCC, and the ratings given here are indicative only. As an approximate guide the terms used equate to the following ranges of values:

Concentration	1	Temperature
Low	Up to $10^{-2}$ M	Ambient
Moderate	Up to 1 M	Below 100 °C
High	Around 1 M	Around boiling
Very high	Near saturation	Above boiling

3. The fracture mode is classified as intergranular (I) where cracks go along the grain boundaries, transgranular (T) where cracks go across the grains, or mixed (M) where there is a combination of the two modes, or where the mode can vary depending on the conditions. There are often circumstances that can cause the fracture mode to change (e.g., chloride SCC of sensitized austenitic stainless steel may give intergranular cracking).

SCC is a complex process with a sensitivity to a multiplicity of interacting environmental, mechanical, and material variables (see Chapter 6.01). It is driven by the local environment, mechanics, and material characteristics. Cracks tend to initiate at microstructural and microchemical inhomogeneities, at stress concentrators, and at locations where modification of the bulk environment has occurred. Indeed, for many systems, crack initiation may not occur directly from exposure to the nominal bulk solution as such but only if preceded by pitting, crevice attack, or intergranular corrosion. These processes create stress

concentrators and, more importantly, establish the critical local solution chemistry for cracking. Once cracking has initiated, the local environment in the crack continues to determine the local electrochemical kinetics and the nature and stability of surface films.

Failures have occurred in almost every industrial sector, and the occasional catastrophic nature of the failures (McHenry *et al.*, 1987) is a permanent reminder to the materials and engineering community of the need to predict the likelihood of cracking and the impact on structural integrity. There is a wealth of knowledge on SCC including numerous ISO testing standards, conference proceedings (Gangloff and Ives, 1990; Shipilov, 2006), and guides (Cottis, 2000). Accordingly, SCC should be a rare event if information were disseminated effectively. Nevertheless, cracking may still occur for the following reasons:

- the operating conditions in service may be altered, for example, for economic reasons;
- transient variations in stress, temperature, or environment chemistry may occur, either from scheduled excursions (e.g., shutdown) or from unintentional fluctuation in system control (e.g., contamination);
- the character of the metal surface may change with time of operation (e.g., precipitation of a scale or deposit) or the material may age (e.g., irradiation effects);
- welding may not be ideal and there may be departures from the weld procedure specification;
- localized corrosion processes, such as pitting, crevice corrosion, or intergranular corrosion, may be initiated and become precursors for SCC;
- concentrated solutions may be generated by evaporation or boiling;
- laboratory testing and predictive tools may be inadequate or of insufficient quality or relevance; and
- monitoring and inspection may not be adequate.

It is evident that there are challenges in design, materials selection, fabrication, environment control, crack detection, system management, and risk assessment in relation to SCC. Fundamental to addressing these challenges is the generation of reliable and relevant laboratory test data. Test methodology is critical to that process. Reliable data enable an informed perspective on the likelihood of crack initiation and subsequent crack propagation rate, limiting unwarranted conservatism at the design stage.

Material	Environment	Concentration	Temperature	Mode
Carbon steel	Hydroxides	High	High	1
	Nitrates	Moderate	Moderate	Ι
	Carbonate/Bicarbonate	Low	Moderate	Ι
	Liquid ammonia		Low	Т
	$CO/CO_2/H_2O$		Low	Т
	Aerated water		Very high	Т
Low alloy steel (e.g., Cr-Mo, Cr-Mo-V)	Water		Moderate	Т
Strong steels	Water ( $\sigma_v > 1200$ MPa)		Low	Μ
C	Chloride ( $\sigma_v > 800 \text{ MPa}$ )		Low	Μ
	Sulphide ( $\sigma_v > 600 \text{ MPa}$ )		Low	Μ
Austenitic stainless steels	Chloride	High	High	Т
(including sensitized)	Hydroxide	High	Very high	Μ
Sensitized austenitic stainless	Aerated water	÷	Very high	1
steels	Thiosulphate or Polythionate	Low	Low	I
Duplex stainless steels	Chloride	High	Very high	Т
-	Chloride + $H_2S$ high	High	Moderate	Т
Martensitic stainless steels	Chloride (usually $+H_2S$ )	Moderate	Low	Т
High-strength aluminum	Water vapor		Low	Т
alloys	Chlorides	Low	Low	Ι
Titanium alloys	Chlorides	High	Low	Т
-	Methanol	-	Low	Т
	$N_2O_4$ high		Low	Т
Copper alloys (excluding Cu–Ni)	Ammoniacal solutions and other nitrogenous compounds	Low	Low	I
	Nitrate	Moderate	Low	Т
	Cupric sulphate	Moderate	Low	Т
Nickel-base alloys	Water		Very high	I
-	Caustic	High	Very high	Ι
	Pb <sup>2+</sup>	-	Very high	Ι
	Chloride + $H_2S$	High	Very high	I

 Table 1
 Stress corrosion susceptible material-environment combinations

#### 11.03.2 GENERAL ASPECTS OF SCC TESTING

There is no generalized analytical approach based on micromechanics, chemistry, and physical metallurgy that would allow prediction of combinations of material and environment conducive to SCC. Avoidance of SCC during service or prediction of the rate of damage accumulation is based largely on past experience and laboratory testing. The challenge, especially in new design, is to predict long-term structural performance from laboratory experimental data obtained over relatively short time periods. Simulating service environments can be difficult and extensive large-scale testing may be prohibitively expensive. Nevertheless, it is essential that the relevant service conditions are represented as effectively as possible in terms of material characteristics, environment, and loading conditions.

However, the detailed representation of these variables depends on the objectives of SCC testing. The more important of these are (Turnbull, 1992):

- materials selection;
- generation of design data;

- quality assurance;
- alloy development;
- evaluation of protective schemes; and
- assessment of relative aggressivity of different environments.

The investigation of mechanisms of SCC represents a complementary activity.

For preliminary materials screening, it may be sufficient to adopt a more aggressive environment or loading mode than in service, provided the basic mechanism of failure is the same. Nevertheless, this would usually be followed by more discrete testing of candidate materials in conditions closer to those expected in service.

#### 11.03.2.1 SCC Test Philosophies

The various SCC test methodologies can be distinguished often on the basis of two design philosophies underlying structural design (Wanhill, 1991):

- the safe-life approach, and
- the damage-tolerance approach.

In the more traditional safe-life approach, structures are designed on the basis of a finite service life with no damage, viz. cracking, developing. Assessment is based on a go/no-go-type evaluation of test results. Since crack development is presumed to have been designed out, there is less emphasis on routine inspection. An example of such an approach would be oil and gas pipelines on the seabed for which regular inspection is not feasible.

The damage-tolerant approach, which has subsequently evolved, requires that any initiated crack remain at a size below the critical size for unstable fracture over the design life of the structure. This can be achieved by ensuring that any pre-existing crack-like defect does not propagate or, if the crack does propagate, that the crack growth rate as a function of crack depth and loading condition is sufficiently well known. Routine inspection and fracture mechanics assessment is imperative. Examples of application of the damage-tolerant approach include pressure vessels and turbine materials in the power industry, and various aircraft components.

## 11.03.2.2 Specimen Configuration and Loading Mode

To reflect the different approaches in laboratory SCC evaluation, testing has been classified (Turnbull, 1992) in terms of either smooth (i.e., plain) or precracked specimens. Testing of smooth specimens would represent a situation in which no initial flaws would be present, whereas the use of precracked specimens would account for the existence of flaws or cracks according to the damage-tolerant approach. This simple discrimination breaks down when considering notched specimens and in testing of welds in the as-welded state. In addition, more use is being made of largescale simulation as exemplified by testing of some pipeline material. The more meaningful distinction is between non-precracked specimens and precracked specimens.

Within that broad classification, the loading mode has also to be considered, and thus four categories can be envisaged:

1. non-precracked specimens – fixed load or fixed displacement tests;

2. non-precracked specimens – dynamic (increasing) load tests;

3. precracked specimens – fixed load or fixed displacement tests; and

4. precracked specimens – dynamic (monotonic) load tests.

Each of these tests has its merits according to the objectives of the test and the need for

qualitative or quantitative information. For example, a material may be immune to SCC under static loading but highly susceptible plastic continuous slow under straining (Parkins, 1972). The use of precracked specimens ensures a conservative approach to the acuity of any physical defects introduced in fabrication and allows crack growth rate determination, at least in the long crack regime. It can also highlight susceptibility of those alloys that depend on development of a localized chemistry for SCC to ensue. For example, SCC of titanium alloys in aqueous solutions may be observed using precracked specimens, which creates a local microenvironment (Wanhill, 1975) that would not be evident from tests under constant load on plain specimens. There are particular challenges in testing of welds in the context of precracked specimens as the location of the crack tip relative to the microstructurally sensitive region could be mismatched. For example, if the tip of the precrack is in the heat-affected zone but the susceptible microstructure is at the fusion boundary, the results may be nonconservative.

On the other hand, the use of specimens that have not been precracked can be more representative of actual surface conditions, provided the surface state reflects that in service. In that context, consideration has to be given to surface preparation with respect to service application and simulated laboratory testing. The process of grinding the surface introduces local microstructural damage and residual stress. For example, even light grinding of solution-annealed stainless steel can result in tensile residual stresses approaching 500 MPa.

The method of testing of welds remains controversial with respect to the use of ground or as-welded specimens. Testing in the as-welded state has the advantages of the natural topography, including features such as step-height changes associated with pipe misalignment, height of root protrusion, natural notches, as well as the natural characteristics of the oxide film, but repeatability may be more varied and variations in thickness along the welded region could be an issue for some tests. An additional factor that has to be considered in testing of welds is the different properties of the weld metal, heat-affected zone, and parent material, which can result in differential strain in the specimen when testing near yield.

The adoption of notched specimens can be controversial as the depth, acuity, and root radius are often arbitrary. In addition, as with precracked specimens, when applied to welds the location of the notch tip relative to the microstructural features in the heat-affected zone may affect the apparent susceptibility of the material. Furthermore, notching can introduce residual stress or microstructural damage at the root depending on how the notch is introduced, for example, mechanical cutting, electric discharge machining, or wire grinding (in case of micronotches).

While much testing is undertaken in conditions of applied stress, additional testing in corrosive environment may be carried out with unstressed samples, to evaluate, for example, the existence of residual stresses and their effect on crack formation and development.

The various methods of conducting SCC tests are specified in detail in the appropriate test standards issued by ASTM, ISO, NACE, and other organizations. Here, only the general characteristics of the various SCC test methods are described. Testing of non-precracked and precracked specimens is considered separately because of their distinctive geometric characteristics and the kind and amount of information they can provide. A detailed overview of the various specimen types and a discussion of the many aspects and problems associated with SCC tests using these various specimen types and loading modes can be found in Turnbull (1992).

#### 11.03.2.3 Environment Control

Whichever type of test configuration is used, it is often the case that the greatest source of variability in results is associated with limitations in environmental control during the test. The three key features are water chemistry, flow rate, and temperature. While temperature has usually been well controlled, water chemistry and flow rate are variables which have often been ill-defined, poorly controlled, and/or monitored. The fundamental requirement in water chemistry control is to ensure that the test environment reflects the intended environment (service simulation or simply a research environment) for the duration of the test. This includes not only the solution composition and pH but also the dissolved gas content.

Clearly, the ratio of volume of solution to metal area and the rate of reactions determines the rate of change of the bulk environment composition. In preparing the solution, the baseline water quality depends on the application, with distilled or deionized water being adequate for preparing artificial seawater for example, but high-purity water of conductivity less than  $0.1 \,\mu\text{S cm}^{-1}$  is often necessary for power industry applications. It is also important to recognize that the chemicals used should be at least of analytical reagent-grade quality. It is prudent to check their assay and to consider a higher grade where the concentration of minor constituents might be significant, as when using concentrated solutions. This is a consideration also when testing in strongly acidic or alkaline solutions.

In addition to testing under controlled, steady, environmental conditions in the laboratory, it is important to recognize that SCC may initiate in service from transient conditions, such as variations in oxygen and chloride concentration. Recent evidence (Ritter and Siefert, 2004; Zhou and Turnbull, 2006) suggests that once corrosion or SCC has initiated, restoration to normal operating chemistry in service does not necessarily result in a reduced crack growth rate or crack arrest. More work to account for solution chemistry transients and indeed temperature transients is required.

Where feasible, the electrode potential should be monitored. Monitoring of the bulk solution pH is also important. Conductivity measurement should be made when testing solutions of initially low conductivity to ensure that this is maintained. The extent to which the monitoring is on-line or involves a sampling process on a regular basis is system specific. Clearly, oxygen monitoring should be an on-line process in most cases. Deaeration is a requirement in many tests but too often the extent of deaeration is not defined or measured. In many systems, use of plastic tubing can severely limit the extent of deaeration. For testing in H<sub>2</sub>S environments for oil and gas applications, enclosure of the test system in a nitrogen cabinet may be essential to achieve sufficient deaeration, typically less than 10 ppb, but ideally to about 2 ppb. At elevated temperatures in low-conductivity water, carbon steels exhibit a rapid transition in corrosion potential at oxygen concentration between about 30 and 60 ppb. Thus, control of the oxygen concentration is critical.

The most challenging tests are those designed to simulate boiling and evaporative conditions as it can be particularly difficult to simulate all features of service conditions. This means setting up simulated evaporation tests as best as can be reasonably achieved or testing in simulated concentrated environments.

The solution flow rate adopted in tests is often not considered but can affect several factors: the corrosion potential, the near-surface solution chemistry, the solution chemistry in a crack, and, on a more mundane level, maintenance of the bulk solution chemistry in a complex recirculation loop. In well-stirred solutions, mixing ensures maintenance of the bulk chemistry at the metal surface, whether in service or in the laboratory. The possibility of solution chemistry changes local to the metal surface is certainly an issue for steel in the active state with a high corrosion rate. In the passive state, with a relatively low dissolution rate, surface chemistry changes would tend to be more modest but would depend on the magnitude of the passive current density.

Where possible, the corrosion potential should be monitored, though in undertaking standardized testing such as the NACE TM0177 tests, this may not be readily feasible and may not be so critical because of the defined nature of the test.

#### 11.03.2.4 Time-Dependent Issues

Laboratory tests are inherently short compared to the lifetime of the component or structure but it is important to be aware of factors that can compromise the results if the test time is too short and leads to a nonconservative prediction. Typical time-dependent processes of relevance include localized corrosion, hydrogen uptake, changes in the characteristics of surface films with exposure time, transient immersion, irradiation damage in nuclear applications, and the formation of grain boundary oxides over long periods.

The measurement issue with localized corrosion arises because of the conflict between minimizing exposure time, to reduce testing costs, and the recognition that the depth of attack will progressively increase with time, albeit at a continually decreasing rate.

In relation to hydrogen uptake, a fundamental question is how long should the duration of a laboratory test be to ensure that hydrogen uptake is sufficient to reflect behavior in service, for which exposure times are of the order of years. To illustrate this point, the effective diffusivities of hydrogen in various steels (Turnbull, 2001) at 20 °C are summarized in **Table 2.** These data represent values for similar cathodic polarization conditions. It should be emphasized that the effective diffusivity is dependent on the extent of hydrogen uptake and should not be regarded as a constant at a particular temperature. The spread in values in **Table 2** is about 5 orders of magnitude. Hence, there can be potential problems in laboratory testing in ensuring that representative data are obtained with respect to steady-state conditions of hydrogen charging. The key issue is the distance between the site of cracking and the primary source of hydrogen atoms. If the latter

 
 Table 2
 Hydrogen atom diffusivities for various steels under cathodic protection at ambient temperature

Material	$D_{\rm eff}  (cm^2  s^{-1})$
Pure iron	$7.2 \times 10^{-5}$
AISI 4340	$1.7 \times 10^{-7}$
13Cr MSS	$6.0 \times 10^{-9}$
22Cr DSS	$2.8 \times 10^{-11}$

is remote, then test times need to reflect this or pre-exposure should be considered. To predict the primary source of hydrogen atoms, some insight into the electrochemistry of the system is necessary. The kinetics of hydrogen atom generation can vary between the crack tip and the surface external to the crack because of differences in local pH, concentration of other species (e.g.,  $H_2S$ ), and electrode potential. The significance of these factors depends on whether the alloys are in the active or passive state (see Chapter 6.02).

For alloys in the active state, that is, with no protective passive film on the surface, hydrogen entry is not limited to local sites but can occur over the whole surface. The kinetics of reaction on the external surface are likely to dominate at the more negative cathodic potentials and in solutions which contain a reactive species that would be readily consumed in the crack before reaching the crack tip (Turnbull and Saenz de Santa Maria, 1990). A bulk solution which is acidic or which contains  $H_2S$  would be an example of the latter.

Consequently, when utilizing fracture mechanics specimens, it is necessary to consider the primary source of hydrogen atoms. If the source is on the surface external to the crack, it is important to calculate the time for the externally generated hydrogen atoms to diffuse to the crack front and attain the steady-state concentration. A rough estimate can be made using Fick's law and an appropriate effective diffusivity determined for equivalent charging conditions. For low-alloy high-strength steels, the exposure time may be as long as a year for a 25 mm thick compact tension (C(T)) specimen. Recognizing this factor, it is essential to calculate the time to steady state, to pre-expose specimens when appropriate (if the diffusivity is low), and to use the lowest thickness compatible with maintaining plane-strain conditions when using fracture mechanics specimens.

Reliable measurement of hydrogen embrittlement of corrosion-resistant alloys in the passive state is a matter of some uncertainty and controversy. Diffusion coefficients are particularly low for most austenitic and ferriticaustenitic duplex alloys (Table 2), and the time to charge specimens to steady state could be very long, except at high temperatures. In that context, it should be emphasized that, for corrosion-resistant alloys, cracking is not necessarily dependent on general hydrogen charging unless crack initiation is at internal microstructural sites or defects. This is because the presence of an oxide film has a major retardation effect on hydrogen entry at ambient temperatures and hydrogen entry is most significant in regions of localized dynamic straining. Hence, when the metal is in the passive state, it may reasonably be assumed that longterm exposure is not required in most cases.

#### 11.03.3 NON-PRECRACKED SPECIMENS

In conducting a test program to select a material for engineering service, based on the concept of safe life, usually one or two types of tests are adopted, often based on a standard for the industry. In contrast to a single qualification test, a more advanced approach would be to identify the envelope of meaningful conditions under which SCC might occur and the likelihood of overlap with service operational conditions. This approach focuses more attention on the concept of intelligent risk assessment.

A wide variety of plain, notched, or aswelded specimens are in use for SCC testing, and are described in detail in the various test standards. Apart from the generally used tensile specimens, many of these specimen types are associated with the corresponding method of mechanical loading as described below.

#### 11.03.3.1 Constant Load Tests

The most important characteristic of these tests is that the load is maintained constant throughout the whole period of testing, in contrast to constant-displacement testing, for which some load relaxation occurs. The specimens in constant load tests can be cylindrical or flat, sometimes notched, sometimes as-welded, and are usually loaded in tension. In principle, there are two kinds of constant load tests, that is, a specimen in a self-loading frame with a spring system to provide quasi-constant stress (e.g., proof ring) and a specimen loaded by an external load frame.

Testing with an external load frame is often done with deadweight loads and is the most reliable method. The number of load frames required can be minimized by testing chains of specimens which may be connected by loading links suitable to prevent unloading upon failure of one of the specimens. However, testing can be expensive when a range of variables or materials needs to be evaluated. The more compact quasi-constant stress testing frames are cheaper and take up less space. Whichever method is used, consideration has to be given to the detailed procedure. Preloading prior to expoto the environment can sure lead to nonconservative results if creep is important. For example, duplex stainless steels undergo significant creep and the period between loading and exposure to the environment may have an influence on apparent susceptibility as the creep rate will be changing with time. For quasi-constant stress systems, loading of the specimen usually precedes exposure to the environment, whereas with deadweight machines the load can be applied on exposure.

In constant-load SCC tests, usually the time to total failure,  $t_{\rm f}$ , is evaluated for a variety of applied initial stresses, thus providing an estimate of the threshold stress,  $\sigma_{th}$ , below which total failure due to SCC does not occur (Figure 1). The stress determined by this method is not the threshold stress for initiation per se (though it could be) but the threshold stress below which any initiated crack does not propagate. This is the appropriate definition of the threshold stress, similar to the concept of the fatigue strength in fatigue testing. However, for pragmatic reasons, the tests are terminated after an arbitrary chosen time and confidence in attaining a threshold cannot be 100%. Thus, the surface is often inspected for cracks. The existence of a crack may be considered to represent a failure since there is uncertainty as to whether the crack is nonpropagating. For oil and gas applications, it is common also to load the specimens to 90% of the actual 0.2% proof stress, usually for 30 days. Observation of any cracking at the appropriate magnification is considered a failure.

It is sometimes argued that there is no such concept as a threshold stress for cracking. This is somewhat misleading and is based often on tests on plain specimens where SCC is preceded by intergranular corrosion or pitting such that the stress required to initiate a crack decreases with exposure time as the depth of the damage increases. However, there are many situations where SCC is not preceded by such damage and stress is required to activate localized straining



**Figure 1** Influence of the initial stress,  $\sigma_{init}$ , on the time to failure,  $t_f$ , measured in constant-load tests on tensile specimens of the high-strength aluminum alloy AA 2024 T351 in a chromated 3.5% sodium chloride solution (Dietzel, 1991).

sufficient to rupture protective oxide films. In these cases, the concept of a threshold stress is clearly meaningful. However, it is recognized that there is a time factor in such assessment and it is feasible that long-term changes in the material could induce failure not picked up in short-term tests. This does not preclude the concept of a threshold stress but simply qualifies it relative to the test methodology.

An approximate estimate of the crack velocity can be derived from these tests. The depth of largest crack is measured on the fracture surface, or on sections through a specimen that has not proceeded to total failure. The crack velocity is then estimated by dividing the crack depth by the time of testing. This parameter assumes that crack initiation occurred at the start of the test and that the deepest crack also initiated at that time, which is not necessarily the case. Thus, SCC crack growth velocities are typically underestimated by this method, and this becomes an untenable approach if localized attack is a precursor to cracking. A more accurate measurement of the average crack growth velocity is feasible if crack initiation can be detected by using a crack monitoring system, for example, one of the electrical resistance measurement methods discussed in Section 11.03.5.1.

#### 11.03.3.2 Constant-Displacement Tests

Constant-displacement tests – sometimes also called constant-strain tests, which is not entirely correct - are widely used. The specimens are stressed in a self-loading frame. Although SCC testing under constant displacement is straightforward and inexpensive, it has some disadvantages: The specimens are usually loaded before environmental exposure, and again this preloading may lead to nonconservative results due to stress relaxation induced by creep. However, the extent of relaxation depends on the loading mode. In a bend specimen, relaxation may be less significant because only the outer fibers may be highly stressed and there is a stress gradient through the specimens. The underlying elastically stressed material tends to resist deformation of the outer fibers. Thus, creep may be constrained, in contrast to a constant-displacement tensile test or a constant-load test.

The most common types of smooth specimens used in constant-displacement tests are U-bend, bent beam, and C-ring specimens, but self-loaded tensile specimens are also used. The preparation and use of these specimens are described in ISO, ASTM, and other standards and procedures (see Section 7.02.5.8.1).

U-bend and the reverse U-bend (taking a pipe section and bending it so that the inner surface is in tension) are particularly severe tests with high levels of plastic strain. The reverse U-bend test is currently under development as an ISO standard. It is used primarily in the nuclear industry.

The appropriate standards should be consulted for the details for all types of tests. However, advances in the method of testing are being made, especially with respect to testing of corrosion-resistant alloys, both plain parent material and welded specimens, and these advances are yet to be incorporated in the standards. These alloys show a departure from linearity in their stressstrain behavior with no defined yield stress. Correspondingly, when testing using three- or four-point bend or C-ring specimens, calculation of the required deflection based on elastic response of the material is not applicable. Accordingly, it is necessary to strain-gauge the specimen to determine the required deflection. For testing of plain parent material, this needs only to be done once for a group of similar specimens, but for welds it is necessary to strain-gauge each specimen on both sides of the weld.

For four-point bend specimens, the objective is to achieve a specific value of the strain (typically 0.2% plastic strain) in the plain parent plate specimen or adjacent to the weld, as appropriate. To achieve that, the total strain (elastic and plastic) to give that degree of plastic deformation needs to be identified using flexural data obtained in a separate test on the parent material (see example of Figure 2). (Load and force are used interchangeably in this document according to the common usage in application of the different test methods and should be considered formally equivalent.) In testing of welds, deflection is stopped when the first strain gauge registers the required total strain value. Any specimen strained beyond the intended level should be discarded.

It is advisable to generate the stress-strain behavior using actual bend specimens of the



**Figure 2** Illustration showing determination of total strain ( $\varepsilon_{tot}$ ) to be applied to achieve 0.2% plastic strain ( $\varepsilon_{pl}$ ) in four-point bend testing; the elastic strain is  $\varepsilon_{el}$  (Turnbull and Nimmo, 2004).

parent material. Tensile stress-strain data may not be appropriate and may give significant errors for some systems.

#### 11.03.3.3 Slow Strain Rate Tests

The slow strain rate test (SSRT), also called the constant-extension rate test (CERT), is widely used (Parkins, 1979) and is considered as a severe SCC test though, as discussed below, this can be a misleading concept. The test involves loading a specimen under conditions of a progressive increase in strain while it is exposed to a specified environment. SSRTs may be conducted in tension or in bending, on initially plain or notched specimens.

In most slow strain rate testing, the elongation of the gauge section of the specimen is not measured directly. Rather, the test method involves the application of a constant displacement rate to the crosshead of a tensile test machine. Thus, the total displacement includes a contribution from the displacement of the shoulders of the specimen and of the load train. Because these displacements can vary from one test system to another, the calculated strain on the gauge section of the material at any time is sensitive to the test system. In one system (Hinds and Turnbull, 2006), the actual strain measured in the elastic region (by strain-gauging) was 15 times smaller than that calculated conventionally from the crosshead displacement measurement. In other words, the displacement of the specimen in the elastic region was in this case small compared to that of the load train, which was highly compliant. The strain rate of the specimen in the elastic region was also about a factor of 15 less than the nominal value and would be expected to vary from one test system to another, despite similar values of the nominal strain rate. However, once yielding occurs, most of the increase in displacement in the crosshead is associated with the plastic deformation of the specimen gauge section and use of the nominal strain rate is more reasonable (up to the onset of necking). For this situation, the differences between test systems should be less significant, though work hardening means that there is still some contribution from the elastic displacement of the load train.

For initially smooth specimens, the strain rate at the onset of the test is clearly defined, within the above constraints, but once cracks have initiated and have started to grow, the effective strain rate cannot be measured due to strain localization, though approximate estimates may be derived. Necking in a ductile material stressed in tension is a further example of strain localization, and the effective strain rate in the necked region may increase by as much as an order of magnitude, which can cause the strain rate to move in or out of the critical region.

The most important characteristic of the test is the relatively slow strain rate applied. Yet, a principal advantage of the test is the rapidity with which the SCC susceptibility may be assessed. In many material/environment systems, a nominal tensile strain rate of  $10^{-5}$ - $10^{-6}$  s<sup>-1</sup> is appropriate to promote SCC, but the absence of cracking in tests conducted at such rates should not be construed as indicative of immunity to cracking until tests have been performed at a wider range of strain rates. At very low strain rates, susceptibility may be increased or diminished depending on the mechanism of cracking and the conditions of testing, as illustrated by Figure 3 and, for example, reported by Parkins, 1993. The recovof resistance in certain materialery environment combinations is usually attributed to repassivation effects, which are assumed to be more rapid than the generation of fresh surface areas due to straining.

Comparison between identical specimens exposed to the test environment and to an inert environment may be used for assessing the susceptibility to SCC. The parameters commonly measured are:

- (plastic) strain-to-failure;
- ductility loss, for example, reduction in area or elongation to fracture;
- maximum stress achieved; and
- area bounded by nominal stress/elongation curve representing the fracture energy.



Figure 3 Results of slow strain rate tensile tests performed at various strain rates on the same combination of material and environment as shown in Figure 1; the Y-axis shows the ratio of fracture energy,  $E_{\rm f}$ , measured in environment (solid squares) to fracture energy measured in air (open squares),  $E_{\rm f}({\rm SCC})/E_{\rm f}({\rm air})$ . The recovery of this ratio at very low strain rates, as indicated by the dashed lines, was not observed here, but is reported in the literature and then attributed to repassivation effects, for example, in Parkins (1993) (Dietzel, 1991).

The ratio of the parameter measured in the environment relative to the value measured in an inert environment gives an index of susceptibility. Figure 3 is an example of data generated in a series of SSRTs. These tests could, in principle, also give an estimate of the threshold stress,  $\sigma_{th}$ .

The optimum choice of parameter depends on the characteristics of the SCC failure process and it may be useful to employ more than one in appropriate circumstances. For example, if failure occurs prior to the tensile strength, the choice of reduction in area may not be the most appropriate because the change in area reduction prior to reaching the ultimate tensile strength is usually small. Similarly, for materials that do not exhibit significant work hardening, the maximum stress achieved is not a useful criterion because the stress does not change much with increasing strain. In general, plastic strain-to-failure is a most useful index of susceptibility to SCC. The rationale for the adoption of plastic strain-to-failure is that the calculated strain is less affected by the contributions from the load train as noted above and the strain rate is also closer to the nominal value. Thus, comparison between different test systems and laboratories is more reliable. The method of determination of the plastic strainto-failure can be illustrated using Figure 2 by replacing  $\varepsilon_{tot}$  with the failure strain and using the same analytical approach to separate the elastic and plastic components.

Traditionally, SSRT has been used as a ranking or screening test, or for mechanistic studies, and is considered severe because SCC initiates often at high plastic strains-to-failure or after necking, conditions not likely to be experienced in service. However, it can and should have more significance than that for the simple reason that there are a number of systems in service for which slow dynamic straining may play a key role in SCC. Examples would include pipeline movement, and thermal stresses in pressure vessels and associated pipe work during startup. For such cases, it is important to explore the effect of strain rate and assess whether failure occurs at stresses that might be achievable in service. For some systems, failure has been shown to occur close to the 0.2% proof stress if the strain rate is low enough (Hinds and Turnbull, 2006). The judgment in evaluating the risk of failure is to assess the likelihood of such strain rates and to account for the role of the service history. The latter is important for work-hardening materials as prior strain will affect the extent of plastic flow at a given applied stress (see below).

Cyclic slow strain rate testing (similar to ripple loading) was introduced (Erlings *et al.*,

1987) as a means of sustaining dynamic straining without an increase in total strain. In the cyclic SSRT, the stress on the specimens is cycled between preset load limits at a strain rate of  $10^{-6}$  s<sup>-1</sup>. The load limits can be varied according to the purpose of the test but initially were based on cycling between 80% and 100% of  $R_{P0,2}$ . Since the minimum and maximum loads are fixed, the minimum and maximum strain values will change because of the induced work hardening. The cyclic frequency would be typically  $1.25 \times 10^{-4}$  Hz but the cycle period typically decreases slightly during the test since it is the strain rate, not the frequency, that is usually controlled. Cyclic loading is maintained typically for a period of 14 days corresponding to about 150 cycles. At the end of this period, the specimen is removed and inspected for cracks at a magnification of  $40 \times$  using low-powered optical microscopy. Although limited studies have been conducted using this approach, the application to work-hardening corrosion-resistant alloys (duplex stainless steels and martensitic stainless steels) suggests that the test has little merit for such materials. These materials work-harden so much in the first cycle that subsequent loading is wholly elastic (apart from some creep of the duplex stainless steel). Nevertheless, the observation highlights the potential role of service history in determining the likely susceptibility of materials to SCC under slow dynamic straining conditions. Dynamic straining, in the nonlinear region, at a rate not conducive to SCC can reduce the likelihood of cracking if followed by dynamic straining at a much lower, and potentially more damaging, rate because the loading will then be predominantly elastic, within the bounds of likely service stresses.

Average stress corrosion crack velocities for initially plain specimens may be estimated approximately from the depth of the deepest crack measured on the fracture surfaces of specimens that have failed or sections through specimens for which the test had been interrupted prior to failure, divided by the time of testing or the time from onset of plastic deformation. As in the case of constant-load experiments, caution should be taken since SCC crack growth velocities are typically underestimated by this method due to the fact that the exact time of crack initiation is not known. It is not commonly used for engineering purposes. Here again, the use of an electrical resistance measurement technique, as described in Section 11.03.5.1, can help to determine crack initiation and to give a better estimate of the SCC crack growth velocities, as has been shown by Strieder et al. (1994).

#### 11.03.3.4 Breaking Load and Linearly Increasing Stress Tests

The breaking-load and linearly increasing stress tests (LISTs) are not standardized, but are interesting alternatives to the above-discussed methods, to which they are to some extent complementary. In the breaking-load test, the specimens are self-loaded or stressed in a self-loaded frame, exposed to the environment, and are subsequently subjected to a residual static strength test, which quantifies the damage due to SCC (Sprowls et al., 1984). A further development of this test method is the ASCOR (automated stress corrosion ring) test proposed by Schra and Groep (1993). Cylindrical or sheet specimens are stressed in strain-gauged loading rings and subjected to the corrosive environment. Continuous monitoring of the load enables the detection of crack initiation and the construction of stress-lifetime curves.

The LIST, introduced by Atrens et al. (1993), is similar to the SSRT. One essential difference is that LIST is under load control whereas the SSRT is under displacement control. In the LIST, the specimen, exposed to the environment, is subjected to a linearly increasing (engineering) stress until the specimen fractures. The SSRT and LIST are nominally identical in the elastic region (though the stress on the specimen is well-defined, the strain on the specimen in most SSR tests is not because of the dominance of displacement in the load train) and are similar in the region of early crack growth, although the rates of the two tests become different as soon as there is some crack initiation or some plasticity. The LIST is completed when the specimen fractures. Fracture occurs in the LIST when the stress corrosion crack reaches the critical crack size as determined by the applied stress. At this crack size, the SSRT specimen is still in a stable condition, and a significant additional testing period can be required in the SSRT. Thus, a stress-strain curve measured in an LIST terminates at the maximum stress, whereas the stressstrain curve in an SSRT can continue long after the maximum stress. Consequently, over a third to half the test duration in an SSRT can occur after the maximum stress. The stress corrosion cracks yaw open in the SSRT during this period. This means that an LIST can save 1–2 days of testing time for an SSRT of duration 3-4 days.

Ramamurthy and Atrens (1993) have shown that LIST allows a relatively straightforward measurement of the threshold stress for SCC,  $\sigma_{th}$ , using an electrical potential drop technique. Subsequently, Winzer *et al.* (2006) have shown that the SSRT can also be used to measure the threshold stress for SCC when using this electrical potential drop technique: at the threshold stress the measured potential drop begins to increase faster than linear when the potential drop is plotted against the engineering stress in the specimen.

Thus, both LIST and SSRT can identify the occurrence of SCC and both can measure the threshold stress for SCC. The LIST is often one-third to one-half shorter in duration. The longer duration of the SSRT means that there is larger area of SCC on the fracture surface. In contrast, the LIST allows identification and study of the early stages of stress corrosion crack growth.

It should, however, be noted that the threshold stress for SCC measured in an LIST may be somewhat nonconservative compared with service. For example, Australian natural gas transmission pipelines have been operated such that the stresses in service are maintained below the threshold stress for SCC as part of their structural integrity strategy (Atrens et al., 2004). The threshold stress has been typically evaluated with the approach suggested by Parkins et al. (1993) of the specimen subjected to a sustained load plus a slow (typically  $\sim 10^{-4}$  Hz) ripple of 10-20% while the specimen was exposed to a 'high'-pH environment  $(1 \text{ N} \text{ Na}_2\text{CO}_3 + 1 \text{ N})$ NaHCO<sub>3</sub> at 75 °C and  $-0.65 V_{SCE}$ ). With this approach, the threshold has been evaluated to be  $\sim 80\%$  of the yield stress (Atrens et al., 2004; Parkins et al., 1993). In contrast, the LIST gave a threshold stress for SCC somewhat above the yield stress for similar pipeline steels exposed to the same 'high'-pH environment (Wang and Atrens, 1996). Thus, the LIST can give a quick indication of the service threshold stress, provided its limitations are taken into account.

#### 11.03.4 PRECRACKED SPECIMENS – THE FRACTURE MECHANICS APPROACH TO SCC

In damage-tolerant design practice, initial flaws already existing or assumed to exist in a structure or component are taken into account, and fracture mechanics concepts are applied to characterize the initiation and growth of cracks from these flaws. This includes cracks which are caused or enhanced by a corrosive environment. Usually, stress corrosion cracks are considered to be brittle, that is, they occur generally at stresses below general yield and propagate in an essentially elastic body, even though local plasticity may be necessary for the cracking process. Hence, linear elastic fracture mechanics (LEFM) concepts are applied, and the (planestrain) stress intensity factor in the opening mode,  $K_1$ , is used to quantify the stress situation at the crack tip in a precracked specimen or structure which controls the initiation of a

crack and its further extension. For more ductile alloys and thinner sections for which the principle of small-scale yielding becomes invalid, elastic-plastic fracture mechanics (EPFM) is more appropriate, and EPFM parameters such as the crack-tip opening angle/crack-tip opening displacement, CTOA/CTOD, and the *J*-integral are used to define the crack tip stress state (see Chapter 11.02).

#### 11.03.4.1 Linear Elastic Fracture Mechanics

Experimental evidence has shown that, for a given material/environment combination, a unique relation exists between  $K_1$  and the growth rate of a stress corrosion crack. The usual way of representing this relationship is to plot crack growth rates, da/dt (or v), as a function of  $K_1$  (Speidel, 1971). In the typical schematic diagram, that is, the v-K curve shown in **Figure 4**, a lower shelf value of  $K_1$ , that is, the stress corrosion threshold,  $K_{1SCC}$ , characterizes the magnitude of the stress intensity factor at which the first measurable crack extension occurs.

Under increasing K conditions, once the threshold,  $K_{ISCC}$ , is exceeded, cracking initiates, and the growth rate becomes strongly dependent on K (region I). The  $\nu$ -K curve may pass through a distinct plateau at which the crack growth rate is virtually independent of K. In this region (region II), transport and electrochemical processes are the rate-limiting parameters,



Figure 4 Effect of the stress intensity factor,  $K_{\rm I}$ , on the stress corrosion crack growth rate, da/dt in the region of subcritical crack growth (schematic). After Speidel, M. O. 1971. Current understanding of stress corrosion crack growth in aluminium alloys. In: The Theory of Stress Corrosion Cracking (*ed.* J. C. Scully), pp 289–344. NATO, Brussels.

controlling the crack growth kinetics and determining the absolute level of the plateau. A second region strongly dependent on K (region III) occurs as the critical stress intensity approaches the stress intensity factor corresponding to unstable fracture. Here, pure mechanical rupture dominates over subcritical crack extension caused by SCC. This situation is often associated with attainment of the fracture toughness of the material in air,  $K_{\rm lc}$ , but it is not necessarily selfevident that the K value for unstable fracture of a running crack is the same as that determined in a conventional fracture toughness test and indeed may be lower. In investigations of certain material/environment combinations, regions I and III may be absent; in other cases, no real plateau may be observed.

The parameter  $K_{1SCC}$  is defined as the threshold stress intensity factor for sustained crack growth in the sense that above this value any initiated crack will continue to grow, and below. growing cracks will arrest. However, while the definition is clear, there are intrinsic measurement issues that create uncertainty as to its application. For example, crack arrest is conceptually well understood but demonstrating experimentally that a crack has stopped depends on the resolution of the crack-measurement method, its long-term stability, and the period of time over which measurement is made. Thus, it is possible pragmatically only to define the threshold in terms of an acceptably low crack growth rate. This definition of acceptably low crack growth rate should not be arbitrary but should be related to the application and crack detection and monitoring capability. For example, a growth rate less than  $10^{-10} \,\mathrm{m \, s^{-1}}$ corresponding to a crack increment of roughly 3 mm per year, is sometimes used in defining a threshold. This is a relatively high crack growth rate and is more for laboratory convenience than for practical assistance to industry. Stress corrosion crack growth rates as low as  $3 \times 10^{-12} \text{ m s}^{-1}$  have been measured in some systems (Zhou and Turnbull, 2006), and crack growth rates much lower than this may become of particular importance, for example, for nuclear waste depository systems where structural integrity of the containers has to be guaranteed for 10 000 years or even more.

The value measured for the threshold can also vary according to whether an increasing K or decreasing K test is conducted, depending on the methodology used. In an increasing K test, the crack evolves from a fatigue precrack which will have a crack-tip opening different from that of the subsequent stress corrosion crack. Furthermore, the former will be transgranular while the SCC mode may be intergranular, so that the initiation of a stress corrosion crack

#### Stress Corrosion Cracking



Figure 5 Scanning electron micrograph showing the transition from a fatigue precrack (lower region) to a stress corrosion crack (upper region) in an austenitic stainless steel (AISI 316H) in 100 ppm Cl solution at 90 °C; the arrow indicates the direction of crack extension (Dietzel, 1999).

requires a transition in fracture mode. An example of this can be seen in **Figure 5**, where a scanning electron micrograph of the transition region from fatigue precrack to SC crack in an austenitic stainless steel (AISI 316H) in a 100 ppm Cl<sup>-</sup> solution at 90 °C is shown.

#### 11.03.4.2 Requirements of Fracture Mechanics SCC Testing

Fracture-mechanics-based SCC tests are performed with the primary aim of determining the threshold,  $K_{ISCC}$ , and the rate of crack growth, da/dt. The specimens used to achieve this goal contain initial cracks, so that the stage of crack formation from an initially smooth surface is precluded, and the problem of separating the environmental influence on both crack initiation and extension is avoided.

It is not fully clear to what extent the presence of the corrosive environment affects the plastic deformation that is required for the initiation of SCC, though evidence reported by Cooper (2001), Bruemmer and Thomas (2001), and Dietzel and Schwalbe (1990) suggests that SC crack-tip openings are much smaller than predicted by LEFM. Nevertheless, current fracture-mechanics-based SCC test standards demand that the specimen dimensions must be sufficient to maintain predominantly triaxial (plane-strain) conditions in which plastic deformation is limited to the vicinity of the crack tip. The minimum requirements with respect to specimen thickness, B, precrack length, a, and ligament length, W - a (W is the specimen width), are thus

in accordance to those applied to plane-strain fracture toughness tests in air, that is,

$$a, B, (W-a) \ge 2.5 \left(\frac{K_1}{\sigma_y}\right)^2$$
[1]

where  $\sigma_{\rm v}$  is the yield strength of the material.

Because of the time dependence of the SCC process, SCC tests must allow for sufficient time according to the kinetics of the mechanics/metallurgy/chemistry interactions and related transport factors causing crack initiation and growth. For some materials, notably steels, crack growth incubation times can be very long for low stress intensity factors. Too short a test duration would therefore lead to an overestimation of  $K_{ISCC}$  and to nonconservative predictions. As a consequence, ASTM and ISO standards for determining K<sub>ISCC</sub> from tests under constant load or constant displacement recommend test durations up to 10<sup>4</sup> hours. A new ISO test standard in which a rising load or rising displacement technique is applied can help to some extent to accelerate the laboratory testing of precracked samples (see Section 11.03.4.6) and has some similarity in concept to the slow strain rate and the LIST test in the case of smooth-specimen SCC testing. However, the concept of acceleration should be considered with caution. The test is shorter but different values of  $K_{\rm ISCC}$  may be achieved as a consequence of the different loading conditions because of the sensitivity of the threshold to the applied strain rate (see later). Determination of the threshold is required over a range of crack-mouth opening displacement (CMOD) or loading rates. Furthermore, the shorter durations of this test may be an issue if there is a slow time-dependent process, such as hydrogen uptake from bulk charging. Under these conditions, this test may not properly reflect that and may be nonconservative.

#### 11.03.4.3 Loading Mode

 $K_{\rm ISCC}$  data can be determined using:

- crack initiation tests under constant load,
- crack arrest tests on constantly displaced (self-loaded) specimens, and
- rising load or rising displacement tests.

The essential advantage of constant load, and even more of constant displacement tests, are the moderate requirements with respect to the equipment needed for performing these tests. Nevertheless, a number of shortcomings are inherent to both these modes of loading. Although the necessary test times as recommended in the respective test standards can be quite long, there often remains uncertainty as to whether the K values measured in these tests really represent the threshold  $K_{1SCC}$  value of the material/environment combination. Interlaboratory test programs in which these techniques had been applied to investigate nominally identical material-environment combinations in various laboratories have revealed a high degree of scatter between the obtained values of  $K_{ISCC}$  (Wei and Novak, 1987; Yokobori et al., 1988; Dietzel, 1999). Also, there are cases when there are systematic differences between measurements of  $K_{\rm ISCC}$ under constant load and under constant displacement. Another potential problem arises from the fact that testing is performed under static loading conditions, whereas in some materialenvironment combinations dynamic loading (i.e., increasing plastic deformation) appears necessary to initiate SCC (Ford, 1988). A key unresolved issue is the extent to which cracks continue to grow when the dynamic component ceases but the static load remains. In some systems, crack arrest ensues (Ritter and Seifert, 2004; Zhou and Turnbull, 2006).

#### 11.03.4.4 Constant-Load Tests

A wide choice of constant-load specimen geometries is available to suit the form of the test material, the experimental facilities available, and the objectives of the test. Complete overviews of these specimen types can be found in the respective ASTM and ISO standards (see **Table 3**). The specimens can be used for the determination of  $K_{ISCC}$  by the initiation of a stress corrosion crack from a pre-existing fatigue crack using a series of specimens loaded to different levels of stress intensity, and to some extent for measuring of crack growth rates, da/dt.

Constant-load specimens can be loaded during exposure to the test environment in order to avoid the risk of unnecessary incubation periods associated with change in crack-tip strain rate. In constant-load tests, once the crack has initiated, the crack extension results in increasing crack opening so that there is less likelihood that oxide films either block the crack or wedge it open. On the other hand, because of the increasing stress intensity at the crack tip resulting from the growing crack, the failure mode often shifts from environmental cracking to ductile rupture, so that crack growth rates measured do not necessarily represent those associated with SCC. Subsequent inspection of the fracture surface is necessary to ensure reliable SCC crack growth rate data. The tests require an external loading system. In the case of bend specimens, this can be a relatively simple cantilever beam arrangement, whereas specimens subjected to tension loading require deadweight-loaded testing machines similar to those used in creep rupture experiments. Here, chains of specimens connected by loading links that prevent unloading if one of the specimens should fail can be used in order to minimize the complexity. The size of the loading systems makes it difficult to test constant-load specimens under operating conditions, although they can be tested in environments that are bled off from operating systems.

Constant-load specimens can be of two distinct types:

- Specimens in which the stress intensity increases with increasing crack length; these are suitable for the determination of  $K_{1SCC}$  and, to some extent, for measuring crack growth rates,  $(da/dt)_{SCC}$ , as a function of  $K_1$ .
- Specimens, in which the stress intensity is effectively independent of crack length, can be used for fundamental studies of stress corrosion mechanisms.

Constant-load specimens in which K increases as the crack grows can be subjected to either tension or bending loading. Depending on the design, tension-loaded specimens can experience stresses at the crack tip which are predominantly tensile (as in remote tension types such as the center-crack plate) or contain a significant bending component (as in crack line loaded types such as compact specimens). The presence of significant bending stress at the crack tip can adversely affect the crack path stability during stress corrosion testing and can facilitate crack branching in certain materials. Bend specimens

	Table	3	SCC	test	standards
--	-------	---	-----	------	-----------

Smooth specimens		
Constant load		ASTM G 49-85 (2000) ISO 7539 Part 4 (1989) NACE TM 0177-05
Constant displacement	Bend specimens	ASTM G 39-99 ISO 7539 Part 2 (1990) NACE TM 0177-05
	C-ring specimens	ASTM G 38-01 ISO 7539 Part 5 (1989)
	Uniaxially strained tension specimens	ACE TM 0177-05 ASTM G 49-85 (2000) ISO 7539 Part 4 (1989) NACE TM 0177 05
Constant total strain/plastic strain specimens		ASTM G 30-97 (2003) ISO 7539 Part 3 (1989)
Dynamic straining/slow strain rate test		ASTM G 129-00 ISO 7539-7 (2005) NACE TM0198-04
Precracked specimens Constant load		ASTM E 1681-99
		ISO 7539 6 (1989)
Constant displacement		ASTM E 1681-99 British Standard BS 6980 ISO 7539 Part 6 (2003) NACE TM 0177-05
Rising load/rising displacement		ASTM G 129-00 ISO 7539 Part 9 (2003)
Welds		ASTM G 58-85 (1999) ISO 7539 Part 8 (2000)
Guide to testing procedures		ISO/DIS 7539 Part 1 (in preparation, revision of ISO 7539 Part 1 (1988))

can be loaded in three-point, four-point, or cantilever bend (C(B)) fixtures.

Constant-K, constant-load specimens can be subjected to either torsion loading, as in the case of the double-torsion single-edge cracked plate specimen, or tension loading, as in the case of contoured double-cantilever-beam (DCB) specimens. Although loaded in tension, the design of the latter specimens produces crack line bending with an associated tendency for crack growth out of plane, which can be reduced by the use of side grooves.

#### 11.03.4.5 Constant-Displacement Tests

Constant-displacement specimens are usually self-loaded and hence require no external stressing equipment. Their compact dimensions also facilitate exposure to operating service environments. Like constant-load specimens, they can be used for the determination of  $K_{ISCC}$  by the initiation of stress corrosion cracks from the fatigue

precrack, in which case a series of specimens must be used to pinpoint the threshold value. More common is the determination of  $K_{ISCC}$  by the arrest of a propagating crack, since under constant-displacement testing conditions the stress intensity decreases progressively as crack propagation occurs. This means that, unlike in constant load or in rising-load/rising-displacement SCC tests, the  $\nu$ -K curve is traversed in the direction of decreasing stress intensity factor. In principle, a single specimen will suffice, but in practice the use of several specimens – not less than three – appears necessary.

The procedure for determining  $K_{ISCC}$  by constant-displacement tests on self-loaded DCB specimens is illustrated in Figures 6a and 6b. In this example, three specimens were stressed by bolt-loading to various initial K values, then immersed in the corrosion environment, and the crack extension was recorded over about 1.5 years. From the crack increments measured at discrete time intervals, crack growth rate values,  $\Delta a/\Delta t$ , were calculated and plotted



Figure 6 Determination of the threshold value,  $K_{ISCC}$ , from tests on bolt-loaded DCB specimens. a, Crack extension measured on four specimens (one reference test in air, three specimens tested in ASTM D1141 substitute ocean water) for a total exposure time of 460 days; the labels at the three curves tested in ASTM D1141 indicate the stress intensity factors applied at the start of the test. b, Curves of crack growth rates vs stress intensity factor,  $K_{I}$ , as calculated from the data shown in Figure 6a; the different colors/symbol correspond to the different curves shown in Figure 6a (material: aluminum AA 7010).

against the actual values of  $K_{I}$ , leading to a unique value of  $K_{ISCC}$  determined from crack arrest. As indicated in **Figure 6a**, intermediate periods of apparent crack arrest may be observed before the cracks started to grow again. If  $K_{ISCC}$  were derived from such a first (apparent) crack arrest, it would have been overestimated by nearly a factor of 2.

Other problems arising in constant-displacement tests can be:

- The applied loads can only be measured indirectly by displacement changes.
- Oxide formation or corrosion products can either wedge open the crack surfaces, thus changing the applied displacement and load, or can block the crack mouth, thus preventing the ingress of corrodent, and it can impair the accuracy of crack-length measurements by electrical resistance methods.
- Crack branching, blunting, or growth out of plane can invalidate crack-arrest data.
- Crack arrest must be defined by crack growth below some arbitrary rate, which can be difficult to measure accurately (though, arguably, the same concerns apply to the constant-load method and are intrinsically associated with the definition of K<sub>ISCC</sub>).
- Elastic relaxation of the loading system during crack growth can cause increased displacement and higher loads than expected.
- Plastic relaxation due to time-dependent processes within the specimen can cause lower loads than expected.
- It is sometimes impossible to introduce the test environment prior to application of the

load, which can retard crack initiation during subsequent testing.

 Too high a value can be assigned to K<sub>ISCC</sub> if the K decrease during the test is too high.

On the other hand, it should be pointed out that self-loaded constant-displacement specimens offer significant economic benefits, so that their use is often the most practical solution for SCC investigations.

#### 11.03.4.6 Rising-Load and Rising-Displacement Tests

In dynamic SCC testing, the stress intensity factor at the crack tip is increased while the specimen under test is immersed in the test environment. The tests can reveal cases of SCC susceptibility that would remain undetected in tests under static loading. They may also reflect changes in loading associated with plant startup, for example.

In the rising-load  $K_{ISCC}$  test, which was the first of these accelerated fracture-mechanicsbased SCC tests, acceleration is achieved by applying a constant loading rate (McIntyre and Priest, 1972; Clark, Jr and Landes, 1976). A modified version of this test technique is the step-loading test in which the load is increased in discrete increments if no indication of crack initiation is observed in a predetermined time interval (Raymond and Crumly, 1982).

More recently, a displacement-controlled modification of the rising load  $K_{ISCC}$  test has been introduced and standardized by ISO in



**Figure 7** Crack extension measured in a compact specimen under rising displacement at a rate of  $5.6 \times 10^{-11} \text{ m s}^{-1} (0.2 \,\mu\text{m h}^{-1})$  in a chromated 3.5% sodium chloride solution (material: aluminum AA 2024 T351); the figure shows the signal obtained using the electrical resistance measurement technique as described in Section 11.03.5.1 (Dietzel, 1991).

which a constant displacement – or extension – rate is applied. The loading mode of the rising displacement test is thus similar to that of the SSRT on smooth or notched specimens. While the rising-load  $K_{\rm ISCC}$  test, like the LIST on smooth specimens, appears particularly suited for the determination of thresholds, rising-displacement tests foster the study of SCC failure mechanisms. As in the case of smooth specimen testing, the key parameter is the magnitude of the applied loading or displacement rate, or the time interval between successive load steps. The rates need to be low enough to allow for the environmental cracking to develop without being overridden by pure mechanical rupture.

Typical load-line displacement rates in rising displacement tests are the order of  $1-10 \,\mu\text{m} \,\text{h}^{-1}$ . Despite these low rates, dynamic tests have an accelerating nature and can yield information about the SCC susceptibility of a material-environment system within reasonable test times. Figure 7 contains data from a rising-displacement SCC test of a compact specimen in which the crack length was continuously measured using an electrical potential drop technique as described in Section 11.03.5.1. Although the displacement rate applied in this test was extremely low, that is,  $5.6 \times 10^{-11} \text{ m s}^{-1} (0.2 \,\mu\text{m h}^{-1})$  measured in the load line of the C(T) specimen, crack initiation was observed after only about 15 days, and the subsequent measurement of crack extension enabled the determination of the velocity of subcritical cracking (region II in the da/dt vs K curve). In Figure 8, the thus-determined v-Kcurve is compared to the data generated on self-loaded DCB specimens of the same material as described in connection with Figure 6b.

To evaluate  $K_{ISCC}$  from rising-displacement tests, a number of identical specimens are tested



Figure 8 Comparison of the da/dt vs K data of Figure 6b (colored symbols) and a v-K curve (line) determined in a rising displacement test at a compact specimen using the same material/environment combination. The arrows indicate the direction in which the curve is followed in the two different types of test.

at different displacement rates. In each of these tests, the onset of cracking is monitored, usually by an indirect crack-length measuring technique as described in Section 11.03.5.1, and a curve of the type shown in Figure 8 is established. The threshold for the onset of cracking is sensitive to the displacement rate, highlighting the fact that  $K_{\rm ISCC}$  is not an intrinsic parameter but is dependent on the mechanical loading conditions as well as the environment. At low enough displacement rates, a lower shelf of the sigmoidally shaped curve is established that gives the minimum value of  $K_{\rm ISCC}$  for this system. For the material-environment combination that was investigated in this case, the value obtained for  $K_{\rm ISCC}$  corresponds to that obtained from DCB specimens, which may not always be the case.

In order to maintain the accelerating nature of dynamic SCC testing, it is necessary to limit the number of tests which have to be performed for evaluating  $K_{ISCC}$  to a minimum. This requires that the loading rate which corresponds to the lower shelf regime of the curve in Figure 8 and which yields the lower shelf value of  $K_{\rm ISCC}$  can be determined in a straightforward manner. ISO standard 7539-9 is based on the assumption that the displacement rate,  $(dq/dt)_{SCC}$  (Figure 9), at which a rising-displacement test in a corrosive environment should be performed in order to determine  $K_{\rm ISCC}$  can be derived from the ratio of the measured crack growth velocity in a rising-displacement test in air or in an inert environment,  $(da/dt)_{inert}$ , and the crack growth velocity in the plateau region for SCC,  $(da/dt)_{SCC}$ , by

$$(\mathrm{d}q/\mathrm{d}t)_{\mathrm{SCC}} < \frac{(\mathrm{d}a/\mathrm{d}t)_{\mathrm{scc}}}{(\mathrm{d}a/\mathrm{d}t)_{\mathrm{inert}}} \cdot (\mathrm{d}q/\mathrm{d}t)_{\mathrm{inert}}$$
[2]

Crack Growth Measurement



**Figure 9** Influence of the displacement rate, dq/dt, on the stress intensity factor at crack initiation,  $K_{1th}$ , measured in rising-displacement tests on compact specimens (circled symbols); for comparison, the results of long-term constant-displacement tests with a duration of 10 000 h on double cantilever beam specimens are shown (triangles) (Dietzel, 1991). Reproduced from Dietzel, W. 1996. ESIS guidelines for fracture mechanics based stress corrosion testing. *Technol. Law Insur.* 1, 151–157, figure 2, with kind permission of Springer Science and Business Media.

As a first approach, ISO 7539-9 recommends that

$$(\mathrm{d}q/\mathrm{d}t)_{\mathrm{SCC}} \le 0.5 \cdot \frac{(\mathrm{d}a/\mathrm{d}t)_{\mathrm{scc}}}{(\mathrm{d}a/\mathrm{d}t)_{\mathrm{inert}}} \cdot (\mathrm{d}q/\mathrm{d}t)_{\mathrm{inert}}$$
[3]

The value of  $(da/dt)_{SCC}$  can be obtained from tests which avoid long incubation periods by applying high stress intensity factor values. These can be constant-displacement tests on selfloaded specimens, for example, DCB or wedge opening load (WOL) specimens with the tests being interrupted after a sufficient amount of crack propagation has been observed. The crack growth velocity  $(da/dt)_{SCC}$  can also be determined from step-loading tests or, as has been shown, even the average crack velocity data evaluated from SSRTs on smooth specimens may be used for this estimation (Strieder *et al.*, 1994).

#### 11.03.5 CRACK GROWTH MEASUREMENT

The measurement of crack initiation and growth is an important aspect of SCC testing. For a fracture-mechanics-based investigation of SCC, the crack length is, together with the applied force, the key parameter for calculating the stress intensity factor. *In situ* measurement of the crack length provides useful information about the onset of cracking, the actual crack length or depth, and about the instantaneous crack growth velocity. The electrical resistance measurement technique discussed in the following is also useful in tests on smooth specimens (Winzer *et al.*, 2005). For smooth specimens, this electrical resistance or potential drop technique enables a more precise identification of crack initiation and hence a quantitative evaluation of these tests without the danger of underestimating crack growth velocities, as discussed in Section 11.03.3.4 (Figure 10).

Optical methods of measurement are often precluded by the environment and test chamber and, in any case, apply only to the surface length of a crack. Enhancement of crack visibility by removal of corrosion products may perturb the local electrochemistry and is thus problematic. Methods that measure the average crack length across the thickness of the specimen such as electrical resistance or compliance techniques are generally preferred. AC and DC potential drop (ACPD and DCPD) measurements are suitable but care has to be taken to ensure that the application of a current has no detectable influence on the electrochemistry and the rate of crack extension and to eliminate galvanic effects. Precracked specimens can also use compliance methods based on the measurement of displacement across the notch or of strain in the back-face of the specimen opposite the notch.

#### 11.03.5.1 Electrical Resistance Measurement Methods

#### 11.03.5.1.1 DC potential drop method

The specimen is electrically insulated and a constant current passed through it, across the



Figure 10 Potential drop curves for five cylindrical tensile specimens (material: magnesium AZ91) tested in SSRTs in laboratory air and in distilled water.

crack plane. Typically, the current density used on a ferritic steel specimen is of the order of  $10^4-10^5$  A m<sup>-2</sup> on the net section, but higher current densities may be required on metals of lower resistivity. The potential drop between two points on either side of the crack plane is monitored and related to the crack length through a calibration, derived either experimentally, analytically, or numerically. In the latter cases, experimental verification is desirable.

The method is well established and proprietary equipment with the necessary stability and reliability is readily available. It may not be well suited to large specimens or those of low resistivity because of the high currents that are required.

Potential sources of error with the DCPD method are as follows: (1) thermal resistivity changes and (2) thermal e.m.f.'s.

A comparator technique may be used to overcome these problems (Dietzel and Schwalbe, 1986). The potential drop across the crack is normalized by the potential drop, measured either between points on a second specimen similar to the first, electrically in series with it and physically close to it, subjected to the corrosive environment but not mechanically loaded (Figure 11a), or between a second pair of probes on the specimen under test. The latter, though experimentally easier, requires care in calibration as both the measured potentials may vary with crack length. If a second specimen is used, it is placed under identical environmental conditions as the test specimen to ensure similar transient thermal behavior, etc.



Figure 11 a, Circuit for DCPD measurement of crack lengths on compact specimens in SCC tests in aqueous environments, using a reference specimen and a pulsed current with switching of polarity; b, pulsed current sent through both the test specimen and the reference specimen. Reproduced with permission from Dietzel, W. and Schwalbe, K.-H. 1986. Monitoring stable crack growth using a combined AC/DC potential drop technique. Z. Materialprüfung/Materials Testing 28(11), 368–372.

Thermal effects can also be minimized by using the reversing-polarity technique illustrated in **Figure 11b**. The method provides a measure of the average crack length across the specimen and is well suited to automatic data collection and machine control.

#### 11.03.5.1.2 AC potential drop method

ACPD methods fall into two categories: lowand high-frequency systems.

The low-frequency systems (typically operating in the range 10-100 Hz) are essentially derivatives of the DC method. The use of phase-sensitive detection systems enables a high signal to noise ratio to be obtained and thus the sensitivity is enhanced. Thermal e.m.f.'s also cease to be a problem. However, calibrations are required as with the DC method.

High-frequency systems (5–8 kHz) make use of the localization of current flow to the 'skin' of the specimen that occurs at these frequencies. This minimizes the current requirements and leads to a linear relationship between voltage and crack length, which is independent of specimen size. The method is thus particularly suitable for measuring cracks in large specimens.

The electronics of AC systems are relatively complex, and there may be difficulty in achieving the required long-term stability. Depending on the particular characteristics of the system, great care may be necessary to avoid spurious signals due to pickup. Thus, the physical loop formed between the probe wires and the specimen surface should be kept as small as possible and the probe wires should be twisted together or miniature screened/shielded cable used. Wires should be screened to prevent their movement and the probe and field current wires well separated.

Incorporating the higher-frequency methods into automatic monitoring systems is particularly convenient due to the linear voltage-to-crack length relationship obtained.

**Figures 12a–12d** demonstrate that for steel specimens, ACPD systems may offer the additional advantage in that, unlike in the DCPD signal, at least for ferritic steels a distinct minimum of the ACPD signal is observed which can be related to crack initiation (Venkatasubramanian and Unvala, 1984). This can help to identify the early stages of crack development.

#### 11.03.5.2 Compliance Methods

The use of compliance techniques for determining crack lengths is limited to precracked specimens. Two methods are in use: 1. measurement of the displacement per unit force across the notch and

2. measurement of the 'back-face strain' (BFS) per unit force.

These methods provide a measure of the average crack length and are well suited to automated data acquisition and machine control.

In method 1, a suitable transducer, for example, clip-on extensometer or LVDT (linear variable displacement transducer) is mounted across the notch mouth. The displacement per unit force is determined and related to the crack length by calibration. From the thus-determined compliance, the actual crack length is then determined using suitable calibrations. Alternatively, the unloading compliance method, which is one of the standard techniques in elastic-plastic fracture mechanics, has been successfully applied to the problems of SCC (Anderson and Gudas, 1984).

The BFS method is similar in principle. The transducer is a strain gauge bridge located on the back-face of the specimen, that is, the face opposite the notch. This method has been particularly successful with C(T) specimens and similar sensitivity would be expected with single-edge notch (SE(B)) specimens. Both displacement transducers and strain gauges should be kept out of contact with the solution to preserve their integrity and to avoid galvanic effects.

#### 11.03.6 LIMITATIONS OF THE LEFM APPROACH TO SCC

The applicability of LEFM to SCC and the use of  $K_1$  as the driving force parameter relies on the assumptions of limited plasticity and of predominant plane-strain conditions. In cases of SCC in which sufficient plasticity occurs so that neither plane-strain nor linear elastic conditions are satisfied, K is no longer meaningful, and elastic-plastic fracture parameters appear more suitable.

To apply LEFM, the specimens must satisfy the minimum size requirements imposed by the LEFM concept (see eqn [1]). For lower strength and/or more ductile materials, this can result in large specimen dimensions, particularly with respect to the specimen thickness.

According to Judy and Goode (1970), an assessment of the applicability of LEFM can be made on the basis of so-called ratio analysis diagrams. These diagrams are based on the ratio between fracture toughness and yield strength,  $K_{\rm I}/\sigma_{\rm y}$ , of a material under consideration. If this ratio is high, that is,  $K_{\rm I}/\sigma_{\rm y} > 0.1$ , high stresses and large cracks are required to



**Figure 12** Results of electrical potential drop measurements of crack lengths on a compact steel specimen using ACPD and DCPD systems at the same time (Dietzel and Schwalbe, 1986): (a), ACPD signal vs load-line displacement,  $v_{LL}$ ; (b), DCPD signal vs load-line displacement,  $v_{LL}$ ; (c), load vs ACPD signal; (d), load vs DCPD signal. Reproduced with permission from Dietzel, W. and Schwalbe, K.-H. 1986. Monitoring stable crack growth using a combined AC/DC potential drop technique. Z. Materialprüfung/Materials Testing 28(11), 368–372.

cause fracture, and the LEFM approach is justified. For intermediate values, that is,  $0.1 > K_{\rm I}/\sigma_{\rm y} > 0.05$ , a combination of high stresses and small flaws, low stresses and large flaws, or intermediate stress levels and flaw sizes is critical. In this region, a more refined application of fracture mechanics as outlined in Sections 11.03.6.1 and 11.03.6.2 may be required.

If the ratio is below this range, that is,  $K_{\rm I}/\sigma_{\rm y} < 0.05$ , fracture can initiate from very small defects at moderate or low stress levels, and the fracture mechanics concept seems not to be appropriate and could become nonconservative. Here, the use of an approach which combines the threshold value  $K_{\rm ISCC}$  with the threshold stress,  $\sigma_{\rm th}$ , determined from smooth specimens, according to Section 11.03.6.3 could be a solution.

#### 11.03.6.1 The J-Integral

In recent years, the elastic-plastic J-integral concept has increasingly been applied in

fracture mechanics approaches to SCC (Anderson and Gudas, 1984; Abramson *et al.*, 1985; Dietzel *et al.*, 1989). Figure 13 shows, by way of an example, *J*–*R* curves determined from constant extension rate tests on precracked compact specimens in which various load-line displacement rates,  $dv_{LL}/dt$ , had been applied to precracked compact specimens that were immersed in a corrosive environment. The rates spanned more than 4 orders of magnitude, that is, between 0.1 µm h<sup>-1</sup> (2.78 × 10<sup>-11</sup> m s<sup>-1</sup>) and 6 mm h<sup>-1</sup> (1.7 × 10<sup>-6</sup> m s<sup>-1</sup>).

The figure demonstrates that the value of the *J*-integral measured at crack initiation dropped significantly when the displacement rate was reduced. Also, the slope of the crack growth resistance curves decreased. This can be attributed to the increasing test duration which allows for sufficient time of the corrosive environment, here a 3.5% sodium chloride solution, to promote intergranular SCC (IGSCC) requiring only little mechanical energy to be maintained.



Figure 13 J-R curves determined on precracked compact specimens of the aluminum alloy AA 2024 T351 (S-L orientation) in chromated 3.5% NaCl solution using various constant extension rates, measured in the load lines of the specimens (Dietzel, 1991).

#### 11.03.6.2 Crack-Tip Opening Angle/ Displacement

In addition to the *J*-integral, there is increasing interest in two other elastic–plastic fracture parameters, that is, CTOA and CTOD. These parameters are particularly suited for structural integrity assessments of thin-walled structures, and they have successfully been applied for investigating the initiation and growth of stress corrosion cracks.

CTOD, often designated as  $\delta$ , is specified as the relative displacement of the crack surfaces normal to the original, undeformed crack plane at the tip of the fatigue precrack. In practice, this parameter can be determined at the surface of a specimen or a component, for example, by using a specially designed clip-on gauge which measures the CTOD over a gauge length of 5 mm ( $\delta_5$ ) (see Chapter 11.02).

In SCC experiments which are performed in aggressive environments and/or at elevated temperatures, a direct measurement of the variable  $\delta_5$  is tedious, since the clip-on gauge would have to be immersed in the corrosive environment during the duration of the test, and hence would need careful protection against corrosion. Calibration experiments performed at a number of materials and under various environmental conditions have confirmed that excellent agreement exists between the directly measured  $\delta_5$  data and values which were

calculated from the load and the CMOD. The expression correlating  $\delta_5$  and the CMOD is derived from the British Standard 5762 and modified with respect to crack extension (Schwalbe, 1995):

$$\delta_5 = \frac{K^2}{2\sigma_{\rm v}E'} + \frac{0.6\Delta a + 0.4(W-a)}{0.6(a_0 + \Delta a - 0.4W + z)} v_{\rm pl} \quad [4]$$

In this modified CTOD, the motion of the rotation center of the specimen due to crack extension is taken into account. In eqn [4],  $v_{pl}$  is the plastic portion of the CMOD, E' the Young's modulus for plane strain, and z is the distance between the load line and the actual measuring position for v.

The CTOA,  $\psi$ , is the angle subtended by the flanks of an extending crack. The extension criterion of the model of Rice et al. (1980) assumes that, after an initial transition period, crack extension proceeds in such a way that a geometrically similar crack profile is maintained near the crack tip, that is, that the CTOA remains constant, independent of the amount of crack extension. More specifiextension criterion cally, the is the requirement that a critical opening displacebe maintained at a small ment,  $\delta_{\rm C}$ , characteristic distance,  $r_m$ , behind the crack tip (Figure 14);  $r_m$  is comparable to the size of the fracture process zone within which decohesion occurs at the crack tip. The crack is thus assumed to extend at a CTOA,  $\psi_{\rm c}$ , with

$$\Psi_{\rm C} = \frac{\delta_{\rm C}}{r_{\rm m}}$$
[5]

When this model and the associated analysis are extended to SCC, it is assumed that the magnitude of the CTOA,  $\psi_c$ , which is governed by the fracture process in the process zone, depends on the crack extension rate, da/dt, and decreases in the presence of an aggressive environment.

In order to determine the CTOA,  $\psi$ , according to its definition in **Figure 14**, the distance  $r_m$  needs to be held constant. This is usually done using finite element analyses, whereas in experimental studies this is often not feasible. Instead, a measure of the CTOA can be derived from measuring the change of the CTOD,  $d\delta_5$ , with increasing crack extension, da, using

$$\tan \Psi \approx \frac{\mathrm{d}\delta_5}{\mathrm{d}a} \tag{6}$$

In principle, the crack-tip strain rate,  $d\varepsilon_{tip}/dt$ , would be the ideal parameter for characterizing the fracture micromechanisms since this rate



Figure 14 Definition of the crack-tip opening angle (CTOA),  $\psi$ .



**Figure 15** Values of the tangents of the CTOA,  $\psi$ , determined in rising-displacement tests on compact specimens of a high-strength steel (FeE 690T) subjected to hydrogen embrittlement due to cathodic protection (Dietzel, 1991).

controls the time-dependent corrosion-deformation interactions taking place in the process zone at the crack tip. Because of the singularity of the stress field at the crack tip, this parameter is, however, difficult, if not impossible, to determine. So the CTOA, the CTOD, and, in particular, the rate of change of the CTOD,  $d\delta/dt$ , are useful alternative parameters.

In Figure 15, values of the CTOA have been determined from tests performed using precracked compact specimens of a high-strength structural steel using various constantdisplacement rates. The displacement rates have been converted into rates of change of the CTOD,  $d\delta_5/dt$ , as a measure of the applied deformation rate. The values of  $d\delta_5/da$ , corresponding to the tangents of the CTOA,  $\psi$ , are plotted on the vertical axis. A significant decrease of the CTOA due to the increasing influence of SCC was observed at low deformation rates,  $d\delta_5/dt$ . The reference tests in air showed no such change in CTOA with decreasing deformation rate. The drop in CTOA was caused by hydrogen embrittlement resulting from the ingress of atomic hydrogen into the steel. A similar effect of the displacement rate on the CTOA was observed when testing material/environment combinations in which the failure mechanism was IGSCC (cf. Section 11.03.4.2).

In principle, CTOA and CTOD as EPFM parameters reflect the influence of the corrosive environment on crack initiation and growth in the same manner as the *J*-integral. Yet, CTOA and CTOD have two major advantages over the *J*-integral. First, these two parameters can directly be measured on components that are to be assessed with respect to structural integrity, including SCC, and hence the transferability of data generated using laboratory specimens to real structures is facilitated. Second, CTOA and CTOD are parameters that are well suited for the structural assessment of thin-walled structures.

#### 11.03.6.3 Shallow Cracks

The applicability of fracture-mechanics-based SCC test data to real components can be impaired by the fact that the crack-tip chemistry can be considerably different from the electrochemical conditions outside the crack. This is an issue of importance particularly for small cracks (typically less than 1 mm in depth). Here, differences in crack-tip electrochemistry from that in deep cracks may arise in some systems, in which case the principle of similitude with respect to the application of the stress intensity factor would no longer be valid, even if LEFM were applicable to define the mechanical driving force.

Kaufmann (1979) has shown that below a certain crack size, the use of the fracture mechanics concept becomes invalid and that the LEFM threshold parameter KISCC nonconservative (Figure becomes 16). Instead, a combination of the SCC threshold stress,  $\sigma_{\rm th}$ , determined on smooth specimens, and  $K_{ISCC}$  can lead to a short crack design criterion which specifies the minimum crack size for the use of the K concept. Figure 16 further demonstrates that crack initiation values which had been determined on specimens containing shallow surface cracks using both LEFM and the threshold stress,  $\sigma_{th}$ , approach yielded results which fall into the transition region between the two lines forming the SCC limit (Dietzel and Schwalbe, 1993). This limit represents the boundary between propagating and nonpropagating (or noninitiating) stress corrosion cracks. The challenge remains to be able to determine



**Figure 16** The threshold stress,  $\sigma_{th}$ , as a 'short crack' design criterion. Threshold data,  $\sigma_{th}$ , measured on smooth tensile specimens, and  $K_{ISCC}$  data, determined from fracture mechanics SCC tests on precracked specimens, are combined to yield an assessment of the critical minimum size of a semielliptical surface flaw for the occurrence of SCC. The lines defining the upper boundary of the SCC-affected area result from tensile and fracture toughness tests in air; the two red triangles in the hatched area result from SCC tests on two surface cracked specimens (material: high-strength aluminum alloy AA 2024 T351, environment: chromated 3.5% NaCl solution; Dietzel and Schwalbe, 1993).

the growth rate of cracks in the short crack regime and to derive an expression for the relevant mechanical driving force to transfer the data to service conditions.

#### 11.03.7 THE USE OF SCC DATA

#### 11.03.7.1 Predicting the SCC Behavior of Materials and Structures

As a screening parameter for susceptibility classifications of materials, SCC threshold values  $\sigma_{th}$  and  $K_{ISCC}$  are widely accepted for providing guidance to develop and/or select materials that exhibit sufficiently high threshold values for applications in which SCC must be prevented. Together with crack growth data, these values yield information about the severity of environments that can promote SCC, and about the effectiveness of counter-measures and protection means.

Crack growth rate, da/dt, versus  $K_I$  data can be used to establish allowable subcritical crack growth for both new designs and existing structures, that is, to decide whether a period of safe crack extension exists, and if so, to specify inspection intervals for parts assumed or known to have flaws. SCC growth life predictions are in principle performed by integrating da/dt versus  $K_I$  data, thus yielding the time when the critical crack size is reached. This in turn requires that the K values of components can be calculated and that residual and/or assembly stresses are either known or are negligible. A rough estimate of the significance of subcritical crack growth may be obtained from the value of da/dt in the plateau region of the *v*-*K* curve.

Figure 17 is an example in which data that had been obtained on part through-cracked specimens were used to predict the time of crack initiation and the SCC crack growth in plates which contained small semielliptical surface cracks, which in turn simulated a defect in a large structure. The fact that the predicted failure time of 110 h came close to but slightly underestimated the time at which the crack could be observed on the back side of the plate, that is, after 140 h, indicates the capability of the fracture mechanics approach to transfer stress corrosion data from small-scale laboratory specimens to real components and structures.

Using the threshold parameters  $\sigma_{th}$  and  $K_{\rm ISCC}$  allows prediction of the combinations of stress level, flaw size, and shape which lead to SCC. They may be used as a design criterion for ensuring no SCC growth in service, provided that the stress levels, minimum detectable flaw sizes, and environmental conditions are well defined, and that the service loads are essentially sustained, that is, cyclic loading is not significant. Figure 18 illustrates in a schematic way how  $K_{\rm ISCC}$  and  $\sigma_{\rm th}$  values can be included in an assessment of structural integrity based on the failure assessment diagram (FAD). In this case, the fracture toughness of the material under consideration,  $K_{mat}$ , is replaced by  $K_{ISCC}$ . A matter of discussion remains the question whether the threshold stress for SCC,  $\sigma_{th}$ , determined on smooth tensile specimens of the same material and in the same environment, should in this approach be regarded as the new cutoff line on the stress axis,  $L_{\rm r}$ , or whether the original cutoff line pertaining to the final plastic collapse of the structure under consideration should be maintained. Because of subcritical crack growth due to SCC and depending on the susceptibility of the material/environment combination being investigated, the acceptable region can be significantly reduced as compared to the fracture behavior in air.

#### 11.03.7.2 Studies of SCC Mechanisms

Rising-displacement tests using precracked specimens have also proved to be well suited for studying SCC mechanisms. Measurement of the CTOA or, equivalent to this, the ratio between crack growth velocity and the applied deformation rate allows a comparison with models that simulate the mechanism leading to SCC.



**Figure 17** Prediction of residual lifetime with respect to SCC for a structure containing a semielliptical surface crack. The da/dt value measured on a compact specimen (region II of v-K curve) is used to predict the time after which the surface crack in a plate will reach the back-face of the specimen. With a plate thickness B = 5 mm, an initial crack depth of 2.7 mm, and a crack growth rate  $(da/dt)_{SCC,plateau} = 6 \times 10^{-6}$  mm s<sup>-1</sup>, the calculated residual life time was 110 h, and a measurable crack of  $\sim 2$  mm length was observed at the back-face of the specimen (see inset photograph) after about 140 h.

In Figures 19-21, an example is given in which the hydrogen embrittlement of steel was studied in rising-displacement tests at precracked specimens and was simulated by a diffusion model. A comparison of two J-Rcurves measured in air and in the corrosive environment indicated that the fracture resistance of the material was significantly reduced by the ingress of atomic hydrogen (Figure 19). At low displacement rates, the increasing influence of the hydrogen embrittlement led to an acceleration of the crack growth velocity observed at a certain deformation rate compared to the crack growth velocity measured in air at the same deformation rate, as can be seen from Figure 20.

As Figure 20 indicates, this increase in crack growth velocity can be simulated by model calculations of the hydrogen diffusion. Here, a diffusion model was used which assumed that the hydrogen entered at the area of bare metal generated at the crack tip due to the cracking process. It was further assumed that the diffusion in the metal was impeded by traps which were caused by plastic deformation, that is, the plastic zone ahead of the crack tip (Dietzel *et al.*, 2005).

An extension of this model taking into account the stochastic nature of the local cracking process caused by hydrogen embrittlement was applied to further mimic the morphology of the crack front at various displacement rates under these experimental conditions. Figure 21 is a comparison of crack fronts which have been observed in hydrogen embrittlement experiments on compact specimens both at a fairly high and at a low load-line displacement rate (300 and  $0.3 \,\mu m h^{-1}$ ) and of crack fronts (Pfuff



**Figure 18** Schematic presentation illustrating the inclusion of  $K_{ISCC}$  data into a failure assessment diagram (FAD). By substituting the fracture toughness of the material,  $K_{mat}$ , for  $K_{ISCC}$ , and the cutoff line on the stress axis,  $L_r$ , for the SCC threshold stress,  $\sigma_{th}$ , an acceptable region with respect to nongrowing SCCs is defined; this region can be significantly smaller than the original acceptable region as determined from tests in air.



Figure 19 J-R curves measured in air and in a corrosive environment promoting the ingress of hydrogen due to cathodic polarization (material: high strength steel, environment ASTM D1141 substitute ocean water) (Dietzel and Schwalbe, 1990).

and Dietzel, 2005). The development of the crack front at a load-line displacement rate of  $0.3 \,\mu\text{m}\,\text{h}^{-1}$  is also demonstrated in the video (Pfuff and Dietzel, 2005). In the simulation, the hydrogen is entering from the crack tip at the bottom of each of the two pictures, and the diffusion coefficient decreases with the amount of plastic deformation in the material due to mechanical straining.

#### 11.03.8 GUIDE TO SELECTION OF MECHANICAL SCC TEST METHOD

The test method selected depends critically on the application, purpose of the test, and pragmatic issues of cost and expediency. It is



**Figure 20** Crack growth velocity, da/dt, measured as a function of the applied deformation rate,  $d\delta_5/dt$ , in tests performed in air and in corrosive environment promoting hydrogen embrittlement; the solid curve was obtained from simulation based on a diffusion model (material and environment as in **Figure 19**) (Dietzel *et al.*, 2005).

not possible to be prescriptive, not least because there may be industry-specific standards that define the requirements. **Table 4** summarizes the types of test, the nature of the result, and possible applications. In **Table 4**,  $\varepsilon_{pl}$  is the plastic strain to failure, RA is the reduction in area,  $\sigma_{th}$  is the threshold stress for cracking,  $t_f$  is the time to failure, da/dt is the crack growth rate, K is the stress intensity factor, and  $K_{ISCC}$  is the threshold stress intensity factor for SCC.

For screening purposes (environment for material), the SSRT is often used as a first choice because it is rapid and mechanically severe. In interpreting the results, an absence of any SCC should not be considered to indicate immunity unless testing is conducted over a range of strain rates including very slow rates down to  $10^{-8}$  s<sup>-1</sup>.



**Figure 21** Crack front morphology. a, b, Observed in experiments at displacement rates of 300  $\mu$ m h<sup>-1</sup> (a) and 3  $\mu$ m h<sup>-1</sup> (b) at high-strength steel in ASTM D1141 substitute ocean water under cathodic charging (crack growth direction from bottom to the top). c, d, Results of model calculations assuming the same displacement rates; the pictures show the crack front (black) and the hydrogen distribution ahead of the crack front (magenta: highest, orange: lowest hydrogen concentration) (Pfuff and Dietzel, 2005).

The method is used less often at elevated temperature testing when a large number of variables have to be assessed when identifying domains of cracking/no-cracking in complex and variable environments. In general, slow strain rate testing is not used for qualifying a material for service as there are no agreed acceptance criteria. Similar considerations apply for LIST.

Constant total strain tests have a variety of uses because they involve simple restraining jigs, are readily accommodated in autoclaves, and enable multiple testing. Within that category, U-bend tests offer mechanically severe test conditions for sorting/screening purposes. Other bend tests, particularly four-point bend, are used commonly in the oil and gas industry for assessing pipeline steels and tubular products; they are particularly suitable for testing of welded specimens with one surface in the as-welded condition. The applied stress is often set at the 0.2% proof stress for the material.

Most commonly, the test results are based on a cracking/no-cracking approach and can be used for qualifying a material for service. The test duration is often set pragmatically at 30 days for many standard tests in the oil and gas industry, the assumption being that if it is going to crack, some indication would have appeared in that timescale. That is not always the case, particularly where there is some other timedependent precursor to cracking, for example, pitting, intergranular corrosion. When testing tubular products with cracks possibly initiating and propagating in the longitudinal direction, for example, due to hoop stresses (longitudinal seam welds) C-ring specimen under constant displacement would be appropriate.

Constant total strain tests can be conducted equally with tensile specimens as quasi-constant load uniaxial tests. Here, tests may be conducted to follow the evolution of cracks from an initially plain surface or with varying applied

Type of test	Test result	Typical test times	Engineering usage
Slow strain rate, linearly increasing stress	$\varepsilon_{pl}$ and RA ratios	Function of strain rate, 2–10 days	Classification of susceptibility Screening Relative aggressivity of environments
Constant total strain:			environments
Nominally elastic (two- to four-point bend, C-ring, O-ring, uniaxial	$\sigma_{\rm th}$ , cracked/not cracked	Variable but often 10–90 days	Classification of susceptibility Screening Relative aggressivity of environments
			Design (e.g., pass/fail acceptance for service criteria)
Plastic-elastic (U-bend, reverse U-bend)	Cracked/not cracked		Classification of susceptibility Screening
			Relative aggressivity of
Constant load	$\sigma_{\rm th}, t_{\rm f}$	10–90 days	Classification of susceptibility
			Screening
			Relative aggressivity of environments
Deserves and the size has a de			Design (threshold stress)
Increasing K specimens	$da/dt$ vs K, $K_{ISCC}$	10-125 days	Classification of susceptibility
Decreasing K specimens Constant K specimens			Design criterion Life prediction
Rising load/displacement with increasing K specimens			Inspection intervals

**Table 4** Nature of results from SCC tests and their applications

initial load to determine threshold stress conditions. Test times are highly variable depending on the application and purpose. Moreover, in all constant total strain tests, stress relaxation is a concern and should not only be accounted for when assessing threshold stresses in particular, but also in pass/fail-type of tests.

Constant load tests would be the preferred tests for many applications as the stress is well defined and sustained through the test. The limitation is simply one of ease of use and cost when testing a large range of variables or materials for autoclave applications.

In adopting fracture mechanics test specimens, the assumption is that crack-like defects will be present initially or will be initiated in service. The application often determines the adoption of such an approach. The purpose of the test can vary from materials ranking using  $K_{\rm ISCC}$  (e.g., self-loaded DCB testing for carbon steels in H<sub>2</sub>S environment in the oil and gas industry) to determination of design data and life prediction in terms of threshold stress intensity factor and crack growth rates.

For determination of the threshold stress intensity factor for cracking, methods may be based on constant displacement at the loading line (DCB, WOL) or constant load. The former, decreasing K, methods have the advantage in that they allow determination of the threshold stress intensity factor for arrest of a growing stress corrosion crack within the resolution of the crack-measurement method. They also do not require a loading machine. The constantload type of test based on precracked specimens may require a higher stress intensity factor to initiate a crack if there is a crack mode change, for example, from the transgranular fatigue crack to an IGSCC.

Rising-load or rising-displacement  $K_{ISCC}$ measurement has the virtue of not only being a potentially accelerated test method but also one that accommodates the transient dynamic straining that may be experienced in service. The loading/displacement rate is the key variable, and it is prudent to test for a range of rates to assess the impact on  $K_{ISCC}$ . The lowest value may be conservatively considered for

Document	Title
EFAM GTP-SCC 02	The GKSS test procedure for performing stress corrosion tests using precracked specimens, 2003, GKSS 2002/21, GKSS-Forschungszentrum Geesthacht GmbH, Geesthacht, Germany
EFC 17	Corrosion-resistant alloys for oil and gas production: guide on general requirements for test methods for H <sub>2</sub> S service, 2nd edition, European Federation of Corrosion, 2002, Maney Publishing
ESA ECSS-Q-70-36A	Material Selection for Controlling Stress Corrosion Cracking, 1998, ESA Publications Division, European Space Agency, Noordwijk, The Netherlands
ESA ECSS-Q-70-37A	Determination of the Susceptibility of Metals to Stress Corrosion, 1998, ESA Publications Division, European Space Agency, Noordwijk, The Netherlands
ESIS DI-92 Fracture	Control Guidelines for Stress Corrosion Cracking of High Strength Alloys, 1992, European Structural Integrity Society, (available from: GKSS- Forschungszentrum Geesthacht GmbH, Geesthacht, Germany)
NLR CR 89213 L	A Test Plan for Sustained Load Fracture Control Verification, 1986, National Aerospace Laboratory NLR, Amsterdam, The Netherlands
NPL Report DEPC MPE 007	Methodology for determining the resistance of welded corrosion resistant alloys to stress corrosion using the four-point bend method, 2004, National Physical Laboratory, Teddington, Middlesex, UK

 Table 5
 SCC test procedures and documents

assessment purposes. A more sophisticated test machine is required but the test times can be a fraction of that required under constant-load condition. This type of test has the further advantages that it has the potential to include EPFM approaches where required, and to study the mechanisms of SCC including simulation/modeling.

A number of standards and procedure documents which are listed in **Tables 3** and **5** offer detailed guidelines for SCC testing. The lists are not complete, but they may provide some assistance in selecting an appropriate document for SCC testing.

#### 11.03.9 CONCLUDING REMARKS

There is often good agreement between results obtained using different SCC test methods in terms of relative performance. However, differences can occur due to, for example, differences in environmental variables, such as the electrode potential, pH, flow rate, solution composition, and in the time of testing. The specification, monitoring, and, where appropriate, control of environmental variables are necessary requirements for improved reliability of data. But, despite appropriate indications in the various standards, the increased complexity and costs of testing can influence the experimental rigor.

Even if all the environmental variables are monitored or controlled as specified, results from individual test methods can differ because of variations in the timescale of testing. Differences can be associated with the extent of pitting, changes in surface films and deposits, variations in open circuit potential, extent of hydrogen charging, and variations in local chemistry in cracks. The economic pressure for accelerated testing is well recognized, but these factors must be considered and understood in interpreting the relevance of the results.

Concerning the application of these test data, SCC fracture control can be a particular problem for high-strength alloys, and may require verification by extensive small-scale laboratory testing as well as full-scale service qualification tests. In this chapter, the requirements for SCC testing have been defined, categorized, and discussed. The ways in which fracture mechanics and nonfracture mechanics test data and parameters can be used for such SCC fracture control have also been described. Difficulties and limitations in the practical application can especially occur with respect to really controlling SCC, that is, to establish subcritical crack growth allowables.

Such control of SCC may not always be feasible, and hence SCC fracture control will in these cases rather have to be directed to prevent SCC in service. The prevention of SCC is, of course, always the best starting point for any SCC fracture control plan, whereas in many other situations SCC is unavoidable but controllable. Here, guidance on the appropriate steps to take when a stress corrosion crack has been detected in service and an assessment of the implications for structural integrity is required (Wanhill, 1991). Documents that aim at providing such guidance are in preparation by ISO and CEN committees.