# A METHODOLOGY TO DETERMINE THE SCC BEHAVIOUR OF STEELS IN EPFM REGIME: APPLICATION AND VALIDATION

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The J-Integral is the most commonly used test to characterise the toughness of materials showing elastic-plastic mechanisms at fracture. This methodology is starting to be used in the characterization of subcritical cracking phenomena such as stress corrosion cracking (SCC) or hydrogen induced cracking (HIC) when they are supported by elastic-plastic mechanisms. An analytical methodology based on EPRI formulation allows to determine the characteristic parameters of the initiation conditions for cracking as well as the crack propagation rates. In this work this methodology is checked in different environments and materials presenting a sufficiently understood, and even modelled, SCC behaviour . A supporting fractographic SEM study has been performed to help in the analysis of the variable behaviours depending on applied loading rate obtained for each material and environmental condition used.

## INTRODUCTION

J-integral based toughness tests are the most widely accepted for the characterization of the elastic-plastic fracture behaviour of metallic materials (McMeeking (1)). The ease of performing these tests, along with their reliability, have extended their use to fields such as stress corrosion cracking (SCC) where subcritical cracking in elastic-plastic regime exists (Evans and Parkins (2) and Vassilaros et al (3)). Conventional DCB constant displacement tests offered important limitations in the characterization of such behaviours, both in representativeness, as they are based on LEFM concepts, and in accuracy, as they are based on crack length measurements from the crack tip movement, difficult to follow due to its slow rate and developed local plastic deformation. So, it was decided to use an EPFM analysis to better understand the SCC and HIC behaviour of microalloyed and low-alloyed SCC steels.

To perform this analysis a theoretical study was first done ((4) and Alvarez et al (5)) to correlate the data obtained from a conventional J integral test, load-displacement (COD) curve, with those variables that define subcritical environment assisted cracking, i.e. critical propagation conditions at threshold, crack propagation

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rate, da/dt, and the transition conditions from subcritical cracking to final stable or unstable fracture. In order to select the testing conditions that this analysis would need, proof tests were performed showing the clear influence of the loading displacement rate first on the load-COD curves and then on the HI cracking behaviour. The application of this methodology requires its validation, by checking it for different material and environmental conditions, the behaviour of which is sufficienly understood and even modelled (Gutiérrez-Solana et al (6)).

## ANALYTICAL METHODOLOGY

The analytical methodology used to determine the elastic-plastic fracture behaviour of a material from the load-COD relationship obtained in a test was proposed by Shih and Hutchinson (7) as a fuction of sample geometry and material mechanical behaviour. Based on EPFM concepts, this EPRI adapted methodology (Kumar et al (8)) provides the parameters which characterise the crack driving forces as a function of crack advance by considering the mechanical behaviour of the materials, defined through a Ramberg-Osgood law. Applying these values to the EPRI method it is possible to obtain the load-COD behaviour curves for any crack length and specimen geometry for plain stress or plain strain conditions.

Therefore by comparing the experimentally obtained load-COD curve for a material sample with those proposed by EPRI for each crack length, as shown in Figure 1a, the crack advance law for the sample tested can be defined as a function of load or COD, and then of time through the loading history, for the extreme conditions of plane strain and plane stress. Similarly, if the crack length evolution is known in the experimental test through the compliance evolution of the specimen, a theoretical prediction of the load-COD curve in plane strain and plane stress can be done enabling their comparison with the experimental curve in order to determine its current stress state (Figure 1b).

To apply this methodology to performed tests, first the stress state conditions of the tests are determined by comparing their experimental behaviour with the theoretical plane stress and plain strain behaviours. Then, under the corresponding conditions, crack length evolution is obtained and its correlation with time is used for determining the crack propagation rate. The fracture parameter chosen to correlate with the cracking process characterization is the stress intensity factor, $K_I$.

## MATERIAL

The methodology has been applied to two types of steels, used also to validate it by comparison with conventional SCC tests. First, two water quenched and tempered microalloyed steels of high (E690) and medium yield strength (E500), both with a bainitic microstructure. Second, a HSLA 4140 steel, under three different heat treatments: normalized in air (Series A), oil-quenched (Series B) and oil-quenched and tempered (Series C), to provide different, intergranular (IG) or transgranular (TG), SCC mechanisms (6). The treated coupons show bainitic, martensitic and tempered martensitic structures for Series A, B and C. The chemical composition and mechanical characteristics of these steels, necessary for the analytical support of the experimental results, are shown in Tables 1 and 2, respectively.

The high resistance to SCC of the microalloyed steels in sea water under different conditions has been clearly demonstrated and associated with their highly elastic-plastic behaviour (4). The high resistance was observed even for more aggressive environments and hydrogen induced cracking (HIC) processes. Table 3 presents the HIC characterisation of these steels cathodically charged with hydrogen from 1 to 10 mA/cm$^2$ of current density as determined by conventional methods using DCB samples (4). The more susceptible behaviours to SCC in sea water of all 4140 microstructures are also shown in Table 3, which therefore provides the reference results for the new methodology.

TABLE 1- Chemical composition in weight percent of the steels.

| *Steel* | *C* | *Si* | *Mn* | *Ni* | *Cr* | *Mo* | *Cu* | *Sn* | *Al* | *V* | *Ti* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| E690 | .06 | .23 | 1.36 | .58 | .12 | .20 | .10 | .003 | .017 | .048 | .002 |
| E500 | .135 | .241 | 1.1 | 1.52 | .50 | .46 | .18 | .009 | .078 | .002 | .002 |
| 4140 | .39 | .25 | .71 | .23 | .94 | .16 | .14 | .014 | .014 | - | .013 |

TABLE 2- Mechanical characteristics of the different steels and treatments.

| Steels | E500 | E690 | 4140A | 4140B | 4140C |
|---|---|---|---|---|---|
| Hardness (HV) | 202 | 292 | 330 | 670 | 380 |
| $s_{y\,0.2}$ (MPa) | 530 | 840 | 690 | 1210 | 1085 |
| $s_u$ (MPa) | 640 | 915 | 1150 | 2100 | 1150 |
| $e_{max\ load}$ (%) | 9 | 6.5 | 10 | 2.5 | 8 |
| $\alpha$ | 2.55 | 1.56 | 0.002 | 0.67 | 1.39 |
| n | 9.86 | 17 | 6.6 | 4.59 | 17 |

## EXPERIMENTAL PROCEDURE AND RESULTS

J integral type tests were performed in aggresive environments on compact specimens with a thickness of 25 mm and a 2.5 mm side grooving, using a slow strain rate testing machine at velocities ranging from 8.4x10$^{-10}$ to 4.1x10$^{-7}$ m/s to analyse the influence of the displacement velocity.

The hydrogen rich environment used with the microalloyed steel was obtained

TABLE 3- Results of the SCC conventional characterization of the different steels.

| Material | Current density ($mA/cm^2$) | $K_{Iscc}$ ($MPa.m^{1/2}$) | $(da/dt)_{II}$ (m/s) | Fracture type |
|---|---|---|---|---|
| E690 | 1 | 47 | $1\text{-}2x10^{-7}$ | IG+TG |
| E690 | 5 | 38 | $1x10^{-7}$ | IG+TG |
| E690 | 10 | 18 | $2x10^{-7}$* | IG+TG |
| E500 | 1 | >100 | No crack | - |
| E500 | 5 | 45 | $1x10^{-7}$ | TG |
| E500 | 10 | 37 | $1x10^{-7}$ | TG |
| 4140A | * | 50 | $2.5x10^{-7}$ | TG |
| 4140B | * | 8 | $3x10^{-6}$ | IG |
| 4140C | * | 40 | $2.2x10^{-7}$ | IG+TG |

(*) Test made in a solution 3.5%NaCl in distilled water.

by cathodic polarization in an electrochemical cell with a platinum anode and using the specimen as the cathode in a 1N solution of $H_2SO_4$. A potentiostat was used to maintain a constant current independent of the environmental variations . In order to know the behavioural influence of hydrogen on cracking in elastic-plastic regime, tests were performed using variable current densities (1, 5 and 10 $mA/cm^2$) for each of the materials. The environment used with the low alloy steels was a solution of 3.5% NaCl in distilled water.

A fractographic analysis with electron microscopy (SEM) was made on the fracture surface of each specimen to obtain its typology, i.e. fracture mechanisms, to compare with those obtained by conventional testing. Also, reference tests were performed in air to determine the cracking processes of these steels under inert conditions (4).

The analytical methodology has been used considering the samples under plane strain conditions, because this has been shown to be closest to the real behaviour. Curves like those shown in Figure 2 which represent the crack propagation rate against COD or $K_I$, based on the experimental P-COD curves, have been obtained in all cases. In these curves the $K_I$ values were obtained from the elastic part of the J integral, $J_e$. Using these curves it is possible to define the behaviour through various parameters. A threshold stress intensity factor, $K_{th}$, exists at which crack propagation is initiated; this first stage of the cracking process, subcritical, is characterised after determining the crack propagation rate $(da/dt)_{sc}$ as a function of COD or $K_I$. This subcritical cracking process extends to a critical $K_I$ value, $K_c$, where an increase in crack rate suddenly occurs.This $K_c$ value initiates the second stage cracking process, the critical one, associated with specimen unloading instability. At this stage crack propagation rate is termed $(da/dt)_c$. The results of most of the tests carried out for both type of steel are presented in Table 4.

TABLE 4- Results of the J integral type tests performed.

| MATERIAL | Current density ($mA/cm^2$) | Displacement velocity (m/s) | $K_{th}$ ($MPa.m^{1/2}$) | $(da/dt)_{sc}$ (m/s) | $K_c$ ($MPa.m^{1/2}$) | $(da/dt)_c$ (m/s) |
|---|---|---|---|---|---|---|
| E690 | 5 | $4.1x10^{-7}$ | 85 | $1\text{-}3x10^{-6}$ | 135 | $1.5x10^{-4}$ |
| E690 | 5 | $4.1x10^{-8}$ | 52 | $2x10^{-7}$ | 90 | $1x10^{-6}$ |
| E690 | 5 | $8.2x10^{-10}$ | 38 | $5x10^{-8}$ | 45 | $2.5x10^{-6}$ |
| E500 | 5 | $4.1x10^{-8}$ | 65 | $2x10^{-7}$ | 80 | $2x10^{-7}$ |
| E500 | 5 | $8.2x10^{-10}$ | 35/46 | $2x10^{-8}$ | - | - |
| 4140A | * | $4.1x10^{-8}$ | 50 | ** | - | - |
| 4140A | * | $8.2x10^{-10}$ | 71 | ** | - | - |
| 4140B | * | $4.1x10^{-8}$ | 18 | $3x10^{-6}$ | 34 | $2x10^{-5}$ |
| 4140B | * | $8.2x10^{-10}$ | 7 | $2x10^{-7}$ | 13 | - |
| 4140C | * | $4.1x10^{-8}$ | 110 | $1.5x10^{-7}$ | 175 | $2x10^{-6}$ |
| 4140C | * | $8.2x10^{-10}$ | 50 | $2x10^{-8}$ | 88 | - |

(*) Test made in a solution 3.5%NaCl in distilled water.
(**) Unstable propagation.

## ANALYSIS AND CONCLUSIONS

The results of the tests performed for the same material-environmental conditions show that the displacement velocity has a significant influence on the overall cracking behaviour. It is therefore important to establish the appropriate loading rates that characterize SCC in the same way that conventional testing does. However, the displacement velocity has no influence in the tests performed in air. So, the results obtained also show the effect of hydrogen presence and concentration, proportional to current density, on the cracking resistance of the same steel using the results obtained from tests performed at the same displacement velocity under different levels of hydrogen charging or in air. These influences are then conveyed to all the previously defined behaviour characterization parameters for both materials tested.

For both microalloyed steels when the displacement velocity decreases, so does the initiation of subcritical cracking, $K_{th}$, reaching similar values to the one obtained in conventional constant displacement tests at the slowest velocity (8.2x $10^{-10}$ m/s). At the same time, the subcritical crack propagation rate, $da/dt_{SC}$, also decreases finally stabilizing at the conventional test's characteristic values for displacement velocities lower than $4.1 \times 10^{-9}$ m/s. For all the displacement rates, subcritical cracking is typical of bainite transgranular cleavage with an important presence of intergranular fracture. The final cracking processes, after maximum load, only corresponds to the conventional fracture mechanisms by microvoid coalescence at the faster tests. So, for the slower rates $K_c$ values don't represent the limit for SCC when final fracture is reached. This limit is obtained only at the fastest velocity.

For the low alloy steel, independently of treatment, the specimens tested at the slower velocity present a behaviour similar to conventional SCC, both in values of threshold and crack propagation rates and local fracture mechanisms. Series A specimens had a fracture surface with a massive presence of cleavages, conditioned by the dynamic effects which provoke instability in crack propagation. Series B specimens propagated under intergranular mechanisms and the specimens of series C present a transgranular fracture surface with hydrogen induced tearing.

Once the developed methodology has been checked and validated for the characterization of the SCC of these steels with variable microstructural states in different environments, it can be concluded that J-integral type tests, with the support of an analytical methodology designed for the evaluation of crack propagation rate in elastic-plastic regime, can be succesfully used to determine the hydrogen induced cracking behaviour of microalloyed and low alloy steels in aggressive environments. Besides material variables and environmental influences, the loading displacement velocity affects this behaviour in a decisive way. So lower than a critical displacement rates are needed to obtain stable values, similar to those obtained under constant displacement conventional tests. The critical velocities are not constant. They depend on the susceptibility of the material to cracking processes, on the environment and on the geometry of the specimen and the crack, which defines the relationship between external displacement rate and local strain rate. These types of SCC and HIC processes found in these steels to be changeable with displacement rate question the use of universal behaviours obtained by conventional methods.

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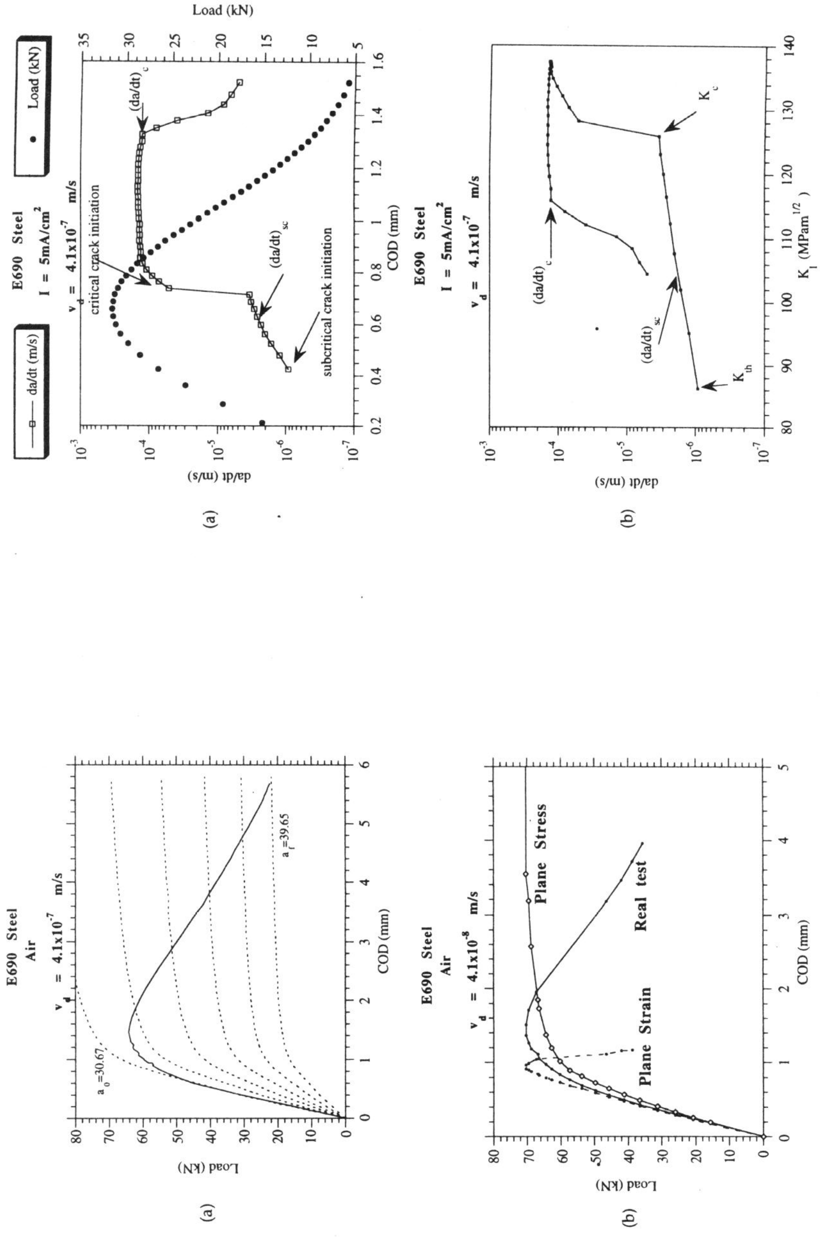


Figure 1. (a) Obtention of crack evolution from Load-COD curves. (b) comparisons of real behaviour with plane strain and plane stress states.

Fig. 2. Definition of crack characteristic parameters.