AN INVESTIGATION ON THE THRESHOLD STRESS INTENSITY FACTOR FOR ENVIRONMENT ASSISTED CRACKING OF A Ni-Cr-Mo STEEL BY A SIMPLE TECHNIQUE

K.K. RAY and G.R. RAO

Department of Metallurgical Engineering,
Indian Institute of Technology, Kharagpur-721 302, India

ABSTRACT

The influence of tempering temperature on the threshold stress intensity factor for environment assisted cracking (K_{IEAC}) of a Ni-Cr-Mo structural steel in 3.5% NaCl solution has been investigated. The threshold load for subcritical crack initiation has been determined using rising step load bend (RSLB) test. But the magnitude of K_{IEAC} has been estimated using a notch root dependent correction factor. The findings demonstrate that the order and the trend of variation of K_{IEAC} with tempering temperature for this structural steel are in close agreement with some earlier results which have been obtained by long duration experiments. The variation of K_{IEAC} with the hardness of the different microstructural states of the material has also been discussed.

KEYWORDS

Environment assisted cracking (EAC), threshold stress intensity factor for EAC (K_{IEAC}), Ni-Cr-Mo steel, tempering temperature, rising step load bend (RSLB) test.

INTRODUCTION

High stength Ni-Cr-Mo structural steels are known to be susceptible to subcritical cracking in presence of aggressive environments. There is a growing concern to characterise this susceptibility to environmental assisted cracking by a threshold stess intensity factor (K_{IEAC}) in order to use the latter parameter for structural integrity analysis. Extensive efforts by round robin test programmes have been made over the last two decades as to recommend standard practice for determining K_{IEAC} using wedge opening loaded (WOL), cantilever beam (CB) (Wei and Novak, 1987, Yokobori, Watanabe and Aoki, 1988) and double cantilever beam (DCB) specimens (Sponseller, 1992). The estimation of K_{IEAC} using fatigue-precracked conventional specimens (WOL, CB and DCB) requires long test durations of

4000-7000 h (Yokobori and others, 1988) depending on the material, the environment, and the initial stress intensity factor(K_{IO}) for a single test.

In eighty's emerged an alternate rising step load bending (RSLB) technique (Raymond and Crumly,1982,Raymond and associates,1987,Crumly,1988), by which K_{IEAC} can be estimated in a much shorter duration. The requirement of fatigue precracked specimens is also inherent in this technique. The values of K_{IEAC} estimated either by the long duration conventional tests or short duration RSLB test depend on K_{IO}, and test results obtained on machined notched specimens are questionable owing to the crack tip root radius (ρ) dependence of K_{IEAC} (Chu,Hsiao and Li,1979). The aims of this investigation are three fold: i)to elucidate a new principle for determining K_{IEAC} , ii) to determine K_{IEAC} of a steel having different microstructural states and iii) to examine the appropriateness of the suggested technique by comparing results obtained by this technique with those obtained from conventional tests for similar materials.

EXPERIMENTAL PROCEDURE

Material, Heat Treatment and Specimen preparation

The chemical composition of the steel is shown in Table 1. The as-received material was in the form of a billet with cross section 125x125 mm. Test coupons in T-L orientation measuring 11x11x55 mm dimensions having a 45° V-notch of 5 mm depth and side grooves of 3 mm depth were fabricated for K_{IEAC} testing. A series of approximately 12x12x12 mm dimension samples was also made for microstructural examinations and hardness testings. All specimens were soaked at 840°C (1113 K) for 30 min and oil quenched. The specimens were then grouped into different sets and were tempered in salt baths at 200, 250, 300, 350, 400 and 500°C for time duration of 30 minutes. The K_{IEAC} test specimens were then subjected to final grinding and notch cutting using a diamond cutter to achieve the specimen configuration as shown in Fig.1.

TABLE 1 Material Composition

Element	C	Mn	Si	Cr	Ni	S	P	Mo	V
Wt%	0.4	0.9	0.28	0.55	0.52	0.011	0.033	0.2	0.003

Metallographic Examination and Inclusion Characterisation

The prior austenite grain size was measured using linear intercept method following ASTM standard E 112 and was found to be 9.6 μm. A comparative assessment of the microstructures was made using an optical microscope. All the heat treated samples revealed fully tempered martensitic structures indicating appropriate quenching operation. The volume fraction of the inclusions was determined using Japanese standard method GO 555 and was found to be 0.4%.

Hardness Test

Microhardness measurements were carried out using a digital microhardness tester LECO DM 400. Fifty measurements were made for each differently heat treated specimen using a load of 50g for 15sec with the help of a Vicker's pyramid indentor.

Fracture Tests

The side grooved Charpy type specimens (Fig.1) were subjected to three point bend loading following ASTM E 399 in a four ton Universal Testing Machine in air and for environmental study in a specially designed test rig as shown in Fig.2. For measurements of K_{IEAC}, the load was increased in steps of 10 Kg and each specimen was immersed in fresh aqueous solution of 3.5 % NaCl (pH approximately 5.5). The loads at which the samples broke were recorded for computing apparent fracture toughness in air (K_{IX}) and apparent fracture toughness in 3.5% NaCl solution ($K_{IEAC}(\rho)$).

Fractographic Studies

Typical fracture surfaces of K_{IX} and K_{IEAC} specimens were examined using a CAMSCAN scanning electron microscope, and a series of fractographs were taken to understand the fracture morphology at the specimen notch tip.

An Outline of the Simple Technique

The major difference in the principles for determining K_{IEAC} by the conventional (using CB, WOL and DCB specimens) and the RSLB technique is as follows: in the former, one determines K_{IEAC} at a point below which the rate of crack growth is insignificantly small, whereas in the latter, one estimates K_{IEAC} at a point where crack initiates in aggressive environments. In both these test methodologies fatigue-precracked specimens are taken in order to initiate crack from a sharp tip. But under the influence of K_{IO} and the aggressive environment, the tip of a fatigue crack is expected to get blunted by uncertain magnitudes. Under these conditions a definite small ρ value should exist at the crack tip. The dependence of K_{IEAC} on ρ has been shown to be (Chu, Hsiao and Li, 1979) as:

$$K_{IEAC}(\rho) = A(\rho)^{1/2} \qquad \text{....(1)}$$

Equation(1) indicates that if one determines K_{IEAC} at a known ρ value, it would be possible to estimate K_{IEAC} for an alternate crack tip root radius. Thus it is possible to determine K_{IEAC} using a blunt notch specimen with known ρ value using RSLB technique, and then compute the value of K_{IEAC} corresponding to a sharp notch configuration similar to the one prevailing in a fatigue precracked specimen stressed for EAC under any particular K_{IO}. For structural steels, the critical value of ρ ahead of fatigue precracks under aggressive environments is ≈ 27 μm (Chu, Hsiao and Li, 1979). Using this critical value of ρ and that at the blunt notch tip, an attempt has been made to determine K_{IEAC}. In this report critical stress intensity factors determined from the threshold loads for the blunt specimens are designated as $K_{IEAC}(\rho)$, whereas, the calculated values corresponding to ρ=27 μm are referred as K_{IEAC}.

RESULTS AND DISCUSSION

Microhardness Investigation

An extensive investigation was carried out on the microhardness of the samples with different microstructural states. This had a two fold objective: i) to characterise the homogeneity of the generated microstructures, and ii) to acquire an indirect knowledge about the strength of the differently heat treated specimens. To understand the homogeneity of the microstructures, histograms of related microhardness (defined as a ratio of a particular hardness to the average of the set to which it belongs) values were prepared. A typical histogram is shown in Fig.3, which indicates Gaussian distribution. The nature of this relative microhardness distribution classifies the tempered microstructures as pseudo-single phase ones (Ray and Mondal, 1992).

The strength of a material can be obtained by multiplying the average microhardness of a material with a suitable conversion factor. A conversion factor of 3 was obtained by correlating the strength and hardness data of an earlier report (Pawlowski, Mazur and Gorczyea, 1991). The conversion factor has been obtained from strength and macrohardness, and was used here for microhardness results. The appropriateness of this has been verified by checking the proportionality between macro and microhardness (taken at 50gms load) values for this type of materials (Ray, Ghosh and Bhaduri, 1993).

The average microhardness values of the different samples were plotted against the corresponding tempering temperatures in Fig.4, which reveals an inflection in the plot indicating the region of tempered martensitic embrittlement. Similar plots in earlier reports (Pawlowski, Mazur and Gorczyea, 1991), however, present a monotonic decrease of hardness with tempering temperature. Coincidentally, when this investigation was carried out similar results of microhardness variation with tempering temperature also came out from a series of experiments on EN18, EN19 steels (Ray, Ghosh and Bhaduri, 1993). These results yield an inference that a plot between microhardness and tempering temperature can reveal the tempered martensitic embrittlement zone around 300°C. This view has been substantiated using some recent results on dilatometric measurements.

CRITICAL STRESS INTENSITY FACTOR MEASUREMENT

Three apparent critical stress intensity factors have been estimated- $K_{IEAC}(\rho)$, K_{IEAC} and K_{IX}. The measurements of $K_{IEAC}(\rho)$ have been done using the fabricated set-up (Fig.2). The threshold loads in these tests have been determined by loading the samples (in 3.5% NaCl solution) discontinuously with a load increment of 10 Kg and holding at each stage for 30 minutes. The load at which the specimen broke was recorded for the computation of $K_{IEAC}(\rho)$. A typical load time record is shown in Fig.4. The value of $K_{IEAC}(\rho)$ has been computed using the expression (Kies and others, 1965):

$$K_{IEAC}(\rho) = \frac{4.12\ M\ (1/\alpha^3-\alpha^3)^{1/2}}{(B.B_N)^{1/2}\ (W)^{3/2}} \quad ....(2)$$

Where α = 1-a/w, M = bending moment = PL/4, P = load of fracture, L = span length, a = notch length, W = width of the specimen, B = thickness of the specimen and B_N = reduced thickness. The estimation of K_{IEAC} has been made using the values of $K_{IEAC}(\rho)$ and eqn.(1). The value of ρ in the fabricated specimens was 320 μm whereas a value of ρ = 27 μm has been considered for K_{IEAC} calculations. Following an earlier investigation (Peterson and others, 1967), an apparent fracture toughness of the material in air (K_{IX}) was estimated using same loading sequence and using the eqn.(2) on identical side grooved Charpy test specimens (Fig.1).

A plot of K_{IX} and $K_{IEAC}(\rho)$ versus tempering temperature was made in Fig.5. The variations of these apparent critical stress intensity factors with tempering temperature are similar, and both the curves indicate minimum stress intensity factor values at 300°C. The relative difference between K_{IX} and $K_{IEAC}(\rho)$ is more at lower tempering temperatures corresponding to higher strength values of the material; this trend is in good agreement with an earlier report (Peterson and others, 1967).

In two earlier investigations (Pawlowski and others, 1991, Peterson and others, 1967), variation of K_{IEAC} with strength has been examined in similar quenched and tempered materials. Using the hardness strength conversion factor of 3 and using K_{IEAC} values a similar study was made in Fig.6. The orders of the estimated K_{IEAC} values are in good agreement with one set of earlier observations (Peterson and others, 1967), whereas the nature of variation of the estimated K_{IEAC} values with strength is similar to the second one (Pawlowski and others, 1991). The variations of K_{IEAC} (Pawlowski and others, 1991, the present) with tempering temperatures had been examined in Fig.7 to substantiate the latter aspect. However, to ascribe the reasons for the observed differences in the K_{IEAC} values between the two sets of observations in Fig.7, is a difficult task.

For each tempering temperature atleast three K_{IEAC} values have been determined as to acquire a knowledge of the standard deviation of the results. A typical histogram of K_{IEAC} values for the microstructural state corresponding to tempering at 400°C is shown in Fig.8. Interestingly the average value of K_{IEAC} for these specimens having a strength level of 1257 MPa is 19 ± 3.3 MPa√m as against the lower bound value of a similar material having a strength level of 1264 MPa is 21.7±1.4 MPa√m. The reported results of the interlaboratory test programme have consumed millions of hours compared to only about 60 hours in the current investigation to generate the data. It can thus be concluded that RSLB technique coupled with blunt notched specimens and considerations for ρ-correction, leads to a simple principle and a simple technique for K_{IEAC} measurement of materials with good reliability.

The scanning electron microscopic examinations revealed some differences at the crack tip of K_{IEAC} and K_{IX} test specimens. In K_{IEAC} samples distinct transverse crackings were observed unlike in K_{IX} test specimens. This could have been originated due to commonly observed crack branching under environment assisted cracking.

SUMMARY

A simple technique for measuring the threshold stress intensity factor of a material in aggressive environment using Charpy type specimens has been developed. The order and the nature of variation of K_{IEAC} estimated by the developed technique against the tempering temperature or the estimated strength of the varying microstructural states, are in good agreement with earlier reports on similar materials. The variation of K_{IEAC} with tempering temperature delineates the tempered martensitic embrittlement zone.

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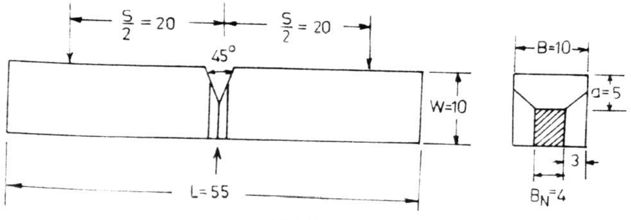
REFERENCES

Chu,W.Y., Hsiao,C.M. and Li,S.Q. (1979). A New Engineering Fracture Toughness Parameter $K_{Iscc}(\rho)$. Scripta Metallurgica, 13, 1057-1062.
Crumly,W.R. (1988). Screening Tests for Hydrogen Stress Cracking Susceptibility: Hydrogen Embrittlement Prevention and Control. ASTM STP 962, 173-177.
Kies,J.A., Smith,H.L., Romine,H.E. and Bernstein,H. (1965). Fracture Testing of Weldments: Fracture Toughness Testing. ASTM STP 381, 328-356.
Pawlowski,B., Mazur,A. and Gorczyea,S. (1991). The Effect of the Tempering Processes on the Susceptibility to SCC of High Strength Steels. Corrosion Science, 685-691.
Peterson,M.H., Brown,B.F., Newbegin,R.L. and Groover,R.E. (1967). Stress Corrosion Cracking of High Strength Steels and Titanium Alloys in Chloride Solutions at Ambient Temperature. Corrosion, 23, 142-148.
Ray,K.K. and Mondal,D. (1992). Some Aspects of Deformation Behavior of Coarse Multiphase Metallic Materials. Metallurgical Transactions, 23A, 3309-3315.
Ray,K.K., Ghosh,A. and Bhaduri,A. (1993). Unpublished work.
Raymond,L. and Crumly,W.R. (1982). Accelerated, Low-Cost Test Method for Measuring the Susceptibility of HY-Steels to Hydrogen embrittlement. Proc. Conf. on Current Solutions to Hydrogen Problems in Steels, Washington,D.C, U.S.A., 477-480.
Raymond,L., Raymond,L. and Associates (1987). Evaluation of Hydrogen embrittlement. Metals Hand Book, 13, 283-290.
Sponseller,D.L. (1991). Interlaboratory Testing of Seven Alloys for SSC Resistance by the Double Cantilever Beam Method. Corrosion, 48, 159-171.
Wei,R.P. and Novak,S.R. (1987). Interlaboratory Evaluation of K_{Iscc} and da/dt Determination Procedures for High Strength Steels. ASTM J. Testing and Evaluation, 15, 38-51.
Yokobori,T., Watanabe,J. and Aoki,T. (1988). Evaluation of the K_{Iscc} Testing Procedure by Round Robin Tests on Steels: Fracture Mechanics: Eighteenth Symposium. ASTM STP 945, 843-866.



(All dimensions are in mm)

Fig.1 A typical test specimen configuration.

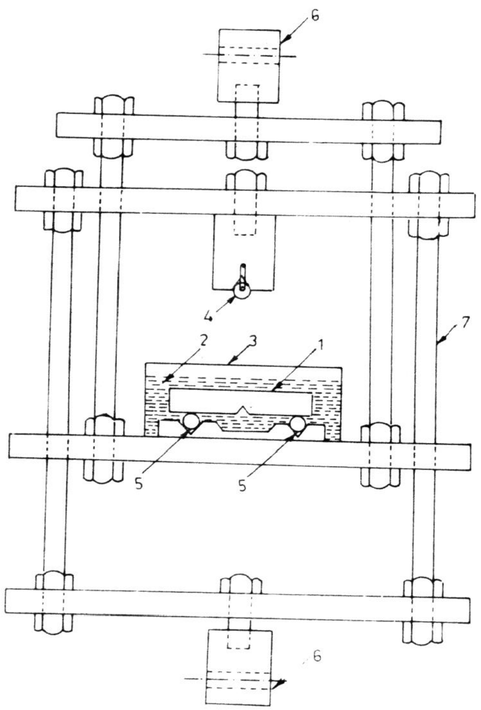


Fig.2 The test rig for K_{IEAC} determination
1. Specimen
2. 3.5% NaCl solution
3. Solution container
4. Upper roller
5. Lower roller
6. Attachments to the rams of a tensile machine
7. Loading frame.

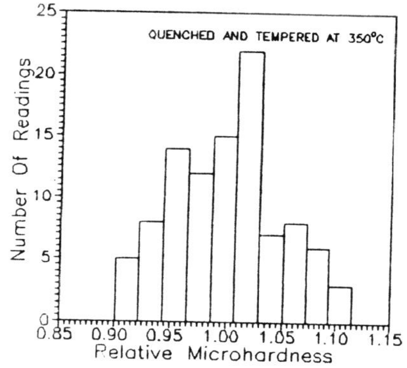


Fig.3 Typical distribution of relative microhardness values in a specimen

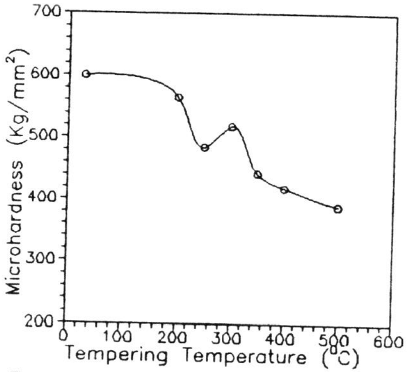


Fig.4 Variation of microhardness with tempering temperature.

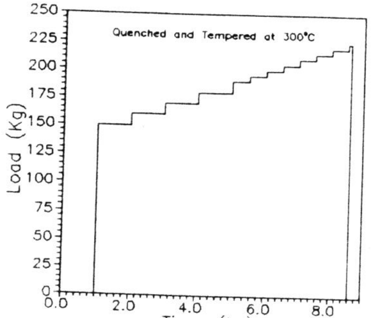


Fig.5 Typical load–time plot for a rising step load bend test

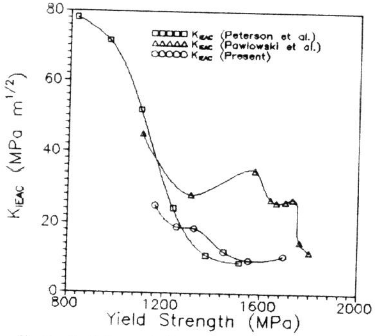


Fig.6 Variation of K$_{IEAC}$ with strength as reported in different investigations

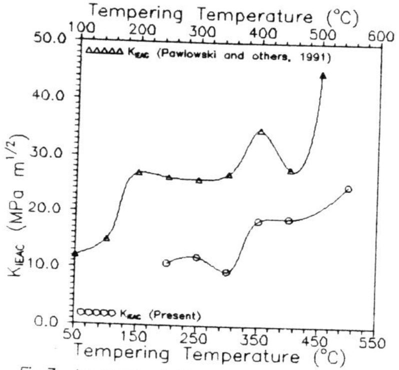


Fig.7 Variation of K$_{IEAC}$ with tempering temperature

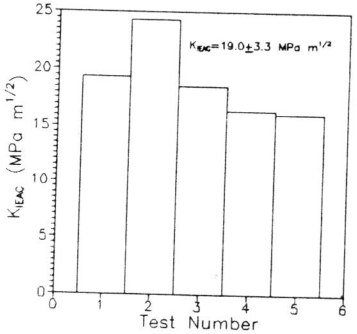


Fig.8 A typical histogram of K$_{IEAC}$ values for samples oil quenched and tempered at 400°C