# Validation of Circumferential Notched Tensile (CNT) Test Procedure for KISCC Determination

O. Phillips Agboola, Filiz Sarioglu and Cafer Kızılors

***Abstract*—It is well known that a great proportion of failures of industrial structures operating in a corrosive environment occur by stress corrosion cracking (SCC). Life prediction of such components involves the application of linear elastic fracture parameters such as stress intensity factor, $K_I$ that is a measure of material's resistance to crack propagation. $K_{ISCC}$ is the threshold value of the stress intensity factor below which a present crack stays stationary under the stress corrosion conditions. Therefore, determination of the threshold stress intensity ($K_{ISCC}$) is essential during material selection for any mechanical engineering design. $K_{ISCC}$ has traditionally been determined using specimens of fracture mechanics with geometries named as compact tension (CT) and double cantilever beam (DCB) test specimens. Such specimens are generally expensive to manufacture and test, requiring relatively expensive testing systems. A rapid and cost-effective determination of KISCC is of great interest to design engineers. Recently in literature alternative test piece geometry called Circumferential Notch Tensile (CNT) specimen has been proposed for producing $K_{ISCC}$ data. Smallness of CNT specimens reduces the cost of the test because of requirement of small loading devices to obtain the desired stress intensities and small amounts of testing material. In this study, for constant loading, a set-up has been designed and manufactured. CNT specimens were produced from AISI 4140 steel after austenizated at 850°C; water quenched and tempered 450°C. The corrosive environment was 33% NaOH (caustic) at 80oC. Specimens were loaded under constant loads until they are broken. Results showed that $K_{ISCC}$ of AISI 4140 was determined as 55 MPa√m. In literature this value in the same environment was given as 59 MPa√m for compact tension specimen produced from the same steel where crack growth was monitored by the potential drop method. Thereby results validate the CNT testing technique for determination of $K_{ISCC}$.**

***Index Terms*— AISI 4140, Circumferential Notched Tensile (CNT) Test, LEFM, Stress Corrosion Cracking (SCC), Threshold stress intensity (KISCC)**

## I. INTRODUCTION

Despite the introduction of polymers and composites in recent years, metals remain important in structures because of their strength, stiffness, toughness and tolerance of high temperatures, but in practice, metals are subject to all forms of corrosion activities (except the noble metals e.g. gold, platinum). Stress Corrosion Cracking (SCC) is an insidious form of corrosion; it produces a marked loss of mechanical strength with little metal loss; the damage is not obvious to casual inspection and the stress corrosion cracks can trigger fast mechanical fracture and catastrophic failure of components and structures [1]. Undetected stress corrosion cracking of in service component has long been responsible for major safety concerns, waste in production time, and cost in the maintenance of the material in some of major industries, such as the chloride in marine application and caustic SCC in alumina processing and pulp industries[2-5]. The stresses can be the result of the crevice loads due to stress concentration, or can be caused by the type of assembly or residual stresses from fabrication. Cold deformation and forming, welding, heat treatment, machining and grinding can introduce residual stresses, the magnitude and importance of such stresses is often underestimated [6]. The residual stresses set up as a result of welding operations tend to approach the yield strength. The build-up of corrosion products in confined spaces can also generate significant stresses which is often times overlooked. In order for SCC to occur, a susceptible material, an environment that will cause cracking of that material and a high enough stress or stress intensity factor is required (Figure 1.)

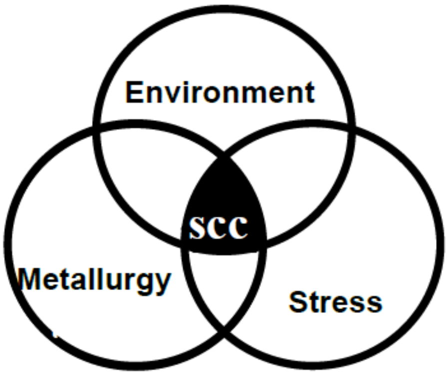

Figure 1. Venn diagram for SCC [1, 7]

In the past, numerous test methods have been developed to assess the susceptibility to stress corrosion cracking. Initially only smooth specimens were used to measure the time-to failure. Time to failure of these specimens is predominantly governed by the crack initiation period

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followed by the crack growth period until fracture. Since the mid-1960s there has been an increasing use of pre-cracked specimens. These specimens enable the application of fracture mechanics concepts to stress corrosion crack growth. One of the parameters which characterize the susceptibility of materials to SCC growth is KISCC, which is the threshold value of the stress intensity factor for the onset of stress corrosion crack growth. KISCC has traditionally been determined using specimens of fracture mechanic with geometries named as compact tension (CT) and double cantilever beam (DCB) test specimen. Such specimens are generally expensive to manufacture and test, requiring relatively expensive testing systems.

Caustic stress corrosion cracking of high strength low alloy (HSLA) steels has been of continuing concern for many Industrial fields: the alkali hydroxides fabrications, oil refineries, paper industries, alumina processes, and generally all the processes that imply the utilization of caustic products especially at high temperature. In order to establish the limit of employ of various HSLA steels in caustic media many laboratory tests have been conducted. The effect of tempering temperature on the microstructure of HSLA was especially studied in the works of Gutierrez-Solana et al [8], Parkins et al [9], Sarioglu [10] and Arup, and Parkins [11]. But the work of Rihan, Raman and Ibrahim [12] in recent publications proposed a new method for determining KISCC, and claimed that the smallness of CNT specimen reduces the cost of the test because of requirement of small loading device to obtain the desired stress intensities and small amount of testing material. The new method claims that the CNT specimen achieves plane strain condition and low plasticity despite its small size [12]. Fracture toughness values determine using CNT specimen are claimed to be within ± 3% of the data generated using the ASTM compact tension (CT) specimen [12,13]. Accepted results have been achieved using 9.5 and 15 mm diameter CNT specimen, whereas for the same material, fracture toughness ($K_{IC}$) determination, using standard CT specimens, requires widths up to 80 mm.

## II. EXPERIMENTAL SET - UP

The CNT specimen was machine from AISI 4140 steel bars to the dimensions stated by Rihan, Raman and Ibrahim [11], in order to eliminate the machining scratches, the surface of each specimens were prepared with 600-grit emery paper, the specimen were later heat treated. The specimens were divided into three batches (A, B&C). A & B batches were heated to 850 °C for one hour then oil quenched. Following quenching, Batch A was tempered for one hour at 450°C to develop a range of strength. The batch B was tempered at 650°C for one hour to get another range of strength. Batch C was allowed cooling in the furnace to room temperature. (Table 1)

After oil quench, the specimens were carefully cleaned and degreased (since they were oil quenched) in acetone to avoid contamination of the solution, and to ensure homogeneous and reproducible surfaces for comparative evaluations. High precautions were taken to keep the integrity of the specimen surface after final preparation; that is, avoid finger print and rough handling that could affect the finish surfaces.

Table 1 Different heat-treatments applied to the AISI 4140 steel

| | Batch A | Batch B | Batch C |
|---|---|---|---|
| Austenizing Temp ($^oC$) | 850 | 850 | 850 |
| Austenizing Time (Hour) | 1 | 1 | 1 |
| Cooling after Austenizing | Oil quenched to room Temp | Oil quenched to room Temp | Cooled to room Temp in Furnace |
| Tempering Temp ($^oC$) | 450 | 650 | - |
| Tempering Time (Hour) | 1 | 1 | - |

Figure 2. CNT specimen

For the needed environment, 33% sodium hydroxide at 80 $^oC$ was used as corrosive medium. One Kilogram of NaOH reagent was mixed with two liters of distilled water to form the 33% sodium hydroxide solution. The CNT test specimen (Figure 2) was inserted between the chucks of the CNT test rig, the solution is then poured into the corrosion chamber then it is heater to 80oC before the load is applied as seen in Figure 3.

The controller unit displayed the current temperature as measured by the thermocouple in the corrosion chamber.

## III. DETERMINATION OF $K_{IC}$ BY THE CNT METHOD

The pre-cracked CNT specimen was subjected to a constant load, until failure. The relationship between the stress intensity factor (KI) and time-to-failure (Tf) was established and the threshold stress intensity factor (KISCC) is determined [2-5, 12]. An accurate determination of KI was the key to the successful application of the technique.

The value of KI is determined from the fractured specimen, using Equations 1 - 9: [4]

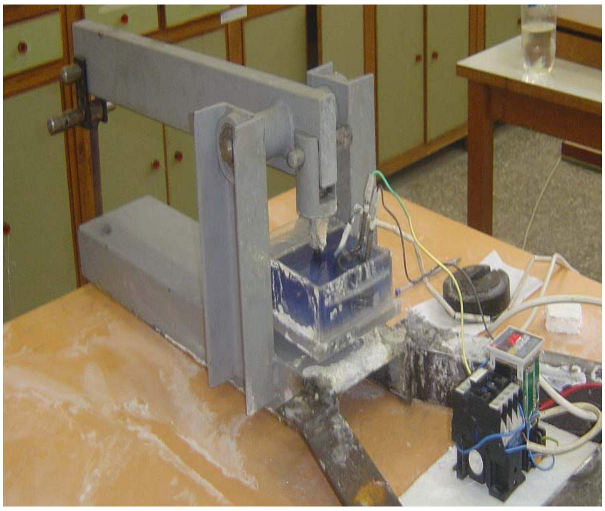

Figure 3. The Test Rig

$$K_1 = (\sigma_t + \sigma_b)\sqrt{a\pi F_o} \qquad (1)$$

$$a = \frac{D-d}{2} \qquad (2)$$

$$\sigma_t = \frac{4P}{\pi D^2} \qquad (3)$$

$$\sigma_b = \frac{16P\varepsilon}{\pi D^3} \qquad (4)$$

$$F_o = F e^{\propto\left(\frac{a}{D}\right)} \qquad (5)$$

$$F - \frac{1.25}{\left[1-\left(\frac{2a}{D}\right)^{1.47}\right]^{2.4}} \qquad (6)$$

$$\propto = 22.188 e^{-4.889\left(\frac{2a}{D}\right)} \qquad (7)$$

F and $F_o$ are geometric functions (no unit) for round specimens, with and without eccentricity, respectively. The term 'α' is a constant (with no unit), and its value can be calculated from Equation (7). P is the applied load in Newton; D, the specimen diameter; d, the equivalent ligament diameter; and a, the effective crack length where all dimension were in meters. Linear elastic fracture mechanics is applicable with the assumption that the materials deform in an elastic brittle manner. In reality, engineering materials undergo some plastic deformation at the crack tip before they fail. The LEFM approach is only valid if the size of the plastic zone is negligible compared to the specimen geometry. Therefore, specifications for determining the validity of measuring $K_I$ were developed. The validity requirements for measuring $K_I$ are

$$a_f \geq 2r_y \text{ and } \frac{\sigma_N}{\sigma_y} \geq 2.5 \qquad (8)$$

Where

$$a_f = \varepsilon + \left(\frac{D-d-2a_m}{2}\right) \qquad (9)$$

$\sigma_N$ is the nominal applied stress (in $N/mm^2$=Pa) in the final ligament, and $\sigma_y$ is the 0.2% offset tensile yield stress (Pa). Validity limits require the fatigue crack depth to be at least twice the Irwin plastic zone, and the average stress across the ligament after fatigue cracking should not exceed 2.5 times the yield strength [4, 5, 11]. These limits were also applied to those cases where the final ligaments were eccentric to the specimen centerline. With an eccentric ligament, the maximum nominal stress considered was then a combination of tensile and bending stresses, which should not exceed 2.5 times the yield strength. Further, for such eccentric ligament cases, the fatigue crack depth considered for the purposes of validity was taken to be the greatest depth of the crack.

## IV. RESULT

A plot of the calculated values of KI against time-to-failure (Tf) of heat treated 4140 steel in 33% caustic solution at 80 °C is shown in Figure. 4.

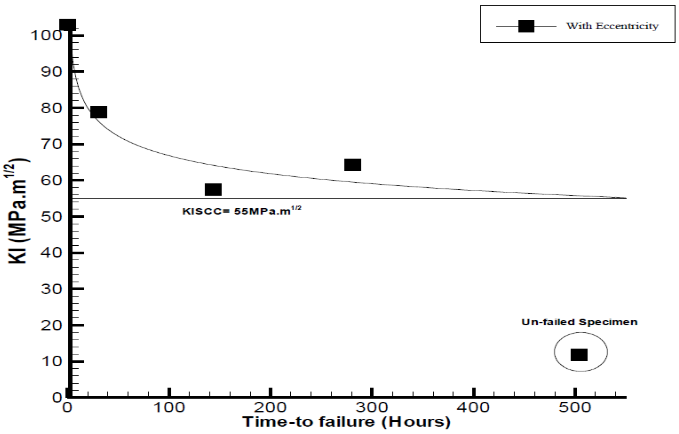

Figure 4. Stress Intensity Factor ($K_I$) versus Time –to Failure ($T_f$) of AISI 4140 in 12.5M NaOH at 80°C

As shown in Figure.4, $K_{ISCC}$ was determined at 80 °C to be 55 MPa√m, which is the minimum $K_I$ required for propagation of SCC crack. The specimen in the region below 55MPa√m should be immune to SCC. Indeed, as shown in Figure.4, a specimen placed in this region did not fail even after 504 hours of testing. The overall fracture surface shows an area of fatigue pre-crack ahead of the machined notch as well as the areas of cracking caused during the CNT test. The areas of machined notch, fatigue crack, SCC, and mechanical failure were identified on the fracture surface in Figure. 5. The $K_{ISCC}$ values of 55 MPa√m for AISI4140 Steel in 80oC NaOH solution suggest that once cracks initiate (in this case the pre-crack introduced), this material will easily suffer caustic cracking, that is, there will be crack propagation along this initial crack. This inference is in line with common cases found in literature [10]. There are few

$K_{ISCC}$ data available for AISI4140 Steel in $80^{o}$C NaOH solution.

Figure 5 Overall fracture surface of a AISI 4140 Steel CNT specimen tested in $80^{o}$C NaOH solution.

## V. CONCLUSION

In view of validation of the novel circumferential notch tensile testing technique for determination of the threshold stress intensity for stress corrosion crack propagation (KISCC) CNT tests were carried out on a high strength 4140 steel in 33% sodium hydroxide solution at 80oC temperature. The conclusions of this study are:

1. Experimental work carried out using CNT techniques with the constructed SCC test machine, to investigate the susceptibility of AISI 4140 steel to caustic stress corrosion cracking have suggested that the material suffered caustic cracking at different KI values in a NaOH solution at 80oC.
2. The KISCC of AISI 4140 steel in 33% NaOH solution at 80oC has been determined to be 55 MPa√ m.
3. The KISCC generated using CNT techniques is very close with KISSC generated in literature using the same material and environment but with compact tensile techniques, which was 59 MPa√ m.
4. The experimental CNT testing is relatively fast and cost-advantageous approach for generating the KISCC data.

## REFERENCES

[1] 1. Cottis, R.A. Guides to Good Practice in Corrosion Control, by National Physical Laboratory, p1 (www.npl.co.uk)

[2] 2. Ibrahim R.N., Rihan, R., Raman, R.K.S., validity of a New Fracture Mechanics techniques for the Determination of the Threshold Stress intensity Factor for Stress Corrosion Cracking (KIscc) and Crack Growth Rate of Engineering Materials. Engineering Fracture Mechanics,2007

[3] 3. R. Rihan, R.K. Singh Raman and R.N. Ibrahim, Determination of crack growth rate and threshold for caustic cracking (KIscc) of a cast iron using small circumferential notched tensile (CNT) specimens, Mater Sci Engng A 425 (1-2) (2006), pp. 272–277.

[4] 4. R. Rihan, R.K. Singh Raman and R.N. Ibrahim, Circumferential notched tensile (CNT) tests for generating KIscc data for cast iron vessels used in hot caustic solutions, Int J Pressure Vessels Piping 83 (5) (2006), pp. 388–393

[5] 5. R. Rihan , R.K. Singh Raman and R.N. Ibrahim , Circumferential notched tensile (CNT) testing of cast iron for determination of threshold (KIscc) for caustic crack propagation, Mater Sci Engng A 407 (1–2) (2005), pp. 207–212

[6] 6. Metal Handbook, 10th Edition, Properties and Selection: Irons, Steels, and High Performance Alloys. pp 148-155

[7] 7. Richard W. Hertzberg, Deformation and Fracture Mechanics of engineering Materials, Third Edition,pp381-386

[8] 8. J. Gonzalez, F. Gutiérrez-Solana and J.M. Varona. Met. Mat. Trans. 27A (1996) 291

[9] 9. R.N Parkins and P.M.Signh, Stress Corrosion Crack Coalescence, Journal Corrosion, Vol 46,No 6,1990

[10] 10. Filiz Sarioglu, The Effect of Tempering on Susceptibility to Stress Corrosion Cracking of AISI 4140 steel in 33% Sodium Hydroxide at 80oC,Material Science and Engineering A315 (2001) 98-102

[11] 11. Arup, H and Parkins, R. N. Stress Corrosion Research, NATO, 1979

[12] 12. R.K. Singh Raman: Metall. Mater. Trans. A, 2005, vol. 36A, p. 1817.

[13] 13. R.K. Singh Raman and B.C. Muddle: Int. J. Pressure Vessels Piping, 2004, vol. 81, pp. 557-61.