

# The Determination Of $K_{ISCC}$ Of Mild Steel In Hot Caustic By Using Small Circumferential Notched Tensile (CNT) Fracture Toughness Specimens

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**ABSTRACT:** A new technique for rapid and cost-effective determination of stress intensity factor ( $K_I$ ) and fracture toughness ( $K_{IC}$ ) using small circumferential notch tensile (CNT) specimens is presented and used to determine the crack growth rate of an ex-service component. The paper highlights the scope and advantages of extending this technique for assessing the susceptibility of ferrous alloys to stress corrosion cracking (SCC). A modified CNT testing rig is designed and constructed based on an existing room temperature sustained load crack testing rig by adding facilities for testing SCC susceptibility at different temperatures. The new testing rig simplifies and speeds up the testing procedure as well as provides considerable cost advantage over the conventional fracture mechanics techniques. The paper also presents results of the SCC tests carried out on mild steel (AISI 1020) using this modified CNT rig.

## 1. INTRODUCTION

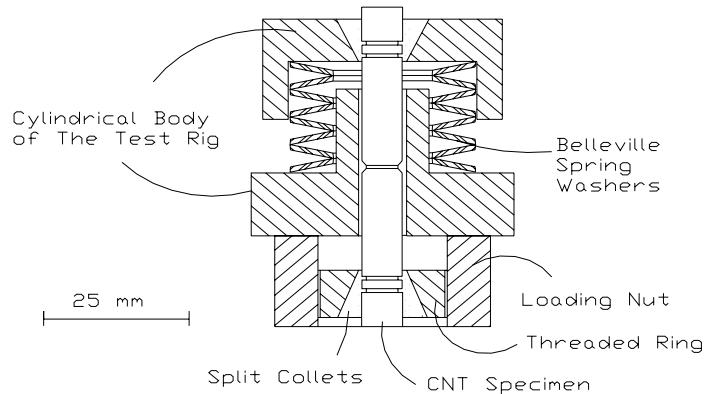
Stress corrosion cracking (SCC) is believed to be the most dangerous form of corrosion-assisted failure of materials, since the stress corrosion crack can lead to sudden failures. SCC of in-service components has long been responsible for major safety concern, lost operating times and materials maintenance costs in the major Australian industries, e.g., chloride SCC in marine applications and processing industries, and caustic SCC in alumina processing and pulp and paper industries (Singh Raman and Muddle, 2003a, 2003b, 2004). Hence, life assessment of in-service components, particularly those exposed to in-service corrosive environments, generally involves assessment of their susceptibility to SCC. Susceptibility to SCC is also a major consideration for design engineers while selecting construction materials for corrosive environments.

Fracture toughness ( $K_{IC}$ ) is a measure of a given material's resistance to crack growth. Determination of stress intensity factor ( $K_I$ ) and its threshold value for crack propagation, is of considerable importance for selecting materials for any mechanical engineering design.  $K_{IC}$  has traditionally been determined using standard compact tension (CT) test specimens, which are expensive to manufacture and require relatively longer testing times. Technique of determination of fracture toughness using circumferential notch tensile (CNT) specimens has been developed in the last decade (Ibrahim, 1989, 1999; Ibrahim and Price, 1997; Ibrahim and Stark, 1990; Stark and Ibrahim, 1986). The CNT specimen is the smallest possible specimen that can produce valid plane strain crack loading conditions.

This paper discusses advantages of determining fracture toughness and crack growth using circumferential notch tensile (CNT) testing. The paper also presents modification of the usual CNT testing rig (Ibrahim, 1989; Stark and Ibrahim, 1992) (that have been used for general  $K_{IC}$  determination) into a SCC testing rig, with a view to simplifying and speeding up the testing procedure as well as providing a considerable cost advantage over the conventional fracture mechanics techniques for determination of  $K_{IC}$  under SCC-susceptible condition (i.e.,  $K_{ISCC}$ ). The paper also presents results of SCC tests carried out on mild steel (AISI 1020) and the  $K_I$  values using the modified CNT test rig.

## 2. CIRCUMFERENTIAL NOTCH TENSILE (CNT) TESTING TECHNIQUE

The room temperature sustained load CNT testing rig and the typical CNT specimen are shown in Figures 1a and b respectively. Ahead of the notch of the CNT specimen a fatigue pre-crack is developed by subjecting the specimen to a controlled rotating bending fatigue machine (Ibrahim, 1989, 1999; Ibrahim and Price, 1997; Ibrahim and Stark, 1987, 1990; Stark and Ibrahim, 1986). The pre-cracked CNT specimen is then subjected to a constant load, until the specimen fails.  $K_{IC}$  is determined by correlating the stress intensity ( $K_I$ ) with one of the crack propagation parameters, viz., crack-growth velocity or time-to-failure ( $T_f$ ) (Stark and Ibrahim, 1992). The  $K_I$  is determined from the fractured specimen, using a method described elsewhere (Ibrahim, 1989; Ibrahim and



Stark, 1987, 1990; Stark and Ibrahim, 1986).

Fig.1a The room temperature sustained load circumferential notch tensile (CNT) testing rig

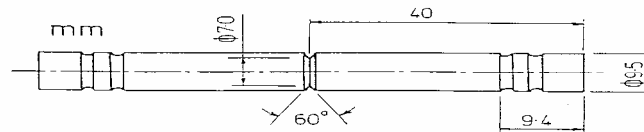


Fig.1b The CNT specimen (9.5 mm diameter version)

The use of CNT specimen has many advantages. The small cross-section of the specimen makes it possible to achieve quite high levels of loading by using moderate loads. This means either small load frames can be used or small machines are possible for applying the mechanical loads. Reducing material requirements, which is a critical requirement when microstructures of limited size such as welds are to be investigated. The relevant microstructure need only be produced in the small volume of metal in the notched region. In many cases material over 20 mm or even 10 mm thickness may not be available. This is characteristic of many pressure vessels and structural steels, especially expensive materials such as stainless steels or duplex stainless steels. If post exposure or aged samples are to be taken out of old structures, there is also likely to be quite severe size restrictions. The CNT specimen is cheap to produce because of its small cylindrical shape reducing the cost of each specimen by up to a factor of 10 (c.f. CT specimen).

The CNT specimen achieves plane strain conditions and low plasticity despite its small size (Pickles, 1983). This is important because, for valid  $K_I$  testing, it is required that the conditions appropriate to linear elastic fracture mechanics exist. For this condition to be the case, only a minimal amount of plastic flow during fracture is permitted. Fully valid plane strain specimens are generally required to be relatively large and bulky in order to satisfy the high restraint requirements for test validity.

A reproducible crack-geometry is generally achieved by fatigue pre-cracking. It is important to note that the shape and stress intensity of fatigue crack tips produced in CNT specimens are highly reproducible and measurable.

Fracture toughness values determined using CNT specimens are claimed to be within  $\pm 3\%$  of the data generated using the ASTM compact tension (CT) specimen (Ibrahim and Price, 1997; Stark and Ibrahim, 1986). In tests conducted earlier at Monash University, acceptable results have been achieved using 9.5 and 15 mm diameter CNT specimens, whereas for the same material, fracture toughness ( $K_{IC}$ ) determination, using standard CT specimens, requires widths up to 80 mm.

### 3. DETERMINATION OF $K_{ISCC}$ BY CNT METHOD

#### 3.1 Modification of CNT testing rig for SCC Monitoring

The original CNT testing rig (shown in Figure 1b) that was designed for testing in air required considerable modification for testing in aggressive environments such as high temperatures caustic solutions. Modification includes a corrosion cell (made out of Monel 400), facilities for heating of the corrosive solution, temperature control and thermal insulation. The modified rig (shown in Figure 2) also consists of facilities for electrochemical testing, such as the reference and counter electrodes.

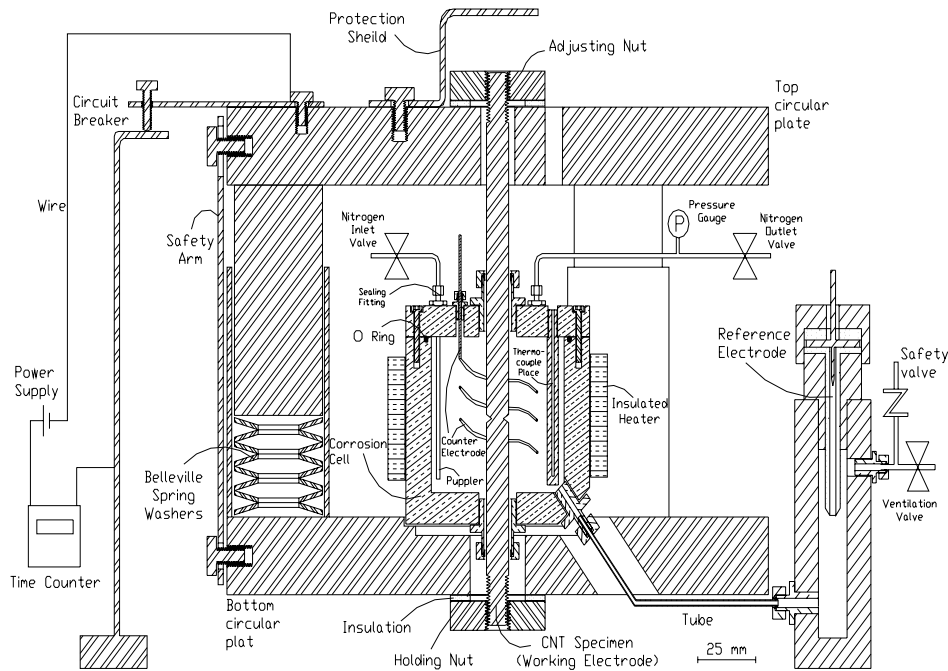


Fig.2 Modified CNT rig for simultaneous mechanical and electrochemical testing

### 3.2 Determination of $K_{ISCC}$ of mild steel in caustic solution

The material used in this investigation is a common mild steel (AISI 1020 grade). Its chemical composition is illustrated in Table 1. The yield stress ( $\sigma_y$ ) was determined experimentally to be 613 MPa. CNT specimens (shown in Figure 3) were machined out of 9.5 mm diameter rods of mild steel (AISI 1020 grade) with 190 mm length to suit the modified test rig.

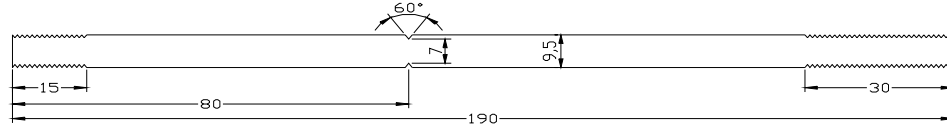


Fig.3 CNT specimen for the modified rig (all dimensions in mm)

Table 1 Chemical composition (wt%) of AISI 1020 grade mild steel

C	P	Mn	Si	S	Ni	Cr	Cu
0.23	0.012	0.84	0.180	0.026	0.01	0.02	0.01

The specimens were subjected to fatigue pre-cracking, using a rotating bending fatigue machine. Then the surface of the specimen was polished with 800/1200 grit paper. The pre-cracked specimen was washed with ethanol and dried before being installed into the rig. The corrosion cell was filled with the given caustic solution (500gpl, i.e., 12.5M NaOH). The test rig was assembled as shown in Figure 2. The test rig was heated to the test temperature (100°C and 150°C) before applying the tensile load to the specimen. This procedure ensured that the applied load does not change as a result of the expansion of the specimen and surrounding metallic fittings. The specimen was held at a given load until it failed. The time-to-failure ( $T_f$ ) is recorded at the failure of the specimen. Tests were carried out at different applied  $K_I$ .

A plot of the calculated values of  $K_I$  against the time-to-failure ( $T_f$ ) is shown in Figure 4. The minimum determined values of  $K_I$  until the time of writing this paper (as shown in Figure 4) are 42.9 MPa.m<sup>1/2</sup> at 100°C and 27.7 MPa.m<sup>1/2</sup> at 150°C, which indicates that the corrosion rate and the susceptibility to SCC increases with increasing temperature as stated in the literature (Bohnenkamp, 1969; Mazille, 1972; Rihan, 2001). Despite that the CNT rigs are still running under different  $K_I$  values, these values are comparable with that of the literature. The  $K_{ISCC}$  of mild steel (AISI 1018) in 12.5 M at 92°C has been determined (Singbeil and Tromans, 1982) (using double cantilever beam (DCB) specimens) to be less than 18 MPa.m<sup>1/2</sup>. In another study (Sriram and Tromans, 1985),  $K_{ISCC}$  of a mild steel (ASME SA-516 Grade 70) at 92°C has been determined (using DCB specimens) to be 29 MPa.m<sup>1/2</sup> in 2 M NaOH, 21 MPa.m<sup>1/2</sup> in 4 M NaOH, and less than 20 MPa.m<sup>1/2</sup> in 8 M NaOH. While the difference in the calculated  $K_I$  values in the present study with  $K_{ISCC}$  values (using double cantilever beam (DCB) specimens) in the literature is attributed to the significant differences in the operating temperatures, steel grade, and the electrochemical conditions etc. it is emphasized that further experimental work is continuing in order to obtain the threshold values under different temperatures.

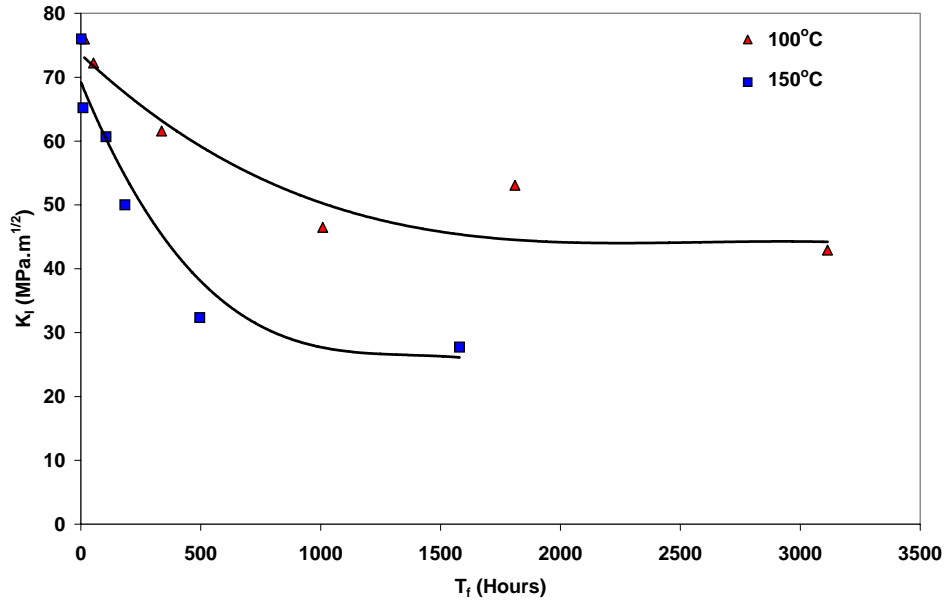


Fig.4  $K_I$  versus  $T_f$  of mild steel (AISI 1020) in 12.5 M NaOH at 100°C and 150°C

#### 4. FRACTOGRAPHY

Fracture surface of the failed specimens was cleaned using a special cleaning solution and fractography was carried out using scanning electron microscopy (SEM), in order to investigate the fractographic evidence of stress corrosion cracking. Fractographic features of the specimen tested using a  $K_I$  value of  $\sim 65 \text{ MPa.m}^{1/2}$  are shown in Figure 5.

The overall fracture surface (Figure 5a) shows an area of a uniform pre-crack ahead of the machined notch as well as the areas of cracking caused during the CNT test. Crack propagation in the area just ahead of the pre-crack appears to have been intergranular, followed by a region of overload ductile failure (Figure 5b). Indeed, at a higher magnification, a region of transition from intergranular cracking to the pure mechanical failure (ductile dimples) could be clearly seen in Figure 5c. The area of intergranular crack propagation (seen more clearly in Figure 5d) and secondary cracking confirmed intergranular caustic cracking.

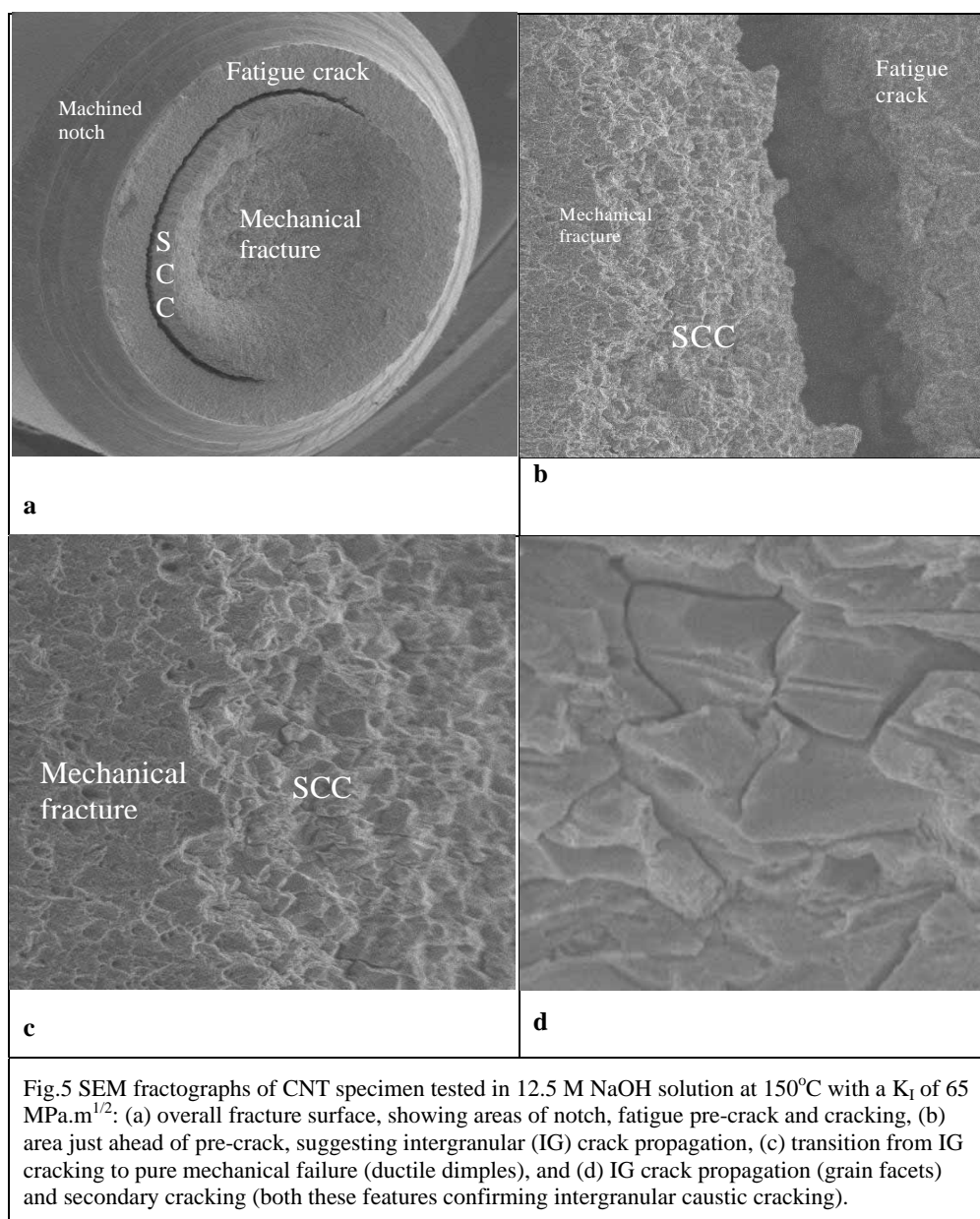


Fig.5 SEM fractographs of CNT specimen tested in 12.5 M NaOH solution at 150°C with a  $K_I$  of 65 MPa.m<sup>1/2</sup>: (a) overall fracture surface, showing areas of notch, fatigue pre-crack and cracking, (b) area just ahead of pre-crack, suggesting intergranular (IG) crack propagation, (c) transition from IG cracking to pure mechanical failure (ductile dimples), and (d) IG crack propagation (grain facets) and secondary cracking (both these features confirming intergranular caustic cracking).

## 5. CONCLUSIONS

Experimental work were carried out, using CNT testing approach and newly modified SCC test rig, to investigate the susceptibility of mild steel (AISI 1020) to caustic stress corrosion cracking have provided  $K_{ISCC}$  data at two different temperatures. The lowest obtained  $K_I$  values until the time of writing the paper were 42.9 and 27.7 MPa.m<sup>1/2</sup> at temperatures of 100°C and 150°C respectively, which is comparable with the  $K_{ISCC}$  values reported in the literature. The experimental CNT testing has shown potential for emerging the  $K_{ISCC}$  data as a simple, fast (compared with other fracture mechanics techniques) and cost-advantageous approach. The experimental CNT testing has showed that the susceptibility of mild steel (AISI 1020) to caustic stress corrosion cracking increases with increasing temperature.

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