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# The development of in situ fracture toughness evaluation techniques in hydrogen environment

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## ABSTRACT

Fracture toughness and fatigue properties of pipeline steels play a critical role in developing advanced high-pressure hydrogen infrastructure for alternative fuel pipelines program. The reliability of structure components, particularly resistance to damage and failure in the intended service environment, is highly dependent on the selected materials. An effective surveillance program is also necessary to monitor the material degradation during the course of service. Steels have been proven to be desirable for hydrogen infrastructure. However, hydrogen embrittlement is an important factor that limits steel performance under high-pressure hydrogen conditions. Furthermore, many conventional fracture testing techniques are difficult to be realized under the presence of hydrogen, in addition to the inherent specimen size effect. Thus it is desired to develop novel in situ fracture toughness evaluation techniques to study the fracture behavior of structural materials in hydrogen environments. In this study, a torsional fixture was developed to utilize Spiral Notch Torsion Test (SNTT) methodology. A fatigue pre-crack procedure of SNTT approach was also developed and demonstrated for weldment. The in situ testing results indicated that the exposure to H<sub>2</sub> significantly reduces the fracture toughness of 4340 high strength steels by up to 50 percent. Moreover, in-air simulated heat-affected zone specimen by Gleeble demonstrated a significant fracture toughness reduction of 75 percent in samples which illustrated the effect of welding on the fracture toughness.

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## Introduction

### Background on fracture toughness evaluation

The Mode I (tensile opening mode) stress-intensity factor at the onset of rapid crack propagation under plane-strain conditions is defined as fracture toughness,  $K_{IC}$ , a controlling reference parameter used in design to avoid catastrophic brittle fracture. American Society for Testing and Materials

(ASTM) standard test methods, *Standard Test Method for Plane-Strain Fracture Toughness of Metallic Materials (E399) [1]*, *Standard Test Method for Measurement of Fracture Toughness (E1820)*, and *Standard Test Method for Determining Threshold Stress Intensity Factor for Environment-Assisted Cracking of Metallic Materials (E1681-03)*, are widely used to determine fracture toughness of metallic materials, using compact tension and compact disk tension specimens, and wedge opening load specimen, having thickness and volume sufficient to ensure the plane-strain condition at the crack front. However, meeting the specimen

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size requirements is difficult and impractical because structure systems materials to be investigated may be geometrically unsuitable and/or have insufficient volume for making the standard specimen. Therefore, use of small specimens for  $K_{IC}$  measurement is essential for application to pressure vessel safety surveillance.

Despite the international efforts on the development of small specimen testing techniques, no methods currently exist for direct measurement of  $K_{IC}$  for small specimens without a concern for size effect. A new method, designated as Spiral Notch Torsion Test (SNTT), is developed recently by the authors to measure the intrinsic fracture toughness ( $K_{IC}$ ) of structural materials [2–6]. The SNTT overcomes many of the limitations inherent in traditional techniques and makes it possible to standardize fracture toughness testing. The SNTT system operates by applying pure torsion to uniform cylindrical specimens with a notch line that spirals around the specimen at a 45° pitch. The  $K_{IC}$  values are obtained with the aid of a three-dimensional finite-element computer code.

In the old days, welds were almost always blamed for any structure-joint failure [7]. But researchers soon identified another suspicious region, heat-affected zone (HAZ), between the steel and the weld [8,9]. And they realized it was this vulnerable HAZ that had been causing much of the trouble. The detailed microstructure and the associated complex fracture morphology of weldment are shown in Fig. 1. The high hardness contours shown in Fig. 1a are associated with HAZ materials. The weld microstructure consisted of 3 regions; i.e., weld metal, HAZ, and base metal. The HAZ can be

further divided into several sub-regions with obviously different microstructures. The HAZ microstructure of a single-pass weld on martensitic steel can be defined as follow:

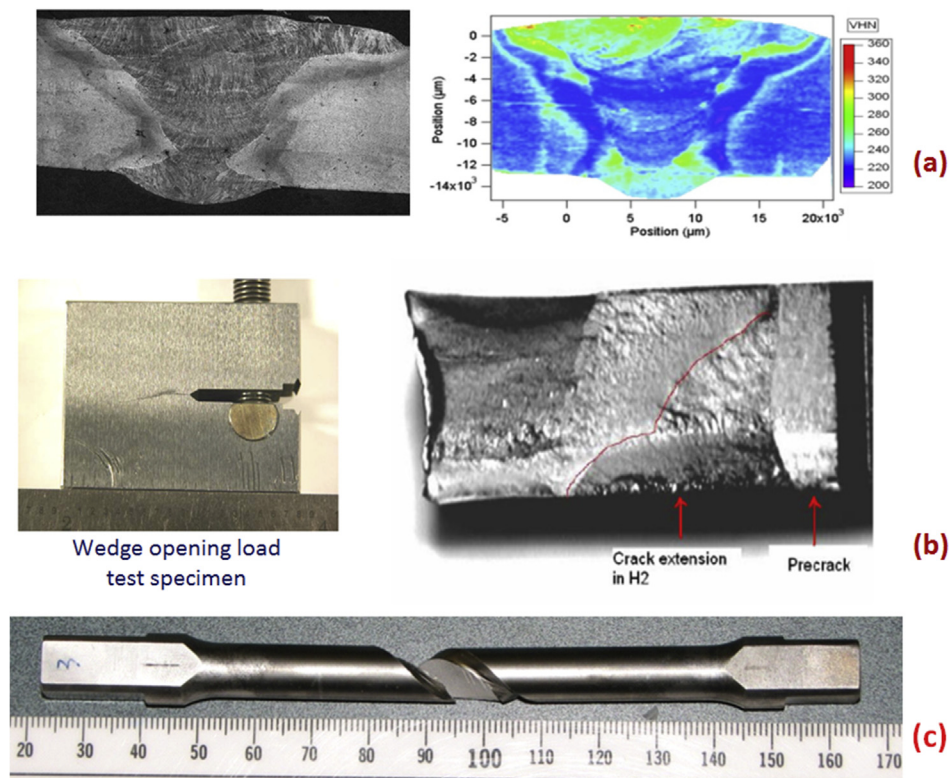
HAZ-1: subcritical region, where microstructure similar to that of the base metal,

HAZ-2: constitute the very fine microstructure,

HAZ-3: the grains are finer than that of base metal, but larger than in HAZ-2,

HAZ-4: coarse grain region located next to the fusion line, the highest hardness resided for ferrite steel.

Complications in HAZ testing and enormous data scatter have discouraged researchers from addressing the subject of HAZ fracture toughness [8–12]. For instance, to generate a credible HAZ fracture toughness data, it will require that the tip of the fatigue-pre-crack located at the coarse-grain region of the HAZ specimens. However, in general, the fatigue crack is advanced toward the soft part of metal; therefore, avoid the brittle coarse-grain region of HAZ. The lack of details of material properties for weld-HAZ, in addition to no consensus standards for HAZs toughness evaluation, has cast a great uncertainty regarding the integrity assessment of the weldment. Furthermore, pressure vessel steel weld-HAZ has potential for reheat cracking in the grain growth zone [13], and this reheat cracking is not only confined to the transformable steels but also in Ni-based alloys and austenitic stainless steels. Also, cold cracking (or hydrogen induced cracking) [14] and lamellar tearing are typically appeared in the HAZ. An



**Fig. 1 – (a) Distribution of micro-hardness shows highly non-uniform HAZ and weld cross section of X-80 weld, (b) Fractured X80 weld specimen in hydrogen shows significant distortion of crack growth and only cracked on one side of the specimen, (c) fractured SNTT weld sample shows smooth and uniform fracture surface contour.**

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