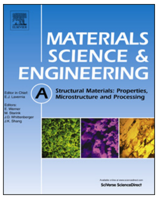
Materials Science & Engineering A 651 (2016) 859–868

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea



Investigation of hydrogen assisted cracking in acicular ferrite using site-specific micro-fracture tests

Walter L. Costin [a], Olivier Lavigne [a,*], Andrei Kotousov [a], Reza Ghomashchi [a], Valerie Linton [b]

[a] School of Mechanical Engineering, The University of Adelaide, SA 5005, Australia
[b] Energy Pipelines Cooperative Research Centre, Faculty of Engineering, University of Wollongong, NSW 2522, Australia

ARTICLE INFO

Article history:
Received 21 September 2015
Received in revised form
13 November 2015
Accepted 14 November 2015

Keywords:
EBSD
Micromechanics
Steel
Welding
Hydrogen embrittlement
Stress intensity factor threshold

ABSTRACT

Hydrogen assisted cracking (HAC) is a common type of failure mechanism that can affect a wide range of metals and alloys. Experimental studies of HAC are cumbersome due to various intrinsic and extrinsic parameters and factors (associated with stress, hydrogen and the materials microstructure) contributing to the hydrogen crack kinetics. The microstructure of many materials consists of diverse constituents with characteristic features and mechanical properties which only occur in very small material volumes. The only way to differentiate the effect of these individual constituents on the hydrogen crack kinetics is to miniaturise the testing procedures. In this paper we present a new experimental approach to investigate hydrogen assisted crack growth in a microstructural constituent, i.e. acicular ferrite. For this purpose, sharply notched micro-cantilevers were fabricated with a Focus Ion Beam within this selected microscopic region. Acicular ferrite can be found in many ferrous alloys including ferritic weld metal and has specific features that control its intrinsic susceptibility to HAC. These features were characterised via Electron Backscatter Diffraction and the specimens were subsequently loaded under uncharged and hydrogen charged conditions with a nano-indenter. The outcomes of the testing, demonstrated that the threshold stress intensity factor, K_{th}, to initiate crack propagation in acicular ferrite ranges between 1.56 MPa $m^{1/2}$ and 4.36 MPa $m^{1/2}$. This range is significantly below the values of K_{th} reported for various ferrous alloys in standard macro-tests. This finding indicates that the mechanisms and resistance to HAC at micro-scale could be very different than at the macro-scale as not all fracture toughening mechanisms may be activated at this scale level.

1. Introduction

Hydrogen assisted cracking (HAC) is a prevalent failure mechanism that affects most metals and alloys. Johnson in 1875 was the first to report about the deleterious effects of hydrogen on the mechanical properties of iron and steels [1]. This phenomenon is historically referred to as Hydrogen Embrittlement (HE) because macroscopically the presence of hydrogen in metals generally leads to reduction in fracture energy and ductility promoting brittle failure. The term HAC was later suggested by Beachem who argued that hydrogen assisted crack propagation may imply microscopic deformation processes that are not necessarily the result of the cessation, restriction, or exhaustion of ductility [2], however, from a mechanistic point of view both designations refer to the same phenomenon.

HAC can be classified as either Internal Hydrogen Assisted Cracking (IHAC), which is the main focus of the current paper, or Hydrogen Environment Assisted Cracking (HEAC). In IHAC, atomic hydrogen can be introduced throughout the microstructure by manufacturing operations (casting, welding, surface-chemical cleaning, electrochemical machining, electroplating, and heat treatment) as well as due to environmental exposure (cathodic electrochemical reactions or gaseous hydrogen exposure). Subcritical crack growth can occur if a hydrogen charged material with a susceptible microstructure is subjected to a sufficient stress level. Meanwhile, HEAC involves the conjoint action of mechanical loading and chemical reaction, i.e. the stress is necessary during the hydrogen uptake to facilitate cracking. Therefore, IHAC and HEAC are distinguished by the source of the supplied hydrogen but share common aspects, i.e. cracking occur in both cases when the three causal conditions are simultaneously present: (1) susceptible microstructure, (2) sufficiently high levels of hydrogen and (3) stress [3].

Hydrogen is widely considered as one of the future sources of

---

* Corresponding author.
E-mail addresses: Olivier.lavigne@adelaide.edu.au, lavigneolivier@hotmail.com (O. Lavigne).

http://dx.doi.org/10.1016/j.msea.2015.11.044
0921-5093/© 2015 Elsevier B.V. All rights reserved.

clean energy [4]. A large scale of production, storage and transportation of hydrogen is likely to be escaladed in the next few decades. Because of the technological importance of HAC for the past, present and future applications, greater amount of efforts have been directed to the development of experimental and theoretical approaches to predict and evaluate the effect of hydrogen on the life expectancy and integrity of structural components [2,3,5–9]. However, HAC continues to plague applications of, specifically, high strength metals and alloys [3]. Various, often controversial views [7,10–14] have been proposed to describe the physical mechanisms by which hydrogen actually affects plasticity and fracture resistance in metals. Detailed reviews of these approaches can be found in [3,15]. However, it is the Linear Elastic Fracture Mechanics that provides a basis allowing the incorporation of hydrogen cracking failure mechanism into structural integrity management [16]. In accordance with this fracture theory pioneered by Naval Research Laboratory in 1965 [17] a single stress related parameter, the stress intensity factor, K, governs the crack initiation and crack growth rates. This concept was confirmed by many other studies over the past 60 years [3,18–22]. From the structural integrity point of view three characteristics of the fracture behaviour have to be identified experimentally: (a) a threshold stress intensity factor, K_{th} (or other terminology), below which crack does not propagate, (b) crack growth rates (da/dt) at $K > K_{th}$ and (c) fracture toughness K_C. Effective laboratory methods have been developed and standardised [23] to determine the threshold and kinetics for hydrogen assisted cracking, which are necessary for the safe design of structures subjected to the risk of HAC. IHAC testing procedures may involve several stages: a fracture mechanics specimen, typically fatigue pre-cracked, which can be stressed under either constant or rising load, crack mouth opening displacement or constant K. These procedures are substantially more complex and time consuming in comparison with the standard fracture testing. For example, the identification of the threshold stress intensity factor, K_{th}, requires several thousand hours of testing time [23].

Due to a wide range of parameters affecting the HAC phenomenon (such as the rate and mode of loading, constraint conditions, hydrogen charging method, temperature, specimen size, diverse microstructural factors, etc.) the outcomes of conventional HAC tests demonstrate a large scatter [5]. In the development of experimental methods to characterise HAC over the past 50 years, a great effort has been directed to eliminate these diverse parameters influencing the reproducibility of the test results. In addition, the utilisation of the stress intensity factor and plane strain conditions as well as controlled hydrogen charging allowed an accurate measure of the mechanical stress and environmental conditions, two main factors affecting HAC. However, the microstructure of metal and alloys (which is the third main factor affecting HAC) typically consists of diverse constituents with specific features and mechanical properties that only occur in microscopic volumes. To investigate the HAC susceptibility of such small volumes it is therefore necessary to significantly reduce the size of the specimens down to the characteristic size of the individual microstructural constituent.

Katz et al. [24] were the first who proposed the application of nano-indentation to examine the micro-mechanisms of hydrogen related failures. This idea was extended by Barnoush and Vehoff [5] who presented a method for determining the onset of plasticity in very small volumes (perfect crystals) subjected to hydrogen charging. Several different materials were tested with this method. For example, it was found that hydrogen reduces the critical stress required for the onset of plasticity. However, this method is not capable of providing a quantitative evaluation of fracture properties similar to conventional fracture tests, such as stress intensity factor threshold or crack growth rates. It seems that the only viable approach for a direct quantitative evaluation of the fracture properties of very small material volumes is the miniaturisation of fracture specimens and testing down to micro-scale [25].

The miniaturisation of fracture tests became possible over the past decade with an increasing efficiency and accuracy of Focused Ion Beam (FIB) workstations, which has resulted in a reproducible fabrication of micro-scaled specimens. Several researchers have already employed FIB micromachining to fabricate micro-pillars for performing compression tests on uncharged and hydrogen charged specimens [26,27]. These experiments demonstrated the hydrogen-deformation interrelationships at microscopic scales. However, FIB micro-machining also allows fabricating standard fracture specimens with microscopic dimensions [28,29]. This idea has been realised in the current work to investigate the effects of hydrogen on the crack kinetics in a selected microstructural constituent with particular features. Sharply notched micro-cantilevers were fabricated with a FIB into a localised region fully consisting of acicular ferrite which is a common microstructural constituent of ferritic weld metal and alloys. This microstructural region has characteristic features that largely determine its mechanical properties as well as its susceptibility to HAC. The micro-cantilevers were characterised via Electron Backscatter Diffraction (EBSD) and subsequently tested under uncharged and hydrogen charged conditions with a nano-indenter. However, HAC fracture testing standards, developed for macro-examinations, could not be followed precisely in our experimental campaign, specifically in terms of the test duration to identify the onset of hydrogen cracking or K_{th}, fatigue pre-cracking and realisation of plane strain conditions. Constant loading was applied for twelve hours to simulate the time delayed nature of the IHAC in a micro-scale specimen. Unexpectedly, the fracture mechanics characteristics derived from the present tests were found to be very different to the corresponding values reported for conventional sized fracture specimens.

2. Material and methods

Weld metal is an example of a material that typically consists of diverse microstructural constituents with distinctive features and mechanical properties. The properties of individual constituents can only be revealed when testing small material volumes, and are therefore not directly assessable with conventional methods of mechanical testing [5,30]. It is well-known that the presence of hydrogen can significantly compromise the structural integrity of the weld metal (as well as of the heat affected zone). Hydrogen Assisted Cold Cracking (HACC), which is a particular manifestation of IHAC, is a well-known weld failure mechanism that may occur after the deposited weld has cooled down to temperatures below 200 °C. A critical structural defect can be developed within minutes to even days after welding [31]. Due to its time delayed nature the onset of Weld Metal Hydrogen Assisted Cold Cracking (WM HACC) may be undetected and, eventually, result in catastrophic failure.

2.1. Material-microstructural constituent

A weld metal specimen was sourced from API 5 L grade X70 samples welded with 4 mm diameter E6010 cellulosic electrodes. The welding procedure is presented in details in [32]. Under these welding conditions, the yield strength of the weld metal is expected to range between the yield strengths of the E6010 electrode (420 MPa) and the X70 parent metal (480 MPa).

A localised region of the weld metal specimen consisting of acicular ferrite was identified for further micro-fracture tests (Fig. 1a and b). This microstructural constituent represents a