Mechanics Research Communications 37 (2010) 230–234

Contents lists available at ScienceDirect

Mechanics Research Communications

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

On hydrogen-induced Vickers indentation cracking in high-strength steel

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ARTICLE INFO

Article history:
Received 28 November 2009
Received in revised form 5 January 2010
Available online 14 January 2010

Keywords:
Vickers indentation
Hydrogen embrittlement cracking

ABSTRACT

The present study investigates hydrogen embrittlement cracking (HEC) upon Vickers indentation in a high-strength steel. When an indentation test is applied to the high-strength steel, i.e., steel that has absorbed hydrogen, several cracks appear around the impression, whereas as-received steel with no hydrogen absorption does not produce any cracks. An experimental/computational framework is used to elucidate the mechanism of such indentation cracks caused by hydrogen embrittlement. We use the acoustic emission technique (AET) to clarify at which point during the test the crack initiates. In parallel with the experiment, finite element analysis (FEA) is carried out in order to compute the stress field around the impression. Based on the combined results, we discuss the mechanism of crack initiation and the critical stress required to nucleate the crack. The findings of the present paper may be useful for characterizing local contact fracture properties, which are often seriously deteriorated by hydrogen embrittlement.

1. Introduction

Vickers indentation of a brittle solid having a high hardness, such as materials in which the ratio of the yield stress to Young's modulus (σ_Y/E) is relatively high (e.g., glass and ceramics), usually induces a complicated crack morphology (Cook and Pharr, 1990; Lawn, 1993), whereas indentation onto engineering metallic materials, including high-strength steels, does not produce such cracks. These cracks are caused by tensile stress that develops due to finite remnants of the plastic deformation associated with the impression. In other words, an indentation impression induces large compressive plastic strain, leading to tensile residual stress that develops in an infinite elastic field surrounding the impression. The tensile stress induced by Vickers impression is reported to be strongly dependent on the value of σ_Y/E for the material (Cook and Pharr, 1990; Feng et al., 2007; Zeng et al., 1995; Zhang and Subhash, 2001). However, the tensile stress reaches approximately half the yield stress at a maximum (Cook and Pharr, 1990), because the elastic field in which the tensile stress develops is infinite. In brittle solids, the tensile strength is generally lower than the compressive strength (which is related to the compressive yield stress). When the indentation-induced tensile stress reaches the tensile strength, or the critical stress for crack nucleation, of the material, cracks nucleate around the impression.

In contrast, in engineering steels, including high-strength steels, the tensile strength is higher than the yield stress, and the yield stresses for tension and compression are the same. Therefore, the Vickers indentation-induced tensile stress cannot reach the tensile strength of such materials. In other words, Vickers crack nucleation requires the tensile strength (critical stress for cracking) to be lower than the compressive yield stress, so that the relationship between the compressive yield stress and the tensile strength is a key parameter.

Environmental species, including hydrogen, often affect the mechanical and strength properties of steels. High-strength steels tend to suffer from hydrogen embitterment, which leads to a significant decrease in the critical tensile stress for crack nucleation. Such critical stress causing Mode-I fracture is responsible for hydrogen embrittlement, resulting in degradation of the apparent "tensile strength", "fracture strain", and "fracture toughness". In contrast, degradation of the mechanical properties upon compression loading (e.g. macroscopic hardness) rarely occurs in a hydrogen environment. Therefore, hydrogen may reduce the critical stress for crack nucleation to below the compressive yield stress, which leads to hydrogen-induced cracking due to Vickers indentation. For high-strength steels containing hydrogen, the ability to determine whether Vickers indentation produces cracks would be useful for investigating the structural integrity of the material with respect to hydrogen embrittlement, which is an urgent problem in the development of hydrogen energy system. To our knowledge, there has been no report on this subject.

The purpose of the present study is to investigate hydrogen-induced cracking in high-strength steel due to Vickers indentation. Since indentation tests generate a morphology that is typical of contact fracture, which is similar to foreign object damage (Chen,

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doi:10.1016/j.mechrescom.2010.01.001

2005; Lawn, 1993), the present study clarifies the mechanism of hydrogen-induced contact fracture, which is a potential fracture mode when steel is used as a contact member in a hydrogen environment.

2. Materials and experimental methods

The material used in the present study is 18% Ni maraging steel (680*HV*1), which is a low-carbon martensitic steel. In order to achieve a high strength, we subjected the steel to solution heat treatment (820 °C for 3 h) followed by air-cooling to room temperature. Subsequently, the steel was aged at 500 °C for 4 h and air cooled to room temperature. The yield stress and tensile strength are 2.40 and 2.45 GPa, respectively (Boyer and Gall, 1990). This steel has superior tensile strength, but is very susceptible to hydrogen embrittlement (Reddy et al., 1992). The specimen is a disk-shaped plate with a diameter of 20 mm and a thickness of 1 mm. After mechanically and electrochemically polishing the specimen surface, cathodic charging was performed in a buffer solution of sodium acetate (0.20 mol/L CH_3COOH + 0.17 mol/L CH_3COONa, pH = 4.7) with a current density of 3 A/m^2. After cathodic charging, the total hydrogen content in the material was measured by thermal desorption spectroscopy (TDS-KU, ULVAC). Here, the hydrogen content, which is calculated by integrating the TDS profile (hydrogen release rate as a function of temperature or time), is denoted as H_c.

Indentation testing was carried out using a hydraulic servo-controlled fatigue testing machine (EHF-FG1KN-4LA, Shimadzu Corp.) equipped with a diamond Vickers indenter. The indenter impresses the specimen surface with an indentation force of up to 300 N at a rate of 1 N/s. Subsequently, the indentation force is reduced at a rate of 1 N/s until $F = 0$ N. To elucidate the mechanism of crack initiation, the acoustic emission (AE) method is used during the test to monitor crack nucleation *in situ*. Although, the existence of pop-in or pop-out events in the indentation curve is useful in determining the occurrence of indentation fracture, such signals are not always present in the indentation curve (Cook and Pharr, 1990; Yonezu et al., 2005). If indentation cracking occurs in the steel of the present study, detection of crack initiation by the AE technique, in conjunction with stress analysis (post analysis) using the finite element method (FEM), is useful for elucidating the cracking mechanism. In the preset study, two AE sensors (Type-PICO; PAC Inc.) are mounted on the specimen surface. The output signals are amplified by 40 dB using pre-amplifiers (NF9913; NF circuit block) and are digitized by a personal computer. Other details of the AE measurement have been reported in previous studies on the indentation fracture mechanics of brittle materials (Yonezu et al., 2006).

3. Results and discussion

3.1. Hydrogen-induced crack morphology

Vickers indentation was applied to the specimen that had absorbed hydrogen as a result of cathodic charging for 72 h (H_c = 49.1 ppm). Fig. 1 shows optical photographs of the impression and its surrounding. In the surface photograph of Fig. 1a, four cracks propagate from the corners of the impression. This is quite similar to surface cracks (radial or half-penny-shaped cracks, HPC) observed in brittle materials subjected to Vickers indentation (Cook and Pharr, 1990; Niihara, 1983). Fig. 1b shows a close-up view of the radial crack path observed by scanning electron microscopy (SEM: JSM-S300S, JEOL Ltd.). The crack grew in a zigzag manner, indicating that it propagates along grain boundaries. Such intergranular cracks are often experienced in maraging steel when the steel suffers from hydrogen embrittlement (Reddy et al., 1992). As such, crack formation by Vickers indentation is thought to be caused by hydrogen embrittlement. In addition, we observed the transverse section of the impression, as shown in Fig. 1c, which reveals that, near the impression, three cracks initiated in the direction parallel to the specimen surface (two cracks on the left side and one on the right side). This type of crack is a lateral crack produced by normal tensile stress acting on the crack surface. It is also noted that the crack perpendicular to the specimen surface cannot be observed beneath the impression, and thus the surface crack (Fig. 1a) is regarded as an radial crack, which is recognized to be shallow crack (Niihara, 1983). Therefore, the present crack system consists of radial cracks and lateral cracks. These cracks are produced by normal tensile stresses, the fields of which change during the process of indentation loading/unloading. Radial cracks tends to initiate under loading and extend during unloading, while lateral crack initiates and grows during unloading (Cook and Pharr, 1990; Zhang and Subhash, 2001).

However, steel with no hydrogen absorption (as-received specimen: H_c is less than 0.05 ppm) did not exhibit any cracking around the impression. Therefore, it is concluded that hydrogen in the steel caused a decrease in the critical stress to nucleate cracking, resulting in Vickers crack formation. In order to clarify the present mechanism of crack nucleation, the timing of crack initiation will be identified using the AE technique next.

3.2. Mechanism of crack nucleation

Fig. 2 shows the changes in cumulative AE count and AE amplitude (peak to peak) V_{pp}, as well as the indentation force, as a function of time during the test. When the test started, numerous AEs (approximately 270 events) with various amplitudes were detected up to approximately $F = 50$ N. A number of these AEs are expected to be produced by radial surface cracking, because the loading process induces radial cracks or HPC (Cook and Pharr, 1990). However, subsequent AEs were absent from $F = 50$ N, but nine AEs were generated at the end of the unloading process. Note that contact friction by indenter penetration (particularly the loading process) also produces AEs (Yonezu et al., 2005). If the present AE sources are due to contact friction, then such AEs must be recorded continuously in the entire range of the loading process. However, they were not observed at $F > 50$ N, and so some of the detected AEs may originate from crack nucleation. Thus, the results of AE measurement suggest that the cracks are expected to nucleate at the initial stage of indenter penetration and the final stage of indenter withdrawal.

In order to clarify the driving force of both radial and lateral crack initiations, we performed a numerical simulation based on the finite element method (FEM). A three-dimensional model of one-quarter on the specimen was generated as shown in Fig. 3a. The model contains more than 20,000 nodes and four-node elements, and a mesh convergence study was carried out. The calculation was performed using commercially available code (Marc 2005 r3 and Mentat II 64-bit). A rigid contact surface was used to simulate the rigid indenter of the Vickers diamond indenter. Coulomb's law of friction is assumed with a friction coefficient of 0.15 (Bowden and Tabor, 1950). The mechanical properties of "as-received" maraging steel are used in the FEM computation because the macroscopic hardness, which is related to the plastic properties in compression, does not change with hydrogen content and is constant at $HV = 674$. A similar trend has been previously reported (Reddy et al., 1992). Referring to a earlier study (Boyer and Gall, 1990), we assumed that the stress–strain curve obeys the work-hardening law, indicating that Young's modulus, the yield stress, and the work hardening exponent are 210 GPa, 2.4 GPa, and 0.025 GPa, respectively. These elastoplastic properties were