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Crack habits in metal/hydrogen interactive systems

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ABSTRACT

Purpose: In terms of structural integrity aspects, interactive insights into metal/hydrogen systems become self-explanatory. In fact, ample of local critical events are involved namely, from sub-critical slow crack growth up to a total delayed failure.

Design/methodology/approach: Mainly, crack-tip vicinity characterization became essential from both, chemical and mechanical factors, as related to the complicated aspects of crack stability. Note that the interactive situation has been currently investigated, imposing as such new local conditions. In fact, the current study adopted a highly comprehensive methodology in view of fracture physics. Thus, the material selection included iron-based (Fe-3%Si) and zinc single crystals. For the sake of background only, polycrystalline austenitic stainless steels were also investigated regarding hydrogen interaction affecting mechanical properties. Hydrogen charging has been performed either by electrolytic cathodic charging or, by high temperature/pressure gaseous methods. Fracture mechanics methodology was mainly implemented by utilizing external or internal hydrogen interaction procedures. Experimentally, research activities have been conducted by novel techniques, such as ultra-high visualization techniques and by using basic plasticity information like crack-tip dislocation emission and structures.

Findings: Enhanced crack extension was established and the slow subcritical crack extension was traced in physically well-defined crack systems. Fracture mode transitions occurred due to the deformation/environment interaction. For example, in iron-based single crystals the unique cleavage mode emphasized the embrittlement impact. The broadness of the current study enabled a more local fundamental approach aimed to understand the crack-path habits. Here, the role of the crack-tip shielding beside the crack arrest potentials have been considered.

Research limitations/implications: The dominating micro-mechanisms of hydrogen-related fracture have been thoroughly reviewed in the literature. Despite the remarkable research efforts involving coupled theory and physical findings, critical experiments still remain the key issue in order to establish more of any basic general concepts. Clearly, the hydrogen/deformation interactions have many facets involving broad service implications.

Practical implications: The issue of hydrogen embrittlement or the possible decrease of fracture resistance causing severe mechanical degradation requires special attention.

Originality/value: The current investigation includes nano-mechanical probes on top of surface probe microscopy. This technique offered additional critical experiments aimed to resolve the scaling relationship. Moreover, the study assisted to reveal local/global insights related to the interaction conditions involved. One of the values to be mentioned resulted even from the subcritical crack path input. The crack path habits with hydrogen interaction served as critical information. In this context, crack path findings supported the assessment of the possible viable micro-mechanical interactive embrittlement models. Basically, the crack path and the crack-tip front varied with the different crack systems. The important role of plasticity in the cleavage mode formation has been substantiated. Alluded to the above implies that critical experiments might eventually provide the building blocks for modeling efforts that can truly simulate and anticipate embrittlement events. It is demonstrated that huge gaps in knowledge exist preventing appropriate bridging of scales. This has often led to controversy when addressing multiple affected microstructures. This multiplicity occurs due to numerous second phases and interfaces that can interact in various modes with aggressive environment affecting both localized flow and fracture. Only by breaking the microstructure down to its individual building blocks can the scale bridging be appropriately dealt with.

Keywords: Single crystals; Hydrogen; Interaction; Crack path; Plasticity; Cleavage

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MATERIALS

1. Introduction

Continuous studies into the understanding of metal/environment interaction have been facilitated by critical experimental findings beside advances in computational/simulation capabilities. Significant developments along those avenues resulted from the self-explained concern regarding issues of structural integrity management. In this context, aggressive agents like hydrogen that might require dedicated attention and comprehensive input are included. Simply put, what are the consequences of elastic- plastic solids affected by environment interaction? The current combined investigation program has been supported by experimental/theoretical interface allowing, as such, to reveal various typical events within the framework of a local material approach. Those events include, for example, enhanced cracking initiation, subcritical slow crack growth, running cracks and alternating local ductile/brittle transition and phase stability changes. Generally, various powerful views on the dominating micro mechanisms of hydrogen enhancing fracture have been vigorously discussed and thoroughly reviewed in the literature [1-7]. Clearly, the compact measures of experimental/theoretical efforts addressing those interactive problems are well-recognized [8-14]. However, the remarkable research activities are normally centered on critical experiments that by definition remain always the key link for further progress. It is well known that hydrogen/deformation interactions have many facets with broad implications. The current study focuses also on small volume research as confined to nano-scale probing and to fine features observations. In fact, the paper intends to clarify the potential of such proposed avenues for analyzing localized flow and fracture occurrence. Moreover, the study is outlined and constructed in a way combining micro/macro scale aspects, based mainly on the extended background.

2. Materials, experimental and work methodology

Material selection included mainly iron-based single crystals. For the purposes of comparative study and background extension, AISI 300 class of polycrystalline metastable austenitic stainless steels was also tested. Moreover, observations also have been conducted on zinc single crystals, mainly in order to expose the critical role of plasticity in shaping the fracture mode process. Basically, fracture mechanics methodology was followed in pre-fatigued mini-compact single-edged notched specimens. Clearly all data have been achieved after the specimens' calibration in cases of no- and post-hydrogen interaction. Notice that hydrogen charging was performed by either electrolytic cathodic charging or by temperature/pressure gaseous charging methods. Since the single crystals specimens were orientation with respect to well defined, various crack systems could be selected and specified in order to establish the mechanical behavior, including the crack path characterization. In addition, crack stability and crack rates have been revealed, beside fracture mode transition on top of fine scale information of plasticity in terms of slip trace analysis and crack-tip dislocation emission and structures mapping. The broad information served also the desire to establish the mechanical environment at the crack-tip. With respect to the cleavage formation due to environment/deformation local interaction - SEM images and Selected Area Channeling Patterns have been utilized, including simulations. Finally, Acoustic Emission techniques assisted to study the nature of the sub-critical slow crack extension, bursts, etc. (Table. 1). Regarding the polycrystalline stainless steels, the issue of phase stability has been investigated by using Mossbauer spectroscopy and X-Ray diffraction. For those materials, nano-probe measures of deformation and fracture were also achieved by nano-indentation and nano-scratch tests. These efforts combined, with ultra-high resolution observations provided critical information on hydrogen enhancing slip localization and habits. In the framework of fine features resolutions, more have been revealed on the role of hydrogen affecting dislocation nucleation, hardening or softening, as well as on steps or typical spacing morphologies of the slip bands.

Table 1.
Critical experiments assisted by nano-probe measurements

Scratch-induced fracture	Grain boundary Second phase interface Cleavage planes
Indentation-induced delamination	True surface energy Dislocation shielding Grain-boundary segregants
Indentation induced plasticity	Dislocation nucleation Indentation caused effect Slip band characterization Plastic zone size

$$da/dt_{(110)} < da/dt_{(100)}; \quad \text{local } \varepsilon_{(110)} > \text{local } \varepsilon_{(100)} \tag{1}$$

If enhanced localized plasticity had been responsible for crack growth – the two inequalities should have been similar rather than opposed.

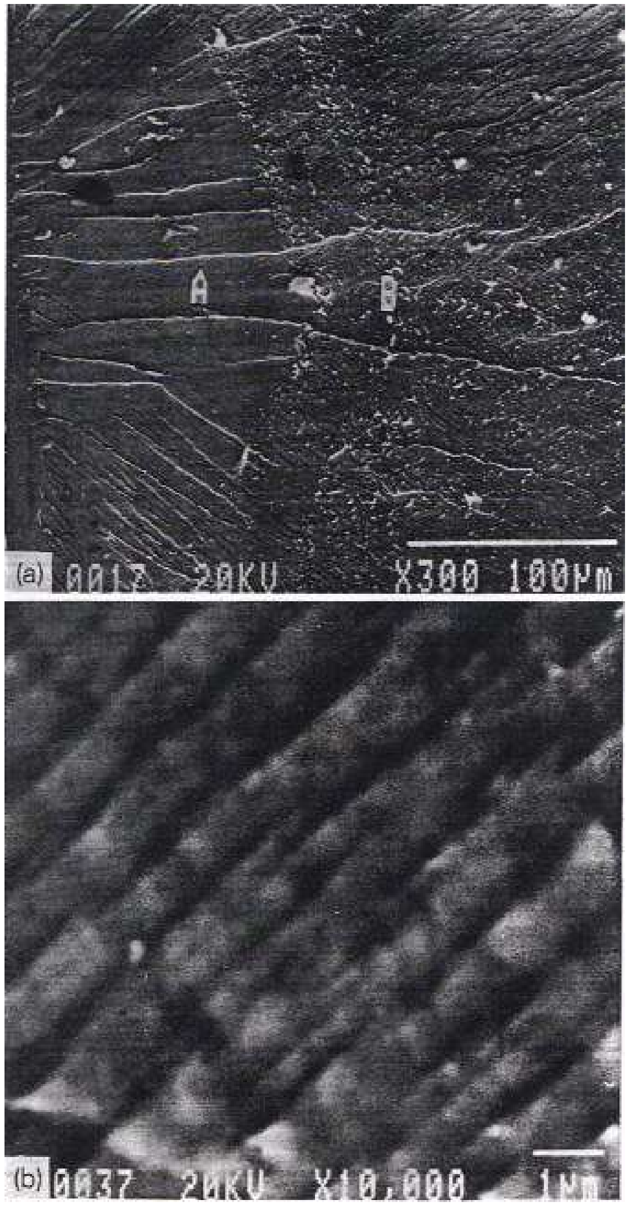

Fig. 1. SEM image of mini single-edge notched fracture mechanics specimen. Post-hydrogen surface, region (A)-slow crack growth, region (B)-running crack. (001) <010> crack system

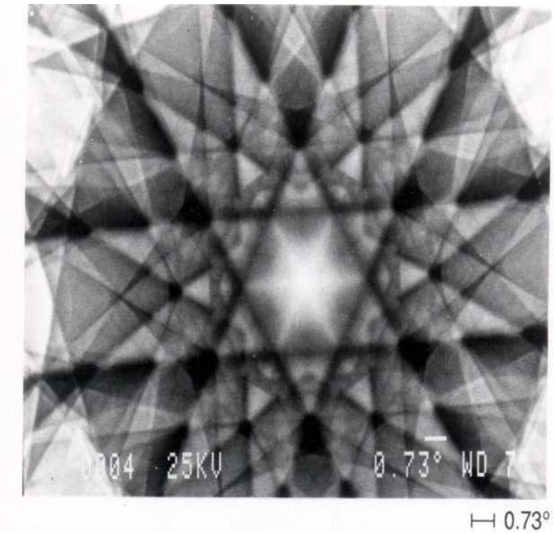

Fig. 2. SACP for zinc single crystals cleavage

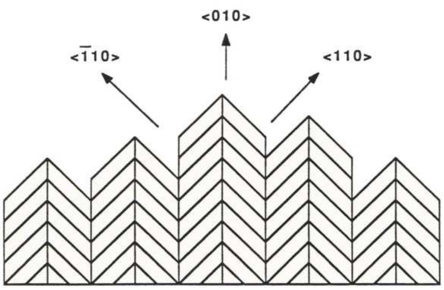

Fig. 3. Schematics of specific crack system demonstrating herringbone growth

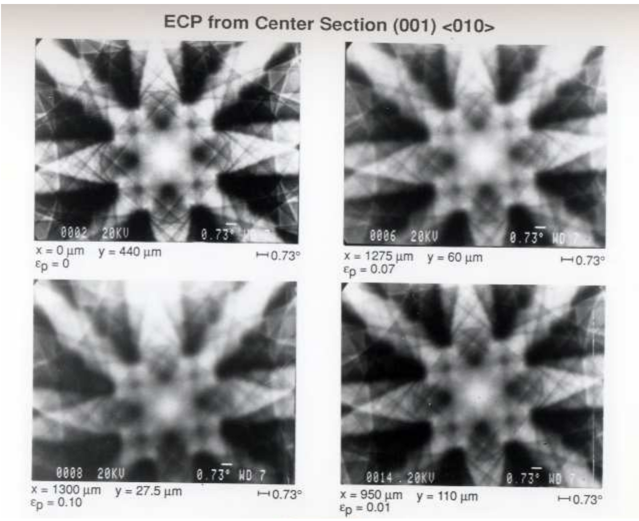

Fig. 4. SACP from center section of (001) <010> crack system

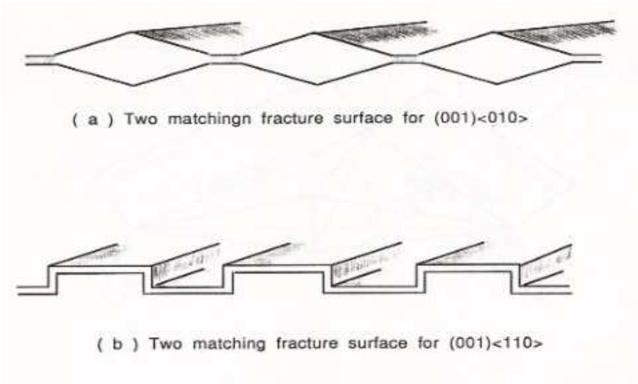

Fig. 5. Two crack systems

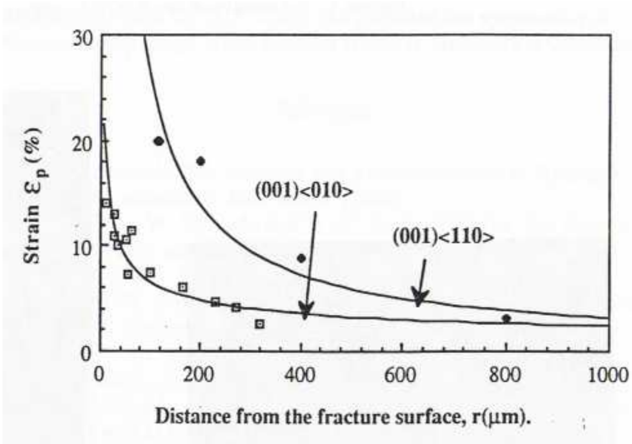

Fig. 6. Measured plasticity for two crack systems on the (001) cleavage plane

3. Description of achieved results

Generally, the sound physical view regarding Deformation/Environment interaction issues still remains a very challenging research topic. Specifically, in the case of the hydrogen agent the enhanced mechanical events turn out to be highly involved on various fronts. For example, hydrogen interaction in elastic-plastic solids might enhance crack initiation leading to crack extension in the form of sub-critical slow crack propagation. Thus, further progress on the basic level requires advance exploration of the micro-mechanical processes within the framework of fracture physics. The current study intends to track and characterize the crack habits in more fundamental circumstances. Due to the environmental interaction a semi-brittle fracture resulted in the unique fracture mode of cleavage. For a better understanding of cleavage, zinc single crystals have been supplemented, allowing a comparative study to be conducted .In the iron-based single crystals the fracture mode transition became apparent, namely from micro-void coalescence to the well-defined cleavage fracture mode. This outcome was confirmed even in relatively thin single crystals specimens. It was also found that with hydrogen threshold value of 4-18 MPa 1/2 was established, while under higher stress intensity factor, plateau crack extension rates of about 70nm/s were measured. Note that the crack extension was discontinuous, evolving a sub-critical slow crack growth behavior. In this context, the SEM images indicated a substantial occurrence of anisotropic plasticity that has been accompanied by differences in the crack growth habits (Fig. 1) .It was also found that the exact hydrogen charging method did not affect the basic micro mechanisms of the cracking process. The variations here were more closely associated with the crack growth kinetics. The Acoustic Emission findings correlated with groups of periodic bursts of stress wave emission. These groups have scaled with observed steps of 1 micron during an average time of 13 s' between a series of crack extension events. In fact this was a typical behavior of an initiation-propagation-arrest followed by a re- initiation process on a cleavage plane. The discontinuous crack extension behavior could be described as reproducible ultra-fine scale features of crack arrest.

Thus, it was argued that the slow crack extension process has evolved by unzipping mechanism along atomic rows with a finite hydrogen transfer and uptake occurring in-between sequential arrest points. This model results in a semi equilibrium micro-mechanism of fracture considering only a minor excess energy involved, and as such activating the periodically crack stability upset. Experimentally, this yielded the measured crack extension rats of 1 to 10 m/s. Consequently, the acoustic emission observations remained consistent with these measured velocities through the trapped diffusivity of hydrogen and the crack jump distances. The comparison between various crack systems in single crystals indicates the important role of plasticity in crack growth habits. These findings have been consistent with the information that was revealed by the slip trace analysis. Here to mention also that the dislocation images substantiated the well-known crystal plasticity behavior .The Selected Area Channeling Pattern (Fig. 2) provided fundamental information regarding the anisotropic plastic flow, thus enhancing the cleavage mode formation. Fig. 1 illustrates the findings from SEM channeling results as obtained from images of mini-compact single crystal specimens exposed to Deformation/Hydrogen interaction. Basically, the crack path and the crack front where highly dependent on the exact crack system, as well as the local plastic strain causing the crack rate dependency in the crystal orientation (Fig. 3). The typical herringbone fracture surface mode occurred only in specific crack systems. Experimentally based, the role of plasticity on cleavage as formed by the hydrogen interaction has been thoroughly discussed. It was found that higher plasticity resulted in the slow sub-critical crack extension regime as compared to the running crack section. In a consistent fashion, plasticity was more severe in the crack system in which crack resistance orientation was more prevailing. Thus, on the microscopic (001) cleavage plane, the crack extension rate in the (110) was less than in the (100) (Fig. 4). The finding that the local strain in (001) is lesser than in the (110) direction is striking and highly important (Figs. 5-6). Note that this information is directly connected to the examination of the possible proposed viable micro-mechanisms in the Deformation/Hydrogen embrittlement process. As already mentioned, the aforementioned concept of plasticity enhancing cleavage additional study on zinc single crystals has been supplemented. Here, even at early stages of plasticity cleavage formation was revealed. Nevertheless, it became apparent that the measured plastic strain enhancing cleavage in zinc is relatively minor in comparison to the iron-based crystal after environmental interaction.

4. Conclusions

The prior sequential model developments concerning the brittle fracture assisted the experimentally based analysis of the Deformation/Hydrogen interaction case. In particular, crack initiation and extension, slow crack growth, crack path; fracture mode transition caused by the environment enjoyed the current local approach in single crystals. These systems provided more fundamental tracking in terms of crystal plasticity argumentations.

In the iron-based single crystals it was concluded that post-hydrogen interaction resulted in crack initiation and extension and variations in the slow subcritical crack growth. In addition, the changes were highly dependent on the specific crack systems. These included the crack habits and the path on top of the crack front.

By following the fracture mechanics methodology much better controlled experimental conditions have been achieved quantitatively.

Refined information regarding the directional dependency of the plastic strain and the corresponding crack rates turned out to be highly important. Note that this kind of data could have been obtained by utilizing various crack systems of single crystals. In fact, this information was directly connected to the proposed viable micro-mechanism of the hydrogen embrittlement process.

Acoustic emission tracking indicated the discontinuous nature of the subcritical slow crack growth. This allowed better understanding of the current semi-equilibrium fracture. It was concluded that a possible micro-mechanism involved in forming such event is associated with periodical crack stability upset.

The role of plasticity in fracture mode transition was confirmed experimentally. Namely, in post-hydrogen interaction plasticity enhanced the cleavage fracture mode. It became apparent that in zinc single crystals even at the early stage of plasticity the fracture mode of cleavage was formed. Clearly, in zinc, less severe plastic strain was measured, compared to the iron-based single crystals.

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Additional information

The current study, on mainly iron-based single crystals under Deformation/Hydrogen interaction, has centered particularly on enhanced crack formation, crack stability, subcritical behavior, crack path and front, fracture mode transition, the role of plasticity in the cleavage fracture mode and the directional dependency of the crack path.
Nevertheless, the author recognizes the complimentary intensive input regarding the environmental interaction in polycrystalline systems (8, 9). Those Experimental Simulation activities are mentioned due to the new dimension of the localized approach. Basically, advanced computational numerical abilities have been used, as well as nano-mechanical probes utilization.

Moreover, visualization by novel techniques including Atomic Force Microscopy has followed nano-indentation and micro-testing techniques. In addition, a crack-tip dislocation emission study has been conducted, assisted also by TEM observations.

Selected issues related to this paper are planned to be presented at the 22nd Winter International Scientific Conference on Achievements in Mechanical and Materials Engineering Winter-AMME'2015 in the framework of the Bidisciplinary Occasional Scientific Session BOSS'2015 celebrating the 10th anniversary of the foundation of the Association of Computational Materials Science and Surface Engineering and the World Academy of Materials and Manufacturing Engineering and of the foundation of the Worldwide Journal of Achievements in Materials and Manufacturing Engineering.

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