Local/global eyes into the crack path behavior in metal/hydrogen interactive system

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ABSTRACT. *Fundamentally, the prediction abilities of the crack-path are anchored in the comprehensive understanding of the fracture process. Generally, fracture comprehension remains a broad and complex field that requires the engagement of a wide range of functional scales. By considering the specific application, the local/global approach becomes almost inevitable. In small volume segments the local approach is self-explained although quite demanding. However, even in a global application the problem origins of fracture or fatigue are highly localized by nature. The aforementioned notion recognizes that frequently global material findings are initiated or dominated by local events. The current study is centered on two elements that might ease the crack-path analysis. First, in iron-based semi-brittle material, single crystal systems have been selected that reduces as such the micro-structural complexities. Secondly, with deformation/environment interaction a very sharp crack-tip situation prevailed, allowing a sub-critical crack extension to be enhanced on a macro-cleavage plane. In the current experimental framework, topics like the crack-tip mechanical environment, the crack-tip shielding potential, crack advancement and arrest, directional rate dependency in a slow crack growth condition have been thoroughly discussed. The study insights regarding the unique fracture mode of cleavage have been assisted by fracture mechanics methodology, crystal plasticity observations, simulation activities and by ultra-fine features visualization. The tracking of the crack-path enabled an additional input to be revealed with respect to the consistency of proposed viable deformation/hydrogen micro-mechanism models.*

INTRODUCTION

With respect to deformation/hydrogen interaction it has been established that micro-plasticity remains a critical trigger-link in semi-brittle elastic solids. Theoretical/experimental interface activities indicated that in pre-fatigued, crack-tip deformation occurred with enhanced crack extension confined to the macro-cleavage plane. Thus, in B.C.C. crystal structure even the unique fracture mode of cleavage is ill-defined [1-3]. Basically, the crack-path analysis in brittle fracture might facilitate further explorations with an attempt to minimize some of the critical variables. In this context of bottom/top approach regarding the brittle fracture the local approach is also included. Particularly, into the crack-path issue, activities down to the atomic scale might ultimately be followed and even become highly attractive in single crystal systems [1].

Nevertheless, by utilizing such procedure other difficulties emerge. For example, the well expressed dilemma of how to formulate the appropriate near-atom model methodology. The latter recognizes the critical requirement to adopt only well confirmed and reliable inter-atomic potentials. Note that the current study also confirmed that even in "brittle cleavage" the solely perception of atoms debonding along crystallographic phases is at least shortcoming. This finding accentuated as such the anisotropic habits of the subcritical crack-path. In contrast to the local approach, the global continuum model still requires always to fulfill explicit fracture criteria. This has been based for example on the energy density methodology [4, 5]. In the light of the crack stability equation according to fracture mechanics formulation, the driving force and the resistance components might dominate the crack-path behavior. For example, in polycrystalline systems variables like environment, thermal effects and local embrittlement segregation might result in inter-granular fracture. In addition, remote dynamic load might cause crack branching [6] or mixed mode conditions adding more complexities [7]. The present investigation followed scales above the atomic resolution but is basically confined to localized argumentations. This has been manifested by the assistance of the fracture mechanics contribution that enabled to establish the crack-tip mechanical environment in various single crystals crack systems.

MATERIAL AND EXPERIMENTAL PROCEDURE

Marginal semi-brittle Fe-3wt%Si single crystals have been selected. The material as received consisted from crystal bars geometry of 23 mm in diameter with <001> and <110> zone axes. By Laue back reflection X-ray technique the exact orientations have been determined providing as such the basis of the specimens preparation. Beside standard mechanical properties characterization, fracture mechanics, mini-compact pre-fatigued crack specimens have been utilized. In order to reduce prior plastic deformation effects, Electric Discharged Machining (EDM) was used to all specimens and crack systems. For reliable crack-tip conditions, final crack-tip sharpening was supplemented by compression-compression fatigue at low temperature of 143 K^0 with R ration of 0.1 and by cycle frequency of 3Hz. This stage was conducted on closed loop servo hydraulic testing machine with controlled temperature and environmental chamber. Hydrogen as an aggressive agent caused dramatic changes regarding the sub-critical crack stability. In fact, hydrogen enhanced crack extension with unique behavior of alternatic ductile/brittle transition. The striking implication was expressed by a consisted crack propagation that was confined on a macro cleavage plane. Thus, beside experiments that were conducted with no hydrogen, tests have been performed in post hydrogen charged specimens providing important information on deformation/hydrogen interaction. Here to mention that the exact hydrogen charging method either by temperature/pressure or cathodic charging have indicated similar findings and that changes due to hydrogen charging methods including internal/external beside fugacity degree affect more the crack extension kinetics or the level of the initial damage. Under hydrogen interaction the plastic strain field was measured by applying the Selected Area Electron Channeling Pattern (SACP), technique, assisted by SEM. Images were taken from both, the free external surfaces and from the crack or surfaces parallel to the fracture surface. In order to achieve comprehensive SACP information regarding the near fracture surface features. Controlled series of layers have been removed by electro-polishing in given layers thickness followed by SEM images. The electron channeling, fine width features were measured by micro-densitometer that enabled to determine the local plastic strain by following previous calibration procedure. Accordingly, the strain field was assessed by the mapping of isostrain contours supplemented by experimentally based additional information. These included slip traces analysis with the general notion of the crystal plasticity behavior. The fracture mechanics specimens were loaded by open mode up to a given K_I stress/strain field. Hydrogen interaction under loaded conditions caused enhanced crack-extension under such sustained load situation. The sub-critical crack growth was tracked also by acoustic emission supplemented by fractography study with special attention to the arrest potential and the crack orientation. Due to the inhomogeneous plasticity behavior at crack-tip vicinity, the various crack systems emphasized the anisotropic strain values as well as changes in the crack extension rates that were also directionally dependent.

EXPERIMENTAL AND SIMULATION FINDINGS

For Fe-3wt%Si single crystals the following properties have been obtained, namely the Young modulus $E_{<100>} = 1.32 \times 10^5$ MPa, the yield stress at ambient temperature σ_{ys} = 296 MPa with strain hardening exponent of 0.38. (8, 9) Generally, the B.C.C. crystal structure is strain-rate sensitive material with 48 non-dependent slip systems. Currently, for the sake of briefness, only two crack systems are emphasized, namely {001}[010] and {110}[110] in terms of the crack plane and the crack orientation respectively. As mentioned already, hydrogen interaction induces cracking under sustained load. In fact, this was achieved in a given range of stress intensity factors. Under interaction, cleavage fracture mode formed with relatively slow discontinuous crack extension process with average crack propagation rate of 1.6×10^{-8} m/s. Figures 1 illustrate ductile fracture with no hydrogen interaction and Fig.2 demonstrates the transition to the semi brittle fracture with hydrogen.

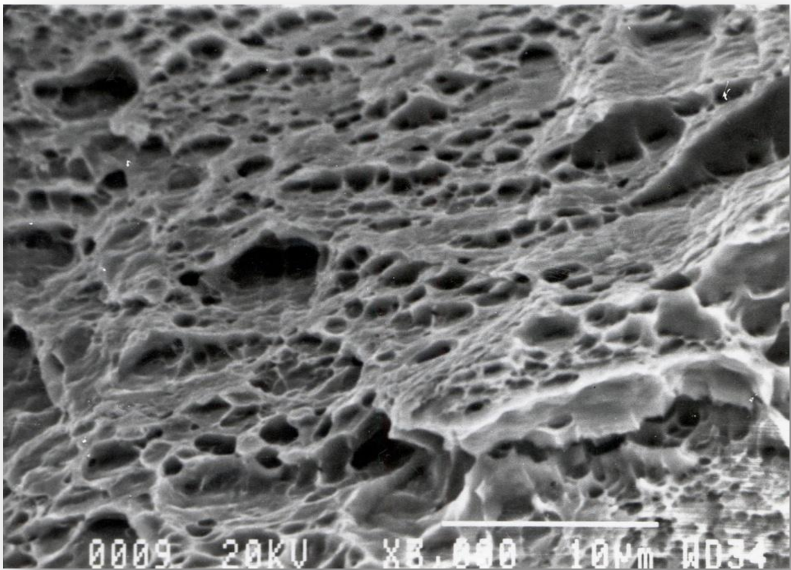

Figure 1. SEM from single crystal (001)<010> system
microvoid coalescence

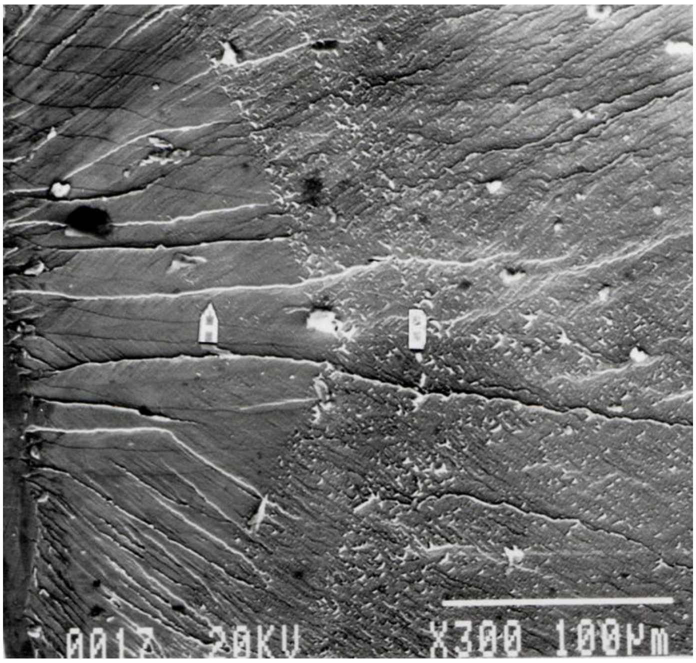

Figure 2. Fractography, sustained load with hydrogen, (001)<010> crack system, AE A to B

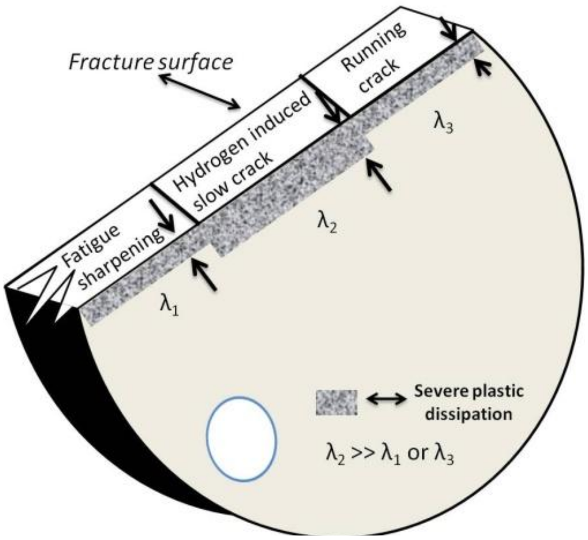

Figure 3. Mini compact, single crystal specimen – post deformation/hydrogen interaction

Mainly the SACP images and measurements manifested that hydrogen interaction enhances beside plasticity cleavage fracture in both directions, <010> and <110>. With respect to the crack along <010> typical crack extension patterns have been revealed. It was found that the crack advanced along two orthogonal <110> directions. This local combined propagation resulted in a typical herringbone pattern with alternative tilted surfaces similar to the crack-path that was observed in other stress corrosion cracking cases [10, 11]. Thus, the crack front was shaped as zigzag front, parallel to two <110> alternate directions. In addition, ridges formation divided the slow crack growth into individual ribbons. Another striking result is now in order. By means of novel visualization techniques consistently ultra-fine features have been identified in the form of striation lines parallel to the local <110> crack front direction. Striations with about 1µm in spacing were attributed to arrest marks in an unzipping crack extension mechanism. Important support of this interpretation was also confirmed from acoustic emission information that provided direct evidences to the discontinuous nature of the investigated subcritical slow crack growth. In contrast, the crack along <110> directions differed substantially and was characterized by various elements. First, flat fracture surface was observed with no herringbone pattern and thus no surface tilting dominated the crack extension. However, the fracture surface study demonstrated the inhomogeneous features of the <110> crack growth direction. In the specimen mid-section the crack growth was confined to the <110> while all the ledges or river line remained parallel to the mentioned direction. Actually, the direction change revealed once again that the herringbone mode is characterized by tilted surfaces across a middle spine. Also this information supports the significant role of the crystal plasticity in shaping of the crack-path behavior. Consequently, crystallographic origins affect strongly the crack orientation. Clearly, this is not the only factor and various others become apparent like plane stress in contrast to plane strain conditions. The latter can cause also the crack tunneling phenomenon that has been addressed on a local scale. [12] Finally the SACP strain measurements turned to be highly deductive indicating the differences between the various crack systems mainly by changes in terms of the imposed plasticity under environmental interaction. As indicated by the SACP even in cleavage, extensive plastic deformation is confined to a very thin layer close to the fracture surface. In the open mode of loading conditions the strain gradient becomes experimentally apparent implemented also by the crack extension rate. Accordingly, further insights into the macro-cleavage mode remain highly important and desired regarding the crack-path aspects.

DISCUSSION

The enhanced crack extension by deformation/hydrogen interaction calls for developments into further understanding of the involved phenomena that are associated with hydrogen embrittlement (HE). In this context, discontinuous brittle crack becomes very prominent as the proposed approach to model the sub-critical crack growth in iron-based single crystals. Regarding HE a couple of powerful views have been addressed mainly on the dominating micro-mechanisms of hydrogen related fracture. The specific input of the present investigation is centered on the addition of environmental interaction insights on basic science standpoint as related to the crack-path behavior. Note that the current case is in quasi-brittle slow crack extension that facilitated fundamentally the use of a local approach. The material selection of single crystals allowed a combined experimental program to be pursued. Here, the study enjoyed the assistance of fracture mechanics methodology, SACP technique and acoustic emission tracking. The SACP provided spatial resolution of 5 µm and a depth resolution of less than 100 nm [13]. Although not in the scope of the current study, general discussion on HE was addressed by Lii et al [14], Chen et al [15] and Katz et al [16] with emphasis on the mechanical response transition due to hydrogen interaction. The HE studies emphasized the important formulation of the crack-tip mechanical environment beside the chemical concentration. Only briefly, with the proposed viable micro-mechanisms of HE, like (HEDE), Hydrogen Enhanced Decohesion or (HELP) Hydrogen Enhanced Localized Plasticity the benefit of the crack-tip analysis based on the exploration of fracture physics should be recognized. More localized background was achieved by the crack-tip dislocation emission model. This approach explained insights regarding the crack-tip shielding effects that enabled to connect the local stress field to the global stress intensity factor. In fact, the crack-tip mechanical environment was developed by numerical simulation with consistent support of slip trace analysis confined to visualization and dislocation structure confirmation. Probably the striking results of the crack-path tracking in the current Fe-Si single crystals originated from both, SACP study and the directional dependence of the crack extension rate. Still in recalling the suggested viable micromechanism of HE the following remarks are in order. For example, the SACP technique indicated that plasticity (i.e. dislocation structure and their activities) was more severe in the local crack resistant orientation. Thus, considering the macro cleavage plane {001} it was found that,

$$da/dt_{<110>} < da/dt_{<100>}$$

and

$$\varepsilon^{local}_{<110>} > \varepsilon^{local}_{<100>}$$

Where ε is the local microplasticity

Thus, If the (HELP) micro-mechanism remained responsible for the crack growth the two inequalities should have been similar rather than opposed. Accordingly, the crack-path study became relevant also to the basic local origins of the crack propagation stage beside the contribution to significant insights up to a combined embrittlement micro mechanisms.

SUMMARY AND CONCLUSIONS

In fatigue pre-cracked Fe-3wt%/Si single crystals, it has been established that deformation/hydrogen interaction induced cleavage that resulted in a discontinuous subcritical slow crack growth. This crack extension obeyed velocities that partitioned into crack nucleation, rapid growth and arrest that were followed again by renucleation stage processes. Sequential events revealed that sufficient driving force via hydrogen interaction is required at the nucleation site. Moreover, plastic dissipation become essential in maintaining an arresting potential. These combined factors produced stepwise crack growth in a quasi-fashion behavior confined to the cleavage plane. At different crack systems the crack-paths were tracked indicating the anisotropic behavior of the crack orientation. Based on different crack systems indicated that the crack front stabilization was highly dependent on the specific crystallography. The important role of crystal plasticity aspects actually motivated the current measurements as supplementary information. Therefore, the following in concluded:

1. In hydrogen enhanced cracking of the macro-cleavage plane in iron-based single

crystals provided more insights regarding the crack-path behavior.

2. By adopting a local approach the dominant role of crystal plasticity on the crack-tip front has been revealed.
3. The selected area channeling pattern technique information contributed to better understanding of the cleavage mode fracture.
4. Beside the differentiation between the various crack systems patterns, the near cleavage plastic strain in <110> direction was higher by a factor of three as compared to the <010> direction.
5. The current interaction resulted in a discontinuous slow crack growth that was analyzed and explained. Thus, the stages of the crack extension were explored with emphasis on the crack arrest potential.
6. The study of the crack-path habits provided its share to the embrittlement proposed micro-mechanisms caused by hydrogen.

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