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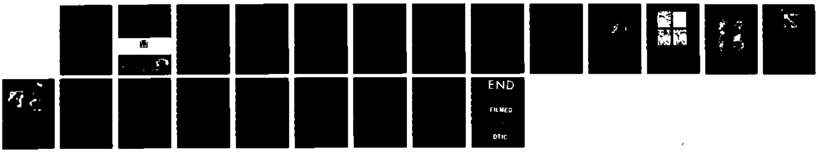

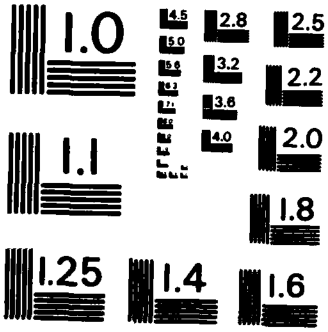

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The Role of Metallurgical Variables in Controlling Hydrogen Embrittlement

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ABSTRACT

The evidence for trapping by mobile and stationary defects in ferrous alloys is reviewed and new results presented to demonstrate that the trap theory of hydrogen embrittlement can rationalize the many different ways by which hydrogen modifies subsequent mechanical properties.

Introduction

The preferential interaction of hydrogen with such features as free surfaces, grain boundaries, local and global stresses and strain fields, and chemical heterogeneties such as precipitates is describable by the general term "trapping". This concept has important ramifications both in terms of the thermodynamics and kinetics of the entry, solubility and diffusion (permeability) of hydrogen and the variable concentrations concomitant with the presence of a trap population of differing strengths. It has now been established in many investigations and for a broad number of model and alloy systems that the presence of such features can impact significantly on the entire range of mechanical properties associated with deformation and fracture (1-10). Furthermore, paradigms are now being developed (2,9,10) in response to earlier suggestions (7,8) that hydrogen embrittlement, as manifested by a degradation of tensile ductility, fracture toughness or fatigue crack initiation or growth, is controllable either by decreasing the population of trapped hydrogen at potential crack sites (C_H), or by increasing the critical concentration necessary to initiate failure at a given trap, C_K.

In this paper the concept, validity and limitations of the "trap theory of hydrogen embrittlement" will be discussed using ferrous systems as the primary exemplar, although a number of non ferrous cases would have served equally as well. The bulk of the results will come from work either directly supported or stimulated by the long term, quality support of the Office of Naval Research at Carnegie-Mellon University and a number of other universities and research laboratories.

Fundamental of Hydrogen Trapping

Pressouyre has extensively developed the concepts of trapping (11,12) and the allied concepts of repellers and obstacles (2,9,13); others have also provided reviews of hydrogen trapping primarily in iron and steels (14,15).

The general viewpoint here, as elsewhere (8), is that trapping can be schematically described as in Figure 1. Five different types of trapping centers can be present either as independent entities or capable of interacting amongst each other:

1. The surface is a trap of multiple origins, including surfaces oxides and work functions. Except for its role as a kinetic barrier to hydrogen entry, it is believed less important than sub-surface traps in influencing a change in embrittlement susceptibility. However, it may be more important in its affect on strain localization or other dislocation related characteristics, where surface sources could dominate.

2. The lattice is, of course, a generalized trap in that there is a finite (but in iron a small) activation energy for interstitial diffusion. This intrinsic effect could become important at high, non-equilibrium hydrogen fugacities due to a concentration dependence of the lattice diffusivity.

3. There are mobile traps which for a large number of experimental conditions are reversible in nature in that the dislocation type of trap can gain or lose hydrogen within a time scale less than or of the order of the length of a mechanical test. Differences are possible when the dislocations are present as sessile arrays.

4. There are static, reversible (r) traps which can rapidly lose or gain hydrogen from dislocations, assuming the absence of significant kinetic barriers to such an interchange. Type 1 are weaker than dislocations and trap less hydrogen than dislocations and type II are stronger and trap more. Examples of the former could be substitutional impurities, while for the latter, examples include high angle grain boundaries or dislocations of other types.

5. Irreversible (r) traps have a much higher trapping energy than either the thermal energy, kT, or any other heterogeniety and can irreversibly keep their hydrogen for the length of a test, thereby acting as a strong hydrogen sink. Note that all of these trap centers have an influence diameter which we consider as the maximum range over which a hydrogen atom is said to be trapped and over which different traps can interact with one another.

More recently Pressouyre (13) has suggested that features with different properties than those illustrated in Figure 1 can also interact with hydrogen. These are illustrated in Figure 2.

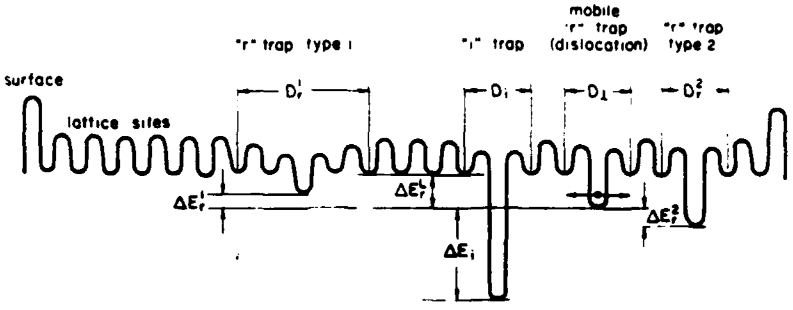


Figure 1. Schematic of general trap types illustrating comparative trapping energies and influence diameters.

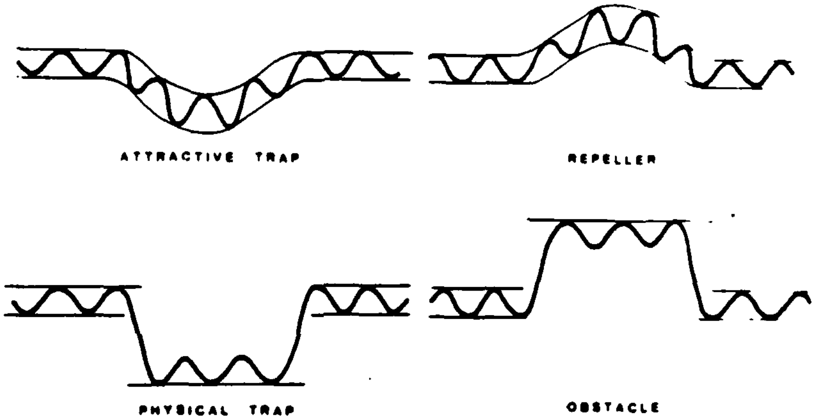


Figure 2. Schematic of the broaden class of heterogeneities capable of interacting with hydrogen: traps, repellers and obstacles.

The depicted attractive and physical traps are like those described by Figure 1; however, counterparts to these also exist. For example, repellers are the opposite of an attractive trap in that a repulsive force exists relative to hydrogen. Examples include atoms with a positive interaction coefficient with hydrogen (11), or local compressive stress fields. A hydrogen obstacle is the opposite of a physical trap and is most likely a discontinuity through which hydrogen has no significant mobility, such as a coherent particle with no hydrogen solubility.

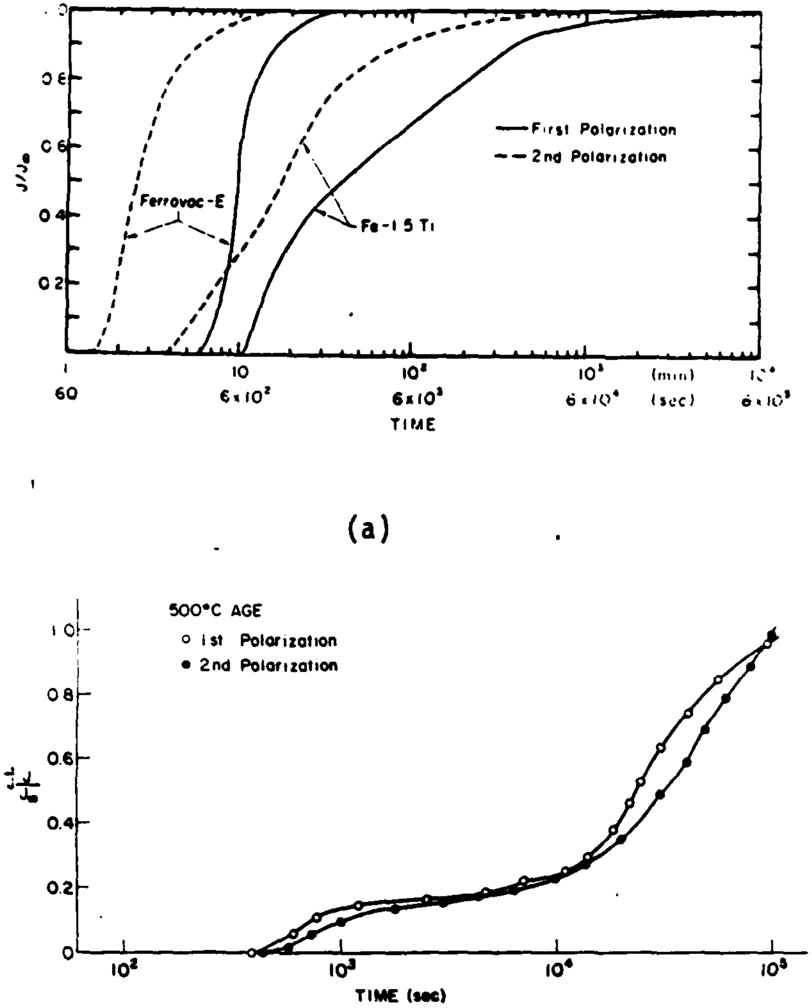
While it is likely that, in general, most heterogeneities will combine mixed characteristics, the advantage of the above characterizations is that they allow the properties of each trapping feature to be identified in terms of measurable (if as yet unknown) thermodynamic or kinetic parameters. Further, as discussed in detail elsewhere (9), the critical trapping parameters which impact on mechanical properties can often be more easily identified and analytically described. Such parameters include the ability of traps to saturate and the rate at which this can be achieved; the change in trap strength with time and the possible transformation of traps to anti-traps as a function of hydrogen concentration or deformation. The major points to be appreciated in the context of this development is that trap characteristics are not fixed properties and that the evolutionary or changing nature of traps must be a part of any attempt to model the role of microstructure on hydrogen embrittlement.

Evidence for Microstructural Trapping

There are a number of techniques available for identifying and monitoring the extent and location of hydrogen trapping. The electrochemical or gaseous permeation test can provide considerable data on trapping types and capacity and while such studies should ideally be carried out on simple model systems so that the specific trap species can be known (8,16), useful results can also be obtained in quite complex alloy systems (17,18). For example, Figure 3(a) shows consecutive transients for purified iron and an Fe-0.15% Ti alloy, while 3(b) depicts the behavior for a higher strength Fe-Ti HSLA alloy (18). In 3(a), it is seen that the first permeation transient can be considerably slower than subsequent ones both at breakthrough and during the rise to saturation. This shift has clearly been shown to be a direct result of the presence of strong, irreversible traps that fill during the first cycle and retain their hydrogen during subsequent permeation cycles. As discussed in detail elsewhere (16), the extent of such differences can be quite variable, depending on alloy content, microstructure and temperature, but such strong traps when present can impact significantly on the degree of embrittlement, both positively and negatively.

The second characteristic, seen in 3(a), and much more strongly in 3(b), is the presence of an inflection point and a considerable plateau region in the flux-time behavior. The presence of both features has been shown to likely result from irreversible and/or reversible trapping (10). The plateau itself is taken as evidence for strong reversible trapping, for conditions of a low hydrogen release rate and trap coverage. The value of both Pressouyre's (11) and Iino's (10) analyses is that they permit estimates to be made of capture and release rates and trapping energies, and permit a direct monitoring of how the trapping characteristics change with heat treatment. As will be described shortly, this approach has been successfully used to rationalize the role of a changing microstructure on the relative susceptibility of alloys to hydrogen induced embrittlement (17,18).

A newer monitoring technique for evaluating trapping and transport by mobile dislocations has been developed (19,20), making use of concurrent straining during hydrogen permeation through a membrane of material of interest. A very extensive study has recently been carried out by Hwang (20,21), who monitored hydrogen flux behavior in single crystal purified iron of special orientations, chosen so that the trapping and transport behavior of screw, edge and mixed dislocation could be separately analyzed. Some of these results are shown in Figure 4 for both screw and edge dislocations. For the former, the discrete discontinuous permeation pulses were attributable to the rapid release of a core population of hydrogen when the entire dislocation line intersected the monitored crystal surface. For the latter (as well as for the mixed case), the intersecting dislocation segments are now kinks travelling along the screw dislocation, since the screw continues to be the predominant source of strain at room temper-

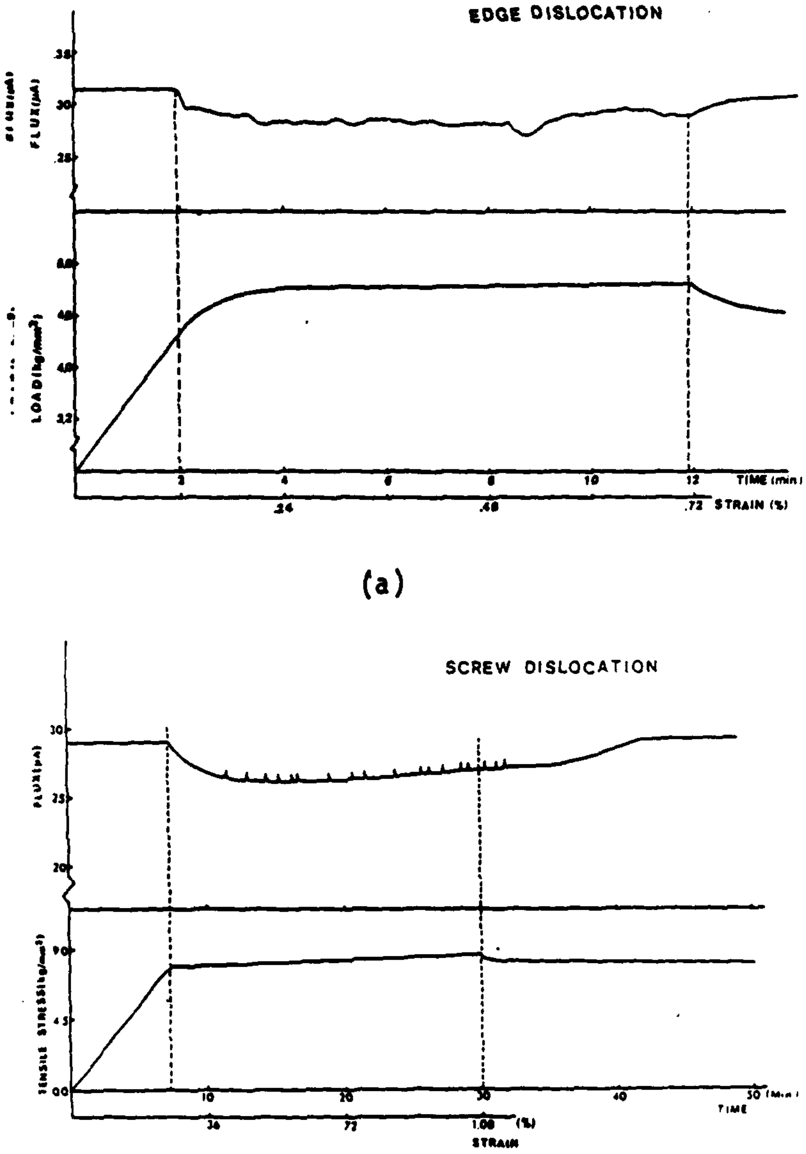


(a)

(b)

Figure 3. a) Normalized first and second permeation transients for purified iron with and without the addition of titanium. The existance of irreversible trapping and the role of substitutional alloy additions on permeability. c) Normalized first and second permeation transients through a quenched and 500°C aged Fe - 0.05C - 0.22 Ti alloy.

ature for crystals of this purity. The hydrogen flux in this case is more continuous, reflecting the high emission frequency of kinks on the monitoring surface. The strain rate dependence was also studied; at slower rates edge kinks have the greatest capacity to transport hydrogen consistent with their higher binding energy, while at higher strain rates the edge kinks can achieve a great enough velocity to break away from the hydrogen (20) and screws now dominate the transport process. These are compelling results in that they clearly demonstrate the significant contribution of dislocation transport, even in ferrous materials of high lattice diffusivity.



(a)

(b)

Figure 4. Typical changes of the steady state hydrogen flux due to concurrent deformation for:

a). edge dislocation
b). screw dislocation

Further, the different response of edges and screws may be of importance when considering hydrogen-dislocation interactions in regions like the plastic zone in the vicinity of a crack.

A final technique to mention is one that is most direct and persuasive, viz. tritium microradiography, which has clearly revealed the spectrum of hydrogen traps in bcc ferrous alloys (22,23), as well as in austenitics and other fcc alloys (24). Some typical behavior is illustrated in Figure 5, showing trapping on decorated dislocation walls in an Fe 1.5%Ti alloy (5a); on $Cr_{23}C_6$ in an Fe-9% Cr alloy (5b) and on dislocations in a 304L stainless steel (5c). Figure 6 reveals the role of strain on the extent of hydrogen trapping in a cathodically-charged spheroidized 1320 steel. Significant trapping only occurs beyond the necking strain and this has important ramification on the way hydrogen accelerates the ductile fracture process in such an alloy (25), as will be discussed. Further, this serves as clear proof that trap character can change, in this case due to strain.

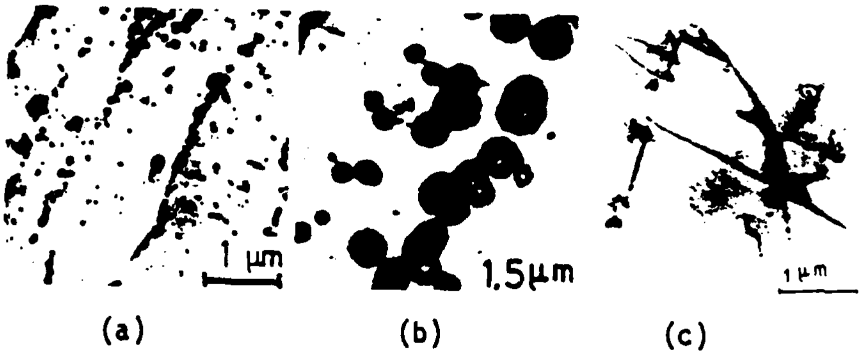


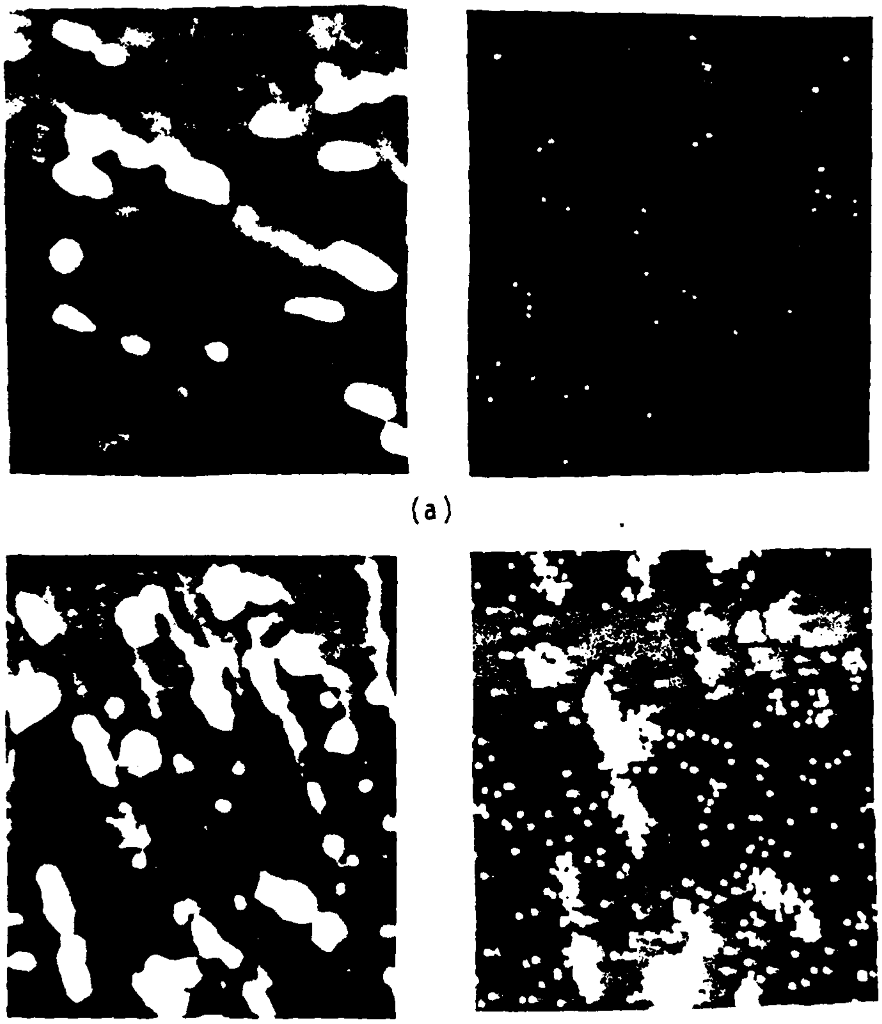
Figure 5. Trapping sites as revealed by high resolution tritium microradiography in ferritic and austenitic steels. Hydrogen revealing silver grains appear as dark dots.

a). replica of Fe - 1.5% Ti
b). replica $Cr_{23}C_6$ in Fe - 9%Cr
c). TEM of 304L stainless steel.

Effect of Trapping on Mechanical Properties

The first effect to be discussed will be how hydrogen when trapped or while being transported by mobile dislocations modifies the deformation response of ferrous alloys. This is most often considered for high purity iron, which generally is softened in the presence of hydrogen near room temperature, while being hardened at lower temperatures (26). The former effect is believed due to the enhanced mobility of screw dislocations, while the latter may be caused by hydrogen clusters, which hinder the sidewise motion of non screw kinks. The softening also leads to an enhanced stage 1 regime for properly oriented single crystals. The modifying effects of hydrogen on work hardening have also been reported (27).

Support for a strong effect of hydrogen on the character and response of screw dislocations is evident from the dislocation transport studies of Hwang (20), described earlier, as illustrated in Fig.7. This micrograph supports previous suggestions that hydrogen reduces cross slip of screw dislocations, leading to more planar arrays. Of perhaps more interest are the TEM results illustrated in Fig.8, which show the dislocation behavior in the vicinity of inert dispersoids in screw-oriented iron single crystals (20,28). It is quite apparent that straining such crystals in the contin-



(a)

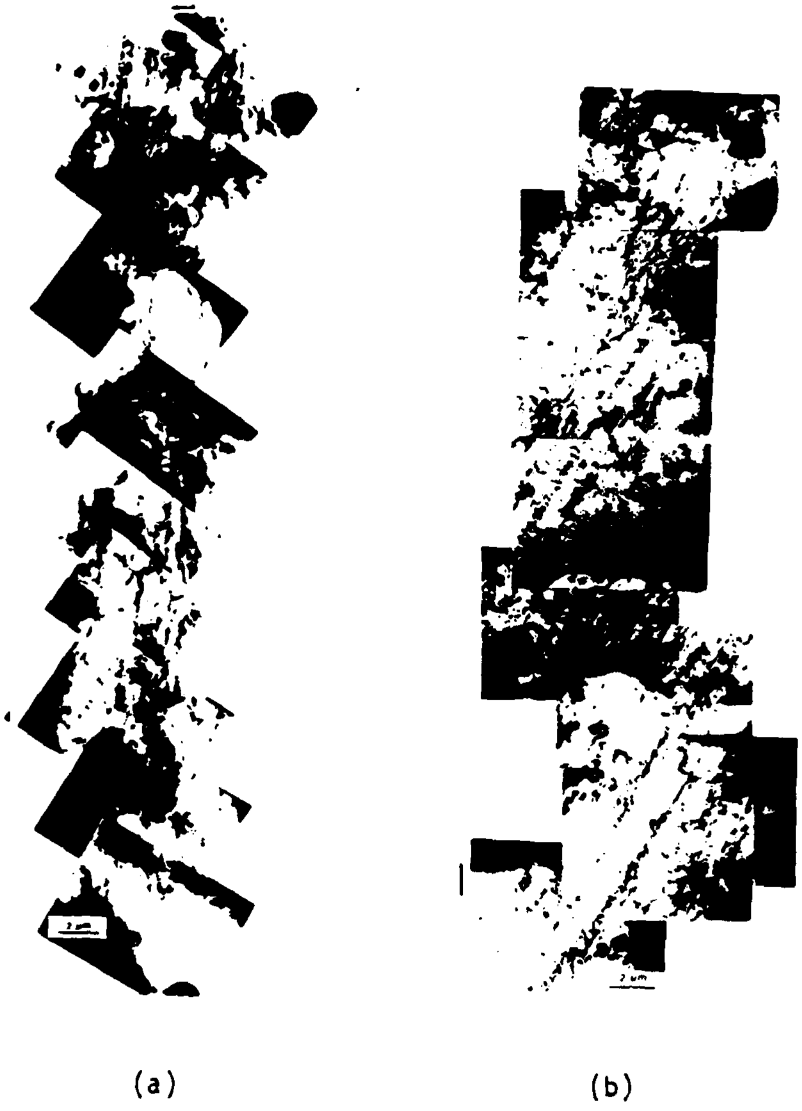
(b)

Figure 6. Trapping in the viscinity of iron carbide particles in a 1320 Mn containing steel as revealed by tritium microradiography. The extent of hydrogen trapping is revealed by white dots on SEM mapping image.

a). unstrained
b). strained beyond necking

uous presence of a cathodically-generated hydrogen environment produces a dense localized tangle of dislocations absent in hydrogen-free material. or in the rest of the hydrogen-containing sample and is akin to reported effects of hydrogen enhancing macroscopic strain localization.(3). An analysis of these results revealed that localization was associated with the presence of a third slip system,absent in the matrix, and it was suggested that the presence of an anti-twinning primary slip system catalyzed the formation and growth of strain localization (20,28). Importantly, in the same study, it was shown that the orientation of the band was identical to subsequently produced hydrogen-induced cracks.

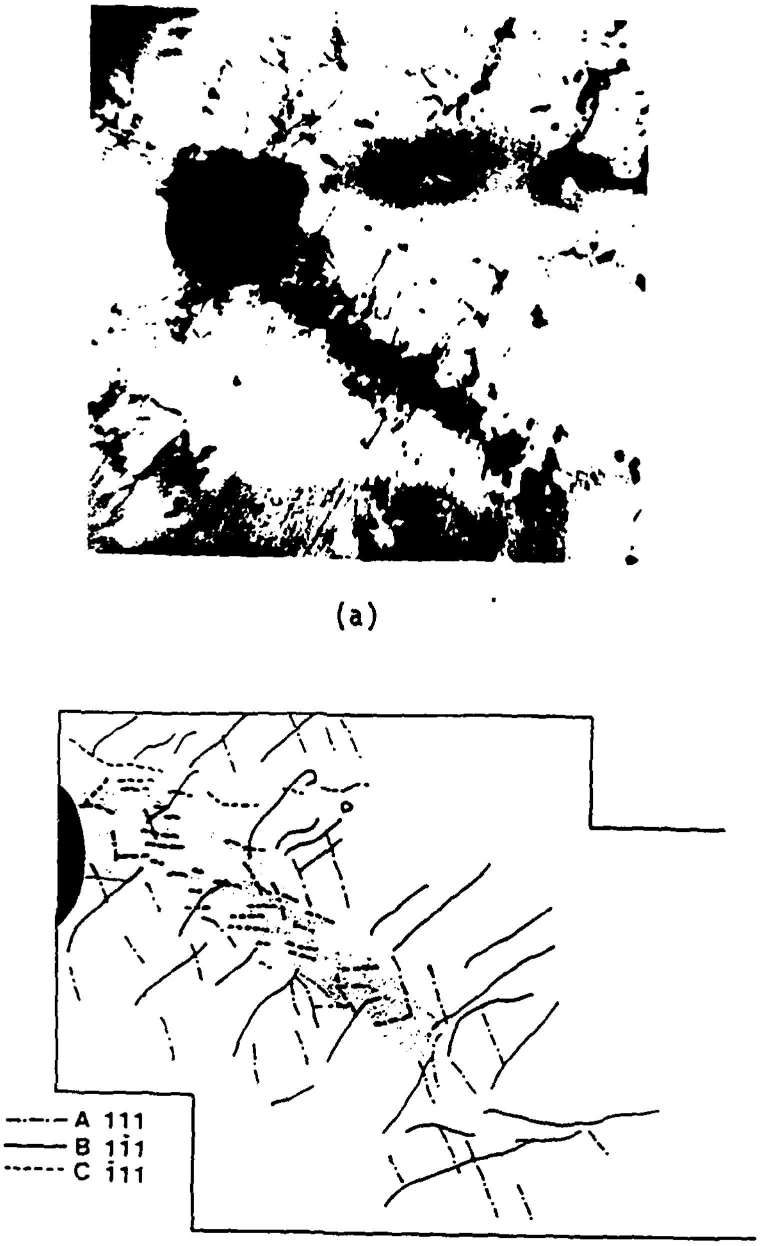
These results have now been extended to and demonstrated in a polycrystalline spheroidized 1320 steel. Fig.9 shows the early stage, at about 4% strain, and the further development of the strain localization band, at about 8% strain; in this case the bands emanate from iron carbide particles (29). Note the discontinuous nature of the band, suggestive of the appearance of hydrogen induced cracking and void growth frequently seen in



(a) (b)

Figure 7. Dislocation structure in a screw-oriented iron single crystal after about 5% easy glide strain

a). hydrogen free region
b). hydrogen containing region of the same crystal as a).



(a)

(b)

Figure 8. Hydrogen induced strain localization in the same single crystal shown in 7b).

a). TEM of localization band emanating from a silicon rich spherical particle.

b). A sketch of dislocations on different slip systems participating in the strain localization structure.

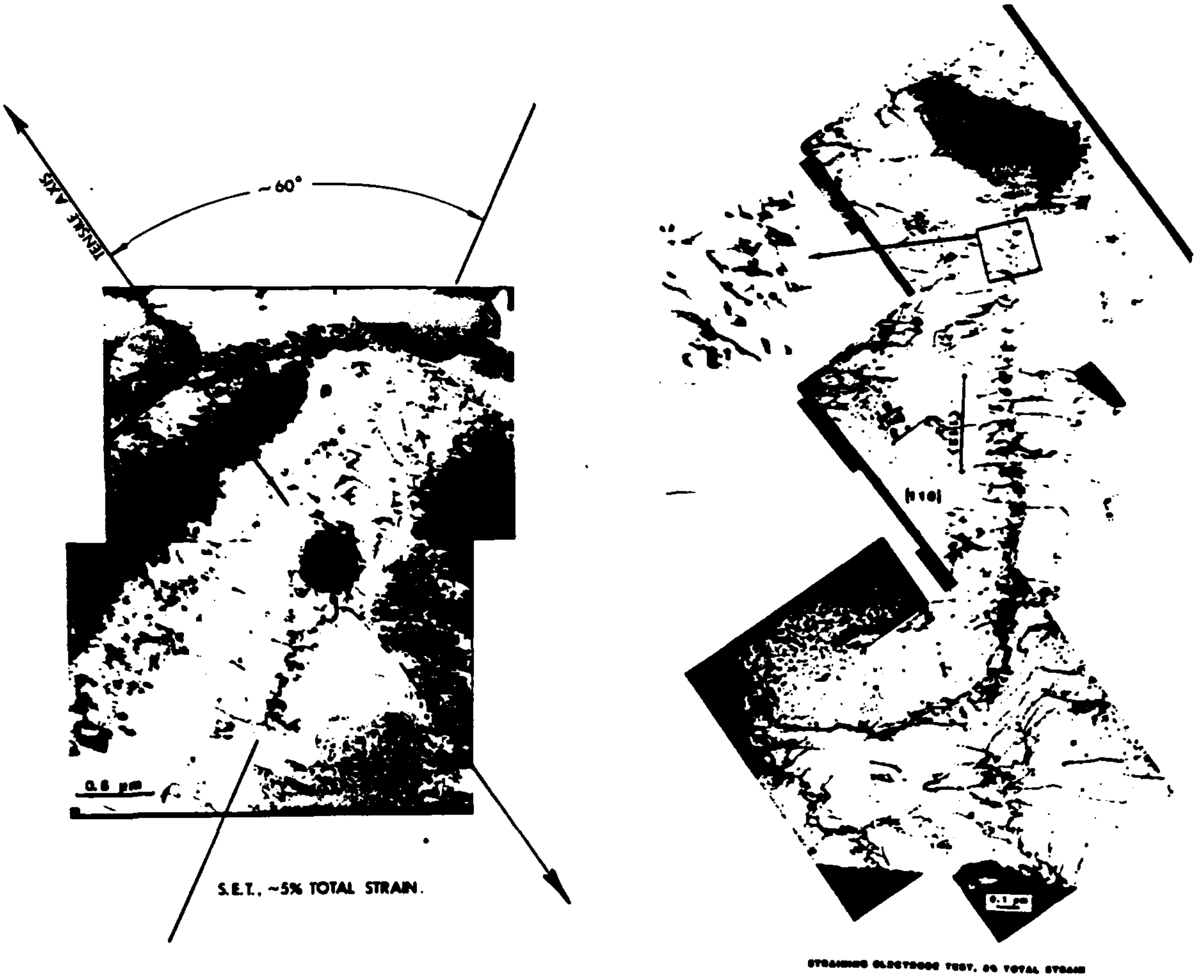


Figure 9. Strain localization in a polycrystalline spheroidized 1320 steel. The diffracting conditions were such that only 2 of the 3 operating slip systems within the band were imaged. Note the discontinuous nature of the band, particularly at lower strains.

a). 5% total strain
b). 8% total strain

steels. The importance of such observations, beyond their fundamental scientific interest, is that they serve to unify behaviors that have previously appeared poorly established or conflictual. Thus, hydrogen can be trapped at mobile and stationary defects, which themselves can mutually interact; it can be redistributed to different sites; it not only can enhance slip, but it can localize it; and each of these can combine for different sets of conditions to enhance or degrade toughness or ductility. While we are not yet in a position to generalize these events, we can use them to rationalize the hydrogen behavior of a number of different ferrous alloys.

For example, a study has recently been completed in a titanium containing low carbon HSLA steel, where the response to hydrogen and microstructure in both tensile and fracture mechanics tests was assessed (18, 30). Following quenching, the alloy was aged at successively higher temperatures, producing the following microstructural sequences: cementite nucleation at lath boundaries at about 400C; clustering of Ti(C,N) plus grain boundary cementite below 500C; preemption of cementite by Ti(C,N) formation at higher temperatures, followed at still higher temperatures by precipitate coarsening and coherency loss. The effects of these different microstructures with similar strength levels, in cathodically charged tensile and on double cantilever beam fracture mechanics specimens are summarized in Figs. 10 and 11, respectively.

The tensile results were interpreted as being strongly dependent on the transition from primary cementite to primary TiC or Ti(C,N) formation below 500°C and to the rejection of metalloids to prior austentie grain boundaries at higher temperatures. In the context of the present focus on trap types, the behavior is separable on the basis of the strength and competition between traps. When cementite is the primary microstructural constituent, the trapping is strong, but reversible in nature (see Fig.3(b)). Presumably the kinetics of detrapping is such that such sites are not efficient enough to significantly improve hydrogen response compared to the as-quenched structure. The precipitation of very fine coherent TiC particles on the other hand are strong irreversible traps whose presence significantly improved ductility. However, at still higher temperatures, P rejection from the growing carbide to grain boundaries created regions with an even stronger interaction for hydrogen than the carbides. The resultant total loss of ductility concomitant with a 100% intergranular fracture suggests that such catastrophic embrittlement requires both hydrogen and metalloids to coexist at sufficient concentrations at grain boundaries. Suprisingly, this embrittlement occurs even though a high density of irreversible, fine (and therefore presumably innocuous) traps are present in the microstructure (29). The apparent deleterious effect of P is in conflict with the prediction of Pressouyre (2,9,13) that this element should act as a hydrogen repeller, improving susceptibility by reducing the extent of segregation. These current results and others (9) suggest that P can act very differently than the behavior predicted solely from thermodynamic considerations, when present as a segregant to grain boundaries and interfaces.

A quite different microstructural response is observed when the hydrogen effect is monitored by changes in the stress intensity for initiating crack growth and/or the subsequent rate of growth (Figure 11). (The associated very discontinuous nature of the crack response is beyond the scope of this paper, but has been treated elsewhere (18).) Hydrogen embrittlement was found to be most severe for the microstructural conditions of negligible deep trapping and a high concentration of short circuit diffusion paths, while resistance was greatest in the presence of deep trapping, which produced both a lower quantity of diffusible hydrogen and slower kinetics of internal redistribution. This type of microstructural response is much more in the spirit of the trap theory of hydrogen embrittlement (8), since in the more susceptible condition, C_K, is reached more quickly, while in the more resistant condition, C_H, is significantly reduced.

The difference between these results and tensile behavior indicates the importance of kinetics in determining the usefulness of trapping to ameliorate embrittlement. In fracture mechanics type specimens, continuous deformation is not involved, reducing possible redistribution by dislocation transport. The fact that time is now a less critical variable implies that long range lattice diffusion through the matrix and the concomitant ease of redistribution in response to local stress changes should control the embrittlement process. Because such variable behavior exists, the comparison of short term tensile testing to long term hydrogen induced cracking can be quite useful as a probe to assess the influence of trapping. The fact that trapping is not effective in reducing embrittlement if the aging process itself degrades other previously innocuous traps (e.g. grain boundaries) to become preferred sites for crack initiation and propagation seems reasonable, but it is not clear why this should not be manifested also under fracture mechanics conditions.

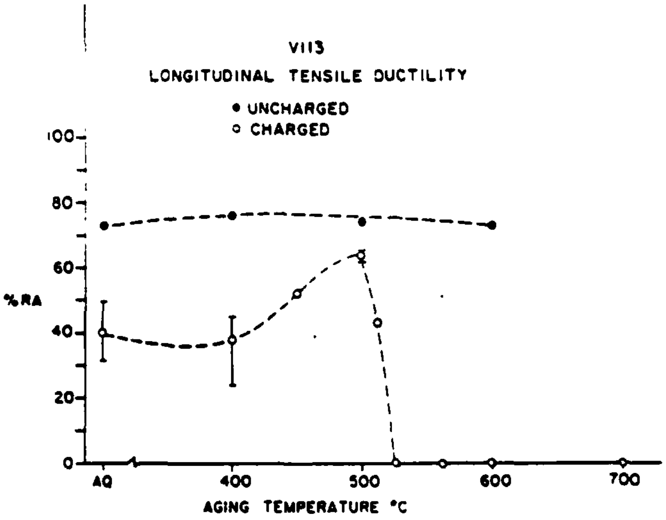
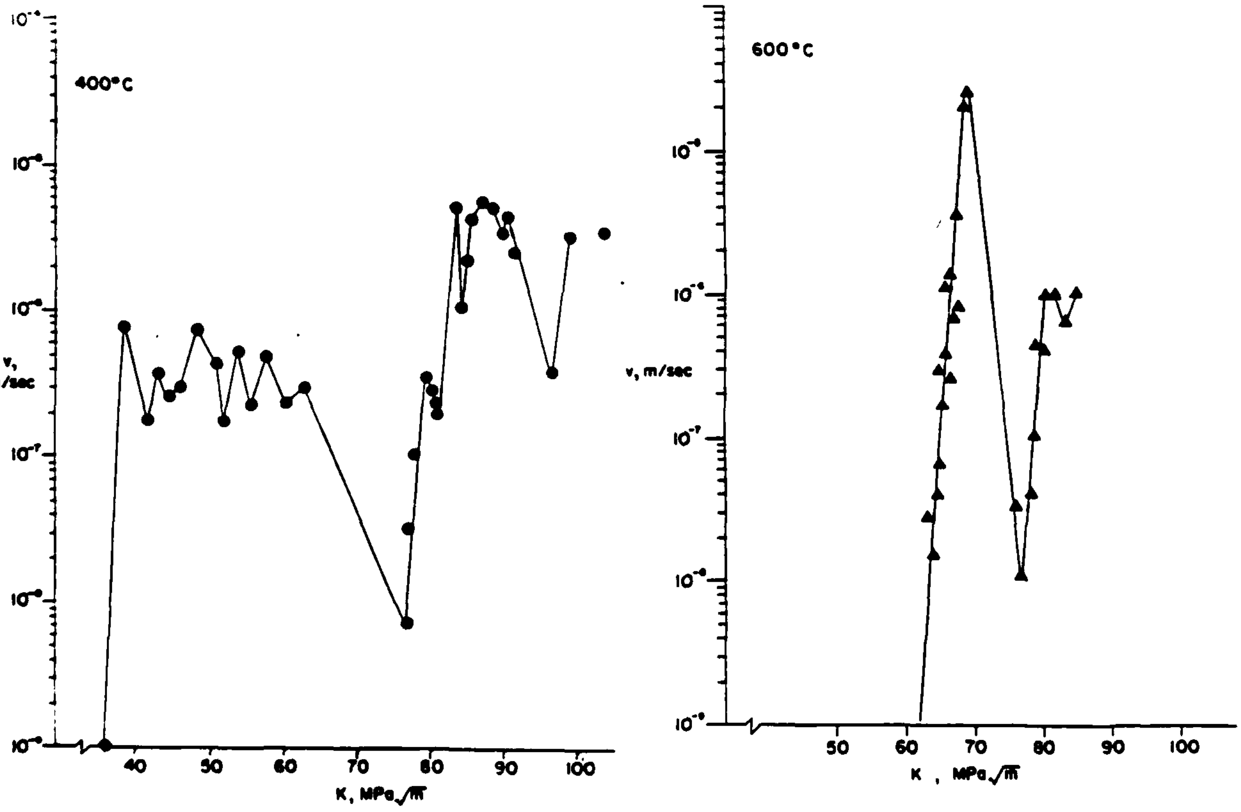


Figure 10. Comparison of internally charged and uncharged ductility response in tensile specimens of a Fe-0.05C - 0.22Ti alloy as a function of aging temperature.



(a) (b)

Figure 11. Stress intensity dependence of hydrogen induced cracking in the same alloy as Figure 10.

a). Quenched and aged 1 hr at 400°C
b). Quenched and aged 1 hr at 600°C

The final example to be presented is the hydrogen response of HY130 steel, a low carbon "clean" steel containing about 5% nickel. We have previously shown, as illustrated in Figure 12, that heat treatment can have a dramatic effect on the threshold and crack growth rate under conditions where hydrogen is continuously being introduced from aqueous solution (17). A very detailed characterization of the microstructure was directed primarily at identifying the possible contribution of a varying amount of retained austentite. For this steel, the retained austenite varied from about 3% to 10% and was mechanically unstable, converting to untempered martensite at strains of the order of those found in the plastic zone in the vicinity of the crack tip (17,30). The increasing presence of such austenite was found to be beneficial and could be correlated with the energy consumed in the transformation, which reduced the mechanical driving force for crack advance (30). The threshold stress in turn scaled with the apparent solubility of hydrogen, derived from an analysis of permeation transients (12). The solubility itself increased with the initial level of retained austenite, suggesting that the presence of the fcc phase is beneficial, particularly at larger concentrations where there is a greater probability of it being present near the crack and in the plastic zone. In terms of trap theory, the retained austenite acts as a solubility trap and a diffusion obstacle, with the net result being a reduced C_H. However, retained austenite need not be always beneficial. In fact, in cases where the austenite is mechanically stable and is present as an interlath film, it was shown to degrade hydrogen performance in 300 M, a silicon modified 4340 steel (17).

Summary

The evidence continues to mount persuasively that the hydrogen performance of ferrous (and likely many non-ferrous alloys) is controlled by the distribution and redistribution of hydrogen to heterogeneities. These can be innocuous traps which, if fine enough and plentiful enough, can reduce C_H. Alternatively these sites can be localized precursors to crack or void formation and can lead to a reduced C_K, if hydrogen's presence accelerates the onset of localized plasticity. As is always necessary when analyzing such a complex subject, some cautionary notes are required. Stress state and testing technique variations can modify expected performance, as can unexpected synergistic interactions between trap features which independently can be beneficial. Nevertheless, the picture which has developed of hydrogen-related trapping and changes in dislocation response, lends credence to an earlier claim that alloy design, through microstructure and compositional control, to improve hydrogen embrittlement resistance is both feasible and attainable (7).

Acknowledgements

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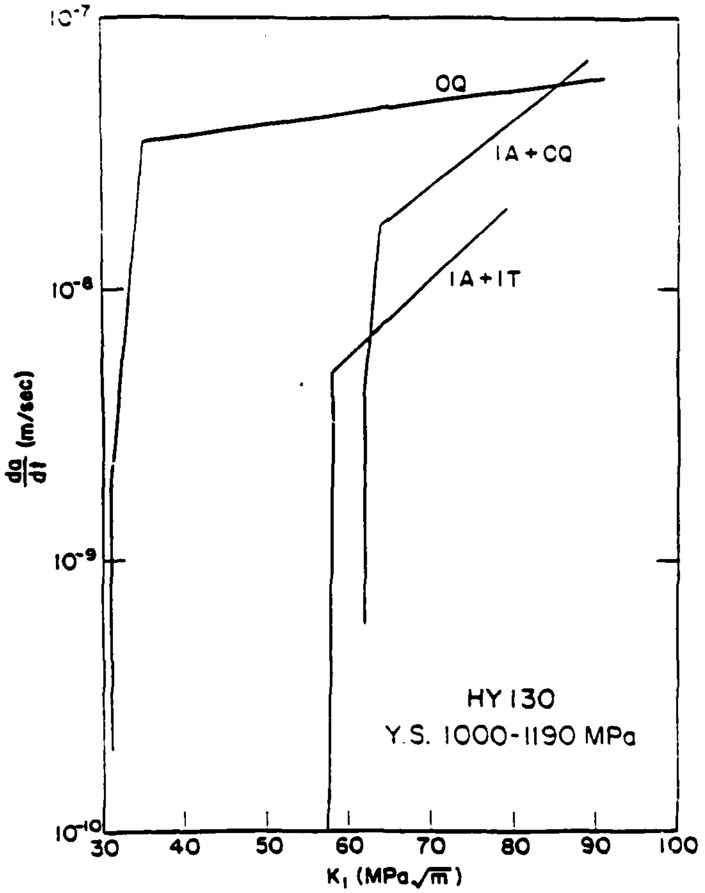

Figure 12. The effect heat treatment on stress corrosion crack initiation and growth in a .09C 5Ni HY 130 steel tested in 3% NaCl with zinc coupling to increase the hydrogen activity. The symbols designate the following heat treatments

OQ: Austenitized 1 hr. at 900°C, oil quench
IA + OQ: OQ treatment + 1 hr. at 675°C, oil quench
IA + IT: OQ treatment + 1 hr. at 675°C, isothermally transformed 3 hrs. at 375°C.

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)
The evidence for trapping by mobile and stationary defects in ferrous alloys is reviewed and new results presented to demonstrate that the trap theory of hydrogen embrittlement can rationalize the many different ways by which hydrogen modifies subsequent mechanical properties.